

Improving the Performance and Sustainability of Additively Manufactured AlSi7Mg

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Statement of Originality

This is to certify that to the best of my knowledge, the content of this thesis is my own work. This thesis has not been submitted for any degree or other purposes.

I certify that the intellectual content of this thesis is the product of my own work and that all the assistance received in preparing this thesis and sources have been acknowledged.

James H. Warner

Authorship Attribution Statement

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With the support of my supervisors, I designed the study, collected and analysed the data, wrote the manuscript, and implemented any corrections as suggested by co-authors and reviewers.

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With the support of my co-authors, I designed the study, collected and analysed data, wrote the manuscript, and implemented any corrections as suggested by co-authors and reviewers. C.V. Funch collected EDS data using SEM and TEM and wrote the results section on these experiments. K. Sisco collected and analysed data using ThermoCalc simulation and APT and wrote the results section on these experiments.

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Abstract

Additive manufacturing represents an increasingly popular group of technologies that are being used to manufacture components in a way that reduces material waste while allowing for the simple customisation of complex, bespoke parts. Powder bed fusion (PBF) is one such technology used primarily for the processing of metals, where the mechanisms involved create significant potential for property control by the manipulation of processing parameters. The popularity of aluminium alloys in a wide range of applications has naturally resulted in its adoption as one of the most popular alloy classes for PBF. Given the excellent processability of aluminium-silicon alloys during PBF manufacturing these have seen some of the most attention, with AlSi7Mg one of the more popular.

While PBF technology has seen significant development in recent times, the complexities involved means that it has not yet reached full maturation. Scores of processing parameters are relevant to the properties of parts manufactured by PBF, with great focus having already been placed on laser parameters. This leaves many significant parameters yet to be fully elucidated and therefore requiring significant investigative work, with the location of parts within the build volume one such parameter. Heat treatments are also commonly applied to additively manufactured parts both to relieve residual stresses and improve properties, however, these are not often tailored for additively manufactured components. The nature of the powder spreading inherent to PBF techniques also raises the question of powder reuse. Given significant quantities of powder remain unmelted after processing, how this powder is reused will influence both the behaviour of the powder in the build chamber and the mechanical properties of parts manufactured in subsequent builds.

The aim of this work is to improve the understanding of each of these factors on the properties of additively manufactured AlSi7Mg, by investigating the effects of part placement within the build volume, tailoring heat treatments for additively manufactured AlSi7Mg, and

developing a robust powder reuse method justified by more than just ease of powder handling. In combination, these investigations will improve both the part performance and sustainability of this additively manufactured alloy.

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CHAPTER 1

Introduction

Aluminium is the most commonly used non-ferrous metal in the world due to its combination of properties including low density, excellent strength to weight ratio, good corrosion resistance, and excellent conductivity. However, as pure aluminium is generally a soft and ductile material, alloying is common. This has resulted in the creation of a number of alloy classes each with varying elemental additions commonly including copper, manganese, silicon, magnesium, and zinc. The addition of these elements can variously improve the hardenability, weldability, corrosion resistance, and strength of aluminium, enabling the use of these alloys in a wide variety of applications such as in the aerospace, aeronautical, and automotive industries.

The manufacture of components for applications in these industries is often complex; freedom in design is required for proper part optimisation which for many traditional manufacturing methods is difficult or results in large amounts of waste. The recent rise in popularity of additive manufacturing has provided new manufacturing technologies enabling the production of geometrically complex components while reducing waste. Additive manufacturing encompasses a number of technologies that produce parts by the addition of material, rather than traditional subtraction, to build up a component layer by layer according to a computer designed model. This allows for the manufacture of complex geometries otherwise impossible to produce by traditional manufacturing methods, and with alterations to part geometry resulting in minimal changes to part cost [1], also allows for the simple customisation of parts. Furthermore, many components manufactured by additive manufacturing have the potential to see savings in time, energy, and material usage, reducing the cost per part in comparison to traditional manufacturing techniques [2]. For these reasons, additive manufacturing continues to grow in popularity.

This disruptive technology is being used to manufacture components in a variety of materials, with metals becoming increasingly common. This is primarily done by powder bed fusion (PBF), where a thin layer of powder is spread before being melted to shape by either a laser or electron beam, with the continued cycle of powder deposition and melting gradually building a part. Reuse of the powder from this manufacturing process can further increase material utilisation and thereby continue to reduce the cost of parts manufactured by this technique [3].

While the majority of aluminium parts continue to be produced by traditional manufacturing techniques, the increasing popularity of additive manufacturing has resulted in aluminium becoming one of the most popular alloy classes for PBF alongside titanium alloys, steels, and nickel-based alloys. Aluminium does, however, come with a number of difficulties in processing as its high reflectivity and conductivity reduces the laser absorbance of powders, resulting in poor melting and solidification cracking of many aluminium alloys manufactured with PBF [4, 5]. For this reason, aluminium-silicon alloys have received some of the most attention within the literature, as the inclusion of silicon reduces the temperature range for solidification and therefore reduces the solidification cracking of the alloy, improving its processability. Research initially focused on AlSi12 and AlSi10Mg alloys due to their success in casting brought about by their proximity to the Al-Si eutectic point and correspondingly narrow solidification window [6]. However, new aluminium alloys are continuing to be adapted and developed for PBF processes, with one such alloy being AlSi7Mg, especially popular for aerospace and automotive applications due to its improved performance at high temperatures.

While the processability of AlSi7Mg is generally understood, there remain many ways to improve its performance when producing additively manufactured parts. This is as the number of processing parameters involved in PBF is immense with over 100 commonly cited in the literature [7–9], with the most commonly considered being laser parameters such as laser power, scan speed, and hatch spacing [8]. This leaves a considerable amount of research as of yet untouched with great potential to improve the properties of additively manufactured parts, with AlSi7Mg being no exception to this. This thesis therefore aims to investigate a number of ways to improve the performance of additively manufactured PBF by increasing the understanding of a number of processing parameters, as well as to improve its sustainability by better understanding the effects of powder reuse in PBF.

A detailed review of the literature is included in Chapter 2. This includes the introduction of additive manufacturing with a focus on the processing of metals using powder bed fusion. Focus then shifts to the processing of aluminium, particularly aluminium-silicon alloys. A review of their processability, typical microstructures, mechanical properties, and heat treatment routes are included.

Chapter 3 involves a detailed literature review regarding powder reuse within powder bed fusion processes, with an emphasis on the attributes of different powder reuse methods. This includes the introduction of common powder characterisation and mechanical property investigation methods, in particular light of their use in the powder reuse literature. An evaluation of the most common powder reuse methods is presented, along with a comparison to determine their relative worth.

The experimental methods and workflows used in this thesis are included in Chapter 4. This includes detail on the sample production, heat treatment, powder characterisation, mechanical property testing, and microscopy tests that were conducted for this research.

In Chapter 5, an investigation on the effects of different location parameters within the PBF build chamber are investigated. These are the location of the part on the build plate, its distance from the build plate, and its orientation, size, and surface finish. Mechanical testing and microstructural investigations are conducted in order to show that, unlike what is presented in much of the literature, these parameters are of enough importance to necessitate consideration during the manufacture of PBF components.

Chapter 6 investigates the heat treatment of AlSi7Mg manufactured with PBF, particularly the effects of ageing and changing solution treatment temperature prior to ageing. The proper characterisation of the microstructure and solute distribution during these processes allows for a genuine understanding of ageing behaviour, and the optimisation of heat treatment routes for PBF AlSi7Mg.

The refreshing powder reuse method is examined in detail in Chapter 7, investigating changes in powder characteristics, part properties, microstructure, and mechanical properties. With a unique focus on the powder reuse method used, this allows for a proper understanding of how the characteristics particular to this powder reuse method will change part properties.

Chapter 8 then presents a brief look at the single batch powder reuse method, with the aim of comparing this to the refreshing powder reuse method as presented in Chapter 7. Given identical tests are used in both chapters, a true comparison of these powder reuse methods can be presented for the first time. While this comparison is made using AlSi7Mg, the principles can be extended to any alloy system utilising reused powder in PBF applications.

To conclude, key highlights of these findings are presented in Chapter 9. Also included are proposals for further investigations into AlSi7Mg manufactured by PBF.

Literature Review on the Additive Manufacturing of Aluminium

2.1 Introduction to Additive Manufacturing

Additive manufacturing relies on the continual deposition of material to create a functional part, in comparison to more traditional manufacturing methods which are primarily subtractive. This results in a reduction of waste when manufacturing parts additively [10], which has helped to push the development and popularity of these new methods. Additive manufacturing uses this material deposition to produce three-dimensional objects from computer designed models, either in production of a new part or in the repair of existing items [11]. This deposition happens layer by layer to gradually build a part, leading to the technology also being called layer technology, alongside other names such as rapid prototyping or 3D printing [12].

Additive manufacturing began as a method of rapid prototyping due to its speed in moving from prototype design to production due to the high degree of available customisation, a significant advantage over traditional manufacturing techniques such as casting. Additive technologies also allow for the production of geometrically complex parts, allowing for simple conversion from design idea to replicable product [13]. Manufacturing runs for additively manufactured parts are typically of low volume and high complexity, where the unique advantages of these techniques are most fully realised [14]. These include the production of aeroplane and automobile parts due to the capability of producing geometrically optimised lightweight parts, as well as the manufacture of fuel cells due to the precise deposition of very thin layers of material [15]. The high level of customisation available with these techniques also means that they are commonly used in medical professions, especially for customised dental and surgical implants, with the ability to still meet relevant industry standards [14, 16].

There are many techniques and materials that are currently being used in a variety of additive manufacturing technologies. The main categories of materials in widespread use are polymers, metals, and ceramics, however composite and functionally graded materials are also used [17]. This variety allows for the development of a range of products for numerous applications. This is aided by the significant number of additive manufacturing techniques available, each of which is typically used for only some materials. These techniques are classified according to their material input requirements which are either filaments, powders, or sheets. Some examples of common additive manufacturing techniques are stereolithography, fused deposition modelling, directed energy deposition, powder bed fusion, sheet lamination, and binder jetting [13].

Metallic processes are some of the most prominent aspects of additive manufacturing being currently investigated [18], however, with the continual uptake of additively manufactured metallic products, a greater understanding of both their processes and properties are required. This review will focus on metal additive manufacturing, with a particular focus on aluminium-silicon alloy systems.

2.2 Metallic Additive Manufacturing

Given the popularity of metals in additive manufacturing a range of metals and alloys are now being used, especially titanium alloys, nickel alloys, aluminium alloys, and steels, with steels accounting for approximately one third of academic publications [19]. The compositions of these alloys are typically designed for high weldability and castability in order to improve their processability, with many alloy compositions being taken directly from casting [20, 21]. As such, there is also continual work on the development of new alloys for use specifically in the field of additive manufacturing [22].

Manufacturing metals using additive techniques primarily focuses on optimising density to maximise properties [23], and as such considerable research has focused on maximising density [24–27]. This has then flowed onto work in optimising part properties by a variety of means, including the manipulation of input material characteristics [28–31], processing parameters [32–35], and post processing treatments [36–39]. A brief overview of the state of these topics, among others, is provided in the following sections.

2.2.1 Metal Additive Manufacturing Techniques

A variety of techniques are available to process metals additively, the most common being directed energy deposition (DED) and powder bed fusion (PBF), which can be further split between laser (LPBF) and electron beam based energy sources [40]. These common manufacturing systems can be further subdivided into powder or wire based systems, depending on their material feedstock [41].

2.2.1.1 Direct Energy Deposition

DED systems incorporate the direction of energy to generate a melt pool at the point where a feedstock is deposited, allowing for the gradual build-up of material layer by layer, essentially an extension of welding technology [42]. This grants much more flexibility than other systems due to the changing deposition location. These systems come with a range of variations, with the power source being either a laser or electron beam, the feedstock material either powder or wire, and either the part or deposition source moving while the other remains stationary [43]. Examples of some of these systems are shown in Figure 2.1. DED lends itself to larger parts than other metallic additive manufacturing systems with considerably faster deposition rates and larger possible build volumes, at the cost of lower resolution. It is also useful for the repair and retrofitting of parts due to the manoeuvrability of the deposition head [44].

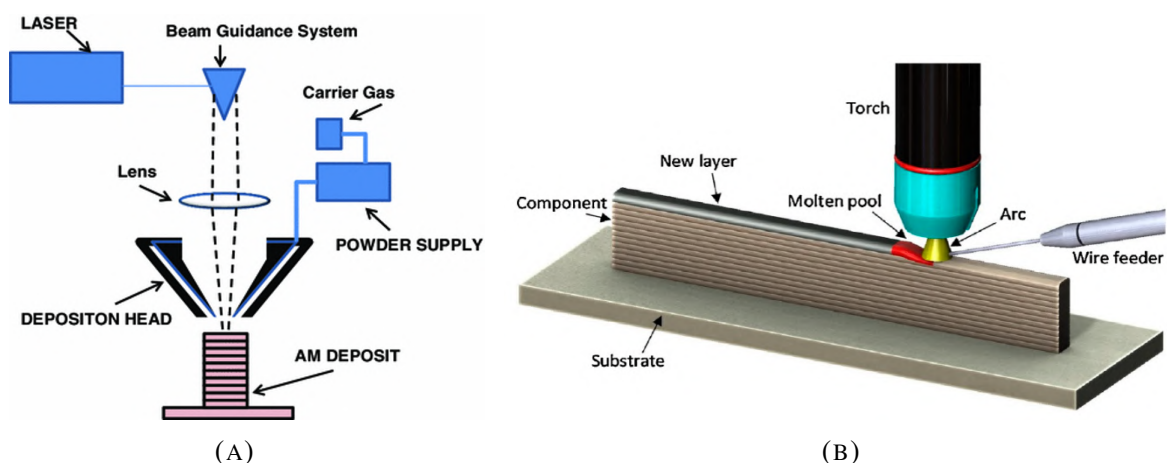


FIGURE 2.1. (a) Illustration of powder fed DED system (reproduced from [43]), (b) Illustration of wire fed DED system (reproduced from [45]).

2.2.1.2 Powder Bed Fusion

Powder bed fusion is the other main method for metallic additive manufacturing, involving the repetitive spreading of a thin layer of powder before the selective melting of layers using a focused, high-energy source to melt the required part to shape. A variety of specific PBF techniques have been developed, the most significant being selective laser melting (SLM) and electron beam melting (EBM). The main difference between these techniques is the energy input source with both SLM and EBM requiring high energies to melt powder into the desired shape and produce parts with high densities, by using lasers and a focused beam of electrons as their energy sources, respectively. The fine particle size and thin layers spread allow for high resolution using these techniques, with layers typically being in the range of tens of microns thick. While allowing for high resolution parts, PBF techniques typically have smaller build volumes and higher build times than DED. Due to its popularity and relevance to the work carried out in this thesis, the remainder of this review will focus on powder bed fusion techniques. A schematic of laser powder bed fusion is shown in Figure 2.2.

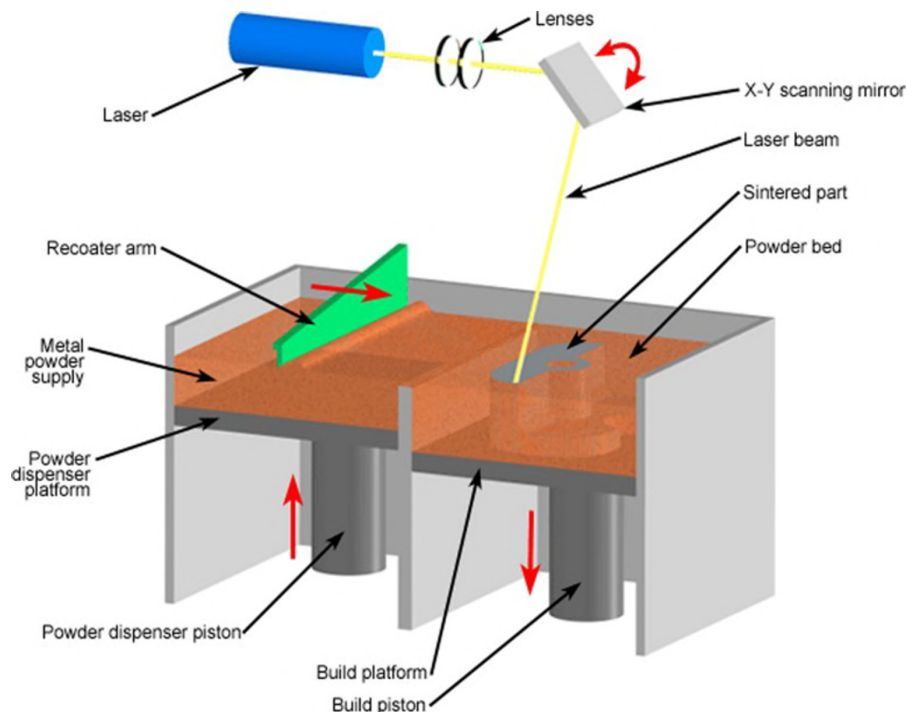


FIGURE 2.2. Illustration of a laser-based powder bed fusion system (reproduced from [42]).

2.2.2 Feedstock Materials

The varying natures of metallic additive manufacturing techniques means that different material inputs are required, the most common being wire or powder feedstock. The quality of material input has been seen to affect properties such as part chemistry, yield strength, fatigue strength and density [46–49].

There has been considerable focus on powders due to their use in both PBF and DED. This work begins with investigation into the production process of powders, typically the atomisation method used and the process parameters involved. Water atomisation is known to produce relatively poor quality powder compared to other atomisation methods due to their high oxygen contents and irregular morphology [50–52], while plasma atomisation produces the best properties due to the particles high sphericity and low porosity [28]. Gas atomisation is the most common method for production due to its good balance between powder properties and production costs [51]. It has been seen that a manipulation of parameters such as gas type, pressure or nozzle shape, among others, have the ability to change powder properties in each of these methods [53–56].

Further research has investigated how these powder properties affect the manufacturing process itself, with special attention paid to how powders flow in PBF. It is known that larger particles and narrower particle size distributions have improved flowability [57–60], although this effect does not always lead to an increase in part density [60, 61]. Powder morphology also has been seen to change other mechanical properties such as hardness, tensile and fatigue strength [62, 63], however this is beyond the scope of this review. Changes in powder oxygen content also influence the yield and tensile strengths of materials, although these properties can be either improved or reduced depending on the material [59, 64–67]. While the influence of powder oxygen content is somewhat understood, the majority of the literature remains focused on the role of particle size and morphology over any other powder characteristic [60, 61, 68], with current research continuing this trend [69–73]. This leaves considerable room for further investigation into more novel fields such as adjusting powder microstructure and chemistry for an improved manipulation of part properties.

The question of the influence of powders has led to the issue of powder reuse, especially regarding whether reused powder will influence part properties and whether this will have a

positive or negative effect. The literature regarding this research question is later reviewed in Chapter 3.

2.2.3 Process Parameters

Process parameters in PBF have been the focus of intensive research in the literature, with a special focus on laser parameters which are considered particularly important [73, 74]. The optimisation of these parameters is typically carried out to improve part density and mechanical properties [34, 75], as well as to minimise the amount of laser energy input to create a safer and more energy efficient process [76].

Laser power is one of the most commonly manipulated parameters in LPBF and as such is one of the most researched parameters. This is often in pursuit of high-density parts, and in general higher laser powers result in higher density parts [34, 77] although this is highly reliant on the type and number of defects produced, with alloy composition also playing a role. Laser power has been further linked to other properties such as surface roughness [34] and part chemistry [75]. Similarly, parameters such as laser scan speed, layer thickness and hatch spacing have each been seen to influence factors such as build time [78] melt pool geometry [79], and part geometry [80], resulting in changes to tensile strength [81] and porosity [82, 83].

While these parameters have been extensively studied due to their relative importance, there remain dozens of other parameters that also play a role in the properties of PBF manufactured parts [8, 9]. An example is the location of a part on a build plate, which is typically determined by ease of production rather than any meaningful consideration of properties [84]. Due to factors that vary with build plate location such as changing laser focal length [85], gas flow direction and velocity, and powder recoating inconsistencies, it has been demonstrated that the location of a part on a build plate may influence its properties. Due to the current comparative lack of research surrounding this parameter, among others, further research is needed to ensure a more holistic understanding of the powder bed fusion process.

2.2.4 Material Microstructures

Powder bed fusion is defined by the slow deposition of material with strong thermal gradients, high cooling rates, and varying thermal cycles, resulting in complex microstructures [86, 87]. These microstructures are typically different from those of the same materials manufactured using other methods; Figure 2.3 shows the different microstructures produced by casting and PBF of AlSi10Mg. Microstructures of PBF components tend to be characterised by fish-scale like melt pool boundaries typically visible by optical microscopy, as in Figure 2.4a, each containing fine grains oriented by directional solidification by heat flow [87], as in Figure 2.4b. Grain nucleation is enhanced at the borders of melt pools where the molten liquid meets the previously solidified material, meaning grains continue to grow towards the centre of the melt pool along the direction of heat. The repeated heating and cooling of these layers by the continual passing of the laser means grains continue to grow along the build direction, forming a columnar microstructure [88].

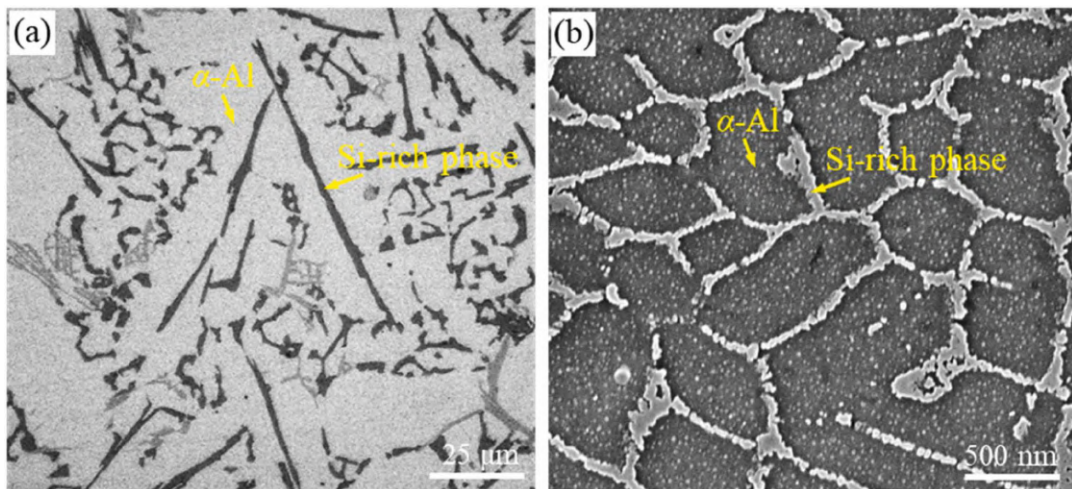


FIGURE 2.3. Comparison of AlSi10Mg from different manufacturing methods: (a) as cast (b) as-built LPBF (reproduced from [79]).

The extent of grain refinement within PBF produced materials is dependant primarily on cooling rates, with high cooling rates producing fine grains [6]. Since the cooling rate is dependent on the energy input, dictated by parameters such as laser power and scan speed, this allows for the manipulation of grain size by parameter modification. For example, a high laser power and low scan speed will increase the energy input into the powder bed and increase the laser-material interaction time, thereby increasing the temperature of the

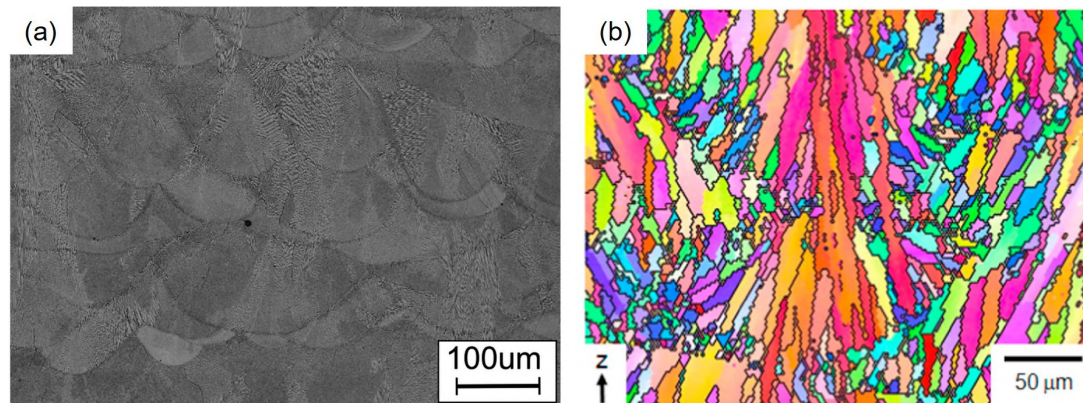


FIGURE 2.4. Typical microstructures of PBF parts: (a) fish-scale like melt pools, (reproduced from [89]), (b) typical grain structure within these melt pools (reproduced from [90]).

melt pool and increasing the solidification time. This creates a lower temperature gradient, resulting in a coarse microstructure [6]. The inverse is also true of a low laser power and high scan speed. This provides for an interesting area of potential research given the correlation between microstructure and material strength.

Within the main grains are cellular structures formed by the rapidly moving solidification front rejecting some elements at the solidification interface [87, 91], as shown in Figure 2.3b. This is especially noticeable in aluminium-silicon alloys, where the silicon forms cell walls surrounding an aluminium matrix interior. These high cooling rates also tend to create metastable phases [87] and so phases such as martensite are often formed [88]. These factors mean that PBF creates unique microstructures that must be carefully understood for parts to be properly used. This also allows for the tailoring of microstructure to customize material properties by controlling process parameters [92].

2.2.5 Mechanical Properties

Generally, PBF parts have high residual stresses due to their rapid solidification and so tend to have relatively higher strengths when compared to other manufacturing methods. This strength is further enhanced by the fine microstructures [93], however, is at the cost of ductility, which tends to be lower than that of traditionally manufactured parts.

Mechanical anisotropy is common due to the columnar texture of grains [94], however it is also influenced by the size, shape, and orientation of pores, particularly keyhole and lack of fusion defects [93]. An example of such anisotropy is shown in Figure 2.5 and Table 2.1, which show the results of the work by Suryawanshi et al. [94] on AlSi12 where it is evident that the orientation of specimens influences the properties of PBF materials. The yield and ultimate strengths of each condition showed minimal change depending on orientation; however, the elongation was considerably affected in each case tested. The melt pool boundaries were also seen to increase the tortuosity of the crack paths thereby increasing the material's fracture toughness, especially in vertically oriented samples. This indicates that the level of anisotropy is dependent on the property tested and is likely reliant on defects such as pores or poor surface quality.

TABLE 2.1. Summary of mechanical properties of PBF AlSi12 processed either as: AS (as processed), HS (heat treated), or CS (as processed with checkerboard hatch style). Samples were loaded parallel ($\parallel$) to the laser tracks or perpendicular ($\perp$) to the laser tracks (adapted from [94]).

Test Condition	Yield Strength (MPa)	Tensile Strength (MPa)	Ductility (%)	K_q (MPa $\sqrt{m}$)
AS $\parallel$	270.1 $\pm$ 10	325 $\pm$ 20	4.4 $\pm$ 0.7	46.7
AS $\perp$	274.8 $\pm$ 8	296.1 $\pm$ 20	2.2 $\pm$ 0.3	37.9
HS $\parallel$	153.4 $\pm$ 5	228 $\pm$ 13	5.3 $\pm$ 0.7	21.7
HS $\perp$	150.3 $\pm$ 17	210.1 $\pm$ 20	4.2 $\pm$ 0.3	19.3
CS $\parallel$	262.4 $\pm$ 17	330.7 $\pm$ 15	3.9 $\pm$ 0.6	47.0
CS $\perp$	276.6 $\pm$ 15	302.7 $\pm$ 15	2.3 $\pm$ 0.3	34.5

While anisotropy with respect to part orientation is the most commonly presented justification for the variation in part properties present in PBF parts, there are many other reasons that are beginning to be investigated. These include the distance of a part from the build platform [95–97], the location of a part on the build platform [96, 98, 99], build platform preheating [100, 101], specimen size [102], inter layer time [97], as well as more traditional reasons such as atmospheric oxygen and moisture content [96, 102], and energy input [95, 97]. These factors tend to vary significantly between materials, processing parameters, and PBF machines, and so deserve considerable research in order to develop a full understanding of their impacts on PBF parts' mechanical properties as well as the reasons behind these impacts.

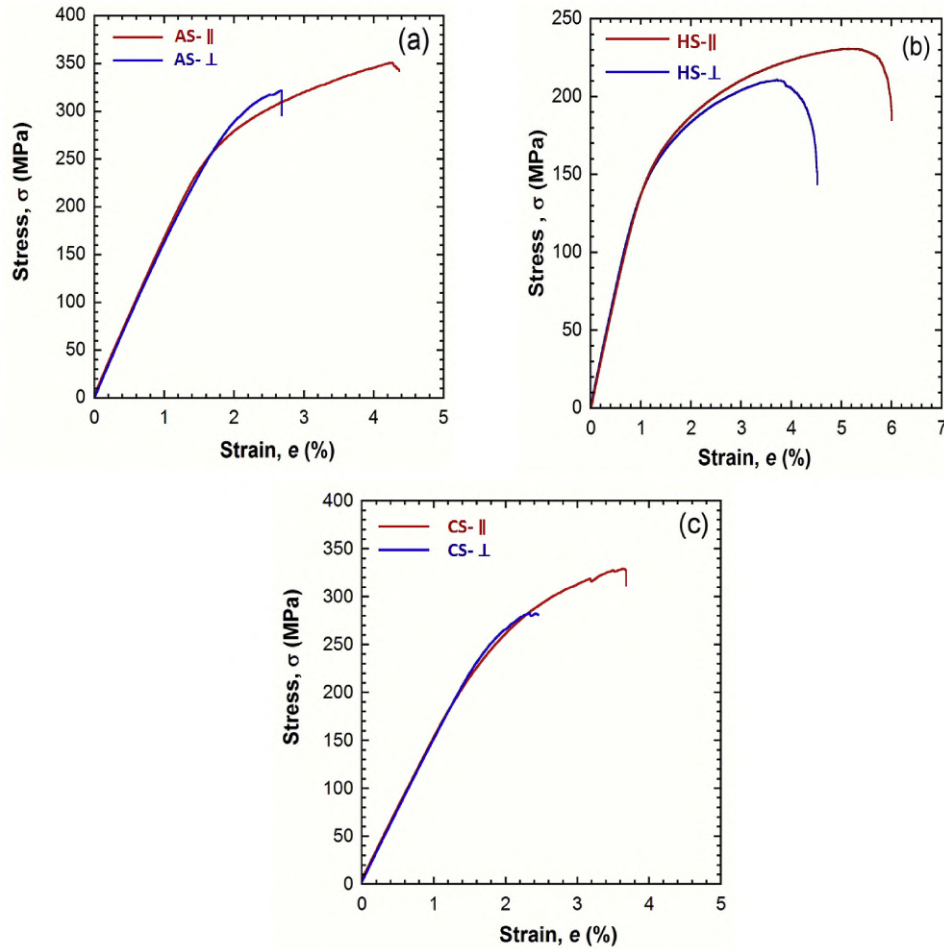


FIGURE 2.5. Representative stress-strain curves of AlSi12 in different conditions: (a) AS (as processed) (b) HS (heat treated) (c) CS (as processed with checkerboard hatch style). Samples were loaded parallel ($\parallel$) to the build direction (vertically oriented samples) or perpendicular ($\perp$) to the build direction (horizontally oriented samples) (reproduced from [94]).

2.3 Powder Bed Fusion of Aluminium Alloys

The growth of additive manufacturing has resulted in the diversification of materials available for production by these methods, and powder bed fusion is no exception to this. Aluminium alloys are popular in a range of industries due to their low density, good corrosion resistance, high strength, and good hardenability [93]. This has naturally led to their adaption in PBF and further opened up the possibility to manufacture advanced lightweight parts with potential applications in automotive, aeronautical, aerospace, and defence.

As the focus of this research has been the AlSi7Mg alloy, the rest of this literature review will focus on the processing of aluminium alloys, primarily Al-Si alloys.

2.3.1 Processability

Due to the high thermal gradients and strong segregation that occur during PBF, cracking is a major issue impeding the successful manufacture of aluminium and is one of the most significant barriers to the transition of existing alloys to PBF processes. Cracking occurs at the end of solidification when solid crystals are still separated by intergranular liquid, when due to the shrinkage associated with cooling from liquid to solid, the lack of shear resistance in this liquid enables the separation of grains and a crack to form [103]. This issue is made more pertinent in alloys with a large difference between solidus and liquidus temperatures and few eutectic points, meaning alloy chemistry is a major risk factor [100]. Due to this process's reliance on chemistry, cracking has particularly been an issue when attempting to additively manufacture alloys in the 2000, 6000, and 7000 series [104]. The addition of additives to alloys has been the most common approach to reduce cracking [105, 106], with silicon being one of the most popular. Due to its improved printability, the Al-Si alloy system is the most popular aluminium alloy currently in use for PBF production [104].

One of the most common issues facing the processability of aluminium is its high reflectivity and low laser absorption, with the reflectivity being estimated to reach 91% [6]. This means that to produce fully or near-fully dense parts much higher laser powers are needed than theoretically required [105], with insufficient input energies likely to result in defects such as lack of fusion porosity, which have detrimental effects on mechanical properties [86]. This issue of reflectivity is being investigated in a number of research studies. These include preheating powder to improve its laser absorptivity, pre-sintering powder with multiple laser passes to reduce laser power requirements, and changing the chemistry of the powder [93, 107].

Another of the issues regarding the processability of aluminium alloys by PBF is the preferential evaporation of some alloying elements. While this is an issue for many alloy systems [6] this is particularly a problem for magnesium and zinc, both commonly alloyed with aluminium. Given their respective evaporation temperatures are 1090 °C and 907 °C, with

the melt pool regularly reaching 1200 °C, although this is dependent on the laser parameters used [103, 108] their evaporation is common. This is of especial concern given the typically low amounts of these elements present in feedstock material, and their significant role in strengthening mechanisms. The main proposition to control this is the careful selection of laser parameters in order to avoid overheating [6], while the doping of alloys with extra amounts of vaporisation-prone elements has also been successfully trialled [4] such that upon evaporation these elements remains at their intended levels.

2.3.2 Alloy Systems

There are currently a limited number of aluminium alloys commercially available for PBF as it has been thought that there is little advantage in developing these alloys for PBF, as they are already relatively cheap and easy to process with traditional manufacturing techniques [109]. This is compounded by the fact that many aluminium alloys are difficult to weld, have high reflectivity and are prone to cracking [109, 110].

These difficulties have meant that the most common class of aluminium alloy for PBF applications are aluminium-silicon alloys due to their success in casting procedures [1, 111]. For this reason, AlSi10Mg has received some of the most attention in the literature. The prevalence of some common classes of aluminium alloys are explained in the following sections.

2.3.2.1 Aluminium-Copper Alloys

Al-Cu alloys are a high strength group of aluminium alloys stemming from the 2xxx series of alloys, originally designed as wrought alloys to be manufactured with techniques such as forging and rolling [112]. Due to the limited geometries available from these methods, PBF is a promising means of manufacturing parts for the automotive, aerospace, and defence industries with more complex geometries, as these are industries where this alloy is already in use. This is especially true given the difficulty in creating complex structures with traditional manufacturing techniques with this alloy group [5].

As is typical of aluminium alloys produced using PBF, cracking is an issue when the correct laser parameters are not selected. This is especially problematic for Al-Cu alloys given their

larger solidification range than most aluminium alloys [113]. This was observed by Zhang et al. [5], who found that, at an energy density of 340 J/mm^3 , samples of 99.5% density could be produced, while lower energies resulted in porous and crack filled samples. In comparison to many aluminium alloys, this is a high energy density requirement for acceptable densification.

Mechanical testing of as-built Al-Cu alloys follows a similar trend to that of other PBF materials, where strengths are generally higher than those of their traditionally manufactured counterparts. This was found to be true in comparison to cast material for both hardness and tensile strength by Zhang et al. [5]. This was attributed to the fine microstructures, high dislocation density, and supersaturated solid solution typical of PBF aluminium. However, given this is an age hardening alloy there remains further potential for strengthening with the application of heat treatments tailored for PBF Al-Cu parts.

2.3.2.2 Aluminium-Zinc Alloys

Al-Zn alloys are based largely on the 7XXX series of aluminium alloys, an attractive class of alloys due to their high strength and excellent age-hardenability [114]. Due to this high strength, the 7XXX series is popular for aeronautical and aerospace applications where high strength and low weight are required. However, their poor weldability, as well as other properties such as their poor fluidity, high thermal conductivity, and high solidification shrinkage rate, mean they do not inherently lend themselves to PBF processes [115], often requiring modification to their constituent elements to become processable.

This is largely due to the susceptibility of these alloys to crack during production, which can be improved by the addition of further alloying elements. One example was the addition of 1 wt.% zirconium, which along with a reduction in solidification cracking led to increased grain refinement [4, 115]. Other elements that have been added include silicon and scandium, although the mechanical properties from these new alloys still do not meet those of conventionally manufactured parts [114].

One of the significant issues with processing Al-Zn alloys by PBF is zinc's low boiling point of 907°C compared with aluminium's 2470°C , which results in a loss in strength due to lowered precipitate growth and hardening as the amount of zinc in the part is lower than that of the powder. This was observed by Mertens et al. [4] who found a reduction of 50% of the

zinc content when producing Al7050. As such, these authors added an extra 7 wt.% zinc to ensure the correct alloy composition in the final product.

While these are all promising developments in the PBF of Al-Zn alloys, this research is still in its early stage and has yet to reach full maturity since it has mainly focused on the production of crack free components with stable chemical composition. As such, widely available 7XXX series alloys optimised for additive manufacturing are not yet available.

2.3.2.3 Aluminium-Scandium Alloys

Given that many aluminium alloys are susceptible to both hot cracking and over-ageing during stress relieving heat treatment [116], the chemical composition of aluminium alloys is in constant development. One of the most promising, and regularly used, elemental additions is scandium, typically included alongside larger elemental concentrations of elements such as magnesium, zinc, and copper due to their prevalence in traditional aluminium alloys. Scandium has gained interest as an alloying element as these new alloys have a small solidification temperature range and the tendency to form a super saturated solid solution of scandium in aluminium. This saturation then promotes the growth of Al_3Sc precipitates which greatly improve the material's grain refinement and strength [116, 117].

A more recently developed PBF specific alloy is Scalmalloy, touted as being able to provide both high strength and ductility [118]; its strength has been shown to be greater than that of traditional wrought aluminium alloys [119]. While still limited in its use, Scalmalloy has been used as a basis for the development of newer PBF designed Al-Sc alloys [117, 120] which shows great promise for future applications in aerospace due to its light weight, corrosion resistance, and improved strength [119]. Part of the study on these new alloys focuses on the development of Al-Sc alloys with minimal scandium content, due to its high cost and relatively low supply [120].

2.3.2.4 Aluminium-Silicon Alloys

The most commonly available commercial aluminium alloys for PBF are near eutectic Al-Si alloys. The silicon content reduces these alloys solidification temperature range and therefore reduces cracking due to the near eutectic compositions [18], as seen in Figure 2.6. For this

reason, these alloys are also popular in casting, and as such some of these alloys have been brought directly to PBF from more traditional manufacturing methods. The pre-existing knowledge of this alloy and the reduction of solidification cracking are some of the main reasons for Al-Si alloys popularity in PBF.

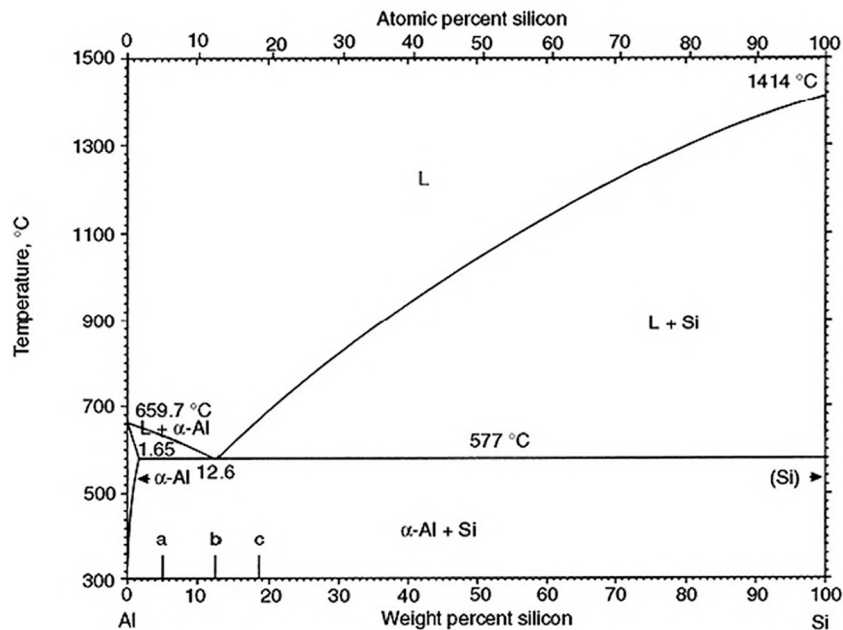


FIGURE 2.6. Al-Si phase diagram (reproduced from [121]).

Given the popularity of this alloy system and the focus on AlSi7Mg for this thesis, Al-Si alloys will be the focus for the rest of this section of the literature review. Further detail on their microstructure, mechanical properties, and heat treatment routes is given in the following sections.

2.3.3 Microstructure of Aluminium-Silicon Alloys Produced Via Powder Bed Fusion

As with other materials, the microstructure of aluminium alloys manufactured using PBF is dictated by complex thermal histories. This produces a unique microstructure involving melt pool tracks, columnar grains, cell structures, and precipitates.

The largest scale features in aluminium PBF microstructures are the melt pool tracks, visible at low magnifications using optical microscopes. In the X-Z and Y-Z planes this produces a

characteristic fish-scale morphology, where each melt pool is visible along with remelted and heat affected zones. In the X-Y plane, overlapping melt pool tracks are visible. Examples of these are shown in Figure 2.7. These melt pools are visible due to the fine cellular network of silicon throughout the as-printed material, which reheats with multiple laser passes resulting in a coarser structure along the melt pool boundaries [20]. This coarsening creates zones within each melt pool: the melt pool core, melt pool boundary, and heat affected zone, with different properties thought to be attributed to each zone. The size of these melt pools is dictated mainly by the layer thickness, with their arrangement dependant on the scan strategy used [122].

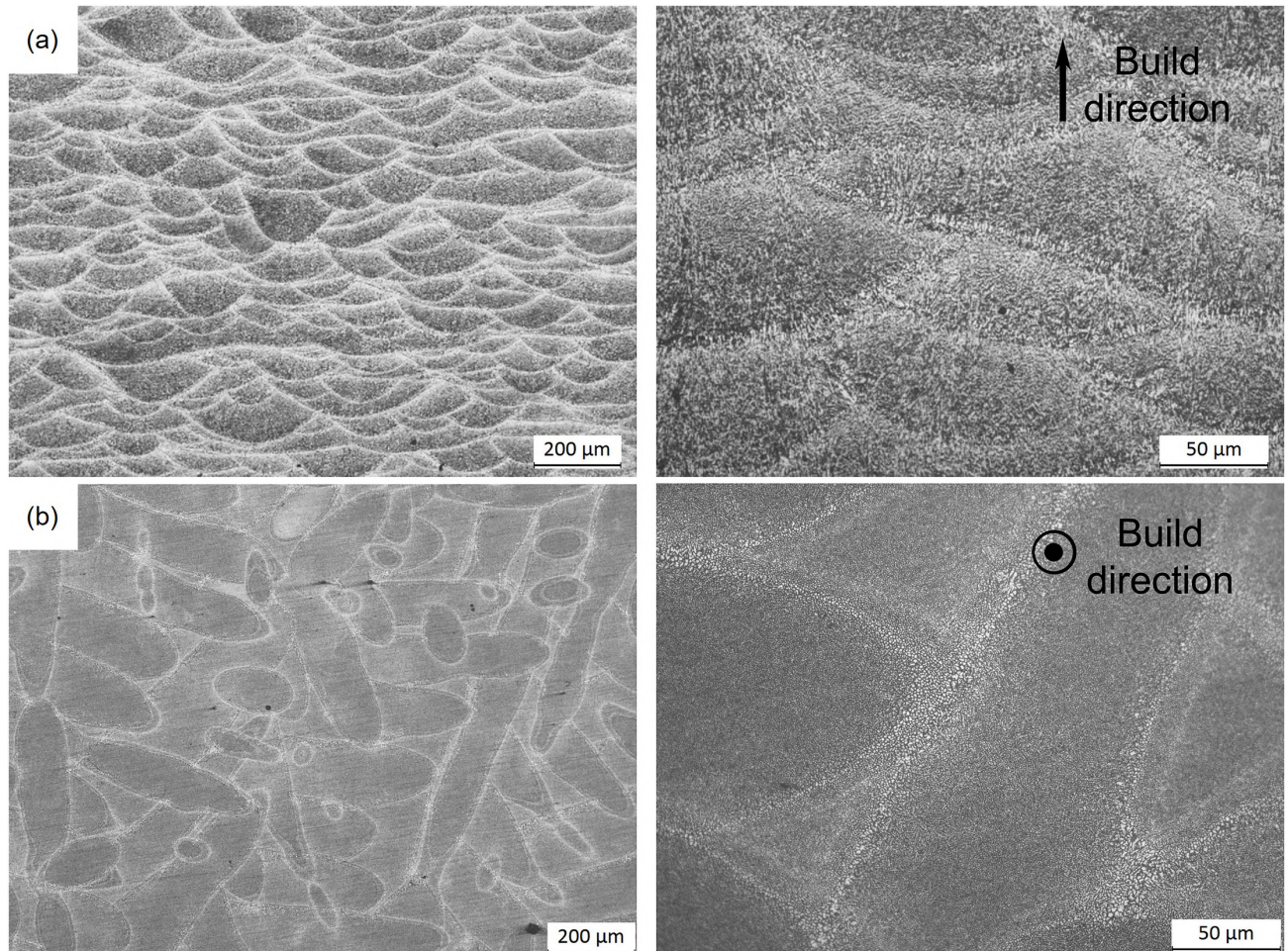


FIGURE 2.7. Optical micrographs of the microstructure of AlSi7Mg (etched with HF 1%) (a) X-Z plane (b) X-Y plane (reproduced from [20]).

Grain growth within the melt pool is dependent on the solidification front direction and the thermal gradients present, creating an elongated grain structure growing perpendicular to

the melt pool boundary, typically in the build direction [1] to create a [100] texture [86]. During the remelting of parts of these melt pool tracks during subsequent laser passes, refined equiaxed structures at the edge of melt pools can be reduced to further accommodate the growth of equiaxed grains throughout the part. It has been proposed that grain boundary strengthening plays a minimal role in the strength of PBF AlSi10Mg [86], however it is more likely that since factors such as heat treatment, build plate temperatures, and laser parameters do not significantly influence grain size [123], grain boundary strengthening provides an insignificantly changing contribution when these parameters are manipulated.

Within Al-Si alloy systems the cellular structure is characterised by borders of silicon and cells of aluminium, due to the relationship between the solute concentration of silicon in aluminium, the effect of temperature on this relationship, and the rapidly moving solidification front inducing high cooling rates [1, 124]. During solidification, the solute concentration in the liquid is increased because of the silicon's rejection from the solidified material. However, due to the high cooling rates the solubility of silicon in aluminium is increased, reducing the amount of silicon in the liquid as well as the amount of undercooling. This then favours the development of a cellular network, where aluminium solidifies in a cellular morphology first while the silicon is segregated to the boundaries [93]. 3D observations of these cellular structures reveal that these cells are approximately 500 nm in size and tubular, as shown in Figure 2.8, and are thought to significantly strengthen the material by Orowan mechanisms [20].

Within the cell walls, the majority of the matrix is comprised of a super saturated solid solution of solute silicon within the aluminium matrix. The amount of silicon within this matrix has been found to be 3% [125], considerably higher than the theoretical solubility of 1.6%, although this is likely dependent on the level of substrate heating temperature used during manufacture. The amount of silicon found in the matrix is dependent on the cooling rate of the material, driven by the small size of the melt pool [86, 126]. Also present are Al-Mg or Si-Mg clusters containing a varying amount of each element [125], although due to the range of Mg:Si ratios observed in the literature it is unlikely that there is a significant amount of the strengthening Mg_2Si phase present [86].

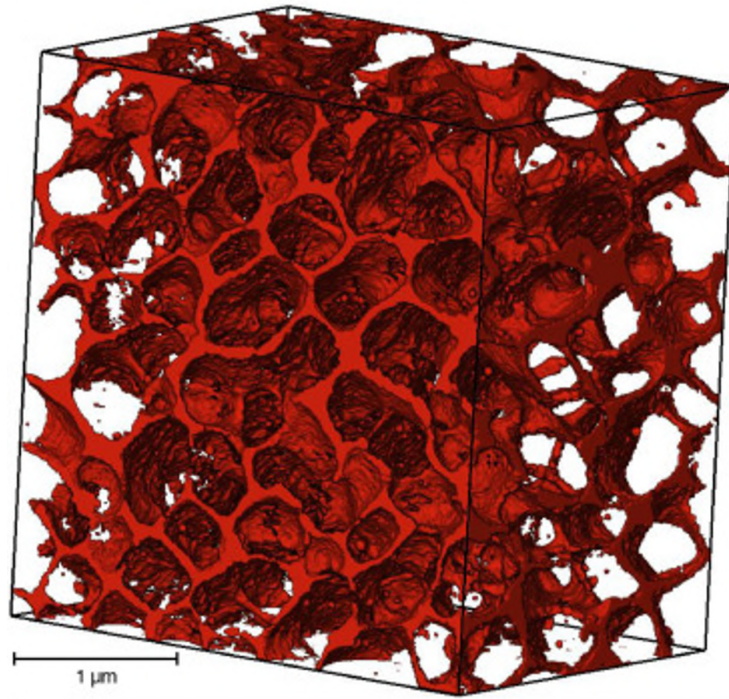


FIGURE 2.8. FIB/SEM tomography of silicon rich phases in PBF ALSi10Mg (reproduced from [123]).

2.3.4 Mechanical Properties of Aluminium-Silicon Alloys Produced Via Powder Bed Fusion

Mechanical properties are some of the most important aspects of materials science investigation and as such have been regularly investigated throughout the literature. Three of the most common of these are hardness, tensile strength, and fatigue strength, each of which are elaborated upon below.

2.3.4.1 Hardness

Due to the ease of testing and the simple sample requirements hardness testing is commonly conducted on PBF parts, with findings regularly showing that PBF parts tend to be harder than their traditionally manufactured counterparts. Considerable variation exists between studies regarding the hardness of aluminium alloys, to be expected given the variety of machines, processing parameters and starting powder properties used. This suggests that the hardness of PBF aluminium alloys is reliant on the processing parameters used.

The hardness of additively manufactured aluminium alloys tends to be between 130-150 HV, higher than that of cast alloys of similar compositions [6]. While the fine microstructure plays a role in this, the effect of residual stresses cannot be dismissed [127], suggesting that heat treatments aimed at eliminating residual stress must be carefully tailored in order to minimise reductions in hardness.

2.3.4.2 Tensile Strength

The tensile strength, similar to the trend for hardness values, tends to be higher for PBF manufactured parts when compared to traditionally produced parts due to the fine microstructure, presence of sub-grain boundaries, and presence of the silicon cellular network [128]. This also results in parts with relatively low ductility. While this trend is evident, an overview of the literature makes it difficult to determine a relationship between tensile properties and laser and build platform parameters.

The strength of PBF aluminium alloys is thought to be affected by a number of factors, one of the most significant of these being Orowan strengthening. Wu et al. [128] observed by transmission electron microscopy (TEM) the pinning of dislocations by silicon rich particles in cell centres, with others also observing similar results [129, 130], with this effect changing depending on precipitate size [123]. While these observations are becoming more frequent, dislocation entanglement is more commonly seen along the boundaries of the silicon network [130]. The amount of silicon in solid solution also affects the strength of aluminium-silicon alloys due to the lattice deformation caused by the inclusion of silicon atoms (radius of 0.118 nm) in the aluminium (radius 0.143 nm) matrix, hindering dislocation motion [126, 129]. Finally, the growth of precipitates is commonly seen to improve the strength of aluminium alloys, particularly the Mg_2Si phase which is induced by ageing Al-Si-Mg alloys. This will be discussed further in Section 2.3.5.

One of the issues processing aluminium alloys, especially high strength alloys, is their susceptibility to cracking during solidification, resulting in lower strength than other manufacturing systems. The addition of nanoparticles to the powder feedstock has been seen to reduce this issue. Martin et al. [106] pioneered this by adding Al_3Zr nanoparticles to 7075 aluminium, making this material processable with a tensile strength 80% higher than AlSi10Mg and within the expected bounds of wrought parts. The addition of these nanoparticles also resulted

in a uniform and equiaxed grain structure with a size of 5 μm due to the introduction of many nucleation sites, further increasing tensile strength. The addition of chemical additives to reduce cracking in other studies has similarly resulted in an increase in tensile strength [131–133], suggesting that processability improvements may also increase strength. While research such as this shows great promise for improving the properties of PBF parts, so far safety protocols around handling nanoparticles during PBF are lacking which presents a significant oversight for future implementation.

2.3.4.3 Fatigue Strength

While in general the mechanical properties of PBF produced parts are comparable to those of conventionally produced components this is not the case under cyclic loading [134], of special concern given fatigue is a common failure method. This poor fatigue performance is typically attributed to factors such as surface roughness, residual stress, and porosity, all common issues with PBF [1] which act as stress concentrators, reducing fatigue life. This is especially an issue given the ring of near-surface porosity evident in many PBF parts [135]. Another key challenge for additively manufactured metals subject to fatigue loading is the significant scatter seen in the data [86].

The initial level of porosity seen in PBF parts is one of the major issues affecting fatigue life. Uzan et al. [136] found that the fatigue resistance of wrought Al 6061-T6 was higher than PBF AlSi10Mg parts even after a variety of heat treatments, albeit the wrought Al 6061-T6 had a slightly higher yield strength, with higher yield strengths tending to improve fatigue life. This change in fatigue resistance is likely due mainly to the larger initial pore size of the PBF parts and the role these play in crack initiation [137], which is also the reason behind the considerable scatter in aluminium PBF fatigue data.

Heat treatments have the potential to influence fatigue life, however these processes can have both positive and negative influences. Heat treatments can improve the yield strength of aluminium-silicon alloys as well as reduce residual stresses from the manufacturing process, both of which will improve fatigue strength. This is, however, coupled with the fact that many high temperature heat treatments will increase porosity by expanding any trapped gasses within the structure [20], thereby increasing the size of already present gas pores. These

contrasting effects mean that if fatigue life is to be a critical issue for a PBF aluminium part, the heat treatment chosen needs to be carefully considered.

2.3.5 Heat Treatment of Aluminium-Silicon Alloys Produced Via Powder Bed Fusion

While aluminium is becoming an increasingly valuable material in PBF, post-processing heat treatments for the most part have been limited to traditional methods designed for components with microstructures distinct from PBF parts. PBF processes tend to form parts with strongly supersaturated solid solutions, high residual stresses, directional solidification, and in-situ heat treatment from successive heating and cooling cycles [111], each of which contribute uniquely to the mechanical properties of these materials. Therefore, the application of traditional heat treatments may be detrimental to the properties of PBF parts. This is particularly pertinent given the decomposition of the metastable cellular structure by many heat treatments, accompanied by the destruction of low angle grain boundaries by the movement of the many dislocations found in the as-built structure [1]. While this structure decomposes, minimal grain refinement has been reported in the literature [90] meaning that many treatments result in a loss in strength.

The heat treatment of aluminium alloys is driven primarily by precipitation hardening [111], as many alloys used in PBF have compositions optimised for the development of precipitates. Examples of this are the addition of Mg to Al-Si systems, and the addition of Sc to Al-Mg alloys.

Given the majority of research on additively manufacturing aluminium has focused on aluminium-silicon alloys, it follows that much of the initial research into heat treatments has been conducted these systems. The first trend to emerge in this research was the application of traditional heat treatments, such as T6, with the manipulation of temperatures and times to tailor these processes for PBF [111]. This further developed into more tailored treatments, such as T5 and annealing, to account for the unique microstructures of these components. Further analyses of the microstructural changes induced by these heat treatments have tended to focus on the precipitation of the Mg_2Si phase and the change in morphology of the silicon network [111].

2.3.5.1 Annealing Treatments

Annealing treatments are regularly conducted on PBF parts as a means of stress relief prior to part removal from the build plate in order to eliminate warping [138]. This is as the intense heat applied by the laser and corresponding high thermal gradients results in contraction stresses upon solidification, which if released by premature removal of a part from the build plate may result in distortion [139]. These stress relieving procedures tend to take place at temperatures ranging between 250 °C - 300 °C, higher than those used for ageing, for times of up to two hours.

While stress relief is the primary objective of these annealing treatments, microstructural changes also occur. This is primarily the agglomeration of silicon along the cell wall boundaries, with solute drawn from the aluminium matrix and small silicon particles within the cell interiors breaking down to further grow these boundaries [38, 140]. This means that while the cell network is still present, it becomes coarser and more discontinuous [38] and the extent to which this happens is a function of temperature, as seen schematically in Figure 2.9. The temperature at which the transition from the growth of the cell boundaries to their destruction occurs is debated, with temperatures ranging from 240 °C to 290 °C being presented in the literature [111]. Further works have indicated that the exact temperature for the destruction of the silicon network is dependent on other processing parameters such as laser power or build plate preheating temperature.

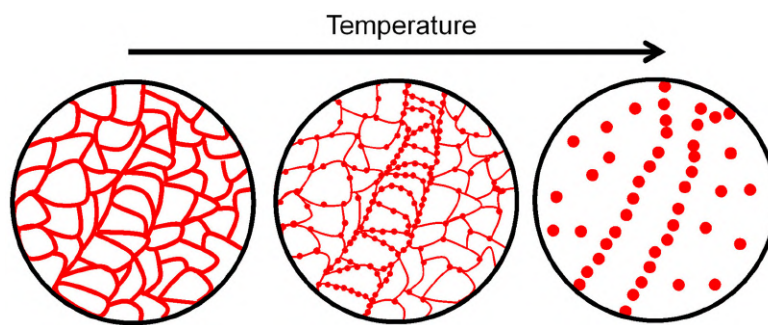


FIGURE 2.9. Schematic of the silicon network in an annealed Al-Si alloy, with increasing temperature from left to right (reproduced from [111]).

Other microstructural changes are limited, as although some studies have observed a change in grain size after a stress relieving annealing treatment is conducted, the majority observe no

change [111]. The changes that have been observed were also small enough that they can be attributed to the variability of local grain size rather than any significant structural changes.

While annealing has been the traditional method of stress relief, the use of heated build plates has been seen to lower residual stresses, creating the potential to omit stress relieving annealing treatments altogether. The manufacture of parts using different build plate temperatures has been seen to reduce residual strains by changing lattice parameters, with higher temperatures reducing stresses by a greater amount [101] as heated build plates promote the diffusion of silicon from the matrix to the cell boundaries, reducing the silicon in solid solution. Silicon in solid solution contracts the matrix and increases residual strain, as measured by x-ray diffraction (XRD) and backed up by energy dispersive x-ray spectroscopy (EDS), and so a reduction of solute silicon will reduce residual stresses. This effect is dependent on the build plate temperature, with a higher build plate temperature promoting greater silicon precipitation and so reducing the amount in solid solution to a greater extent.

These treatments almost exclusively lower the strength of PBF Al-Si alloys, although the extent to which this occurs depends largely on the degree of degradation of the silicon network. As mentioned previously this relies largely on annealing temperature, with an example of these tensile properties as a function of annealing temperature shown in Figure 2.10. This reduction in strength is partly a result of the lowering strain hardening with annealing, due to the reduced ability of the silicon cell boundaries to create dense areas of geometrically necessary

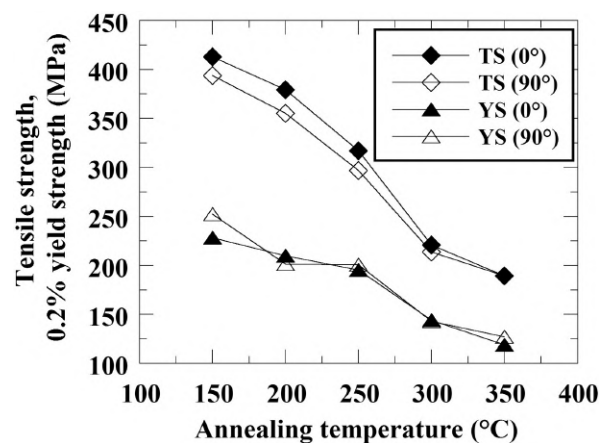


FIGURE 2.10. Evolution of the tensile strength (TS) and yield strength (YS) of AlSi7Mg annealed at different temperatures and for different specimen orientations. 0° refers to vertically oriented samples and 90° refers to horizontally oriented samples (reproduced from [140]).

dislocations that would slow the motion of more mobile dislocations [129]. This strength reduction does, however, result in increased ductility as well as a transition from brittle to ductile fracture [111], although anisotropic behaviour is not fully eliminated as demonstrated in Figure 2.10. For these reasons, stress relieving parameters must be carefully selected in order to preserve the mechanical properties for the application, while still eliminating residual stresses that can cause warping.

2.3.5.2 T5 Treatments

T5 treatments are less common in the literature than T6 treatments and are based on direct ageing without a solution treatment step. The interest in direct ageing comes from the significant loss of strength imposed by solution treatments due to the reduction of solute silicon and the coarsening of the silicon phase, two major contributors to the strength of as-fabricated PBF parts [111]. It has also been seen that a solution treatment will increase the porosity of a part due to the expansion of porosity associated with trapped gas from the manufacturing process [20]. T5 treatments attempt to remove these changes by omitting the solution treatment step, although similar porosity increases have been observed at higher ageing temperatures [141]. Since direct ageing is a single step treatment, the opportunity for parameter optimisation is limited in comparison to other heat treatment methods, with ageing time and temperature the main parameters to be changed.

On the larger microstructural scale, direct ageing has minimal effect on the grains of additively manufactured aluminium alloys [142, 143]. It also has minimal effects on the visible melt pool structures and the cell network, as shown in Figure 2.11, as ageing typically takes place at temperatures where silicon diffusion is slow [111, 138]. While the effect on the substructure is minimal, the silicon precipitates along the edge of these cells have been seen to grow during ageing at slightly higher ageing temperatures with Cerri et al. [144] observing their growth in AlSi10Mg aged at 200 °C for 6 hours. This corresponded with an increase in ductility of 1.9% but a decrease in yield strength of 30.5 MPa, due to the reduction of solute silicon and the low number of precipitates created that are insufficient to induce an increase in strength. While these silicon particles grew, work using XRD found that they are relatively small compared to other heat treatment methods [145].

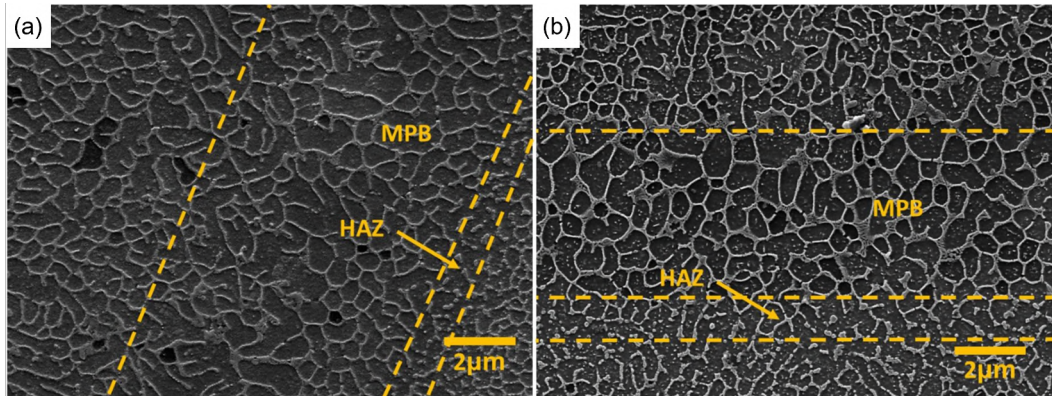


FIGURE 2.11. Silicon cell structure in AlSi10Mg: (a) as-printed, (b) after artificial ageing for 6 hours at 170 °C. MPB refers to the melt pool boundary, and HAZ refers to the heat affected zone (reproduced from [146]).

Strengthening is typically attributed to the growth of the Mg_2Si phase, which is precipitated through the following sequence:

SSSS of Al $\rightarrow$ GP zones formation $\rightarrow$ precipitation of β'' phase $\rightarrow$ precipitation of β' phase $\rightarrow$ precipitation of stable β - Mg_2Si phase

A number of studies have carried out differential scanning calorimetry at temperatures ranging between 0 °C - 500 °C, confirming the growth of Mg_2Si at typical natural ageing temperatures up to 200 °C [138]. Past these temperatures, the destruction of the silicon cell network begins to negate any strengthening effects of precipitate growth.

Depending upon the conditions used in direct ageing, the growth of Mg_2Si precipitates has been found to occur at varying stages. Casati et al. [145] observed the growth of 20 nm long lath-shaped particles after four hours of ageing at 160 °C, which were identified as metastable β'' - $\text{Mg}_4\text{Al}_3\text{Si}_4$ precipitates, coherent with the aluminium matrix. This contrasts with the more common observation that the more stable Mg_2Si particles grow to provide considerable strengthening effects to the alloy even at similar temperatures [20, 143]. Still other studies have found no evidence of the Mg_2Si phase at all [38]. This suggests that while the precipitation sequence of Mg_2Si is known, its exact formation process through ageing of Al-Si alloys is not completely understood.

While these effects of ageing time and temperature are becoming understood, it is also known that other factors such as the use of a heated build plate [147] or a delay between manufacture

and ageing [148] will change the effectiveness of ageing treatments. This is as these processes reduce the amount of silicon in the matrix before ageing, reducing its effectiveness, while also partially relieving residual stresses from the manufacturing process, further weakening the material. There is also the potential for Mg_2Si precipitates to grow during PBF, resulting in over-ageing during T5 treatment [20]. While this has been observed in the literature, thermal histories during PBF manufacturing are complex and so need to be better understood in order to properly utilise in-situ ageing processes.

Given T5 treatments aim to increase the strength of aluminium alloys, tensile and hardness tests are regularly carried out in the literature on direct aged aluminium. Casati et al. [148] observed in AlSi7Mg an increase in hardness from 101 HV to 137 HV after four hours of ageing at 160 °C, although this was reduced to 130 HV if the sample was left at room temperature for a week between manufacture and ageing. This increase in hardness corresponded to an increase in yield strength from 257 MPa to 309 MPa, and tensile strength from 398 MPa to 411 MPa. Similar results on Al-Si alloys have been observed by others in the literature, with changes in strength being ascribed to increased precipitate volume and subsequent Orowan strengthening [39], changing solid solution strengthening [38], a reduction in residual stresses [142], changing cell size and morphology [140], and changing dislocation interactions with the silicon network [141].

2.3.5.3 T6 Treatments

One of the most common heat treatments of Al-Si alloys is a T6 treatment, involving a high temperature solution treatment to dissolve pre-existing precipitates and phases followed by quenching, then a low temperature ageing process to grow the required precipitates for strengthening. Examples of the evolution of this microstructure are shown in Figure 2.12. Many studies focus on the optimisation of T6 parameters, typically by varying solution and ageing temperatures and times, then determining optimal properties by measuring the material's hardness. However, there is minimal research on the effect of solution treatment temperature for PBF parts, with most studies using temperatures between 500 °C and 550 °C with minimal justification [149–152].

One of the notable effects of a solution treatments is the destruction of the melt pool network and the corresponding melt pool centre, melt pool boundary, and heat affected zones. Although

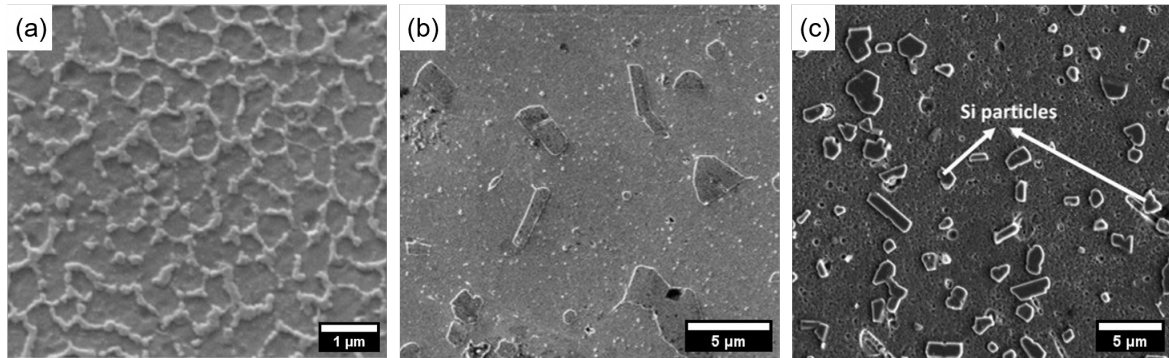


FIGURE 2.12. SEM images of (a) an as-built AlSi10Mg PBF part (reproduced from [138]), (b) AlSi7Mg solution treated at 540 °C for one hour (reproduced from [153]), (c) AlSi10Mg after a conventional T6 treatment (reproduced from [154]).

this phenomenon is regularly commented on, it is rarely discussed in any depth. While the breakdown of this network is common there is minimal effect of the solution treatment on the grain size and texture, with the columnar grains parallel to the build direction characteristic to PBF microstructures preserved in solution treated samples due to the pinning effect of silicon particles along grain boundaries [38].

During solution treatment, the main effect is the destruction of the cellular network and the growth of large silicon particles, accompanied by the reduction of solute silicon. The number and size of these particles depends on the solution time and temperature, with longer solution treatment times creating larger particles due to Ostwald ripening [111], however they tend to coarsen preferentially at grain boundaries for short solution treatment times before distributing more evenly for longer treatments [142]. A schematic of this process is shown in Figure 2.13. Since the majority of movement of silicon from matrix to precipitates occurs in the first half hour of the solution treatment [155], this has led to many authors investigating the effects of short solution times in heat treatments tailored for PBF components [20, 148]. While this is generically true, it has been shown that the silicon distribution after solution treatment depends on the alloy composition [141], and so heat treatments must be tailored for each independent alloy rather than individual alloy classes.

An increase in the size of the silicon particles and the reduction in their number reduces localised stress and strain during tensile loading. This, along with a reduction in residual stress, results in an increase in ductility that has been seen to reach up to 25% in a solution

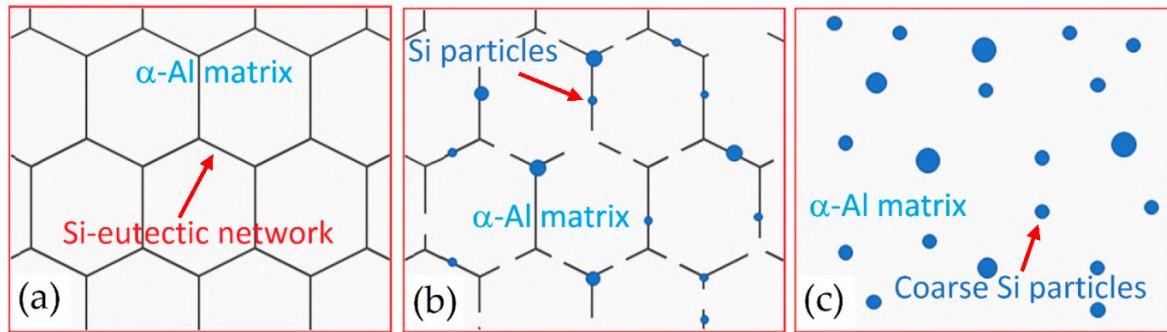


FIGURE 2.13. The process of silicon coarsening in solution treated Al-Si alloys: (a) as-built cell structure with silicon at the cell boundaries, (b) silicon beginning to preferentially coarsen at these cell boundaries, (c) final microstructure of coarse silicon particles distributed through the aluminium matrix (reproduced from [138]).

treated AlSi12 alloy [155]. While this is significant, the poor strength that correlates with such impressive ductility means that an ageing treatment is necessary for these materials to be readily applicable.

During the same solution heat treatment, the growth of iron rich phases is also commonly observed. These are typically β - $\text{Al}_9\text{Fe}_2\text{Si}_2$ needle-like phases or the π - $\text{Al}_{18}\text{Fe}_2\text{Mg}_7\text{Si}_{10}$ phase, although spherical precipitates of silicon and iron also form [20, 142, 149]. These phases are considered to embrittle the material due partly to their high aspect ratios and so are often undesirable, although the addition of small amounts of beryllium has the ability to reduce this effect by transforming these precipitates into less damaging, nodular shapes [148]. For high iron content alloys, introducing manganese has a similar effect.

After artificial ageing, a number of precipitates grow to contribute to the increase in strength from the solutionised state due primarily to the silicon and magnesium contained in solid solution [140]. Since at ageing temperatures the diffusion rate of iron is lower than that of silicon and magnesium [142], primarily this is the Mg_2Si phase, although the exact amount of this strengthening phase is highly dependent on the magnesium content [141] and the extent to which ageing has occurred. Therefore, the ageing process after solution treatment tends to exhibit similar mechanisms to samples that have been direct aged.

While solution treatments are known to decrease the strength of aluminium alloys and increase their ductility, alongside a reduction in anisotropy [39], the final properties of a T6 treated

alloy are complex. The level of strength recovered during ageing differs between studies, however when compared with the as-built properties most studies observe a reduction in strength [20, 38, 39, 140]. While this is the case for strength, ductility is usually improved by T6 treatments, even with differing solution and ageing times and temperatures [20, 156], although some exceptions do exist [140]. This improvement in ductility is accompanied by a more ductile fracture, attributed in part to the destruction of the silicon cell network [38].

Within the same heat treatment conditions, differences in properties were observed between AlSi10Mg and AlSi7Mg. Cerri et al. [141] saw that after a solution treatment of 505 °C for four hours followed by ageing at 175 °C for four hours, AlSi10Mg reached a hardness of 97 HV whereas the as-built hardness was 110 HV, while the AlSi7Mg's hardness of 111 HV after T6 was higher than the 105 HV reached by their samples 300 mm from the build plate. This was attributed to the alloys' differing magnesium contents and therefore different levels of Mg_2Si growth, suggesting that ageing times and temperatures need to be tailored for particular alloys. This is of especial significance when considering tailored properties required for particular applications.

2.4 Conclusion

Metallic additive manufacturing, and powder bed fusion in particular, demonstrate significant potential to dramatically alter the manufacturing industry, particularly in relation to the production of geometrically complex parts for low volume manufacturing runs. Regarding this, there has been considerable investigation into PBF in order to mature it as a mainstream manufacturing method, with significant progress being made. This currently allows for a wide range of materials to be produced by PBF, with a good base of knowledge developed allowing for a valuable understanding of processing-property relationships.

This is also true for aluminium. While research has focused on the Al-Si class of alloys due to their castability, many classes of aluminium alloys are now being examined for PBF manufacturing. This does not mean, however, that research into Al-Si alloys is by any means complete, with a significant number of research topics still requiring development.

One of these is the effect of production parameters on part properties and microstructures, such as part height, build plate location, specimen orientation, part size, and surface finish. This is especially true given that these factors are seen to have different effects depending on the PBF machine, processing parameters, and materials used, and so an understanding of the mechanisms behind these influences is required. Such an understanding would allow for the production of parts that are of a high standard, but also allow for the manipulation of these inputs to design and produce parts that use these factors to a parts advantage. As such, further research into these areas is key.

Another significant aspect requiring further research is heat treatment, specifically in regard to the modification of traditional heat treatment methods to suit additively manufactured parts. This is particularly true in relation to Al-Si components, where significant amounts of their strength are derived from the rapid solidification microstructures inherent to the process, which are broken down during many traditional heat treatments. Therefore, an improved understanding of the mechanisms of these heat treatments is required, as well as understanding how factors such as heated build plates and solution temperatures will change microstructures and mechanical properties. The development of such an understanding will allow for the improvement of the final properties of PBF produced Al-Si components, improving their performance in real world applications.

CHAPTER 3

Literature Review on the Reuse of Powder in Powder Bed Fusion

One of the issues relating to PBF is the reuse of powder, given a substantial amount of powder remains unconsumed at the completion of each manufacturing run. This powder must be reused in order to improve the efficiency of the PBF process, as powder reuse results in a significant reduction in cost as well as providing supply chain and environmental benefits. This is a readily accepted fact within the literature.

The quality of used powder is, however, often an unknown quantity affected in a number of ways, with many factors changing the properties of a powder batch especially in cases of long-term powder reuse.

A significant amount of research has been published regarding the effects of powder reuse using a variety of techniques and materials, however little attention has been paid to the method of powder reuse utilised. This chapter reviews the literature in order to compare the effectiveness of different powder reuse methods, with consideration of both powder and part properties. This forms the literature review for Chapters 7 and 8 of this thesis, where powder reuse is considered as a means of improving the sustainability of AlSi7Mg, with a specific focus on the effect of the powder reuse methodology employed.

This chapter contains research that has previously been published as the article “Strategies for powder reuse in powder bed fusion: A review”. The paper was authored by J.H. Warner under the supervision of G. Proust and S.P. Ringer.

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3.1 Introduction

Additive manufacturing is a term representing a number of technologies that allow for the production of objects by the deposition of material rather than subtraction, as is the case in many traditional manufacturing techniques [109]. The popularity of additive manufacturing has its origins in its ability to produce components of geometric complexity that would otherwise be difficult or impossible to create through conventional manufacturing techniques such as machining [15]. More recently, there is a recognition that additive manufacturing is a scalable route to production of new alloy materials. One of the more common additive manufacturing technologies is powder bed fusion, which utilises the continued deposition of thin powder layers selectively melted by either a laser or electron beam according to a CAD design until a final product is built. PBF is primarily responsible for the industrialisation of metal additive manufacturing, and applications in the aeronautical, biomedical, automotive, and aerospace industries now abound [1, 157].

One of the considerable advantages with PBF is the ability for powder to be reused. Commonly, a significant fraction of the machine's powder stock will remain unused in the production of a given part. The amount of powder remaining unconsumed varies between builds, depending on part geometry and build chamber dimensions.

The reuse of powder presents both economic and environmental benefits. It has been estimated that Nickel Alloy 718 powder can cost about 90 USD/kg [158], while more expensive alloys such as Ti-6Al-4V can cost up to 400 USD/kg [159]. This pricing and the issues around resource utilisation demand a powder reuse strategy, as the process would be unviable if the non-consumed powder were to be wasted. Since the powder cost typically comprises 10-30% of the build cost [160, 161], substantial increases in the cost per part follow from not reusing powder. This is especially true when some powder suppliers recommend that only virgin powder be used [162]. Moreover, compliance with increasingly sophisticated emissions regulations require reductions in the energy consumption of manufacturing processes and this is a key focus for industry. Since it has been estimated that aluminium requires 224 MJ of energy to atomise 1 kg of powder [163], powder reuse is able to assist in meeting these requirements. In addition to this, powder reuse represents a significant easing of the logistical considerations surrounding powder based additive manufacturing. Lower powder quantities

need to be purchased, shipped, stored, and tracked, reducing both the administrative load accompanying inventory management and the risk associated with storing large quantities of potentially dangerous material. These considerations all factor especially into industrial applications, where cost and efficiency are particularly important. The lack of powder reuse in industrial applications therefore has the potential to complicate the oversight of powder based additive manufacturing, as well as increase both the associated costs and environmental footprint.

Notwithstanding the clear economic and environmental drivers to reuse powder, this remains an issue because the effects on the melt pool behaviour from changes in the powder batch are not fully understood. The interaction between the energy source and the melt pool generates new powder types in the form of evaporated metal and powder spatter [159, 164]. These newly generated powder particles form by the evaporation and ejection of material from the melt pool and subsequent solidification in the chamber atmosphere before settling back on the powder bed, where they are typically not consumed in subsequent laser or electron beam scans. These particles differ from the virgin powder in morphology, porosity, and surface oxidation state [165]. Notwithstanding the separation of some of this newly generated powder via sieving, their inclusion into the feed will change the properties of the powder batch as a whole, and so influence the fidelity of the as-built microstructure from one build to another.

As such, considerable effort has been put into understanding the effect that powder reuse has on both the powder and built part properties. This research has been wide ranging covering a number of materials including mainly steel, aluminium, titanium and nickel alloys [166, 167], but bronzes [168] and tantalum [169], among others, have also been studied. Many characterisation techniques have been used on these powders in order to determine changes in shape and size distributions, flow characteristics, oxygen content, the phases present, and microstructure, among others [170–174]. However, the bulk of the literature continues to use relatively simple powder characterisation techniques. Many tests have also been conducted on parts manufactured by PBF utilising reused powders such as tensile strength, fatigue strength, hardness, density, surface roughness and microstructural analysis [175–180].

This research into both reused powders and manufactured parts, although extensive, remains difficult to compare. As interest in this area is growing, comparisons are of vital importance yet currently studies lack consistency regarding powder reuse methods, materials, machine

parameters, characterisation techniques, measured properties, component geometry and powder reuse cycles [181]. As such, results are often cited as being inconsistent between studies [102] making definitive conclusions on the effects of powder reuse strategies difficult.

The most significant of these inconsistencies is the powder reuse method employed in each study. While some reuse practices are well laid out within the research protocol [182–184] others state simply that powders have been reused [185, 186] meaning that few genuine conclusions can be drawn connecting changed powder and part properties with the powder reuse schemes employed. There have also been a range of reuse methods employed in studies, however little focus has been placed on the relative effectiveness of these strategies. This means that industrially standardised powder reuse methods remain a distant goal. While qualitative conclusions can be drawn, as of yet there has been no study into a comparison of powder reuse methods within the literature that would enable reuse guidelines to be written. This review will analyse the results from a number of powder reuse studies to determine the effects that the employed powder reuse method has had on powder and manufactured components, with the aim to recommend a general approach for future research and industrial applications.

3.2 Powder Reuse in the Literature

Recent years have seen a more concerted effort to attempt to understand the effect of powder reuse on powder characteristics and part properties. Figure 3.1 shows the number of powder reuse studies published by year, and Figure 3.2 represents an overview of the literature categorised by material.

To be able to properly understand how powder and parts change with powder reuse, reuse methods must be properly quantified and analysed, yet one of the main issues within the literature is the incomplete information provided regarding powder reuse methodologies. Many papers simply say that powder has been reused, with little mention of powder reuse methods or the number of times a powder is reused [170, 179, 186–189]. If powder reuse methods are not effectively described, it becomes impossible to either replicate past methods or determine the true extent to which reused powders are different from pristine powders and how that affects part quality. Also commonly overlooked in methodology reporting is the

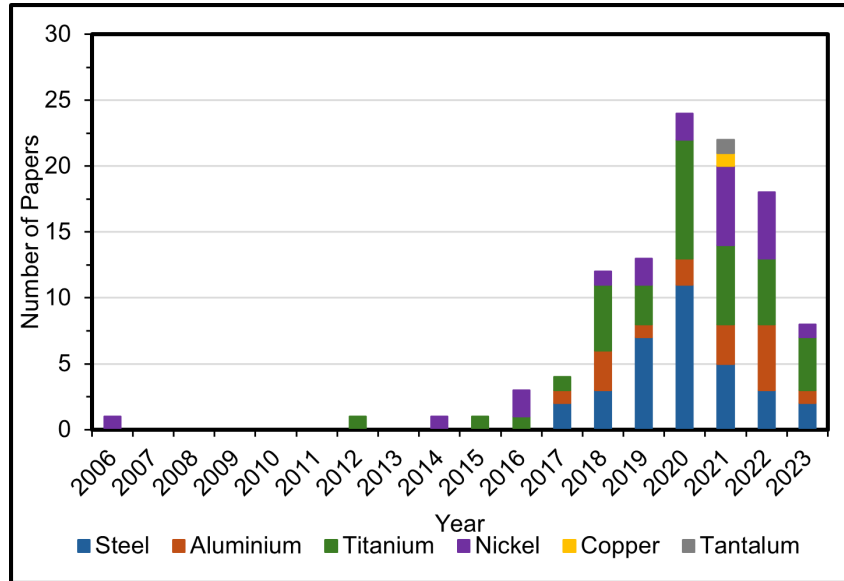


FIGURE 3.1. Number of powder reuse studies published by year and material.

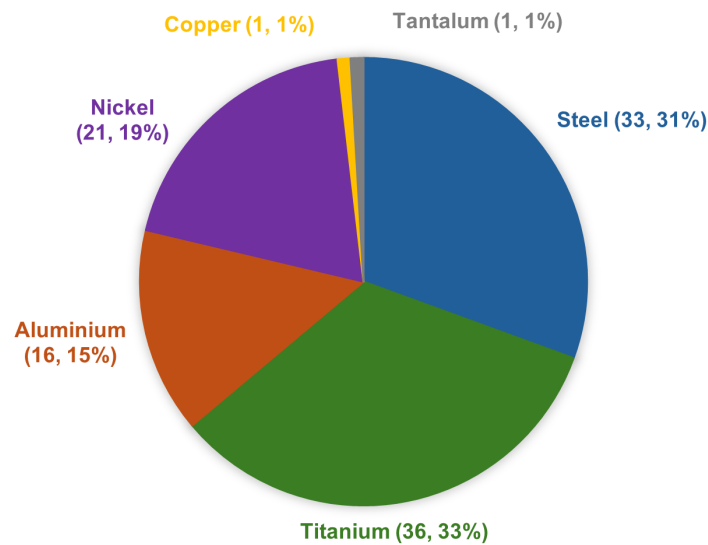


FIGURE 3.2. Graphical representation of the materials investigated in powder reuse studies. The first number represents the number of publications and the second number the percentage of the available of literature on powder reuse.

extent to which powder is consumed within a build. While it is commonly noted that spatter production is one of the leading causes of powder degradation [190, 191], the amount of melt area within a powder reuse cycle, and therefore amount of spatter generated, is also rarely commented on. As this is one of the main aspects influencing powder degradation with reuse, it is crucial that melt area is recorded during future research.

Despite these significant oversights, many pieces of literature do describe the powder reuse strategies employed with enough information to enable conclusions to be drawn. The following sections outline the three most commonly used powder reuse methods. Due to the differences between these reuse methods comparison is difficult, and so each will be treated independently throughout this review.

3.2.1 Single Batch and Collective Ageing Methods

The single batch method involves taking an initial batch of powder and reusing it in successive runs until there is no longer enough powder to complete a print. Figure 3.3 shows the amount of powder available for each successive run. Between runs the powder is sieved to remove agglomerates or sintered particles produced in previous runs, but no additional powder is added to the powder batch. Due to the reduction in powder available for subsequent builds, a reduction in build height or area is common [173, 184].

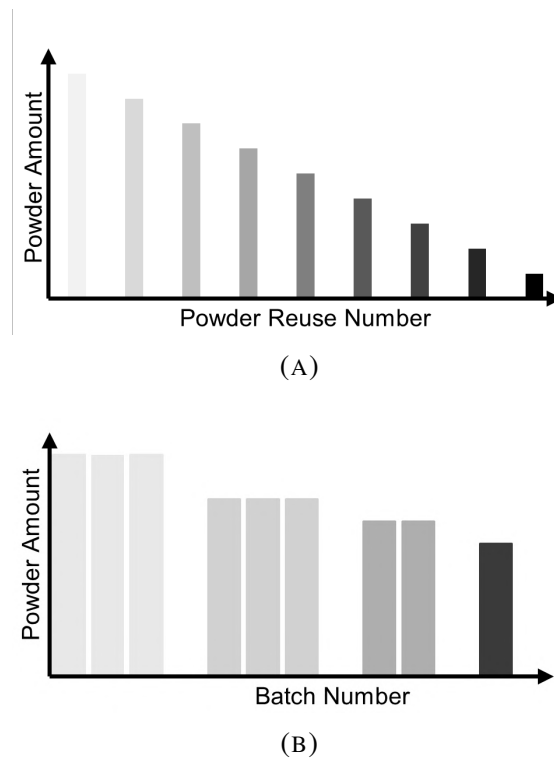


FIGURE 3.3. Graphical representation of the: a) single batch and b) collective ageing methods. A lighter colour represents relatively unused powder and a darker colour represents highly used powder.

The collective ageing method is similar to the single batch method, however, takes place on a larger scale. Here, multiple batches of powder are used to print builds, with powder of the same age being mixed together for use in subsequent builds. Therefore, one cycle is comprised of numerous builds using powder of the same age, as illustrated in Figure 3.3b.

The single batch method is the most commonly used method in academia due to the expected acceleration in powder degradation, attributed to the lack of introduction of unused powder [192]. The collective ageing method is less commonly used as similar levels of powder degradation take longer to occur, increasing study times. It is, however, a viable alternative to the single batch method in industrial settings for larger production runs.

These powder reuse methods are deterministic in nature as the processing history of all powder particles is both identical and known as powders of different ages have not been mixed. This gives the powder high traceability which is important when understanding the processing history and thermal treatments of a powder batch. Traceability is an especially important consideration in industries such as the aerospace industry, where quality control is one of the main factors that influences powder reuse [3, 178]. This deterministic nature of the single batch and collective ageing methods is another reason for their popularity within the literature.

3.2.2 Top Up Method

Another powder reuse method that has been employed is the top up method. This involves placing sieved or virgin powder on top of powder remaining in the powder supply chamber to maintain the level required for a new build. This powder is not mixed with the powder already present in the machine resulting in a layered powder bed, with different sections having different processing histories. An example schematic of the composition of builds using the top up method is shown in Figure 3.4, where it can be seen that builds of differing heights will especially affect the powder composition of following builds.

The top up method has relatively simple powder handling procedures as there is no mixing of powder batches, meaning the only handling involves powder sieving and drying. A reduction in powder handling is advantageous as it is thought that this is when the majority of powder oxidation occurs [193, 194].

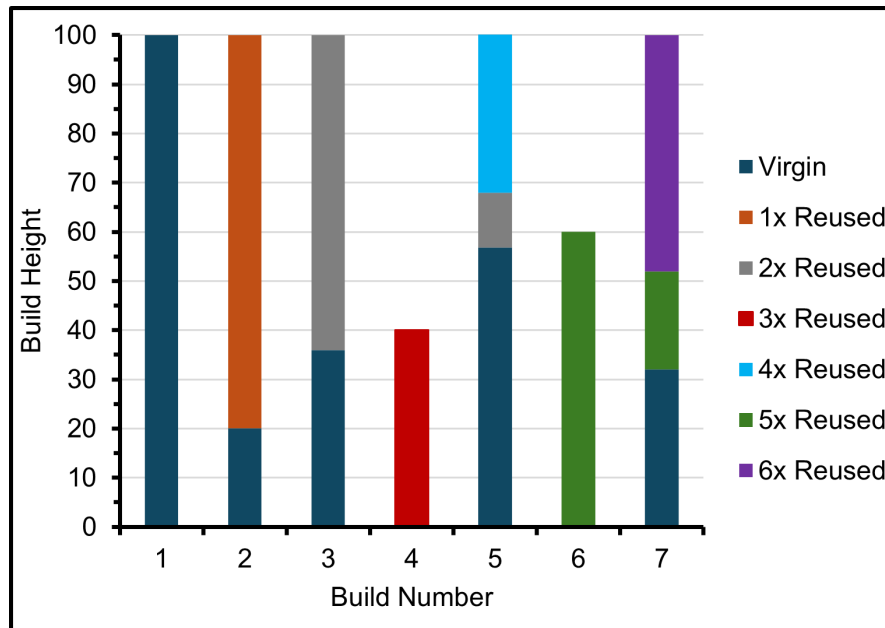


FIGURE 3.4. Graphical representation of the top up method. Each colour represents a powder that has been through the processing chamber a certain number of times, producing a layered build.

This method has poor traceability, with inconsistent layering of the powder bed making it difficult to determine the processing history of the powder bed, in turn complicating property prediction of parts manufactured using this powder. This poor powder traceability has meant that this reuse technique has seen only limited use within the literature.

3.2.3 Refreshing Method

The refreshing method is one of the more common powder reuse techniques employed in the literature. It involves completing a build and then replacing the material lost in the build and sieving processes with virgin powder, known as the ‘refreshing’ stage. This virgin powder is mixed in with the remaining powder to create a homogeneous mix. Refreshing can take place either after every build, or after a predetermined number of builds so that there is always enough powder for new builds. Schematics of these variations on the refreshing method are shown in Figure 3.5.

This method is considered probabilistic as the exact composition of each powder batch cannot be precisely determined due to the continual introduction and mixing of powder. The exact

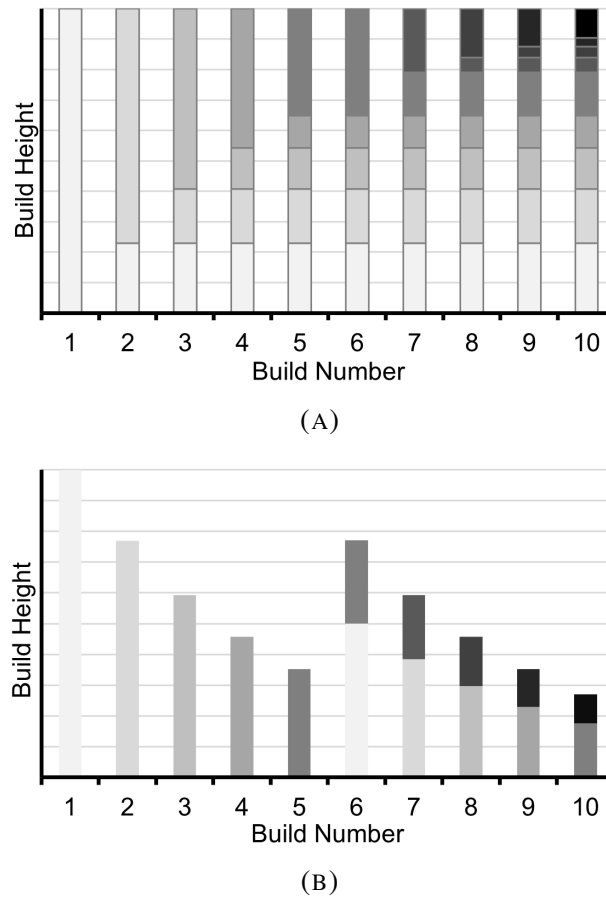


FIGURE 3.5. Graphical representation of the powder composition of builds when using the refreshing method: a) The continuous refreshing method b) The frequent refreshing method. A lighter colour represents relatively unused powder and darker colour represents highly used powder.

number of times each powder particle has been used cannot be known due to the differing histories of individual particles, rendering any interpretation of the powder composition probabilistic. This can be problematic in industries that require high traceability in their powder stock [178]. Frequent powder characterisation may help overcome this setback.

3.3 Common Properties Investigated in Powder Reuse Studies

Powder characteristics influence the final properties of manufactured parts [59, 62, 195] and as such, one of the key areas of interest in powder reuse studies has been the evolution of powder

characteristics with reuse. Various techniques are employed to investigate characteristics such as powder size, flow, shape, chemical composition, density and microstructure [68, 196]. Due to the significant range of tests available and the limited scope of individual research projects, most powder reuse studies focus only on a few powder properties.

While advanced powder characterisation techniques are crucial to furthering the understanding of the effects of powder reuse on powder particles, the current research is not yet comprehensive enough. As particle size distribution (PSD) and oxygen content are the most commonly measured powder characteristics, their analysis allows for a basic comparison between the majority of powder reuse studies while more comprehensive methods continue to advance in popularity.

Powder reuse remains an uncertain practice due also to the lack of clarity surrounding the properties of parts manufactured using reused powder [197]. Therefore, powder reuse studies will often also investigate the properties of parts such as tensile strength, fatigue strength, hardness, roughness, and density, the most common of these being tensile strength.

The following sections provide an overview of the three most commonly tested powder and part characteristics, explaining their testing, representation, and significance in powder reuse studies. Discussion on observed trends and relationships will be addressed in Section 3.4 of this chapter.

3.3.1 Particle Size Distribution

The PSD of a powder batch is commonly investigated due to the simplicity of measurement and its influence on properties [59, 61, 198]. PSD gives the range and quantity of particles within a sample and can be measured by many methods such as sieving, scanning electron microscope (SEM) imaging, laser diffraction, static image analysis, and dynamic image analysis. Each technique has advantages and disadvantages, and data from each cannot be directly compared as the particle size measured is somewhat dependant on the method used [68].

PSD is commonly measured as it is understood to have the potential to change as a powder stock is reused [176, 199]. This has been attributed to reasons such as the preferential

consumption of fine powders [200], the production of large spatter particles due to melt pool phenomena [159], and the creation of agglomerate particles [201].

There are a number of ways in which PSD data can be represented. Particle size is given in either volume or equivalent diameter and the two most common graphical representations are by a probability distribution function or a cumulative distribution, both represented in Figure 3.6.

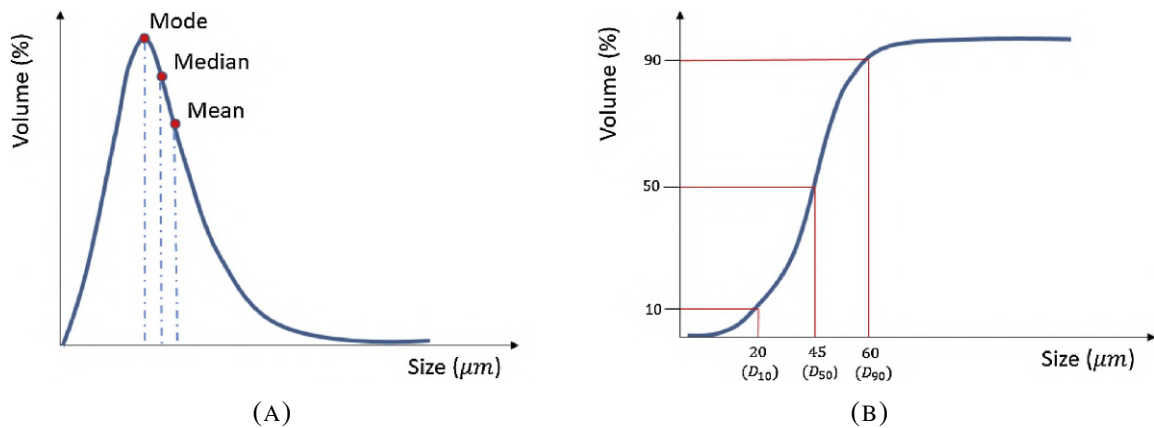


FIGURE 3.6. Graphical representation of the particle size distribution data: (a) a Gaussian probability distribution function, (b) a cumulative distribution (reproduced from [61]).

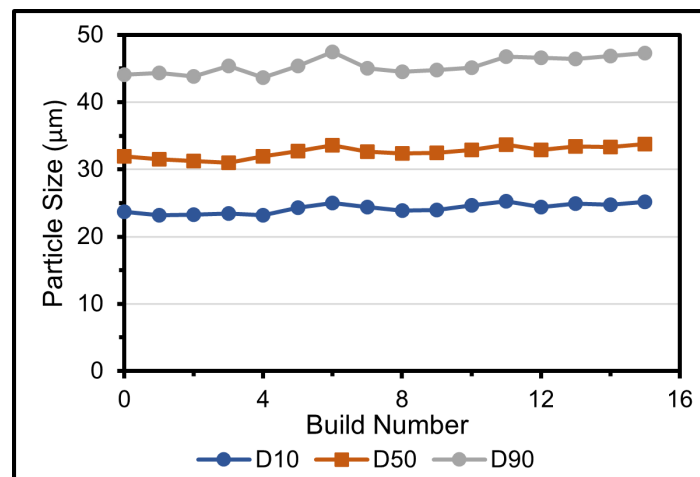


FIGURE 3.7. Graphical representation of D10, D50, and D90 values of a powder as they change during a reuse study (adapted from [200]).

PSD is numerically represented by D10, D50, and D90 values, each representing the diameter below which a certain percentage of particles, either 10, 50, or 90%, fall. For example, D10

= 15 μm means that 10% of particles have a diameter smaller than 15 μm . In a powder reuse study where powder sizes are expected to change, these values can be plotted for trend observation as demonstrated in Figure 3.7.

3.3.2 Powder Oxygen Content

A significant consideration in PBF is powder oxygen content, as this can influence properties such as the microstructure of powder and parts [47] as well as tensile strength [202]. This is especially important for titanium alloys at elevated temperatures [189] due to titanium's reactivity and affinity to pick up oxygen. For this reason, Ti-6Al-4V alloy powders have oxygen limits designated by ASTM F2924-14 (2021) [203], the standard dictating part and feedstock properties for powder bed fusion processes involving Ti6Al4V. This limits powder oxygen inclusion to 0.2%.

The oxygen content of powders can be measured using techniques such as inert gas fusion, combustion, and energy dispersive X-ray spectroscopy [68]. Such analysis typically yields results such as those displayed in Figure 3.8, which shows results from a powder reuse study conducted by Tang et al. [204].

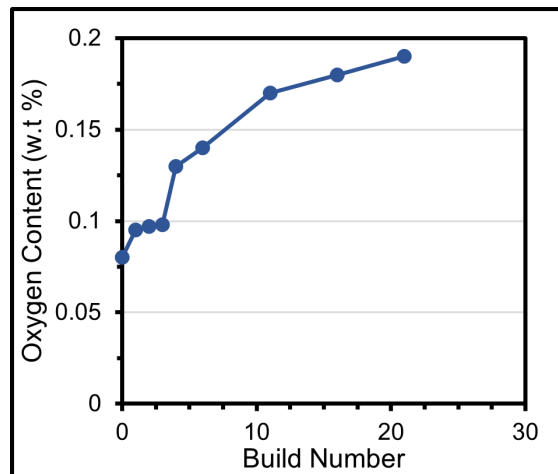


FIGURE 3.8. Typical example of mass oxygen content measurement from a powder reuse study (adapted from [204]).

While bulk oxygen content is an important consideration, oxygen is most commonly observed on the surface of particles and as such there is also interest in investigating powder surface chemistry, often using techniques such as X-ray photoelectron spectroscopy and Auger

emission spectroscopy. The results of similar surface investigations are shown in Figure 3.9. While changing powder chemistry, this growth in oxides also has the potential to change powder morphology, as seen in Figure 3.9a. These new localised growths, due to potential local chemical heterogeneities, reduce the circularity of particles, potentially influencing the flow of particles during powder spreading and therefore their packing within the build chamber [182].

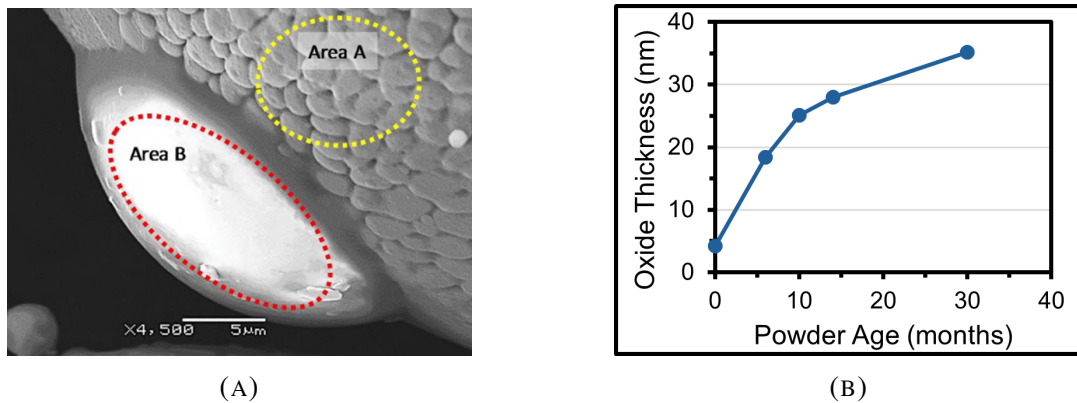


FIGURE 3.9. (a) SEM image demonstrating localised oxide concentration (reproduced from [205]) (b) Oxide layer thickness on AlSi10Mg powder (adapted from [194]).

3.3.3 Tensile Properties

Tensile properties are some of the most commonly investigated mechanical properties of additively manufactured materials. While different tension testing techniques are used throughout the literature, the extent of available data allows for comparison between studies to determine the effect of powder reuse methods on tensile properties.

Within powder reuse studies different tensile properties are investigated, the most common being tensile strength, yield strength, and ductility. However, tensile specimens are manufactured in a multitude of ways. Some authors use vertically [171] or horizontally [173] orientated specimens, giving different properties due to the columnar microstructure associated with PBF [206], while others manufacture specimens in multiple orientations to compare these anisotropic properties [64]. Samples have also been produced in a range of sizes using both standard [174] and non-standard specimen dimensions [204], and have undergone a variety of heat treatments post manufacturing. These factors naturally result in varying properties [207,

208] and therefore the comparative reliability of some published data could be questioned in the context of a comparative study.

3.3.4 Other Properties Commonly Investigated

While Sections 3.3.1 to 3.3.3 detail some of the most common properties of parts and powders investigated in powder reuse studies, there remain a plethora of tests conducted in other studies. Some of the most popular testing includes: powder flow and morphology due to their effects on the quality of the powder layer spread within the build chamber [209]; powder density and microstructure due to their effects on melt pool behaviour and final part density [26]; part density due to its prevalence as a goal in idealised PBF processing [23]; fatigue strength due to the extensive use of fatigue prone parts in applications such as in the biomedical industry [210]; and hardness due to the simplicity of testing and its relation to properties such as tensile strength. These characteristics and properties are tested due to their importance to both the manufacturing process and part performance [68, 109, 211, 212]. While these, among other, properties are tested to truly understand the effect of powder reuse in PBF, they have not been widely studied and not enough data for each powder reuse method is available to realise a substantial comparison of reuse techniques. Therefore, only some of these characterisation techniques/properties are touched upon throughout this review.

3.4 The Effects of Powder Reuse Methods on Common Properties

This section will evaluate the current literature in relation to the effect of powder reuse on powder and part properties. Ultimately, the acceptability of powder reuse relies on understanding its effects on both powder and part properties and as such a holistic understanding is required based on multiple studies covering a range of materials.

3.4.1 The Single Batch and Collective Ageing Methods

3.4.1.1 Particle Size Distribution

In single batch and collective ageing methods the entire powder batch has exactly the same processing history with no new powder introduced throughout the powder's life cycle. As such, it is expected that there will be some changes to PSD. The literature relating to the single batch and collective ageing powder reuse methods presents a range of results typically correlating to the material studied.

Steel alloys have the greatest range in results. A number of studies found that all the D10, D50 and D90 values increased by approximately 2% to 7% [173, 184, 200]. The most significant of these findings was made by Sutton et al. who observed that the D5 value of AISI 304L increased from 3.28 μm to 14.53 μm , which was attributed to the continual consumption of fine particles which were not replaced, whereas large particles are continually generated as spatter and replace those consumed in the build process [184]. This reduces the relative number of fine particles within the powder batch, thereby increasing the D5 and D10 values. In contrast to this, other studies on steel powders observed a decrease in all powder size measurements [171, 213], with each value decreasing by approximately 1% to 8%. However, both these studies consider these results to be insignificant with the change in PSD attributed to the fine sieve size used by each. This removed the majority of large particles formed as spatter or remaining from the virgin powder, reducing the overall size of the powder and therefore the PSD. Other studies fall in between these two extremes, with changes in particle size dependant on the size range of the particles themselves. Sartin et al. found a statistical difference in the PSD of 316L stainless steel, where the D10 value of the powder decreased while the D90 value increased, leading to a wider PSD [214]. These results together indicate that the reuse of steel alloys will change the PSD of a powder batch due to factors such as sieving, with reuse exacerbating changes induced by these processes.

When Ti-6Al-4V powders have been reused, a narrowing of the PSD has been regularly observed. Alamos et al. observed a noticeable reduction in both fines and coarse powders after the first build cycle, after which the PSD remains relatively constant. This was attributed to sieving removing large particles and smaller particles being lost as dust clouds during handling and sieving operations [162]. Quintana et al. added to this explanation by hypothesising that

the reduction in large particles was due to the breakdown of satellite particles during sieving [65]. Work by Ghods et al. showed the most significant reduction in PSD range, with the measured D90 value reducing 23% from 110 μm to 84.3 μm [215]. The authors attributed this reduction in D90 to sieving, and a slight increase in D10 was again thought to be due to the preferential consumption of fine particles and the ease at which they are lost during sieving and handling procedures, a similar conclusion to Soltani-Tehrani et al. [190]. Other studies have found alongside a reduction in fine particles an increase in large particles, such as the study conducted by O'Leary et al. [216]. Here all measurements of powder size increased due to the increased presence of large particles as a result of agglomeration and spatter.

Studies conducted on Inconel 718 are inconsistent in their findings, with some, such as those conducted by Gruber et al. [217] and Rock et al. [218], finding that the PSD of Inconel 718 narrows [217] or expands [218]. Gruber et al. observed an increase in D10 size of 15% from 19.62 μm to 22.63 μm after 20 builds and a decrease in D90 size of 9% from 55.23 μm to 50.3 μm . Similar explanations as those presented in other studies are given for these changes, with a reduction in fine particles occurring due to preferential consumption and loss during powder handling and processing, and the creation of satellite particles from fine particles. Rock et al. found that large particles were created by the melting procedure at a fast enough rate to increase the D90 value of the powder with constant reuse, thereby increasing the range of the PSD [218]. This suggests that the laser-melt pool interaction will influence PSD, with parameters such as laser power and scan speed affecting the number and size of new particles created. The only consistency in studies on Inconel 718 was an increasing D50 size [217–220].

Limited research has been conducted on the effect of powder reuse on aluminium alloys using a single batch powder reuse method, with works by Del Re et al. [221] on AlSi10Mg and Smolina et al. [222] on AlSi7Mg0.6 being the only pertinent studies. Del Re et al. observed a decrease in all powder size measurements across nine builds, with a decrease in mean size of 15% from 32.09 μm to 27.19 μm , a similar reduction in the D50 of 10% from 27.7 μm to 24.9 μm , and a reduction of 9% from 12.8 μm to 11.7 μm for the D10. These reductions were attributed to a lower number of large particles in the first few builds as these larger particles were sieved out. Contrastingly, Smolina et al. found no significant changes in any powder size measurements, with SEM imaging showing near identical particle size and shape. It

was noted by Del Re et al. [221] that different sieving practices could give different results, suggesting that the sieving method and size employed may influence the change in PSD. This aligns with the conclusions of many studies on Ti-6Al-4V, where sieving was noted as a significant reason for PSD change.

The range of results observed in the literature indicate that there is no simple resolution to the question of whether PSD changes under a single batch or collective ageing powder regime. Factors such as build volume and laser parameters influence the amount and size of new powder produced during the build process, and powder handling and sieving procedures affect the number of large particles removed and small particles lost. These need to be more carefully monitored and their influence on PSD better understood to gain insight into their effect during powder reuse. Regular recording of the sieve size used in reuse studies and analysis of sieved waste powder would greatly aid in this matter.

Also of importance is material type. While changes in large particle presence can be manipulated through a deliberate choice of sieve size, fine particles tend to be lost as dust during powder handling procedures, which is particularly evident for Ti-6Al-4V. These preliminary results suggest that lighter materials may be more prone to losing finer particles due to their lower density and the ease at which this powder is disturbed, resulting in an increasing PSD the more a powder is reused.

From these studies no clear trend can be found regarding how PSD changes when subject to a single batch or collective ageing powder reuse regime. While the majority of powder size measurements increased in each of these studies, there were a considerable number of values that saw statistically significant decreases. The only consistency appears in studies on Ti-6Al-4V where PSD regularly narrowed. It is clear that further work is required with more consistent powder reuse methodologies to confirm any trends evident in powder reuse studies.

3.4.1.2 Powder Oxygen Content

The chemical composition of powders in PBF is carefully monitored, most notably oxygen content. A number of single batch and collective ageing powder reuse studies have investigated the bulk oxygen content of powder, and their results are displayed in Figure 3.10.

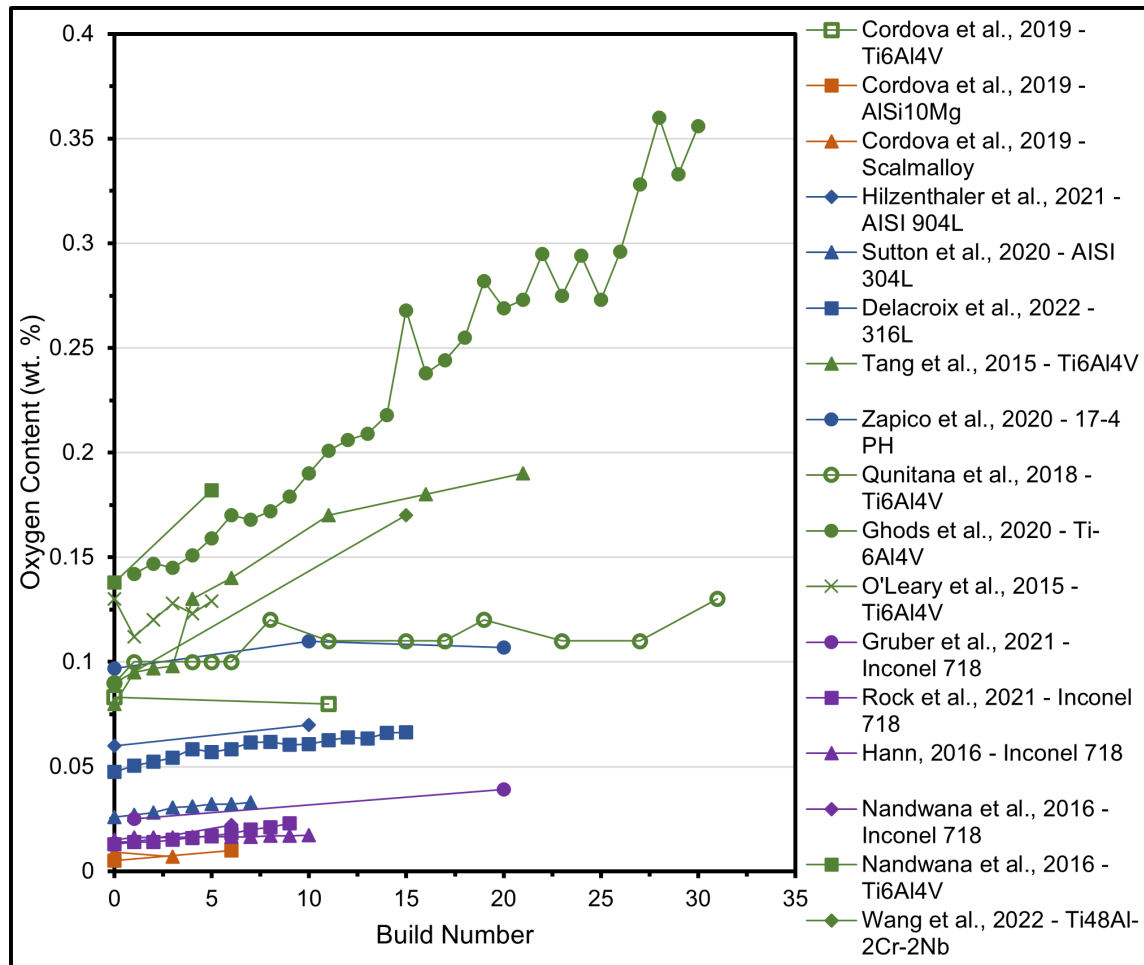


FIGURE 3.10. Plot of oxygen content of powders observed in single batch and collective ageing powder reuse studies. Green points represent titanium alloy data, blue points represent ferrous alloy data, purple points represent nickel alloy data, and orange points represent aluminium alloy data. All measurements are in wt.%, and data is drawn from studies as referenced [65, 166, 171, 184, 200, 204, 205, 215–220, 223].

The majority of studies demonstrate an increase in the oxygen content of powder, however the extent to which this occurs varies. The notable exception to this finding was research by Cordova et al. which showed minor decreasing oxygen content with powder reuse for Ti-6Al-4V and Scalmalloy [166]. These findings were due to a low number of reuse cycles and measured values falling within their respective scatter bands, rendering these results statistically insignificant.

The increase in oxygen content has been attributed to the affinity of some materials to react with oxygen, especially those using aluminium or titanium-based alloys. Cordova et al.

found that the oxygen content in AlSi10Mg almost doubled over six production runs [166] and attributed this to the low free Gibbs energy associated with the production of Al_2O_3 [224]. These authors contrastingly found that Scalmalloy did not experience the same level of oxygen pick up, however this was due to the low number of builds conducted as used and virgin powder characteristics were very similar. They hypothesised that further reuse of this aluminium based alloy would see an increase in oxygen content similar to AlSi10Mg. A similar idea was presented by Cao et al. in their study of Ti-6Al-4V. These authors found that aluminium preferentially oxidated on the surface of powder particles due to its affinity for oxygen and low free Gibbs energy [29]. Therefore, the amount of oxidation and its form depends on the affinity for alloy constituents to react with oxygen. However, even powders with higher oxidation resistance can increase in oxygen content with reuse [225], with studies on nickel alloy powders observing a doubling in oxygen concentration [30], implying that oxygen affinity is not the only factor to be considered.

Most authors explain that the increase in the oxygen content of powder is due to the accumulation of spatter within the powder bed during the melting process. Many studies have found that the largest particles within samples have the highest oxygen content [65, 184, 205, 217, 218] and so its continual generation increases the overall powder oxygen content. This is agnostic of material type. Spatter oxidation was emphasised by Quintana et al. [65] who observed that these particles were more prevalent near melted parts, and Soltani-Tehrani et al. [190] found that spatter was more prevalent near the gas outlet, suggesting that the laser melting process and associated spatter production is a major contributor to increasing oxygen levels. The prevalence of this theory within the literature suggests that spatter is likely the main source of oxygen pick up in powders. This is as molten material, such as spatter when ejected from the melt pool, has a higher affinity to oxygenate than solid powder and so its generation will lead to a noticeable increase in powder oxygen content.

The other main source of oxygen is atmospheric conditions during powder handling procedures such as removal of powder from machines and sieving. This exposure to air allows for both absorption of atmospheric oxygen [204] as well as adsorption of atmospheric moisture [220]. Water molecules adsorbed from the atmosphere break down under elevated temperatures, increasing powder oxidation in the build chamber. This method of oxygen pick up is commonly quoted in powder reuse studies [204, 215, 220, 226], however tends to be

associated more regularly with materials that readily oxidise, such as titanium and aluminium alloys. This indicates that this method of oxygen pick up should be further investigated in less reactive materials to determine if it is a relevant mechanism for these alloys, even if on a smaller scale.

It is evident that the majority of powder reuse studies employing the single batch or collective ageing methods exhibit increasing oxygen content, and this is most prevalent in titanium alloys. The most common reasons for this include oxygen and moisture absorption and adsorption during powder handling processes, and spatter accumulation within the powder bed. As this powder is not replenished during its lifetime, it would be expected that the constant increase in spatter and continual exposure to atmosphere would cause the powder oxygen content to increase at a consistent rate. However, this does not seem to be the case. Most studies surveyed showed that while powder oxygen content continued to increase during later builds, it was often at a decreasing rate. Since spatter particles increase at a consistent rate, assuming constant build geometries and processing parameters, the rate of oxidation for other powder particles must therefore reduce. Many alloys are able to form oxidised surface layers meaning that after a certain level of oxidation further growth is hindered [83, 182]. This suggests that the oxidation of a powder batch may plateau when a certain level of oxide growth is complete, however this peak depends upon the material used according to its potential for oxidation.

3.4.1.3 Tensile Properties

Changes in tensile properties due to powder reuse are often attributed to changing material characteristics such as oxygen content, part density, void density and type, and surface roughness [204, 227]. As such, a range of responses have been observed in the literature regarding the tensile behaviour of materials when utilising a single batch or collective ageing method of powder reuse.

Minimal data exists in the current literature regarding the elastic modulus of materials during single batch or collective ageing powder reuse studies. Data from these few studies are displayed in Figure 3.11. Hilzenthaller et al. found in their study of AISI 904L superaustenitic steel that the change in elastic modulus was within the error range for the tests and so powder reuse was found to have no effect on this value [171]. Schur et al. found similar results as

their measured values for the elastic modulus of Ti-6Al-4V saw insignificant change [64]. Therefore, preliminary results suggest that utilising a single batch or collective ageing powder reuse method will have no effect on the elastic modulus of materials. This is an expected result due to the reliance of elastic modulus on the chemical composition of a material, which is not expected to change to a high enough degree during powder reuse to bring about noticeable changes in the elastic modulus.

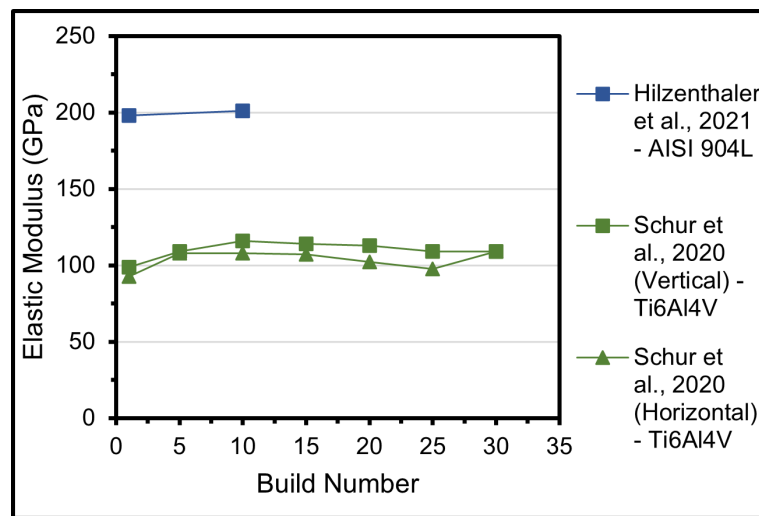


FIGURE 3.11. Elastic modulus observed in single batch and collective ageing powder reuse studies [64, 171]. Green points represent titanium alloy data, and blue points represent ferrous alloy data.

Yield strength is more commonly investigated, and the results of single batch and collective ageing studies are shown in Figure 3.12. The majority of results indicate only minor changes in yield strength. Most studies do not provide explanations as to why the yield strength remains constant with reuse [162, 171, 219]. Delacroix et al. found no increase in the yield strength in their study of 316L stainless steel despite observing grain refinement in their specimens with the average grain size decreasing from 108.5 μm to 80.5 μm over 14 builds, a decrease of 25.8% [200]. The Hall-Petch relationship indicates that this reduction should increase the yield strength of the material, as has been observed in other additively manufactured parts [109]. However, the lack of change in strength suggests that yield strength was influenced by more than simply grain size. Relatedly, other studies have found no change in the yield strength attributed to the unchanging chemical composition of the powder [213]. In contrast, it has also been reported that yield strength decreases with powder reuse, though explanations for these observations are unclear [221, 228]. It has, however, been more commonly reported

that yield strength increases with powder reuse, which in reactive materials such as titanium alloys has most commonly been associated with increasing oxygen content. This is most clear in research conducted by Schur et al. [64], who found that over 30 build cycles with Ti-6Al-4V the yield strength increased from 739 MPa to 1050 MPa. This was due to the increase in oxygen content of the parts, with oxygen atoms impeding slip during loading of the samples. Similar proposals have been put forward by others in the literature [65, 190, 204], with explanations for increased yield strength including increased dislocation density [225]. A comparison of single batch and collective ageing studies shows there is no clear relationship between yield strength and the age of powder, with the majority of the literature finding no change in yield strength as powder is reused. The exception to this appears to be Ti-6Al-4V where most studies observed an increase in yield strength due to the increased presence of oxygen.

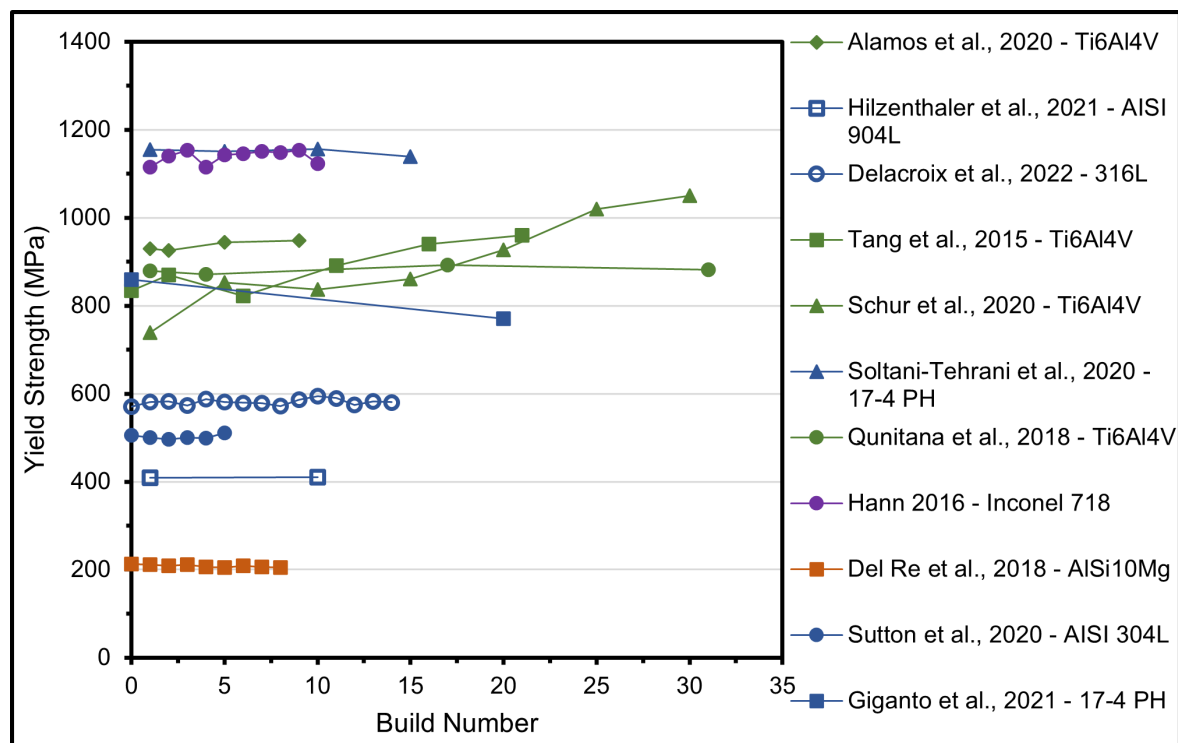


FIGURE 3.12. Yield strength of samples in single batch and collective ageing powder reuse studies [64, 65, 162, 171, 184, 200, 204, 213, 219, 221, 227, 228]. Green points represent titanium alloy data, blue points represent ferrous alloy data, purple points represent nickel alloy data, and orange points represent aluminium alloy data.

The results of tensile strength follow very similar patterns to those of yield strength, with most studies finding no change with powder reuse as seen in Figure 3.13 [30, 162, 171, 213, 219, 222]. This lack of change in tensile strength has been attributed variously to a good choice of process parameters [200] or only small changes in powder oxygen content [65], although these findings could easily be due to the limited number of reuse cycles conducted in these studies. However, a few studies have found that tensile strength increases with powder reuse. Schur et al. [64] observed an increase in tensile strength in Ti-6Al-4V from 857 MPa to 1120 MPa in the horizontal direction and from 859 MPa to 1130 MPa in the vertical direction, both over 30 builds. This occurred simultaneously to an increase in yield strength and powder oxygen content. Similar conclusions were drawn by Tang et al. [204], where increased tensile strength was also attributed to increased oxygen content. The fact that both these studies used Ti-6Al-4V suggests that the changing oxygen content, likely due to increased powder oxygen content during reuse, is the cause for this increase in tensile strength and so is most likely an

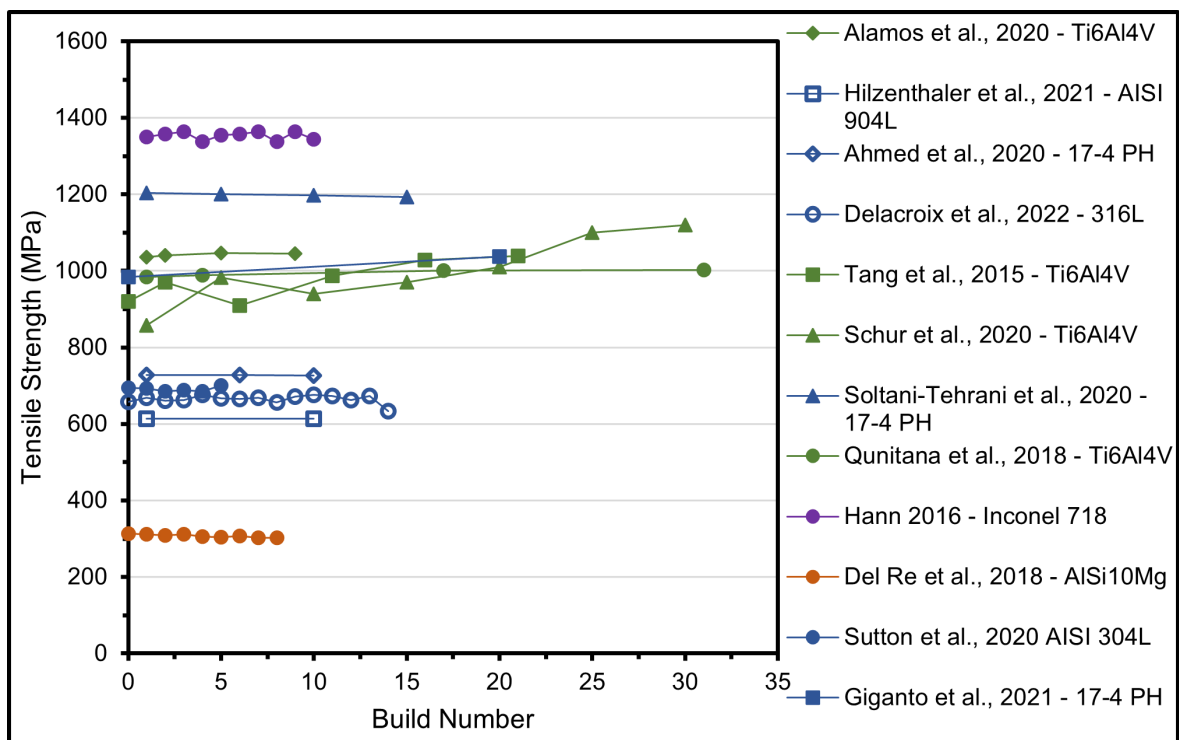


FIGURE 3.13. Tensile strength of samples in single batch and collective ageing powder reuse studies [64, 65, 162, 171, 173, 184, 200, 204, 213, 219, 221, 227, 228]. Green points represent titanium alloy data, blue points represent ferrous alloy data, purple points represent nickel alloy data, and orange points represent aluminium alloy data.

issue in reactive materials. A review of these studies suggests that the utilisation of a single batch or collective ageing powder reuse strategy will have little to no effect on the tensile strength of specimens, with the material used having a significantly greater role.

Changes in elongation with powder reuse follow accepted material science theory in relation to strength properties. Holistically, the literature is divided as to whether powder reuse using a single batch or collective ageing method will increase or decrease the ductility of printed specimens, with many studies observing increases in elongation [65, 171, 213, 221, 227, 228] while others observe decreases [30, 64, 162, 173, 190, 200, 204, 219]. However, most of these values fall within the standard deviations of measured values and so are deemed statistically insignificant. For those finding changes in elongation, typically increases in strength correspond to reductions in elongation, and vice versa. This was most evident in the study by Schur et al. on Ti-6Al-4V [64] where the significant increases in strength observed corresponded with a reduction in elongation at failure from 9.28 % for virgin powder to 3.53

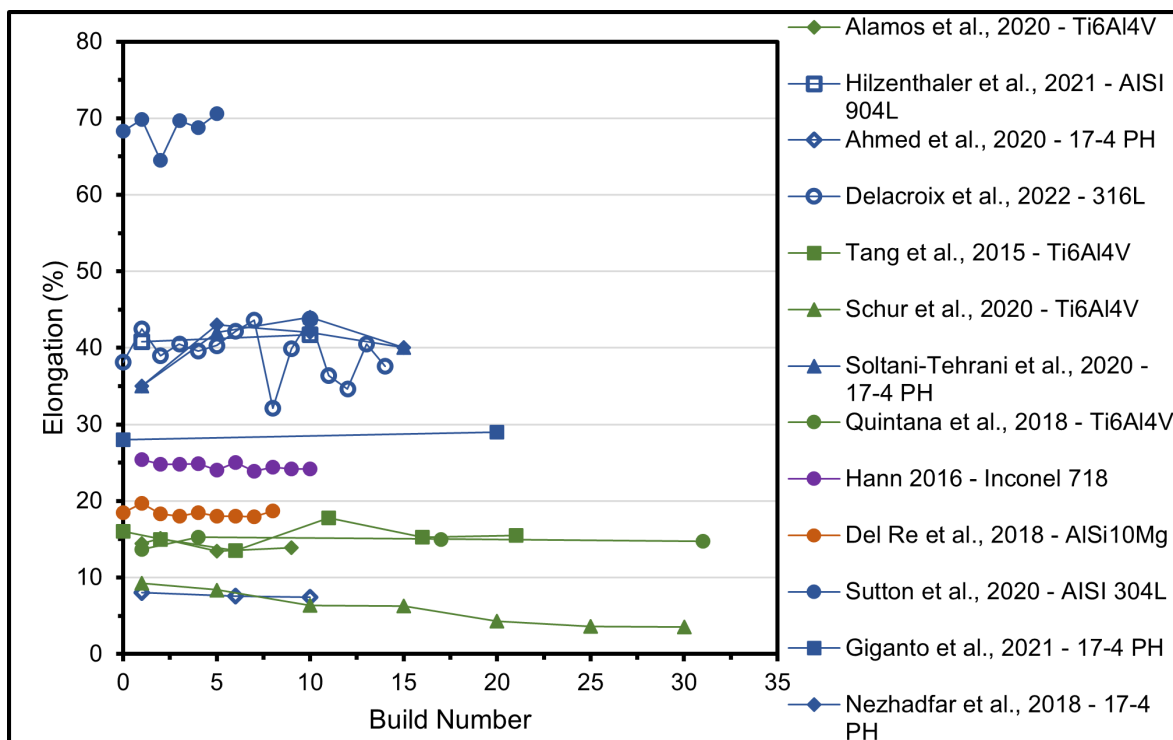


FIGURE 3.14. Elongation at fracture of samples in single batch and collective ageing powder reuse studies [64, 65, 162, 171, 173, 184, 200, 204, 213, 219, 221, 227–229]. Green points represent titanium alloy data, blue points represent ferrous alloy data, purple points represent nickel alloy data, and orange points represent aluminium alloy data.

% for powder reused 30 times. Similarly, Alamos et al. [162] observed a small decrease in elongation in Ti-6Al-4V from 14.45 % to 13.88 % after nine reuse cycles which matched a slight increase in both yield and tensile strengths. These results have been mirrored in a number of other studies [204, 213, 221]. Two main reasons have been given for changes in the elongation of specimens, and these tend to be material dependant. In studies on stainless steels, specifically 17-4 PH, the main explanation for elongation change is porosity. Soltani-Tehrani et al. [213] proposed that a slight increase in ductility occurred due to a reduction in part porosity, itself a by-product of a reduction in powder cohesion allowing for better spreadability in the powder bed. A similar view was espoused by Nezhadfar et al. [229]. Ahmed et al. [173] contrastingly found that ductility decreased as porosity increased over their ten builds with 17-4 PH, attributing ductility change to particle contamination and poor powder flowability. This implies that changing ductility has a more complicated relationship with porosity than sometimes suggested. The other proposed mechanism for elongation change is oxygen inclusion, as oxygen has been seen to lead to embrittlement, thereby reducing ductility [64, 162]. This view, however, has been limited to studies on Ti-6Al-4V. Since the majority of studies have found either no or negligible change to elongation while using a single batch or collective ageing powder reuse method, these strategies can be seen to not significantly influence the ductility of parts while under tensile loading.

3.4.1.4 Other Significant Properties

While PSD, powder oxygen content, and tensile properties are commonly investigated, a number of other significant properties are also tested within the literature. Some of these properties are discussed here as a preliminary assessment on the effect that powder reuse has on them. However, further data is required to provide meaningful conclusions.

Some of the more extensively studied properties in single batch and collective ageing powder reuse studies relate to powder flow and packing, such as the tap and apparent densities, flow times, and avalanche angle. While these properties often rely on particle size, distribution, and shape, their independent measurement remains important [196, 230].

The tap and apparent densities of powders are often investigated to understand how a powder will settle on the build plate after spreading. The typical response for powders undergoing a single batch or collective ageing powder reuse regime is to increase the tap and apparent

densities with reuse. This was observed by Gruber et al. who found that the tap density of Inconel 718 increased from 4.83 g/cm^3 to 5.06 g/cm^3 and apparent density increased from 4.28 g/cm^3 to 4.61 g/cm^3 over 20 builds [217], which was attributed to a narrowing PSD as well as a reduction in fine particles in the powder bulk. Similar conclusions have been drawn in other studies [184, 213, 221, 229], however these findings are limited to stainless steel and aluminium alloys. This increase in tap and apparent densities theoretically results in denser powder packing in the build chamber, allowing for parts containing lower porosity. In contrast to these studies, Tang et al. [204] found no significant changes in tap or apparent densities over 21 reuses of Ti-6Al-4V, and Quintana et al. [65] observed a slight decline in the tap density of Ti-6Al-4V powder. These contrasting results show that further research into the tap and apparent density of reused powders is required to gain conclusive results, as it has the potential to be materially dependant.

Powder flowability is important to understand as build times and powder bed density rely on it. This can be measured in a number of ways [230]. When utilising the avalanche angle studies tend to find that this angle decreases as powder is reused, representing an increase in flowability. Sutton et al. [184] observed a reduction in avalanche angle from 34.8° to 33.3° over seven builds for AISI 304L stainless steel, and Hilzenthaller et al. [171] found for AISI 904L stainless steel a more pronounced decrease from 43.9° to 35.4° after ten builds. In contrast to this Smolina et al. [222] observed no change in the cohesive index or avalanche angle of reused AlSi7Mg0.6 powder, likely due to the lack of change in powder morphology and size. However, this may also be influenced by the limited number of reuse cycles conducted. Studies focused on nickel, titanium, and aluminium based alloys have found that flow improves with powder reuse using a variety of methods such as Hall flow and flow energy [65, 177, 204, 226]. Explanations for these improvements in flow behaviour include the increased circularity of particles [184], a reduced numbers of fine particles [65, 184, 226], a reduction in powder moisture content [65], and a reduction in particle surface roughness due to wear from powder handling and the formation of smooth oxides [171, 184]. Few instances in the literature have found that powder flow decreases with reuse, with Ahmed et al. having the most significant findings with an increase in flowability energy of $\approx 35\%$ over ten builds with 17-4 PH stainless steel [173]. This was attributed to a reduction in particle sphericity and therefore greater interparticle friction during flow. Together, these findings suggest that a single batch or collective ageing powder reuse method will most likely result in

an improvement of powder flow properties regardless of the material used. However, a greater examination of powder flow is needed in the literature to validate this conclusion. This would be greatly aided by a wider uptake of more sophisticated powder characterisation techniques such as powder cohesion and avalanche angle measurements.

Fatigue strength is an important property of materials, especially in aeronautical industry applications [219], yet it remains rarely studied in single batch and collective ageing powder reuse studies. The few studies investigating fatigue strength have largely found that it is not affected by powder reuse in steel, titanium, nickel, and aluminium alloys [167, 213, 219, 221]. Only in one case has fatigue strength improved with powder reuse, in the specific case of high cycle fatigue testing using machined specimens of 17-4 PH stainless steel [213]. In the same study, low and medium cycle fatigue strengths were found to remain stable for both machined and as-built specimens, likely due to the inherent uncertainty regarding cyclic testing due to its reliance on porosity [221]. Therefore, these limited studies indicate that fatigue strength is unlikely to be affected by single batch or collective ageing powder reuse methods, although significant research is required to draw conclusions.

Part roughness is not often investigated during powder reuse studies. However, it can have serious implications, especially for components undergoing cyclic loading. Quinn et al. [178] found that over 11 builds the average roughness of 316L stainless steel parts increased 48% from 13.2 μm to 19.6 μm , increasing beyond the material data sheet specification of 13 ± 5 μm . Similarly, Barile et al. [231] observed an increase in the average surface roughness of AlSi10Mg parts from 10.2 μm to 10.8 μm over an unknown number of reuse cycles. In these, amongst other studies, surface roughness is seen to increase with powder reuse typically due to changing powder flow, shape, and roughness conditions [178, 221, 228, 231]. However, these observations are limited to steel and aluminium alloys and so the body of research is not yet large enough to provide generic conclusions.

3.4.1.5 Summary

The findings of Sections 3.4.1.1 to 3.4.1.4 are summarised in Table 3.1. This provides a brief overview of all the properties tested in various single batch and collective ageing powder reuse studies. Each property investigated in powder reuse studies is listed in Tables 3.1-3.3, and if a particular property is investigated by a reuse method, it is assigned a symbol. A minus

sign means the property reduces in value (e.g., reduction in yield strength), an equal sign means no change occurs, and a positive sign means the property increases (e.g. improvement in powder flowability). If there is an exceptional change in a property, the symbol is doubled. A blank cell means no data was collected for a particular property and material combination in that reuse method.

TABLE 3.1. Summary of powder reuse study findings for the single batch and collective ageing powder reuse methods. A plus sign indicates an improving or increasing value, a minus sign a declining or reducing value, an equal sign indicates no change to a property, and a blank space means not enough information was available for a judgement to be formed. Double of either sign represents an exceptional change to a property.

Alloy Base	PSD			Oxygen			Tension			Other Properties			
	D10	D50	D90	Oxygen Content	Elastic Modulus	Yield Strength	Tensile Strength	Elongation at Failure	Apparent/Tap Densities	Flow	Fatigue Strength	Roughness	
Ni	=	+	=	+		=	=	=	+	+	=	=	
Fe	=	=	=	+	=	=	=	=	+	+	=	+	
Ti	+		-	++	=	+	+	=	=	+	=	=	
Al				+		=	=	=	+	+	=	+	

TABLE 3.2. Summary of powder reuse study findings for the top up powder reuse method. A plus sign indicates an improving or increasing value, a minus sign a declining or reducing value, an equal sign indicates no change to a property, and a blank space means not enough information was available for a judgement to be formed. Double of either sign represents an exceptional change to a property.

Alloy Base	PSD			Oxygen			Tension			Other Properties			
	D10	D50	D90	Oxygen Content	Elastic Modulus	Yield Strength	Tensile Strength	Elongation at Failure	Powder Bed Density	Powder Chemistry	Part Density		
Fe	=	=	=	=	=	=	=	=	+		=		

TABLE 3.3. Summary of powder reuse study findings for the refreshing powder reuse method. A plus sign indicates an improving or increasing value, a minus sign a declining or reducing value, an equal sign indicates no change to a property, and a blank space means not enough information was available for a judgement to be formed. Double of either sign represents an exceptional change to a property.

Alloy Base	PSD			Oxygen Content	Tension			Other Properties	
	D10	D50	D90		Elastic Modulus	Yield Strength	Tensile Strength	Elongation at Failure	Hardness
Ni				=					
Fe									=
Ti				+		=		+	+
Al				+		-	-	-	-

3.4.2 The Top Up Method

While the top up method remains rarely used in the academic world, it represents the simplest way to reuse powder for PBF processes. As such, the following sections review the current state of the literature regarding how this powder reuse method influences powder and part properties. It is, however, important to note that due to a lack of powder blending any powder properties are highly dependent on powder sampling practices.

3.4.2.1 Particle Size Distribution

Due to the layering of powders when utilising the top up method, it is expected that PSD measurements will vary. The D10 values measured in powder reuse studies are displayed in Figure 3.15. Both the steels reused by Contaldi et al. [232] showed notable increases over nine reuse cycles, with the D10 value for 17-4 PH increasing from 18.6 μm to 21.2 μm and for 15-5 PH increasing from 19.5 μm to 21.3 μm ; increases of approximately 14% and 9%, respectively. This was contrasted by the study by Jacob et al. [182] that found a slight size reduction from 23.98 μm to 22.62 μm over eleven cycles. Contaldi et al. attributed this reduction in finer particles to their tendency to stick to parts and therefore be removed from the build cycle, as well as the increased likelihood for these particles to be blown away by the protective gas flow. Jacob et al. did not see a significant enough change to the D10 value to propose reasons for its variation. The contrasting finding of these two studies and the limited

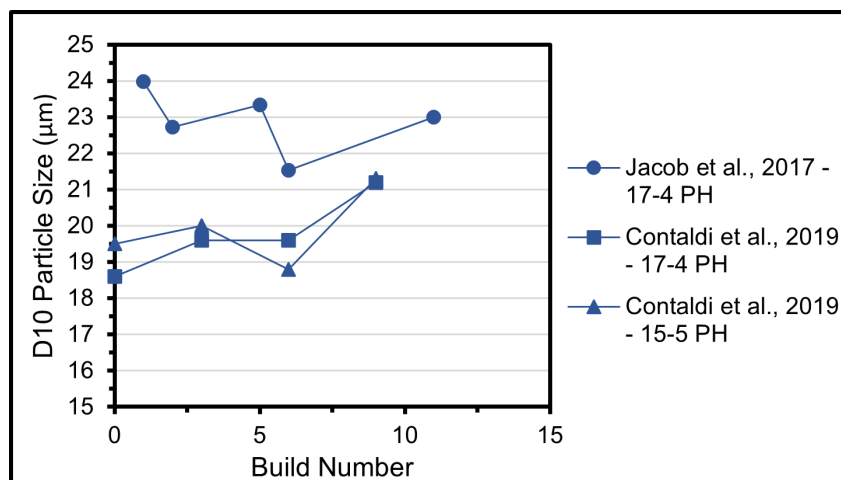


FIGURE 3.15. D10 value of top up powder reuse studies [182, 232].

variety in materials and PBF processes means that no significant conclusions can be drawn regarding how the D10 value of a powder batch will change while using the top up method.

The D50 values observed within the literature show minimal variation and therefore demonstrate that the D50 value does not significantly change with powder reuse, with results shown in Figure 3.16. Powders investigated by Jacob et al. [182] and Contaldi et al. [232] changed less than 5% from their virgin sizes. While the current research shows that D50 size does not change when using a top up powder reuse method, the range of literature is so far too small to draw general conclusions.

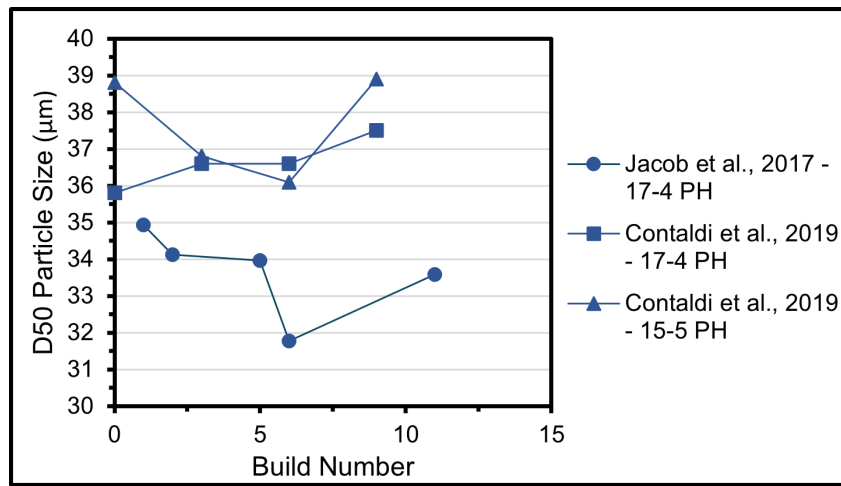


FIGURE 3.16. D50 value of top up powder reuse studies [182, 232].

The results of investigation into the D90 values of reused powders from the top up method are shown in Figure 3.17. In a similar manner to the D50 values from the same studies there is no clear increase or decrease in the D90 values, with Contaldi et al. observing changes in size no greater than 1.6 µm [232] and Jacob et al. finding that all changes to the D90 values fell within their reported measurement uncertainty [182]. Therefore, there are no changes to D90 size when using the top up powder reuse method, however, there is not enough data within the literature to draw generic conclusions.

Along with the inconsistencies in changing D10, D50 and D90 values, the change in PSD shape is also inconsistent. Contaldi et al. [232] found that there was no change in PSD shape for 17-4 PH stainless steel but in the same study found that the PSD of 15-5 PH stainless steel shifted towards larger particle sizes, attributed to the reduction in fines. The stability of the D90 value also aided this shift. Since only one out of the three top up powder reuse cases

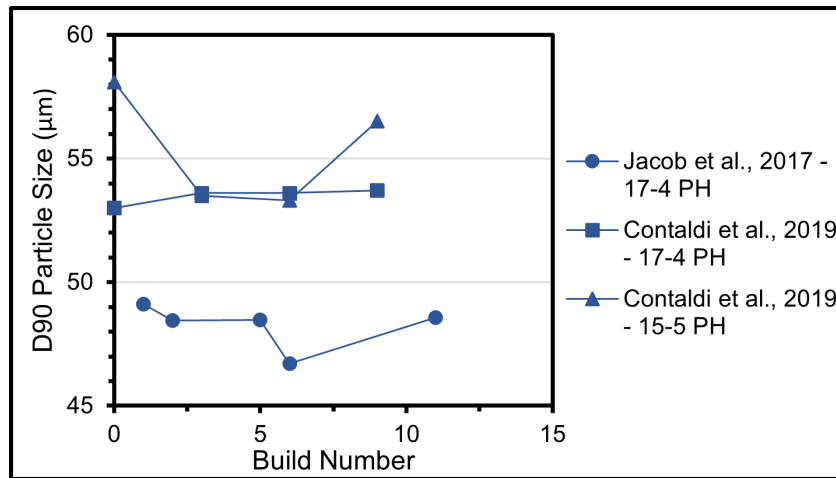


FIGURE 3.17. D90 value of top up powder reuse studies [182, 232].

found a change in PSD shape, based on these studies it is impossible to conclude a trend. These results indicate a significant oversight in the literature; the top up reuse method requires a significant increase in research output.

3.4.2.2 Powder Oxygen Content

Although both studies investigating the top up method of powder reuse investigated the changing chemical composition of their powders, neither monitored the change in oxygen content as a function of reuse time [182, 232]. This means that further research is required to determine the effect the top up powder reuse method has on the oxygen content of powders for PBF.

3.4.2.3 Tensile Properties

In the two studies utilising a top up powder reuse strategy the tensile properties of the materials are well documented, and their results are presented in the following paragraphs.

The elastic modulus is investigated in the study by Jacob et al. on 17-4 PH stainless steel [182], the results of which are shown in Figure 3.18. While the elastic modulus slightly increases over the first six builds, the following five builds show there is no genuine trend as the elastic modulus demonstrates only slight changes, suggesting that this is within normal property variability. This implies that the top up powder reuse method will not affect the

elastic modulus of the material. However, given only one study examines this relationship, further research is required.

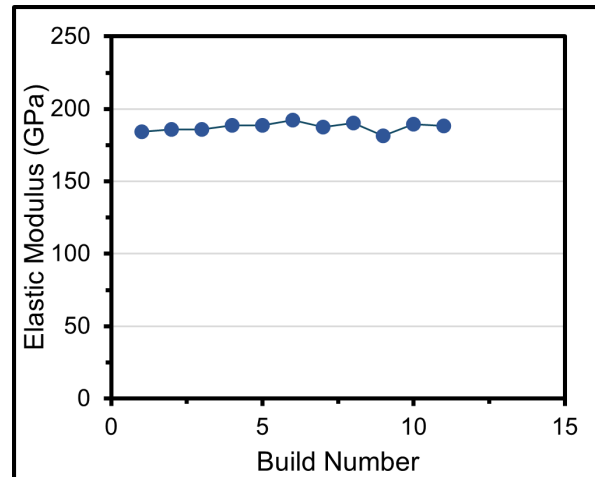


FIGURE 3.18. Elastic modulus of samples in top up powder reuse studies [182].

The yield strength underwent differing changes during top up powder reuse regimes, and these results are shown in Figure 3.19. The only significant result was by Contaldi et al., who observed a considerable increase in the yield strength of 17-4 PH [232]. The yield strength remained steady for the first seven runs however increased by approximately 200 MPa after the eighth run, granting later builds considerably higher yield strengths. This was attributed to the reduction in retained austenite in the microstructure, caused by changing chromium and

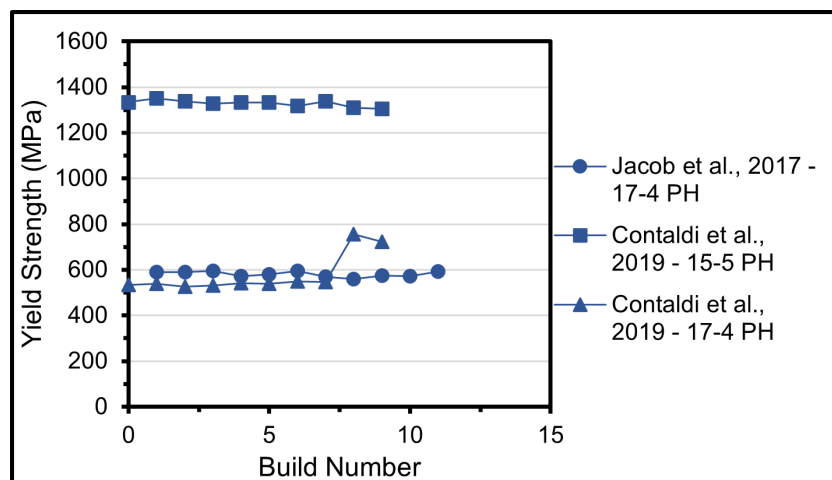


FIGURE 3.19. Yield strength of samples in top up powder reuse studies [182, 232].

nickel equivalents due to evolving powder chemistry, as theorised in the Schaeffler diagram [232, 233]. This suggests that changing powder composition will affect the microstructure and therefore the strength of PBF materials, although this case is limited strictly to steel and its phases. Other materials will likely behave differently and so the effect of the top up powder reuse method on materials other than steel needs to be further researched to determine if similar effects are observed. However, this case indicates the importance of powder chemistry monitoring, which is often a characteristic overlooked in current research.

Results from studies investigating the tensile strength of parts made from powders undergoing a top up reuse method are displayed in Figure 3.20. These plots show no clear relationship between the tensile strength and number of powder reuses. Therefore, from the limited studies available, a top up powder reuse method does not seem to influence the tensile strength of samples.

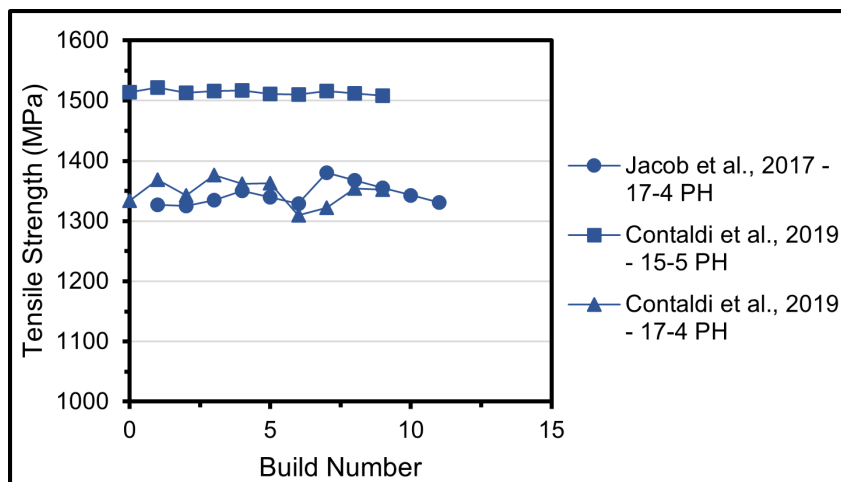


FIGURE 3.20. Tensile strength of samples in top up powder reuse studies [182, 232].

As with the other tensile properties investigated in top up powder reuse studies, there is no discernible relation between the elongation at fracture of samples and powder age. These results are shown in Figure 3.21. The most noticeable result was found by Contaldi et al. [232], when the elongation dropped from 24.1 % after build 7 to 15.55 % after build 8 when investigating 17-4 PH. This occurred at the same time as increases in both yield and tensile strengths, which is expected given known relationships between these tensile properties. This was previously explained as being due to a change in microstructure resulting from a change in nickel and chromium equivalencies. Therefore, the elongation is unlikely to change due to

the top up method of powder reuse unless this method brings about a noticeable change in powder chemistry.

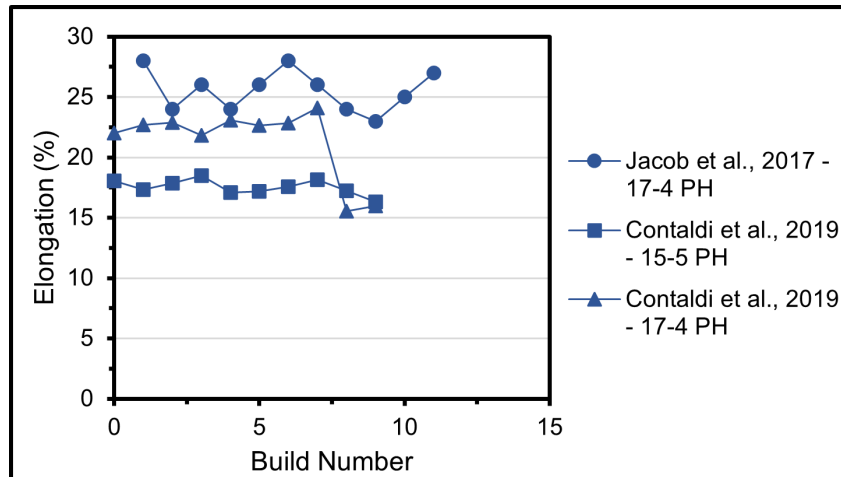


FIGURE 3.21. Elongation of samples in top up powder reuse studies [182, 232].

3.4.2.4 Other Significant Properties

The limited number of studies conducted using the top up powder reuse method means that few other properties of powders or parts have been investigated, and none by more than one study. The following paragraphs therefore review the preliminary research into part density, powder bed density, and powder elemental composition in top up powder reuse studies.

One of the most important factors affecting PBF is the density of the powder bed. Many different characterisation techniques are currently available to measure powder properties that have an effect on powder bed density such as flow and cohesion. However, these do not completely reflect the behaviour of powder within the build chamber [234, 235]. Therefore, a number of studies have recently been conducted to investigate the powder bed density directly [236–238]. Jacob et al. investigated powder bed density throughout their reuse study and their findings are shown in Figure 3.22. They observed that the increases in powder bed density were significant, with increases of 1.6% and 2.3% between builds 1, 5, and 11 [182]. These increases were accompanied by insignificant changes in PSD and so were thought to be due to factors such as powder moisture content. Since the powder bed density was seen to be higher than the apparent density of the powder, it was concluded that it relates more closely to powder tap density. Given the changes observed in this study as well as the likely

significant impact that powder bed density can have on part properties [234], this technique is worthy of more extensive consideration in future literature. However, given that only one study has investigated powder bed density, its link to the top up powder reuse method cannot be properly determined.

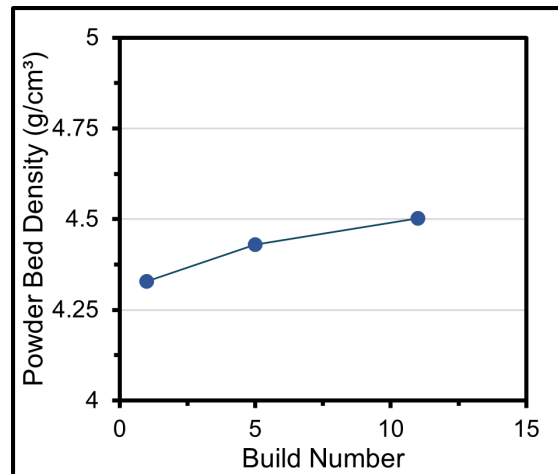
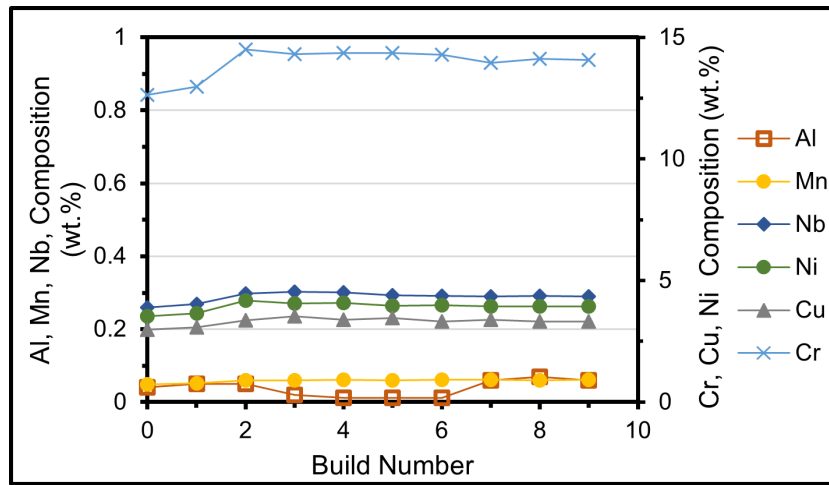


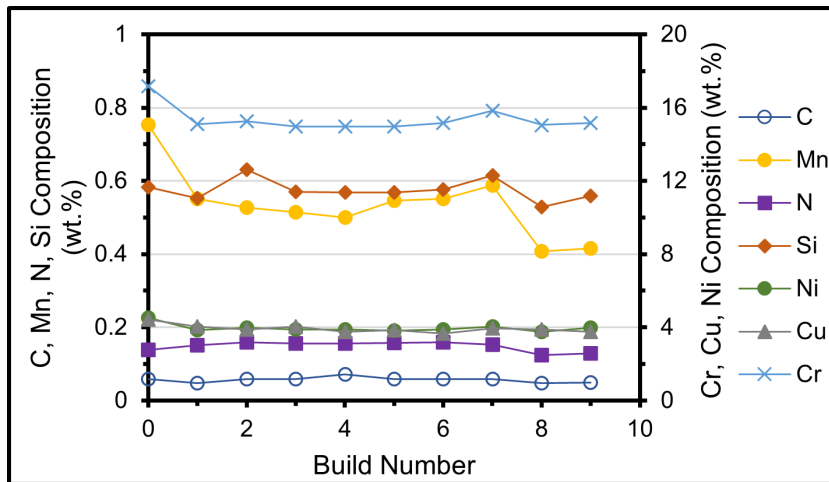
FIGURE 3.22. Powder bed density as measured from capsules by Jacob et al. (adapted from [182]).

The chemical composition of powders was monitored by Contaldi et al. [232] in their study of 17-4 PH and 15-5 PH stainless steels, the results of which are shown in Figure 3.23. They found that these chemical compositions were somewhat stable, however also observed a change in microstructure between builds using powder used eight and nine times. This was attributed to a slight change in powder chemistry, namely chromium and nickel equivalents, resulting in a different microstructure according to the Schaeffler diagram [233]. This led to a significant change in yield strength, implying that powder chemistry needs to be carefully monitored in all alloys for PBF. This study also indicates that a top up powder reuse method has the potential to change the powder chemistry and so in future studies should be carefully monitored.

One of the most important properties during the development of PBF processes is the part density, this property being a particular focus during parameter development [239–241]. It remains a concern within powder reuse studies, with a range of research investigating both the density and porosity of parts printed with reused powder [151, 169, 242]. Jacob et al. [182] remains the only study investigating the top up powder reuse method to consider part density. The relative density of their samples remained above 99.5 % of the specified density of 17-4



(A)



(B)

FIGURE 3.23. Chemical composition of stainless steel in top up powder reuse studies: (a) 15-5 PH (b) 17-4 PH (adapted from [232]).

PH, indicating good results for all their samples. Over the first five builds, no trend in part density was found. After build six, a reduction in part density was noticed and attributed to an increase in the particle size of the powders used, correlating with other research that has also linked changing particle size to part density [61, 201, 243]. However, it was concluded that there was no correlation between part density and powder reuse as all the density values measured by Jacob et al. appeared to be randomly distributed, so any variance was put down to variations within the Archimedes Method. While there appears to be no trend relating part density to the top up powder reuse method, it remains an important property to monitor due to its significance within the additive manufacturing field [244].

3.4.2.5 Summary

The findings of Sections 3.4.2.1 – 3.4.2.4 are summarised in Table 3.2, which provides a brief overview of the properties tested in top up powder reuse studies. However, it is important to note that these findings were made with the observations of only two studies on steel alloys and so further research into both steels and other alloys is required to validate these preliminary findings.

3.4.3 The Refreshing Method

The following sections review the literature surrounding the refreshing method of powder reuse and the effect that this method has on both powder and part properties.

3.4.3.1 Particle Size Distribution

Due to the regular addition of virgin powder it is expected that the PSD will behave differently to the previously mentioned methods, as the powder batch will not have a uniform processing history. The behaviour observed in the literature regarding particle size distribution is discussed below.

The D10 values for refreshing powder reuse studies are shown in Figure 3.24. There is no clear trend within this data, with some studies finding that the D10 value of a powder batch will increase with reuse, while others have found it decreases. However, the majority of studies have found only minor changes to D10 size, with changes of less than 2 μm found in a number of studies [172, 181, 194, 245]. Only a few authors have observed significant changes in D10, with Denti et al. [158] finding a significant reduction in the D10 value from 24.6 μm to 19.7 μm over 1750 build hours of Ti-6Al-4V; however, no explanation for this change was given. Contrastingly, Lee et al. [246] observed a 2.76 μm increase in D10 over nine builds using Inconel 718 which may have been due to an increase in the size of satellite particles. These contrasting results indicate that it is unlikely that the refreshing powder reuse method will change the D10 value of a powder batch, especially given the low changes observed in many studies. However, since few studies have addressed PSD when the refreshing powder reuse method is used, further research is required to solidify these claims.

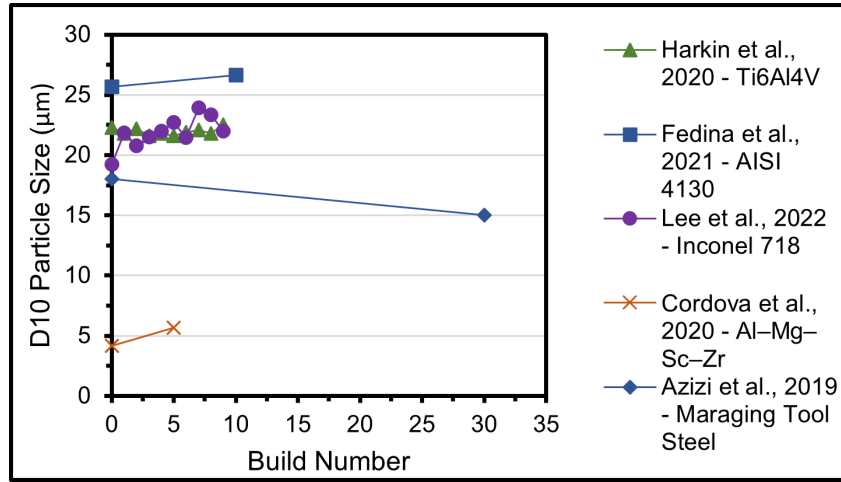


FIGURE 3.24. D10 values of powders in refreshing powder reuse studies [66, 172, 245–247]. Green points represent titanium alloy data, blue points represent ferrous alloy data, purple points represent nickel alloy data, and orange points represent aluminium alloy data.

The D50 values for refreshing powder reuse studies are shown in Figure 3.25, where it can be seen that the majority of studies find minimal change throughout a powder batches life [172, 181, 194, 245, 246]. Cordova et al. [66] and Denti et al. [158] were the exceptions to this, finding an increase and decrease in median particle size, respectively. Cordova et al. observed an increase of approximately 5 µm over five builds in their study of an Al-Mg-Sc-Zr alloy

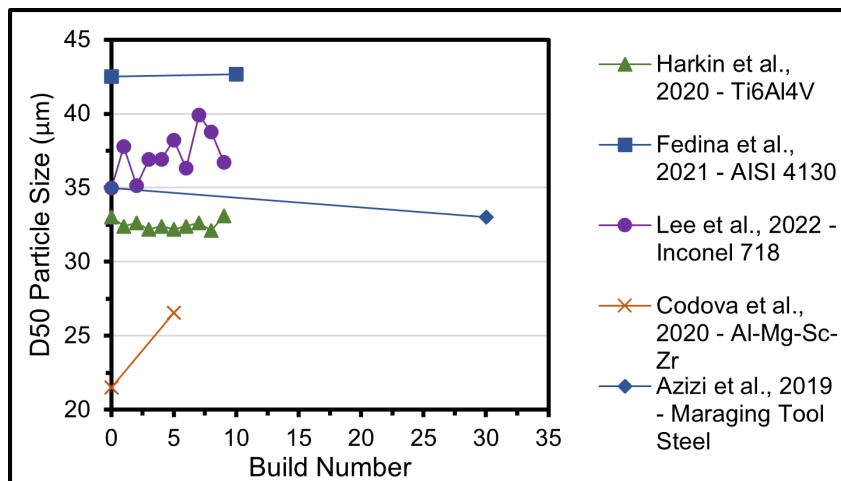


FIGURE 3.25. D50 values of powders in refreshing powder reuse studies [66, 172, 245–247]. Green points represent titanium alloy data, blue points represent ferrous alloy data, purple points represent nickel alloy data, and orange points represent aluminium alloy data.

due to a reduction in the influence of fine particles to the overall PSD. Denti et al. found a reduction from 38.6 μm to 30.6 μm during their study of Ti-6Al-4V, a significant reduction of approximately 20%. The lack of consistency between these two studies, the only ones to report significant changes in D50 value, suggest that changes are not due solely to the refreshing powder reuse method and may be attributed to other factors such as the specific sieving strategy employed [158] or the material used. Therefore, it is likely that the refreshing powder reuse method results in no significant change to the D50 value of a powder batch.

The D90 values for powder studies using a refreshing powder reuse method are shown in Figure 3.26. These again show no consistent trend with most studies finding no considerable change in D90 value [172, 181, 194, 245–247]. This occurred in the study by Fedina et al. [245] on AISI 4130 low alloy steel even though no sieving was employed during the study. Sieving is typically employed to limit an increase in the number of large particles in a powder batch; however, the lack of a change in D90 size indicates that the spatter particles generated during the PBF process were not produced in large enough quantities to noticeably change the PSD, or that they were of a similar size as the largest particles already existing in the powder. Denti et al. [158] and Cordova et al. [66] were again the exceptions to the normal, with both finding changes to the D90 values of their powders, Ti-6Al-4V and Al-Mg-Sc-Zr, respectively. The results of these studies conflict with each other, with Cordova et al. observing an increase

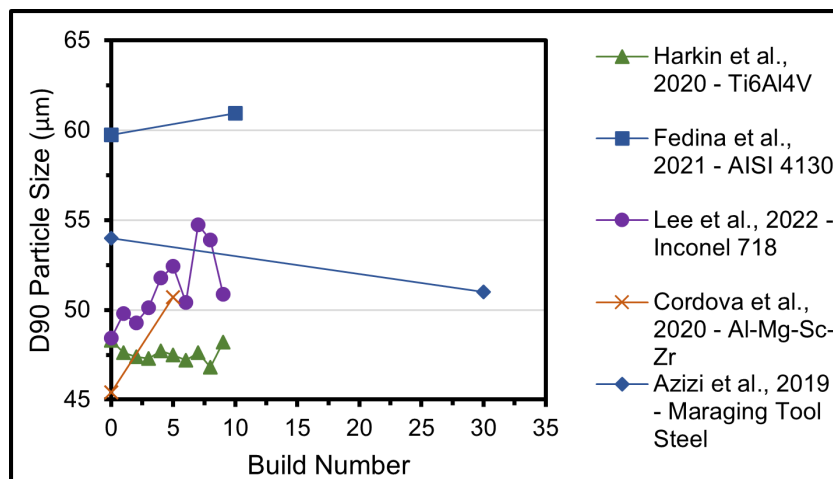


FIGURE 3.26. D90 values of powders in refreshing powder reuse studies [66, 172, 245–247]. Green points represent titanium alloy data, blue points represent ferrous alloy data, purple points represent nickel alloy data, and orange points represent aluminium alloy data.

in size from 45.4 μm to 50.7 μm after five builds, whereas Denti et al. found a decrease in size from 59.9 μm to 47.3 μm after 1750 build hours. It is noteworthy that changes are likely not materially dependant with studies on aluminium alloys resulting in conflicting results, with authors variously finding both increases [66] and decreases [181] in D90 size. Changes in the larger particles of a powder batch have been variously attributed to accumulating agglomeration and spatter [245], laser heat affecting the size and shape of particles [247], and sieving processes removing large particles or breaking down agglomerates [202]. These reasons are similar to those presented by studies using other powder reuse methods. The minimal changes in D90 values during these studies suggests that the D90 will not change due to the refreshing powder reuse method.

Overall, while most studies observed some changes in PSD while employing the refreshing powder reuse method, the changes were not significant enough to determine any relationship between powder reuse and particle size. An approximately equal number of studies found an increase [66, 172, 223, 245, 246] or decrease [158, 181, 194, 247] in particle size, meaning that there is no clear direction in which particle size tends to shift. Therefore, it is likely that a refreshing powder reuse method will not influence the PSD, however, as the majority of studies referenced here run less than ten builds, longer term studies may yield different results. Any changes to PSD are more likely influenced by other factors such as processing parameters or powder handling and sieving procedures, with extended powder reuse exacerbating these changes.

3.4.3.2 Powder Oxygen Content

Most studies investigating the oxygen content of powder when using the refreshing powder reuse method have observed an increase in oxygen content; however, this increase is not universal. The results of these studies are displayed in Figure 3.27. From this graph it is clear that studies by Harkin et al. [172] and Fedina et al. [245] observed relatively consistent increases in oxygen throughout their studies. This was especially true of Harkin et al., where the oxygen content of Ti-6Al-4V powder increased each build cycle until a slight drop after the final build. While Fedina et al.'s build to build findings were less consistent, they still showed a trend demonstrating increasing oxygen content with continued powder reuse using AISI 4130 low alloy steel. Similar results were found by Gruber et al. [248] and Raza et al.

[194], whose studies were conducted in hours and months rather than the typical build number. Over 30 months, Raza et al. documented a powder oxygen content increase in AlSi10Mg from approximately 0.052 % to 0.123 %, an increase of 136%. This considerable increase in oxygen suggests that powders will continue to pick up oxygen throughout their lifetime, although this may not be at a consistent rate. Some refreshing studies have found that oxygen content increases more rapidly in the early stages of powder reuse and then gradually slows during later build cycles [194, 248], which is likely due to surface oxide layers reducing the potential for further oxidation [83].

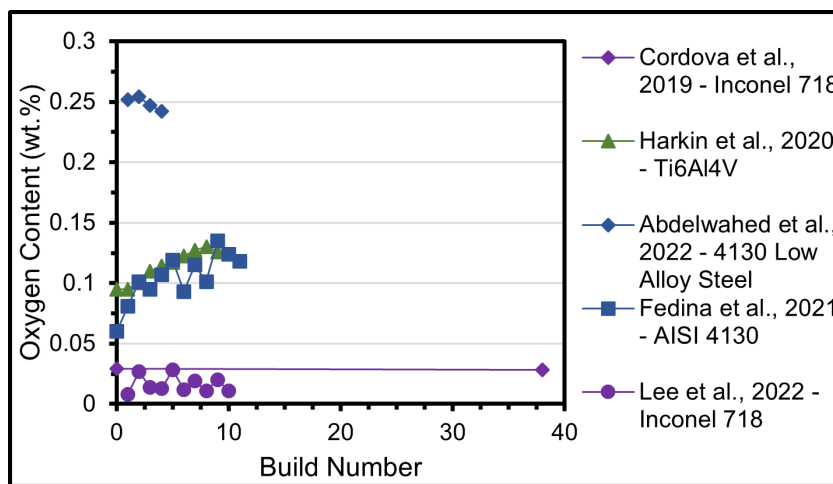


FIGURE 3.27. Oxygen content of powders in refreshing powder reuse studies [166, 172, 206, 245, 246]. Green points represent titanium alloy data, blue points represent ferrous alloy data, and purple points represent nickel alloy data.

Rare studies have found a decrease in the oxygen content of powders with powder reuse, and these go against the findings of the significant majority of the literature. Cordova et al. observed a reduction in the oxygen percentage of Inconel 718, however this was within the scatter band of their measurements and so was deemed insignificant [166]. This, along with findings by Lee et al. [246] that showed no significant change to the oxygen content of a similar powder suggests that the oxygen content of Inconel 718 will not change under a refreshing powder reuse regime, even with long term reuse. This is likely due to the good oxidation resistance inherent to this alloy [249]. Other research has found that the amount of oxygen in a 4130 low-alloy steel reduced by almost 4% during a refreshing powder reuse regime, and this reduction was consistent across multiple build cycles [206]. While no explanation for this was given by Abdelwahed et al., this reduction may be due to the high

initial levels of oxygen present in the virgin powder, at 0.29 wt.%, leaving a lower potential for oxygen to continue to be added to the powder batch. As a greater proportion of refreshing powder reuse studies showed a reduction or no change in powder oxygen content when compared to other powder reuse methods, this method is more likely to result in a lower than usual change in oxygen content. Given the requirement of an oxygen content limit for some metal powders [203], this reuse technique shows great potential. However, due to the still limited number of studies that have investigated oxygen content in refreshed powders, this requires further investigation before its acceptance as a tested theory, especially for reactive materials.

The main way oxygen is introduced into a powder is via surface oxide spots or shells, with the type and manner of oxide formation depending on the alloy used. Raza et al. [194] found a rapid initial growth in an aluminium surface oxide in a refreshed AlSi10Mg powder, attributed to the natural passivation tendency of aluminium. This oxide layer growth is shown in Figure 3.28, with a total growth in oxide thickness from 4 nm to 38 nm over 30 months. During powder reuse, oxides tend to form preferentially. Some of the most common formed are aluminium and titanium oxides, which tend to form even in alloys where they are not the dominant constituent [246]. This suggests that titanium and aluminium based powders will more easily oxidise than other materials, and so particular care must be taken when using these alloys.

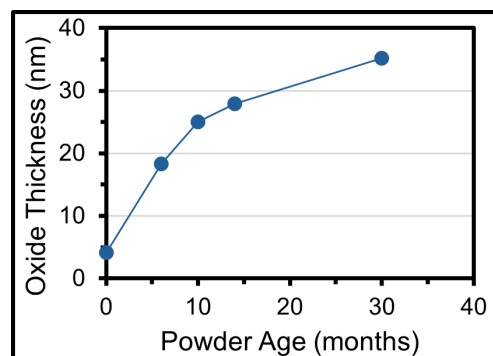


FIGURE 3.28. Oxygen layer thickness of AlSi10Mg powder from a refreshing powder reuse study, up to 30 months of reuse (adapted from [194]).

3.4.3.3 Tensile Properties

As tensile properties are some of the most important to understand due to the prevalence of tensile loading in industrial applications, they have been investigated by numerous refreshing powder reuse studies. Only one study has investigated the elastic modulus of a refreshed powder batch, with Chen et al. [250] finding that the elastic modulus increased from 162 GPa to 167 GPa over ten builds. However, only one specimen was tested for each condition, calling into question the statistical validity of this result. Therefore, future studies should consider investigating the potential change in elastic modulus for refreshed powders, especially if changes in chemistry are noticed.

The yield strength of materials is a more commonly investigated property in refreshing powder reuse studies, and the results of these studies are displayed in Figure 3.29. From this data, there is no clear trend relating yield strength to the number of refreshing cycles. Clearer is the trend relating yield strength change with the type of alloy during reuse studies. Two refreshing powder reuse studies utilising Ti-6Al-4V observed increases in yield strength with powder reuse, the most significant being the findings of Denti et al. These authors observed an increase of approximately 115 MPa over 1740 build hours [158], while Wei et al. found an increase of 46 MPa over seven builds [251]. Work by Yanez et al. [252] contrastingly found no change in yield strength in Ti-6Al-4V reused 12 times. The yield strength of aluminium

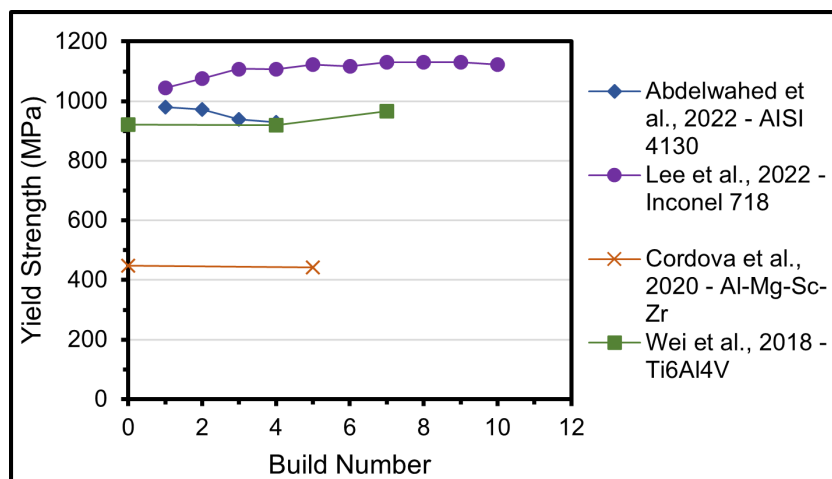


FIGURE 3.29. Yield strength of samples in refreshing powder reuse studies [66, 206, 246, 251]. Green points represent titanium alloy data, blue points represent ferrous alloy data, purple points represent nickel alloy data, and orange points represent aluminium alloy data.

alloys has been seen to decrease with reuse in the two studies investigating such materials [66, 181], however the reductions were not as large as those found by Denti et al. Results for steel powders showed that reuse leads to a reduction in yield strength, however the level of change differs between studies. Abdelwahed et al. [206] found a reduction of 50 MPa in yield strength over four builds for the as-built, vertical specimens of 4130 low alloy steel, a reduction of 5%, whereas the yield strength of maraging tool steel investigated by Sun et al. [253] reduced by 32 MPa after 113 cycles, a reduction of 3%. This suggests that other aspects are likely influencing the yield strength of these powders, such as the exact alloy composition or the powder handling techniques employed. From these refreshing powder reuse studies, it is evident that the material composition will determine how the yield strength will change as a powder is reused.

The tensile strength of materials undergoing a refreshing powder reuse method display very similar results to those of yield strength and are shown in Figure 3.30. Again, alloy type seems to play a significant role in the changing strength. Titanium alloys were again seen to increase in strength as powder was reused, attributed to changing powder microstructure [251]. Aluminium alloys were seen to decrease in strength due to increased porosity [66, 181]. Results using steel alloys were less significant, with Abdelwahed et al. [206] observing a 2% increase in tensile strength, while Sun et al. [253] found an increase of 1% in strength from

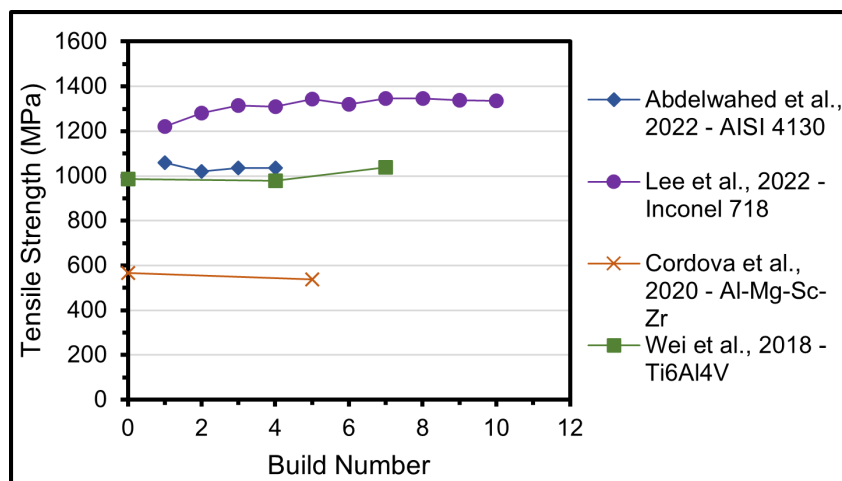


FIGURE 3.30. Tensile strength of samples in refreshing powder reuse studies [66, 206, 246, 251]. Green points represent titanium alloy data, blue points represent ferrous alloy data, purple points represent nickel alloy data, and orange points represent aluminium alloy data.

1115 MPa to 1127 MPa. This suggests that the change in tensile strength of steels caused by reused powders is negligible as it is within measurement errors. These results suggest that the tensile strength of products made with refreshed powder may be changed by this powder reuse method. Whether there is any change, and the extent of this change, will be determined by the alloy composition.

The elongation at fracture of materials that have undergone a refreshing powder reuse strategy are displayed in Figure 3.31. Once again trends appear to be related to material, however, increases in strength are not necessarily matched by a reduction in ductility. Both aluminium studies observed lowered ductility that corresponded to a reduction in both yield and tensile strengths [66, 181], with Cordova et al. [66] finding of a reduction of elongation from 13 % to 11 % over five successive builds. While this was not deemed significant by the authors, a combination of studies finding similar, small reductions in elongation at failure suggest a trend. Similarly, refreshing studies on titanium have found that an increase in strength occurred simultaneously to an increase in elongation, with Denti et al. observing an increase in elongation at failure from 11.8 % to 12.6 % over 1740 build hours [158] and Wei et al. finding an increase from 15.7 % to 16.4 % over seven builds [251]. These changes were minor compared to aluminium results so while this indicates that elongation will increase when titanium alloys are reused in a powder refreshing regime, longer term studies are required

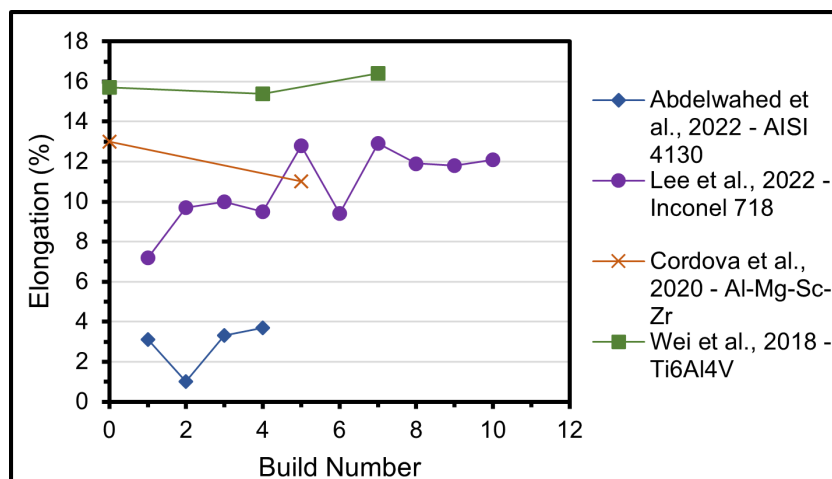


FIGURE 3.31. Elongation of samples in refreshing powder reuse studies [66, 206, 246, 251]. Green points represent titanium alloy data, blue points represent ferrous alloy data, purple points represent nickel alloy data, and orange points represent aluminium alloy data.

to validate these initial results. Steel alloys were again inconsistent in their results, with Abdelwahed et al. [206] finding elongation slightly increased from 3.1 % to 3.7 % over four builds, whereas Sun et al. [253] found an insignificant decrease in elongation over 113 builds of 0.1 %. This again suggests that the tensile properties of steels will not be changed by powder refreshing itself. Therefore, the refreshing powder reuse method will likely affect the ductility of samples, although the alloy composition will determine how this property will change.

3.4.3.4 Other Significant Properties

Due to the significant range of powder characterisation, mechanical, and microstructural analysis methods available to researchers, there remain a number of tests conducted during refreshing powder reuse studies that have not been yet mentioned in this review. A comprehensive study of every technique in every piece of research on this powder reuse method is beyond the scope of this review, however some common investigations are reviewed in the following paragraphs.

One of the aspects of powder characterisation that is not currently extensively investigated is powder microstructure, however in some refreshing powder reuse studies this has been demonstrated to change over time. One example is the study by Pinto et al. [254], who investigated the magnetic properties of 316L steel. They found that an increase in the magnetism of the powder was due to an increase in ferrite, from being non-existent in virgin powder to having a volume fraction of 0.06% in powder reused 16 times. This was hypothesised to cause inhomogeneous powder distribution, creating voids in the powder bed and parts as magnetic particles began to clump together. While the volume fraction of ferrite remained low in this study, continual powder reuse has the potential to exacerbate this issue and cause significant porosity in later builds. Wei et al. [251] also studied the microstructure of a refreshed powder during their investigation into Ti-6Al-4V, finding significant changes. Virgin powder was dominated by a α or martensitic α' phase, which over the course of seven builds transformed into a powder with a fine α and β lamellar structure. This was accompanied by a reduction in the width of X-ray diffraction peaks due to improved crystallinity. These changes were attributed to the heating and cooling cycle in the build chamber and represent a return to the materials equilibrium state and were thought to contribute to an improvement in

tensile properties. Since the change in phases in both steel and Ti-6Al-4V have been attributed to the continual exposure of powder to build chamber processes, whether the production of spatter [254] or continual reheating and cooling of existing powder [251], it is likely that the use of a refreshing powder reuse technique will minimise phase transformation effects due to the continual introduction of unheated, virgin powder.

While the main reason for investigating powder chemistry tends to be measuring oxygen levels, other aspects of powder chemistry can also be monitored. The majority of studies have found that there is no change in chemistry during refreshing powder reuse studies. Rousseau et al. [47] found no significant changes in carbon, nitrogen, and sulfur over four runs using Ti-6Al-4V, however this may be due to the limited reuse runs. Lee et al. [246] and Fedina et al. [245] found similar results, using Inconel 718 and AISI 4130 low alloy steel respectively, observing no changes in minor alloy constituent quantity. The only result of significance was found by Fiegl et al. [181], who observed an increase in hydrogen content from 50 ppm to 80 ppm over 30 months reusing AlSi10Mg0.4. This led to a slight increase in the porosity of printed parts as this hydrogen content is above the hydrogen solubility limit in molten aluminium. While these results show that there is minimal or no change in powder composition, apart from oxygen, when utilising a refreshing powder reuse method none of these studies investigated major alloy constituents. In studies using lighter elemental alloys such as titanium, aluminium, and vanadium, it has been shown that some of these elements may evaporate preferentially from a melt pool, producing spatter of different elemental composition to the powder bulk [191]. Therefore, further research is required into the bulk alloy constituents and whether they change when using a refreshing powder reuse method.

The hardness of materials is another important property, however only a limited number of refreshing studies have investigated this property. Fiegl et al. [181] found that an AlSi10Mg alloy reused for 30 months produced parts with a hardness that lowered from 108 HV to 96 HV. Rousseau et al. observed similar results, with the hardness of Ti-6Al-4V decreasing from 161 HV to 157 HV over 10 build cycles [47]. These reductions in hardness were attributed to a coarsening microstructure [181], and were expected to follow the trends of other mechanical properties with reuse [47]. Similar justification was given by Sun et al. [253] who found that the hardness of maraging tool steel changed depending on measurement location due to the Hall-Petch relationship. Although differing local microstructures and hardness values were

observed, hardness values overall remained stable changing from 36.9 HRC to 37 HRC over 113 builds. This change was within the error measurement of this study. While these three studies provide an insight into the potential changes in hardness when reusing a powder with a refreshing technique, the scope is still too limited to draw generic conclusions. Therefore, further research is required to gain a conclusive understanding.

3.4.3.5 Summary

The findings of Sections 3.4.3.1 to 3.4.3.4 are summarised in Table 3.3. This provides a brief overview of properties tested in the refreshing powder reuse studies. Due to the limited number of studies conducted using the refreshing powder reuse method, many results are unable to be summarised. This is especially true for different alloy systems, as often only one study on a particular alloy has been conducted. Therefore, the summaries in Table 3.3 would be aided by a larger body of research.

3.5 Evaluation of Powder Reuse

3.5.1 The Single Batch and Collective Ageing Methods

The single batch powder reuse method is the most commonly used powder reuse technique employed in academia due to the accelerated degradation of the powder batch since no new powder is introduced [192]. The collective ageing method is less commonly used, however produces similar results to the single batch method while allowing for a greater number of build cycles.

The main advantage of these powder reuse methods lies in their high degrees of traceability, the relative advantage of which is displayed in Table 3.4 alongside relative powder handling simplicity. This means that for a given powder batch the exact number of build cycles and the amount of time spent in the machine can be definitively known.

This traceability does come at a cost, as evidenced in previous sections. The properties of both powders and printed parts were the most volatile in the single batch and collective ageing methods when compared to similar studies using different powder reuse methods. Single batch and collective ageing methods saw the majority of the meaningful changes in PSD,

TABLE 3.4. Comparative summary of each powder reuse method in relation to their powder handling procedures and powder traceability. A plus sign signifies relatively high quality, an equal sign middling quality, and a minus sign relatively low quality.

Powder Reuse Method	Powder Handling Simplicity	Powder Traceability	Powder Waste
Single Batch and Collective Ageing	=	+	-
Top Up	+	-	+
Refreshing	-	-	+

with all materials except steel alloys experiencing significant changes to the powders D10, D50, and D90 values with reuse. While this suggests that the material will affect the rate at which PSD will shift during powder reuse, it is clear that when using a single batch or collective ageing method changes are expected. These changes in PSD then have the ability to affect the printed part density, part surface roughness, and hardness, amongst other properties [255, 256]. Single batch and collective ageing powder reuse methods also saw more regular increases in powder oxygen content when compared to other powder reuse methods. These steady increases degrade the powder batch and are especially problematic when using alloys with allowable oxygen limits, such as Ti-6Al-4V [203]. Powder oxygen content can alter melt pool dynamics during melting resulting in changes to porosity, impact toughness, and tensile strength, amongst other properties, and so is typically not seen as desirable [193, 195].

From a powder handling perspective, the single batch and collective ageing methods are relatively simple to employ. As they are simpler than the refreshing method but more complex than the top up method, the single batch and collective ageing methods are relatively neutral from a powder handling perspective.

One of the issues arising from the single batch and collective ageing powder reuse methods is that there will be leftover powder, in insufficient quantity for another build. This powder has been designated as end-of-life powder and remains a problem within the additive manufacturing field. It cannot be reused without mixing with powder of a different age, thereby rendering the single batch or collective ageing cycle at its end, and the alternatives to mixing are disposal to landfill or scrapping and repurposing, which is unlikely to be profitable [257]. The disposal of powder is also counter to the ideas of recycling and environmentalism, which

are both some of the key reasons behind the investigation into powder reuse. This problem relates solely to these reuse methods and as such provides a significant disadvantage when compared to other powder reuse methods.

The main advantage of a single batch or collective ageing powder reuse method lies in its high traceability. However, the increased changes in properties when compared to other powder reuse methods suggest that the single batch and collective ageing methods are not as effective at preserving the properties of powders and parts manufactured with reused powders. This, in combination with the issue of end-of-life powder means that the single batch and collective ageing methods are not likely to be the first preference of powder reuse method for most applications.

As such, this powder reuse method is mainly relevant for industries with regulation requiring high powder traceability, such as for the aerospace industry [258]. Here, the single batch or collective ageing methods would be most relevant, even though the properties of the powder may degrade more rapidly.

3.5.2 The Top Up Method

The top up method remains severely understudied, with so far only studies by Contaldi et al. [232] and Jacob et al. [182]. This means that the effect of the top up method on many properties, such as powder oxygen content, have not been investigated and so a considerably larger body of knowledge needs to be built up in order to properly assess the viability and effectiveness of this method.

The top up method has poor traceability, as powders of different ages are used to produce parts in the same build cycle. While a graphical representation of the powder layers within builds is shown in Figure 3.4, due to the slight mixing of powders inherent in reloading a PBF machine as well as variability in powder deposition within the build chamber, this is not a perfect representation of powder layering. In reality, there is no clear, linear distinction between powder layers. Therefore, while the approximate powder age of each build can be known, the exact age cannot, resulting in poor powder traceability. This is exacerbated by factors such as changing dose factors, making powder tracing even more complicated.

Due to the limited number of studies investigating the top up method, its effects on powder and part properties are difficult to determine. Preliminary results indicate that regarding PSD, there is a reduction in fines associated with this powder reuse method while other particle size measurements will not change. Tensile properties show considerable variations between studies and therefore further research is required to begin to draw conclusions regarding the effect the top up powder reuse method has on these properties. The limited scope of current studies, especially the fact that they have so far been conducted exclusively on stainless steels, mean that no conclusive answers can be found regarding the effect the top up powder reuse method has on powder or part properties.

The top up method has by far the simplest powder handling procedure of all the existing powder reuse techniques. No special processing of the powder is required apart from sieving, and no mixing or separation of powder batches needs to occur. These two factors greatly simplify this reuse method, meaning that it should be the preferred powder reuse method from a powder handling perspective.

Due to the current lack of research surrounding the top up powder reuse method, it cannot be recommended for widespread use as the effects of this method on powder and part properties is largely unknown. This is especially true when powder traceability is important, as the limited traceability available with this method renders it unfeasible. However, due to its significant advantages from a powder handling perspective, if it is found that the top up powder reuse method has limited effects on powder and part properties then this method has great potential for widespread application. Already, its use should be considered for applications where part properties are not of high concern, such as for artistic applications, initial geometric calibrations and test pieces, and initial stages of prototyping. These are applications where the potential for inhomogeneous property distribution will not affect the performance of an additively manufactured part.

3.5.3 The Refreshing Method

While a number of studies have been conducted investigating the refreshing powder reuse method, research in this area remains limited. Therefore, while the effects of this refreshing

method are beginning to be understood, further efforts are required to understand its effects on specific alloys within specific processing systems.

The refreshing powder reuse method has poor traceability, as a sample taken from a powder batch will have an unknown history due to the mixing of powders of different ages. While probabilistic estimates may be made by assuming a homogenously mixed batch comprised of various samples of known ages, this is not a perfect representation. Therefore, the exact age of the powder cannot be determined, becoming problematic for applications where powder tracing is crucial.

Regarding powder and part properties, the refreshing method seems to have a limited effect. Changes in PSD have been observed in refreshing studies, however, these do not occur as frequently or consistently as in single batch studies. The same is true for powder oxygen content, with a slightly lower average increase when compared to other reuse methods. Regarding mechanical properties, a refreshing powder reuse method has been seen to change tensile properties although these changes are materially dependant. Therefore, it can be seen that the refreshing powder reuse method causes no major effects on the properties of powders or parts manufactured with reused powder, although a consideration of the material used is required.

The powder handling procedures surrounding the refreshing method are more complicated than other reuse methods, and this forms the main drawback to this technique. The addition of virgin powder to a batch, whether every build cycle or after a predetermined number of cycles, adds an extra layer of complexity to the process. Considerations need to be made regarding how virgin powder is introduced to a batch, such as when to introduce this powder and how to minimise its exposure to atmosphere. Thought must also be put into how powder is blended together - if it is blended at all – adding to the time and cost of the process of powder preparation. This added complexity when compared to other powder reuse techniques forms the main disadvantage of the refreshing powder reuse technique.

One of the considerable advantages of refreshing powder is the significant reduction in wasted powder, as end-of-life powder is not a factor. All powder is constantly being used in successive build cycles, therefore producing no waste apart from sieve residue and powder lost during the build procedure. This means that the refreshing method is more environmentally and

economically efficient than particularly the single batch and collective ageing methods, as less powder needs to be disposed of after a production run.

For these reasons, the refreshing method shows great potential for use in a wide range of industries and applications. The efficiency of the method from a powder use perspective as well as the minimal change in powder and part properties make this a desirable process from environmental, economic, and mechanical perspectives. This means that if the issues regarding powder traceability are resolved then this powder reuse method could become the most widely used of all methods. Already powder blending, as required in this method, is a consideration in some industrial standards [258, 259], lending promise to this methods future employment. Therefore, while refreshing is already suitable for applications such as the automotive and biomedical industries, it may also soon be common practice for aeronautical and aerospace manufacturing.

3.5.4 The Current State of Powder Reuse

While the effects of reusing powder during PBF processes are becoming more well known, the growing body of research remains a complex topic and one that is not yet adequately understood. While extensive research has been undertaken, complexities remain due to the large variation in materials studied, as well as the plethora of powder reuse strategies that have been employed over time, resulting in a range of often contradictory results.

In terms of the current state of powder reuse, there are a variety of factors that need to be considered. One of the most important of these is the current stance of industry, namely its common reluctance regarding powder reuse even in the face of its obvious environmental and economic benefits. Yet the most significant aspect remains what current strategies are being employed in powder reuse, as these dominate the way in which industry and academia comprehend the reuse of powder for PBF.

The literature conflicts as to the extent to which powder reuse is industrially employed, with the majority of authors stating that reuse is severely limited [69, 188] while others claim the inverse [182]. While the economic and environmental benefits of powder reuse are well known in both academia and industry [229], in practice it is not commonly enough undertaken. The main reason for this is that industries see the practice as an unknown, and therefore

risky [188]. This is especially true in the biomedical, aeronautical, aerospace, and nuclear industries, each of which have low risk tolerances. This means that any abnormalities in parts can be costly and unsafe, meaning that the reuse of powders is not worth the risk [69, 260].

While there has been a considerable amount of recent research undertaken, powder reuse is still seen as too risky for wide implementation and there are a variety of factors that influence this school of thought. This mainly has to do with the fact that the current body of knowledge is not extensive enough to deem powder reuse a safe practice in high-risk applications [261]. However, this lack of research is not the only reason powder reuse is not industry standard practice. There remains considerable variation within the literature regarding the effects of powder reuse, as evidenced in this review. This can be seen in the many studies that investigate the same material using similar reuse practices and processing parameters but still have significantly different findings. Therefore, universal conclusions are difficult to draw. There are also different powder manufacturing techniques, each of which will produce varying qualities of powder [215] which may respond differently when reused. The extent to which it will affect the body of powder recycling studies is more limited than Ghods et al. [215] suggest, as the vast majority of presently used powders are gas atomised due to this techniques widely accepted balance between product quality and cost.

The most significant reason for a lack of uptake remains the inconsistency within the literature concerned with powder reuse techniques. Concerted effort is required to overcome this obstacle. Reuse methodologies are required to allow repeatability and means of comparison between studies. This needs to be complemented by more advanced characterisation techniques, allowing for a greater understanding of powder degradation and its underlying mechanisms. Until these considerations are met, the state of the powder reuse literature will not be significantly advanced enough to produce standardised powder reuse methodologies that can be implemented in industry.

3.6 Conclusion

Additive manufacturing is a growing field, and metal powder bed fusion is a popular subset of this new manufacturing class. Part of the advantage of additive manufacturing lies in its additive rather than subtractive nature, meaning that less material is wasted during the

manufacturing process. For powder bed fusion, however, this relies on powder reuse, which is often seen as an unknown quantity and therefore a risk in some manufacturing sectors. Therefore, a solid understanding of powder reuse is required to enable its full potential regarding material efficiency.

Powder reuse is currently being investigated by a number of research groups using a variety of different powder reuse methods. The single batch and collective ageing, top up, and refreshing methods are the most popular methods for powder reuse, and each has been shown to have respective advantages and disadvantages. The single batch and collective ageing methods allow for high traceability; however, result in the quickest degradation of powder and part properties. The top up method remains relatively unstudied; however, its simple powder handling procedures give it great potential for applications where mechanical properties are not crucial. The refreshing method allows for the least amount of waste of all the methods with the greatest preservation of part properties; however, the relatively complex powder handling required as well as poor powder traceability means it is not as widely used as its potential suggests. It has been shown that each of these powder reuse methods will likely have their own areas of application given their respective advantages.

Much research remains to be undertaken in the field of powder reuse, particularly regarding the effects that particular reuse methods have on powder and part properties. This is especially true for the top up method, the understanding of which would be greatly improved by further research using a variety of materials along with a wider range of properties tested. Of particular help for the powder reuse topic would be studies where different reuse methods are tested concurrently, to eliminate as many variables between studies as possible. Longer term studies are also required, since many current studies observe small changes in powder and part properties that may become more significant in later build cycles.

CHAPTER 4

Methodology

The following chapter outlines the various methodologies surrounding each of the experimental tests carried out in this research. This chapter has been split into four sections according to the categories of equipment used: part manufacture, powder characterisation, mechanical testing, and part characterisation.

In conjunction with the aim of this thesis, testing was conducted on additively manufactured AlSi7Mg samples. These samples were manufactured according to the process outlined in Section 4.1 of this chapter.

Powder characterisation includes tests that were carried out to characterise the powder used during the powder reuse components of this thesis and are outlined in Section 4.2. Many of these were conducted according to ASTM standards, however, due to the relatively new field of powder characterisation for additive manufacturing and its continual development, many of these tests are not covered by standards.

Mechanical tests, outlined in Section 4.3, were conducted throughout the research for this thesis and were chosen for a variety of reasons that included the suitability of the test for the property being investigated, such as hardness testing to develop ageing curves. However, tests were mainly chosen to allow for the testing of a wide variety of mechanical properties.

Part characterisation in Section 4.4 covers all other tests conducted on parts manufactured during this thesis. This primarily includes microstructural characterisation, however, also includes tests such as those investigating surface roughness and density, which are well known as being important to characterise in additively manufactured parts.

While these four sections provide a general outline for how tests were conducted, not all tests fit into a single category. Therefore, where applicable, explanations in this chapter mention

if tests were conducted upon both parts and powders. These tests are placed in the sections where they were most utilised.

4.1 Manufacturing Processes

All parts for analysis in this thesis were manufactured and heat treated according to the steps described in the following sections.

4.1.1 Part Manufacturing

Parts were manufactured using a dual laser Concept Laser M2 Series 4, with the basic machine parameters given in Table 4.1. These are the standard, recommended processing parameters for AlSi7Mg according to Concept Laser using the ‘hybrid’ processing parameter set, designed as a balance between productivity and surface finish. All parts used in this research that are designated ‘as-printed’ were printed on supports, whereas parts that have been designated ‘machined’ were machined from blocks printed directly onto the build plate. The exception to this were the flexural samples used in Chapters 7 and 8, where the as-printed samples were printed directly onto the build plate and removed using wire electrical discharge machining (wire EDM).

TABLE 4.1. Processing parameters used for part manufacturing.

Parameter	Value
Laser Power	370 W
Scan Speed	1600 mm/s
Hatch Spacing	0.112 mm
Layer Thickness	30 μm
Build Plate Heating	80 $^{\circ}\text{C}$
Gas Type	Argon

The powder used in this research was supplied by AP&C, with this powder’s chemical composition displayed in Table 4.2. This powder was tested according to ASTM E1097-12 and was found to be chemically conforming to standard specifications for AlSi7Mg. The powder was new and unused for manufacturing parts for all components of this research, except for Chapters 7 and 8 which utilised reused powder.

TABLE 4.2. Powder chemistry for part manufacturing. Displayed is the nominal chemistry as measured by the powder supplier, with measurements in wt.%.

Al	Si	Fe	Cu	Mn	Mg	Zn	Ti	Be	O	N
Bal.	7.0	0.06	<0.01	<0.01	0.44	<0.01	0.11	<0.001	0.076	<0.005

Once parts had been manufactured, they were removed from the build plate using wire EDM. Parts that have been designated as machined were also cut using wire EDM into their final shape, except for fatigue specimens where a lathe was used for manufacturing and surface finishing.

4.1.2 Heat Treatment

For heat treatments conducted as a part of this research, a variety of techniques were used. All stress relieving treatments were carried out in a Nabertherm LH 60/14 chamber furnace under an argon atmosphere. Solution treatment was conducted in a salt bath and ageing in an oil bath.

All parts analysed in Chapter 5 were not heat treated. The exception is one set of samples that were stress relieved for two hours at 280 °C. These parameters were chosen due to their suitability according to previous studies on AlSi7Mg [111].

Parts analysed in Chapter 6 underwent a variety of heat treatments. Stress relieving was conducted for two hours at 300 °C, according to manufacturer's recommendations, while solution and ageing times and temperatures were varied according to the investigation carried out.

All parts that were manufactured for Chapters 7 and 8 underwent a stress relief heat treatment for two hours at 300 °C prior to removal from the build plate to eliminate warping resulting from residual stresses. Samples that were T6 treated were solution treated at 530 °C for six hours and subsequently water quenched. These samples were then aged at 160 °C for five hours. This T6 treatment was conducted according to the recommended parameters of the powder supplier, AP&C. The exception to this heat treatment workflow is the fatigue specimens, which underwent no heat treatment prior to testing.

4.2 Powder Characterisation

4.2.1 Particle Size Distribution

Powder particle size distribution was investigated using a Malvern Mastersizer 3000. This machine utilises laser diffraction to measure the equivalent circular diameter of particles, producing D10, D50, and D90 readings. For this research the wet dispersion attachment was used with isopropanol as the solvent, with five measurements taken for each sample.

4.2.2 Hall and Carney Flow

Before each new manufacturing run was started, powder was removed for testing using the Hall and Carney flowmeters. These tests were chosen due to their long term establishment within the literature [68], with both tests used to ensure that powder flow could always be measured by at least one test given the variability of powder flow properties especially when conducting powder reuse research [214]. As powder did not initially flow through the Hall funnel using the static flow method, the dynamic flow method was used, whereas for the Carney flow tests the static flow method was used. Five tests were carried out for each powder state measured to ensure statistical validity. These tests were conducted according to ASTM B213-20 and ASTM B964-23 [262, 263].

4.2.3 Apparent Density

The apparent density of powders was measured according to the procedure in ASTM B212-21 [264] using the apparatus required for the Hall flow test. Five samples were tested for each powder sample, and these tests were carried out concurrently with the Hall and Carney flow tests.

4.2.4 Granudrum

To further determine the powder flow characteristics the rotating drum method was used, using a Granudrum. Approximately 80 grams of powder was used for each of these tests to ensure the drum was half full as required for accurate imaging. This allowed for the measurement

of the powder's avalanche angle, by slowly rotating the drum containing the powder until the slope collapsed to give an indication of how powder will sit while it is being spread by the recoater. Tests were also conducted using the speeds hysteresis mode, allowing for the measurement of the velocity angles and powder cohesion to give an indication of powder flowability, with low velocity angles and cohesion values indicating a powder that flows well. A schematic of this is shown in Figure 4.1. Hysteresis testing was conducted at speeds ranging from 2 rpm to 20 rpm, gradually increasing in intervals of 2 rpm, then decreasing by increments of 2 rpm. Ten measurements were taken for each data point tested using the Granudrum.



FIGURE 4.1. Schematic of different powder flow conditions as measured by the Granudrum, with drum rotation in the direction of the arrows. On the left, powder has a low velocity angle, as seen by the shallow slope of the powder face, and also has a low cohesion value, as seen by the uniformity of the powder face. These are indicative of good powder flow. Powder flow worsens towards the right, with a steeper velocity angle and a wider face indicating worsening powder flowability. Figure is reproduced from the Granudrum user manual [265].

4.2.5 Powder Bed Density

During part manufacturing, hollow cylindrical capsules were made at the corner of each build plate in order to capture powder at these locations. These capsules were 30 mm tall with a radius of 13 mm: a schematic is shown in Figure 4.2. These capsules were removed from the build plate prior to stress relief to ensure that no powder sintering occurred during heat treatment. Analysis involved weighing the capsules while full of powder, empty of powder,

and when filled with deionised water. Using the calculation shown in Equation 4.1, the powder density for each of these capsules was then calculated. These densities were averaged to determine the powder bed density of each manufacturing run, with one capsule taken from each corner of the build plate.

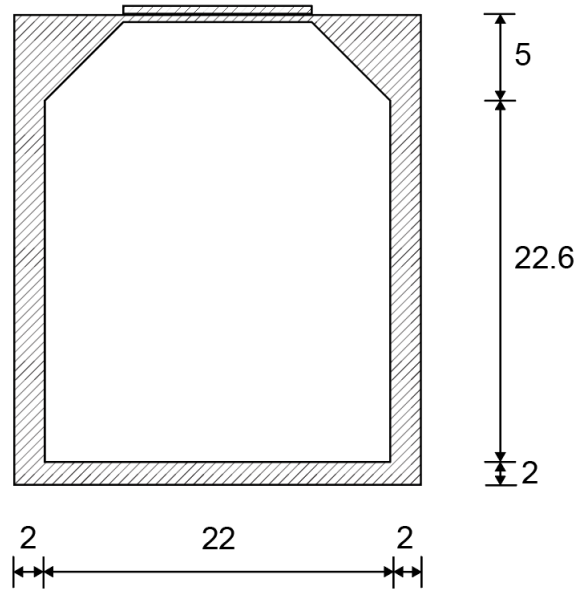


FIGURE 4.2. Cross section of cylindrical powder bed density capsule, including an internal cavity to capture unmelted powder. All dimensions are in mm.

$$\rho = \frac{\text{mass full of powder} - \text{mass empty}}{\left(\frac{\text{mass full of DI water} - \text{mass empty}}{\text{density of water}} \right)} \quad (4.1)$$

4.2.6 X-Ray Photoelectron Spectroscopy

Given powder oxidation typically occurs at the surface of powders [250], surface chemistry analysis was carried out by X-ray electron spectroscopy (XPS). This was completed using a Thermo Fisher K-Alpha XPS, which utilises a monochromated aluminium K-alpha X-ray source. A spot size of 300 μm was used, and analysis was completed using the Advantage software system. All analysis was conducted after spectra were charge corrected to the C1s peak at 284.8 eV.

4.2.7 Bulk Chemical Analysis

Bulk chemical testing of both powders and parts was carried out using inductively coupled plasma atomic emission spectroscopy and Leco combustion. These tests were conducted commercially, external to the university as this equipment was not available at the university during the time this research was conducted.

4.3 Mechanical Testing

The following section outlines the mechanical testing that was conducted as a part of this research. With the exception of hardness testing, three samples were tested for every data point to ensure statistical validity.

4.3.1 Sample Naming Nomenclature

Throughout this thesis, a number of samples were tested in a variety of conditions. These conditions are displayed in Table 4.3 and are used throughout the rest of this thesis. Where more than one condition is relevant for a sample, conditions are hyphenated together, e.g. SR-M-V refers to a sample that was stress relieved, machined to shape, and was vertically oriented in the build chamber.

TABLE 4.3. Nomenclature of samples for mechanical testing.

Condition	Heat Treatment			Finishing Process		Part Orientation	
	As-Built	Stress Relieved	T6 Treated	As-Printed	Machined	Horizontal	Vertical
Abbreviation	AB	SR	T6	P	M	H	V

4.3.2 Hardness Testing

Hardness testing was conducted using a Duramin-40 hardness tester. Vickers testing was carried out for all samples, with a load of 1 kg and a dwell time of 15 seconds. Indentations were located in the centre of each sample, with a minimum of ten indentations taken per sample. These tests were carried out according to ASTM E92-23 [266].

4.3.3 Charpy Testing

Charpy testing was conducted due to its sensitivity to property change and its ability to have a high throughput of specimens [261], and was completed using a CEAST 9050 pendulum impact system according to ASTM E2248-18 [267]. Miniature samples were used, the dimensions of which are shown in Figure 4.3, to ensure that specimens could still be manufactured in the later stage of the powder reuse studies when the powder for part manufacturing was limited.

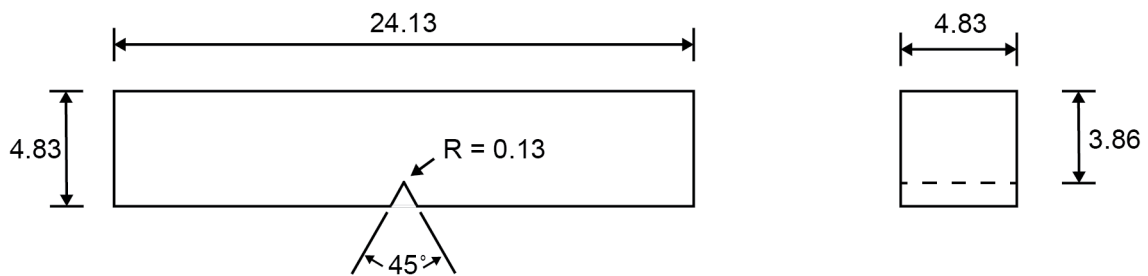


FIGURE 4.3. Mini Charpy specimen dimensions, as per ASTM E2248-18. All dimensions are in mm.

4.3.4 Tension Testing

Tension testing was carried out using an Instron 5567 universal testing machine, with a strain rate of 0.1/min and in accordance with ASTM E8/E8M-24 [268]. Two specimen sizes were used throughout this thesis for a variety of reasons. Both small and large samples were used in Chapter 5 to provide an evaluation on the effect of sample size, while Chapter 6 used large tensile samples. Due to the limited supply of powder for sample manufacture during the later stages of the powder reuse studies in Chapters 7 and 8 of this thesis, both small and large sample sizes were used. These sample dimensions are shown in Figure 4.4.

4.3.5 Flexural Testing

Flexural testing was completed using an Instron 3366 mechanical testing system to conduct three point bending tests using two supports and a former. Testing was displacement controlled with a strain rate of 1.3 /min, with testing terminated at 7 % strain if failure was not reached. Rectangular specimens were used which were 80 mm long and 30 mm wide with a thickness

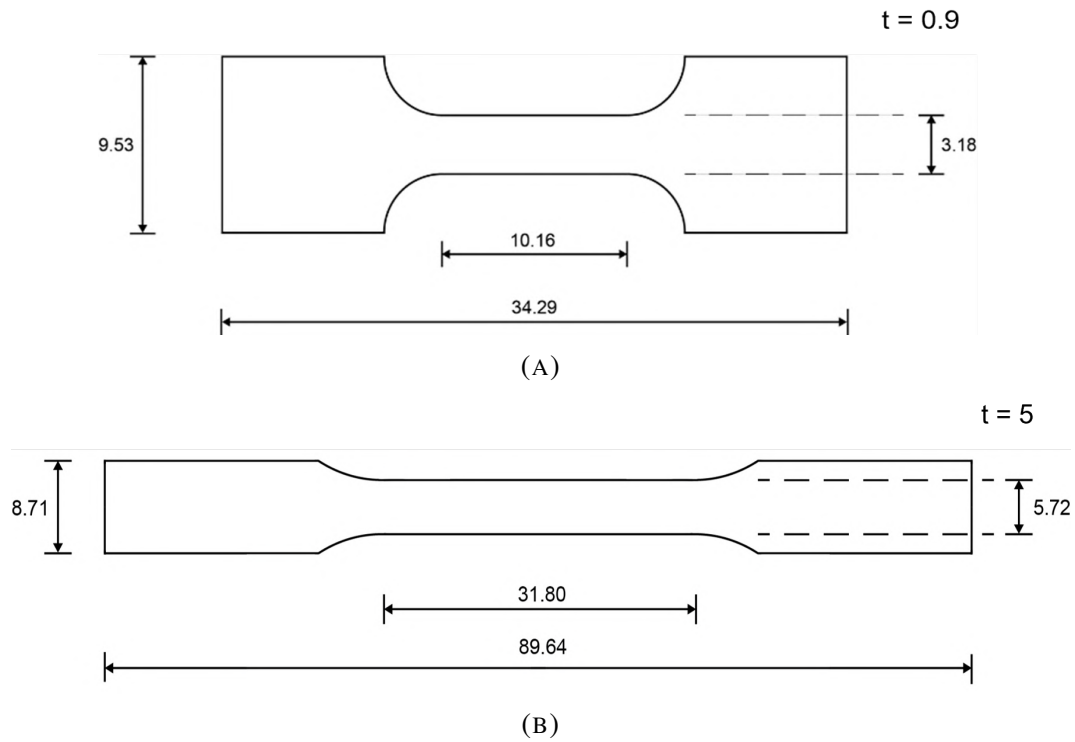


FIGURE 4.4. (a) Customised miniature tensile specimen dimensions, (b) sub-size specimen according to Figure 19 of ASTM E8E8M – 24. All dimensions are in mm.

of 3 mm, as shown in Figure 4.5a. Tests were conducted according to EN ISO 7438:2020 [269] using the set-up shown in Figure 4.5b.

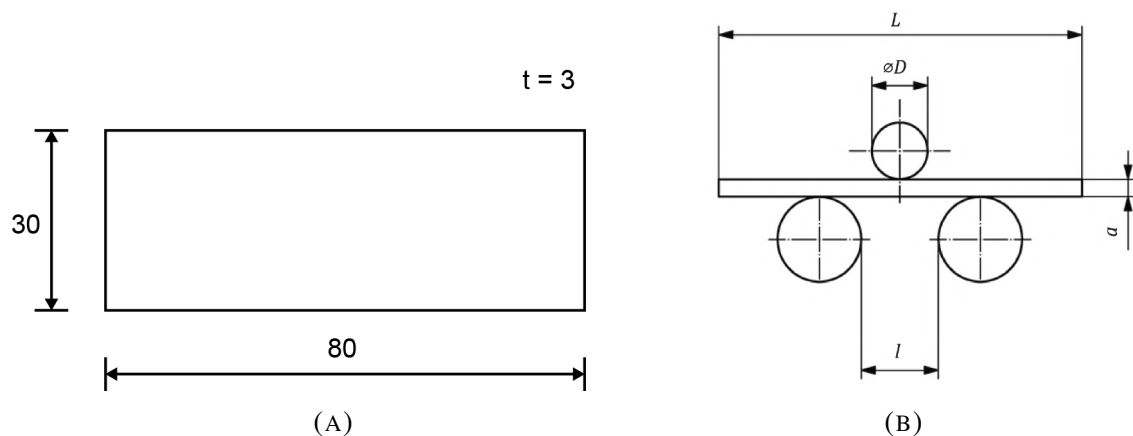


FIGURE 4.5. (a) Flexural testing specimen dimensions, (b) testing apparatus used for flexural specimens. The distance between supports was 60 mm (figure reproduced from [269]). All dimensions are in mm.

4.3.6 Fatigue Testing

Given the reliance of fatigue properties on defects and feedstock characteristics [270] that often change during powder reuse, fatigue testing was included in Chapter 7 of this thesis. This testing was conducted on an MTS 810 material test system, following the procedures as laid out in ASTM E606/E606M-21 [271]. A stress ratio of 0.1 was used at a test frequency of 5 Hz. Samples were machined to shape from additively manufactured cylinders to specimens of a uniform-gauge type with a gauge length of 15 mm, as shown in Figure 4.6, and underwent no heat treatment prior to testing.

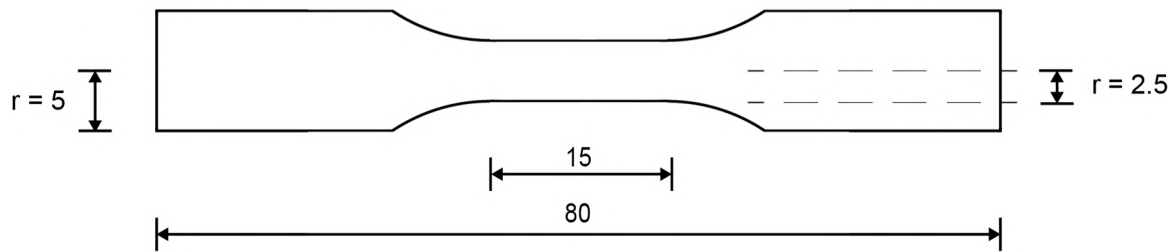


FIGURE 4.6. Cylindrical fatigue testing specimen dimensions used for testing samples manufactured in Chapter 7 of this thesis. All dimensions are in mm, with radii shown where relevant.

4.4 Part Characterisation

The following section outlines the tests that were conducted on parts manufactured as a part of this research. Where these tests were also used on powder samples, this is mentioned. All tests in this section were conducted on witness cubes (13 mm x 13 mm x 13 mm) sized for simple investigation, with sample preparation carried out as required.

4.4.1 Optical Microscopy

Optical microscopy was carried out on printed parts in order to gain an understanding of higher-level microstructural features. This was conducted on a Leica DMI8M microscope, using magnifications of 10x and 20x with images taken using darkfield settings. Samples

were prepared by grinding with silicon carbide papers, before a diamond suspension 3 μm in size and a final polishing step of OPS colloidal silica were used.

4.4.2 Scanning Electron Microscopy

Scanning electron microscopy (SEM) was used to characterise both manufactured parts and powders. Primarily this was by imaging, however, it was also used for chemical mapping and grain characterisation. All samples requiring polishing were first ground with silicon carbide papers before a diamond suspension 3 μm in size and a final polishing step of OPS colloidal silica were used.

Fracture surface images were captured using a Zeiss Evo SEM. An accelerating voltage of 5 kV was used, at a working distance of approximately 30 mm and a spot size of 350 nm.

Imaging of powder samples was conducted using a Zeiss Sigma. An accelerating voltage of 5 kV was used at a working distance of 15 mm to take secondary electron images.

The characterisation of the cell network within the main grain structure of the manufactured alloy was carried out using a Zeiss Sigma, with an accelerating voltage of 20 kV at a working distance of 15 mm. Secondary electron images were taken for comparison between different samples. Where cell size analysis was conducted, ImageJ software was used with cell size measurements taken from seven different imaged locations in a sample. Ten lines were drawn on each image to determine cell size by averaging the number of intercepts along these lines, with lines chosen to intercept equiaxed rather than elongated grains.

Electron dispersive spectroscopy (EDS) was used for qualitative chemical analysis of specimens. This was conducted on a Zeiss Sigma using an accelerating voltage of 20 kV at a working distance of 8.5 mm.

Electron backscattered diffraction (EBSD) was used to analyse the grain structure and crystallographic texture of manufactured parts. This was done in a Zeiss Ultra using an accelerating voltage of 20 kV at a working distance of 15 mm, aperture of 60 μm , and a step size of 1 μm , with data collection and analysis completed using the Aztec suite of software. Grain size measurements presented in all chapters are equivalent circular diameters, with a minimum of 1500 grains measured in each scan.

4.4.3 Transmission Electron Microscopy

Transmission electron microscopy (TEM) was carried out using a JEOL 2200FS with a field emission gun and EDS detector. Scanning transmission electron microscopy (STEM) was conducted with an acceleration voltage of 200 kV. Sample preparation was conducted by twinjet electropolishing with a Struers TenuPol-5 in an electrolyte consisting of 10 % perchloric acid in methanol for as-built and stress relieved samples, while solution treated samples were prepared by dimple grinding on a Fischione Instruments Model 200 dimpling grinder and precision ion polishing on a Gatan PIPS-II at -20 °C. Sample preparation for TEM and the TEM imaging was done by Dr Cecilie Funch (a post-doctoral researcher in Prof Proust's group). The data presented here is part of a paper co-written by the team and enables explanation of the results found in other tests.

4.4.4 Atom Probe Tomography

Atom probe tomography (APT) work was performed on an Inviso 6000, with a dual laser using a 200 pJ laser, 200 Khz pulse rate, and a 2% detection rate. APT tips were prepared by bulk electropolishing in a 25 % perchloric acid solution at 20 kV and finished in a Thermofisher Helios Plasma Focused Ion Beam to mill the electropolished tips to a fine point for data collection. The sample preparation for the APT specimens was conducted by Dr Kevin Sisco (a post-doctoral researcher in Prof Ringer's group) and Dr Cecilie Funch. The APT data collection and data analysis was performed by Dr Kevin Sisco. The data presented here is part of a paper co-written by the team and enables explanation of the results found in other tests.

4.4.5 Micro-computed Tomography

Micro-computed tomography (μ -CT) was used to determine the relative density of samples that had been additively manufactured. For ease of scanning and analysis, these specimens were cylinders specially made for these experiments. For results in Chapter 5 these μ -CT specimens were cylinders of 10 mm diameter with a scan pixel size of 4 μ m, whereas for Chapters 7 and 8 these cylinders were 4 mm in diameter with a scan pixel size of 2 μ m. Scans were collected using a Bruker Skyscan 1272 by Dr Matthew Foley, a staff member of the Sydney Microscopy and Microanalysis at the University of Sydney.

4.4.6 Archimedes Density

Even though the Archimedes density of parts is not as reliable as other techniques for measuring density, it provides a simple means of investigating part density [272]. Archimedes density measurements were therefore conducted to provide a simple initial look at the density of parts manufactured as a part of this research, allowing for a higher throughput of samples than μ -CT. Cubes were lightly polished using silicon carbide paper of grit size 1000 prior to testing to reduce the adhesion of air bubbles to the rough, as-printed surfaces of cubes, with three cubes tested for each data point. Tests were conducted using deionised water, and followed the procedures as set out in the standard ASTM B311-22 [273].

4.4.7 Surface Roughness

Surface roughness measurements were conducted on an Olympus LEXT 5000 laser confocal microscope, using a long working distance lens of 20x magnification. Ten measurements were taken on each surface measured: five parallel to the build direction and five perpendicular to the build direction as shown in Figure 4.7. The measurements taken were Ra values, representing the arithmetic mean deviation. This is a measure of the average height difference for the average surface, i.e. the average distance of the surface from the mean height.

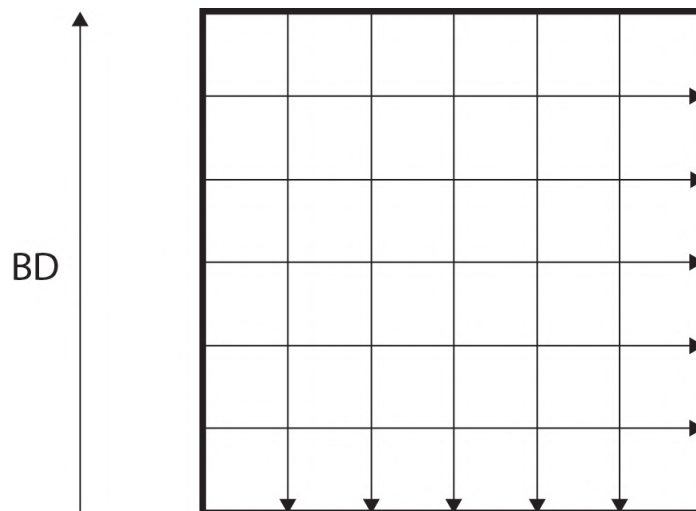


FIGURE 4.7. Measurement lines taken on each surface for surface roughness measurements. Five were taken parallel to the build direction, and five perpendicular.

For measurements in Chapter 5, surface roughness was measured on the side of each sample, as shown in Figure 4.8a. Measurements were taken from a square region (10 mm x 10 mm) of each face at least 1 mm from the edges of the cubes.

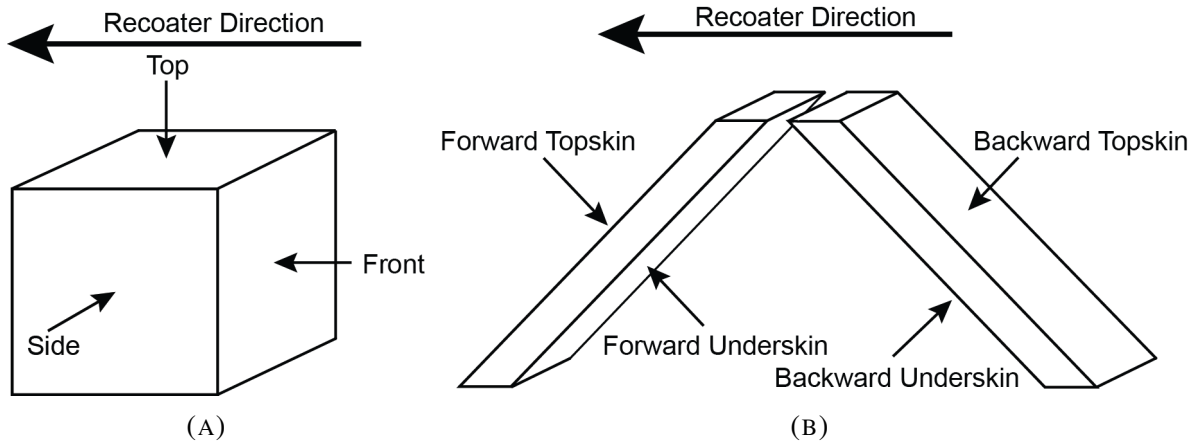


FIGURE 4.8. Parts manufactured for surface roughness measurement: (a) surface roughness cube, (b) surface roughness sloped samples, with nomenclature used throughout this thesis.

For roughness measurements in Chapters 7 and 8, measurements were taken on all surfaces of the cube samples. The front, side, and top of cubic samples were measured with measurements taken from a square region (10 mm x 10 mm) at least 1 mm from the edges. Sloped surfaces were also measured in Chapter 7, with both the top skin and under skin measured. Sloped surfaces designated forward leaned towards the powder chamber, while those designated backwards leaned away from the powder chamber and towards the overflow of the M2. These surfaces are represented in Figure 4.8b. Measurements were taken from a rectangular region (11.7 mm x 21.3 mm) of each face at least 1 mm from the edges.

The Effects of Part Location Within the Build Volume

5.1 Introduction

There has been considerable research investigating the effects of processing parameters on PBF parts, however, this has tended to focus on parameters dictating the volumetric energy density input, which is calculated using the following equation:

$$E_V = \frac{P}{v \times h \times t} \quad (5.1)$$

where P is the laser power, v is the scan speed, h is the hatch spacing, and t is the layer thickness. These parameters are some of the simplest to change in PBF processes and as such have been regularly manipulated to firstly produce fully dense parts, and then create desirable microstructures for optimal mechanical properties. However, there are many more parameters than these that affect part properties, leaving considerable room for improvements.

Other parameters are important to understand so that parts can be manufactured such that their properties are both known and consistent. This is especially true regarding a part's location within the build volume, with factors including the location of a part on the build plate [84, 98, 99] and the height of a part [95, 96, 274] potentially playing a role in the part's properties. This may be due to factors such as uneven powder spreading due to differential powder settlement [275], or build plate preheating leading to in-situ stress relieving and precipitate growth [144]. For these reasons, it is worthwhile investigating the effect that part location within the build volume has on microstructure and mechanical properties.

Similarly, the dimensions of parts may influence the material microstructure and properties. It is known that tensile strength can change with part size due to factors such as the increased

probability of critical defects being present in large samples, while stress concentration distributions may be different depending on size. This is complicated by the porosity present in PBF manufactured parts, especially given the increased porosity near the surface of parts [276]. This porosity will therefore represent a higher portion of the material in smaller parts with a higher surface area to volume ratio, and so it is worthwhile investigating this phenomenon for additively manufactured parts.

This chapter investigates the effects of three factors that may influence the properties of parts: their location on the build plate, their distance from the build plate, and the size, orientation and surface finish of parts. The following sections present the results of this investigation along with discussions of these results.

5.2 The Effect of Build Plate Location

To determine the effect of build plate location on part properties, a manufacturing run was completed with specimens placed on a grid of nine locations on the build plate, as shown in Figure 5.1. The recoater spread powder from right to left in the build chamber, with gas flowing in the same direction. At each location tensile samples, a witness cube, and a density cylinder were manufactured for analysis, the geometries of which are described in Chapter 4.

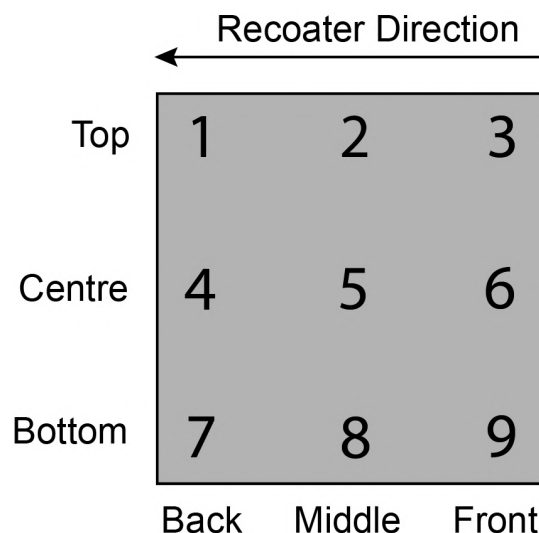


FIGURE 5.1. Build plate layout of the manufacturing run conducted for this work. Each number serves as a reference location for parts taken from each section of the build plate.

5.2.1 Tensile Properties

The tensile properties at each location were tested to determine if there is any relationship between build plate location and mechanical properties. These samples were large tensile samples as per Chapter 4 and shown in Figure 5.2, vertically oriented, printed to shape, and their properties are presented in the following sections.

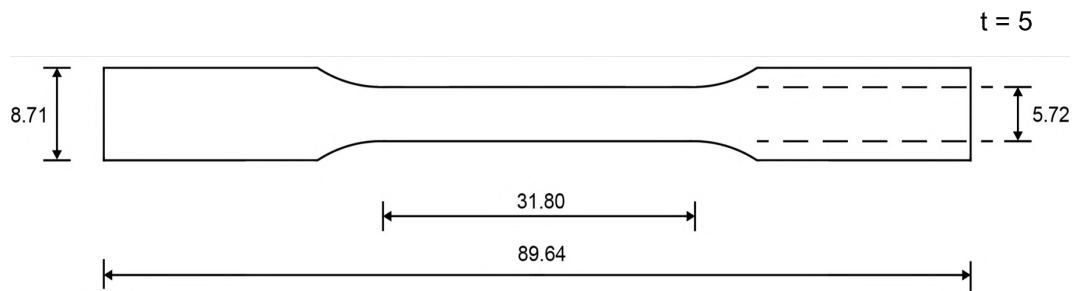


FIGURE 5.2. Dimensions of tensile specimens manufactured at each location on the build plate.

5.2.1.1 Yield Strength

The yield strengths of samples taken from each location on the build plate are presented in Figure 5.3. There is no significant variation in yield strengths across the build plate, with a minimum strength of 192.6 MPa at location 2 and a maximum of 197.0 MPa at location 5.

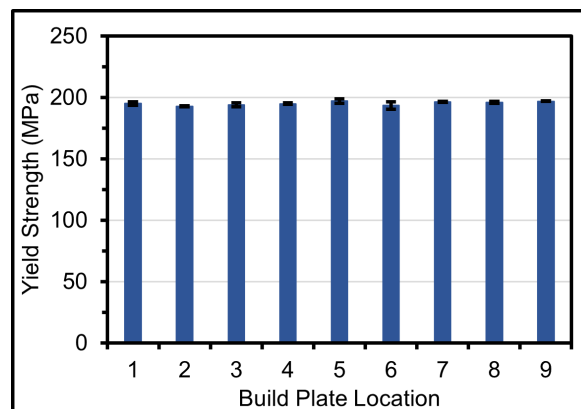


FIGURE 5.3. Yield strength of samples taken from each location on the build plate.

5.2.1.2 Tensile Strength

The tensile strengths of the samples taken from different locations on the build plate are shown in Figure 5.4. In a manner similar to the yield strength there is no significant relationship between the tensile strength and build plate location, with the minimum tensile strength of 362.2 MPa found at location 2, and a maximum strength of 367.9 MPa at location 8.

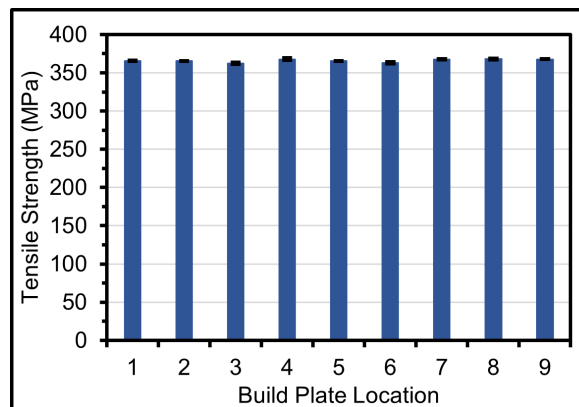


FIGURE 5.4. Tensile strength of samples taken from each location on the build plate.

5.2.1.3 Ductility

The ductility of each of the specimens tested is presented in Figure 5.5, with a maximum ductility of 12.0 % found at locations 8 and 9, and a minimum ductility of 10.7 % at location 5. While this represents greater variation than that found in the yield and tensile strengths, due to the larger standard deviations in these results these variations are statistically insignificant.

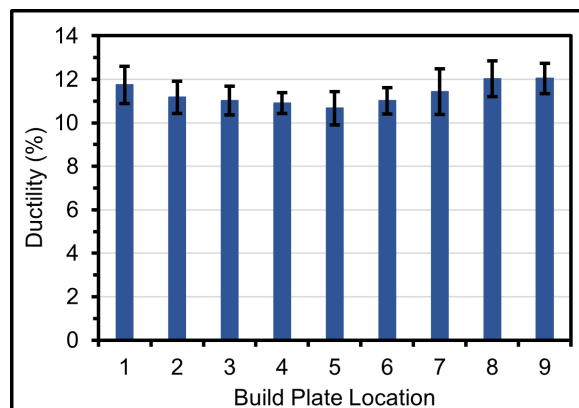


FIGURE 5.5. Ductility of samples taken from each location on the build plate.

5.2.2 Microstructure

5.2.2.1 Grain Size Characterisation and Crystallographic Texture

Characterisation of the grains reveal that there is minimal difference in structure, shape, and texture across each of the build plate locations. Each location has a grain structure that is characteristically dominated by columnar grains growing with the build direction alongside smaller equiaxed grains, as shown in Figure 5.6, following the observations typically made in the literature for parts manufactured by PBF.

There is also no significant difference in grain size, with Table 5.1 showing grain sizes ranging from 9.51 μm at location 1 to 10.49 μm at location 3, with these minor differences attributable

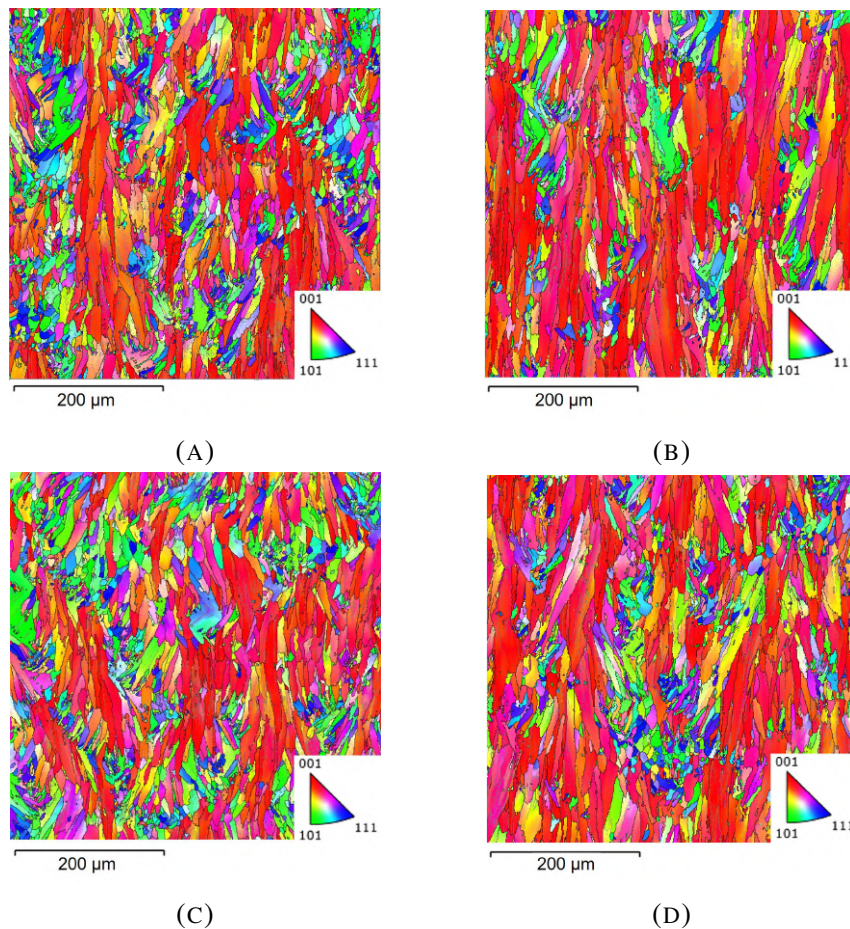


FIGURE 5.6. Inverse Pole Figure (IPF) maps from build plate locations at the corners of the build plate: (a) location 1, (b) location 3, (c) location 7, (d) location 9.

TABLE 5.1. Grain size and maximum pole figure intensity as a function of build plate location.

Location	Grain Size (μm)	Maximum Pole Figure Intensity
Location 1	9.51 ± 7.14	3.54
Location 3	10.49 ± 9.57	5.67
Location 7	9.65 ± 7.29	2.82
Location 9	10.16 ± 8.77	4.73

to random sampling within each specimen and the number of melt pools present in each map area. The large standard deviations associated with each measurement also reduces the significance of these slight differences in grain size.

There is, however, a slight difference in texture in each location, specifically along the recoater direction. Figure 5.7 contains the pole figures from each of the four corner locations of the build plate, showing that locations 3 and 9 have a slightly stronger texture in the $[100]$

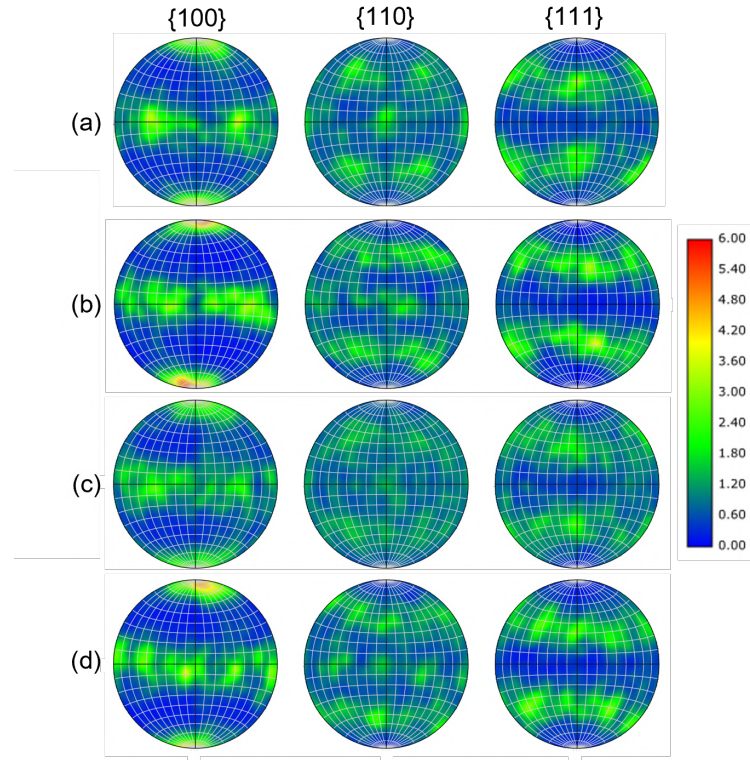


FIGURE 5.7. Pole figures of samples from build plate locations at the corners of the build plate: (a) location 1, (b) location 3, (c) location 7, (d) location 9.

direction than the microstructures at locations 1 and 7. This indicates that the texture is slightly stronger at locations near the beginning of powder spreading.

5.2.2.2 Cell Structure

Within the grains of aluminium-silicon alloys is a network of eutectic silicon, distributed to create a cell structure within the larger grains. A comparison of the cell structure at each of the four corner locations of the build plate indicate that there is good consistency of size distribution, as shown in Figure 5.8. These SEM micrographs show the zones characteristic to the melt pool, with the elongated cells of the large grains nearer the centre of the melt pool and finer, more equiaxed cells nearer the heat affected zones.

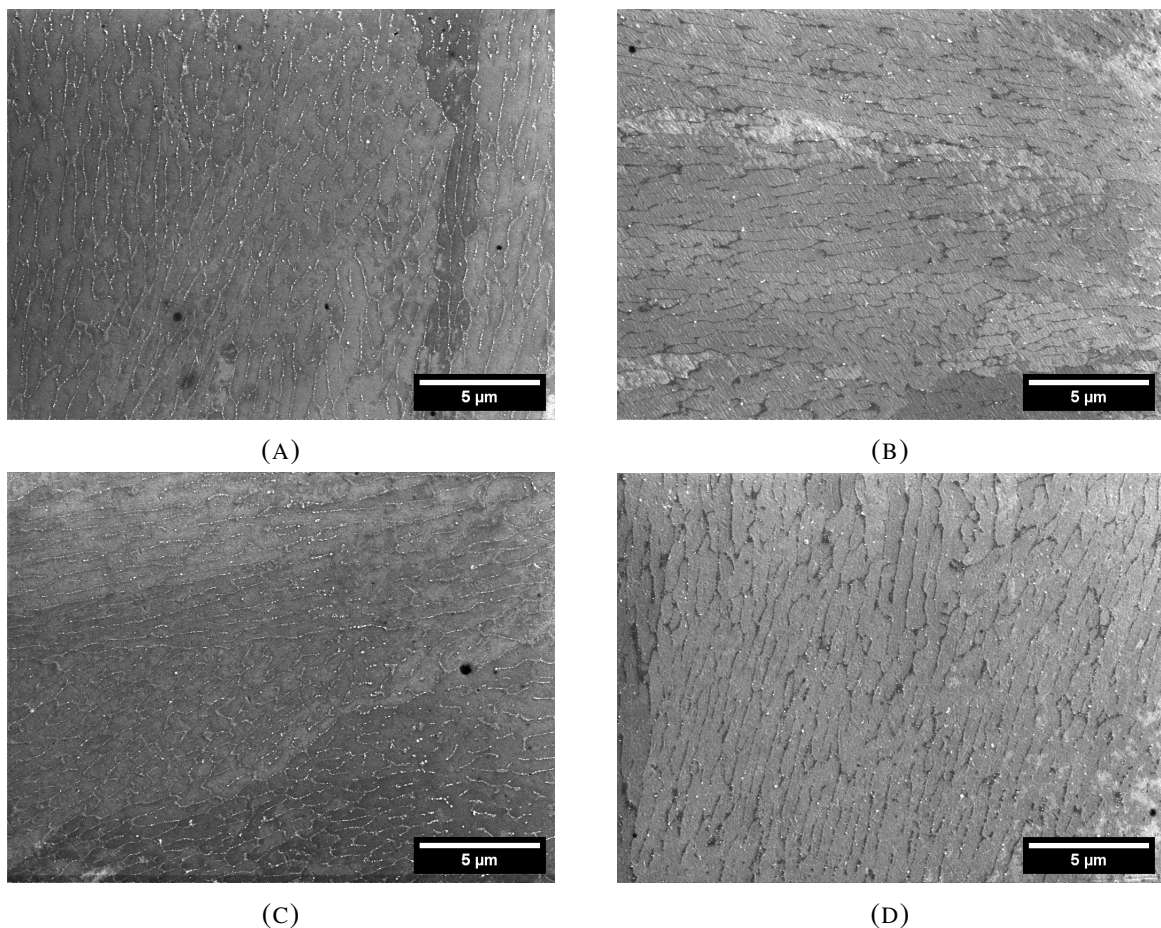


FIGURE 5.8. SEM micrographs of samples from build plate locations at the corners of the build plate: (a) location 1, (b) location 3, (c) location 7, (d) location 9.

5.2.3 Density

Part density as measured by μ -CT scans are displayed in Table 5.2. There is limited variation in density for each location on the build plate, with each sample tested showing high densification. These densities are all equal to or greater than that laid out in the manufacturers data sheet of 99.6 % relative density, indicating good process control over all locations of the build plate.

TABLE 5.2. Part density at the four corner locations of the build plate.

Location	Relative Density (%)
Location 1	99.60
Location 3	99.65
Location 7	99.62
Location 9	99.81

5.2.4 Surface Roughness

The surface roughness at each build plate location, measured by the Ra value from the sides of samples as per Chapter 4, is displayed in Table 5.3. This shows there is minimal difference in surface roughness for each location on the build plate, although an ANOVA analysis yields a p value of 0.00001, indicating that there is a statistically significant effect of build plate location on surface roughness.

TABLE 5.3. Surface roughness for each location on the build plate.

Location	Ra (μm)
1	5.028
2	5.134
3	5.222
4	5.786
5	5.527
6	5.342
7	6.290
8	5.757
9	6.354

A comparison of the sections of the build plate shows no difference in part roughness between the front and back of the build plate along the recoater direction, as shown in Figure 5.9a. There is however a statistically significant difference between the roughness of the top section of the build plate compared with the bottom and centre sections, with a two tailed t-test producing p values of 0.039 and 0.007 when comparing the top to the centre and bottom respectively. These roughnesses are shown in Figure 5.9b.

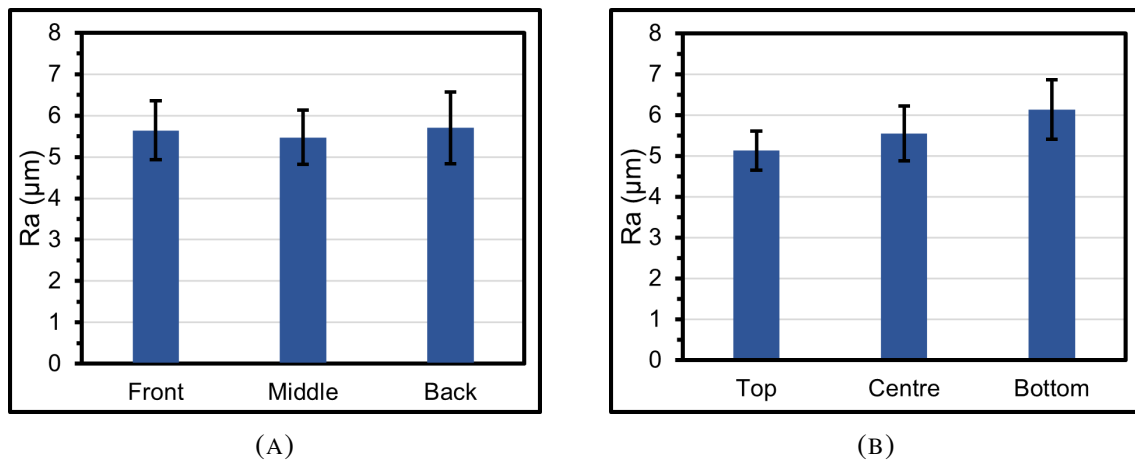


FIGURE 5.9. Surface roughness for each zone on the build plate: (a) from left to right (b) from top to bottom.

5.2.5 Discussion

Overall, there is excellent consistency of properties across each of the locations on the build plate. This is especially true for the tensile properties of samples at each location, with no significant variations in yield or tensile strengths. While there is some variation observed in the ductility, it is not statistically significant.

The microstructure of these samples also shows good consistency, with no significant changes in either the grain size or cell structure. This is to be expected of any well calibrated PBF machine, given these characteristics rely on the melt pool behaviour. Grain size and the distribution of columnar and equiaxed grains within each melt pool is dictated primarily by the rapid cooling rates associated with PBF [277, 278], and so a consistent microstructure is indicative of consistent heat flow across the build plate. Any variations in the grain characteristics that may potentially arise as a function of build plate location may also be mitigated by the use of a heated build plate, as was the case in this study.

The only difference in microstructure across each of the locations on the build plate relates to texture, with samples manufactured closer to the powder hopper, i.e. locations that had powder deposited first during recoating, having a slightly stronger texture than those near the overflow chamber. While this is evident by the average maximum intensity of 5.2 for samples near the powder chamber and 3.18 for those near the overflow chamber, as in Table 5.1, this is likely due to random sampling. This is partly due to the lack of change in grain size. Given the texture of large, columnar grains is much stronger than that of the fine equiaxed grains also found in PBF microstructures, their increased presence would result in an increased texture intensity. While a slight change is seen in the grain size related to part location, there is not a large enough increase in grain size to induce a large change in texture. Since the texture of a material is also known to influence its mechanical properties [279, 280], the lack of change in the tensile properties at each build plate location also suggests there has been no real change to microstructural texture. This is supported by the fact that there is no other change in properties when comparing the front and back of the build plate. Together, these changes indicate the differences in texture observed due to build plate location are the result of random sampling rather than a genuine relationship.

Density measurements conducted by μ -CT show that there is minimal difference due to build plate location. Given that each location demonstrates high levels of densification, this indicates that the production process is stable across all locations, particularly the melting process. Melt pool instabilities lead to the formation of porosity [281] as does defocusing of the laser [282], which has the potential to occur over different locations on the build plate given the differing distances the laser must travel to reach the powder bed. Therefore, the minimal porosity, indicated by the high density, suggests a consistently stable melt pool and focused laser. This also contributes to the consistent tensile properties seen.

While showing statistically significant differences, the surface roughness data indicates only small variation between locations. This is true along the direction of the recoater, with an average Ra value of 5.64 μm at the front locations, and 5.70 μm at the back locations. Given surface roughness is influenced by the particle size distribution [80] and segregation of powders by size during recoating has been observed [275], this indicates that there is minimal differential settlement of powder across the build plate upon recoating.

However, the difference in surface roughness becomes apparent when comparing the top and bottom of the build plate. Given the otherwise consistent properties over the build plate, including the surface roughness as a whole, it is difficult to attribute this change in roughness to any factors. This is especially true given the gas flow and recoater directions are both from the right to the left of the build plate, and therefore will not induce a change in surface roughness from the top to the bottom of the build plate. The most likely explanation for the increased surface roughness at the bottom of the build plate was the closer proximity of the measured surfaces to other parts when compared to samples at the top of the build plate. This increased proximity makes it more likely that spatter particles from the melting of nearby parts will fall along the edges of the tested surfaces. These spatter particles, due to their larger shape, increased agglomeration, and higher oxidation rates when compared to other particles, have the potential to alter the melt pool dynamics and therefore its stability. This, in combination with larger particles sintering to the edge of parts, may increase the surface roughness of parts in close proximity to others, thereby increasing the surface roughness of parts near the bottom of the build plate in comparison to those at the top. This indicates that this change of surface roughness is not attributable to the build plate location. This slight change in surface roughness also did not result in changed tensile properties, indicating that these changes are both small and insignificant when considering mechanical properties.

5.3 The Effect of Part Location Along the Build Direction

Another factor that is not commonly considered during the production of PBF components is the location along the build direction of a part, i.e. its distance from the build plate. There is considerable potential for property variation across a part's height due mainly to heat accumulation within a build volume, which becomes especially pertinent given the increasing prevalence of heated build plates during production. This section therefore examines the hardness along a part 250 mm tall, together with a microstructural investigation, to determine the property inconsistencies over the part height.

5.3.1 Hardness

To determine if there is inhomogeneity along the height of a sample, hardness testing was conducted at 10 mm intervals along a vertically oriented sample. The results of this testing are shown in Figure 5.10. There is a clear reduction in hardness with increasing distance from the build plate, decreasing from 127.7 HV 10 mm from the build plate to 109.4 HV 250 mm from the build plate. This effect is eliminated by a stress relieving treatment of 280 °C for 2 hours, with the hardness staying consistent at approximately 80.9 HV.

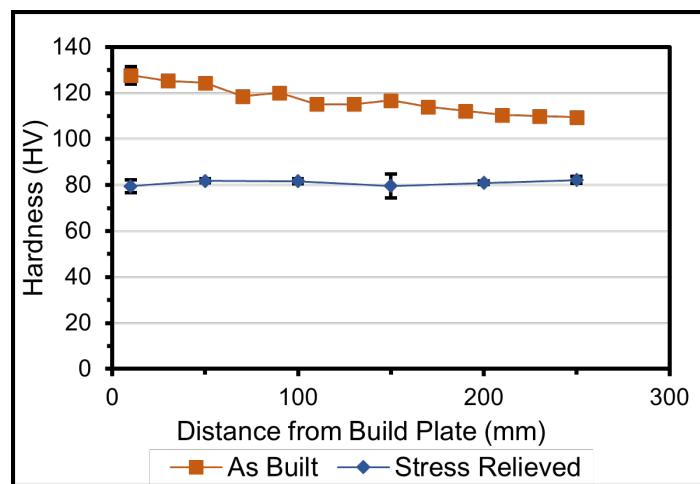


FIGURE 5.10. Hardness of specimens according to distance from the build plate.

5.3.2 Melt Pool Structure

Investigation by optical microscopy allows for a large-scale investigation into the macroscopic melt pool structures of PBF manufactured parts. Figure 5.11 shows darkfield micrographs of samples taken from the bottom, middle, and top of the vertically orientated part. There is minimal difference between these micrographs, with each showing the fish-scale melt pool shape characteristic of PBF components. These are of similar size, shape, and orientation, suggesting no difference in the melting of parts due to distance from the build plate.

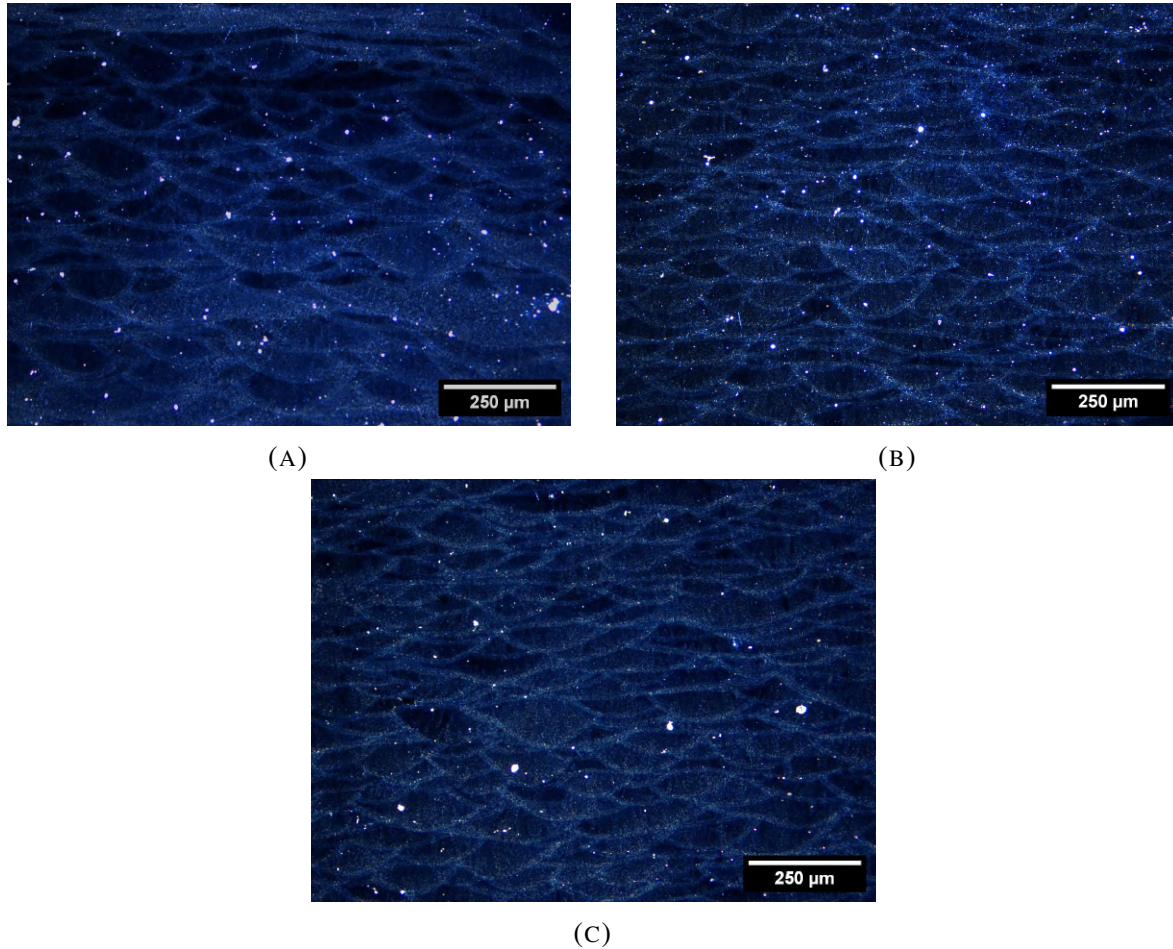


FIGURE 5.11. Darkfield optical micrographs of samples taken from different distances from the build plate: (a) 10 mm from the build plate, (b) 130 mm from the build plate, (c) 250 mm from the build plate.

5.3.3 Grain Size Characterisation and Crystallographic Texture

EBSD maps were taken at representative heights along the build direction, as shown in Figure 5.12. Each map shows a similar grain pattern with groups of columnar grains surrounded by smaller equiaxed grains, each section corresponding to a melt pool. There is no significant difference in this structure depending on the sample's distance from the build plate. This is also true of the grain size at each location, with minimal variation observed between samples. These results are displayed in Table 5.4.

There is, however, a slight difference in the texture of samples taken at varying heights. Figure 5.13 shows the pole figures from samples 10 mm, 130 mm, and 250 mm from the build

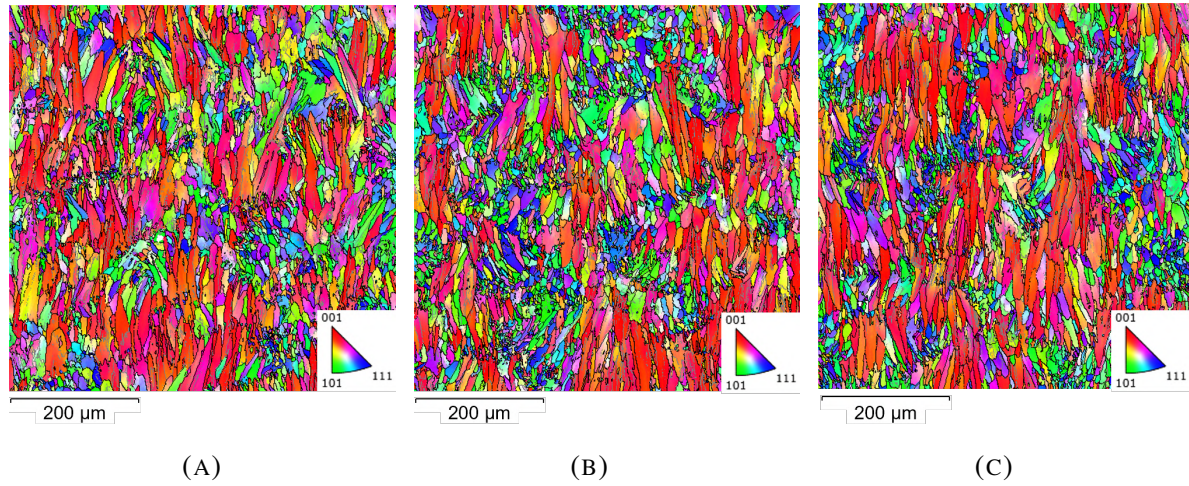


FIGURE 5.12. IPF maps of samples taken from different distances from the build plate: (a) 10 mm from the build plate, (b) 130 mm from the build plate, (c) 250 mm from the build plate.

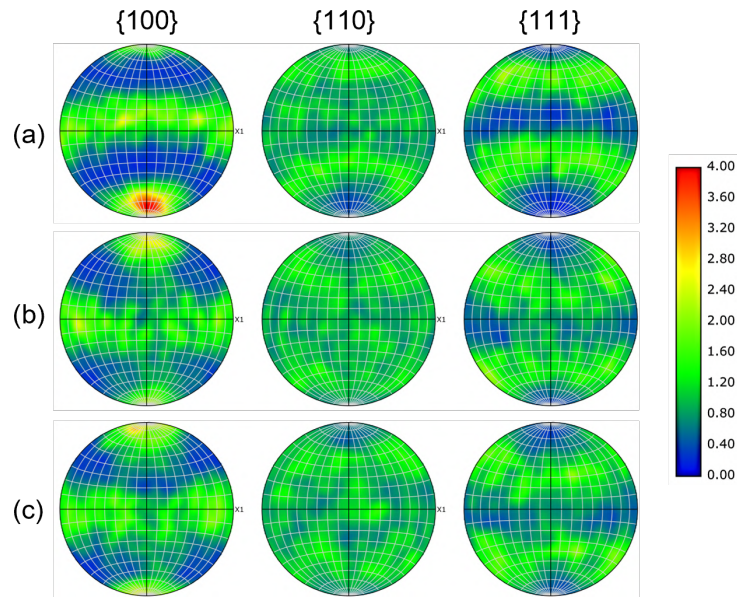


FIGURE 5.13. Pole figures of samples taken from different distances from the build plate. (a) 10 mm from the build plate: (b) 130 mm from the build plate, (c) 250 mm from the build plate.

plate. These pole figures show that the [100] direction is favoured, with a stronger texture for the sample closest to the build plate with a maximum intensity of 4.0, compared to the maximum intensities of 2.69 and 2.97 for the samples 130 mm and 250 mm from the build plate respectively. The maximum intensity values are also presented in Table 5.4.

TABLE 5.4. Grain size and maximum pole figure intensity according to distance from the build plate.

Distance from Build Plate (mm)	Grain Size (μm)	Maximum Pole Figure Intensity
10	9.14 ± 7.2	4.0
130	8.29 ± 6.35	2.69
250	8.86 ± 6.61	2.97

5.3.4 Cell Structure

Representative micrographs of the cell network at various distances from the build plate are shown in Figure 5.14, showing that the morphology of these cells remains roughly similar with both elongated and equiaxed cells present in each sample. The average size of these cells is presented in Table 5.5, indicating that as a sample's distance from the build plate increases,

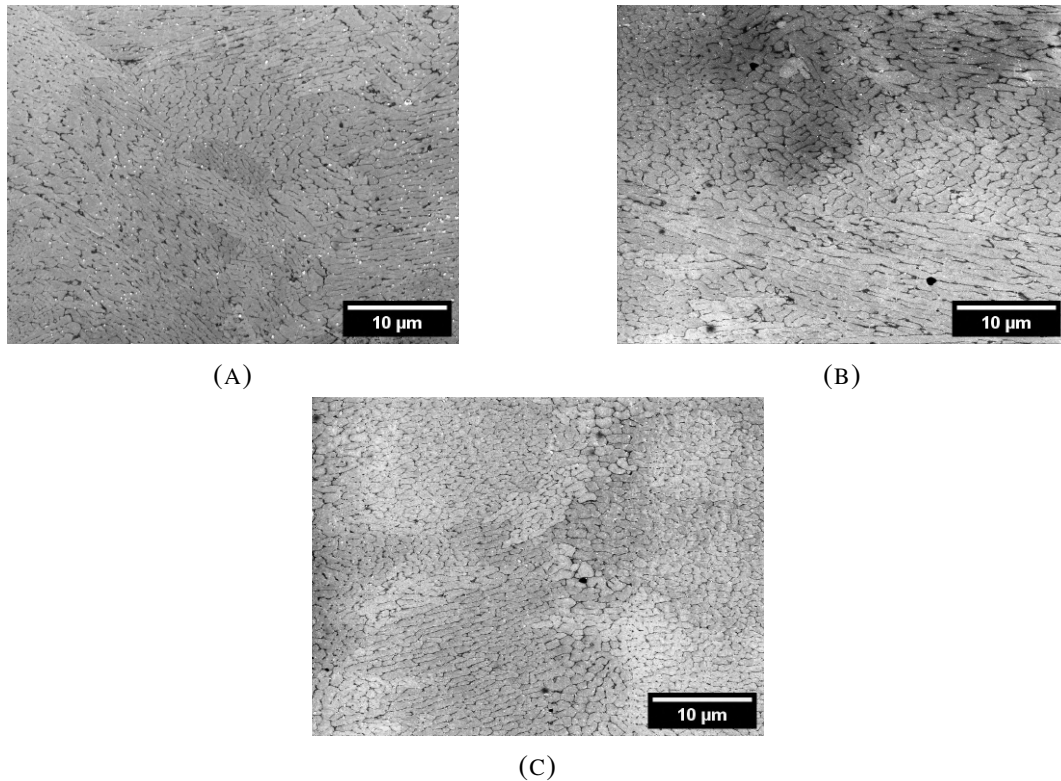


FIGURE 5.14. SEM micrographs of the cell network, taken from different distances from the build plate: (a) 10 mm from the build plate, (b) 130 mm from the build plate, (c) 250 mm from the build plate.

there is an increase in average cell size. Equiaxed cells are $0.636\ \mu\text{m}$ in diameter 10 mm from the build plate, increasing to $1.014\ \mu\text{m}$ 250 mm from the build plate.

TABLE 5.5. Average size of the silicon cell network across different locations in the build height. The images used to calculate these measurements can be found in Appendix A.

Distance from Build Plate (mm)	Cell Size (μm)
10	0.636 ± 0.159
130	0.867 ± 0.218
250	1.014 ± 0.251

5.3.5 Chemical Mapping

Chemical maps obtained by EDS are shown in Figure 5.15. These maps reveal that the silicon has strongly segregated to the walls of the cell structure, as is commonly observed

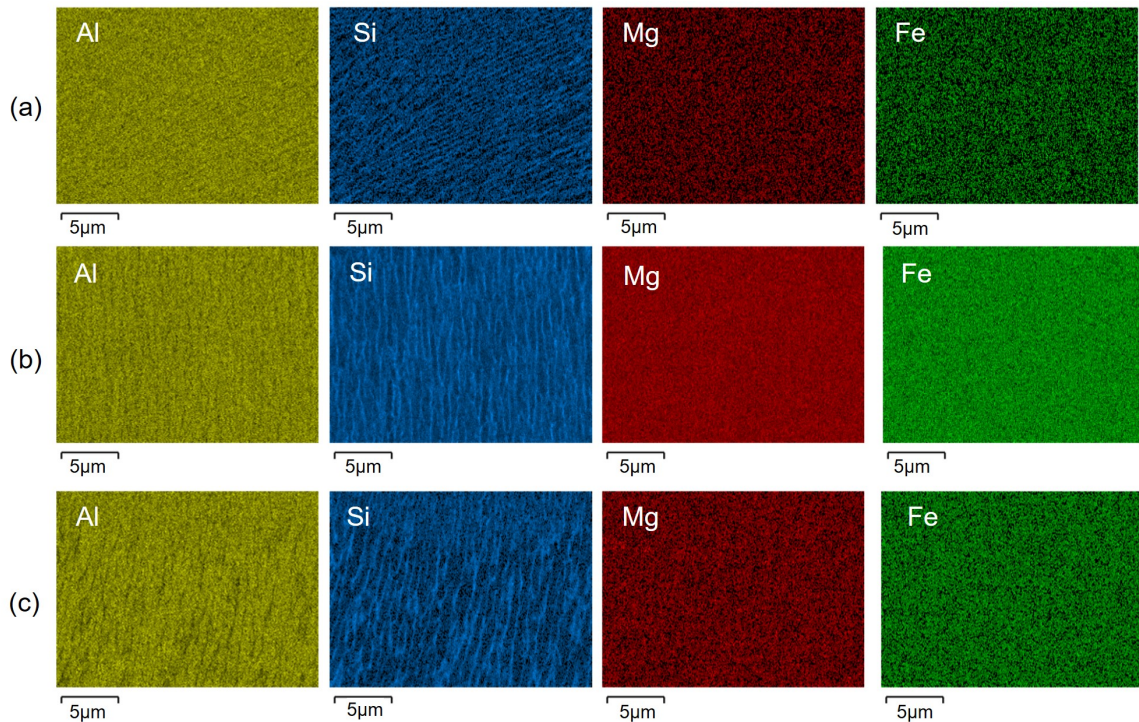


FIGURE 5.15. Chemical maps of samples taken from varying distances from the build plate. (a) 10 mm from the build plate: (b) 130 mm from the build plate, (c) 250 mm from the build plate. Differences in cell sizes in these maps are due to the location of the measurement within the melt pool rather than differences in elemental segregation behaviour.

in the literature regarding aluminium-silicon additively manufactured alloys [283]. These cell walls are also devoid of aluminium, which comprises the bulk of the matrix. The other main alloying elements, magnesium and iron, are present in low quantities and are distributed relatively homogeneously throughout the microstructure. Differences in the cell sizes shown in these maps is due to the location in the melt pool where measurements were taken, rather than any difference in alloy segregation due to sample height.

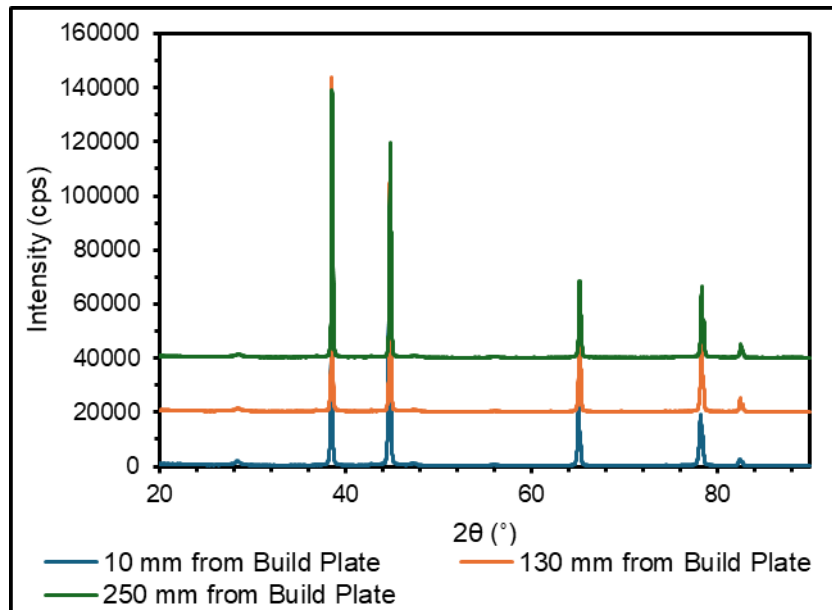
5.3.6 Phase Analysis

To determine any changes in phase that had occurred over the length of the sample XRD analysis was conducted; the obtained spectra are displayed in Figure 5.16. These show that there is no noticeable difference in the spectra as a whole, with peaks for aluminium and silicon showing minimal variation. Figure 5.16b shows a higher magnification spectrum around the main peak for Mg_2Si at $2\theta = 40.2^\circ$, showing that the peak for this phase for the sample 10 mm from the build plate is slightly higher than for other samples. However, as no other Mg_2Si peaks are present in the spectra this cannot be taken as a quantitative assessment.

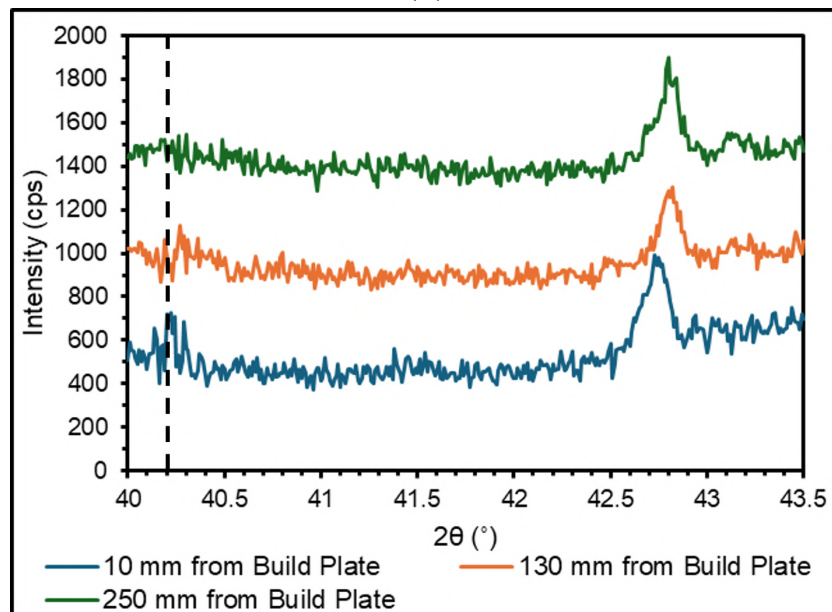
5.3.7 Discussion

The relationship between hardness and location along the build direction is clearly seen in Figure 5.10 where for samples that have undergone no post processing heat treatment, the hardness reduces consistently with increasing distance from the build plate. This suggests that other mechanical properties will also have varying properties depending on a part's distance from the build plate, and an understanding of the reasons for this will allow for the proper manipulation and utilisation of this phenomena.

The presence of varying properties according to part height is eliminated upon a stress relief treatment, showing that this is enough to homogenise the microstructure as well as reducing the overall hardness of the sample. This suggests that any reasons for the variation in hardness values are due to factors that can be eliminated by a stress relief. This also means that for many parts that undergo stress relief prior to their removal from the build plate, this mechanical variation is not a factor that needs to be considered.



(A)



(B)

FIGURE 5.16. XRD diffraction spectra for: (a) an extended 2θ range (b) a range focused on the Mg_2Si peak at $2\theta = 40.2^\circ$, with a dashed line at 40.2° .

While there are many factors that contribute to the changing hardness over the build height, some may be ruled out as being inconsequential. Given the hardness samples were all taken from one location on the build plate it can be assumed that the melt behaviour is consistent

across the build height. This is confirmed by the micrographs of Figure 5.11 which show no difference between the melt pools at the top, middle, and bottom of the sample tested. This suggests that the change in hardness is not related to changes in melt pool size, and by extrapolation this change is also unrelated to the locations and size of various regions within the melt pool such as the melt pool core and heat affected zone.

The same is true for any solid solution strengthening, with the EDS maps shown in Figure 5.15 indicating that there is no noticeable difference in elemental distribution between samples, with strong segregation of silicon to the cell boundaries while the other alloying elements, magnesium and iron, are homogeneously spread throughout the aluminium matrix. The consistency of this chemistry indicates no significant differential strengthening across the sample, with any solid solution strengthening of the silicon, magnesium, and iron within the aluminium matrix being relatively consistent across the build height. The application of a stress relief will, however, influence the amount of solid solution strengthening over the entirety of the build height. The stress relief treatment increases the diffusion of the silicon within the matrix, allowing it to migrate towards the cell boundaries to enlarge the silicon precipitates at these locations. This is evident in TEM-EDS measurements shown in Table 5.6, where the amount of magnesium, silicon, and iron can be seen to decrease in the matrix as a result of a stress relieving heat treatment. The application of a stress relief can therefore be seen to reduce the amount of solid solution strengthening, lowering the hardness of the material and so contributing to the significant reduction in strength evident in stress relieved samples.

TABLE 5.6. TEM-EDS point analysis for the chemical composition of the cell matrix in representative as-built and stress relieved samples. Measurements are taken as an average of 7-14 point measurements. Values are in wt.%.

	Al	Si	Mg	Fe
As-built	98.5 ± 0.2	1.29 ± 0.2	0.18 ± 0.03	0.05 ± 0.02
Stress relieved	99.7 ± 0.03	0.17 ± 0.01	0.12 ± 0.02	0.03 ± 0.02

A comparison of the grain structure of each of the samples reveals that there is minimal difference in grain size for each location tested, with Figure 5.12 and Table 5.4 showing this. This indicates that grain boundary strengthening by the Hall-Petch relationship is not playing a significant role in changing the strength across the build height. The small difference in texture is likely due to the nature of random sampling and the fact that the sample taken

from nearest the build plate was slightly angled during analysis, producing a slightly different texture orientation when measured.

The cell network makes a significant contribution to the strength of additively manufactured aluminium-silicon alloys, with the silicon borders of these cells inhibiting dislocation motion [283]. The results in Table 5.5 show that the cell size increases over the height of the part, from 0.636 μm at the bottom to 1.014 μm at the top. As higher energy inputs have been linked to larger cell sizes [284], it follows that this change in size is linked to the different cooling rates experienced at different locations of the build height. Over the course of a manufacturing run, the continual application of heat by both the heated build plate and the passing of the laser increases the temperature of the powder bed as a whole. This results in a greater residual heat for parts created at higher locations in the build volume, lowering the cooling rate slightly. This lower cooling rate allows for greater diffusion of the silicon through the aluminium matrix during solidification, creating a coarser structure and therefore larger cell size. Similar results have been seen by other authors, whereby heated build plates and the accumulation of heat have lowered the cooling rates and produced finer microstructures [116, 285, 286]. These smaller cells therefore create a greater opportunity for dislocation pile up, increasing the material strength. This small change in cell size can therefore be seen to play a role in changing the hardness of the part at different heights.

These cells are affected by a stress relief treatment, whereby the silicon along these cell walls agglomerates into larger precipitates [139]. These precipitates are still loosely located along the original cell wall boundaries where they become considerably more discrete due to their larger size, while some of the silicon in solid solution with the aluminium matrix is also drawn into these precipitates [111]. These larger and more discrete precipitates are less effective at inhibiting dislocation motion and therefore allow for greater deformation at lower loads, reducing the material's hardness. This effect is consistent over the build height, with the cell structure destroyed regardless of its initial size. This therefore has the effect of both considerably reducing the strength of the alloy, given the significant role these cell boundaries play in strengthening, as well as increasing isotropic behaviour over the length of the build height.

The other strengthening mechanism apparent within these samples is the likely growth of strengthening precipitates on precipitation path of the Mg_2Si strengthening phase. Due to

the small particle size and low presence by volume of this phase, given the low magnesium content of the alloy and the relatively low temperatures and times involved in the production of a single part, this phase cannot be seen by larger scale microstructural investigations such as SEM-EDS and is difficult to quantitatively identify by XRD. Figure 5.16b shows a small peak for Mg_2Si at a 2θ of 40.2° , however, this is the only peak for this phase present over the spectrum in Figure 5.16a meaning that a quantitative and accurate determination of the presence of this phase is impossible. It is, however, likely to be present in small amounts, especially in samples closer to the build plate given their slightly higher intensity peak. As will be discussed further in Chapter 6, these precipitates are likely in the early stages of growth and so are more likely to be GP zones, β'' , or β' precipitates rather than fully formed Mg_2Si precipitates. This is as the heat accumulation during the manufacturing process, a function of the heated build plate and continual passing of the laser, serves as an in-situ ageing treatment for parts. Those nearer the build plate are affected by this most acutely due to the continual proximity to one of these heat sources in the build plate, as well as the longer time remaining at these elevated temperatures. This will promote precipitate growth on a small scale, including the Mg_2Si phase at different stages of precipitation. This phase is known to be the strengthening phase of the AlSi7Mg alloy [111, 145, 287] and so its growth is the aim of ageing treatments carried out as a part of further heat treatment in many applications. The increased presence of this phase in samples closer to the build plate will lead to an increase in strength as the small size of these particles, often in the range of tens of nanometres, allows for an increase in precipitation strengthening. Therefore, the likely increased presence of GP zones, β'' , or β' precipitates in locations near the build plate will contribute to the higher hardness values found in these locations.

Upon stress relief, the continuous growth of these precipitates leads to a reduction in their strengthening effect, with the larger particles formed after a two-hour stress relief treatment less able to inhibit dislocation motion. While this is likely true, further investigation with detailed microscopy at high resolution is needed to confirm this hypothesis. The change in strengthening provided by these precipitates is also likely overshadowed by the considerable loss of strength experienced due to the alteration of the cellular silicon network by the same heat treatment.

Given a stress relieving heat treatment provided a considerable decrease in strength, it follows that the residual stresses within the samples also contributed to the hardnesses measured across the build height. While measurements of these residual stresses were not undertaken as a part of this research, they remain a necessary consideration. Due to the rapid heating and cooling cycles experienced during PBF processing, residual stresses form during processing as a result of the interaction between already solidified material and newly deposited, rapidly cooling material. This induces stresses as seen in Figure 5.17a. During manufacture the

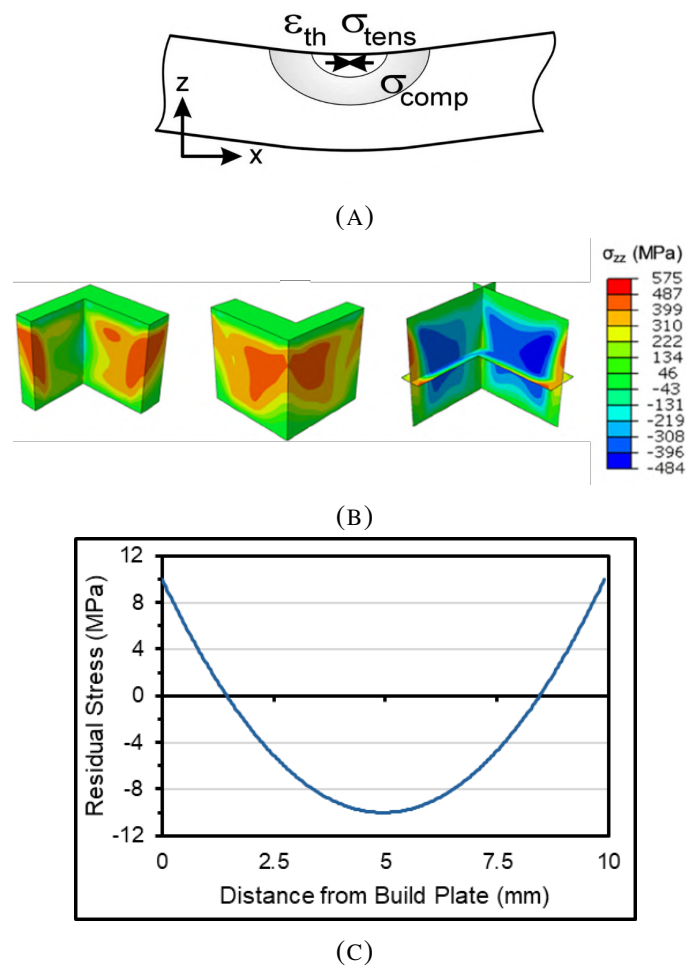


FIGURE 5.17. (a) Stresses and deformation strains on the surface of cooled parts due to the large thermal gradients associated with heating and cooling in PBF (reproduced from [288]), (b) residual stress distribution (σ_{zz}) in stainless steel 316L L-shaped parts that have been removed from the build plate with the build direction vertically upwards (adapted from [289]), (c) example of a typical residual stress distribution over the height of a part, with tensile stresses at top and bottom and compressive stresses in the centre.

stresses experienced in the part are largely tensile, with the build plate experiencing both tensile and compressive stresses [290, 291]. Upon removal from the build plate the distribution of stresses changes, with tensile stresses found near the surfaces of parts, especially at the top surface of parts and near the build plate, and compressive stresses found in the part interior [292, 293]. An example of this in modelled data is shown in Figure 5.17b.

This modelling matches physical results often seen in manufactured parts [294, 295], with a representative example shown in Figure 5.17c. The use of a heated build plate will, however, reduce these residual stresses by reducing thermal gradients [295, 296] with these reductions typically ranging between 40% – 80% of total residual stresses [297] and extreme cases reducing residual stresses to 1 MPa – 5 MPa [298]. Given the standard profile of residual stresses in Figure 5.17c does not match that of the hardness trend in Figure 5.10, it is unlikely that residual stresses play a major role in providing different strength increases across the height of the sample. This hypothesis, however, requires experimental validation.

5.4 The Effect of Part Dimensions and Surface Finish on Tensile Properties

Due to the complex and anisotropic nature of the microstructure of PBF parts, it has been seen in the literature that mechanical anisotropy is common [1, 93]. In part this is due to the preferential growth of grains along the build direction, however, factors such as porosity and surface finish also play a role. These differences are often not considered when manufacturing parts and so must be properly investigated to understand how geometric factors will affect performance. This is especially true regarding specimen size and surface finish, with little consideration beyond generic material properties for how a part will behave under service.

This section will therefore aim to compare tensile specimens prepared under a number of conditions to determine the effects of surface finish and specimen size on AlSi7Mg's tensile properties. These specimens were manufactured in both horizontal and vertical orientations, with a set of small and large samples printed to shape and small samples machined to shape. The dimensions of these specimens are shown below in Figure 5.18

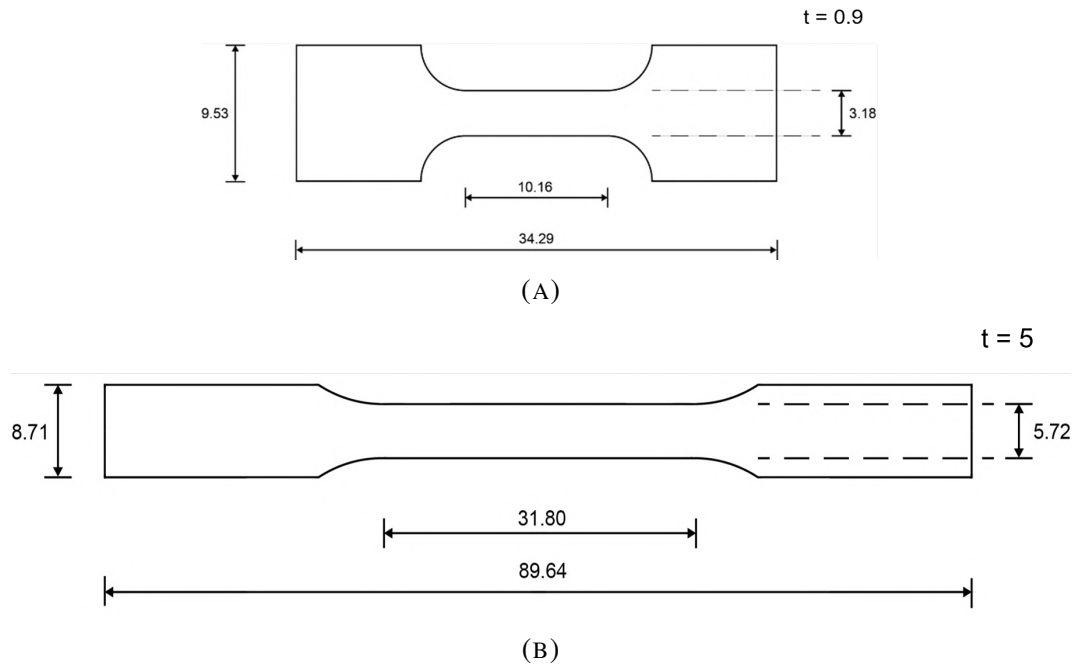


FIGURE 5.18. (a) Small tensile specimen dimensions, (b) large tensile specimen dimensions. All dimensions are in mm.

5.4.1 Yield Strength

The yield strengths of variously manufactured tensile specimens show significant dependence on their manufacturing process. The yield strengths of these specimens are shown in Figure 5.19. It is evident that the yield strength of the small machined specimens is the greatest, with an average strength of 159.7 MPa across both horizontal and vertical orientations. This is compared to the yield strengths of large printed and small printed samples of 134.2 MPa and 122.7 MPa respectively. Part orientation has no effect on the yield strength of parts with the exception of the small machined samples, with the yield strengths of horizontally and vertically oriented specimens being 168.5 MPa and 150.8 MPa respectively.

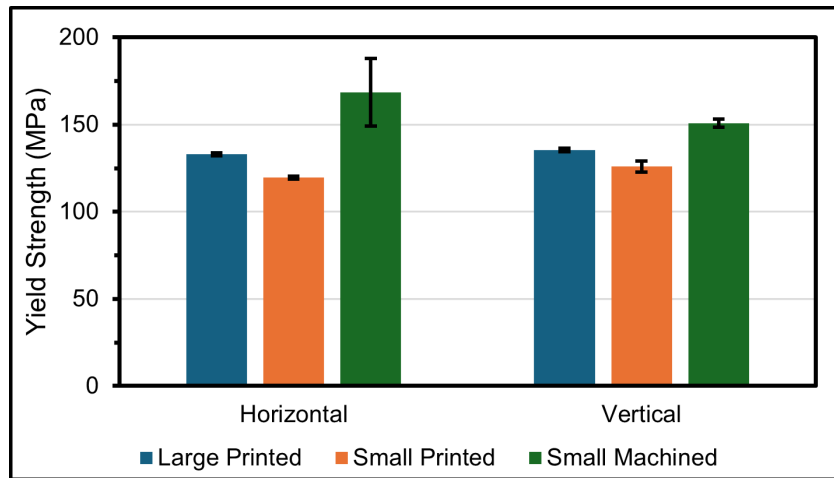


FIGURE 5.19. Yield strengths of various tensile specimens.

5.4.2 Tensile Strength

The tensile strengths of variously manufactured specimens follow a similar trend to that of the yield strength and are displayed in Figure 5.20. Again, the strength of the small machined samples is greater than that of the other manufacturing processes, with a tensile strength of 234.9 MPa compared to 199.5 MPa for the small printed samples and 220.2 MPa for the large printed samples. These differences in strength were present in both vertically and horizontally manufactured specimens.

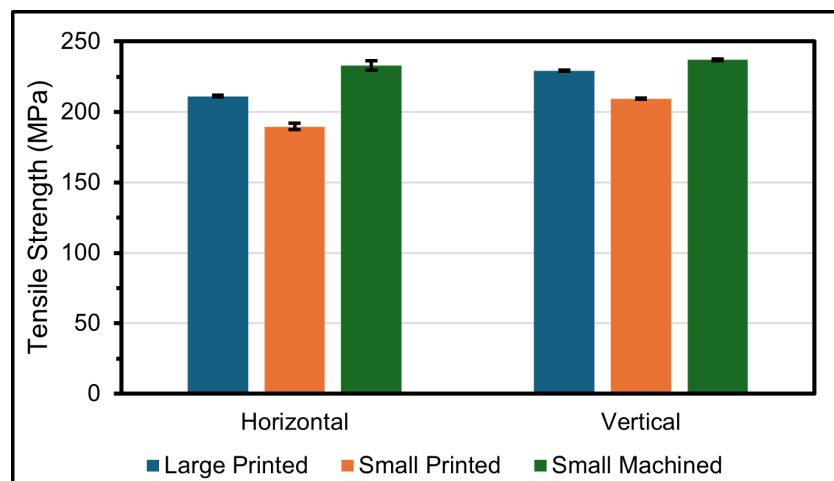


FIGURE 5.20. Tensile strengths of various tensile specimens.

While there are differences in the tensile strength depending on the manufacturing process, there is also a dependence of tensile strength on part orientation for printed samples. While

for the small machined samples the difference was 4 MPa, a difference of only 1.7% that falls within the standard deviation, the differences were more significant for printed samples. For large printed samples vertical specimens were 18.1 MPa stronger than their horizontal counterparts, an increase of 8.2%. A similar increase was found for the small printed samples, with vertical specimens 19.9 MPa stronger than horizontal ones, an increase of 10.0%.

5.4.3 Ductility

The ductility of tensile specimens showed a considerably more varied relationship to their manufacturing processes than other tensile properties. As seen in Figure 5.21, for vertically oriented specimens the relationship between ductility and processing follows a similar trend to that of the yield and tensile strengths. While this same relationship is evident, it is to a much lesser degree given the larger standard deviations in the data as well as the closer proximity of ductility values.

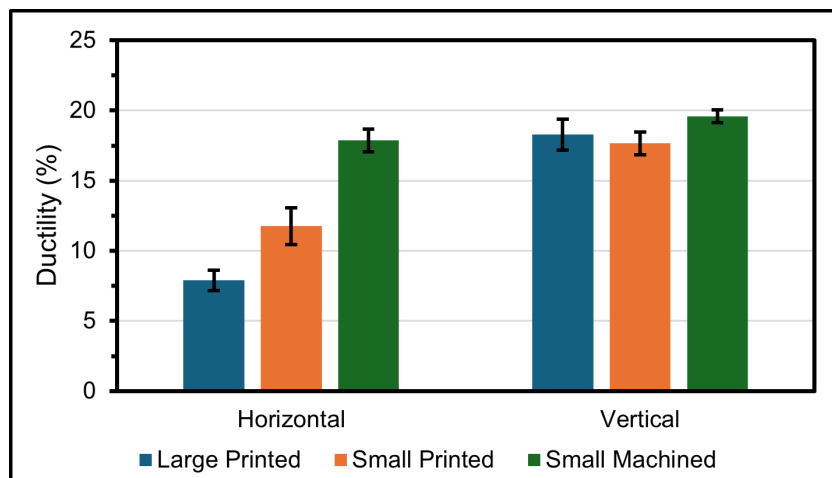


FIGURE 5.21. Ductilities of various tensile specimens.

The ductility of horizontally orientated samples shows a different relationship compared with other tensile properties. Large printed specimens show 9.98 % lower ductility than small machined specimens and there is also a considerable difference of 6.1 % ductility when comparing small printed and small machined samples, showing that a machined surface provides a more significant improvement in ductility than changing sample size.

Unlike other tensile properties, there is a notable difference between ductilities for specimens of consistent manufacturing processes but different orientations. A large printed specimen

shows a significantly more varied ductility when compared to other specimens, with a reduction of 10.4 % ductility when comparing a vertically orientated specimen to a horizontally orientated one. This effect is lessened for small printed samples with a difference of 5.9 % ductility, while small machined samples differ by only 1.73 % ductility.

5.4.4 Fracture Surfaces

Figure 5.22 shows a representative fracture surface for each of the six specimen types tested. These images show that the fracture initiation sites for each specimen differ, with the large, printed, and horizontally oriented specimen in Figure 5.22a showing large indents on the bottom surface of the sample where it was attached by supports to the build plate. As-printed samples in Figures 5.22b, 5.22d, and 5.22e each show significant areas of porosity near the edge of the specimens, while machined specimens in Figures 5.22c and 5.22f show planar fracture surfaces.

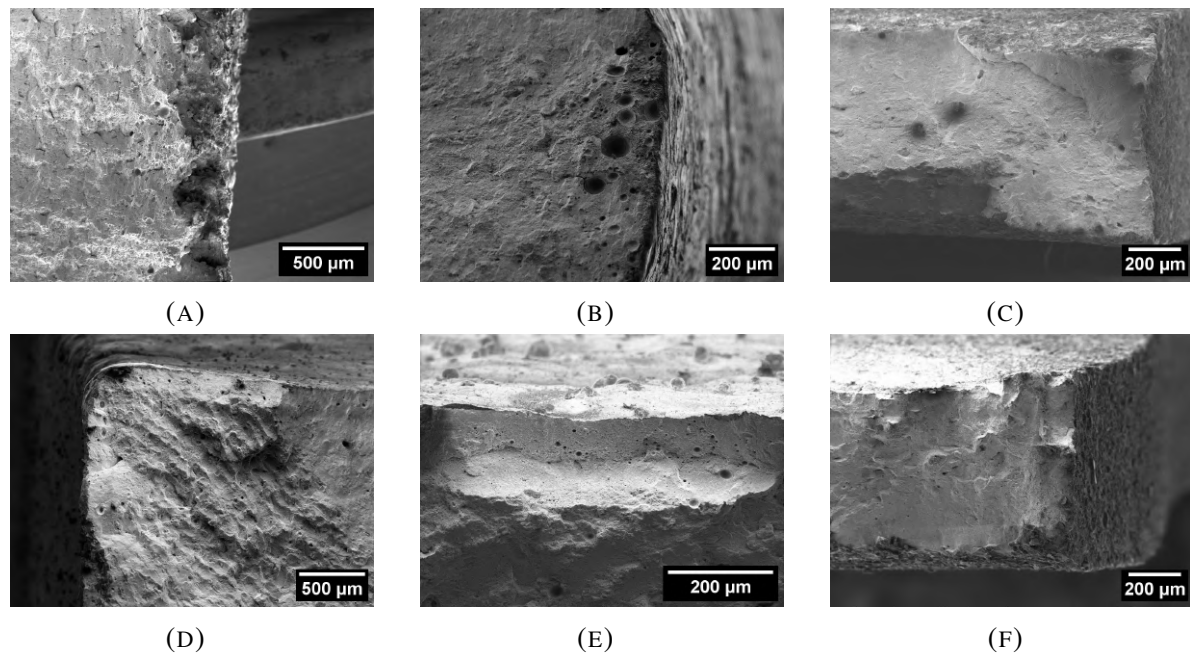


FIGURE 5.22. Representative fracture surfaces showing the failure initiation sites for each type of tensile specimen tested in this study: (a) large, horizontal, printed sample, (b) small, horizontal, printed sample, (c) small, horizontal, machined sample, (d) large, vertical, printed sample, (e) small, vertical, printed sample, (f) small, vertical, machined sample.

While these images show the areas of fracture initiation, Figure 5.23 shows a higher magnification image of a representative fracture surface of these specimens. The areas between pores, edges, and other defects show the dimple morphology typical of stress relieved samples, indicating a relatively ductile failure. This deformation type was consistent across all samples.

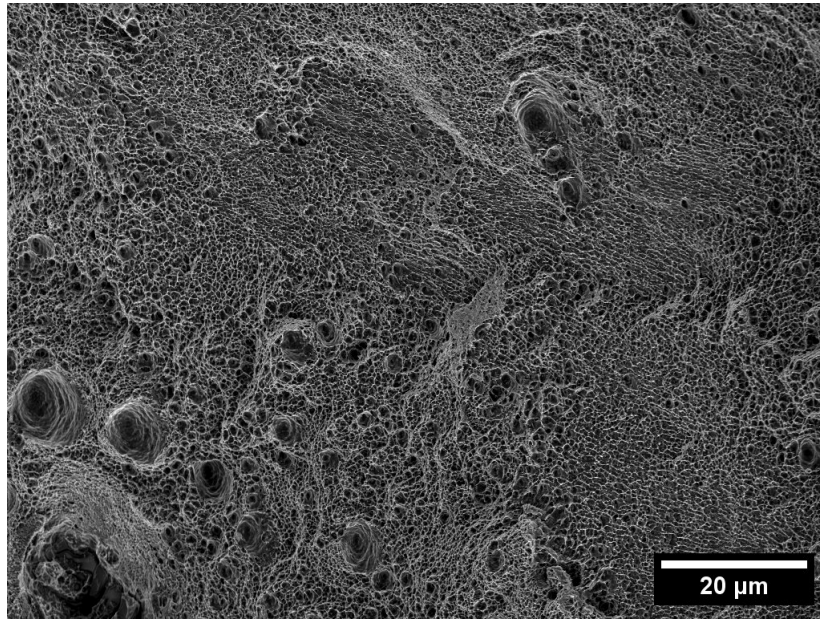


FIGURE 5.23. Representative sample of the internal fracture surface of various tensile specimens.

5.4.5 Surface Roughness

The surface roughnesses of representative samples for each processing condition are shown in Table 5.7. It is clear that the machined sample has a noticeably smoother surface finish which is also more consistent than both of the printed samples. This is as factors affecting surface roughness related to PBF processing, such as layer lines and sintered powders, are removed by machining, with wire EDM producing a more consistent surface finish.

TABLE 5.7. Roughness values for each set of tensile specimens.

Sample	Ra (μm)
Large Printed Sample	5.59 ± 0.99
Small Printed Sample	6.72 ± 0.85
Small Machined Sample	4.36 ± 0.25

5.4.6 Grain Characterisation

To investigate the differences in properties observed between each of the specimens, grain characterisation was carried out to determine if these grains had any effect on properties. Figure 5.24 shows IPF maps taken from the edge and the interior of a printed sample. The grains closer to the centre of the sample reveal the typical microstructure of PBF material, with melt pools clearly characterised by columnar grains with groups of smaller, equiaxed grains surrounding them. This is in contrast to the microstructure at the edge of printed parts with elongated grains present at the very edge, before a transition to elongated grains of a different texture, and then a large region of fine, equiaxed, and randomly orientated grains beginning approximately 200 μm from the edge of the sample. This microstructure is present in the as-printed samples, whereas the machined samples contain only grains similar to that of the interior of the printed specimen.

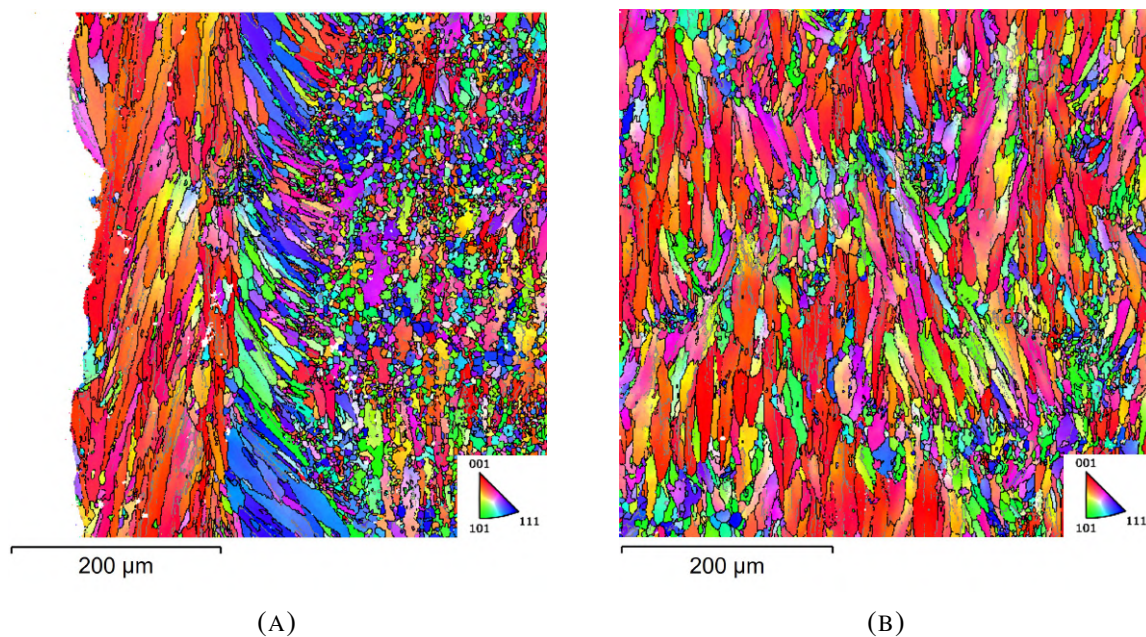


FIGURE 5.24. IPF maps of: (a) edge of a printed part (specimen surface located on the left of the image) (b) interior of a printed part.

5.4.7 Discussion

A comparison of these results suggests that the machining of samples provides for the greatest increase in strength, with the small machined samples having consistently higher yield and

tensile strengths than the as-printed samples regardless of sample orientation. A comparison of the fracture surfaces reveal that this is due to the reduced number of defects, namely voids and poor surface finish due to supports, present in machined samples compared to as-printed ones.

As-printed specimens are weakened by the presence of a ring of porosity that surrounds the edge of these samples, as seen in Figure 5.25. This porosity forms due to the interaction of two different laser parameter sets, those of the bulk and the surface. These parameters are optimised for different conditions, balanced properties and a smooth surface finish respectively, therefore involving different laser powers, speeds, and hatch spacings. The overlap between the areas where these parameters are used, while allowing for full melting and reduced lack of fusion defects, results in a significant amount of spherical gas porosity at their meeting point. This porosity is clearly present in Figures 5.22b, 5.22d, and 5.22e. This porosity appears to be the initiation point for failure, particularly evident in Figure 5.22e, suggesting that its presence has a detrimental effect on part strength. Since these pores exist only at the edge of parts, approximately 100 μm from the surface, these pores are not present in machined samples and so do not result in a reduction in strength for these parts.

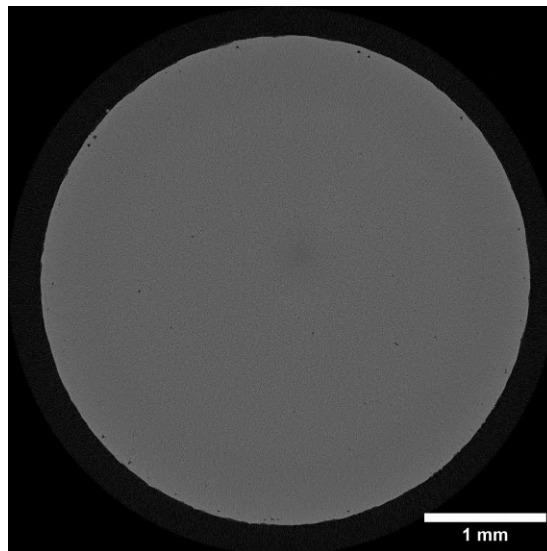


FIGURE 5.25. Cross section of a cylinder of AlSi7Mg produced by PBF showing a ring of porosity near the surface of the part, measured by μ -CT.

Figure 5.22a reveals that horizontally orientated, as-printed samples begin to fail due to the presence of residual support structures on their bottom surface, which serve as stress

concentrators due to their sharp borders on the tensile samples. The remelting of these supports in the first few layers of the production of the tensile samples also has the potential to alter the microstructure and increase the prevalence of voids at these locations, as is the case at the border of surface laser parameters and bulk laser parameters. These stress concentrators then serve as the crack initiation sites leading to early failure, thereby reducing the strength of these samples. These effects are enhanced by the relatively poor surface finish of the as-printed surface when compared to machined surfaces, due to the presence of sintered powder and layer lines. Given that these supports are present on the gauge section of only the horizontally orientated specimens, this can account for the slightly higher strengths shown in vertical tensile samples when compared to horizontal ones.

The main effect of printed, as opposed to machined, samples is a significant loss of ductility. Figure 5.21 shows that for vertically orientated specimens the ductility follows a trend similar to other tensile properties, with machined samples having the greatest ductility of 19.6 %, followed by the large printed specimens with 18.3 % ductility and small printed specimens at 17.7 % ductility. This stands in deep contrast with the horizontal specimens where printed specimens experience a drastic decrease in ductility, especially evident in the large specimens. Figure 5.26 shows a representative stress strain curve of each of the horizontal test cases, indicating that the behaviour of each sample under tensile loading is different. While the small machined samples were able to continue to deform once the tensile strength had been reached, this was not the case for the printed samples. Both printed sample types exhibited signs of early failure, with large printed samples only deforming by up to 2% once their tensile strength was reached, whereas small machined samples deformed by a further 10% past their tensile strength. Given that vertically oriented specimens exhibited more consistent behaviour in all cases, that of continued deformation past the maximum tensile stress, it can be concluded that horizontal, printed specimens fail early. According to the fracture surfaces in Figure 5.22a and 5.22b this is due to residual support structures and internal porosity. Given fracture of the large tensile specimens clearly initiated from these residual supports, their presence can be seen to have a significantly greater effect on a component's reduction in ductility than porosity.

While Figure 5.26 shows the difference in elongation for each sample, indicative of the early failure of printed specimens, it also shows that there is a difference in strain hardening

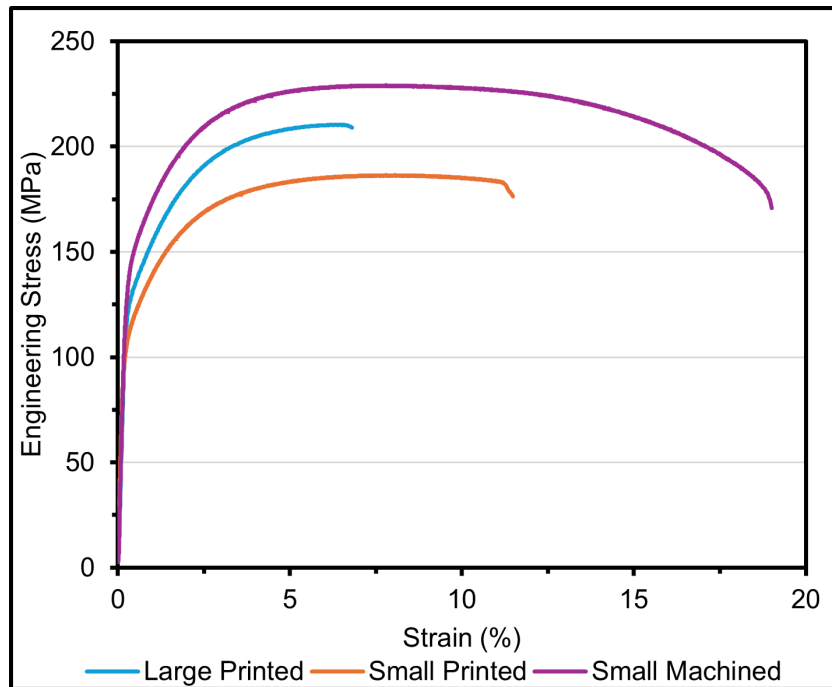


FIGURE 5.26. Comparison of representative stress-strain curves of horizontally oriented tensile samples.

according to the surface finish of the samples. For all vertically oriented samples the increase in stress between yield strength and tensile strength ranged from 57% to 69%. This difference is instead most significant in the horizontally oriented specimens which increased in strength by 59% for all printed samples, while the increase for machined samples was only 38%. These rates are compared in Table 5.8. Given the internal microstructure of all samples are the same, represented by the IPF maps in Figure 5.24b, this significantly different rate of strain hardening between printed and machined samples can be attributed to the microstructure near the surfaces of samples. The grains at the surface of the printed samples are considerably smaller than those in the centre, with a large region of fine, equiaxed, and randomly orientated grains present 200 μm from the surface, as shown in Figure 5.24a. These fine grains present a large number of grain boundaries inhibiting dislocation motion, preventing the onset of plastic deformation to a greater degree than the larger grains in the interior of the samples. The lack of this fine region of grains in the machined samples reduces the amount of strain hardening they experience.

While the rate of strain hardening in the vertical direction is more consistent than in the horizontal direction, there is also greater strain hardening in the vertical orientation in every

TABLE 5.8. Comparison of strain hardening rates for tensile samples of different orientations and processing histories.

(A)		
Large Printed		
	Difference between Tensile and Yield Strengths (MPa)	Ratio between Yield and Tensile strengths
Horizontal	78.3	0.59
Vertical	93.8	0.69
(B)		
Small Printed		
	Difference between Tensile and Yield Strengths (MPa)	Ratio between Yield and Tensile strengths
Horizontal	70.1	0.59
Vertical	83.5	0.66
(C)		
Small Machined		
	Difference between Tensile and Yield Strengths (MPa)	Ratio between Yield and Tensile strengths
Horizontal	64.4	0.38
Vertical	86.1	0.57

case tested. This is likely due to the melt pool structure and the corresponding zones within these melt pools that relate to silicon distribution, as shown in Figure 5.11. Some regions of these melt pools are remelted upon subsequent laser passes, while others are heat affected. In these regions the fine cell structures begin to reform, with larger precipitates of silicon forming rather than the more consistent cell wall boundaries found in the melt pool interior. These regions of larger silicon precipitates can be seen as the lighter regions forming the boundaries of the melt pools. These regions of larger silicon precipitates form weaker zones

of PBF material, since the larger silicon precipitates are less able to hinder dislocation motion than their more continuous counterparts in the melt pool interior. Due to the alignment of the melt pools with the axis of loading in vertically orientated samples, dislocations must pass through both the melt pool interiors and heat affected zones to properly deform, meaning both coarsened silicon precipitates and fine cell walls are able to hinder their motion. This is in contrast to horizontally oriented specimens where dislocations are more readily able to pass through only these heat affected zones which continuously connect horizontally through the sample, moving past larger precipitates that are less able to hinder their motion. This difference in the hindrance of dislocation motion means that vertically orientated specimens are able to strain harden at a greater rate than their horizontally orientated counterparts.

5.5 Conclusion

These results show that, for PBF AlSi7Mg, properties are in some cases reliant on the placement of parts within the build volume. This has been tested according to location on the build plate, distance from the build plate, and the dimensions and surface finish of parts.

The most consistent results appear to relate to the location of parts on the build plate. There is minimal difference between parts taken from nine locations on the build plate, with the yield and tensile strengths, density, surface roughness, and microstructure showing excellent consistency. While the ductility and grain texture, as well as the surface roughness when comparing the top and the bottom of the build plate, show some inconsistencies, these are minimal and make no significant impact on the behaviour of parts.

The properties according to a part's distance from the build plate show much more variation. Parts near the build plate were seen to have higher hardnesses than those at the top of the build, resulting from slight changes in microstructure due to different cooling rates during melting, in-situ ageing, and potentially varying residual stresses. This potentially presents problems for large parts where consistent properties are expected and required. A stress relief treatment was seen to eliminate this issue, however, it also led to a dramatic loss of strength.

It is also clear that the dimensions and surface finish of a part will strongly influence its strength, with large and small, machined and as-printed, and vertically and horizontally

orientated specimens showing significantly different tensile properties. This is most evident in horizontally oriented as-printed samples, where the significant surface roughness present from residual support structures greatly reduces tensile properties, especially ductility. This indicates that the orientation of a part and level of supports used in production need to be carefully considered, otherwise machining of the part may be required. This, however, may not always be a viable option to the geometric complexity of many parts manufactured using PBF.

This then raises the question of how more consistent properties can be attained in PBF produced parts. The simplest method is likely the application of a heat treatment, especially convenient given the equalising affect this has on properties as seen in samples taken from different distances from the build plate, and the existing prevalence of stress relieving treatments to prevent warping when removing parts from the build plate. Stress relieving, however, results in a significant reduction in strength, meaning further heat treatments would be beneficial in regaining some of this lost strength.

Optimising the Heat Treatment of Additively Manufactured AlSi7Mg

Considerable research has been focused on improving the properties of additively manufactured material, including AlSi7Mg, by manipulating in-situ process parameters such as laser power, build plate heating temperature, and powder properties. These have proven successful in a number of ways, resulting in the creation of fully dense parts with unique microstructures and mechanical properties, with ever maturing developments continuing to grow the field of knowledge.

The alternative method to manipulating in-situ parameters for property optimisation is post processing treatments, one of the most common of these being heat treatments. Aluminium age hardening alloys are traditionally treated by direct ageing and T6 heat treatments which take advantage of the precipitation hardening behaviour of these alloys. While the use of these heat treatments is well established in the general literature on these alloys, the unique microstructures and compositions of alloys for additively manufactured materials mean that a direct application of parameters developed for these alloys manufactured under other, more traditional methods may not produce the same results.

As such, this chapter investigates the ageing behaviour of additively manufactured AlSi7Mg, with an especial focus on the effects of solution treatment temperatures prior to ageing. This understanding allows for the proper tailoring of heat treatments to this alloy, improving its mechanical performance.

This chapter contains research that is under review for publication as the article “Effect of Solution Treatment Temperature on the Ageing Behaviour and Mechanical Properties of AlSi7Mg Produced by Laser Powder Bed Fusion”. The paper is authored by J.H. Warner, C.V. Funch, and K. Sisco under the supervision of G. Proust and S.P. Ringer.

6.1 Introduction

Additive manufacturing (AM) allows for the production of geometrically complex parts by the sequential deposition of material in a layer-by-layer manner, with these parts finding applications in a range of industries. Laser powder bed fusion (PBF-LB) is an attractive additive manufacturing technique for the production of metallic components. Aluminium alloys are some of the most popular materials for this technology due to their inherent properties of high specific strength, low density and relatively low cost [35, 93], with aluminium-silicon alloys being especially popular as their good castability makes them suitable for PBF-LB [299]. One such alloy that is becoming increasingly popular is AlSi7Mg due to its good processability, stemming from its narrow solidification range and subsequent limited hot cracking, as well as its heat treatable properties [111].

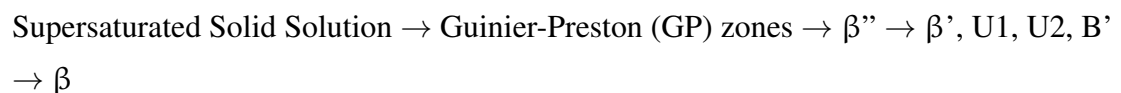
This combination of alloy and manufacturing technique results in a unique microstructure with grain elongation due to grain growth in the build direction, which is the direction of the largest heat flow. Within these grains is a fine cellular network of Si surrounding an Al matrix, formed by the ejection of Si from the solidifying Al due to the rapid solidification rates experienced in PBF-LB [1, 124]. Compared to heat treated states, the as-built (AB) condition has relatively high tensile strengths but low ductility, amplified by the high residual stresses inherent to the PBF-LB process [20, 283], with these alloys being seen to reach tensile strengths of up to 430 MPa and yield strengths of up to 290 MPa [140, 141]. Mechanical anisotropy is also a common issue regularly observed in as-built AlSi7Mg produced by PBF-LB due to the directional cooling of the process and resulting anisotropic microstructure, which often requires further treatment to mitigate [39].

This has resulted in considerable focus on improving the properties of AlSi7Mg by heat treatment. This is often achieved by T6 treatments which involve a solution treatment to promote the homogenisation of the microstructure and dispersion of alloying elements through the matrix, followed by an artificial ageing step which aims to strengthen the material by the growth of precipitates from these now solute elements. These methods are adapted from casting heat treatment processes [20].

T6 treatments have been seen to have a mixed effect on the ductility of Al-Si alloys due typically to the solution treatment step, with some studies observing an increase in ductility

[38, 111, 283, 300], some a decrease [140, 142, 148], others no change [141, 301], and still others observing different effects according to part orientation [20, 39]. A variety of reasons have been given for these changes. The most common of these is the coarsening of the Si network where the larger Si particles require lower stress levels to crack, resulting in early fracture and a corresponding reduction in ductility [148]. Solution treatment also promotes the growth of β -Al₅FeSi and π -Al₈FeMg₃Si₆ intermetallic phases which tend to be highly brittle, with their elongated shapes often forming stress concentrators that inhibit the ductility of the material as a whole [38, 145]. The increased porosity of solution treated parts, caused by the expansion of existing gas porosity within the softened aluminium matrix during solutionising, has also been attributed to a reduction in ductility [142]. Contrastingly, the reduction of elements in solid solution [39], lower residual stresses [283], and the dissolution of fine Si precipitates [283] have all been attributed to an increase in ductility after a solution treatment. Differences in measured ductility may also, however, be affected by the different tensile specimen sizes used by authors within the literature. While changes to ductility are inconsistent after a solution treatment, these changes in ductility are typically accompanied by a loss in strength.

Some of the strength lost by this solution treatment step is then regained by artificial ageing, with the growth of nanoscale precipitates improving the hardness and ultimate tensile strength [143, 301]. This occurs primarily through the Mg₂Si precipitation sequence, which is as follows [302]:



These precipitates start as coherent needles (β'' - Mg₅Si₆ or Mg₅Al₂Si₄), becoming semi-coherent rods or lathes (β' - Al₃Mg₉Si₇), and finally incoherent platelets (β - Mg₂Si) [111, 141]. The strengthening of AlSi7Mg has been variously attributed to the presence of each of these phases, however the degree of precipitate growth is often unclear [20, 39, 142, 300, 301]. Strengthening occurs by Orowan mechanisms, with the growth of these small precipitates throughout the matrix hindering dislocation motion [303]. These small precipitates are considered the main strengthening mechanism for Al-Si-Mg systems, and so have been the focus of numerous ageing studies.

The variation in solution treatment effectiveness and degree of precipitation of the strengthening phases results in a range of strength properties being observed due to T6 treatments. For T6 treated samples in comparison to the as-built state, with a shortened solution treatment time Vanzetti et al. [300] observed a reduction in both yield and tensile strengths from 254 MPa to 218 MPa and 382 MPa to 270 MPa, respectively, with ageing recovering most of the strength loss associated with the solution treatment. This reduction in strength is common for many T6 treatments [38, 140, 141]. While these findings reflect those traditionally thought of being the effect of a T6 treatment, they are not universal. De Menezes et al. [39] observe a different response to T6 treatment according to part orientation, with horizontally oriented specimens reducing in yield strength from 257 MPa to 236 MPa, while vertically oriented specimens increase in yield strength from 216 MPa to 245 MPa, with Pereira et al. [20] also observing anisotropic properties.

While these processes are similar and produce changes in mechanical properties that are relatively consistent, the times and temperatures required for peak strengthening differ, especially regarding solution treatments. These temperatures lie in the range of 500 °C-545 °C, with 505 °C [141], 525 °C [38], 535 °C [20], 540 °C [39], and 543 °C [142] each being tested for a range of durations. The range in temperatures used in the literature suggests that the microstructural evolution of AlSi7Mg due to a solution treatment is not properly understood. As the solution treatment temperature has the potential to influence ageing behaviour by altering the precipitation of the Mg_2Si phase, this temperature should be properly optimised to maximise the effectiveness of artificial ageing, with a microstructural understanding being crucial to this.

Therefore, this work aims to develop an understanding of the effect of solution temperature on the strengthening mechanisms at work in a heat treated AlSi7Mg alloy. To determine this, samples were artificially aged in three conditions: as-built directly after removal from the build plate, after a stress relief treatment, and after solution treatment at a variety of temperatures. Microstructural investigations were then carried out to develop a correlation between the mechanical properties observed and component microstructures.

6.2 Methods

Gas atomised powders from Concept Laser were used for the production of samples analysed in this work, of the composition shown in Table 6.1. The particle size distribution was 25-58 μm , as measured by Concept Laser.

TABLE 6.1. Chemistry of AlSi7Mg powder and parts used in this work, as measured by ICP-AES. Measurements are in weight percent.

	Al	Si	Mg	Fe	Ti	O	Cu	Mn	Zn	Be
Powder	Bal.	6.77	0.42	0.06	0.04	0.015	<0.01	<0.01	0.01	<0.001
Part	Bal.	6.77	0.39	0.06	0.04	0.011	<0.01	<0.01	<0.01	<0.001

Specimens for this research were manufactured using a Concept Laser M2 Series 4 with two 400 W lasers enabled to work in tandem, however, each part was manufactured using only one laser to avoid interaction zones between the lasers. Parameters used include a laser power of 370 W, scan speed of 1600 mm/s, layer thickness of 30 μm and an 80 °C build plate preheating. The scan pattern was rotated 67° between layers, and parts were removed from the build plate using wire electron discharge machining before heat treatment.

Tensile specimens and miniature Charpy specimens were manufactured in the vertical direction, according to dimensions in ASTM E8/E8M-24 and ASTM E2248-18, respectively. Tensile samples were dimensioned according to Figure 19 of ASTM E8/E8M-24, the standard flat tension specimen for powder metallurgy products. Also produced were witness cubes of dimensions 13 x 13 x 13 mm³ for microstructural analysis and the development of ageing curves.

Heat treatments took place using a variety of techniques. Samples designated as-built underwent no heat treatment after removal from the build plate, while stress relieved (SR) samples were heated in a Nabertherm LH 60/14 chamber furnace at 300 °C for two hours in an argon atmosphere. Solution treatment was completed in a salt bath for six hours at 400 °C, 450 °C, 475 °C, 500 °C, 510 °C, 520 °C, 530 °C and 540 °C, followed by water quenching. Ageing was conducted in an oil bath for up to 8 hours at 150 °C, 160 °C, 170 °C, 180 °C and 190 °C, with hardness testing taking place every 30 minutes.

Charpy testing was completed using a CEAST 9050 pendulum impact system, and tests were carried out according to ASTM E2248-18. Tension testing was carried out using an Instron 5567 universal testing machine, with a strain rate of 0.1 mm/mm/min following ASTM E8/E8M-16. Three samples were tested for each case to improve statistical validity. Vickers hardness testing was conducted using a Duramin-40 hardness tester using a load of 1 kg and a dwell time of 15 seconds. Indentations were made on mechanically polished samples with a final step of 2000 grit silicon carbide paper, with a minimum of ten indentations taken per sample. These tests were carried out according to ASTM E92-17.

Electron backscattered diffraction (EBSD) was conducted in a Zeiss Ultra, with an accelerating voltage of 20 kV, aperture of 60 μm and a step size of 1 μm . Samples were polished using diamond suspension with a final step of oxide polishing with colloidal silica. Energy dispersive spectroscopy (EDS) was conducted on the Zeiss Ultra using an Oxford Instruments EDS detector with an acceleration voltage of 20 kV, a 120 μm aperture and an 8.5 mm working distance.

Transmission electron microscopy (TEM) was conducted on a JEOL 2200FS equipped with a field emission gun (FEG) and an Oxford Instruments EDS detector. Scanning transmission electron microscopy (STEM) was conducted with an acceleration voltage of 200 kV. Sample preparation for TEM was done for the AB and SR specimens using twinjet electropolishing using a Struers TenuPol-5 in an electrolyte consisting of 10 % perchloric acid in methanol, while solution treated specimens were prepared by a combination of dimple grinding on a Fischione Instruments Model 200 dimpling grinder and precision ion polishing on a Gatan PIPS-II at -20 °C to avoid overheating of the material.

Atom probe tomography (APT) was performed using an Inviso 6000, with a dual laser using a 200 pJ laser, current 200 KHz pulse rate, and 2 % detection rate to investigate the composition of the matrix and precipitates. The APT tips were bulk electropolished in a 25 % perchloric acid at 20 Kv. A Thermofisher Helios Plasma Focused Ion Beam (PFIB) was used to mill the electropolished tips to a fine point for data collection.

ThermoCalc using the TCAL8 database was employed to model thermodynamic equilibriums for this system using the CALPHAD (Calculation of phase diagrams) methodology to understand equilibrium phase stability and compositions. The determination of equilibrium

FCC Al concentrations were done at the measured bulk composition of the AM part, with all default phases included for the Al-Si-Mg-Fe-Ti system.

6.3 Results

6.3.1 Mechanical Performance

A range of ageing treatments were carried out on samples of different initial states. Figure 6.1 shows the effects of utilising different ageing temperatures on samples that underwent no prior heat treatment, i.e. a T5 treatment. For lower ageing treatment temperatures there is no significant change in hardness, with ageing at 150 °C and 160 °C producing no discernible change. However, higher ageing temperatures led to a continuous decrease in strength, with this effect being more pronounced the higher the temperature and the longer the ageing time. The continuity of this trend regarding both temperature and time suggests that this effect will also be visible at 150 °C and 160 °C when aged for longer times. The consistency of strength reduction evident in the ageing behaviour of the alloy suggests that the as-built condition is already overaged, due to the use of a heated build plate in the manufacturing of these specimens. Continued artificial ageing is worsening this effect.

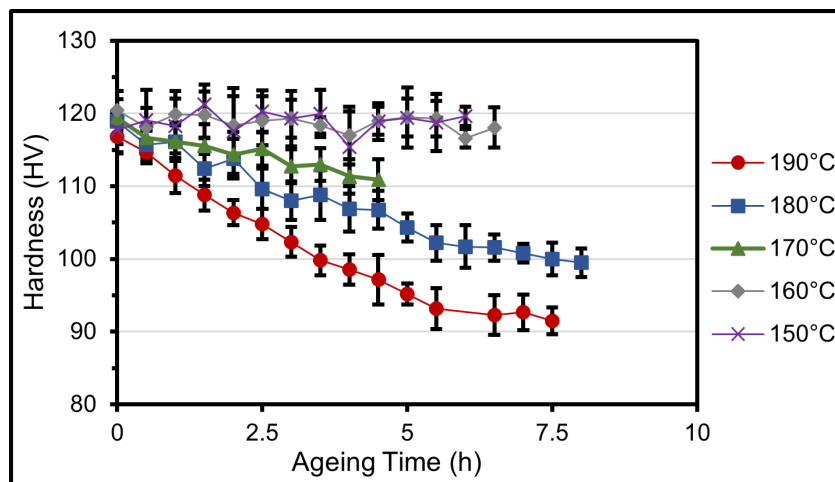


FIGURE 6.1. Ageing curves of samples that underwent no prior heat treatment.

Given the prevalence of stress relieving treatments to eliminate the warping of parts, ageing was conducted on samples that underwent stress relief, as shown in Figure 6.2. The strength

of these samples is considerably lower than those in the as-built state, with the initial stress relief reducing the hardness from ~ 120 HV to ~ 70 HV, with ageing treatments then having no effect on the strength of the alloy. The fact that ageing has no impact on strength indicates that the alloy is already overaged, with any further changes to the microstructure due to ageing having no influence on the mechanical properties.

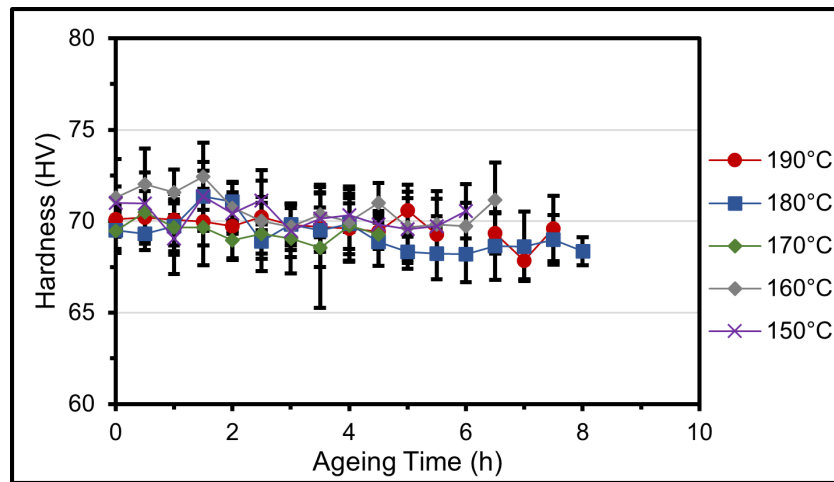


FIGURE 6.2. Ageing curves of samples that underwent a prior stress relief heat treatment.

Samples that underwent solution treatment for six hours at a variety of temperatures were subsequently aged at 160°C for varying times, with the results of these ageing treatments shown in Figure 6.3. The solution treatment significantly decreases the strength of these specimens in comparison to the as-built state, with a reduction in strength from ~ 120 HV to 60-70 HV, in a behaviour similar to the stress relieved samples. There is also a clear distinction in behaviour between samples aged above and below 500°C , with samples below this threshold displaying both poorer initial strengths and minimal ageing behaviour. This implies that the elemental dispersion and precipitate growth at different solution temperatures are not consistent, resulting in different strengthening mechanisms and changing the effectiveness of ageing. The poor, and almost non-existent, age hardenability of these low temperature solution treated samples indicates suboptimal growth of strengthening precipitates. Solution treatment temperatures over 500°C display similar ageing behaviour to each other, with an increase in hardness until a peak is reached between four and seven hours. Given the success of ageing samples solution treated at 530°C , this temperature was selected for further ageing analysis, with a solution time of six hours chosen.

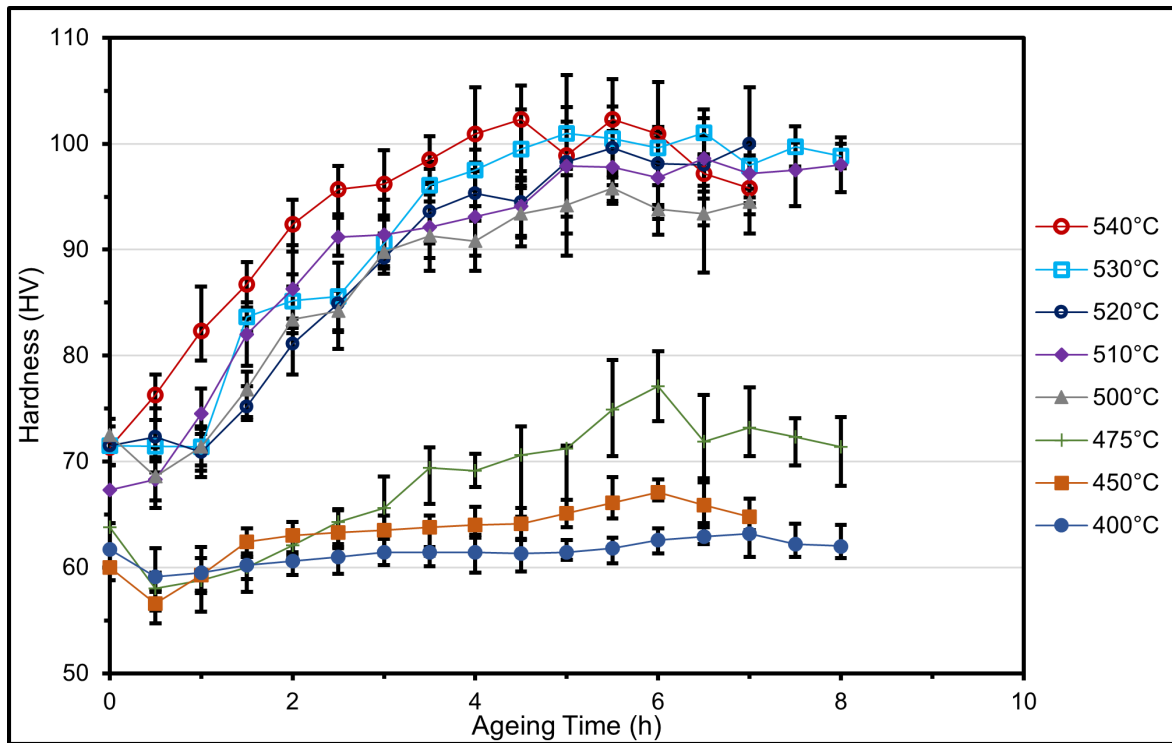


FIGURE 6.3. Ageing curves of samples that underwent solution treatments at a variety of temperatures.

Figure 6.4 shows that ageing was successfully carried out on samples that underwent solution treatment at 530 °C for six hours, with each of the ageing temperatures tested demonstrating a clear age hardening behaviour. The exact nature of this age hardening, however, differs according to the ageing temperature used. Higher temperatures reached their peak hardness quicker than lower temperature ageing; however, these peaks were lower. This is demonstrated most noticeably by ageing at 190 °C where the peak hardness of 90.9 HV was reached after only one hour of ageing, in comparison to ageing at 150 °C where after 7.5 hours a peak hardness of 99.7 HV was achieved. These varied strengthening patterns indicate different precipitate growth behaviours. This is as higher temperatures promote faster diffusion, therefore growing larger precipitates at a later stage in the Mg_2Si precipitation sequence at a faster rate which results in earlier over ageing.

Tensile testing was conducted to determine the properties of as-built, stress relieved, and T6 treated samples, the results of which are shown in Figure 6.5a - 6.5c, with sample dimensions shown in Figure 6.5e. The parameters for the T6 treatment were chosen based on the previous ageing curves, with a solution treatment at 530 °C for six hours and ageing at 160 °C for five

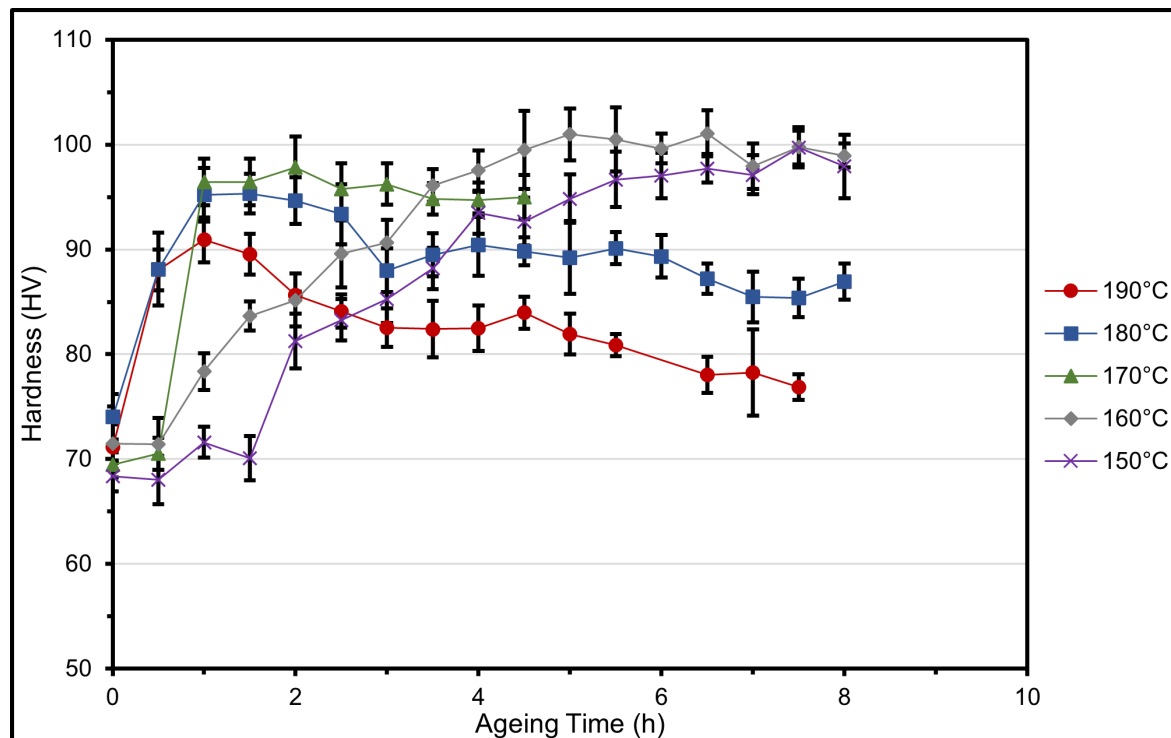


FIGURE 6.4. Ageing curves of samples that underwent a solution treatment of 530°C for six hours.

hours selected. This was based on the high peak hardness reached in a reasonable time frame. These results show that the yield strength of AlSi7Mg can be significantly improved by a T6 treatment, with an increase from 199.5 MPa to 233.3 MPa when compared to the as-built state. This behaviour is different, however, for the tensile strength, with T6 treated samples seeing a reduction in strength from the as-built state from 364.9 MPa to 282.5 MPa. This indicates that there are different strengthening mechanisms at work in these specimens each with different effects on the yield and tensile strengths. An increase in ductility is also observed for the T6 samples increasing from 11.65 % in the as-built state to 15.44 %. Stress relieved samples behaved as expected and in a manner that reflected their hardness, with this treatment leading to a significant reduction in both yield and tensile strengths and a corresponding increase in ductility when compared to the as-built state.

Charpy testing was conducted to determine the energy absorption of each of the heat treated states, the results of which are shown in Figure 6.5d. This shows that any heat treatment results in a reduction in absorbed energy with this being especially true for T6 treated specimens, as T6 treated specimens absorbed only 20 % of the energy that their as-built counterparts did.

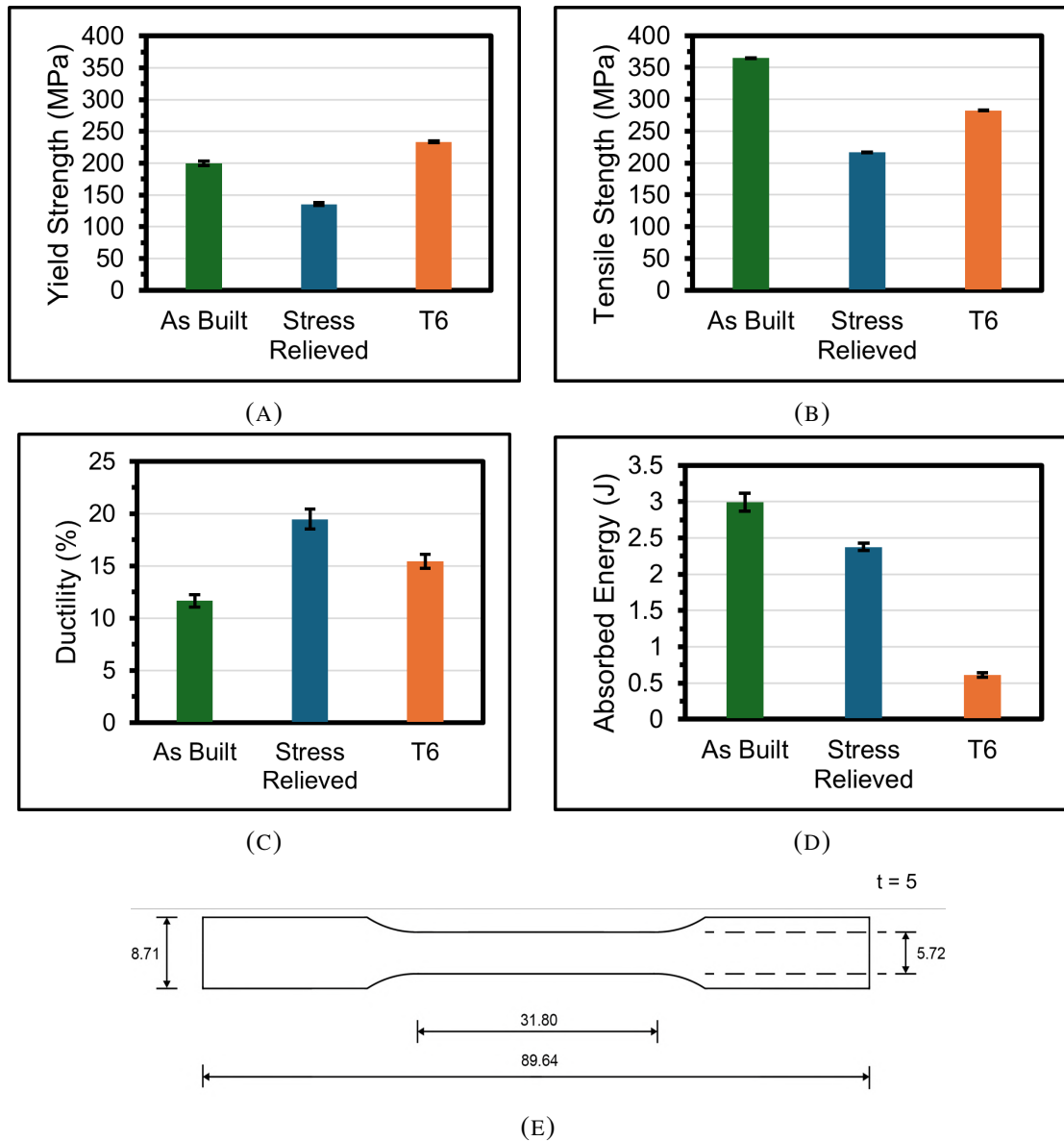


FIGURE 6.5. Mechanical properties of as-built, stress relieved, and T6 treated samples: (a) yield strength, (b) tensile strength, (c) ductility, (d) absorbed energy. The dimensions of tensile samples used in these tests are shown in (e).

6.3.2 Microstructure

The microstructure of the material as a function of the applied heat treatment is illustrated using EBSD in Figure 6.6. At this level of magnification, no differences can be detected between the different heat treatment conditions. In the as-built condition, the microstructure consisted of elongated grains aligned approximately along the build direction i.e. the direction

of the largest heat flux. The crystallographic textures of each condition can be found in Figure 6.7. The as-built condition can be seen to have the $[100]$ direction aligned with the build direction. These characteristics are overall unchanged between each heat treatment condition. No significant grain growth of these elongated grains was observed, even at the highest solution treatment temperature (Figure 6.6e and 6.6f). Similarly, the grains remain elongated and do not experience recrystallisation, which is also reflected in the unchanged crystallographic texture in Figure 6.7.

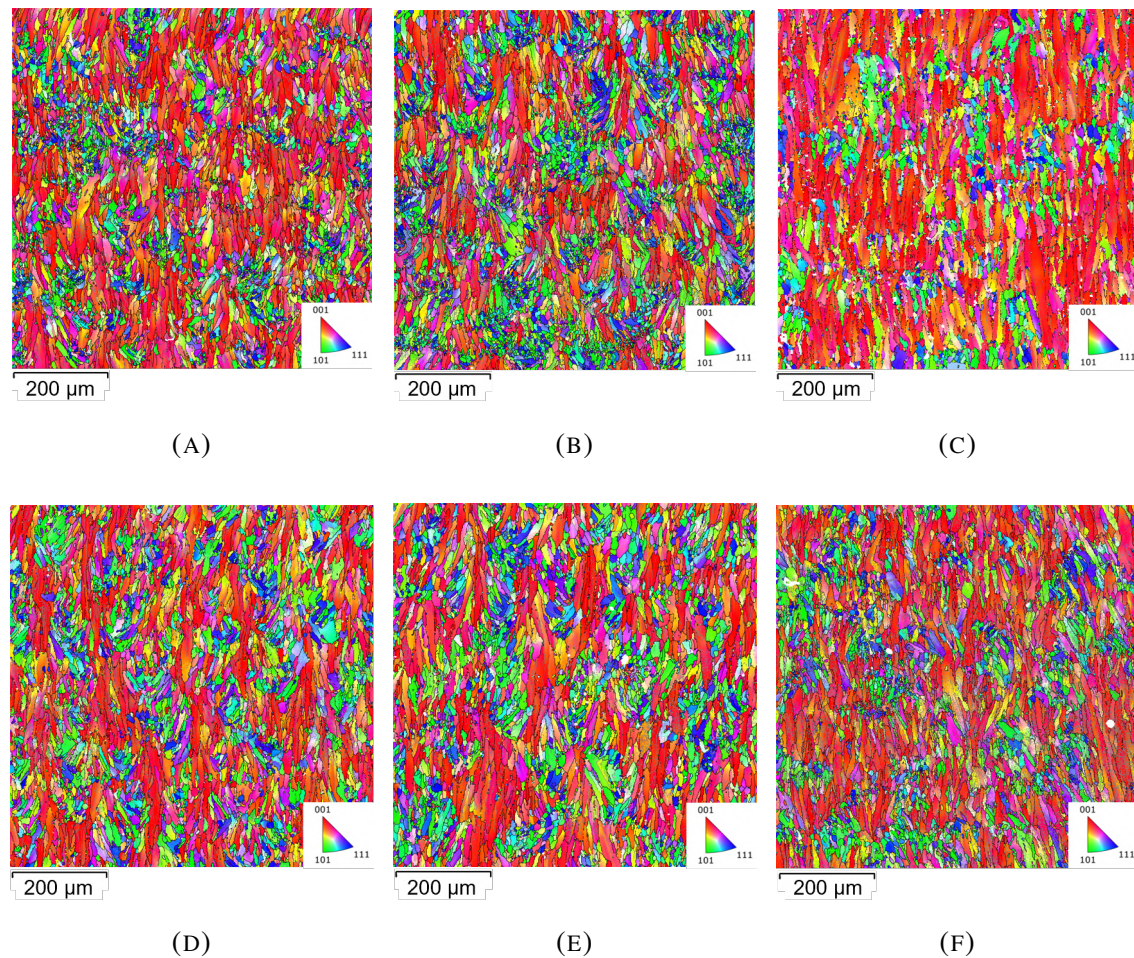


FIGURE 6.6. Inverse Pole Figure (IPF) maps of samples at different stages of heat treatment: (a) as-built, (b) stress relieved, (c) solution treated 400 °C, (d) solution treated 500 °C, (e) solution treated 530 °C, (f) solution treated 530 °C and aged. Build direction is from bottom to top.

Compositional maps for the different solution treatment temperatures using SEM-EDS can be found in Figure 6.8. Only slight segregations can be observed at this length scale for the as-built and stress relieved conditions resembling a cellular network. However, using

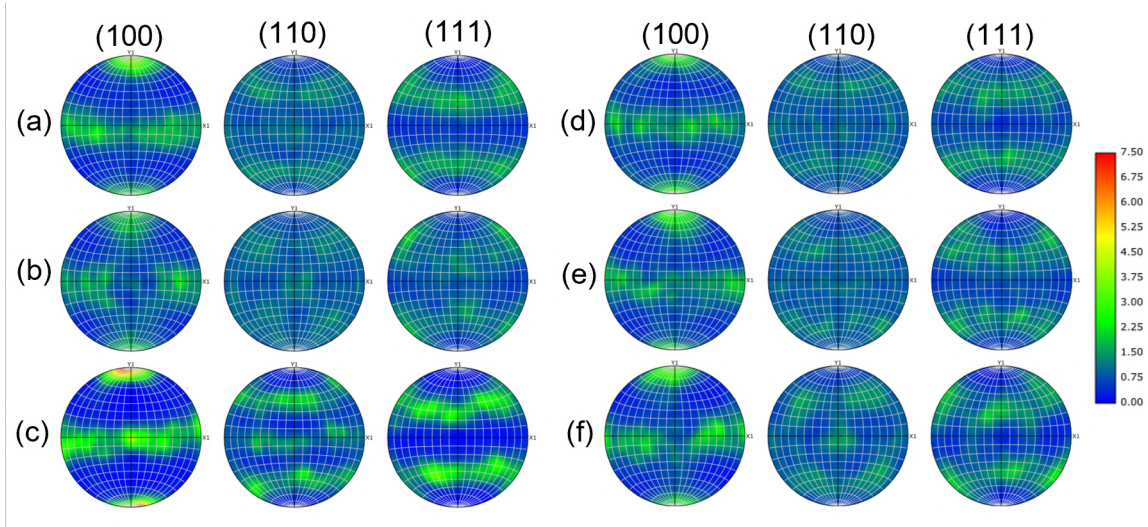
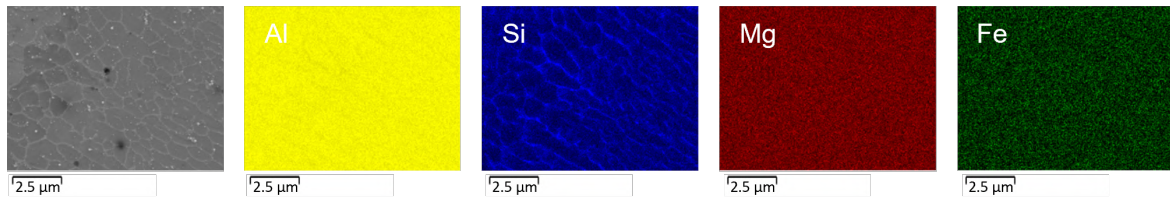


FIGURE 6.7. Pole figure plots of samples at different stages of heat treatment: (a) as-built, (b) stress relieved, (c) solution treated 400 °C, (d) solution treated 500 °C, (e) solution treated 530 °C, (f) solution treated 530 °C and aged.

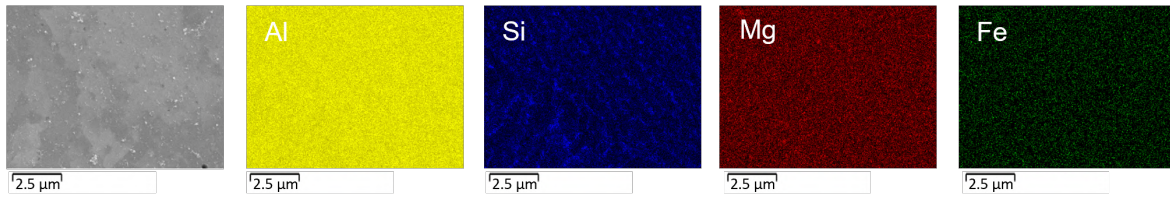
TEM-EDS as seen in Figure 6.9, a fine continuous cell network is formed in the as-built condition with segregations of Si and Mg to the cell walls.

In the stress relieved condition, these uniform continuous cell walls are redistributed into more distinct precipitates. These precipitates are still located at the boundaries of the original cell network and are characterised by a segregation of primarily Si to these precipitates. Some of the precipitates also experience simultaneous segregation of Mg and Fe. The solution treatments are not only conducted at higher temperatures but also for longer durations and the precipitates formed during these treatments are both bigger and more distinct. At 400 °C (Figure 6.8c and Figure 6.9), the microstructure consists of large irregularly shaped particles of pure Si and smaller particles with segregations of Si, Fe and Mg. Mg and Fe tend to segregate simultaneously with particles rich in Fe also being rich in Mg. As the solution treatment temperature is further increased to 500 °C and 530 °C (Figure 6.8d -6.8e and Figure 6.9d -6.9e), similar irregular pure Si precipitates are found along with rod shaped Si-Fe-rich precipitates. These rod shaped particles are generally located adjacent to the larger particles of pure Si. Furthermore, some smaller round Mg rich particles are found in the two highest solution treatment temperatures. After the low temperature ageing treatment (Figure 6.9f), the microstructure is characterised by irregular pure Si particles and Si-Fe-Mg particles as observed in the samples treated at lower solution temperatures. In the solution treated

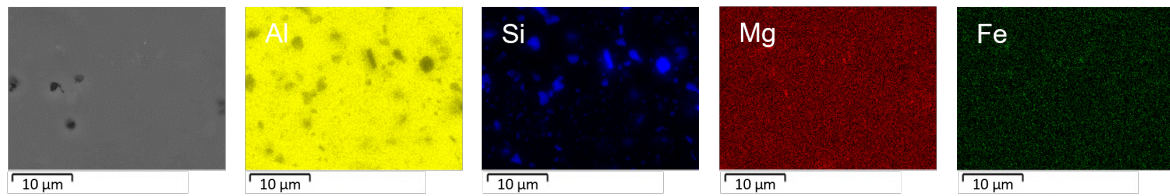
(a) As built



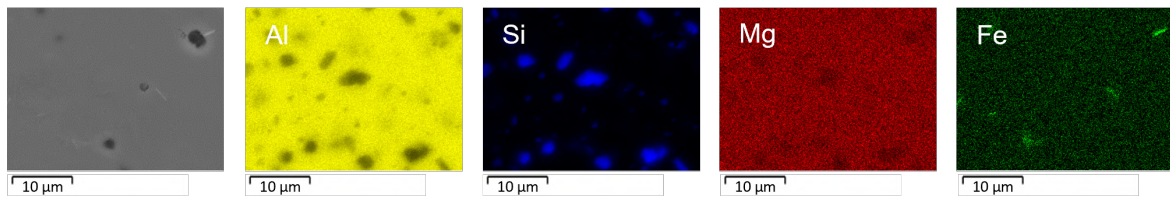
(b) Stress relief



(c) 400 °C solution treatment



(d) 500 °C solution treatment



(e) 530 °C solution treatment

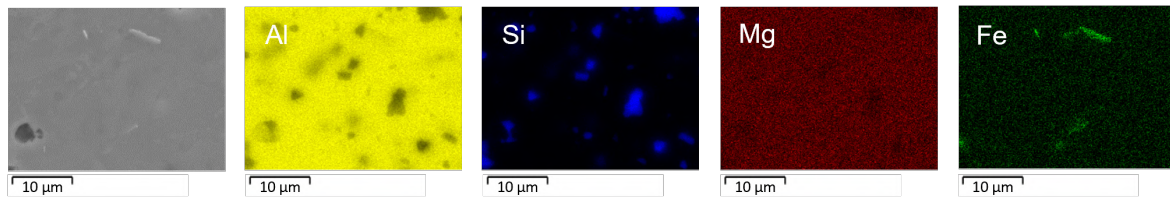
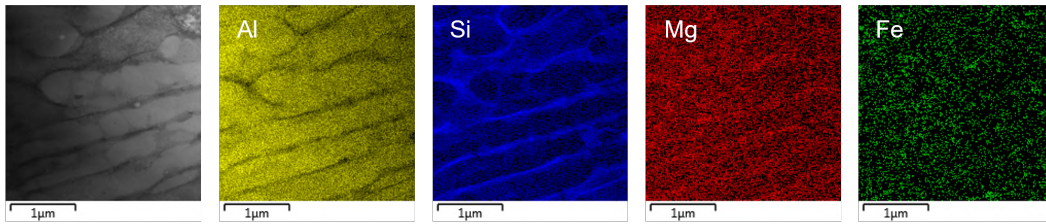


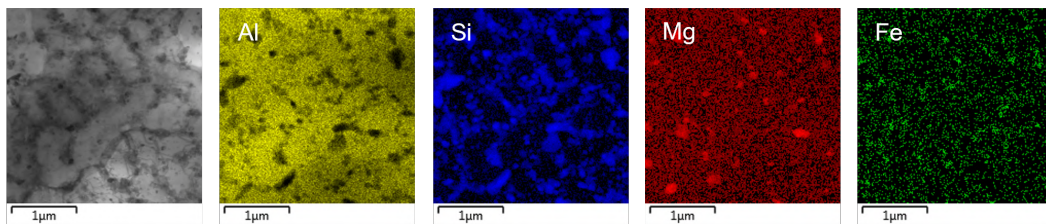
FIGURE 6.8. SEM-EDS maps of samples at different stages of heat treatment: (a) as-built, (b) stress relieved, (c) solution treated 400 °C, (d) solution treated 500 °C, (e) solution treated 530 °C.

specimens, large holes are observed across the samples. They are however not porosities from the PBF-LB process, but rather voids left behind by removal of these pure Si particles by the metallurgical sample preparation process.

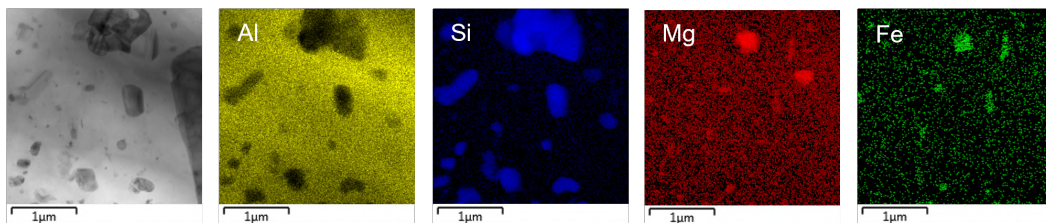
(a) As built



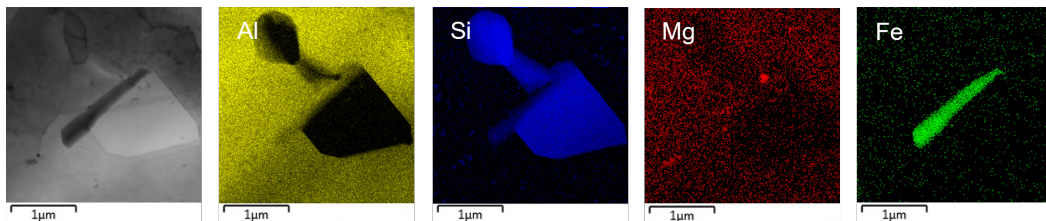
(b) Stress relief



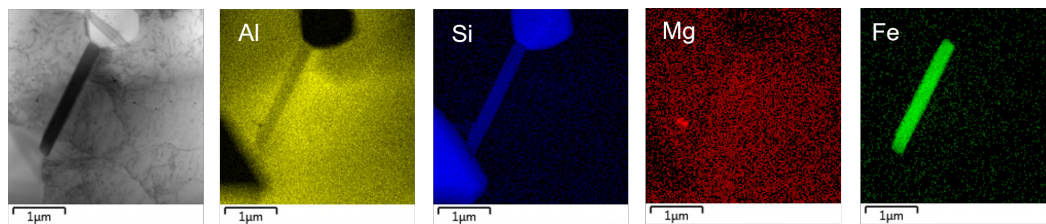
(c) 400 °C solution treatment



(d) 500 °C solution treatment



(e) 530 °C solution treatment



(f) 530 °C solution treatment + ageing

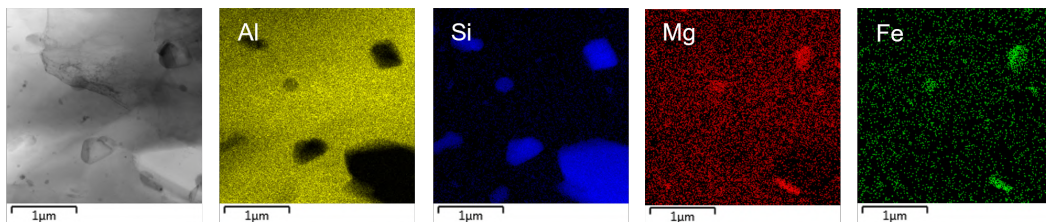


FIGURE 6.9. TEM-EDS maps of samples at different stages of heat treatment: (a) as-built, (b) stress relieved, (c) solution treated 400 °C, (d) solution treated 500 °C, (e) solution treated 530 °C, (f) solution treated 530 °C and aged.

Point measurements were also taken by TEM-EDS to determine the composition of the cell interiors for the as-built and stress relieved samples, as shown in Table 6.2. These show that the already low levels of solute elements in the Al matrix after fabrication are further reduced by a stress relief.

TABLE 6.2. TEM-EDS point analysis for the chemical composition of the cell interiors in as-built and stress relieved samples. Measurements are taken as an average of 7-14 points and are in wt.%.

	Mg	Al	Si	Fe
As-built	0.18 ± 0.03	98.5 ± 0.2	1.29 ± 0.2	0.05 ± 0.02
Stress relieved	0.12 ± 0.02	99.7 ± 0.03	0.17 ± 0.01	0.03 ± 0.02

Atom probe was collected to determine the composition of the matrix after each solution treatment and compared with Thermocalc to determine if locally the expected thermodynamic equilibrium had been reached, as seen in Table 6.3. It is notable that the Mg and Si in the 400, 500 and 530 °C samples never reached the saturation levels predicted by Thermocalc, with sample matrixes also containing a greater saturation of Fe than predicted. This is likely because of insufficient time to diffuse the iron to form β -Al₉Fe₂Si₂ or π -Al₁₈Fe₂Mg₇Si₁₀.

TABLE 6.3. Matrix elemental compositions for solution treated samples. Displayed are compositions measured by atom probe and equilibrium predictions made by Thermocalc (TC). Measurements are in wt.%.

	Mg	Si	Fe	Al
APT 400 °C	0.1495 ± 0.0027	0.2634 ± 0.0008	0.0130 ± 0.0005	Bal.
APT 500 °C	0.3282 ± 0.0011	0.5748 ± 0.0020	0.0171 ± 0.0009	Bal.
APT 530 °C	0.3485 ± 0.0016	0.9445 ± 0.0028	0.0397 ± 0.0015	Bal.
TC Eq. 400 °C	0.176	0.265	5.85x10 ⁻⁵	Bal.
TC Eq. 500 °C	0.416	0.80	0.001	Bal.
TC Eq. 530 °C	0.415	1.05	0.002	Bal.

6.4 Discussion

6.4.1 Microstructural Evolution

6.4.1.1 As-Built Samples

Samples that were aged directly after removal from the build plate show clear signs of over ageing, with samples aged at or below 160 °C showing no change in hardness, while samples aged at higher temperatures all showed a consistent decrease in hardness. This is consistent with over ageing behaviour whereby the desired precipitates grow too large and dispersed to cause any meaningful disruption to dislocation motion.

This indicates that an in-situ heat treatment has already taken place during the PBF-LB process, with the application of a heated build plate and continual heat accumulation from the laser ensuring that the powder bed and the individual parts remain at an elevated temperature. Over the duration of the build this allows for the diffusion of Mg and Si within the matrix and the formation of strengthening phases in the Mg_2Si precipitation sequence [304], as well as some Si particles in the interior of the cells [101]. This in-situ ageing contributes to the high strengths seen in the as-built samples.

Upon the application of higher temperatures during artificial ageing, minimal Mg and Si remains unprecipitated within the matrix to form new particles, with the majority either already precipitated or segregated to the cell wall boundaries as seen in the TEM-EDS results in Figure 6.9. These results show that approximately 1.3 wt.% Si remains in solution within the matrix, lower than the equilibrium solubility of Si in Al of 1.6 wt.% [101] indicating that the as-built specimens are already overaged, with the high Si content of the matrix that is typical of PBF-LB Al-Si alloys drastically diminished by the continual application of heat by the heated build plate. This means further ageing promotes the growth of existing precipitates, reducing their strengthening effectiveness. This effect is accelerated at higher temperatures, with increased temperature and time producing a consistent reduction in hardness. This indicates that a T5 treatment is not always suitable for PBF-LB manufactured components, with over ageing and a reduction in strength a significant possibility.

6.4.1.2 Stress Relieved Samples

The stress relieved condition demonstrated a significantly lower initial hardness than the as-built condition. This is due to the coarsening of the Si cell network brought about by the stress relief, as evidenced by the TEM-EDS results, where the increased discontinuity of this network hinders its ability to strengthen the material. A smaller reduction of the strength would have been achieved by utilising a lower stress relieving temperature, with treatments carried out below 240 °C considered to preserve the cell network structure [111]. The reduction in strength is independent of the grain structure, with a stress relief having no effect on grain size, shape, or texture.

Samples that had been stress relieved prior to ageing showed a complete inability to age harden, regardless of ageing temperature and time. This is due primarily to the already overaged microstructure of the alloy, with Mg and Si rich precipitates along the remnants of the cell walls observable by TEM-EDS. These particles do not provide as significant a strengthening effect as the nanosized Mg_2Si precipitates, with β'' precipitates being approximately 20 nm in length [145], and so contribute to the significant reduction in strength for stress relieved samples when compared to the as-built state.

Further ageing can be seen to have no strengthening effects on the stress relieved samples. This is primarily due to the lack of Mg remaining in solid solution, with EDS showing that only trace amounts of Mg exists in the matrix as Mg-Si precipitates consume the majority of the Mg. This shortage of Mg in the matrix makes the formation of new precipitates in the Mg_2Si sequence difficult, with Ostwald ripening reducing the likelihood of their perseverance. Further growth of these pre-existing particles by the addition of diffused Mg during ageing does not provide any contribution to the strength of the alloy due to their already large size, hence the flatlining strength.

This is supplemented by the lack of Si remaining in solid solution, with the stress relieving treatment facilitating the diffusion of solute Si to grow pre-existing Si precipitates both at the cell walls and in the cell interiors. This means that any Mg remaining in the matrix is not able to diffuse to available Si to form strengthening phases. The consistency of this behaviour across ageing temperatures indicates that to improve this alloys strength by ageing, other heat treatment steps must be conducted between stress relief and ageing treatments.

6.4.1.3 Solution Treated Samples

The initial reduction in strength for the solution treated samples comes for similar reasons to the stress relieved samples, in that a solution treatment brings about the destruction of the Si cell network that significantly strengthens the as-built structure. This effect is more severe for the solution treated samples than those that were stress relieved, with the complete destruction of the cell network and the formation of large, Si precipitates, whereas the stress relieved samples still showed evidence of a cellular network, albeit one with coarser walls. There is also an effect of solution treatment temperature on this initial drop in hardness, with solution treatments lower than 500 °C displaying lower hardness values than samples treated over this temperature. This is attributable to a difference in solid solution strengthening, with high temperature solution treatments retaining a greater amount of solute Si, Mg, Fe, and Ti than low temperature treated samples. Again, the grain structure appears to have no effects on the change in strength for solution treated samples with grain size, shape, and texture unaffected by a solution treatment regardless of temperature.

The solution temperature used during the T6 treatment has a clear effect on the outcome of ageing, with temperatures of 475 °C and below producing poor or no age hardening effects, while an increase in solution temperature to 500 °C resulted in an improvement in peak ageing of 18.7 HV. This peak hardness was further improved by continuing to increase the solution temperature.

This is seen to be the result of the different amounts of Mg and Si in solid solution remaining within the Al matrix after each of these solution treatments. At 400 °C, the equilibrium composition of Mg within AlSi7Mg is 0.176 wt.% which is matched well by the composition as measured by APT of 0.150 wt.%, with quenching after the solution treatment able to minimise the diffusion of Mg out of the matrix. This low level of Mg is accompanied by the relatively low concentration of solute Si, measured at 0.263 wt.%. The low levels of these strengthening elements in solution within the Al reduces the ability of the alloy to age harden, as the strengthening precipitates are less able to form. This contrasts with the solute concentrations in the 500 °C solution treated sample with 0.328 wt.% Mg and 0.575 wt.% Si present within the matrix, values slightly lower than the equilibrium concentrations predicted by ThermoCalc. This difference is likely related to the localised nature of the APT technique and the time required to reach equilibrium at each of the solution treatment temperatures.

The increased presence of these elements in solution results in a greater ability to grow strengthening precipitates, meaning that at solution temperatures of 500 °C and greater age hardening is possible. This effect is further enhanced at higher temperatures where a 530 °C solution treatment where a concentration of 0.345 wt.% Mg and 0.945 wt.% Si in the matrix leads to further age hardening, with a peak hardness of 101.5 HV reached after 6.5 hours, although a plateau in the ageing curve is seen from 4.5 hours onwards.

These differences in solute chemistry are driven primarily by the different precipitates formed within AlSi7Mg at different temperatures. For solution temperatures below 493 °C, according to thermodynamic calculations from ThermoCalc as displayed in Figure 6.10, the main Fe rich phase that forms is the π -Al₁₈Fe₂Mg₇Si₁₀ phase. The presence of this phase is confirmed by the TEM-EDS maps as globular particles rich in Fe, Mg and Si are found in the 400 °C

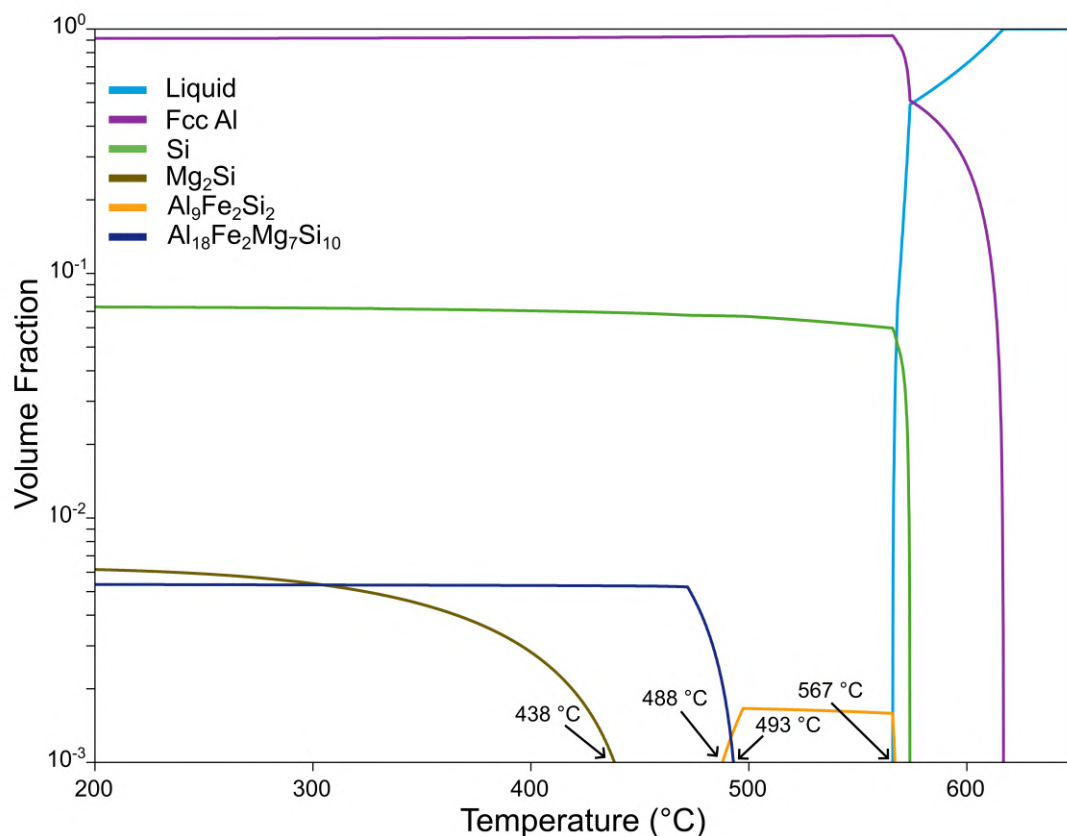


FIGURE 6.10. ThermoCalc phase diagram for the AlSi7Mg alloy with the composition Al-6.77Si-0.39Mg-0.06Fe-0.04Ti (wt.%), which is the composition of parts used in this work as measured by ICP-AES. The temperatures at which important phases begin to form or stop being stable are also displayed.

solution treated samples. Also present at temperatures below 438 °C is Mg_2Si , the formation of which after a solution treatment at 400 °C has minimal improvement on the strength of the alloy due to its large size relative to the strengthening precipitates formed in ageing treatments. The formation of these two Mg containing phases at low solution temperatures reduces the amount of Mg in solid solution. For higher temperatures the equilibrium phases of AlSi7Mg change, and so the amount of Mg in solid solution is increased. For temperatures over 488 °C the $\beta\text{-Al}_9\text{Fe}_2\text{Si}_2$ phase begins to form at the expense of $\pi\text{-Al}_{18}\text{Fe}_2\text{Mg}_7\text{Si}_{10}$. These phases are needle shaped, and their presence and composition are also observable in the TEM-EDS measurements. Given this phase does not contain Mg, this frees the Mg to exist in solid solution within the Al matrix and so improves the age hardenability of samples heat treated at temperatures higher than 488 °C. This trend is consistent until 493 °C, where the $\pi\text{-Al}_{18}\text{Fe}_2\text{Mg}_7\text{Si}_{10}$ phase is no longer present and increasing solution treatment temperatures will not significantly affect phase formation within the alloy.

6.4.2 Mechanical Properties

6.4.2.1 Tensile Properties

The tensile properties of the as-built condition show high strength with the trade-off of relatively low ductility when compared to heat treated samples, as shown in Figure 6.11. The reasons behind this high strength are well understood and are partly attributable to the high residual stresses remaining after the PBF-LB process and the associated high number of dislocations [155], as well as solid solution strengthening. This is primarily by Si which is present in the matrix at a concentration of 1.29 wt.% as measured by TEM-EDS, but also Mg, present at 0.18 wt.%. The majority of the strength, however, comes from the fine cell network present within the grains of the as-built microstructure. These cells are predominantly elongated, following the aspect ratio of the parent grain they reside in and being in the range of 0.3-1 μm in size [145, 305], with the cell walls comprised of nanometric Si particles formed by the ejection of Si from the Al matrix during solidification. These cell walls strengthen the alloy as their fine size hinders the motion of dislocations through these walls, aided by the presence of a dislocation network along these boundaries [130]. Fracture often relates to this cellular structure, as it is reliant on the number of rupture locations available along the failure plane. Due to the many small Si precipitates along these cell walls, there are many

crack initiation sites that eventually begin to form cracks which often mean the cell network remains visible on fracture surfaces, as evidenced in other works [148]. However, the small size of these Si precipitates results in a relatively high localised stress required for fracture [38], meaning they do not provide a meaningful reduction in part ductility. The ductility of as-built samples is more strongly influenced by the amount of Si in solid solution, low angle grain boundaries, and the high dislocation density [283].

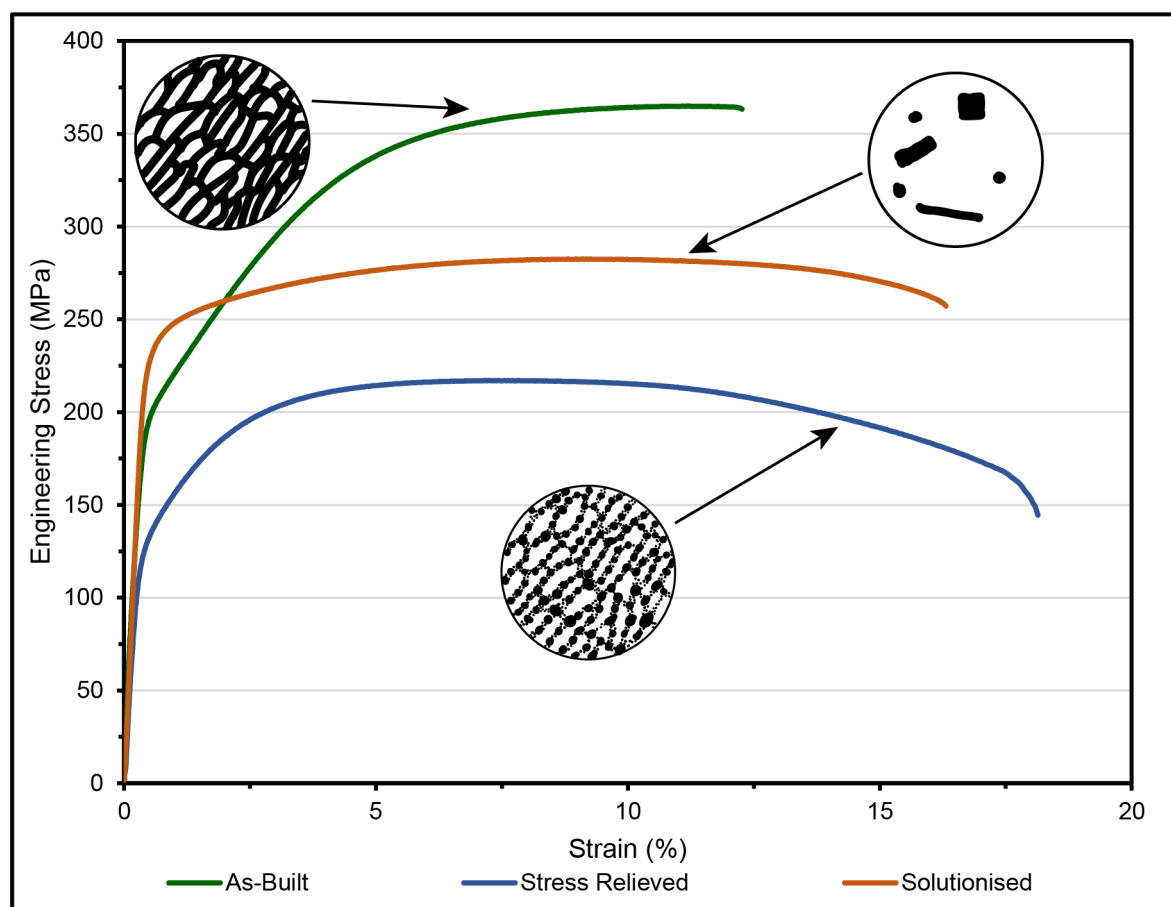


FIGURE 6.11. A representative stress strain curve for each of the heat treatment states tested: as-built, stress relieved, and T6 treated. Also shown are schematics of the microstructure for each specimen; the as-built microstructure consisting of cell walls of nanosized Si precipitates that appear continuous at low magnification; the stress relieved microstructure demonstrating the coarsening of these cell walls due to Ostwald ripening; and the T6 microstructure consisting of large Si particles, elongated Fe rich precipitates, and nanosized Mg₂Si precipitates. Also evident is the difference in strain hardening behaviour for each heat treatment state.

A stress relieving treatment produces a significant reduction in strength accompanied by an increase in ductility, as the stress relieving treatment degrades each of the main strengthening contributions available within the as-built microstructure. These treatments primarily reduce the number of dislocations within the alloy as well as reducing residual stress present from the rapid cooling of the material during the production process. It also softens the Si cell network, with the continuous cell borders beginning to break up as individual particles along these edges consume nearby particles while also drawing Si from solution within the matrix. This creates a more discontinuous network of Si precipitates rather than definitive cell boundaries, meaning the motion of dislocations through this network is less readily hindered due to these particles larger size and greater spread through the matrix. The growth of these particles at the expense of Si within the matrix also reduces the amount of solution strengthening the matrix can provide, further weakening the material. In addition to this reduction in strength, the fewer and larger Si precipitates present in the stress relieved samples increases ductility by reducing the number crack initiation sites and promoting plastic deformation by their reduced ability to hinder dislocation motion.

T6 samples feature different behaviour with their yield strength the highest of the three heat treatment cases tested, while their tensile strength and ductility lies between those of the as-built and stress relieved samples. The unique combination of a higher yield strength but lower tensile strength when compared to as-built samples is explained by the different strengthening mechanisms at work within these heat treatment cases.

While as-built specimens rely on the high volume of dislocations present after manufacturing as well as the fine cell network, T6 treated specimens are strengthened primarily by the presence of precipitates. A number of precipitates are formed during T6 treatments including Si particles, β -Al₉Fe₂Si₂ needle-like phases and the π -Al₁₈Fe₂Mg₇Si₁₀ phase, however strengthening is primarily attributed to the growth of the Mg₂Si based β' and β'' precipitates rather than Fe rich ones [142, 149]. These nanometric sized precipitates pin the few dislocations that remain after the solution treatment, restricting their movement and therefore increasing the stress required for yielding. The small size and scattered dispersion of these precipitates means they are better able to prevent this initial slip than the larger Si precipitates along the cell wall boundaries that are responsible for similar strengthening in the as-built state, meaning the yield strength for the T6 samples is greater than that of the as-built samples.

However, once yielding has occurred and many of the Mg_2Si precipitates have been sheared, the minimal amount of solid solution strengthening and the lack of smaller precipitates of a suitable size to block dislocation motion results in minimal strain hardening for the T6 samples, and therefore a relatively low tensile strength. This is in comparison to the as-built samples where the fine cell size continues to strengthen the material, alongside the large number of dislocations continuing to entangle as they move through the matrix resulting in good strain hardening and a relatively high tensile strength, with similar behaviour seen in AlSi10Mg [306]. The higher level of solute Si in the as-built microstructure has also been linked to improved strain hardening due to dynamic precipitation under loading [307].

6.4.2.2 Impact Toughness

As heat treatments are applied to the material, a continual reduction in impact toughness is observed with the as-built samples having the highest energy absorption of 2.99 J, stress relieved samples 2.38 J, and T6 samples 0.61 J. The reduction in impact toughness is explained by the change in yield strength, precipitates, and porosity.

In general, a high yield strength is seen to result in a low impact toughness, which given the high yield strength of the T6 samples compared to the as-built and stress relieved states and its corresponding low toughness, is one relevant influence on impact toughness. However, the fact that the stress relieved sample, which had the lowest yield strength, was not the toughest of the samples indicates that other factors are also significant.

One of the major contributors to the toughness of the as-built microstructure is the fine cell network. These cells functionally act as fine grains that slow the propagation of cracks, increasing the load required to push through these regions and thereby increasing its toughness. This effect is reduced in the stress relieved samples and functionally non-existent in the T6 samples, given the strong coarsening effect of a stress relief on the cell network and its destruction after a solution treatment. The lack of a cell structure in the T6 samples can therefore be seen to contribute to its low toughness. Similar results have been seen in cast Al-Si alloys, whereby a reduction in grain size by rotary-die equal-channel angular pressing and the presence of Si precipitates smaller than 1 μm were both seen to improve impact toughness [308].

Another major factor in the reduction of the toughness of the T6 specimen is the presence of Fe rich phases, particularly the β -Al₉Fe₂Si₂ phase. In cast AlSi7Mg alloys an increase in Fe content has been seen to reduce the fracture toughness as the increased presence of Fe rich intermetallic phases increases the brittleness of the alloy [309]. This is related to the more brittle and weaker nature of these particles compared to the Al matrix, promoting earlier fracture at the interface between these phases and the matrix. The presence of these precipitates after solution treating at 530 °C, and their preservation through ageing, means that the toughness of the T6 treated material is reduced.

6.5 Conclusion

This work investigated various heat treatments on additively manufactured AlSi7Mg in order to determine the effect of different solution temperatures.

Samples that were directly aged in the as-built state demonstrated signs of over ageing, with ageing at any temperature resulting in a reduction in hardness. This over ageing behaviour was driven by in situ ageing during the manufacturing process due to the use of a heated build plate.

Stress relieved samples were also over aged, with ageing treatments resulting in no changes to sample hardness. A significant reduction in strength in comparison to the as-built samples was also observed due to the breakdown of the Si cell network that provides the majority of the as-built microstructures strength.

The solution treatment temperature used was seen to influence the ability of AlSi7Mg to age, with samples solution treated below 500 °C showing poor or no age hardenability, while samples treated over this temperature were able to age harden as expected. This was due to the formation of the π -Al₁₈Fe₂Mg₇Si₁₀ phase under 493 °C, which reduced the amount of Mg in solid solution at these temperatures, thereby limiting the precipitation of the strengthening Mg₂Si phase. Between 488 °C and 493 °C, the π -Al₁₈Fe₂Mg₇Si₁₀ phase is gradually replaced by the β -Al₉Fe₂Si₂ phase, allowing a greater amount of Mg to be present within the matrix and therefore promoting Mg₂Si precipitation. This resulted in an optimal solution treatment

temperature of 530 °C to be found according to its peak ageing behaviour after ageing at 160 °C.

A T6 treatment was seen to increase the yield strength but reduce the tensile strength of AlSi7Mg when compared to the as-built state, which was seen to be a result of the different strengthening mechanisms at work in each heat treatment state. The ductility was also improved, while the impact toughness decreased.

Given the poor age hardenability of both the as-built and stress relieved states, this suggests that a further heat treatment step is required prior to ageing treatments when a heated build plate is used in the PBF-LB process. The improvement in properties demonstrated by the T6 heat treatment suggests that heat treatment for AlSi7Mg is likely an integral part of PBF-LB process chain.

The Refreshing Powder Reuse Method and its Effects of Powder and Part Properties

7.1 Introduction

One of the advantages of PBF is its ability to create parts of unique and complex geometries, as the layer-by-layer process allows for the gradual deposition of material, reducing the need to design for machining considerations. These complex geometries, in combination with the large powder chamber volumes in PBF machines in relation to the parts manufactured, mean that there is often a considerable volume of unmelted powder remaining after a manufacturing run. This unconsumed powder must be reused in subsequent manufacturing runs to ensure the economic viability of the process. A proper understanding of the effects of powder reuse are crucial to both ensure consistency in part properties between manufacturing runs, but also ensure that minimum standards are met in relation to powder chemistry and final part properties, as required by some standards [203, 310].

Many of the properties of both powders and parts are well investigated in reused powders, as reviewed in Chapter 3 of this thesis. Amongst other properties, studies have variously seen increases [215] and reductions [166] in powder oxygen content, increases [246] and reductions [66] in tensile strength, and improvements [171] and deteriorations [173] in powder flow properties. These inconsistencies can be attributed to a variety of factors such as the material used, number of manufacturing runs, the parts produced in each manufacturing run, and the powder reuse method employed. While many of these factors are acknowledged within the literature, the effect of powder reuse method is rarely considered.

As it stands, there are a variety of powder reuse methods that have been utilised within the literature, as reviewed in Chapter 3 of this thesis. One of the most common of these is the

refreshing method, where new, unused powder is added to a powder batch at regular intervals to replenish the powder consumed in previous manufacturing runs. This creates a powder batch of consistent volume that is regularly topped up and used, in theory indefinitely, with minimal change in powder and part properties due to the continual introduction of unused powder. This method provides great promise for powder reuse in PBF, however needs to be properly investigated to be consistently utilised with an understanding of its effects on powder and part properties.

This chapter investigates the refreshing powder reuse method in detail, involving powder characterisation, microstructural investigation, part property analysis, and mechanical property investigation. This provides a comprehensive assessment of the effectiveness of this powder reuse method for AlSi7Mg, as well as providing a basis for comparison with the single batch method in Chapter 8.

7.2 Powder Reuse Scheme

In order to investigate the refreshing powder reuse method, a number of specimens and blocks for machined specimens were produced in each manufacturing run. The same specimens were manufactured in each run to allow both consistency in the properties tested and also to ensure that the melt volume was constant so there would be no changes to powder or part properties due to a change in spatter and heat generation between manufacturing runs. An image of the build plate produced in each manufacturing run is shown in Figure 7.1.

At the end of each manufacturing run, new powder was added into the old powder batch prior to sieving. Each manufacturing run consumed 18 % of the powder within the build chamber, which was replaced for each subsequent build. However, due to inconsistencies in the amount of powder lost during powder handling processes such as sieving, the amount of powder added for each manufacturing run varied slightly. The powder composition for each build number, as a function of powder age, can be seen in Figure 7.2. Powder batches early in the reuse cycle are comprised of powders with only a few differing powder histories, whereas those in the later manufacturing runs are composed of powders with more varied histories. As powder is continually used and introduced, the batch, as a whole, approaches an equilibrium state where each subsequent manufacturing run shows minimal change in powder history. For

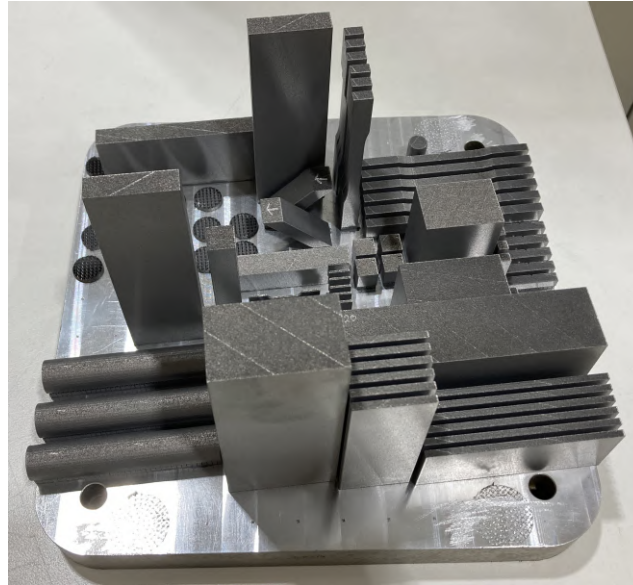


FIGURE 7.1. Build plate manufactured for each manufacturing run in the refreshing powder reuse scheme.

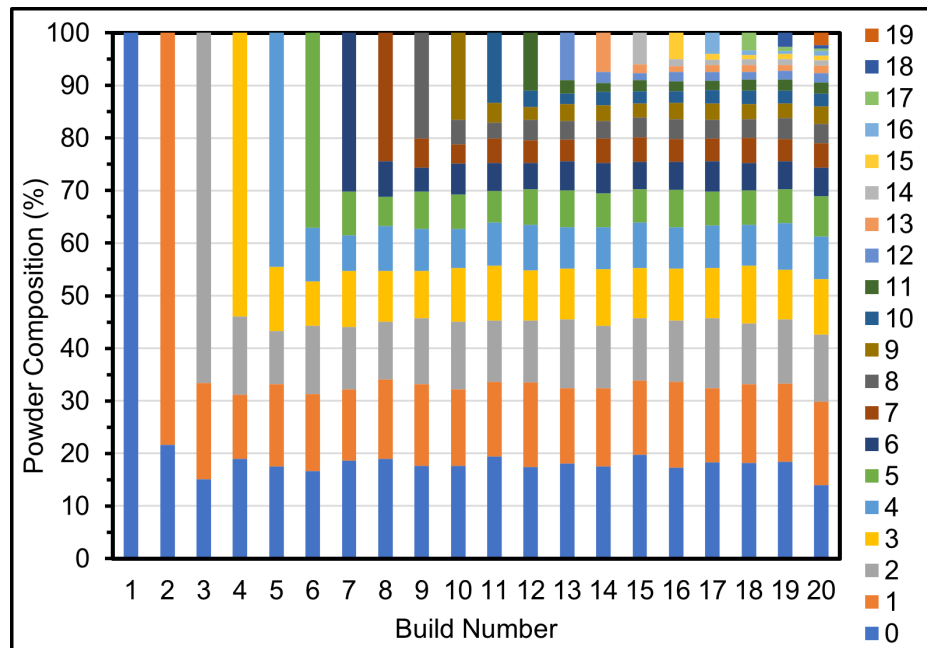


FIGURE 7.2. Powder composition as a function of powder age. Each colour refers to the number of manufacturing runs that a segment of powder has been used for, i.e. light blue (0) corresponds to powder never used prior to the current manufacturing run, while maroon (7) corresponds to powder used seven times prior to the current manufacturing run.

this reason, 20 manufacturing runs were carried out for this investigation, with changes to the powder batch's history in subsequent manufacturing runs deemed to be negligible.

Throughout this chapter each of the manufacturing runs is referred to by the letter R, indicating the use of the refreshing powder reuse method, and a number referencing the manufacturing run itself. R1 refers to the first manufacturing run completed in the refreshing powder reuse study, with R20 being the last.

7.3 Results

7.3.1 Powder Characterisation

7.3.1.1 Powder Imaging

Qualitative assessment of powder quality was conducted by SEM imaging of powder particles, with representative images of each powder batch shown in Figure 7.3. The majority of particles in each case are highly spherical with diameters approximately 20 – 40 μm . Each powder batch shows evidence of the inclusion of some particles that are either noticeably larger, or are some form of agglomerate, satellite, or elongated particle. The prevalence of these non-spherical particles tends to increase with continual powder reuse; however, this is not possible to quantitatively assert using this technique.

7.3.1.2 Particle Size Distribution

The PSD is one of the most common powder characteristics tested within the literature due to its simplicity of measurement and its effects on powder flow properties [68]. The D10, D50, and D90 values of the powder as a function of reuse are shown in Figure 7.4. The D10 size changes from R1 to R5, increasing from 24.2 μm to 25.6 μm , after which it stabilises and shows no significant change. The D50 results have an identical trend where this size increases from 36.8 μm to 39.4 μm after five manufacturing runs, whereupon it also stabilises. The D90 values are similar but increase in size for a slightly longer period, taking until R7 to increase from 55.1 μm to 61.4 μm , whereupon there is no further change in size.

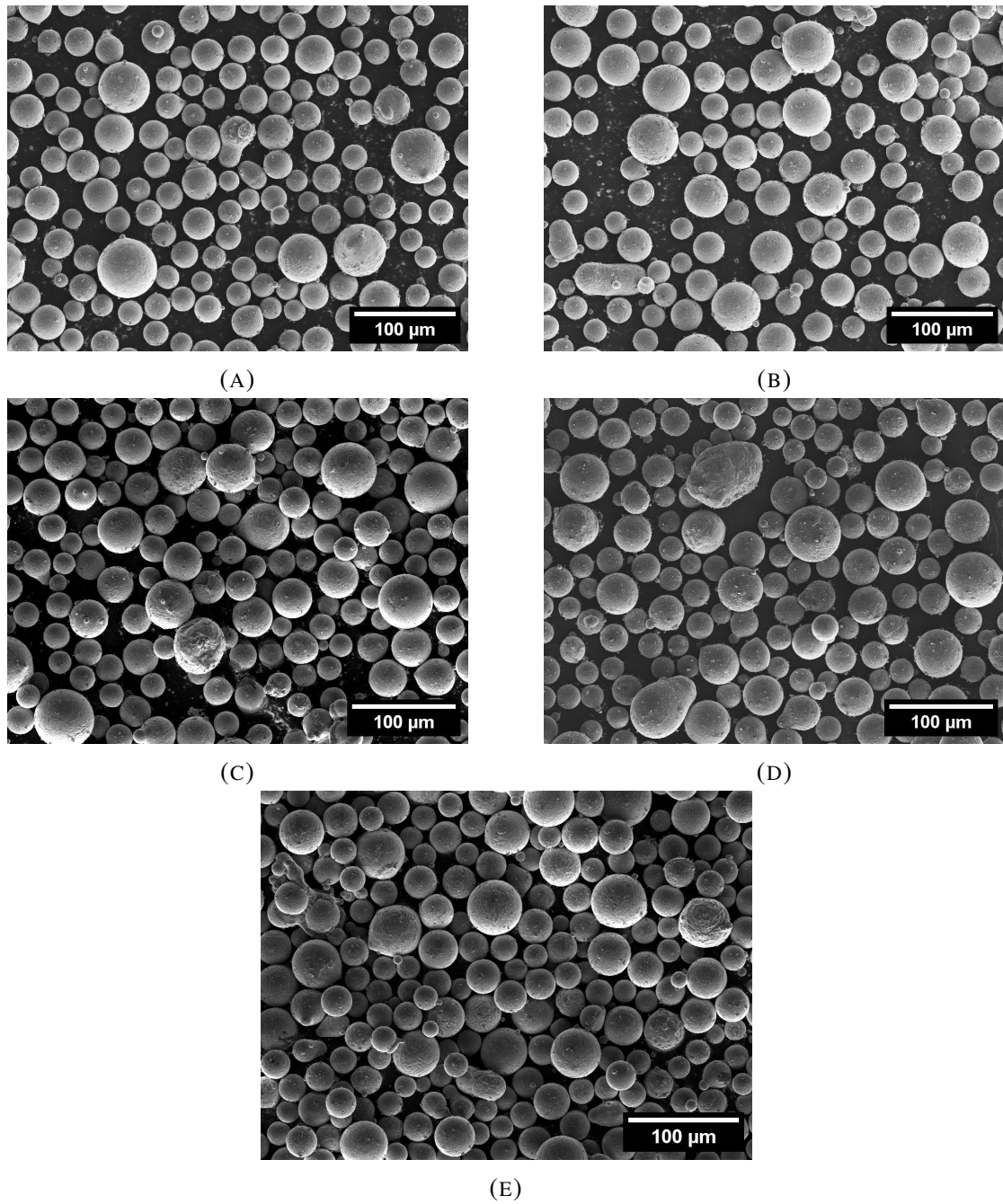


FIGURE 7.3. Representative powder samples of reused powder from the refreshing method: (a) R1, (b) R5, (c) R10, (d) R15, (e) R20. Powders tend to be highly spherical, however present in each powder batch are also non-uniform particles.

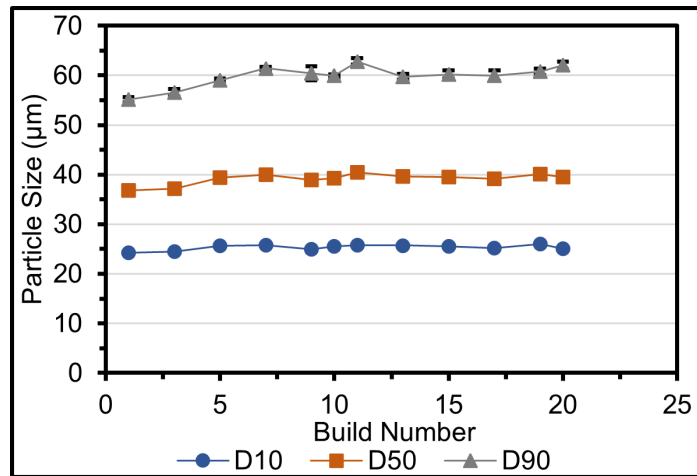


FIGURE 7.4. Evolution of the D10, D50, and D90 values of the powder reused by the refreshing method.

These changes in size result in a slight shift in the PSD, as displayed in Figure 7.5. This shows that the powder initially had a relatively narrow distribution of sizes, evidenced by the higher peak of the R1 in comparison to the other powder batches. As the powder continues to be reused this peak reduces, indicating a widening of the PSD. This is primarily achieved by the increase in large powder particles, as this distribution is seen to become more right skewed, with the left-hand side of these distributions remaining relatively stable while the right-hand side increases in size.

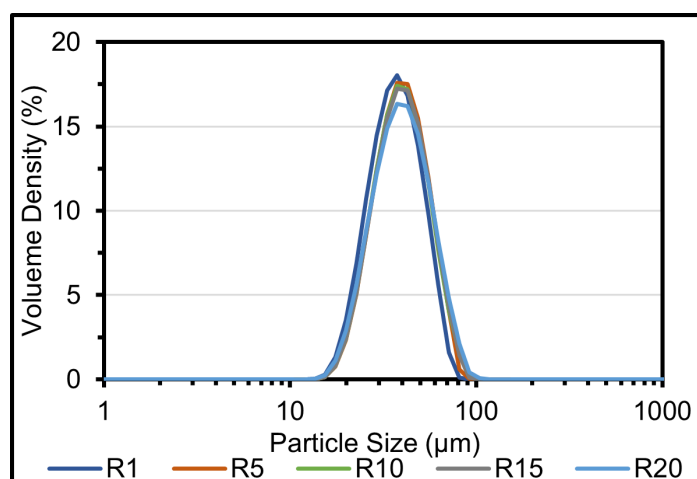


FIGURE 7.5. Distribution of the size volume density of powder reused by the refreshing method.

7.3.1.3 Hall and Carney Flow

Hall and Carney flow measurements were conducted to determine any changes in powder flow behaviour, and the results of this investigation are shown in Figure 7.6. It is clear that there is a distinct improvement in flow time in both tests over the first four manufacturing runs before these times stabilise. The reduction in flow times indicates an improvement in powder flowability. This change in flowability indicates that initially powder was more difficult to spread in the build chamber, but after four manufacturing runs this spreadability improved.

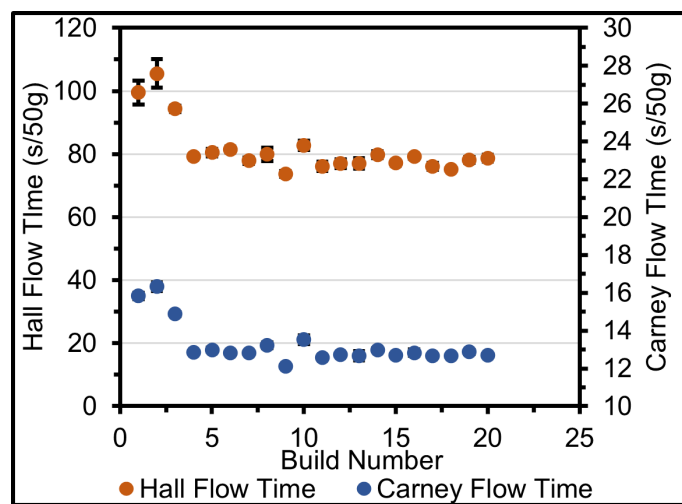


FIGURE 7.6. Hall and Carney flow times of powder reused by the refreshing method.

7.3.1.4 Apparent Density

The apparent density of powder refers to its density after being subject to free fall and is taken as a measure of flowability and packing within the build chamber. The change in apparent density while using the refreshing powder reuse method is shown in Figure 7.7. There appears to be a reduction in apparent density over the first five manufacturing runs before the apparent density becomes more consistent, indicating a reduction in the packing density of powder within the build chamber. The validity of this was confirmed by an ANOVA test followed by a Tukey-Kramer analysis, which showed that the apparent density difference was statistically significant for R1 and R3 in comparison to all other manufacturing runs. Details of this analysis are included in Appendix B1.1. The outlier point likely stems from the manual

process used to measure the apparent density, with an error in mass measurements resulting in a lower than expected apparent density.

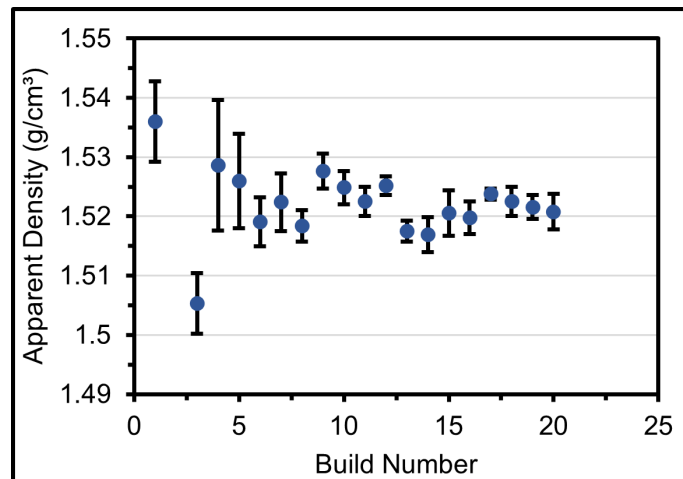


FIGURE 7.7. Powder apparent density of powder reused by the refreshing method.

7.3.1.5 Powder Flowability

Powder flowability measured by the Granudrum produces a number of results to indicate the behaviour of the powder under different conditions. The first is the avalanche angle, as shown in Figure 7.8. An ANOVA analysis was conducted showing that the changes in avalanche angle are statistically significant, with details of this analysis displayed in Appendix B1.2. However, there is no consistent trend relating the avalanche angle due to powder reuse with a reduction in angle until R14, whereupon it increases to reach its starting values in later manufacturing runs. This lack of trend is exacerbated by the large standard deviations seen within the measurements, reducing the likelihood of a meaningful trend relating to powder reuse.

The angle at which powder is able to stably flow at a certain rotating speed is referred to as the sequence velocity angle, when the speed is gradually increased, and the reverse velocity angle, when the speed is gradually decreased. These tests were conducted on reused powders, the results of which are shown in Figures 7.9 and 7.10. While there is considerable scatter amongst the data, these show that the velocity angles are slightly larger at higher speeds compared to lower speeds, as each line slopes upwards. This indicates the powder demonstrates shear thickening behaviour, meaning that at higher rotation speeds, i.e. higher

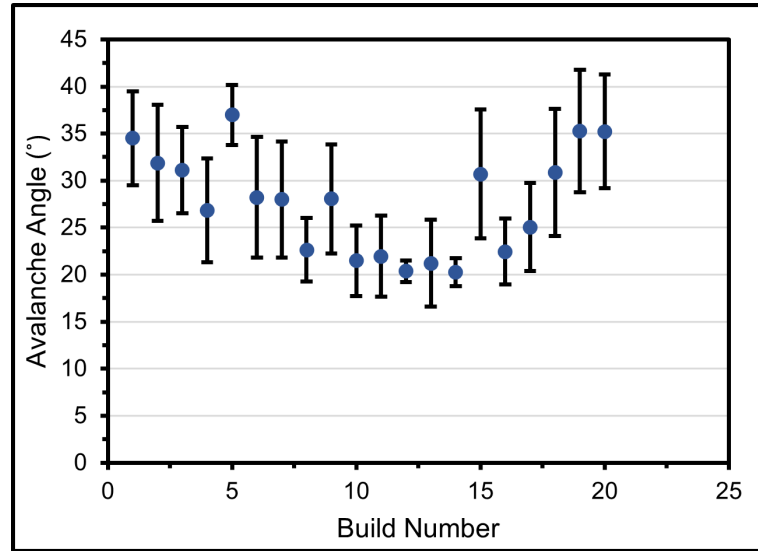


FIGURE 7.8. Avalanche angle of powder reused by the refreshing method.

shear stresses, powder flowability decreases. This behaviour appears to intensify with powder reuse, with the R20 sample having low velocity angles at low rotation speeds in both the sequence and reverse testing modes, and the highest angles at high speeds. This is compared to the R1 sample which demonstrates a more constant flow behaviour with changing speed.

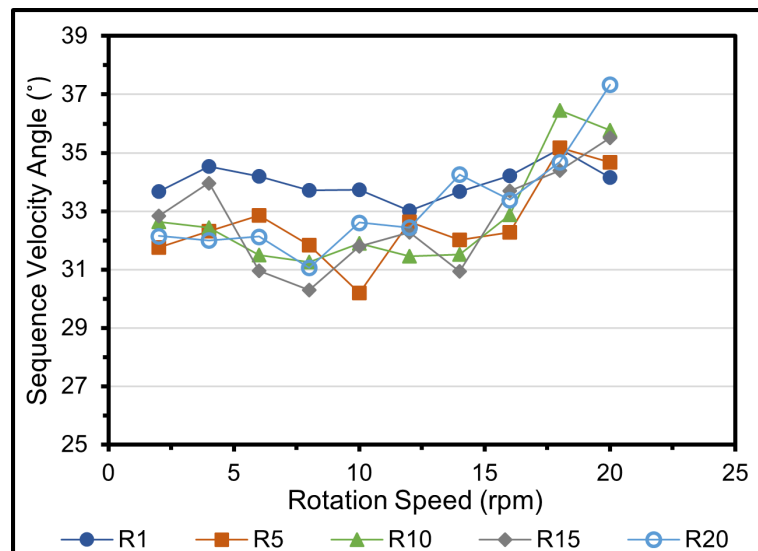


FIGURE 7.9. Sequence velocity angle of powder reused by the refreshing method.

The cohesion of powder while flowing was captured simultaneously with the velocity angle data, with these results shown in Figure 7.11. These results also show shear thickening

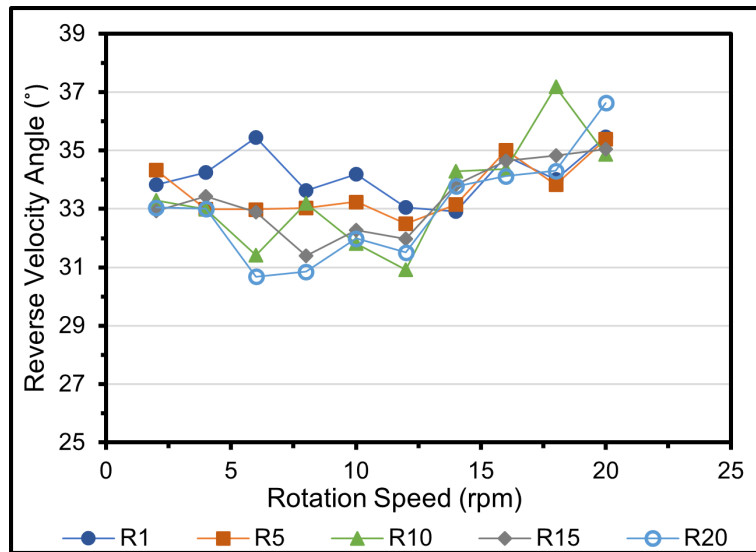


FIGURE 7.10. Reverse velocity angle of powder reused by the refreshing method.

behaviour by an increase in powder cohesion as the rotation speed increases, with this trend clearer than in the velocity angle data. The cohesion lines in Figure 7.11 consistently steepen with increased powder reuse, indicating that stronger shear thickening behaviour occurs as powder is reused.

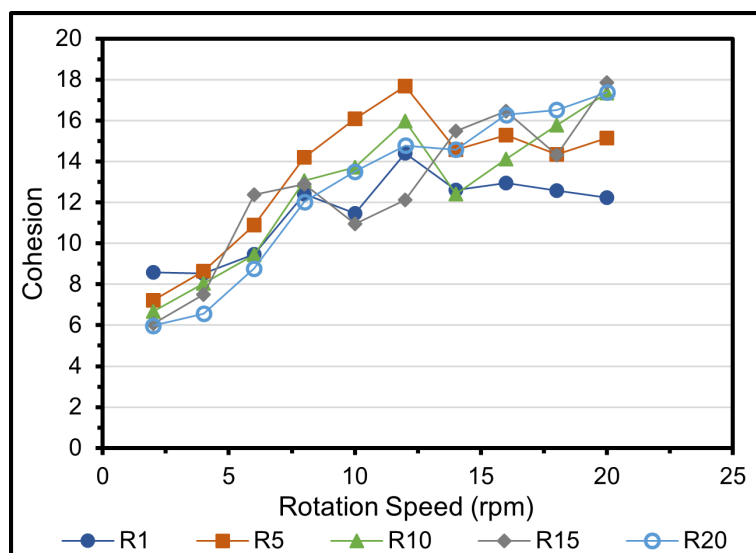


FIGURE 7.11. Cohesion of powder reused by the refreshing method.

7.3.1.6 Powder Bed Density

The powder bed density of each manufacturing run, averaged over the four corner locations of the build plate, is shown in Figure 7.12. This shows a consistent powder bed density across each of the manufacturing runs, with an ANOVA analysis showing no statistical significance related to powder reuse (see Appendix B1.3 for detail) with the scatter in these measurements stemming from a slightly higher powder bed density at the front of the build plate compared to the back, consistent across manufacturing runs.

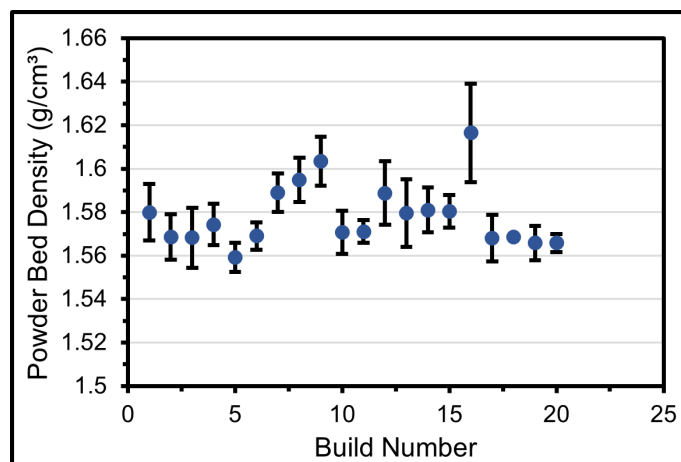


FIGURE 7.12. Powder bed density of powder reused by the refreshing method.

7.3.1.7 Powder Bulk Chemistry

The bulk powder chemical composition shows good consistency with powder reuse, with minimal change in the constituent elements seen in Figure 7.13. Silicon, iron and titanium all show only minor changes in their presence, and none to the degree required to put them outside the required specifications [311]. The oxygen content of the powder is the main element that sees meaningful change, with a reduction from 0.11 wt.% to 0.03 wt.% from R1 to R5. The oxygen content then appears to stabilise, however, three of the lowest four measurements were taken in the last three samples, at R15, R17, and R20. While these changes are clear, it is likely that they are a function of the extended storage times prior to testing rather than any meaningful change as a result of powder reuse, with specimens in earlier manufacturing runs spending longer in storage and therefore containing higher oxygen contents. The magnesium content of the powder also appears to slightly decrease as powder

is reused, with a steady reduction from 0.42 wt.% at R1 to 0.39 wt.% at R20. While this decrease is slight, it may affect the strengthening of the alloy during ageing treatments.

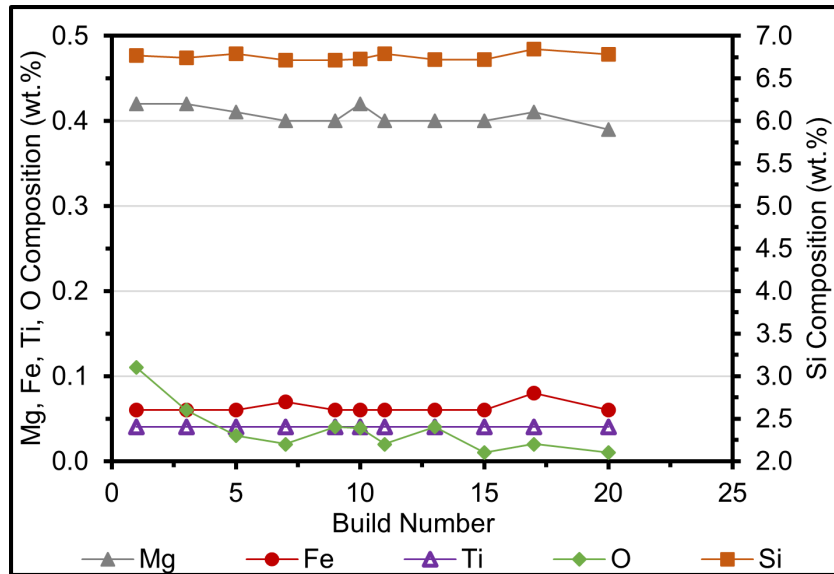


FIGURE 7.13. Bulk chemistry of powder reused by the refreshing method. All measurements are in wt.%.

7.3.1.8 Powder Surface Chemistry

The surface chemistry of the powder was seen to be consistent over the course of reuse, with a slight increase in magnesium content at the expense of silicon from R16 onwards being the only noticeable change. This is displayed in Figure 7.14. The chemical composition was also relatively stable over the 20 manufacturing runs with the surface chemistry dominated by Al_2O_3 , as shown in Figure 7.15. This comprised 70 % – 80 % of the surface of the powder for each manufacturing run, as expected for a readily oxidising aluminium alloy. Pure aluminium and SiO_2 were the least stable of the other compounds measured, however, both had relatively low concentrations in comparison to Al_2O_3 .

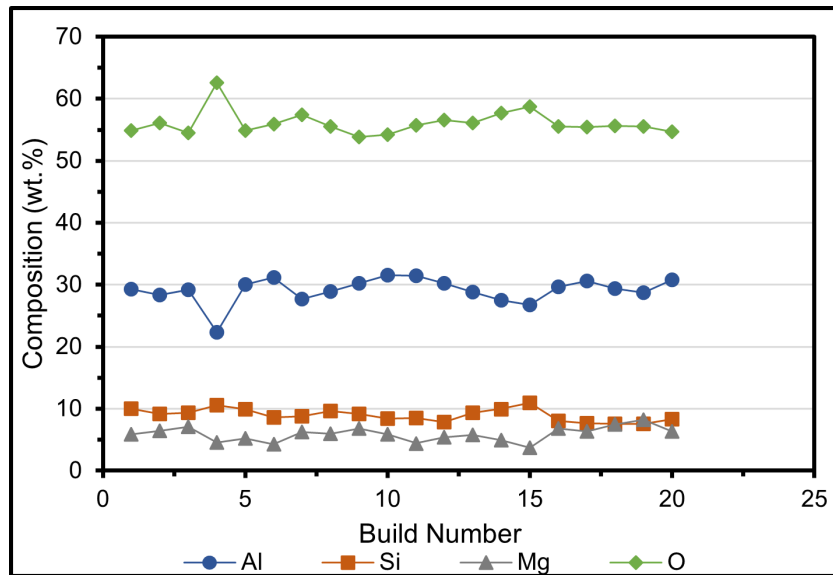


FIGURE 7.14. The presence of different elements on the surface of powder particles, as measured by XPS. All measurements are in wt.%.

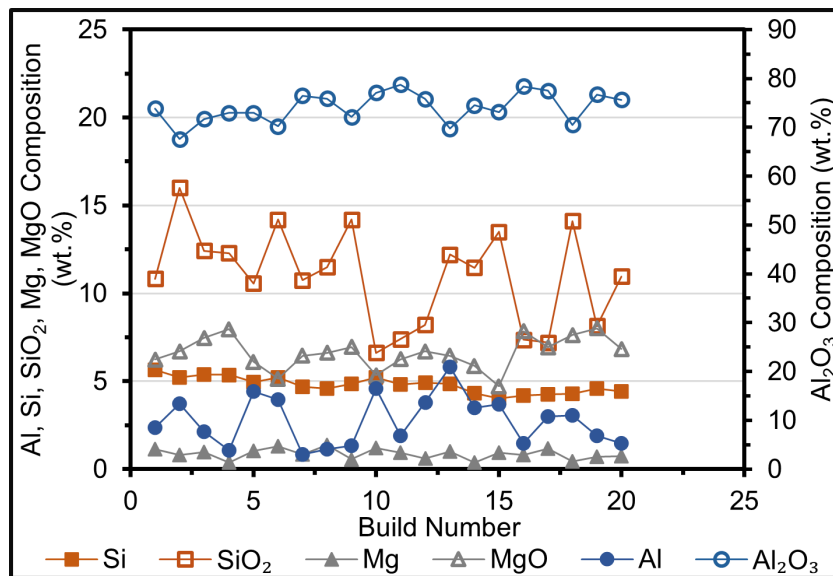


FIGURE 7.15. The presence of different chemical compounds on the surface of powders as a function of powder reuse. All measurements are in wt.%. Peak fitting curves for this data can be found in Appendix C1.

7.3.2 Microstructural Analysis

7.3.2.1 Melt Pool Characterisation

The X-Z cross sections of samples manufactured using reused powder each show the fish-scale microstructure inherent to the PBF process, as shown in Figure 7.16. These show the locations of the melt pools, with no change apparent due to the reuse state of the powder.

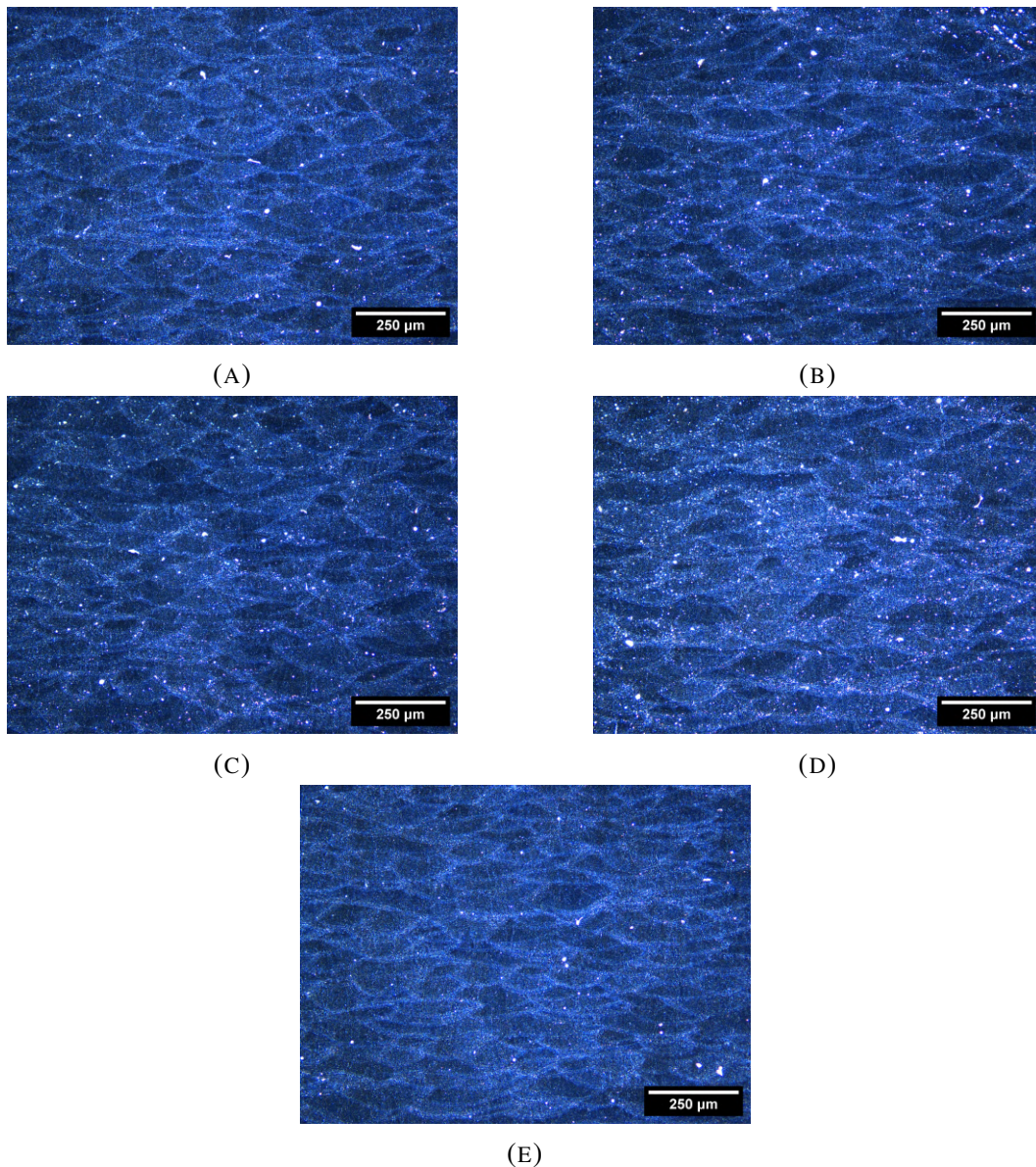


FIGURE 7.16. Dark field optical micrographs of the X-Z plane of samples manufactured with powder at different stages of reuse: (a) R1, (b) R5, (c), R10, (d) R15, (e) R20.

7.3.2.2 Grain Size Characterisation and Crystallographic Texture

Within these melt pools is the characteristic grain structure of PBF materials, with columnar grains in the centres of melt pools and finer, equiaxed grains present along the edges. This is shown in Figure 7.17. The pole figures in Figure 7.18 show that this texture is strong in the [100] direction, with no change in grain size or texture due to powder reuse as shown in Table 7.1.

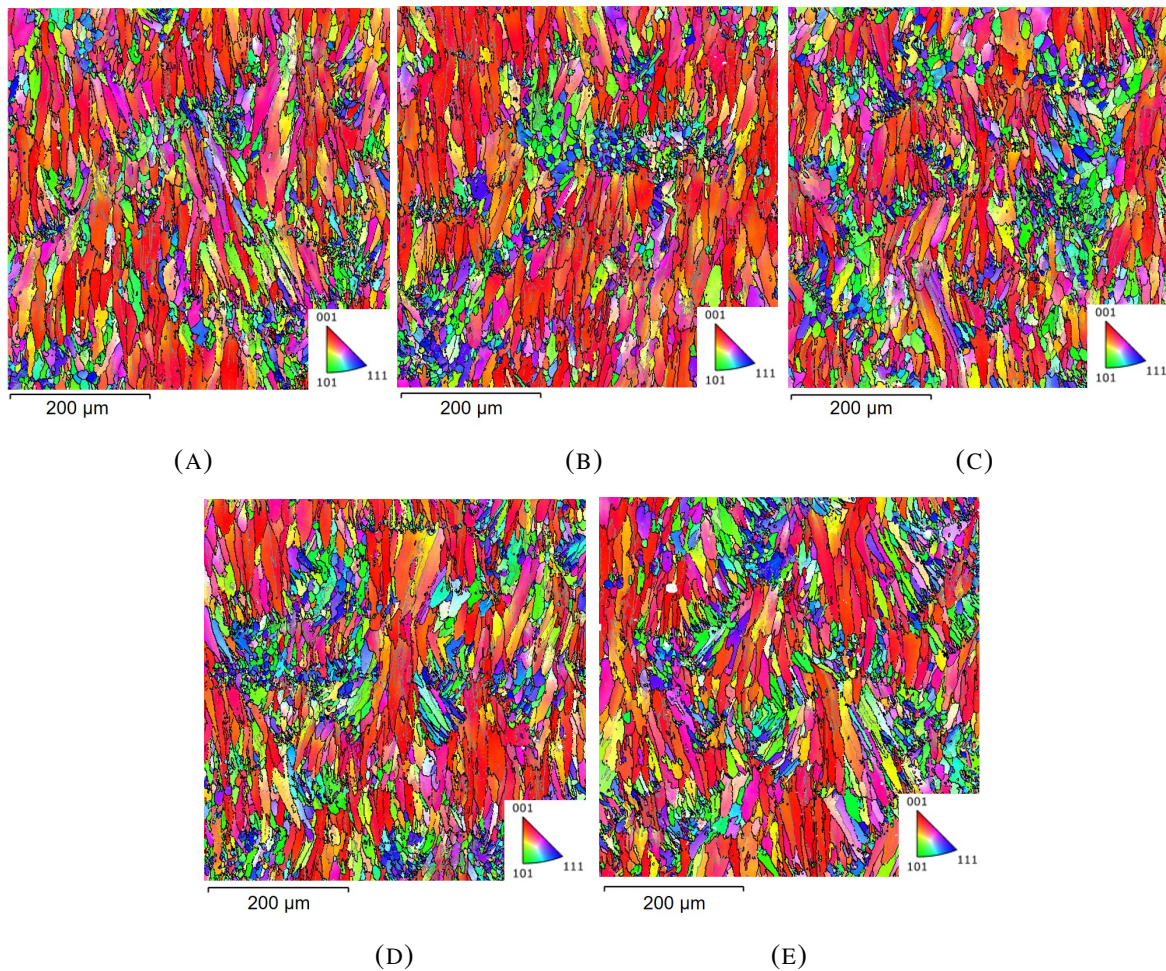


FIGURE 7.17. IPF maps of the X-Z plane of samples manufactured with powders reused by the refreshing method: (a) R1, (b) R5, (c), R10, (d) R15, (e) R20. The build direction for each micrograph is vertically upwards.

TABLE 7.1. Grain size and maximum intensity for the pole figures representing the crystallographic texture of parts manufactured with powders reused by the refreshing method.

Reuse Run	Grain Size (μm)	Maximum Pole Figure Intensity
R1	9.26 ± 7.89	4.45
R5	9.68 ± 8.54	4.77
R10	9.24 ± 7.06	3.76
R15	8.82 ± 6.99	3.73
R20	9.38 ± 7.74	3.62

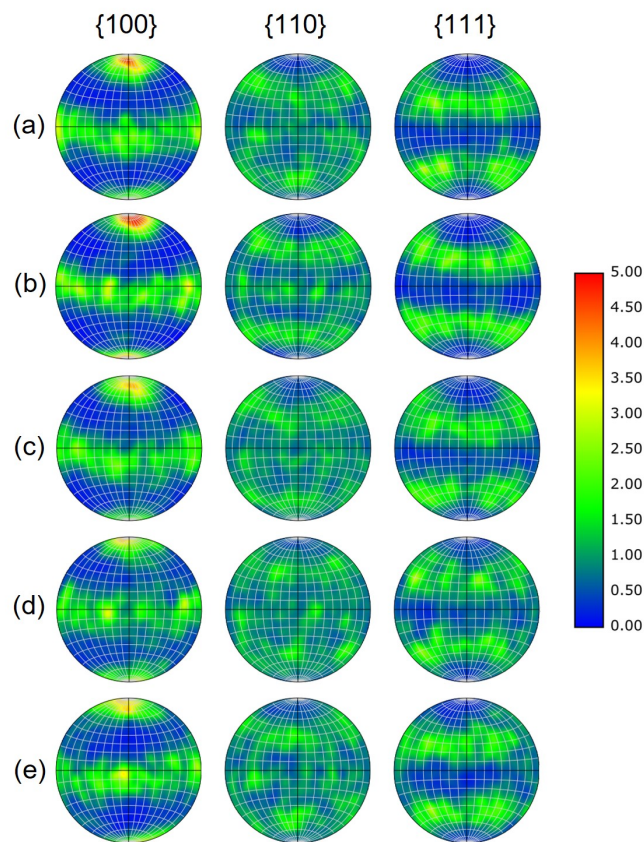


FIGURE 7.18. Pole figures of the X-Z plane of samples manufactured with powder at different stages of reuse: (a) R1, (b) R5, (c), R10, (d) R15, (e) R20.

7.3.2.3 Cell Structure

Within each of the grains is a network of silicon particles forming a cell network. These cells can be either elongated or equiaxed depending on their location within the melt pool, with this network coarsening as the heat effected zone of the melt pool is reached. SEM micrographs of this cell network are shown in Figure 7.19, with both equiaxed and elongated cells shown

for each powder reuse stage. There is no significant evolution in the cell network as a function of powder reuse.

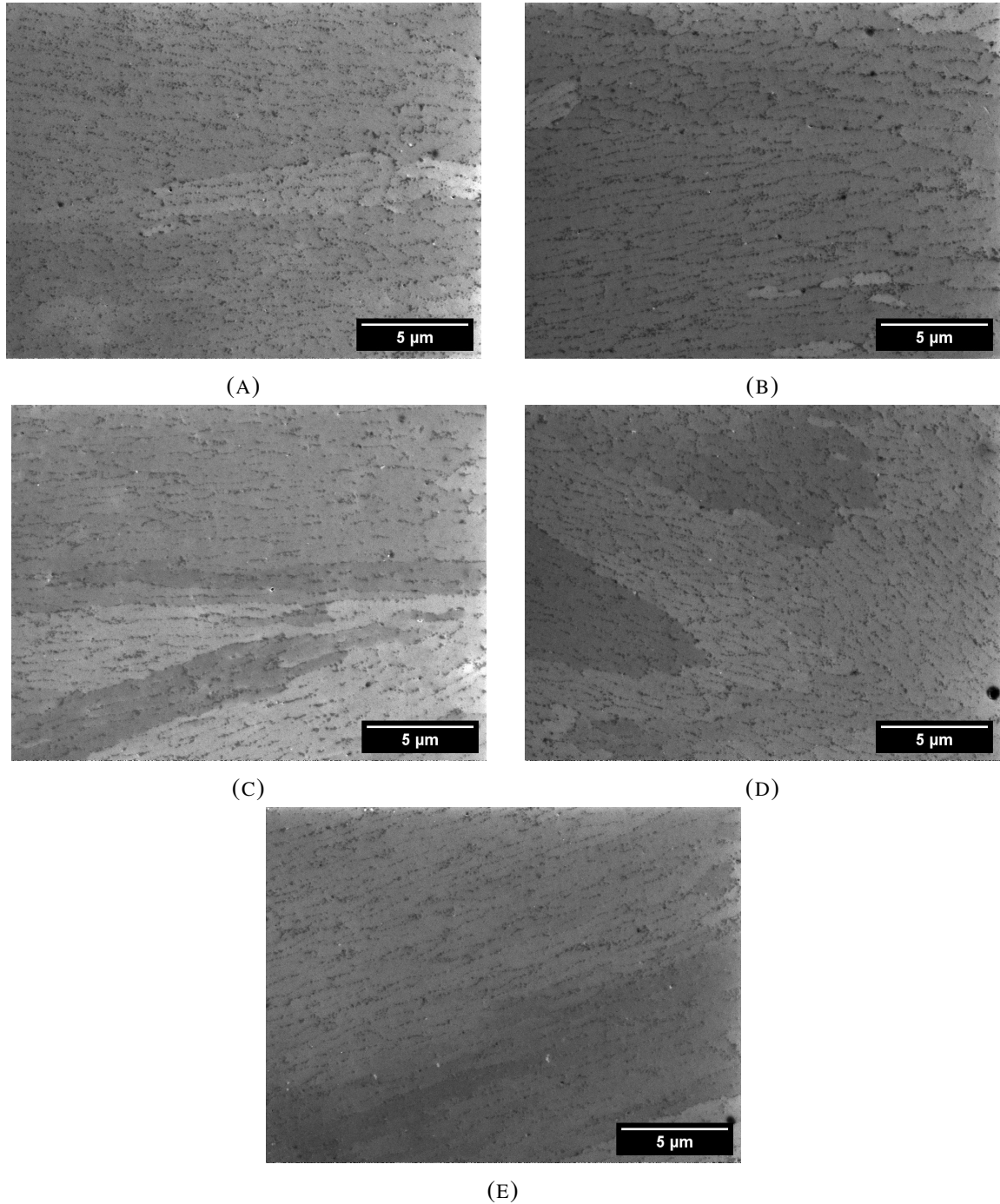


FIGURE 7.19. SEM micrographs of samples manufactured with powders reused by the refreshing method, showing the silicon cell structure within the grains of parts: (a) R1, (b) R5, (c), R10, (d) R15, (e) R20.

7.3.3 Part Properties

7.3.3.1 Part Density

The density of parts shows a slight decrease as powder is reused, as seen in Figure 7.20. The most significant drop is seen in Archimedes testing between R1 and R2, where the relative density decreases from 99.41 % to 99.15 %. The relative density then stabilises with any changes attributable to the error inherent to the manually measured Archimedes process. Micro-computed tomography shows similar results, albeit at a higher resolution than that of Archimedes testing. It shows a gradual decrease from a relative density of 99.98 % to 99.88 % after 20 manufacturing runs, with this decrease in density relatively consistent. Together, these indicate the density of these samples are reducing when manufactured with reused powder, however, it should be noted that these densities measured by μ -CT always remain higher than the powder supplier's specification of 99.6 %.

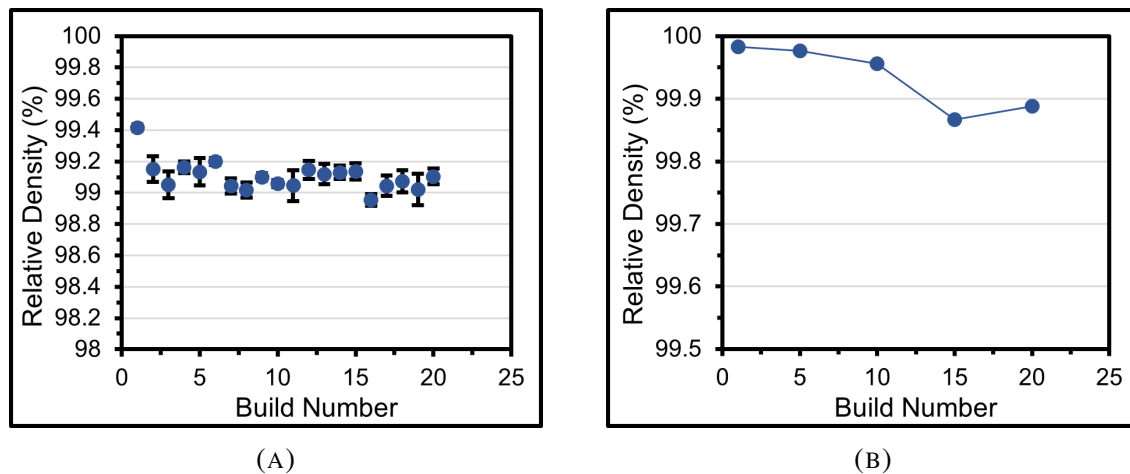


FIGURE 7.20. Relative density of AlSi7Mg when manufactured with powder reused by the refreshing method as measured by: (a) the Archimedes principle, (b) micro-computed tomography.

7.3.3.2 Surface Roughness

The surface roughness of parts, measured from a number of planes as described in Chapter 4, is displayed in Figures 7.21 and 7.22. The surface roughness of parts shows no relation to powder history, with no change outside the standard deviations evident in any location. The exception to this is the sloped surfaces in R11, where build interruptions increased the

surface roughness. These occurred due to faults with the Concept Laser M2 where software issues caused the manufacturing run to pause for short intervals before continuing, producing visible lines on the surfaces of parts resulting from changed solidification behaviour due to the increased time between layer deposition. These did not affect the surfaces in Figure 7.21 significantly as the majority of interruptions occurred once the parts for these measurements were already built.

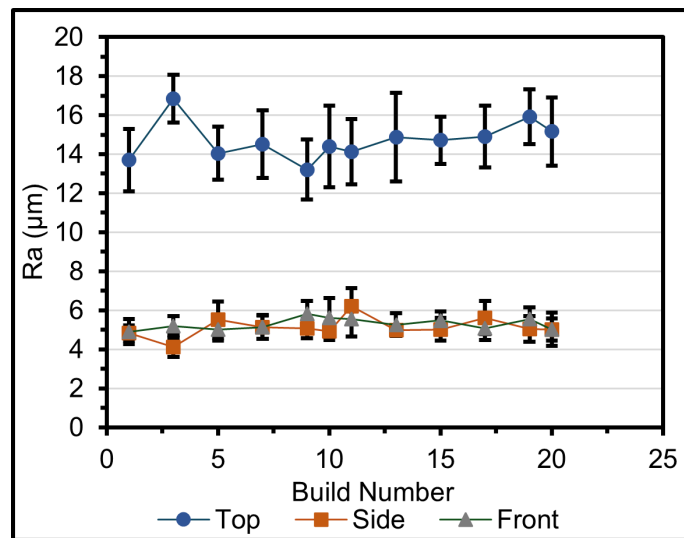


FIGURE 7.21. Surface roughness of planes measured from surface roughness cubes.

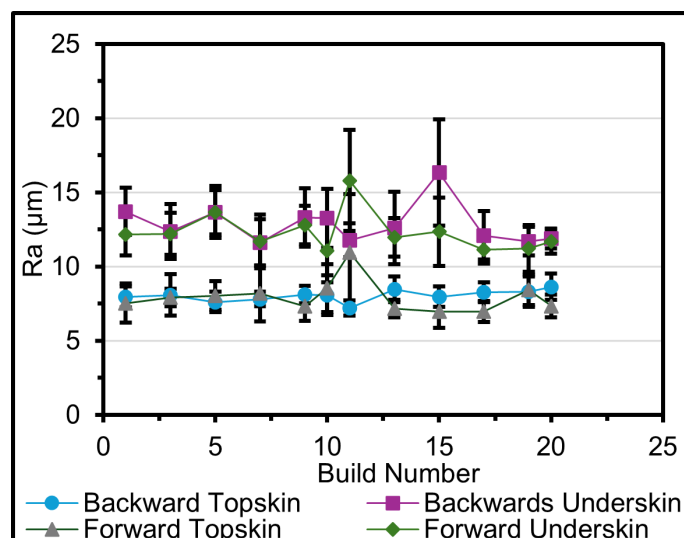


FIGURE 7.22. Surface roughness of planes measured from sloped blocks.

The location of testing, however, is seen to affect the surface roughness of parts. The top surfaces of parts are rougher than the front and sides, which are the smoothest of all surfaces measured, while also having greater variability. Part under skins are also consistently rougher than part top skins, again with greater variability.

7.3.3.3 Part Chemistry

The bulk chemistry of parts as a function of reuse is shown in Figure 7.23, with the main alloying elements represented and aluminium comprising the remainder of the composition. There is a slight decrease in the amount of silicon between R10 and R15, however, since the silicon composition returns to a value similar to the starting composition, this is likely a result of random sampling. Magnesium, however, reduces and then remains at lower levels than the first few manufacturing runs, suggesting its concentration in parts decreases with powder reuse. The most noteworthy change in composition is the increase in oxygen content, which gradually rises from an initial value of 0.015 wt.% to a final value of 0.048 wt.%, a threefold increase. The consistency of this trend suggests that as powder is reused the oxygen content of parts will consistently increase, potentially providing an issue for later parts if powder is reused beyond 20 manufacturing runs.

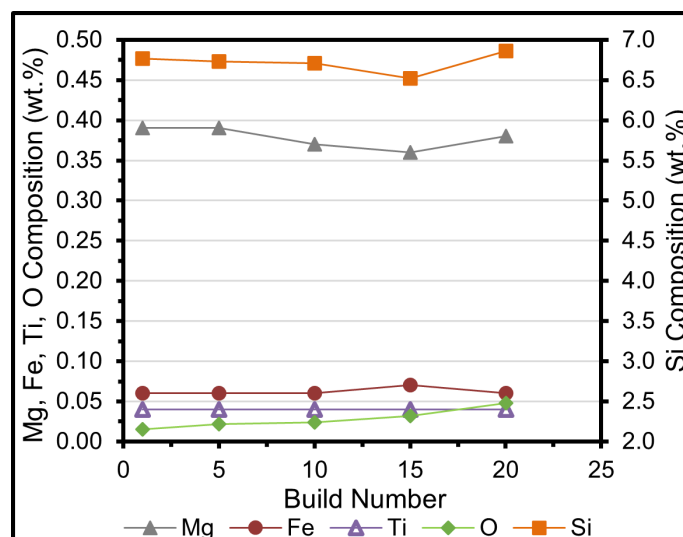


FIGURE 7.23. Bulk chemistry of specimens manufactured with powder reused by the refreshing method.

7.3.4 Mechanical Properties

7.3.4.1 Hardness

The hardness of stress relieved samples manufactured with reused powders is shown in Figure 7.24. An ANOVA analysis, displayed in Appendix B1.4, shows that there is a statistical difference from R1 to R7, whereupon there is no further change. Figure 7.24 shows that this involves a slight increase in hardness from 75.67 HV at R1 to 80.22 HV at R7, whereupon the hardness values steady. This suggests that after the first few manufacturing runs, the hardness of AlSi7Mg stabilises and will thereafter remain consistent.

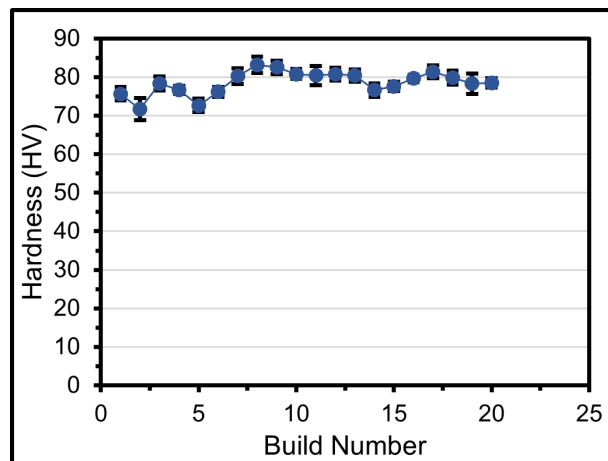


FIGURE 7.24. Hardness of specimens manufactured with powder reused by the refreshing method.

7.3.4.2 Tensile Properties

Tensile samples were tested using both small and large samples, with these sample dimensions shown in Figure 7.25. Results for the large coupons tested are shown in Figure 7.26. It is clear that the vertically oriented samples perform better than their horizontally oriented counterparts, with both strength and ductility being consistently greater for vertical samples, with further extrapolation on the reasons behind this found in Chapter 5. Also evident is a slight increase in both tensile and yield strengths from R1 to R10, however, these increases are small with the greatest increase being in the tensile strength of vertical samples which increase from 229.3 MPa to 242.3 MPa, a change of only 5.7 %. The increase in strength is

matched by a corresponding decrease in ductility. These trends are slightly stronger in the vertically oriented samples.

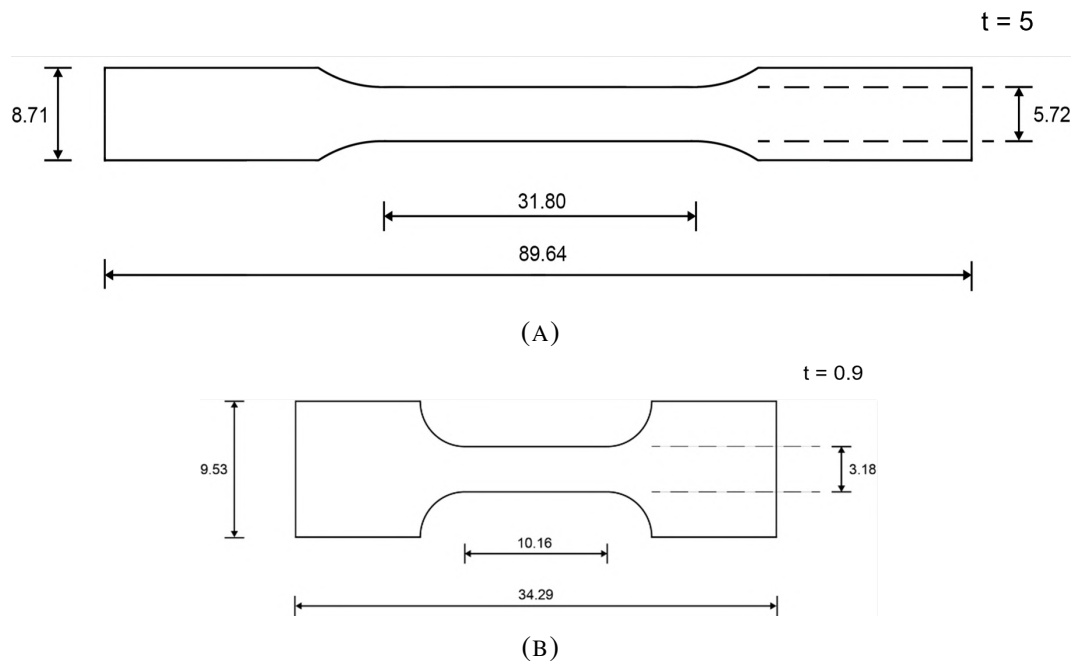


FIGURE 7.25. (a) Subsize specimen according to Figure 19 of ASTM E8E8M – 24, (b) Customised miniature tensile specimen dimensions. All dimensions are in mm.

Smaller tensile samples were also tested in a variety of states to compare the effects of heat treatment, part surface finish, and part orientation on properties while using reused powders. The results of this are shown in Figures 7.27, 7.28 and 7.29. The yield strength shows a clear reliance on a part's manufacturing history, with each treatment case clearly distinct from the others. There is a slight increase in the yield strength of stress relieved parts from R1 to R9, with this trend not present in the T6 treated samples. This increase is also followed by a decrease in yield strength from R11 onwards for the SR-M-V samples.

The tensile strength of parts shows less of a trend than their yield strength relative to manufacturing history, with the exception of the stress relieved, as-printed samples, which were consistently weaker than all other samples tested. The tensile strength of stress relieved samples showed some reliance on powder history, with the strength of SR-P-H samples increasing from 189.6 MPa to 200.4 MPa from R1 to R9, although this effect began to plateau once reaching R5. Similarly, the SR-P-V samples increased in strength from 209.4 MPa

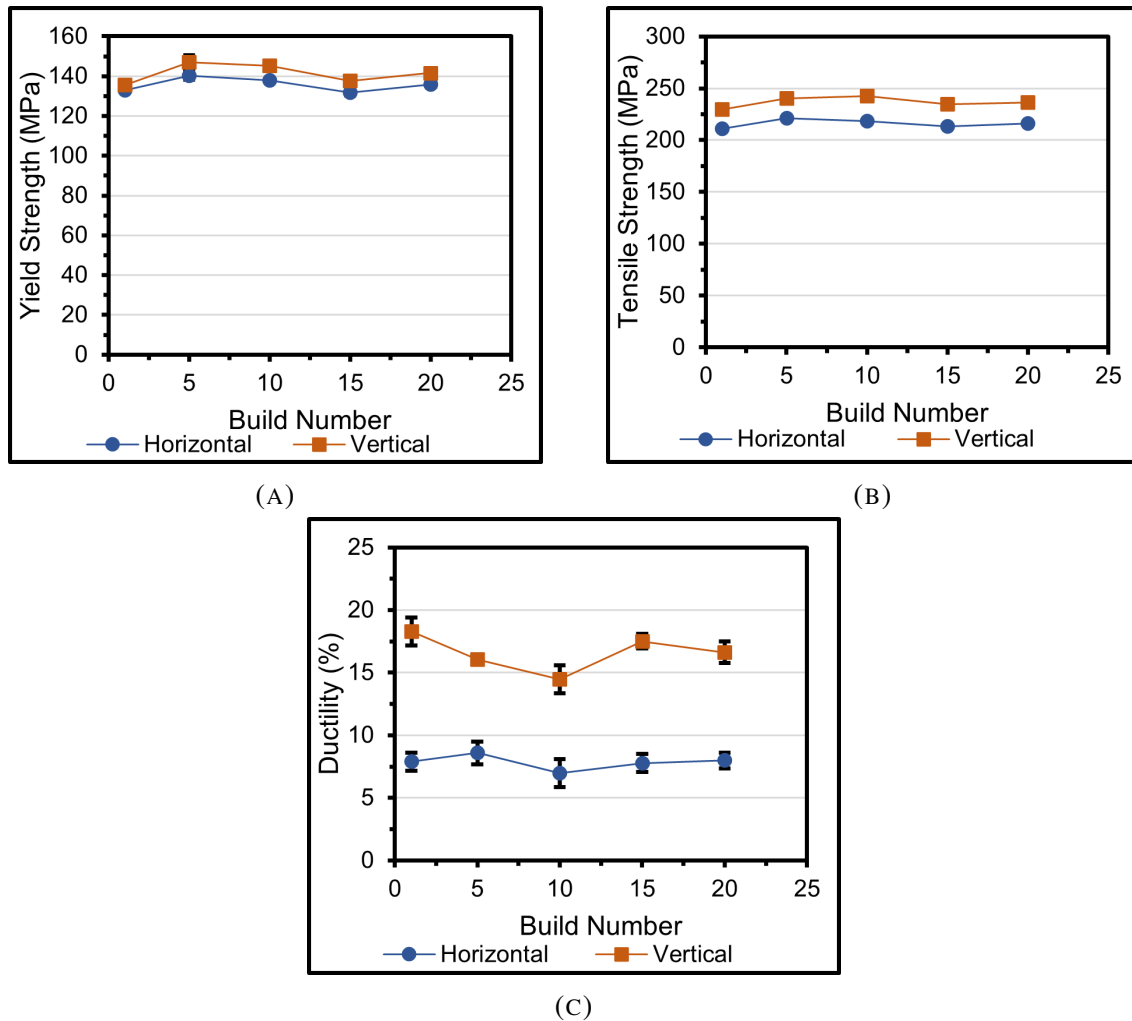


FIGURE 7.26. Tensile properties of parts manufactured with reused powder: (a) yield strength, (b) tensile strength, (c) ductility. These plots represent the results from large coupons. Error bars are present on each plot but too small to be seen in yield and tensile strength plots.

to 224.5 MPa from R1 to R5 before stabilising. T6 samples, however, demonstrated no relationship between tensile strength and the number of powder reuse cycles.

The ductility of parts manufactured with reused powder shows considerable scatter, as evident in Figure 7.29. Horizontally printed samples consistently show the poorest ductility regardless of heat treatment state, while also having the largest standard deviations. No discernible trend can be seen in this data to relate powder reuse to part ductility.

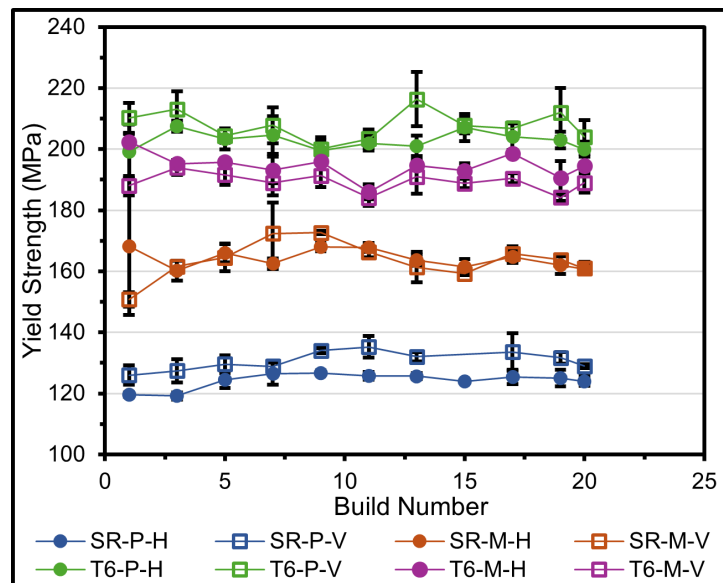


FIGURE 7.27. Yield strength of mini-tension specimens manufactured with powder reused by the refreshing method. Nomenclature is as follows; SR – stress relieved; T6 – T6 heat treated; P – printed to shape; M – machined to shape; H – horizontally oriented; V – vertically oriented.

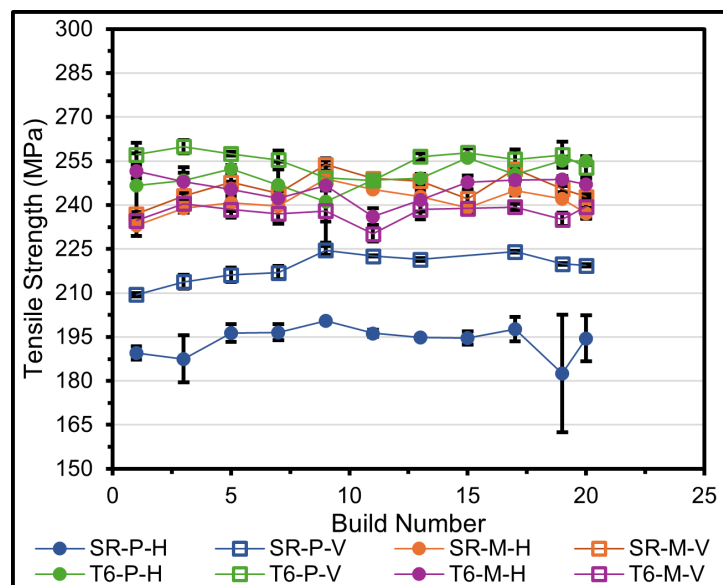


FIGURE 7.28. Tensile strength of mini-tension specimens manufactured with powder reused by the refreshing method. Nomenclature is as follows; SR – stress relieved; T6 – T6 heat treated; P – printed to shape; M – machined to shape; H – horizontally oriented; V – vertically oriented.

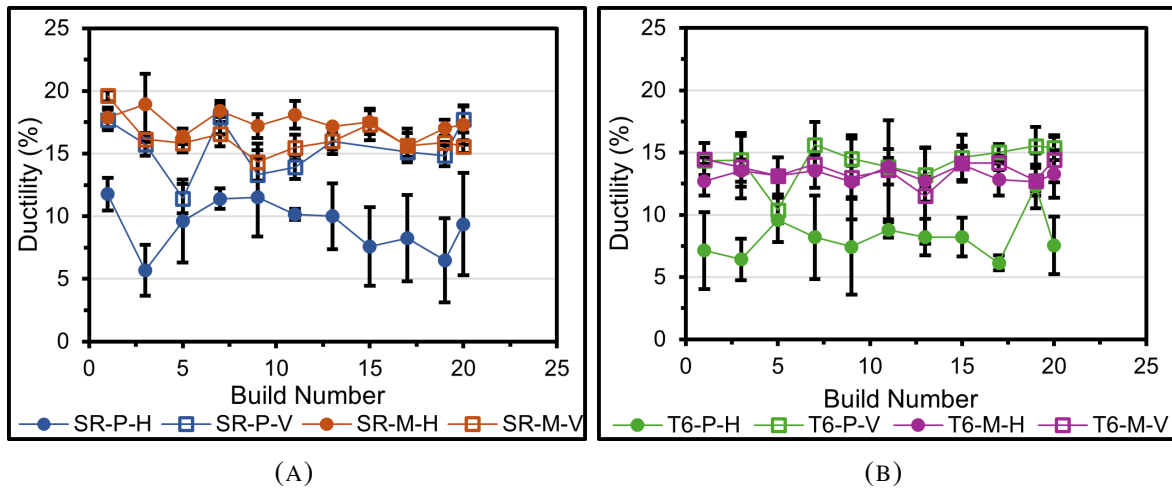


FIGURE 7.29. Ductility of mini-tension specimens manufactured with powder reused by the refreshing method. Nomenclature is as follows; SR – stress relieved; T6 – T6 heat treated; P – printed to shape; M – machined to shape; H – horizontally oriented; V – vertically oriented.

7.3.4.3 Flexural Properties

The flexural properties of specimens printed to shape are displayed in Figure 7.30. These most noticeably show a dependence on the heat-treated state of the specimens with T6 samples having higher yield and flexural strengths than the stress relieved samples, while also having lower ductility. T6 treated horizontal samples are also stronger and less ductile than their vertically oriented counterparts, while stress relieved samples do not demonstrate this anisotropy. These samples also show remarkably consistent properties, with the standard deviations for each tested case typically being within 0% - 2%.

Powder reuse has mixed effects on the flexural properties of AlSi7Mg. Horizontally oriented stress relieved samples show no change in properties with powder reuse, while vertically oriented parts demonstrate a minimal increase in strength from R1 to R5 before stabilising. T6 treated vertical samples show no change with powder reuse, however, horizontally oriented samples show a slight reduction in strength from R1 to R5, before again stabilising.

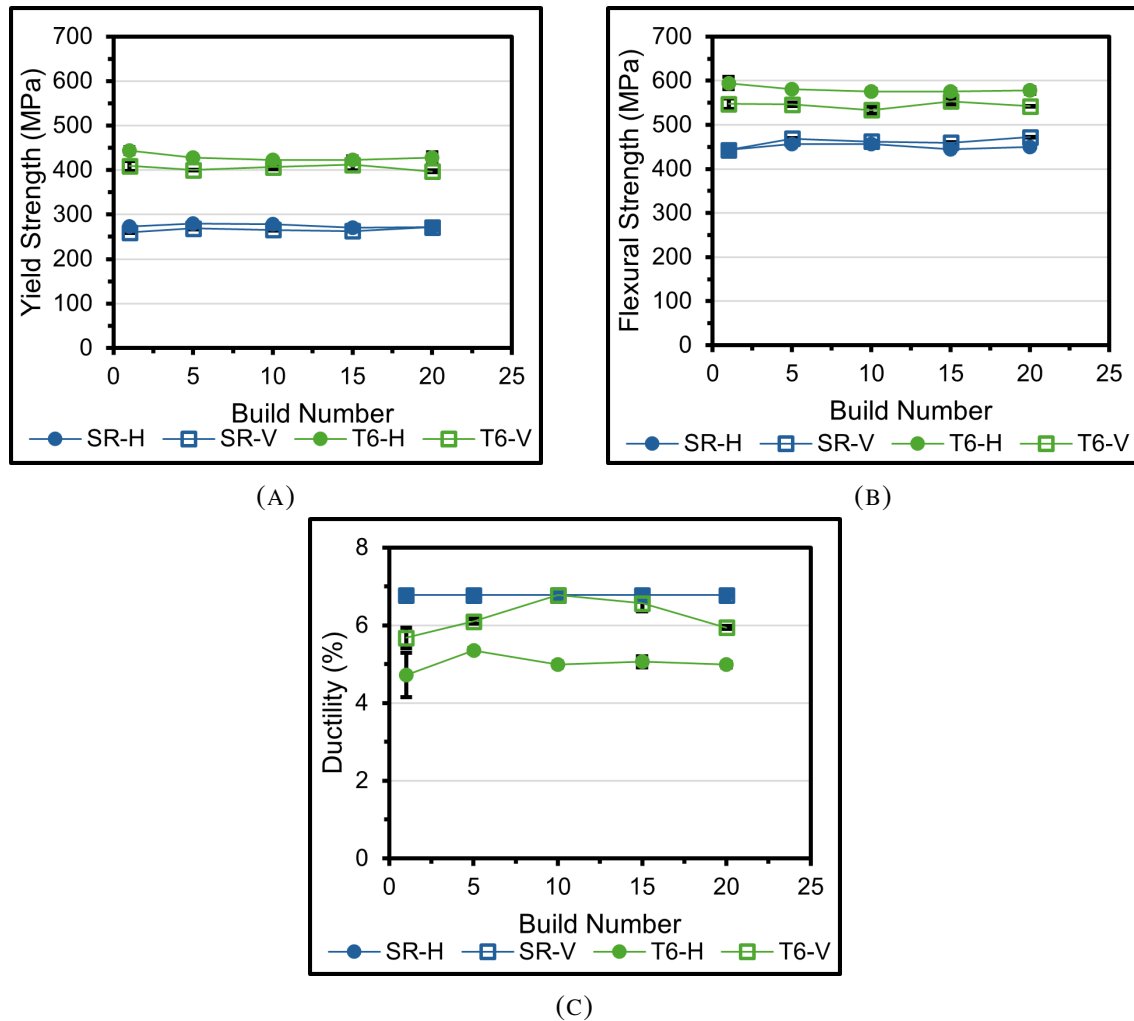


FIGURE 7.30. Flexural properties of parts manufactured with reused powder: (a) yield strength, (b) flexural strength, (c) ductility. Nomenclature is as follows; SR – stress relieved; T6 – T6 heat treated; H – horizontally oriented; V – vertically oriented.

7.3.4.4 Impact Toughness

The impact toughness of samples both as-printed and machined to shape were tested, with these results shown in Figure 7.31. It is evident that horizontally oriented specimens are able to absorb more energy before failure, regardless of surface finish. While ANOVA analysis, included in Appendix B1.5, indicates that differences between specimens tested are statistically significant for all cases but the horizontal, machined samples, there is no relation between powder reuse and impact toughness with the exception of as-printed, horizontally oriented parts. These show a gradual decline in impact toughness as powder is reused and

while other cases show evidence of slight declines, these changes are at a smaller scale and within error measurements.

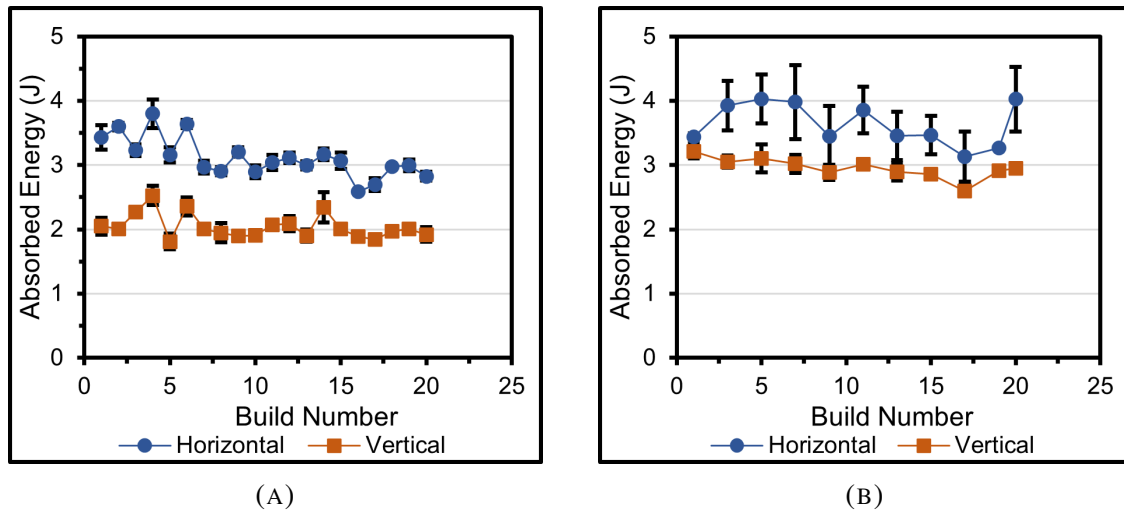


FIGURE 7.31. Impact toughness of parts manufactured with powder reused by the refreshing method: (a) impact toughness of as-printed samples, (b) impact toughness of samples machined to shape.

7.3.4.5 Fatigue Strength

The SN curves of R1 and R20 samples were tested, with samples determined to have reached their fatigue strengths when they withstood one million cycles. These curves are shown in Figure 7.32. While the behaviour of the samples at low and high loads is similar, with samples tested at 300 MPa and 150 MPa showing minimal difference in cycles withstood, it is evident that there is a difference in the fatigue behaviour at middling loads. At 250 MPa these differences are obscured by the deviations between measurements, with these deviations intersecting and so indicating there is minimal difference in fatigue life at this load. At a load of 200 MPa, however, the R20 sample displays a significantly lower fatigue resistance of 298,000 cycles compared to the R1 samples which withstood one million cycles. This indicates that the fatigue life for the R1 samples lies between 200 MPa and 250 MPa, whereas for the R20 samples it lies between 150 MPa and 200 MPa, showing that powder reuse will reduce the fatigue limit of AlSi7Mg.

Fracture surfaces for the fatigue samples that failed are shown in Figure 7.33. These show that failure was initiated at sites of both inclusions and porosity, with the rest of the fracture

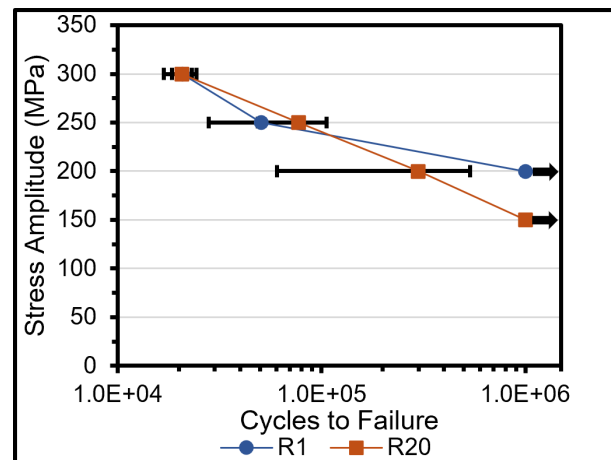


FIGURE 7.32. S-N curves of R1 and R20 samples. R1 samples tested at 150 MPa also reached one million cycles, where testing was halted, but are not shown in this figure for clarity.

surfaces being consistent in that melt tracks are clearly visible in each. The R1 samples in Figure 7.33b and 7.33d show minimal evidence of large pores, with failure initiating from small pores and inclusions. This is in comparison to the R20 samples in Figure 7.33f, 7.33h, and 7.33i, where pores are larger and are likely lack of fusion (LOF) pores given the presence of small particles inside these pores which are likely unmelted powder particles.

Analysis of the inclusions present at some of these fracture surfaces was conducted using SEM-EDS, with these results being shown in Figure 7.34. Figure 7.34a shows that failure of the R1 sample loaded at 250 MPa began near two precipitates: a silicon-oxide and a titanium rich precipitate. The closer proximity of the titanium precipitate to the surface suggests that this played a greater role in initiating fracture. Figure 7.34b displays the fracture surface of the R20 sample loaded at 200 MPa, with the lack of aluminium, magnesium, and silicon within a large area suggesting a LOF defect. This surrounds a silicon particle approximately 20 μm wide that penetrates this defect. This particle is larger than silicon precipitates found in heat treated states as per Chapter 6, meaning that it is unlikely to be a result of in-situ precipitation. This is aided by the fact that this inclusion sits in a LOF defect, meaning melting was not able to properly occur in this region, indicating that this inclusion is not a result of the melting and heating process. This suggests this particle is likely a foreign inclusion in the powder mix, potentially from the recoater used during the powder spreading process.

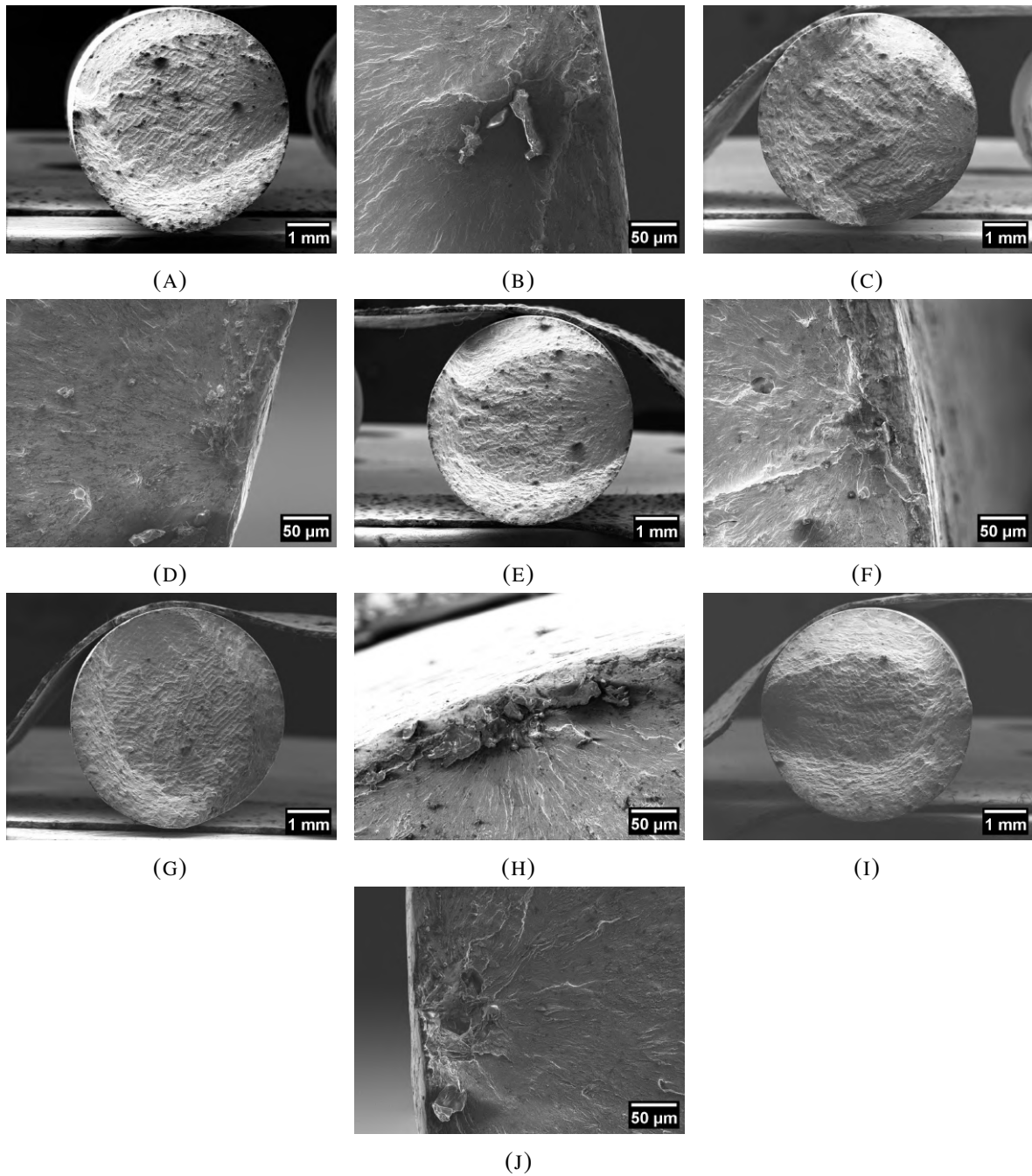


FIGURE 7.33. Fracture surfaces of R1 and R20 fatigue samples: (a) R1, 250 MPa large field of view (FOV), (b) R1, 250 MPa small FOV, (c) R1, 300 MPa large FOV, (d) R1, 300 MPa small FOV, (e) R20, 200 MPa large FOV, (f) R20, 200 MPa small FOV, (g) R20, 250 MPa large FOV, (h) R20, 250 MPa small FOV, (i) R20, 300 MPa large FOV, (j), R20 300 MPa small FOV.

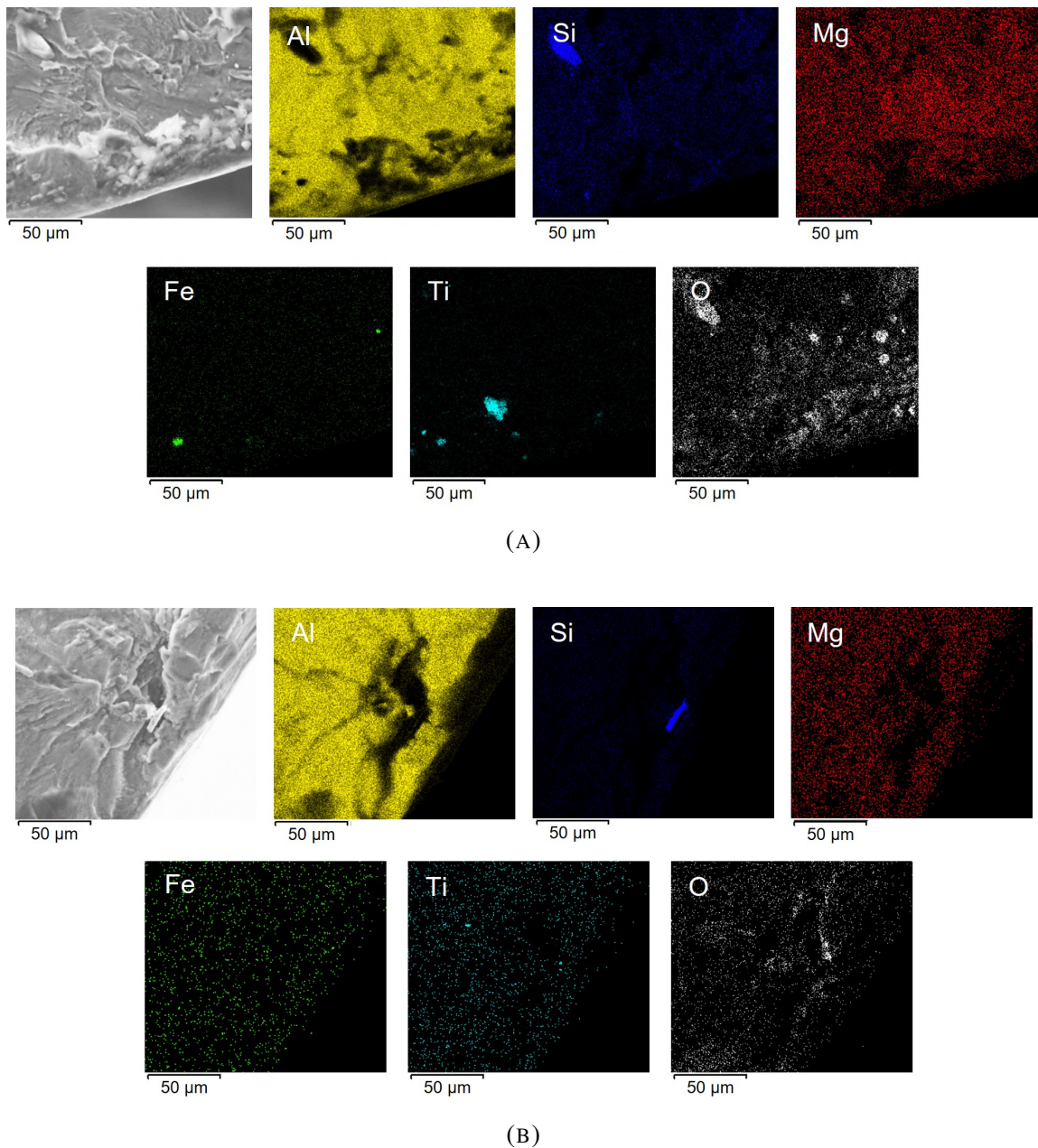


FIGURE 7.34. EDS analysis of fatigue fracture surfaces: (a) R1 – 250 MPa, (b) R20 – 200 MPa.

7.4 Discussion

7.4.1 Powder Characterisation

Powder characterisation encompasses the investigation of different types of powder features, with changes in these features able to provide insight into other aspects of the PBF process

as well as the performance of manufactured parts. In this research, powder characterisation comprises of powder morphology and flow, and powder chemistry.

7.4.1.1 Powder Morphology and Flow

Qualitatively, the powder batches are consistently comprised of highly circular particles of similar size which remain consistent with powder reuse. There is, however, a slight increase in the presence of both large and agglomerate particles as the number of reuses increases. Larger particles are spatter particles, formed by ejecta from the melt pool rapidly solidifying in the chamber atmosphere to form new particles that are often highly spherical and larger than the average particle formed by the gas atomisation process [312], with an example of this shown in Figure 7.35a. However, this spatter is also not always spherical, as in Figure 7.35b. As molten material is ejected from the melt pool, it is often accompanied by solid powder from the powder bed that gets entrained in the vapour plume as a result of gas flow and melt pool instabilities, as well as smaller, quickly solidifying melt pool ejecta. The collision of these solid particles with the still molten ejecta in the build chamber atmosphere results in the creation of new agglomerate particles, either in the form of multiple particles sintered together, or of solid particles joined by highly irregularly shaped material. These processes form new agglomerates that are both non-circular and larger than gas atomised particles, changing the composition of the powder batch. Examples of these powder types are displayed in Figure 7.35c and 7.35d.

The increased presence of these particles can be seen to influence the PSD of the powder. From R1 to R7 the D10, D50, and D90 values all increase, with this effect most pronounced in the D90 size. The fact that this effect is strongest amongst the largest particles suggests an increase in large particles rather than a decrease in small particles, consistent with the qualitative results from SEM imaging. This trend also flatlines after R7 once the D90 value reaches 61.4 μm , implying that the 63 μm sieve used between each manufacturing run is limiting the increased presence of large particles regardless of the rate they are being produced. The increase in large particles also results in a widening and slightly skewed PSD, as shown in Figure 7.5. As powder is reused the peak of the PSD lowers, pointing to a widening distribution. The left tail of this distribution is kept relatively constant by the continual introduction of new powder due to the use of the refreshing powder reuse method, which

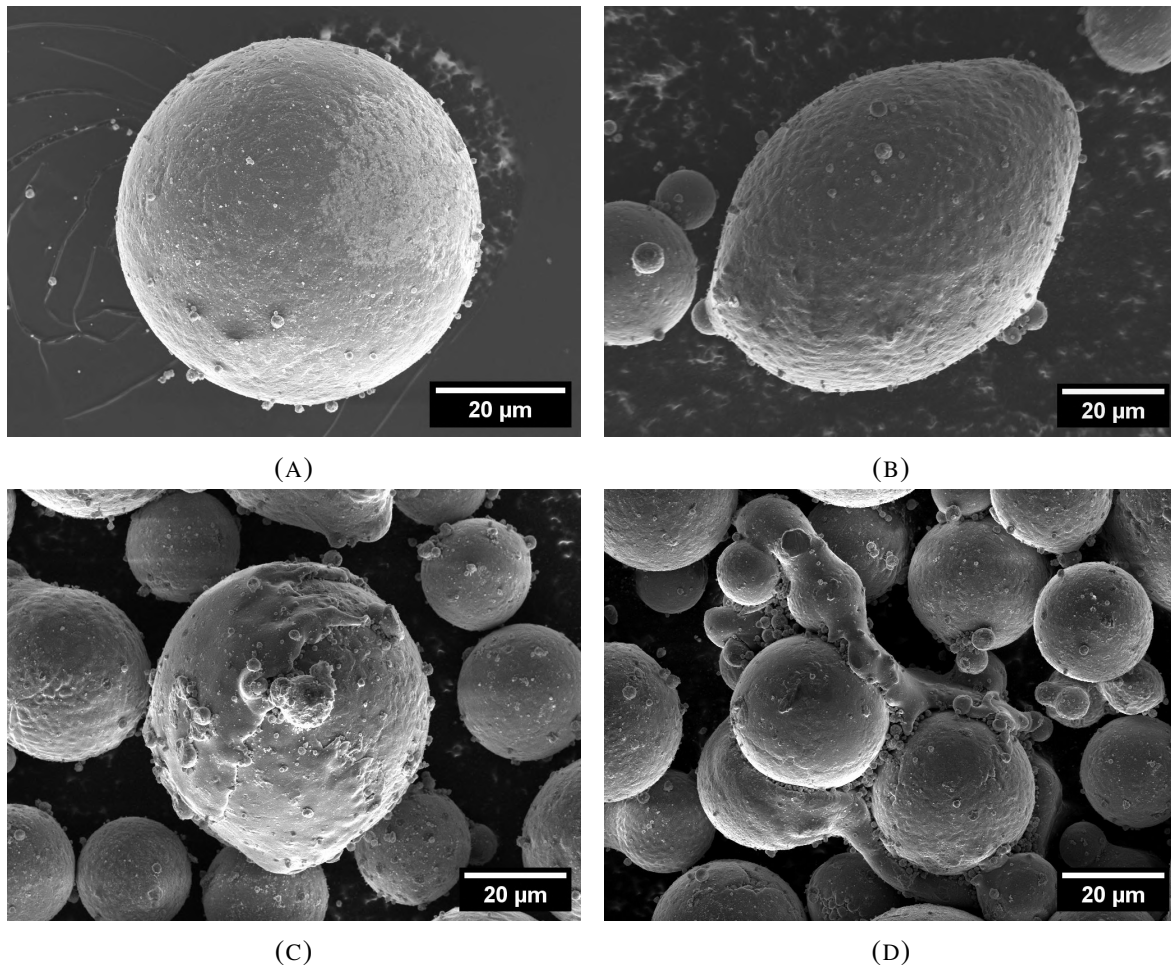


FIGURE 7.35. Examples of different powder types present in the powder batch: (a) highly spherical particle from the gas atomisation process, (b) large spatter particle with low sphericity, (c) spatter particle formed by the collision of a solidified particle with molten ejecta in the build chamber atmosphere that creates a coating over half of the particle, (d) large agglomerate formed by the collision of multiple particles with ejecta within the build chamber atmosphere.

is able to replace the fine powder particles consumed in the PBF process. The right tail of this distribution, however, continues to increase with powder reuse due to the accumulation of spatter and agglomerate particles. The fact that this widening continues even after the D10, D50, and D90 values have stabilised after R7 suggests that large particles are still accumulating, albeit at a slower rate than previously due to sieving. This is likely due to the increased presence of large particles between the D50 and D90 sizes which are less affected by sieving.

This change in the PSD is seen to influence the flow of the powder, most notably in the traditional Hall and Carney flow tests. Both see a reduction in flow times over the first four manufacturing runs, decreasing between 20% – 30% which indicates a significant improvement in flowability. After this initial improvement the flow times stabilise. The earlier stabilisation of powder flowability by the Hall and Carney flow when compared to PSD values suggests that this property is less reliant on larger particles and more so on the PSD as a whole. As the D10 and D50 values stabilise at R5, this implies that once these changes occur, powder flowability becomes more stable. This is as finer particles have a greater influence on the flowability of a powder batch, with fine particles being seen to have poorer flowability than coarse ones [58, 80]. The constant fraction of fine particles after R5 therefore limits any further changes to powder flowability as a result of the changing presence of larger particles.

Flowability measures by the rotating drum technique indicate a slight change in behaviour due to powder reuse, with powder reuse improving flowability. This is firstly shown by the trend for the velocity angles, which is similar to those for the Hall and Carney flow times where powder flow improves by R5 but then stabilises. This is indicated by the consistently higher velocity angles of unused powders at rotational speeds up to 14 rpm, with a higher angle related to poorer flow. According to these tests powder reuse beyond R5 shows minimal change in flow behaviour, with each powder batch demonstrating similar flowability. The cohesion of powder, however, exhibits a more consistent relationship between powder reuse and flowability. At lower rotational speeds the cohesion of powder continually decreases with powder reuse, indicative of better flowability as the powder is more able to move over itself as it rotates. Examples of these powder flow states are demonstrated in Figure 7.36, where images captured by the Granudrum are displayed for poor and good quality powder flow. These velocity angle and cohesion results indicate that the increased presence of larger particles is improving the flowability of the powder, with the reduction of fine particles supporting this change. The increased presence of non-circular particles from accumulating spatter can be seen to not have a significant effect, as these particles increase cohesion and reduce flowability due to their irregular morphology which is a trend not observed in the flowability results. However, at higher rotational speeds the cohesion of the powder increases. The powder therefore demonstrates shear thickening behaviour, whereby at increased rotational speeds the flowability of the powder reduces. This behaviour occurs for powder of all ages; however, the effect increases in reused powder as evidenced by the increasing cohesion gradients. This is

also evident in the velocity angle results, although to a lesser degree. In PBF applications, this means that the recoater velocity must be carefully managed to ensure that powder continues to spread correctly, with slower speeds producing better results. This especially becomes a consideration when using reused powders, where higher recoater speeds may result in increasingly poor recoating. To make use of this, however, a correlation between recoater speed and rotational velocity must first be established.

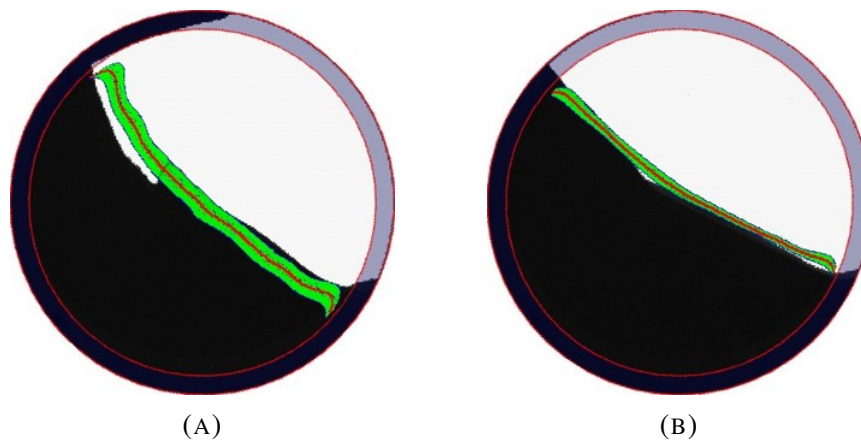


FIGURE 7.36. Examples of powder flow behaviour within the Granudrum at different reuse stages: (a) a powder exhibiting poor flowability due to the high velocity angle and high cohesion, (b) a powder showing good flowability due to the low velocity angle and low cohesion. The average powder surface is shown by the red line, with the velocity angle measured from the gradient of this line. The green region represents the average maximum and average minimum of the powder surface, with a large green area representing a large cohesion value and therefore poor flowability.

The goal of using highly flowable powders in PBF is to ensure good spreading in the powder chamber by creating an even layer with no patches marred by poor coverage. Measurements of powder apparent density and powder bed density seek to quantify this. Powder apparent density showed relatively consistent results as a function of powder reuse. Powders from R1 – R4 showed a gradual decrease in apparent density, with an ANOVA analysis confirming the statistical validity of this observation. Even then, the decrease in apparent density is slight and so likely has minimal impact of powder spreading within the build chamber. Powder bed density, as measured in the build chamber, also shows no trend in relation to powder reuse, with densities staying constant at approximately 1.57 g/mm^3 , although the large deviations may obscure any trends present due to the reuse of powder. This indicates that even though

powder characteristics are changing with powder reuse, they are not significantly influencing powder spreading within the build chamber.

These results show that there is a change to powder as it undergoes a refreshing powder reuse regime. The generation of spatter and agglomerate particles due to sintering and ejecta particles in the build chamber result in an increased presence of larger particles in the powder batch, which results in a widening PSD. This in turn improves the flowability of the powder, although this has no significant effect on the spread of powder in the build chamber. These changes are most prevalent in the first nine manufacturing runs, which suggests that the continual refreshing of the powder eventually homogenises the powder batch to a significant enough degree that any further changes in powder morphological characteristics become negligible.

7.4.1.2 Powder Chemistry

The chemistry of powders stays relatively consistent as the number of reuses increases, with this being the case for both surface and bulk chemistry. For the top ~ 10 nm of the powder surface, as measured by XPS, the chemistry is dominated by alumina which is expected given the rapid propensity for aluminium to oxidise. Alumina forms 70 wt.% - 80 wt.% of the surface chemistry, with the next most dominant compound being silicon oxide, which fluctuates between 6.6 wt.% - 16 wt.%. The magnesium concentration at the surface is also considerably higher than that of the nominal alloy chemistry, as shown in Figure 7.37. The surface chemistry is consistent and shows no change in relation to powder reuse, likely due to the already heavily oxidised surface of the unused powder inhibiting any further chemical change. Given these oxides have considerably higher melting temperatures than their elemental constituents, their presence to any significant depth may negatively impact the meltability of the powder. However, given the adequate values of density, surface roughness, and mechanical properties seen in this research, this is likely not a significant issue for powder reused up to twenty times with the refreshing method.

The bulk chemistry of the powder also shows minimal relation to powder reuse state, with the relative presence of most alloying elements remaining consistent throughout the powder reuse study. This is most importantly true for the main alloying element silicon, the concentration of

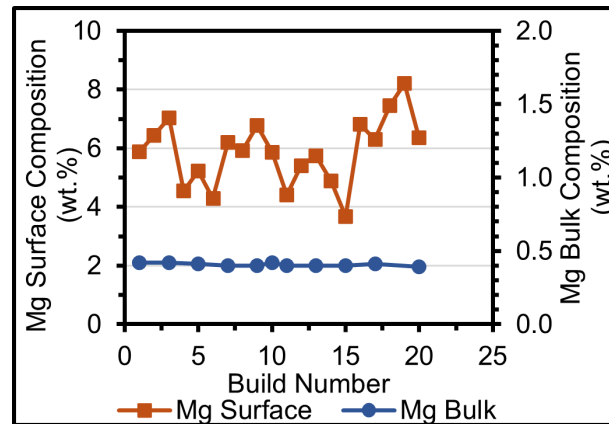


FIGURE 7.37. Comparison of the magnesium content in the bulk powder, measured by ICP-AES, and on the surface of powder, measured by XPS.

which fluctuates between 6.71 wt.% and 6.84 wt.%, with changes within this range showing no trend reliant on powder reuse.

A slight reduction in the magnesium content is seen, with a maximum presence of 0.42 wt.% for R1 and a minimum of 0.39 wt.% for R20, demonstrating a small loss. This slight reduction is due to the low evaporation temperature of magnesium compared to aluminium, meaning it is preferentially evaporated during processing and, as a consequence, new spatter particles have a lower magnesium content than unprocessed powders. This gradually reduces the magnesium content of the powder batch as it is constantly reused, although the limited decline indicates that this change is slow. While this decline is gradual, the already low magnesium content means that the powder begins to fall out of its chemical specification in later reuse runs, with a minimum of 0.4 wt.% magnesium required for this alloy class. This means that the magnesium content of powder needs to be carefully monitored during extensive reuse, where this concentration has the potential to even further reduce.

The presence of oxygen in the bulk composition of the powder also changes with powder reuse, decreasing from a concentration at R1 of 0.11 wt.% to 0.02 wt.% at R7, before once again stabilising. This is in stark contrast to the majority of powder reuse literature, where oxygen pickup is noticed especially in aluminium based alloys [166, 181, 226]. This decrease in powder oxygen content is likely due to the storage time between powder collection and chemical testing for the early manufacturing runs (for this project, samples of powder were collected at the beginning of each manufacturing run and kept in sealed plastic containers;

all the samples were sent at the same time for chemical analysis after the 20 manufacturing runs had been performed). Long term storage allowed for increased pick up of oxygen through both oxide growth and moisture adsorption, with this especially significant for early manufacturing runs given their increased storage time before testing. However, the reduction may also have been due to lowering powder moisture due to the use of a heated build plate. XPS shows no change in powder oxygen content and therefore no change in oxide growth, meaning moisture is a more likely contributor to the high oxygen content, especially given aluminium's propensity to adsorb moisture [313]. Long term storage of powder prior to the first manufacturing run likely results in a level of moisture higher than that which is ideal for processing, potentially contributing to the initially high level of oxygen measured in the powder. The use of a heated build plate during manufacturing runs that lasted 3.2 days resulted in build chamber temperatures that regularly exceeded 100 °C, allowing for the evaporation of any moisture contained in the powder and therefore a reduction in the associated oxygen content. This reduction in moisture would also improve powder flowability, with higher powder moisture content resulting in decreased flowability [314, 315], which was also observed in this research.

7.4.2 Microstructure

Dark field optical microscopy reveals the fish-scale melt pool structure characteristic of PBF manufactured parts, with this structure present in each of the micrographs taken for parts made with reused powder. The consistency of these melt pools between samples indicates that reused powder does not affect the melt pool structure, meaning that the melt pool core, melt pool boundary, and heat affected zones will also continue to be present within the microstructure.

The grain structure of AlSi7Mg parts also shows characteristics that are typical of additively manufactured parts. Columnar grains can be seen in the centre of each melt pool that grow in the direction of heat flow during solidification, i.e. in the build direction. This produces a textured [100] grain structure, with the majority of the grains possessing the same crystallographic orientation. This texture is confirmed by the pole figures. Surrounding these columnar grains are regions of fine, equiaxed grains. These form at the boundaries of melt pools where the dynamics of remelting and faster heat flow result in quicker solidification

than at the melt pool centres, therefore producing finer grains. The rapid cooling associated with the formation of these grains also reduces their textural uniformity, creating a region of randomly oriented texture. Due to the relatively low volume fraction of these grains, however, this does not play a significant role on the texture of the microstructure as a whole. As powder is reused, the shape, size, and texture of the grains do not change significantly. Since grain structure is dictated by cooling rates and heat flow, this indicates that powder reuse will not affect these factors.

One of the characteristic features of AlSi7Mg's microstructure is the presence of a fine network of silicon cells within the grains themselves, the result of rapid solidification and the subsequent segregation of silicon from the aluminium matrix. This silicon cell network is present in each of the samples observed in Figure 7.19, with the non-continuous nature of this network the result of the stress relieving treatment each sample underwent, the effects of which were extrapolated on in Chapter 6 of this thesis. These micrographs show the two main regions within the cellular network: the fine, elongated cells that form in the large, columnar grains; and the larger, equiaxed cells that form at the melt pool boundaries as a result of reheating from subsequent laser passes. The consistency of both the melt pool structure and the grain structure implies that this pattern of elongated and equiaxed cells continues throughout the microstructure of the PBF manufactured parts. Given these cells are consistent across each powder reuse iteration, it is evident that the refreshing powder reuse method has no influence on the solidification behaviour of AlSi7Mg and the subsequent development of the silicon cell network.

Together, these investigations reveal that the refreshing powder reuse method has no effect on the microstructure of AlSi7Mg. This indicates that the melt pool stability, cooling dynamics, and solidification behaviour all remain consistent as evidenced by the unchanging melt pool structure, grain size and shape, and cell structure. This shows that previous changes in powder characteristics such as chemistry and flow, and therefore potential packing and melting behaviour in the build chamber, do not have a significant enough effect to cause any changes to part microstructure. This shows great promise for the proper utilisation of the refreshing powder reuse method.

7.4.3 Part Properties

7.4.3.1 Part Density

The density of parts manufactured by PBF is one of the most widely investigated properties due to its relation to the proper manufacture of material [23]. Measurement of part density by the Archimedes method shows a decrease in relative density from R1 to R2, after which there is no further change. As the relative density at R1 was considerably higher than for any other sample, this may be a result of the manual process of measurement resulting in greater inaccuracy which is also evidenced by the lower relative density measured by the Archimedes method in comparison to μ -CT. This may also obscure any further trends in the density as a function of powder reuse.

Micro-computed tomography reveals a slight decline in part relative density as powder is reused, with this decrease accelerating in later stages of reuse. While the relative density stays at acceptable values, with 99.6 % being the manufacturer's specification and the minimum relative density reached being 99.87 % at R15, the regularity of the reduction in density suggests that this may cause issues in longer term powder reuse.

Part density has been tied to powder flowability, with improved flowability increasing density as this results in more even settlement in the powder chamber, creating a denser powder bed and therefore denser part [201]. Similar is true for the PSD of a powder batch, with a wide PSD being linked to an increased part density due to the better packing of powder within the powder chamber [201]. This is as a wide distribution of powders will allow smaller particles to settle in the spaces between large particles, as in Figure 7.38, decreasing the volume of voids within the powder bed and thereby increasing the density of parts [80]. This contrasts with the findings of this research, where improved powder flowability and a wider PSD did not result in an increase in part density according to μ -CT, with a reduction in relative density observed instead.

Since μ -CT is more suitable for identifying large pores due to its limitations in resolution, the decrease in part density seen is likely due to the increased presence of larger pores, such as keyhole and LOF porosity. The failure initiation sites of the R20 fatigue samples also support this, with fracture initiating from large defects that include a large LOF pore surrounding an

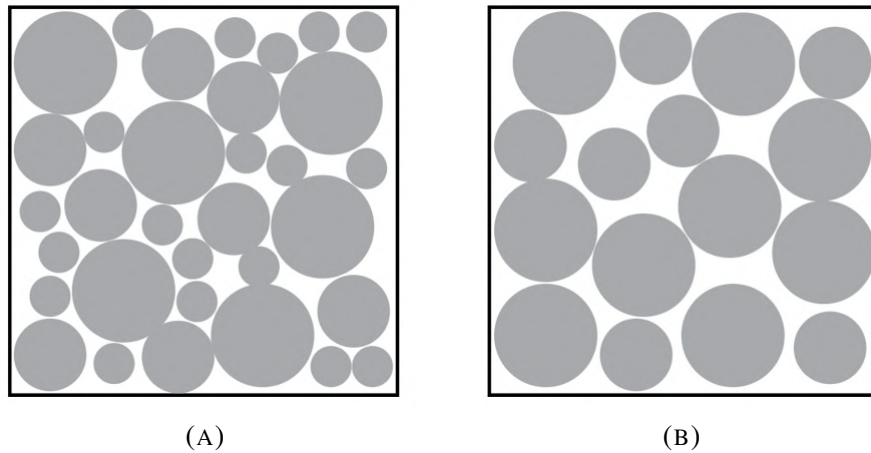


FIGURE 7.38. Schematics of different packing densities of powders: (a) with a high proportion of fine particles and therefore high packing density, (b) with a low proportion of fine particles and therefore low packing density.

inclusion. The increased age of the powder batch therefore likely results in an increase in the presence of foreign inclusions from the sieving and powder recoating processes which increase the prevalence of LOF defects within parts manufactured with reused powders. The increased presence of agglomerate and large spatter particles would also increase the amount of LOF pores with their large size potentially reducing their meltability, therefore influencing melt pool behaviour and inducing further porosity [316, 317]. These factors together result in the reduction in part density observed by μ -CT, with the slight improvements seen in PSD and powder flowability not inducing any significant change to part density.

7.4.3.2 Surface Roughness

The surface roughness of parts shows considerable dependence on the location at which measurements were taken. For samples measured on cubes, the front and side of parts show consistent roughness with minimal variation according to powder reuse. The roughness at these locations was also equal, indicating that the direction a vertical surface faces has minimal impact on its roughness. Noteworthy was the fact that the top surfaces of the cubes had considerably greater roughnesses than the fronts and sides, typically having an Ra value three times that of the front and side. This is as the surface roughness of the top of the cubes is dictated by the hatch spacing of the laser scans, which for these parts was 0.112 mm, in comparison to the layer thickness of 0.03 mm which is the main influence on the roughness of

vertical surfaces. This larger spacing therefore increases the roughness of these top surfaces, with the ratio of the hatch spacing to layer thickness corresponding well to the ratios of surface roughness on these surfaces.

The roughness of sloped surfaces similarly showed no correlation to powder reuse, although they had considerably greater deviations when compared to the cube surface measurements. It is also clear that the under skins of parts have considerably greater surface roughnesses than top skins, with an average R_a of $12.57\text{ }\mu\text{m}$ compared to $7.98\text{ }\mu\text{m}$ for the top skins. This is due to the increased sintering of particles to the under skins compared to the top skins, as when the under skin is melted to shape, particles underneath the surface sinter to it due to the radiant heat from the melt pool. For top skin surfaces this does not occur, as when the surface is melted there is no adjacent powder to sinter to the surface, as it has yet to be spread in the subsequent layers. This accounts for the increased roughness of under skins compared to top skins.

The consistency of roughness for all surfaces in relation to the refreshing powder reuse method is significant, as it indicates that no matter the state of powder the surface roughness of parts will not change. The surface roughness is governed by two main factors: the production parameters used, namely the hatch spacing and layer thickness, and the PSD of the powder. As the laser parameters were kept constant through this study, it follows that the surface roughness as a function of powder reuse should not be influenced by the hatch spacing and layer thickness. However, a shift in PSD has been observed. Given the roughness did not change, this indicates that the changes in PSD were not significant enough to result in a change in R_a . This was likely aided by the utilisation of the refreshing powder reuse method, given the D10, D50, and D90 values were able to stabilise rather than continue to increase as they did from R1 to R7.

7.4.3.3 Part Chemistry

The chemistry of parts manufactured using reused powders shows good consistency, especially in regard to the lower concentration alloying elements iron and titanium, which remain stable at 0.06 wt.% and 0.04 wt.% respectively. More importantly, the silicon content remains stable at approximately 6.7 wt.%, which means the solidification range of AlSi7Mg will remain constant with powder reuse and the processability of the alloy will not be affected.

The main exception to the rule of elemental consistency is oxygen, which continues to increase in quantity as powder is continually reused. This increase is both gradual and consistent, increasing from 0.015 wt.% for R1 to 0.048 wt.% for R20, an increase of 320 %. Due to the powder oxygen content results being influenced by storage conditions, while the increase in part oxygen content is likely linked to that of the powder, this link cannot be explicitly drawn here.

During the early stages of powder reuse, the powder oxygen content is high while the oxygen within the parts is relatively low, as shown in Figure 7.39a. Given the contamination of powder oxygen contents due to long term storage, two observations within the literature can be used to explain the increase in part oxygen content. During powder reuse spatter accumulates in the powder batch, as evidenced by the increasing D90 value, with these larger particles being considerably more oxidised than their smaller counterparts [165, 318]. This increase in spatter contributes to the increase in part oxygen content [319]. The increase in oxygen content in parts may also be attributed to the changing density of parts. A lower density equates to higher part porosity, which is often filled with shielding gas from the chamber atmosphere. While this shielding gas is of high purity, there remains trace amounts of residual oxygen in the gas and when this gas is entrapped within the parts as gas porosity, the overall oxygen content of the parts is increased. This is especially pertinent given porosity often has oxidised surfaces [320, 321], further increasing the oxygen content of parts. This then contributes to the constant increase in part oxygen content with powder reuse, however, it should be noted that this contribution is limited given the small increase in the porosity of parts.

Part magnesium content follows the same trend as powder magnesium content, with a gradual reduction as powder is reused. Also of note is the difference between powder and part magnesium content, as while both gradually decrease with powder reuse, their levels are different. Powder has a magnesium content that varies between 0.39 wt.% and 0.42 wt.%, while the parts regularly have a lower magnesium presence that varies between 0.36 wt.% and 0.39 wt.%. The difference between the two lies between 0.01 wt.% and 0.05 wt.% for each case tested, however, is always lower for the parts than the powder. These differences are shown in Figure 7.39b. The difference in magnesium content between the powder and parts is due to magnesium's low evaporation temperature of 1090 °C, while the melt pool can reach temperatures of up to 1200 °C [103]. This means that during processing, the magnesium

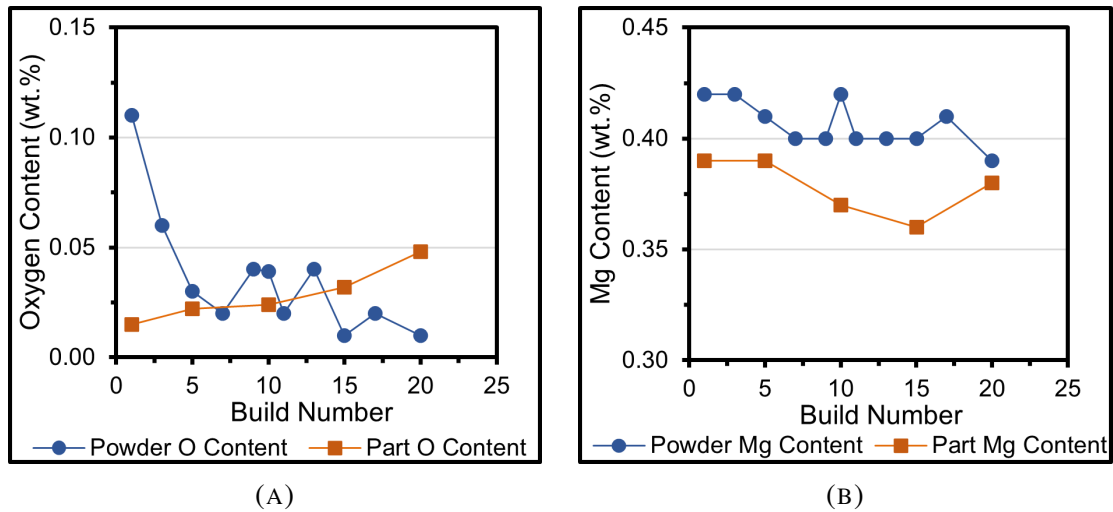


FIGURE 7.39. Comparison of the chemistry of AlSi7Mg powder and parts: (a) oxygen content of powder and parts, where powder oxygen content is affected by long term powder storage, (b) magnesium content of powder and parts.

preferentially evaporates and so its concentration within manufactured parts decreases. This, in combination with the gradual reduction of magnesium content as powder is reused, means that the strengthening of AlSi7Mg by heat treatments may be affected if reused powder is used. This is as the growth of precipitates in the Mg_2Si precipitation sequence is strongly reliant on the continued presence of solute magnesium in the alloy, and if its presence is lowered in reused powder, the amount of precipitate growth able to occur during ageing treatments will also reduce. This means that T6 treatments, as investigated in Chapter 6 of this thesis, may reduce in effectiveness with continued powder reuse.

7.4.4 Mechanical Properties

7.4.4.1 Hardness

The hardness of samples tested under the refreshing powder reuse method indicate an initial rise in hardness, before plateauing after R7. This behaviour is attributable to the contributions of both changing oxygen content and part density.

The presence of oxygen within aluminium can result in the formation of oxide nano-dispersoids such as alumina, which due to its higher strength than aluminium will increase the strength

of the alloy as a whole via dispersion and Hall-Petch strengthening [322]. As discussed in Section 7.4.3.3, this oxygen is introduced by both the oxidation of the powder batch through the production of spatter, as well as inclusion from the trace oxygen content within the nominally inert processing atmosphere. The continual increase in part oxygen content therefore induces a continual increase in strengthening, and therefore an increase in hardness.

This oxide strengthening is, however, reduced in effectiveness by the reduction of part density seen as powder is continually reused. Given the only slight decrease in density over the first five manufacturing runs as measured by μ -CT, the influence of oxide strengthening is dominant which results in increased hardness. However, from R10 onwards the reduction in density is more significant and so the reduction in strength this causes balances the continued increase in strength seen by continual oxygen accumulation, resulting in a plateauing hardness value. This indicates that the refreshing powder reuse method will result in an initial increase in hardness, followed by a longer-term stabilisation.

7.4.4.2 Tensile Properties

The tensile properties of AlSi7Mg manufactured using reused powders are affected by a number of factors, with part orientation, part surface finish, and heat treatment all influencing whether tensile properties change with powder reuse. For all specimens tested the variance in ductility is too large to see a correlation with powder reuse, and so this discussion will focus primarily on the yield and tensile strengths of samples manufactured with reused powders.

The part orientation appears to have no influence on whether tensile properties will change with powder reuse. As-printed, vertically oriented parts have consistently higher yield and tensile strengths than horizontally oriented parts, with this caused primarily by the early fracture induced by the residual supports on the underside of the gauge sections of the horizontal specimens. This was consistent for both stress relieved and T6 treated samples. Upon machining, for stress relieved samples this discrepancy according to part orientation was largely eliminated, while for T6 treated samples, the horizontally oriented samples were stronger than their vertically oriented counterparts. This follows more closely the findings of the literature, where horizontally oriented specimens are typically found to be stronger than vertically orientated ones due to the preferential orientation of the columnar grain structure [39, 323]. These effects are further elaborated on in Chapter 5 of this thesis.

The surface finish of parts, i.e. whether they were printed or machined to shape, also appears to have minimal impact on whether yield and tensile strengths will be influenced by the refreshing powder reuse method. Machined samples are, however, consistently stronger than their as-printed counterparts. This is because as-printed samples have a rougher surface finish due to the presence of layer boundaries on their surfaces which can act as stress concentrators, with this roughness reduced for machined samples. As-printed samples are also weakened by a ring of porosity present near the edge of parts as a result of the interaction between different laser parameter sets, with this porosity not present in machined samples. These two factors together serve to weaken the as-printed samples in comparison to the machined samples, a trend not influenced by powder reuse. Further detail on the effect of part surface finish is presented in Chapter 5 of this thesis.

While part orientation and surface finish influence the yield and tensile strengths of AlSi7Mg independently of the refreshing powder reuse method, this is not true for the heat treatment applied. Stress relieved samples show a slight increase in strength from R1 to R9, most evident in as-printed specimens, while this trend is not present in the T6 treated samples. This is due primarily to the different strengthening mechanisms at work in each of these heat-treated states.

Stress relieved samples were the weakest of the heat treatment states tested in Chapter 6 of this thesis, and this is also true in these results. The strength of these samples is reliant on the remnants of the silicon cell wall network which, while made more discrete by a stress relieving heat treatment, are still present as seen in Figure 7.40a. Stress relieved samples are not able to rely on the other strengthening mechanisms available to as-built samples, with solid solution strengthening, dislocation density, and the presence of nanosized silicon particles within the cell interiors all significantly reduced after stress relief [283]. As powder is reused in the refreshing powder reuse method, manufactured parts have been seen to gradually increase in oxygen content, as discussed in Section 7.4.3.3. In stress relieved parts, this oxygen is able to form oxygen enriched precipitates along the cell walls, as shown in the TEM-EDS maps in Figure 7.40a. These oxides contribute to the strengthening of the alloy by providing an increase in precipitation strengthening, with these precipitates inhibiting the motion of dislocations through the matrix. The continual increase of oxygen as a function of powder reuse means these oxides increase in prevalence in parts manufactured with reused powders,

thereby increasing their strengths. However, the increase in porosity beginning from R10, as per the μ -CT results, reduces the strength of the material and thereafter strengths plateau. Similar effects cannot be seen in the ductility of stress relieved samples due to the large scatter present in the data.

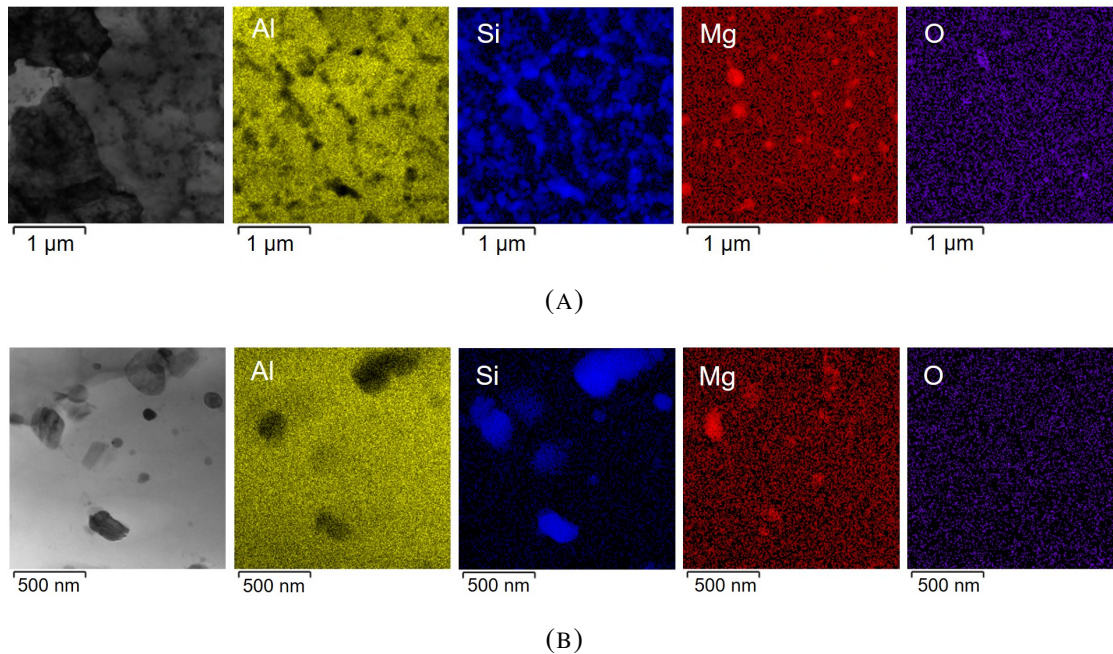


FIGURE 7.40. TEM-EDS measurements of: (a) a representative stress relieved sample (b) a representative T6 treated sample. Magnesium and silicon precipitates are present in both samples, however, oxygen rich precipitates are only visible in the stress relieved sample.

While this is true for stress relieved samples, strengthening with powder reuse is not observed in T6 treated samples. T6 treatment results in the growth of GP zones, β'' , β' and β - Mg_2Si precipitates which provide the majority of the strength for AlSi7Mg in the heat-treated state. Further explanation of this is given in Chapter 6. According to the TEM-EDS maps in Figure 7.40b, oxygen does not form precipitates in the T6 treated microstructure, with silicon and magnesium rich precipitates being dominant and iron rich precipitates also present but not shown here. Instead, this oxygen is present in solid solution at a greater concentration than in the stress relieved samples, as shown in Table 7.2, although this solute oxygen does not provide as much strength as the precipitates grown by ageing treatments. Porosity is the other factor contributing most to part strength, however, the change in part density evident in parts manufactured with reused powders appears to have no effect on the strength of T6

treated parts. This is as the solution treatment step has been seen to greatly increase the porosity of parts by expanding existing gas porosity [142], with this effect dominant over any small increases of porosity as a result of using reused powder. Given that a stress relief sees changing tensile properties with powder reuse while a T6 treatment eliminates these, proper heat treatment can be seen as a way of reducing any of the minimal effects imparted by the refreshing powder reuse method.

TABLE 7.2. Chemical composition of the matrix for stress relieved and T6 treated samples. Values are the average of seven different point measurements taken by TEM-EDS, with all measurements in wt.%.

	Al	Si	Mg	O
Stress Relieved	99.21 $\pm$ 0.05	0.18 $\pm$ 0.02	0.12 $\pm$ 0.02	0.45 $\pm$ 0.04
T6	98.71 $\pm$ 0.22	0.33 $\pm$ 0.12	0.20 $\pm$ 0.02	0.60 $\pm$ 0.16

7.4.4.3 Flexural Properties

The flexural properties of AlSi7Mg appear to be influenced by part orientation, with horizontally oriented parts consistently displaying higher yield and flexural strengths than vertically oriented parts. While this difference is small, it is consistent and is especially noticeable in the flexural strength results. The difference in strength is for reasons similar to those for tensile strength, with the preferential orientation of grains weakening vertically oriented samples. This is as the columnar grain structure is oriented along the build direction, i.e. the vertical direction, meaning that vertically oriented specimens are loaded along the elongated axis of these grains. The elongated nature of these grains serves to weaken these samples by reducing the effective grain size, reducing the amount of Hall-Petch strengthening available when parts are loaded in this orientation. This is in comparison to the horizontally oriented specimens which are loaded along the shorter axis of the grains, providing a greater Hall-Petch strengthening effect. This reduction in strength seen in the vertically oriented samples is, however, compensated by an increase in the ductility of parts for the T6 treated samples. The same effect is likely to occur in stress relieved samples, however, is obscured by the termination of tests upon reaching 7 % strain, which was reached by all stress relieved samples.

Heat treatment is also seen to influence the flexural properties of AlSi7Mg, regardless of powder reuse state. T6 treated samples have both higher strength and lower ductility than

their stress relieved counterparts, with this being consistent for both sample orientations. This is primarily due to the reduction in strength of stress relieved samples caused by the loss of solid solution strengthening, reduction of dislocation density, and coarsening of the silicon cell network, while T6 samples are able to take advantage of the growth of Mg_2Si precipitates after ageing. These strengthening effects are complemented by changes in ductility, with stress relieved samples showing considerably higher ductility than T6 treated samples, as expected in a strength-ductility trade off. These effects are elaborated on in Chapter 6 of this thesis, and so are not discussed in further detail here.

The refreshing powder reuse method can be seen to have no significant influence on the flexural properties with yield strength, flexural strength, and ductility each showing no correlation to the reuse state of the powder. For R1 samples, the horizontal T6 sample exhibits a slightly higher strength than for other reuse states, while the vertical stress relieved sample shows a slightly lower strength than subsequent reuse states. However, given these are individual points and no trends are present from R5 onwards, it is likely that these tests are outliers and there is no true change to flexural properties when utilising the refreshing powder reuse method.

7.4.4.4 Impact Toughness

The impact toughness of AlSi7Mg appears to be most strongly influenced by the orientation of the part, with horizontally oriented specimens consistently having a greater impact toughness than vertically oriented specimens. This is influenced largely by factors relating to the PBF process, namely the layer-by-layer manufacturing. Horizontally oriented specimens fracture across layer lines, with the notch being manufactured perpendicular to these layers meaning the fracture surface must cross these lines. Vertically oriented samples, however, are manufactured with the notch parallel to the layers, meaning that a fracture initiated at this notch is able to travel along one layer boundary rather than being required to intersect multiple boundaries. Fracture is easier along these inter layer boundaries as they are planes of weakness in PBF parts, given this is where lack of fusion defects form [324] and is also the location of the weaker melt pool boundaries with a disrupted silicon cell structure. This is shown in Figure 7.41, with the red lines showing the increased tortuosity of the path across the melt pool boundaries for the horizontally oriented sample as opposed to the vertically

oriented sample. This leads to a reduction in impact toughness for vertical, compared to horizontally oriented, samples. Similar results have been found in other research on Charpy impact behaviour in PBF [325, 326].

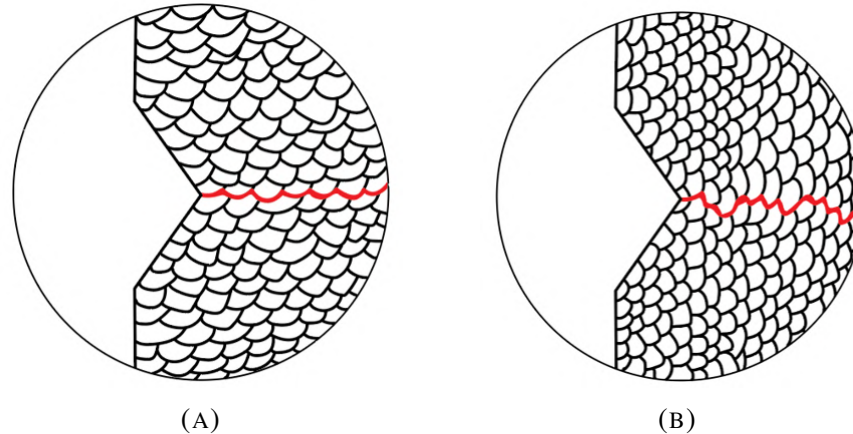


FIGURE 7.41. Paths for fracture across melt pool boundaries for: (a) vertically oriented specimens (b) horizontally oriented specimens. Horizontally oriented specimens show a more tortuous path as the melt pool boundaries do not align with the direction of crack propagation.

Machined samples can also be seen to have a greater impact toughness than samples that were printed to shape, with this effect being most pronounced in the vertically oriented samples. As elaborated on in Chapter 5, this largely has to do with the improved surface finish of machined samples in comparison to as-printed ones, with this being especially true given the notch sensitivity of these tests. Machining produces a higher quality notch than the as-printed samples, with as-printed samples containing a considerable amount of sintered powder within these notches. This greatly increases their roughness and so increases the stress concentration at certain points, promoting fracture with lower energy requirements. This therefore reduces the impact toughness of these as-printed samples in comparison to the machined samples, where such stress concentrators do not exist. This effect is more pronounced in the vertically oriented specimens due to their predisposed reliance on surface quality, owing to their failure along layer boundaries.

Impact toughness shows a mixed relationship with powder reuse, with all sample manufacturing methods showing no clear reliance on powder reuse with the exception of the stress relieved, as-printed, horizontally oriented (SR-P-H) samples. These samples showed a continual reduction in impact toughness as powder was reused, likely due to the increased

presence of oxygen in the manufactured parts. Oxygen embrittles aluminium and so its gradually increasing presence in parts will naturally result in a reduction of impact toughness. While this trend is clear only in the SR-P-H parts it is present to some degree in the other samples, albeit to a lower degree which makes identification of a definite trend difficult. The machined, horizontally oriented parts appear to demonstrate a similar trend to their as-printed counterparts, however, the larger standard deviations and the exceptionally low and high absorbed energy values of R1 and R20 respectively obscure any trend. Similar is true for the vertically oriented samples in both the machined and as-printed states. While an argument could be made for the reduction of impact toughness as a result of the refreshing powder reuse method, this trend is minimal. This is likely due to the reliance of these samples' failures on the inter layer boundaries as planes of weakness, where the effects of increased porosity and a weakened silicon cell network from processing factors dominate those of any change in oxygen content coming from powder reuse. Given the presence of these weak trends, further powder reuse may reveal more definitive trends. However, given the relative stability of the impact toughness for the majority of samples, it appears that the refreshing powder reuse method inhibits any changes to impact toughness due to reused powder.

7.4.4.5 Fatigue Strength

The fatigue strength of AlSi7Mg is clearly affected by the refreshing powder reuse method. This is most clearly seen in the fatigue limit of samples, defined as the stress amplitude for which this alloy can withstand 1,000,000 cycles, which for R1 lies between 200 MPa and 250 MPa, while for R20 samples lies between 150 MPa and 200 MPa. Fatigue life is often dictated by part surface roughness [327], however, given these specimens were all machined to shape this is not the cause for the observed change in fatigue limit. An analysis of the fracture surfaces indicates that this change is instead reliant on the reduction in part density and the corresponding size, number, and type of pores. Fatigue life is known to be influenced significantly by porosity, with PBF parts containing pores generated by a variety of processes [299]. The manifestation of this is, however, different for low and high cycle fatigue behaviour.

Low cycle fatigue (LCF) sees minimal difference for parts manufactured with new and reused powders, with a difference of only a few hundred cycles seen at a stress level of 300 MPa.

The R1 sample fractography reveals that fracture initiated from a small LOF pore, with the failure of the R20 sample starting at a larger, more spherical pore. Given the majority of fatigue life in LCF is spent in crack growth [48], it is clear that the presence of porosity of any size will result in failure after a relatively similar number of cycles. This means that minimal change in LCF life is expected when reused powder is used to manufacture components.

The most notable difference in fatigue life is seen in high cycle fatigue (HCF), similar to other studies [176, 213], as in this regime, fatigue life is most affected by crack initiation rather than crack growth [48]. At a load of 200 MPa, the R1 samples did not fail. This suggests that the majority, if not all, of the one million cycles these samples underwent were spent initiating a crack. Due to the relatively high density in these samples few large pores existed after processing, extending the number of cycles required to initiate a crack and so result in failure. This is in contrast to the R20 samples, which failed after an average of 298,000 cycles, a significant reduction in fatigue life compared to the R1 samples. The fracture surface observed indicates that failure initiated from a poorly bonded foreign silicon inclusion in the matrix, which given the presence of rounded particles in the vicinity, induced a LOF pore. These LOF pores are known to be especially detrimental to fatigue life [328], and so their presence meant that fewer cycles were required to initiate the crack compared to the R1 sample.

The fatigue life of parts manufactured with reused components can therefore be seen to be influenced by changing porosity and inclusions. The reduction in part density over 20 manufacturing runs as observed by μ -CT points to an increase in porosity, increasing the sites for crack initiation and therefore reducing fatigue life, especially for HCF. Similar explanations for changes in fatigue life in PBF parts have been provided elsewhere within the literature [180, 328, 329], with this porosity especially detrimental given these pores are more likely to be LOF in R20 samples than R1 samples. This effect is exacerbated by the inclusion of foreign particles in the powder batch, likely coming from contamination during powder handling procedures and powder recoating during the manufacturing process. These particles will accumulate as powder is reused, creating more sites for potential LOF porosity to form, further inhibiting fatigue performance. The refreshing powder reuse method can therefore be seen to have a detrimental effect on fatigue life, particularly during HCF.

7.5 Conclusion

From the above results, it is clear that the refreshing powder reuse method does have some effect on both the quality of a powder batch and the properties of parts manufactured with this reused powder.

The main changes to the powder batch came as an improvement in the flowability of the powder, resulting from a change to the powder's PSD. A reduction in the number of fine particles in the powder batch and concurrent increase in large powder particles, stemming from the preferential consumption of fine powders and continual spatter production of larger particles, shifted the PSD to both a larger and wider distribution. This shift resulted in improved flowability of the powder, given the better flow properties of large particles compared to small ones. This improved flowability increased the spreadability of the powder within the build chamber, although this did not result in any change to the density of the powder bed. Chemically, powders remained stable, with the exception of a slight reduction in magnesium content, while any potential changes in oxygen content were obscured by the long-term storage of powder prior to testing.

These changes in powder properties resulted in no changes to the microstructure of parts, with the melt pool shape, grain size and texture, and the silicon cell network all remaining consistent when manufactured using reused powder. This indicates that the melt pool dynamics and solidification behaviour were not influenced by the small changes observed in the powder.

The properties of parts showed more of a relationship with powder reuse, with the oxygen content of parts increasing and the density of parts decreasing as powder continued to be reused. Also of concern was the reduction in magnesium content with continual powder reuse, both as a result of powder reuse but also the evaporation of magnesium within the melt pool, resulting in powder and parts from the same manufacturing run having different magnesium contents. In contrast to these changes, the surface roughness of parts was consistent no matter the stage of powder reuse.

These changes to powder and part properties resulted in small changes to the mechanical properties of parts manufactured with reused powders. Stress relieved samples increased in yield strength, tensile strength and hardness as powder was reused due to the changes in

part oxygen content and porosity, although these properties stabilised from R9 onwards. A T6 treatment was then seen to eliminate the changes attributed to manufacture with reused powders due to the different strengthening mechanisms for these different heat-treated states, showing great promise to reduce the potential impact of powder reuse. Horizontally oriented Charpy samples showed a gradual reduction in impact toughness when manufactured with reused powder, although this was obscured for machined samples by larger deviation within the data. Flexural properties were seen to have no relation to the reuse history of the powder, while the HCF life of parts was seen to reduce with powder reuse.

These results indicate that the refreshing powder reuse method shows great promise for industrial application, with minimal change in properties observed over 20 manufacturing runs. Fatigue performance is the exception to this, with a reduction in fatigue life seen after 20 manufacturing runs. Most significantly, the majority of changes to powder characteristics and mechanical properties stabilised once nine manufacturing runs had been completed, insinuating that further manufacturing runs beyond the 20 completed in this study would also show minimal change. A T6 heat treatment also negated any property change observed due to powder reuse, suggesting that the utilisation of the refreshing powder reuse method in tandem with T6 heat treatment will allow for improvement in both the properties of AlSi7Mg parts, as well as the efficiency of the process by allowing for continual powder reuse.

The Single Batch Powder Reuse Method and its Effectiveness in Comparison to the Refreshing Powder Reuse Method

8.1 Introduction

As elaborated on in previous chapters, powder reuse in PBF is crucial to ensure the economic and environmental sustainability of the process. This is especially true given the large amounts of powder that remain unused at the end of a manufacturing run, the waste of which would result in a significant increase in production costs.

When it comes to powder reuse, the alloy system used is the most commonly investigated factor [3, 189, 330]. While this is important, it does not provide a holistic overview of all the factors that will influence reused powder properties. One of the aspects of powder reuse least investigated is the powder reuse method, with little consideration ever given to this in the literature. A poor understanding regarding the influence of powder reuse methods has the potential to result in inefficient methods being used, as others may be more appropriate for a given application. As such, a comparison of powder reuse methods is crucial.

The single batch method is the most commonly used powder reuse method due to its simple powder handling procedures and good powder traceability, as elaborated on in Chapter 3. However, while it is commonly studied, the property changes observed as a result of powder reuse vary significantly. This is seen across a variety of alloy systems and tests, as varied results are seen in observations of PSD [162, 173, 214], tensile properties [64, 200, 219], and powder flowability [65, 184, 222]. The exception to this variability is powder oxygen content, which tends to increase no matter the number of manufacturing runs or alloy system used. This variability implies that the mechanisms behind changing properties with powder reuse are not yet fully understood.

This chapter therefore first investigates the single batch powder reuse method to analyse its effectiveness as a method and determine its suitability for industrial application. A comparison is then made with the refreshing powder reuse method in order to determine the relative effectiveness of these two different powder reuse methods. To complete this, a simplified version of the investigations carried out in Chapter 7 is conducted again involving powder characterisation, microstructural investigation, part property analysis, and mechanical property investigation.

While there are differences in characteristics between the initial powder batches used to test the refreshing and single batch powder reuse methods, both meet the specifications required for AlSi7Mg regarding both chemistry and powder size. While these differences do not invalidate a general comparison of powder batch behaviour during powder reuse, given these powder batches were sourced from the same supplier it does highlight the importance of quality control between powder of different batches.

8.2 Powder Reuse Scheme

To investigate the effect of the single batch powder reuse method and compare it to the refreshing powder reuse method, specimens similar to those in Chapter 7 were produced in a series of manufacturing runs. Specimens used for part, microstructural, and mechanical testing were kept consistent throughout manufacturing. Specimens designed specifically to consume material were also produced to ensure that the parts manufactured were consistently equivalent to 18% of the build volume, ensuring the amount of powder consumed in each manufacturing run remained consistent to allow for accurate comparison between manufacturing runs and powder reuse methods.

The single batch powder reuse method requires that no new powder is added to the powder batch between manufacturing runs, which results in a continual reduction in the amount of available powder. For this reason, each successive manufacturing run reduces in height, as seen in Figure 8.1, starting from 257.7 mm, the maximum build height allowed given the dose factor used, with a tenth and final build height of 29.64 mm. No further manufacturing runs were completed as the powder quantity remaining was insufficient to produce the mechanical specimens required for analysis. Even so, vertically oriented samples were not produced in

all manufacturing runs due to insufficient build height. This reduction in build height also meant that the number and volume of specimens produced solely to consume material also changed, with their volume manipulated to ensure that 18% of the build volume powder was consumed in each manufacturing run to mimic the refreshing powder reuse study in Chapter 7. All samples in this chapter were stress relieved prior to testing, as per Chapter 4.

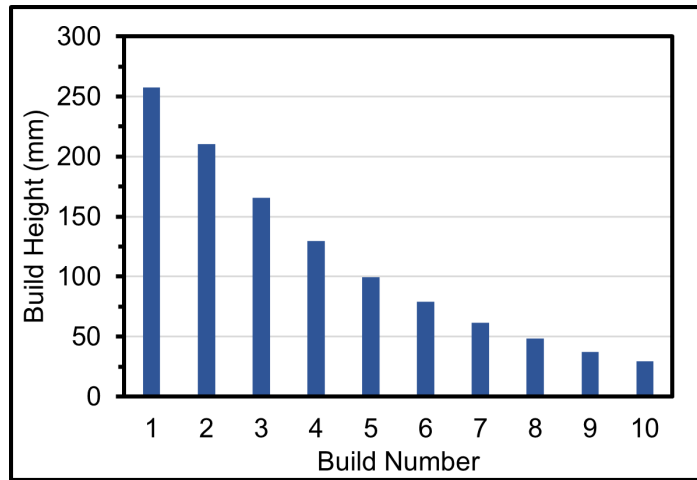


FIGURE 8.1. Build heights for the manufacturing runs conducted using the single batch powder reuse method.

Throughout this chapter each of the manufacturing runs is referred to by the letter S, indicating the single batch powder reuse method was used, alongside a number referencing the manufacturing run itself. S1 refers to the first manufacturing run completed with unused powder in the single batch powder reuse study, with S10 being the last.

8.3 Results

8.3.1 Powder Characterisation

8.3.1.1 Powder Imaging

SEM micrographs of reused powders are shown in Figure 8.2, giving a qualitative view on the evolution of powder as it is reused. Reused powders appear to have a greater prevalence of larger particles, with this especially evident when comparing S1 to S10. The surface morphology of reused powders appears to change with reuse, with the larger particles present

in the S10 powder batch having coarser surfaces in comparison to the unused powder of S1. There is also a slight increase in the prevalence of misshapen particles in the reused powder, although the unused powder also appears to contain a number of elongated and satellite particles.

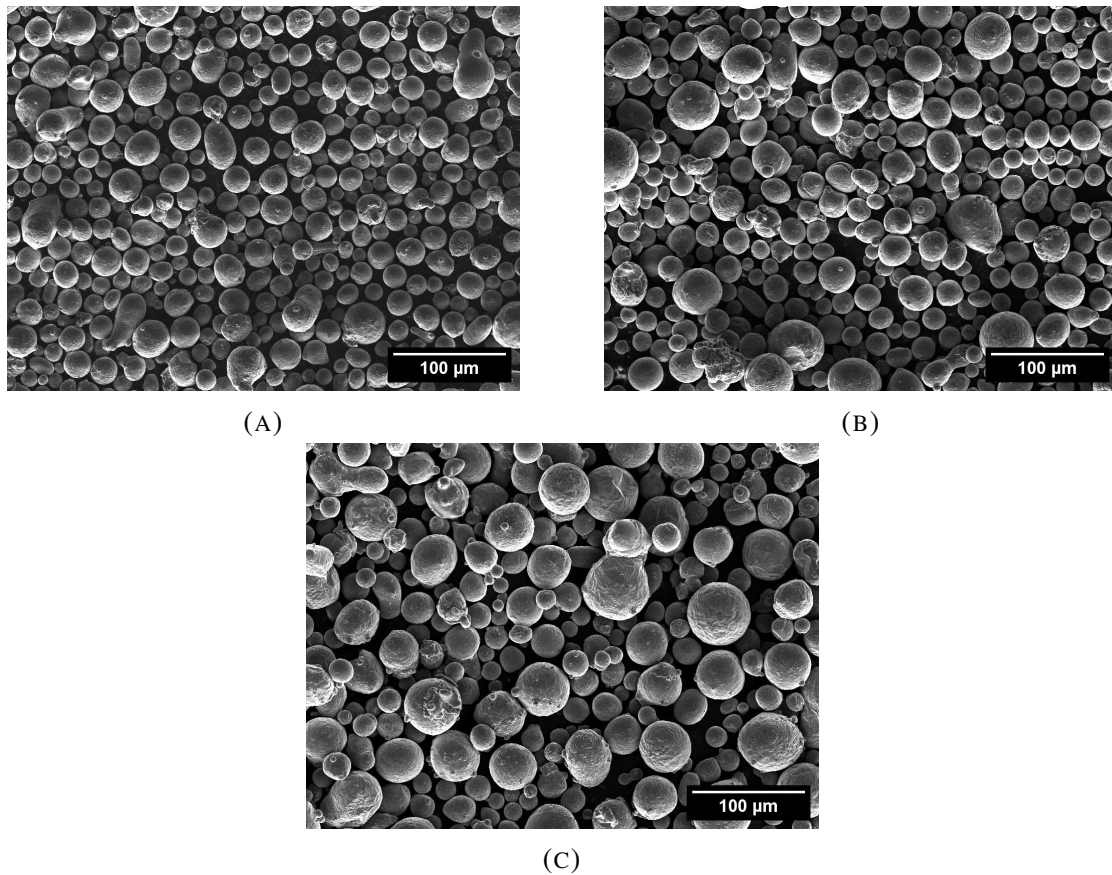


FIGURE 8.2. Representative powder samples from single batch reused powder: (a) S1, (b) S5, (c) S10.

8.3.1.2 Particle Size Distribution

The PSD of powder reused by the single batch method shows consistent change, especially in the measurement of small particles. As displayed in Figure 8.3 the D10 value consistently increases, beginning at 18.3 μm and steadily rising to 20.8 μm by S10, indicating a reduction in the number of fine particles present in the powder batch. The same is true of the D50 value, however, this change slows by S5 after increasing regularly over the first five manufacturing runs. The D90 value follows behaviour similar to the D50 value, increasing between S1 and S5 from 54.7 μm to 61.2 μm before flatlining, albeit with considerable variation.

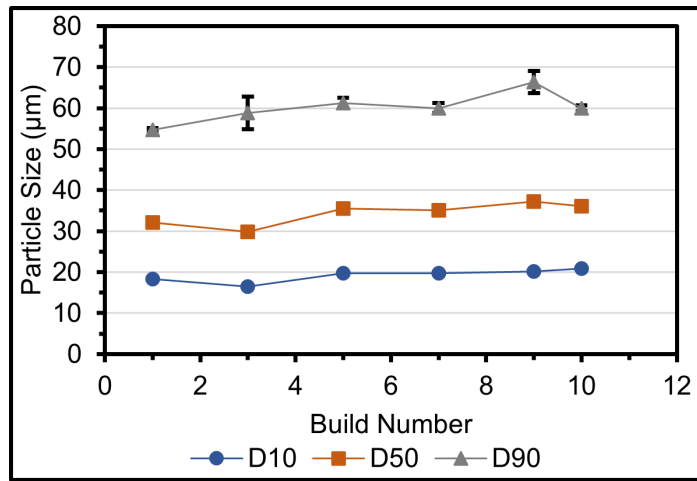


FIGURE 8.3. Particle size distribution of powder reused by the single batch method, represented by the D10, D50, and D90 values.

The changes in PSD are most evident in the volume distribution shown in Figure 8.4. As powder is reused the distribution is shifted to the right by a reduction in the number of fine particles and an increase in the number of large particles. This shift is most significant between S1 and S5, with a smaller shift seen for S10. Between S5 and S10, there is still a small shift to the right, however, this is driven by a reduction in fine particles rather than the influence of both small and large particles. This second shift results in a narrower PSD, as seen by the higher peak of S10 in comparison to the other powder measurements.

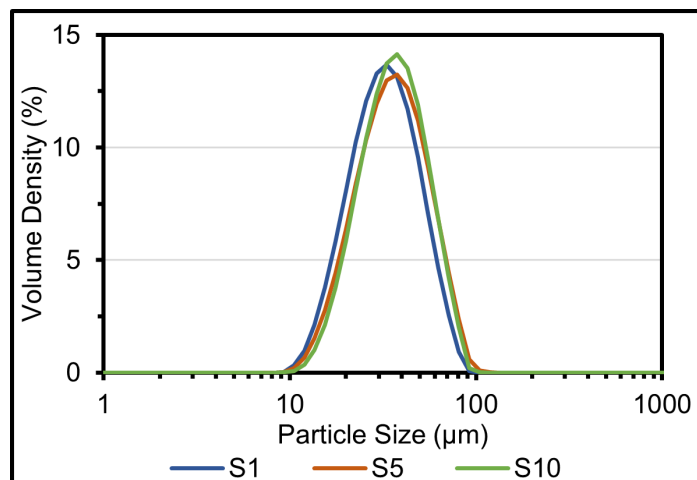


FIGURE 8.4. Particle size distribution of powder reused by the single batch method, represented by the volume distribution of particle sizes.

8.3.1.3 Hall and Carney Flow

The flow times through the Hall and Carney funnels improved as powder was continuously reused, with powder initially not flowing through either funnel even upon tapping. This did not have any noticeable effect on powder spreading in the build chamber. S5 was the first manufacturing run where powder was able to flow through the funnels, and as such there are no flow times for S1-S4 in Figure 8.5. Once powder did start to flow there was a gradual reduction in flow time from S6-S10, with this most clearly seen in the Hall flow tests. S5, however, had a lower flow time than S6-S8 in this test, suggesting that differences in flow times may be related to random sampling.

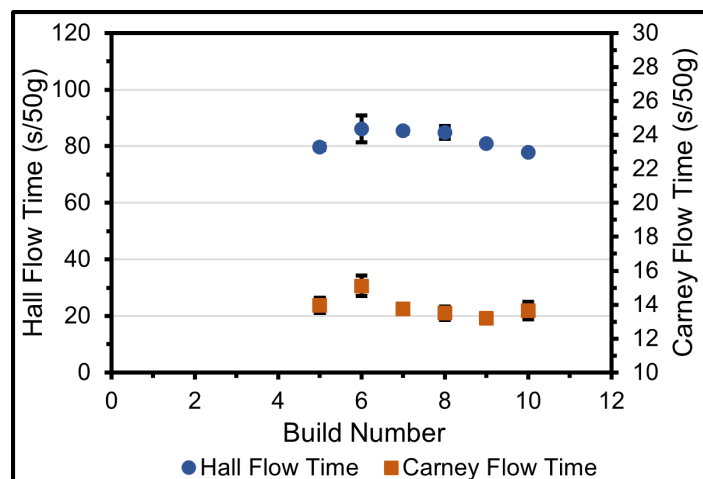


FIGURE 8.5. Hall and Carney flow times for powders reused by the single batch method. As powder did not flow through the Hall or Carney funnels for S1-S4, these values could not be measured.

8.3.1.4 Apparent Density

The apparent density of powder shows no trend with powder reuse, with the minor changes observed appearing to be more related to random scatter than any meaningful influence of powder reuse. These results are shown in Figure 8.6, with testing for S1-S4 unable to occur as powder did not flow through the Hall or Carney funnels.

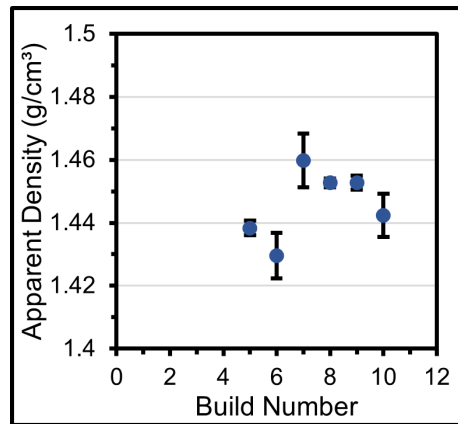


FIGURE 8.6. Apparent density of powders reused by the single batch method. As powder did not flow through the Hall or Carney funnels for S1-S4, the apparent density could not be measured.

8.3.1.5 Powder Flowability

Powder flowability measurements taken with the Granudrum are presented in Figures 8.7 - 8.10. The avalanche angle of powders, shown in Figure 8.7, continues to decrease as powder is reused, an indication of improving powder flowability. While the errors associated with early measurements are large, this does not discount the continuously decreasing avalanche angle. This continual reduction in angle is confirmed by an ANOVA analysis, shown in Appendix B2.1.

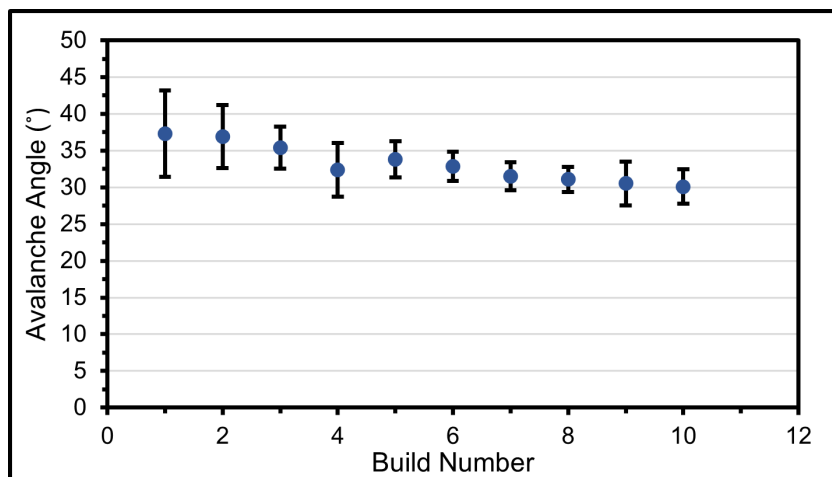


FIGURE 8.7. Avalanche angle of powders reused by the single batch method.

The velocity angles of powders show that powder flowability considerably improves between S1 and S5, with a smaller change between S5 and S10. These changes can be seen in Figures

8.8 and 8.9. The significant reduction in velocity angles from S1 to S5 suggests that the initial powder quality was poor, with any change in powder characteristics able to improve its flowability. Between S5 and S10 powder begins to show an increase in shear thickening behaviour, with the velocity angles for low rotation speeds typically lower for the S10 powder sample than that of S5. This trend then reverses for higher rotation speeds. This indicates better flowability for reused powder at low rotation speeds, but poorer flowability at high rotation speeds.

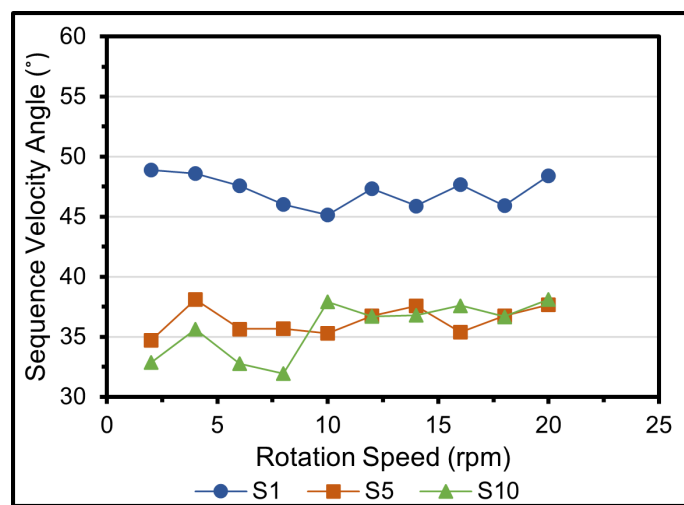


FIGURE 8.8. Sequence velocity angles of powders reused by the single batch method under a variety of rotational speeds.

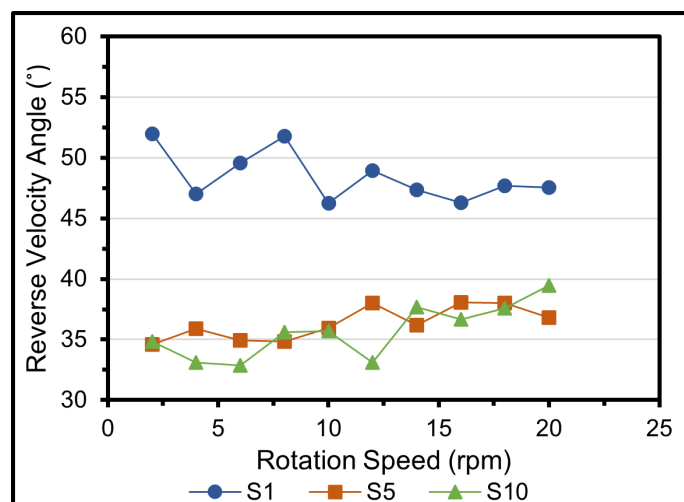


FIGURE 8.9. Reverse velocity angles of powders reused by the single batch method under a variety of rotational speeds.

The same trend is observable in the cohesion of powder reused in the single batch method, as shown in Figure 8.10. The improvement in flowability is clear between S1 and S5 with a dramatic reduction in powder cohesion, and the increase in shear thickening behaviour between S5 and S10 is also clearer than in the velocity angle results. This indicates that lower recoating speeds within the build chamber will take advantage of improved powder flowability, enhancing powder spreadability, especially for reused powders.

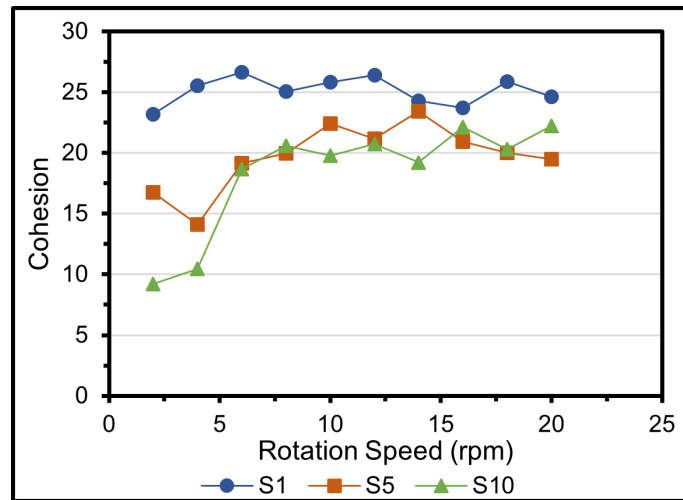


FIGURE 8.10. Cohesion values of flowing powder when reused with the single batch powder reuse method.

8.3.1.6 Powder Bed Density

The powder bed density shows continual increase as powder is reused, as evidenced in Figure 8.11. While this increase is small, from 1.48 g/cm^3 at S1 to 1.59 g/cm^3 at S9, with deviations that are occasionally significant due to the variety of densities across the build plate, this increase in powder bed density is consistent. This indicates that powder spreadability in the build chamber is improving, with the increased amount of powder present in each layer potentially resulting in improvements in part densification.

8.3.1.7 Powder Bulk Chemistry

As seen in Figure 8.12, the majority of the elements present in the AlSi7Mg powder show good consistency as powder is reused. This is true of iron, titanium, and most importantly silicon, which shows a slight decrease over ten manufacturing runs from 6.77 wt.% to 6.67

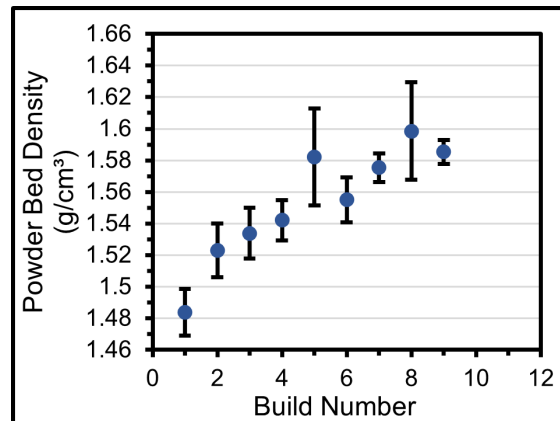


FIGURE 8.11. Powder bed density when utilising powder reused by the single batch method. Capsules were not able to be produced during S10 due to insufficient powder, and so results for S1-S9 are shown.

wt.%. Due to silicon's role in improving the processability of aluminium alloys during PBF, its consistent presence in reused powder indicates that AlSi7Mg's processability will not be significantly influenced by the single batch powder reuse method.

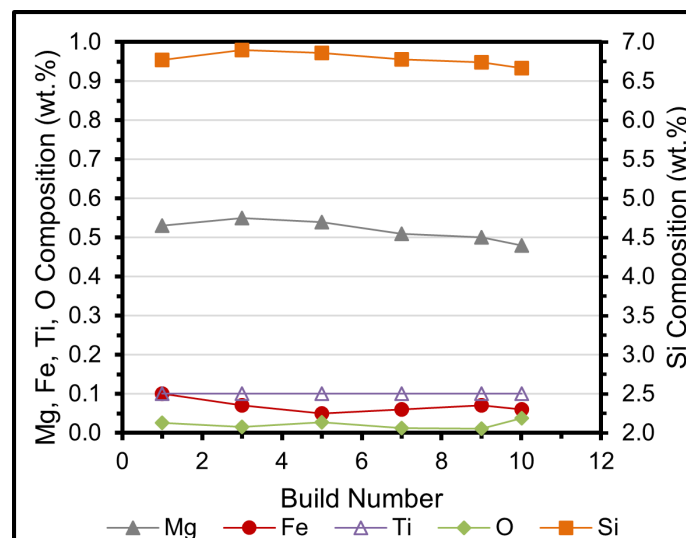


FIGURE 8.12. Bulk chemistry of powders reused by the single batch method. The main alloying elements are shown, with aluminium comprising the balance of the composition.

The oxygen content of powder reused by the single batch method shows great variability, but slightly increases over the course of reuse. S1 contained 0.026 wt.% oxygen, which increased to 0.038 wt.% by S10, an increase of 46%. However, the oxygen content of powders between

S1 and S10 regularly dropped below these values, suggesting that while the oxygen content of the powder is likely increasing, it is also highly dependent on sample selection.

The magnesium content of the powder gradually decreases as powder is continually reused, dropping from 0.53 wt.% at S1 to 0.48 wt.% by S10, a concern given magnesium's role in the precipitation strengthening of this alloy. This reduction occurs primarily from S5 onwards, with the magnesium content slightly increasing from 0.53 wt.% at S1 to 0.54 wt.% at S5, a result of random sampling.

8.3.1.8 Powder Surface Chemistry

The surface chemistry of reused powder is shown in Figure 8.13 and demonstrates a highly oxidised surface, with this oxygen presence varying from 50 wt.% to 56 wt.% over the course of powder reuse. No element showed any relation between surface presence and powder reuse with the exception of magnesium, which began to reduce from S5 onwards, lowering from 8.8 wt.% at S5 to 5.97 wt.% by S10.

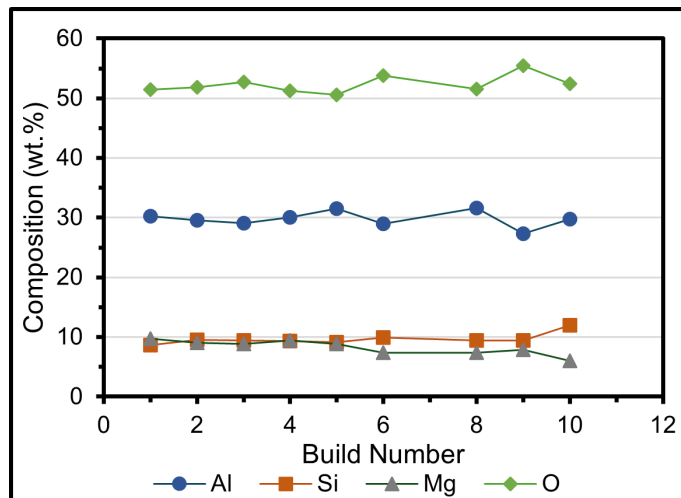


FIGURE 8.13. Surface chemistry of powders reused by the single batch method, by element. XPS survey spectra from which this data was drawn can be found in Appendix C2.

In regard to the chemical compounds present on the surface of reused powders, Figure 8.14 shows that the chemistry is dominated by alumina, which varies between approximately 72 wt.% and 77 wt.% over the ten manufacturing runs tested. These changes show no relation to powder reuse. Also of note is that the presence of magnesium oxide continues to deplete

as powder is reused, reducing from 6.86 wt.% at S1 to 3.16 wt.% by S10. Again, this trend accelerates past S5. The most significant change, however, is the growth in silica, which increases from 6.34 wt.% to 14.43 wt.% over ten manufacturing runs. This is not to the significant detriment of the elemental silicon presence, which only slightly reduces from 5.67 wt.% to 4.73 wt.%, but rather all other compounds present at the surface reduce slightly as the silica presence increases.

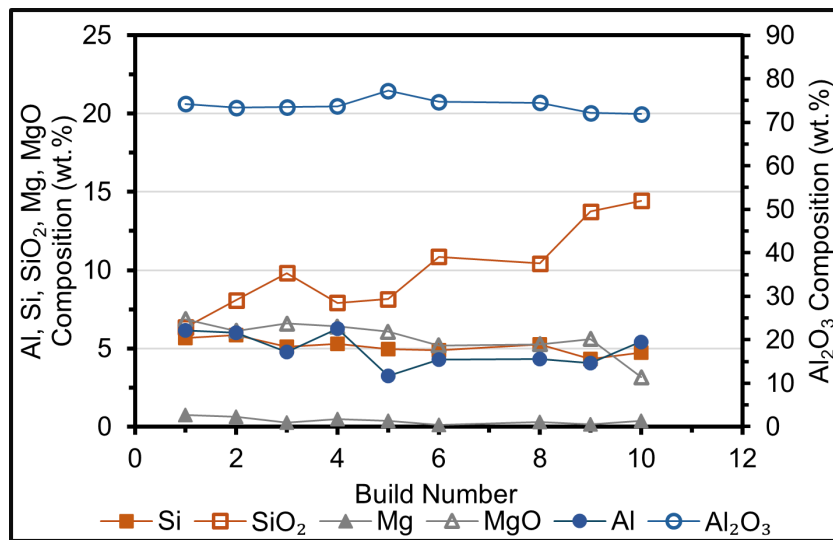


FIGURE 8.14. Surface chemistry of powders reused by the single batch method, by compound. XPS survey spectra from which this data was drawn can be found in Appendix C2.

8.3.2 Microstructural Analysis

8.3.2.1 Melt Pool Characterisation

The X-Z cross sections of parts manufactured with powder from the single batch powder reuse method are displayed in Figure 8.15. These show the fish-scale microstructure characteristic of PBF parts, representative of the melt pools formed by the passing of the laser. No change can be seen between these samples, indicating there is no change to the melt pool structure due to powder reuse by the single batch method.

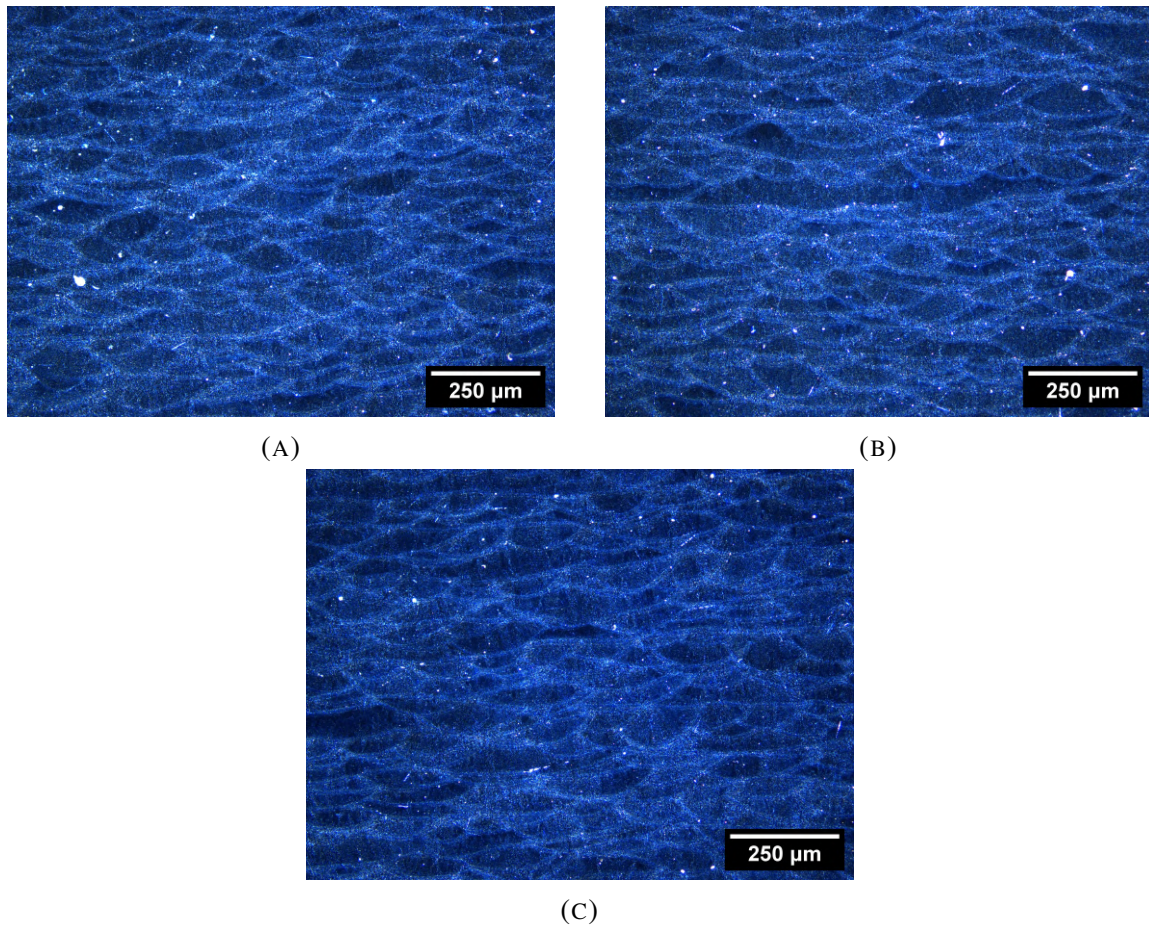


FIGURE 8.15. Optical micrographs of the X-Z planes of parts manufactured with powders reused by the single batch method: (a) S1, (b) S5, (c), S10. The build direction for each micrograph is vertically upwards.

8.3.2.2 Grain Size Characterisation and Crystallographic Texture

The columnar grain structure typical of PBF material remains consistent in parts manufactured with powders reused with the single batch method, with the IPF maps in Figure 8.16 demonstrating columnar grains and smaller regions of equiaxed grains. The size and texture of these grains also show no significant changes as powder is reused, with these values remaining relatively unchanged as shown in Table 8.1. Figure 8.17 shows that these grains, as expected for PBF manufactured parts, show strong texture in the [100] direction.

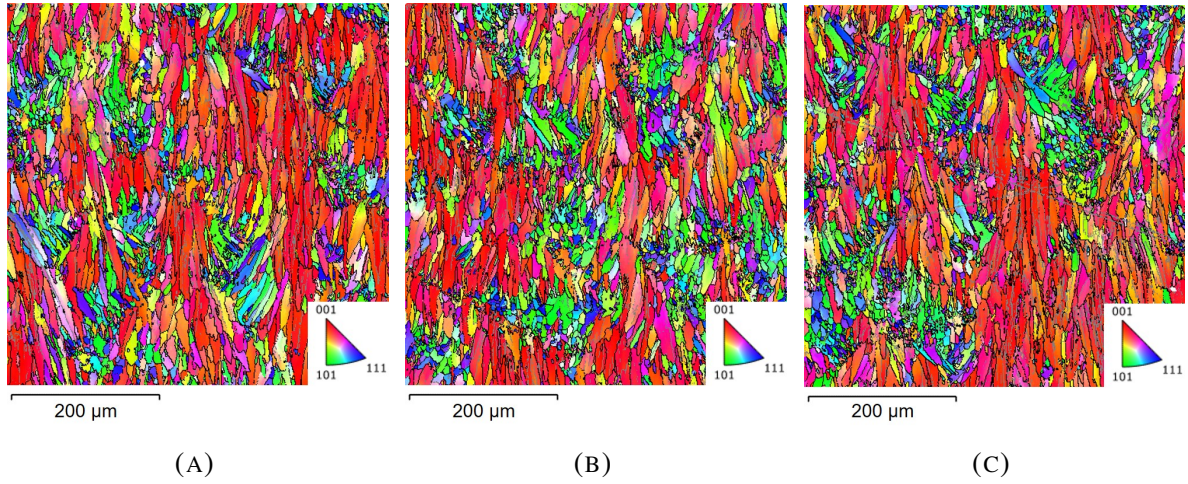


FIGURE 8.16. IPF maps of the X-Z planes of samples manufactured with powders reused by the single batch method: (a) S1, (b) S5, (c), S10. The build direction for each micrograph is vertically upwards.

TABLE 8.1. Grain size and maximum pole figure intensity of parts manufactured with powders reused by the single batch method.

	Grain Size (μm)	Maximum Pole Figure Intensity
S1	9.0 ± 7.16	4.09
S5	8.68 ± 6.63	3.15
S10	8.58 ± 6.87	3.89

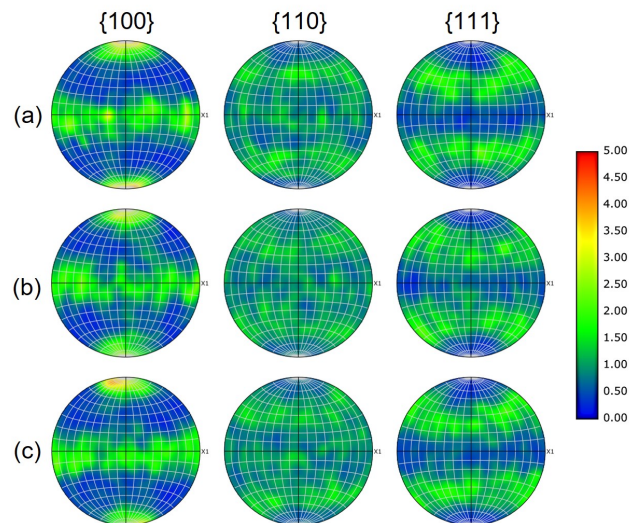


FIGURE 8.17. Pole figures of the X-Z planes of samples manufactured with powders reused by the single batch method: (a) S1, (b) S5, (c), S10.

8.3.2.3 Cell Structure

Within these grains is the silicon cell structure inherent to PBF manufactured aluminium-silicon alloys. Each sample consistently shows the elongated cells present within the large columnar grains that form the majority of the melt pool centres, as well as the more equiaxed cells present in the remelted and heat affected zones of the melt pool. These cell structures are shown in Figure 8.18, with no change evident as a result of powder reuse.

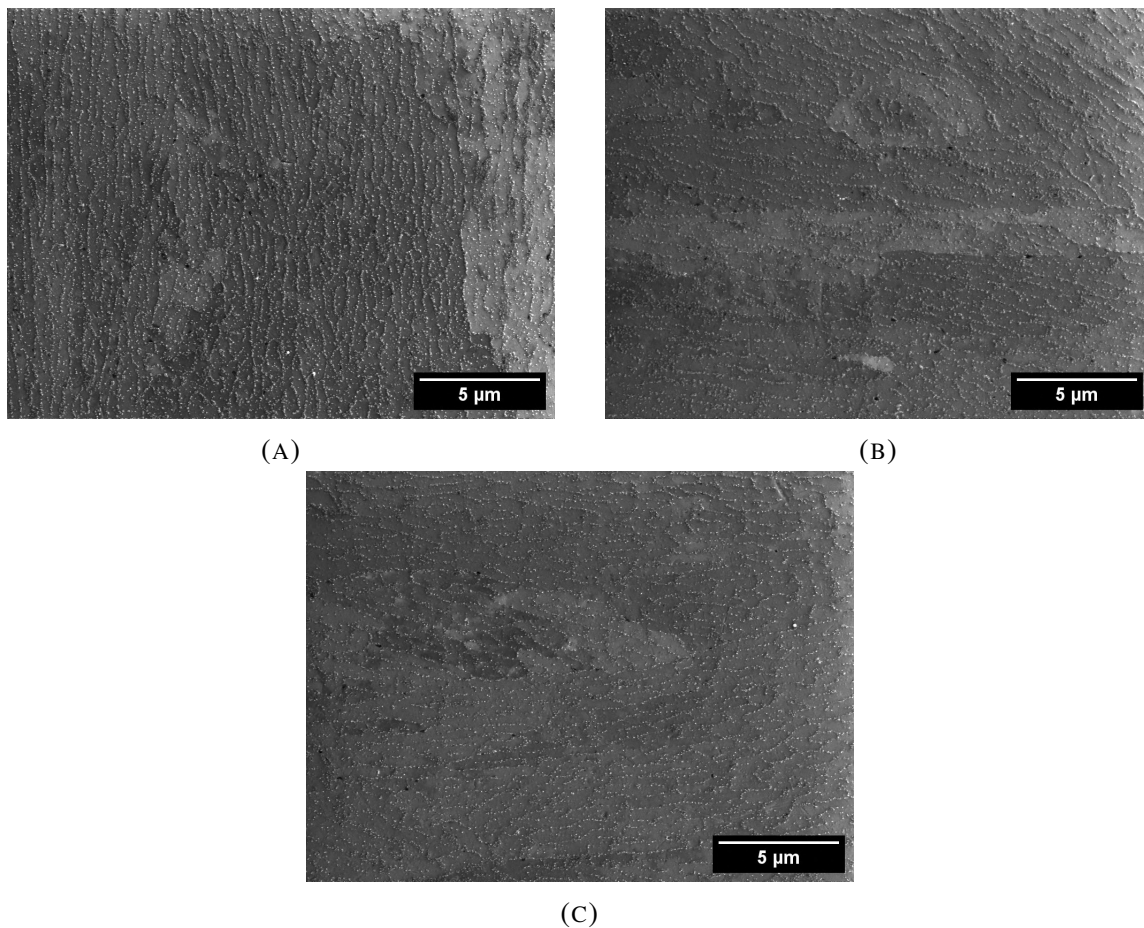


FIGURE 8.18. SEM micrographs of samples manufactured with powders reused by the single batch method, showing the silicon cell structure within the grains of parts: (a) S1, (b) S5, (c), S10.

8.3.3 Part Properties

8.3.3.1 Part Density

The relative density of parts manufactured with powder reused by the single batch method is shown in Figure 8.19. Measurements by the Archimedes method show no change in relative density due to powder reuse, with no trend evident in Figure 8.19a. Some of these points display significant standard deviations that are inherent to the manual measurement process involved, especially S1, the error of which encompasses all subsequent measurements. The relative density does, however, appear to increase with powder reuse when measured by μ -CT, shown in Figure 8.19b. Here, the relative density can be seen to increase from 99.88 % at S1 to 99.94 % for S10. While this increase is slight, it is consistent across all stages of powder reuse. It should also be noted that as per μ -CT measurements, the relative density remains at adequate levels no matter the stage of powder reuse.

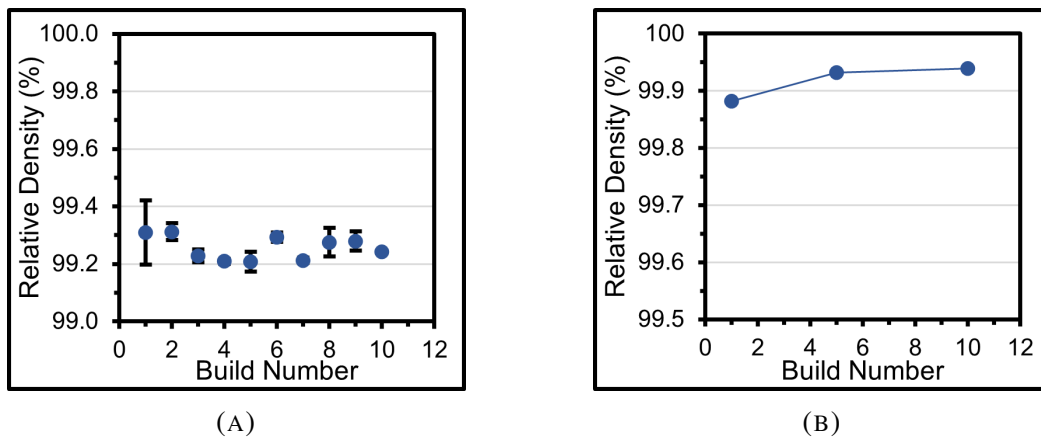


FIGURE 8.19. Relative density of parts manufactured with powder reused by the single batch method, measured by: (a) the Archimedes method, (b) micro-computed tomography.

8.3.3.2 Surface Roughness

The surface roughness of parts shows minimal change as a result of powder reuse by the single batch method, as seen in Figure 8.20. The front and side roughnesses for S1 are slightly higher than those for parts at later stages of powder reuse, however, this change is minimal. The top roughness is also considerably higher than that measured on the front and sides of parts, with significantly greater variation within measurements as seen by the large standard

deviations. This remains consistent with powder reuse and so can be related to processing factors rather than powder history.

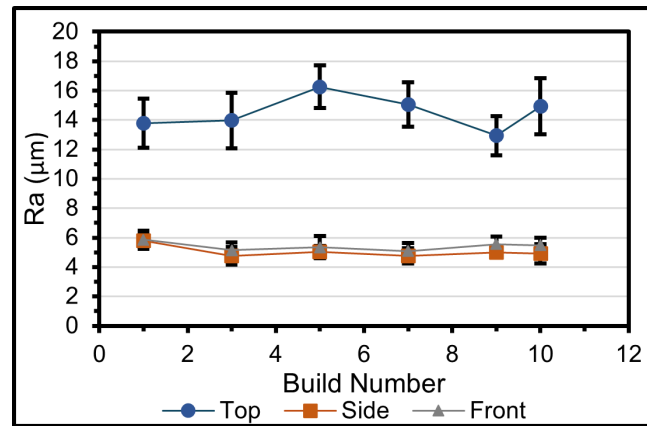


FIGURE 8.20. Surface roughness of parts manufactured with powder reused by the single batch method. All roughness measurements in this figure were taken from a cubic specimen.

8.3.3.3 Part Chemistry

The chemistry of parts manufactured with powder reused using the single batch method remains relatively stable, as shown in Figure 8.21, with silicon, iron, and titanium levels all remaining constant as powder is reused. The magnesium content does, however, reduce between S5 and S10, falling from 0.51 wt.% to 0.47 wt.%.

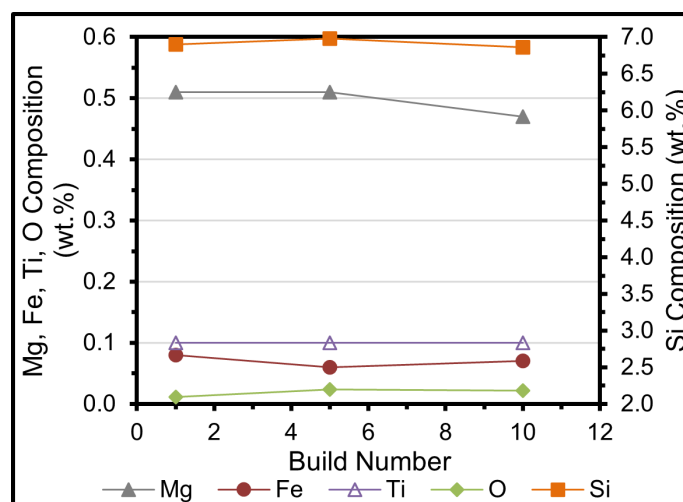


FIGURE 8.21. Chemistry of parts manufactured with powder reused by the single batch method. The balance of the composition is aluminium.

The oxygen content of parts shows a slight increase as powder is reused with this increase most evident in the first five manufacturing runs, where the oxygen content increases from 0.011 wt.% to 0.024 wt.% at S5. There is then no significant change until S10, where the oxygen remains relatively steady at 0.022 wt.%. Overall, this indicates that the single batch powder reuse method results in an increase in part oxygen content over ten manufacturing runs.

8.3.4 Mechanical Properties

8.3.4.1 Hardness

The hardness of parts manufactured with powder from the single batch powder reuse method is shown in Figure 8.22. The hardness of part S1 is slightly lower than that of the rest of the samples, at 76.6 HV instead of the 82.9 HV averaged by the rest of the samples. The hardness of samples increases until S3 where it reaches 84.5 HV and stabilises, with most change lying within the range of standard deviations. The validity of this slight initial increase in hardness is confirmed by an ANOVA analysis, the details of which are displayed in Appendix B2.2. However, the large standard deviation associated with S1 indicates that this may be a result of sampling rather than a genuine trend relating to powder reuse.

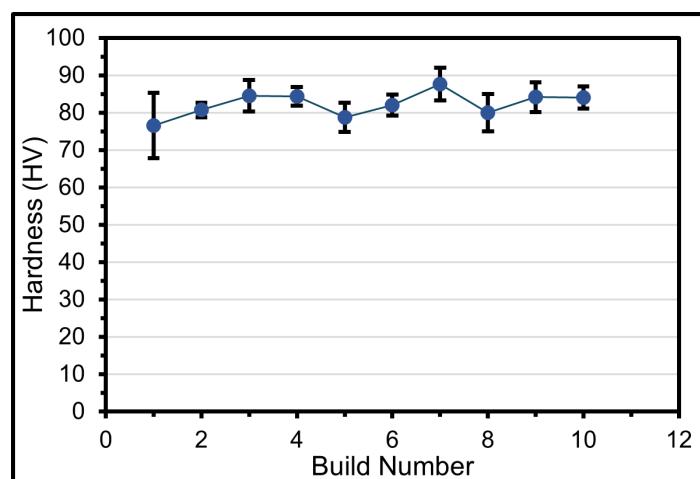


FIGURE 8.22. Hardness of specimens manufactured with powder reused by the single batch method.

8.3.4.2 Tensile Properties

The tensile properties of large tensile specimens, the dimensions of which are displayed in Figure 8.23, that were manufactured with powder reused by the single batch method are shown in Figure 8.24, with vertical specimens tested only for the first five manufacturing runs due to insufficient powder for further production. These results show that vertically oriented specimens perform better than horizontally oriented specimens in both strength and ductility properties in all cases tested, with this difference being especially evident in the tensile strength and ductility of samples. These samples display no evidence of changes in strength or ductility as a result of powder reuse, with samples behaving consistently over the course of powder reuse.

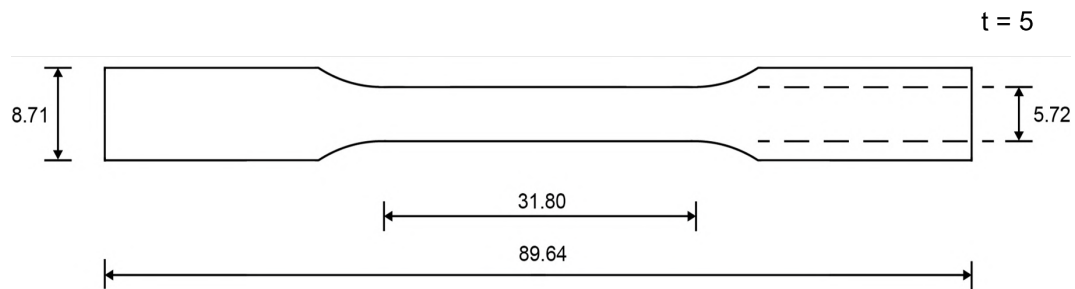


FIGURE 8.23. Dimensions of tensile specimens manufactured with powder reused by the single batch method.

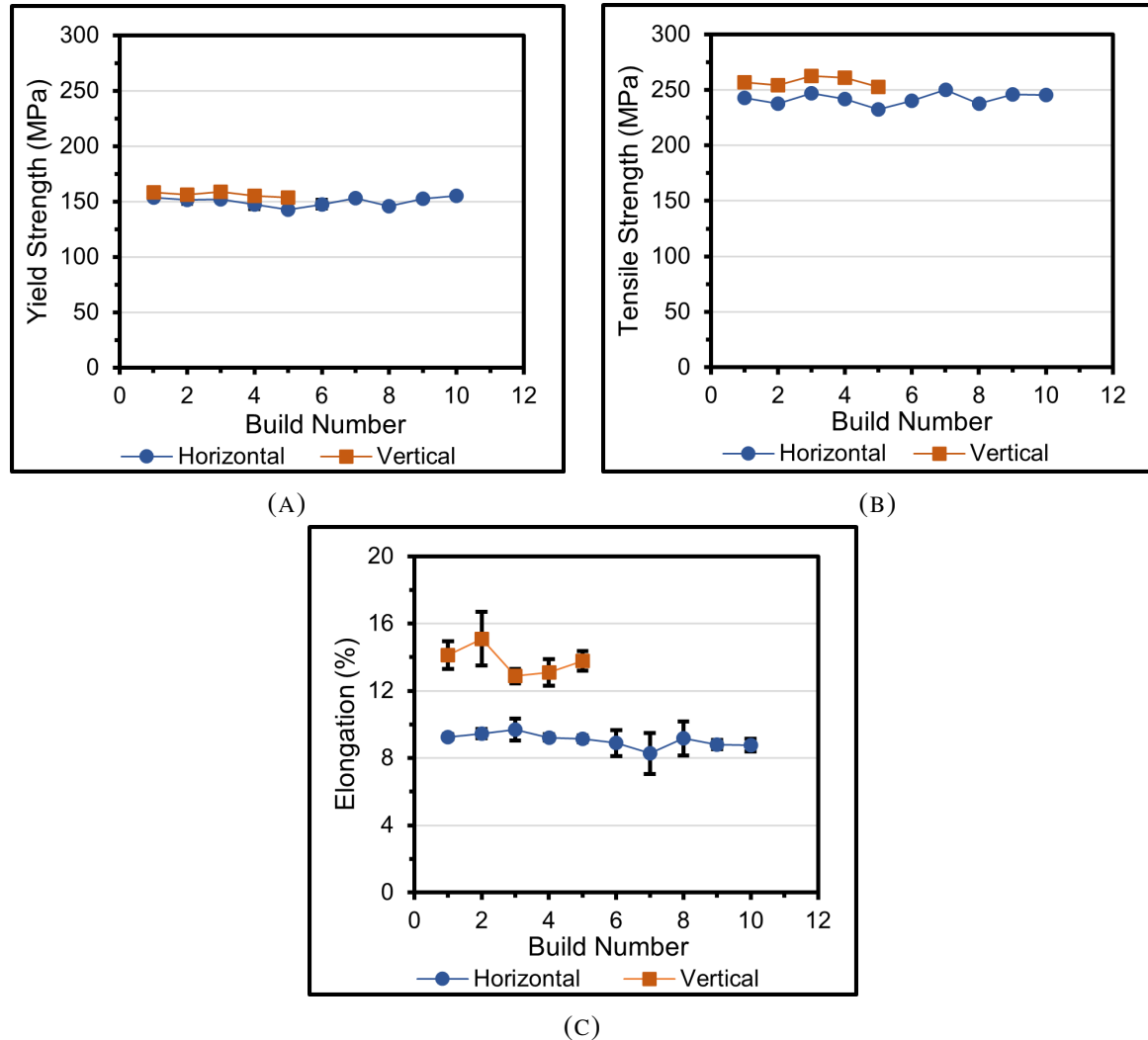


FIGURE 8.24. Tensile properties of parts manufactured with powder reused by the single batch method: (a) yield strength, (b) tensile strength, (c) ductility. Error bars are present on all figures but are too small to be seen for yield and tensile strength plots.

8.3.4.3 Flexural Properties

The flexural properties of parts manufactured with reused powders are shown in Figure 8.25, with vertical specimens tested only for five manufacturing runs due to insufficient powder for further part production. These properties show no change due to powder reuse with all changes small and constantly fluctuating. Part orientation has minimal influence on flexural properties, with the yield strength of horizontal and vertical samples nearly identical and ductility values identical with the exception of S3. Further differences in ductility may, however, arise if

samples are allowed to deform past 7 % strain. The main influence of part orientation was the improved flexural strength of vertically oriented samples. It is also noteworthy that the flexural properties showed remarkable consistency with deviations between samples being minimal, resulting in standard deviations that never exceeded 1% of the property being tested.

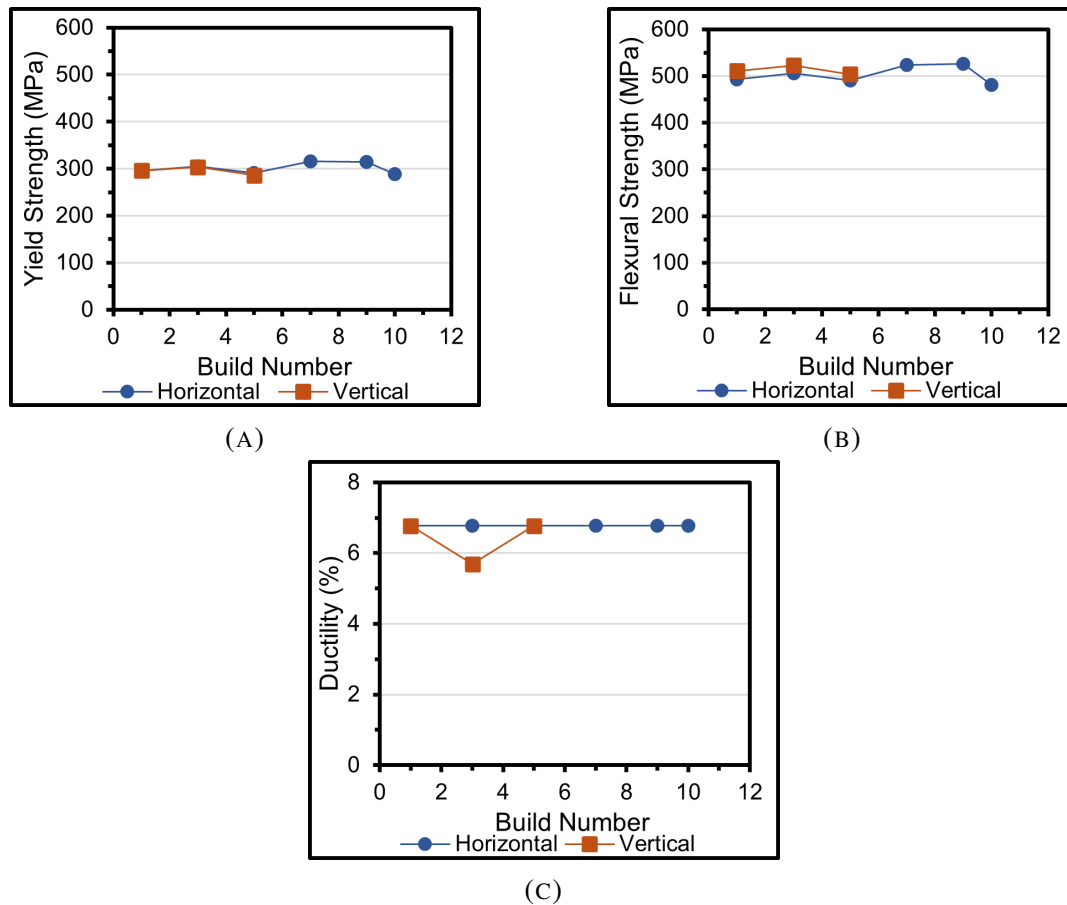


FIGURE 8.25. Flexural properties of parts manufactured with powder reused by the single batch method: (a) yield strength, (b) flexural strength, (c) ductility. Error bars are present on all figures but are too small to be seen.

8.3.4.4 Impact Toughness

The impact toughnesses of samples are shown in Figure 8.26 where it is clear that the strongest influence on impact toughness is the part orientation, with horizontally oriented samples consistently tougher than their vertically oriented counterparts. A slight decrease in the impact toughness is observable for horizontally oriented specimens, with S1 and S2 being the toughest specimens tested, however, any reduction due to powder reuse is slight. ANOVA

analysis, shown in Appendix B2.3, confirms that while there are statistically significant differences between some pairs of values, these do not strongly relate to powder reuse.

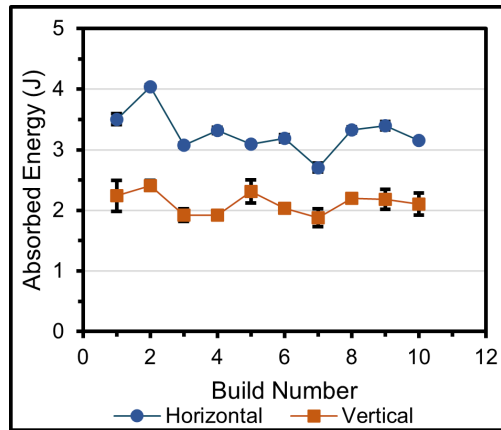


FIGURE 8.26. Impact toughness of parts manufactured with powder reused by the single batch method.

8.4 Discussion

8.4.1 Powder Characterisation

8.4.1.1 Powder Morphology and Flow

As powder is reused by the single batch method, there is a consistently increasing presence of large, rough particles. This is noticeable in SEM micrographs, where S10 powder contains a significantly greater portion of large powders than in the S1 powder batch, which contains a greater proportion of fine powders. The new large powder particles are spatter formed from melt pool ejecta [312], although these spatter particles appear to be considerably rougher than those produced by the refreshing powder reuse method. This, along with the presence of deformed and cracked powders within the S10 batch, suggests that these particles have experienced mechanical damage during sieving and powder spreading over the many manufacturing runs they have been reused in. Examples of these cracked and rough powders are shown in Figure 8.27. Holistically, this results in a degraded powder batch with an increased presence of large particles resulting from powder reuse by the single batch method.

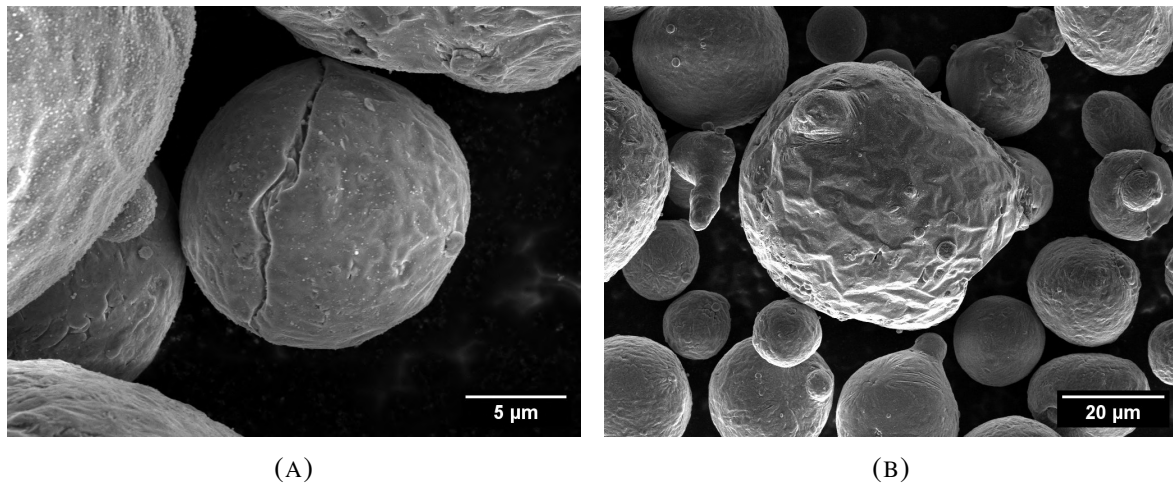


FIGURE 8.27. Examples of misshapen powders that begin to appear in reused powder batches: (a) cracked particles, (b) rough particles.

These changes in the size of powders results in a shift in the PSD. The D10 size of the powder increases over each of the manufacturing runs, rising from 18.3 µm at S1 to 20.8 µm at S10, where in each successive manufacturing run, with the exception of S3, this D10 size increases. This indicates that there is a continual reduction in the presence of fine powder particles in the powder batch, with any newly generated spatter insufficiently small to replace these particles. This is aided by the fact that smaller particles are more likely to be present on the build plate and so are preferentially consumed [200], resulting in an accelerating decrease in their presence in comparison to large particles which are regularly swept into the powder overflow and are continually generated as spatter. Large particles are also seen to continue to increase in presence as per the PSD, with the D90 size consistently increasing from S1 to S5 before stabilising. This stabilisation is due to sieving, with the D90 averaging 61.85 µm between S5 and S10 with a 63 µm sieve used. Sieving removed most particles over 63 µm, thereby limiting the continued increase in large particles in the powder batch.

These changes in the D10 and D90 sizes resulted in the PSD consistently shifting to the right, although the manner of shift is not consistent over each manufacturing run. From S1 to S5 both the D10 and D90 values increase, resulting in a shift of the entire curve but also a widening, seen by the lower peak height. This is as the D90 size increased at a greater rate than the D10 size, increasing the size difference between these two values and so widening the curve. This behaviour changes between S5 and S10, where the PSD curve continues to shift right, however, narrows instead of widens. This is as sieving inhibits the increase in D90

size while the D10 size continues to increase as fine particles continue to be consumed but not replaced in the build chamber, reducing the range between the D10 and D90 sizes and therefore narrowing the PSD. Given the plateauing of the D90 size from S5 onwards, this narrowing of the PSD is expected to continue if this powder is reused further with the single batch method.

These changes in PSD result in improvements in powder flowability, most notably over the first five manufacturing runs. Powder initially did not flow through either the Hall or Carney funnels, indicating very poor flowability, while results from the rotating drum technique showed remarkably different behaviour for S1 compared to other manufacturing runs. This indicates that the initial powder batch was of poor quality, likely due to a high initial moisture content from powder storage that is known to reduce powder flowability [331]. Once powder was able to flow through the Hall and Carney funnels for S5, powder flowability continued to improve, although the flow times for S5 itself were lower than many of the other samples. This trend is due mainly to the reduction of fines, which is known to improve powder flowability during powder reuse [65, 226]. Powder also appears to continue to improve its flowability according to the rotating drum technique, with the avalanche angle consistently decreasing as powder is reused. This is matched by a reduction in the velocity angles of flowing powders at low rotation speeds and is accompanied by an increase in shear thickening behaviour. As fine particles have high cohesive forces between them due to their high surface area to volume ratio [80], the reduction of these fine powders reduces the interparticle friction in the powder batch and so improves flowability.

These powder characteristics interact to result in a steadily increasing powder bed density, with the increase constant from S1 to S9 and no measurement taken for S10 due to insufficient powder to manufacture the required capsules. This increase was most significant from S1 to S4, where the powder bed density increased from 1.48 g/cm³ to 1.54 g/cm³, with powder bed density subsequently increasing to 1.59 g/cm³ by S9. Typically, an increase in powder bed density is attributed to a widening of the PSD, as the broader range of particle sizes present allows small particles to fill the gaps between large particles [255, 332]. Given the widening PSD seen from S1 to S5, this change can be seen as the main contributor to the increased powder bed density in these early manufacturing runs. The continued increase in powder bed density can also be attributed to a widening PSD, although this is obscured in Section

8.3.1.2 as at S10, the D10 value increases significantly while the D90 value decreases slightly in comparison to the previous manufacturing runs. The PSD for S9 follows a trend more similar to that of previous manufacturing runs, as shown in Figure 8.28, indicating that a continued widening of the PSD results in an increased powder bed density. The lower rate of change between S5 and S9 compared to S1 and S5 is evident, with S9 having a closer peak to S5 than S1 does, which indicates a slower increase in powder bed density from S5 to S9 compared to S1 to S5. The other main factor contributing to the changing powder bed density is the improvement in the flowability of powder. Given the initial powder state was poor, the correspondingly poor flowability meant that powder was more readily able to agglomerate during spreading in the build chamber [313, 333], resulting in gaps within the powder bed that reduced the powder bed density. As powder was reused, flowability dramatically improved which resulted in better spreading in the build chamber, with less particle agglomeration resulting in a denser powder bed. These changes were most significant between S1 and S5 according to Hall and Carney funnels as well as the rotating drum method, corresponding to the larger increase in powder bed density over the first four manufacturing runs. While these are the main contributors to the improvement in powder bed density, other factors may also influence powder packing. Powder bed density is also likely increased by a reduction in powder moisture as it is reused by reducing interparticle adhesion [182], as well as a shift to larger particle sizes which generally pack denser than smaller ones [334].

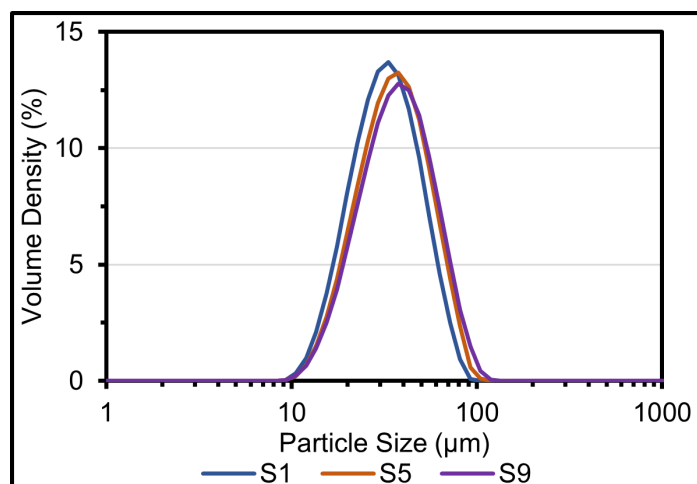


FIGURE 8.28. Particle size distribution of powder reused by the single batch method, represented by the volume distribution of particle sizes. S1, S5, and S9 are shown due to their relevance to powder bed density measurements.

8.4.1.2 Powder Chemistry

The majority of powder chemistry remains consistent in powder reused by the single batch method, with silicon, iron, and titanium levels all showing no meaningful change in bulk chemistry presence. Given AlSi7Mg's reliance on silicon to improve its processability, this indicates that the alloy's solidification range will not change as a result of powder reuse.

The oxygen content of powder increases as powder is reused by the single batch method, however, this trend is not consistent. Powder oxygen content is initially 0.026 wt.% which increases to 0.038 wt.% by S10, with the scatter in the data making the identification of a linear trend impossible. This indicates that the oxygen content of powder is increasing as it is reused, but that measured values are highly dependent on sampling. This oxygen content is increasing due to the accumulation of spatter within the powder batch, as expanded on in Chapter 7.

The change in magnesium content of the powder is of concern, with a reduction from 0.53 wt.% to 0.48 wt.% over the course of ten manufacturing runs. This change is especially significant given the majority of this decrease was seen in the last few manufacturing runs, with the magnesium content decreasing by 0.03 wt.% from S7 to S10. This reduction is due to the preferential evaporation of magnesium from the melt pool, meaning newly generated spatter particles are lower in magnesium content than the powder batch as a whole, which was further elaborated on in Chapter 7. This reduction in magnesium is significant given the strengthening role it plays in precipitation hardening during ageing treatments, with reduced magnesium content lowering the potential growth of precipitates in the strengthening Mg_2Si phase precipitation sequence. While the magnesium levels remain acceptable for the ten manufacturing runs completed in this study, further powder reuse would result in a continued reduction in magnesium and so be of greater concern, as shown in Figure 8.29. This becomes especially pertinent when the collective ageing method is used, with greater potential for powder to be used for an extensive number of manufacturing runs.

The surface chemistry of powders, as expected, is dominated by oxygen due to the reactivity of the main elements of AlSi7Mg, with oxygen varying between 50 wt.% and 56 wt.% of the powder's surface composition. The majority of this oxygen is bound with aluminium as alumina, this being by far the most prevalent compound of the powder surface at approximately

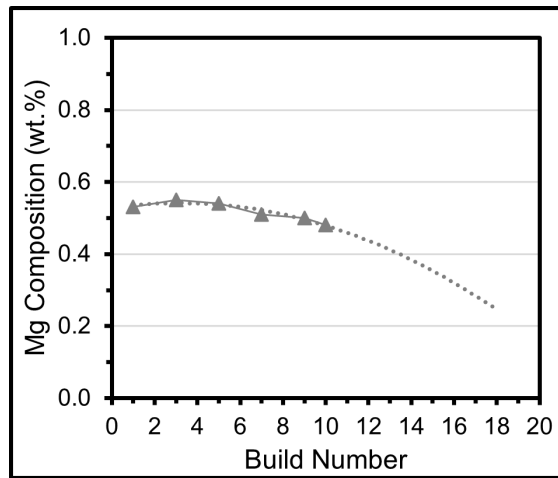


FIGURE 8.29. Powder magnesium content under the single batch powder reuse method, with a projected trend for if powder reuse is continued and magnesium content decreases in a similar manner.

74 wt.%. This also represents the majority of aluminium that is present at the surface. The presence of alumina and oxygen show no significant changes as a function of powder reuse.

The concentration of magnesium oxide does show a significant reduction as powder is reused, dropping by an average of 0.37 wt.% per manufacturing run, a reduction that is consistent no matter the stage of powder reuse. This is in good agreement with the bulk chemistry measurements where the magnesium content is also seen to decrease as powder continues to be reused, although not to this extent.

Interestingly, the surface presence of silica regularly increases as powder is reused, as seen in Figure 8.30, a result not seen elsewhere in the literature. This is not commonly observed as silica is less chemically inclined to form than other oxides such as magnesium oxide, which due to their different free Gibbs energies are more likely to form and thereafter remain stable [194]. It can also be seen that the formation of silica is not at the expense of solely elemental silicon, which reduces as powder is reused but not at the same rate at which silica grows. This increase in silica is therefore likely due to the gradual reduction of other elements on the surface of powder, particularly magnesium, which lowers at a far greater rate than other elements, providing greater opportunity for the silicon on the surface of powders to oxidise.

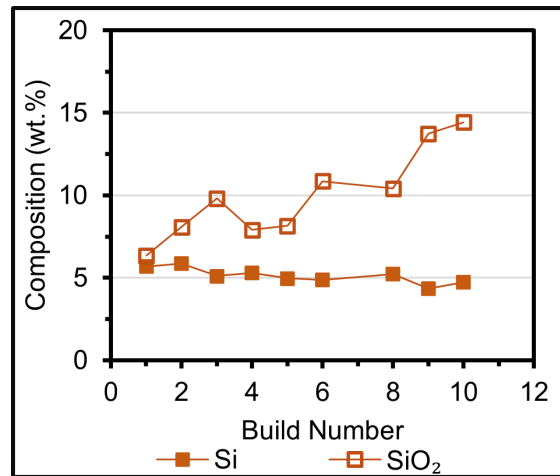


FIGURE 8.30. Powder surface silicon and silica content as a result of powder reuse using the single batch method.

8.4.2 Microstructure

The large-scale microstructure as revealed by dark field optical microscopy shows the fish-scale melt pool morphology typical of PBF parts, with this pattern repeated throughout the sample in the X-Z plane. The consistency of these melt pools indicates that the melting within the build chamber is not influenced by powder reuse, with any changes in surface chemistry and oxide presence not significantly influencing the melting behaviour of the powder.

Grain characterisation via EBSD also reveals that there is minimal change in the grain structure of parts manufactured with powder reused by the single batch method. The small differences in the grain size of each sample are attributable to the standard deviations within measurements, and the differences in maximum texture are also minimal. Each sample tested demonstrates the characteristic columnar structure created by the directional heat flow during cooling, with grains preferentially forming a [100] oriented structure and no difference in this orientation between manufacturing runs. This indicates that the use of powder reused with the single batch method will have no influence on the grain structure of PBF parts. Given the microstructural similarities of parts manufactured with powder reused by the single batch method to the refreshing method, further discussion on the grain characteristics of parts can be found in Chapter 7 of this thesis.

The silicon cell network within these grains also shows no sign of transformation in parts manufactured with reused powders. Still present are the elongated cells within the columnar

grains and the equiaxed cells formed at the melt pool boundaries, with no noticeable change in shape or size developing as a result of powder reuse. Given the microstructural similarities of parts manufactured with powder reused by the single batch method to the refreshing method, further discussion on the cell structure can be found in Chapter 7 of this thesis.

It is clear that the microstructure of AlSi7Mg parts does not change when manufactured with powder reused by the single batch powder reuse method. This shows that the melt pool and solidification behaviour remain the same throughout these manufacturing runs, even given the changes observed in powder chemistry and packing density. This shows good promise for the use of the single batch powder reuse method both academically and industrially.

8.4.3 Part Properties

8.4.3.1 Part Density

The density of parts manufactured by PBF techniques is one of their most important and commonly investigated properties, with production parameters often optimised to maximise density [23]. This remains true for parts manufactured with reused powders. Relative density as measured by the Archimedes method shows no change according to powder reuse, with values varying between 99.21 % and 99.31 %. While the standard deviations within each manufacturing run were low, with the exception of S1 whose deviation encompassed the values of all other measurements, the inconsistency and lack of trend in relative density measurements suggests these were influenced more by the manual measuring method employed than any trend in the relative density of samples themselves. This means any changes, or lack thereof, to density could not be effectively seen using the Archimedes density measurement technique.

Contrastingly, the relative density measurements according to μ -CT show a gradual increase as powder is reused. While this change is slight it is consistently present in all manufacturing runs tested, increasing from 99.88 % to 99.94 % over ten manufacturing runs. This is primarily due to the increase in powder bed density observed as powder is reused, with a denser powder bed improving part density due to the increased volume of material available for melting, resulting in a reduction in part porosity [335]. Powder bed density measurements were seen to increase continually as powder was reused, with this trend most noticeable over the first five manufacturing runs. These results correlate well with those for the relative density of parts,

which also increased most significantly over the first five manufacturing runs, suggesting a strong correlation between these factors.

8.4.3.2 Surface Roughness

The surface roughness of parts manufactured with powder reused by the single batch method shows that roughness is dependent on the measurement location, with the most noticeable difference being between the top surface and the front and side surfaces of parts. As with parts manufactured with powder undergoing the refreshing powder reuse method, top surfaces were considerably rougher than both front and side surfaces. Given this similarity to past results, further discussion on this difference can be found in Chapter 7 of this thesis. While the front surfaces appear to have slightly higher roughnesses than side surfaces, the regularly intersecting standard deviations of these measurements makes the difference between these surfaces statistically insignificant.

The surface roughness of parts shows minimal relation to the reuse status of powder, with no trend evident that relates the two. This indicates that the changes seen in the PSD by the reduction of fine particles and increase in large particles were not great enough to influence the surface roughness, even though a changing PSD is often linked to a changing surface roughness [178, 231]. This indicates that the single batch powder reuse method will not significantly change the surface roughness of parts.

8.4.3.3 Part Chemistry

The chemistry of parts manufactured with powders reused by the single batch powder reuse method shows no change in the majority of elements, with silicon, iron, and titanium all showing no change over ten manufacturing runs. This indicates that the expected phase formations and processability improvements brought by these alloying elements will not change with continual powder reuse.

The oxygen content of parts does, however, gradually increase as powder is reused, rising from 0.011 wt.% at S1 to 0.022 wt.% by S10. This doubling of part oxygen content over ten manufacturing runs is significant, however, there was a slight decrease from 0.024 wt.%

observed from S5, suggesting that the majority of oxygen pick up occurs in the initial manufacturing runs.

As seen in Figure 8.31a, while both powder and part oxygen content increase with continual powder reuse, there is only a slight correlation between the two. This is as the increasing powder oxygen content shows much greater variability than the part oxygen content, with considerable scatter between measurements suggesting that the variability inherent with sample selection plays a role in the oxygen content of a particular powder sample. While there is only a slight correlation between the two trends, the majority of the part oxygen content increase can be attributed to the increasing powder oxygen content. This is as other means of oxygen pick up, such as an increase in powder bed density, are not seen during the single batch powder reuse method and so cannot be attributed to increased part oxygen content.

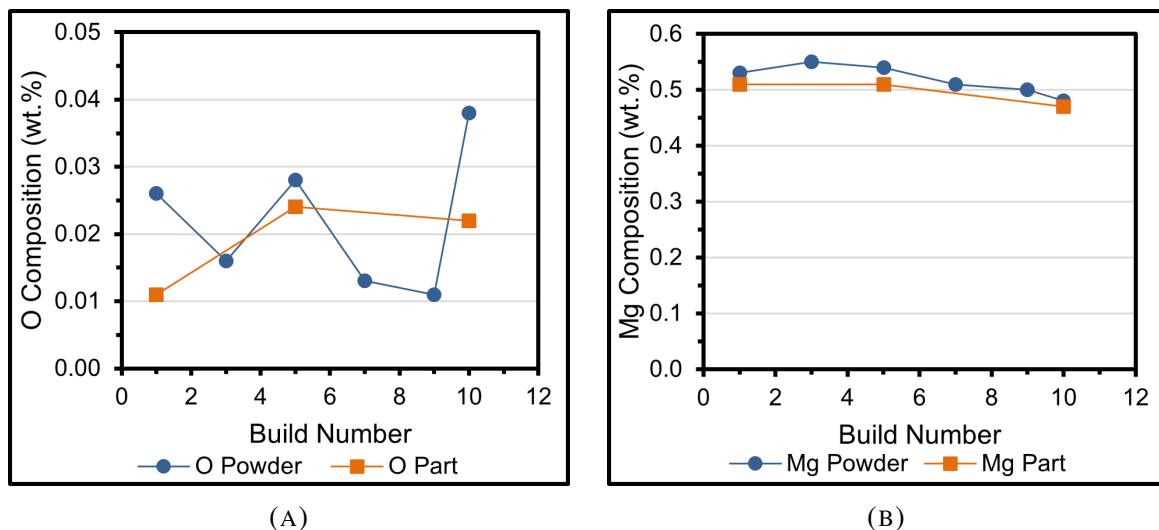


FIGURE 8.31. Comparison of the chemical composition of parts and powders from the single batch reuse method: (a) oxygen content, (b) magnesium content.

Also of note is that the part oxygen content is often lower than that of the powder oxygen content, with this true for every manufacturing run where part oxygen content was measured. This suggests that during processing some of the oxygen in the powder is lost, as seen in other studies, due to the dissociation of oxides within the melt pool, resulting in the loss of oxygen and possibly carbon oxides [336]. Additional oxygen acquired from the build chamber atmosphere during melting is not sufficient to replace the oxygen lost from the

powder, resulting in an oxygen content lower in the parts than in the powder. However, given the variability of powder oxygen content, further data points are required to confirm this trend.

Part magnesium content is seen to decrease with continual powder reuse by the single batch method, decreasing from 0.51 wt.% to 0.47 wt.% from S1 to S10. This effect can be strongly tied to the magnesium content of the powder, which follows the same trend as visible in Figure 8.31b. The reduction in magnesium content accelerates after S5, which is also matched by the powder's magnesium concentration. Also significant is that the part magnesium content is consistently lower than that of the powder. This is to be expected, with the preferential evaporation of magnesium within the melt pool a common occurrence [4, 6, 103] and regularly seen within this research. Longer term powder reuse than that in this study may therefore result in unacceptable losses of magnesium, particularly if initial concentrations are low. This is especially likely to become problematic when the collective ageing method is used, and so careful monitoring of part and powder magnesium content is recommended.

8.4.4 Mechanical Properties

8.4.4.1 Hardness

The hardness of samples manufactured with powder reused by the single batch method showed a slight change from S1 to S3, increasing by 8.0 HV, with this being the only noticeable change related to powder reuse. Even though this increase is evident, given the significant error associated with the S1 measurement and the stabilisation of hardness after S3, it is likely that this trend is only apparent due to this large deviation. Therefore, the hardness of parts manufactured with powder reused by the single batch method shows no change as a result of powder reuse.

8.4.4.2 Tensile Properties

As with parts from the refreshing powder reuse study, the vertically oriented parts manufactured with powder that had been reused according to the single batch method performed noticeably better under tensile testing than horizontally oriented specimens did. Again, this was due to the presence of residual support structures on the gauge sections of these horizontal tensile samples, with the harsh edges of some of these supports initiating premature failure

and so lower ductility, yield, and tensile strengths. Further discussion on this can be found in Chapter 5 of this thesis.

The tensile properties of parts manufactured with powder reused by the single batch method show no change as a function of powder reuse, with this true for both horizontally and vertically oriented samples. While at some stages during powder reuse strengths reduce, with the yield strength of horizontal specimens reducing 6.25% from 152.0 MPa to 142.5 MPa between S3 and S5, this is followed by a subsequent increase to 153.0 MPa two manufacturing runs later, negating any previous changes. Similar fluctuations occur in each property tested with no relation to powder reuse, indicating that reused powder has no effect on the tensile properties of AlSi7Mg.

While this is true for the stress relieved samples tested here, the same may not be true of T6 samples tested with powder reused by the single batch method. This is due to the reduction in magnesium content evident in both powders and parts, which plays a key strengthening role in T6 treated AlSi7Mg as seen in Chapter 6. A continued reduction in magnesium may result in the reduced growth of strengthening precipitates during heat treatment, lowering yield and tensile strengths. Further investigation should therefore be carried out on T6 treated samples manufactured with powder reused by the single batch method.

8.4.4.3 Flexural Properties

The flexural properties of AlSi7Mg are slightly influenced by part orientation, with the flexural strength of vertically oriented parts slightly greater than those of their horizontally oriented counterparts. The yield strength and ductility, however, have no relation to part orientation. The difference in flexural strength follows an unusual pattern, given past findings are typically that horizontally oriented parts are stronger than vertically oriented ones [337], seen most commonly in tensile testing [1, 39, 90, 323]. This is as the columnar grains of PBF microstructures are preferentially oriented along the build direction, i.e. the [100] direction, meaning the loading of horizontally oriented specimens is across the shorter axis of these grains, increasing strength due to both grain size and dislocation network effects [338]. A similar logic can be followed for the cell structure given these cells follow the orientations of the grains they reside in, while other strengthening factors such as solute strengthening, and surface roughness are orientation agnostic. Given these factors cannot explain the higher

strengths of vertically oriented samples, further microstructural investigation is required, however, this lies beyond the scope of this research. Since the differences between the horizontal and vertical flexural strengths are small, the largest being 18.6 MPa, a difference of 3.6%, this is also unlikely to be an issue for most applications. This is especially true given the yield strengths show no difference according to part orientation.

Throughout the course of powder reuse, no difference can be seen in any of the flexural properties due to the use of reused powders. While there is some deviation within the data, with a noticeable increase in both yield and flexural strengths for the horizontal samples for S7 and S9, the consistency of the other samples indicates these results are more likely natural deviations as a result of factors other than powder reuse. This is especially true given the flexural properties of S10 return to levels similar to those of S1 after the increases associated with S7 and S9. These results show that the single batch powder reuse method has no effect on the flexural properties of parts.

8.4.4.4 Impact Toughness

The main influence on the impact toughness of parts manufactured with powder reused by the single batch method is part orientation, with horizontally oriented specimens showing impact toughnesses approximately 35% greater than those of vertically oriented specimens. This is due to the orientation of melt pool boundaries along the horizontal plane, i.e. the plane of fracture for vertically oriented samples, which reduces the tortuosity of the fracture path for vertical samples, thereby reducing their impact toughness. This concept is elaborated on in Chapter 7 of this thesis.

The single batch powder reuse method shows only a minimal relationship with the impact toughness of parts, with a weak trend observable mostly in horizontally oriented samples. Samples manufactured with newer powder can be seen to have slightly higher impact toughnesses than those manufactured later in the reuse cycle, with the toughest two samples tested being S1 and S2. While this is true, many of the later samples, although less tough than S1 and S2, still show adequate toughnesses only slightly smaller than that of these initial values. This suggests that while there may be a slight decrease in impact toughness as a result of the single batch powder reuse method, these changes are small enough that they are unlikely to influence part properties more than the natural scatter associated with this testing.

8.5 Comparing the Refreshing and Single Batch Powder Reuse Methods

8.5.1 Powder Characterisation

8.5.1.1 Particle Size

While both the refreshing and single batch powder reuse methods result in changes to the PSD of a powder batch, the extent to which this occurs differs, as seen in Figure 8.32. The refreshing method results in a gradual increase in the D10 and D50 values until R7, as in Figure 8.32a and 8.32b, due to the continual introduction of new powder that replaces the fine particles consumed in previous manufacturing runs. The single batch method, however, results in much steeper increases in the D10 and D50 values as these lost fine particles are not replaced. This is as these fine powders are continually consumed while large ones are produced as spatter [312, 318], increasing the values of all powder size measurements. The D90 size of particles is less affected by the powder reuse method chosen, although the rate of increase in D90 size is still slightly higher for the single batch than refreshing method as the concentration of fines in the powder batch is reducing at a faster rate. However, both powder reuse methods see a gradual increase in the D90 size until approximately the fourth manufacturing run, where this value stabilises. This stabilisation is due to the use of a 63 μm sieve, which removes the majority of particles larger than this. Some particles larger than 63 μm are able to remain in the powder batch if the shorter of their axes is lower than this sieve screen size, with their high aspect ratios allowing them to pass through the sieve. This indicates that the sieve size will have a greater influence on the upper bound of a powder batch's PSD than the powder reuse method selected.

The volume distribution of the PSD is shown in Figure 8.33 with a clear distinction between the changes in powder batches reused by the refreshing and single batch powder reuse methods, even given their different initial forms. Powder reused by the refreshing method showed a slight increase in the width of the PSD due to the large increase in D90 value until R7, with the increase in D10 value more gradual and also stopping by R5. This is seen by the slight decline in the maximum peak height of the PSD distribution. Powder reused by the single batch method shows similar initial behaviour, with the rapid increase in the D90 value

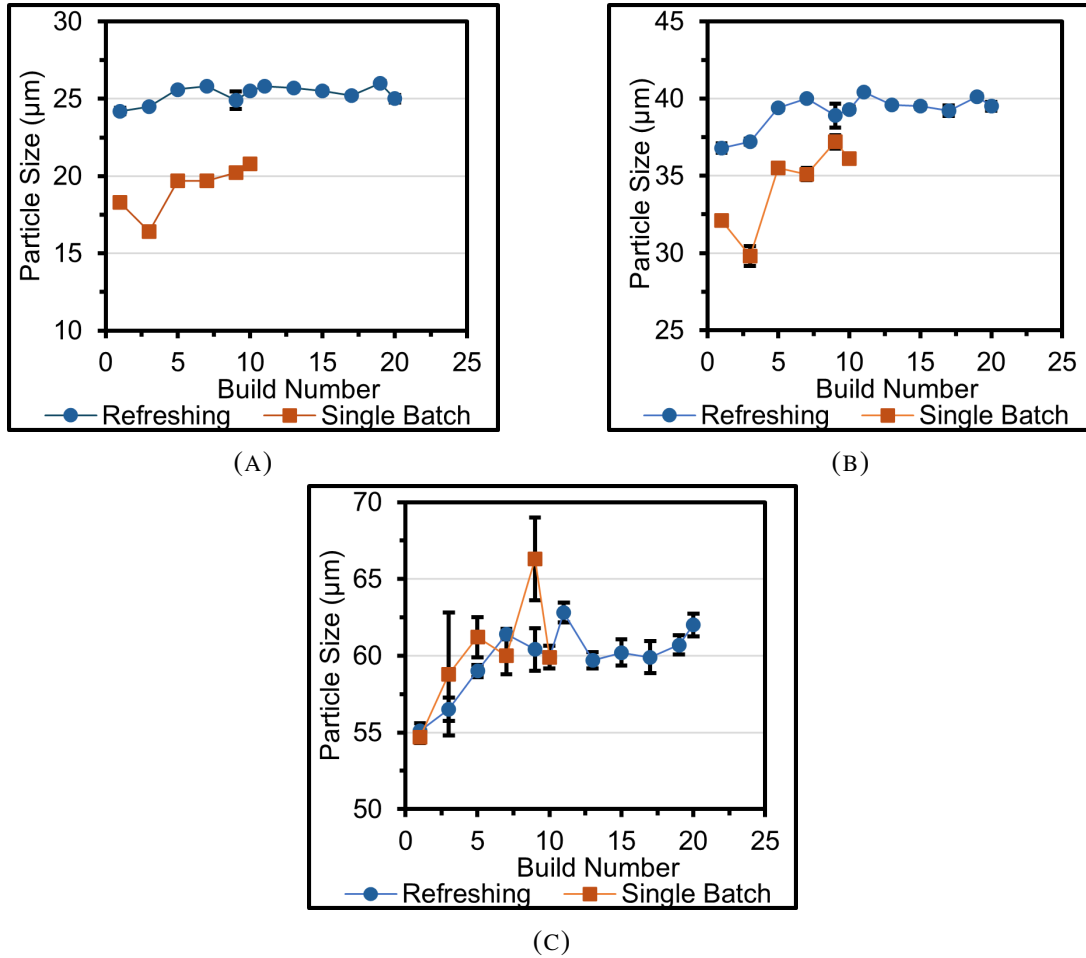


FIGURE 8.32. Comparison of the particle size distribution values for the refreshing and single batch powder reuse methods: (a) D10, (b) D50, (c) D90.

having a greater influence on the width of the PSD than the increasing D10 value, resulting in a wider PSD. However, once the D90 value stabilises and the D10 value continues to increase the PSD curve narrows, as in Figure 8.33. This implies an inflexion point exists for the single batch method, where the initial widening PSD behaviour will shift to narrowing upon the stabilisation of the D90 value.

The PSD of a powder batch can therefore be seen to be influenced by the powder reuse method employed. The refreshing powder reuse method results in a slight increase in all powder size measurements, with this increase gradual and stabilising after a certain number of manufacturing runs, in this case seven. This is due to the sieve size limiting the influence of large particles, while the continual introduction of unused powder limits the reduction in fines. Contrastingly, the single batch method results in a continual increase of all powder size

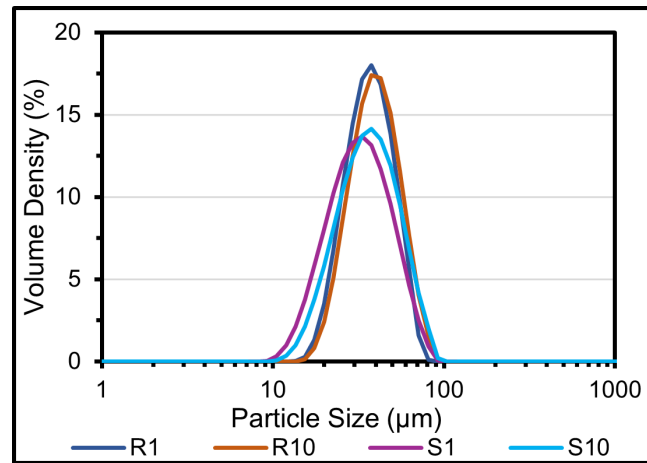


FIGURE 8.33. Volume distribution of the particle size distribution of powder reused by the refreshing and single batch methods initially (R1 and S1) and after ten manufacturing runs (R10 and S10).

measurements, restricted only by the sieve size. This results in a widening then narrowing PSD, which given the continuity of change in the D10 and D50 sizes, would likely continue to narrow in further powder reuse beyond the ten manufacturing runs investigated here. The refreshing powder reuse method, therefore, can be seen to better preserve the initial particle size characteristics of a powder batch than the single batch method.

8.5.1.2 Powder Flowability

No matter the powder reuse method used, powders demonstrated an improvement in flowability with continual reuse. As discussed in Section 8.4.1.1 and Chapter 7 of this thesis, this is due largely to the reduction of fine particles and moisture as powder continues to be reused within a heated build chamber. According to the Hall flow data in Figure 8.34a, this change is most evident in the first five manufacturing runs, which saw a decrease in flow time of 20.3% from R1 to R4 during refreshing powder reuse, and an improvement from initially non-flowing behaviour to freely flowing powder for the single batch powder. Similar can be said of the avalanche angle of powders as in Figure 8.34b, which showed most improvement for the refreshing powder batch between R1 and R4, while the single batch powder continuously improved its flowability. This behaviour can be linked to the PSD of the powder batches, with a narrow PSD and large particles improving flowability [57, 58]. This means that powders reused by the single batch method will continue to improve their

flowability due to the continual reduction in fines as well as the decreasing PSD width after the ninth manufacturing run, as seen by the continually improving Hall flow times and avalanche angles. This trend is not evident in powders reused by the refreshing method as changes in PSD are slower due to the continual introduction of unused powder, with the proportion of fines better conserved. This means that further changes in powder flowability are unlikely. The single batch powder reuse method can therefore be seen to improve the flowability of powders to a far greater extent than the refreshing powder reuse method, which is potentially of great benefit if initial powder flowability is poor.

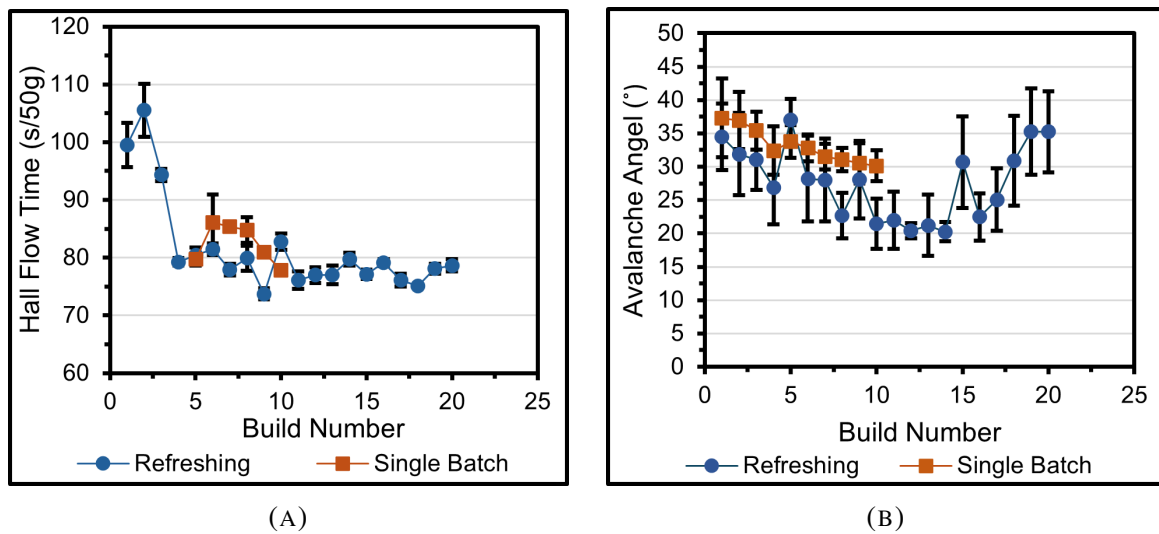


FIGURE 8.34. Powder flowability measurements of powders reused by the refreshing and single batch methods: (a) hall flow time, (b) avalanche angle.

8.5.1.3 Powder Bed Density

The powder bed density of reused powder batches is reliant on the powder reuse method used, with the single batch method creating far more significant changes than the refreshing method. This is shown in Figure 8.35, where the powder bed density is seen to consistently increase for the single batch method, whereas the refreshing method shows no change due to powder reuse. The increase evident in the single batch powder is due to the widening PSD over the first nine manufacturing runs and the constant increase experienced by every measure of particle size, with large particles being known to pack denser than fine particles [334]. This is in contrast to the refreshing method where no change in the powder bed density can be seen, with the smaller changes in PSD and particle flowability insufficient to influence powder

bed density to any significant degree. This suggests that the powder bed density will be more strongly influenced by the single batch powder reuse method, although this may also be due to the poorer initial powder quality for the single batch method. Further testing with either longer term reuse of the single batch powder or testing powders of the same initial quality would help elucidate this hypothesis.

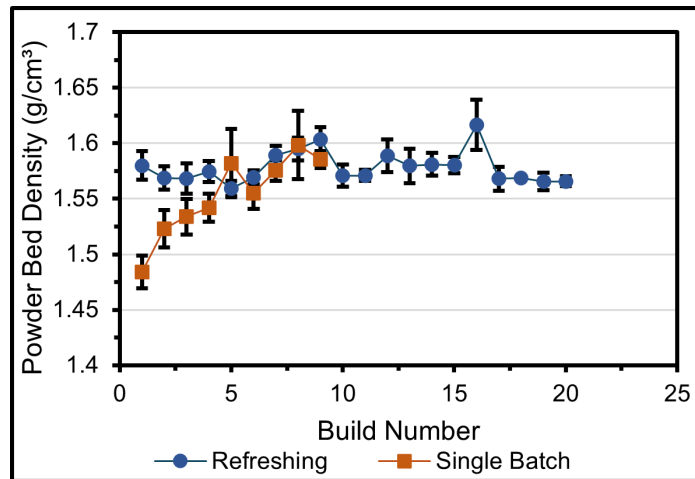


FIGURE 8.35. Powder bed density as a function of powder reuse for the refreshing and single batch methods.

8.5.1.4 Powder Chemistry

The oxygen content of reused powders continuously changes as powder is reused, although these results are obscured in the refreshing powder samples by the initial high oxygen contents caused by long term storage. The single batch method shows a slight increase in powder oxygen content as it is reused, however, there remains considerable scatter within the data suggesting that these changes may be as much a function of random sampling as a genuine trend relating to powder reuse. These results differ compared to those of other powder reuse studies which generally report an increase in powder oxygen content for both refreshing [172, 245] and single batch powder reuse methods [166, 171, 215, 217], seen across a variety of material types. Given the minimal change in the oxygen content of single batch powder and the lack of change seen in later stage refreshed powder, as evident in the last refreshing data points in Figure 8.36a, it appears that careful powder management can be a way to reduce the oxygen pick up of reused powder. This is especially true given these powders were AlSi7Mg, where the reactivity of aluminium makes this alloy especially prone to oxidation.

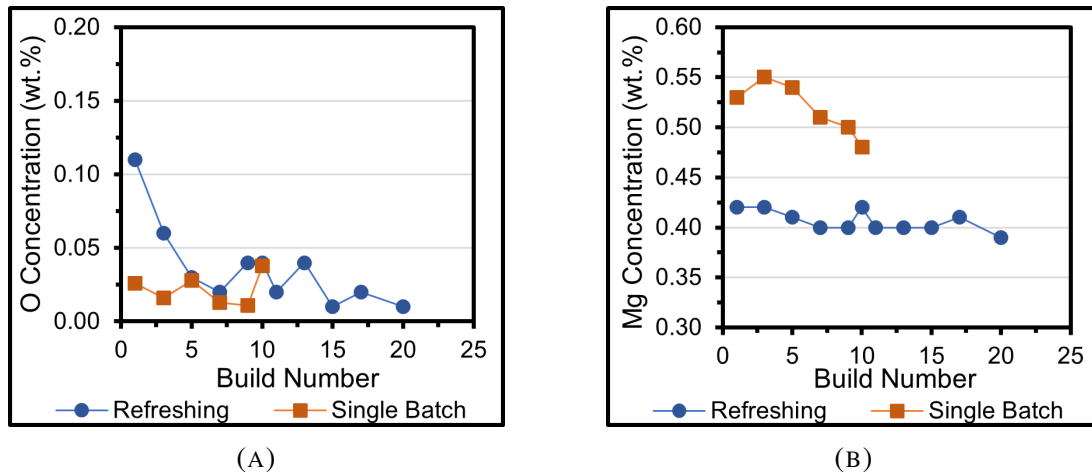


FIGURE 8.36. Powder bulk chemistry as a function of powder reuse for the refreshing and single batch methods: (a) oxygen content, (b) magnesium content.

The magnesium content of powders during reuse according to Figure 8.36b appears to be of greater concern than oxygen content, with both powder reuse methods seeing a reduction in magnesium content. For the refreshing powder reuse method this reduction is minimal, although the consistency of decline and its lowering to below the alloy composition limit of 0.4 wt.% means this remains concerning. For the single batch reuse method, the decline in magnesium content occurs significantly faster than for the refreshing method, falling 9.4% over ten manufacturing runs at an accelerating rate. This will likely reduce the strengthening of AlSi7Mg manufactured with powder reused by this method due to its reliance on the formation of magnesium rich precipitates, which a reduction in magnesium content will inhibit. These differences occur as reused powder continually loses magnesium by its evaporation from the melt pool and therefore has a reduced presence in spatter. With the continual addition of new powder by the refreshing method, which is magnesium rich in comparison to spatter, this reduction in magnesium content is slowed. As this refreshing does not occur during the single batch method, the decline in powder magnesium content is more significant. This means that for powders with either low initial magnesium contents or parts undergoing heat treatments reliant on magnesium, the continual use of the single batch method to reuse powder is not recommended, with the refreshing powder reuse method recommended instead.

8.5.2 Part Microstructure

The microstructures of parts manufactured with reused powders from both reuse methods demonstrated great consistency no matter the powder age. Every part showed the characteristic fish-scale melt pool structure typical of PBF parts, with no noticeable changes between manufacturing runs. Examples of these are shown in Figure 8.37a and 8.37d. This indicates good consistency of melting throughout the structure, regardless of changing powder characteristics such as surface chemistry and powder bed density. This is especially promising given that changing powder size can affect powder bed laser absorptivity [339] and therefore melting behaviour, however, with no changes observable in these results it is clear that the small changes to powder due to reuse did not significantly affect its melting.

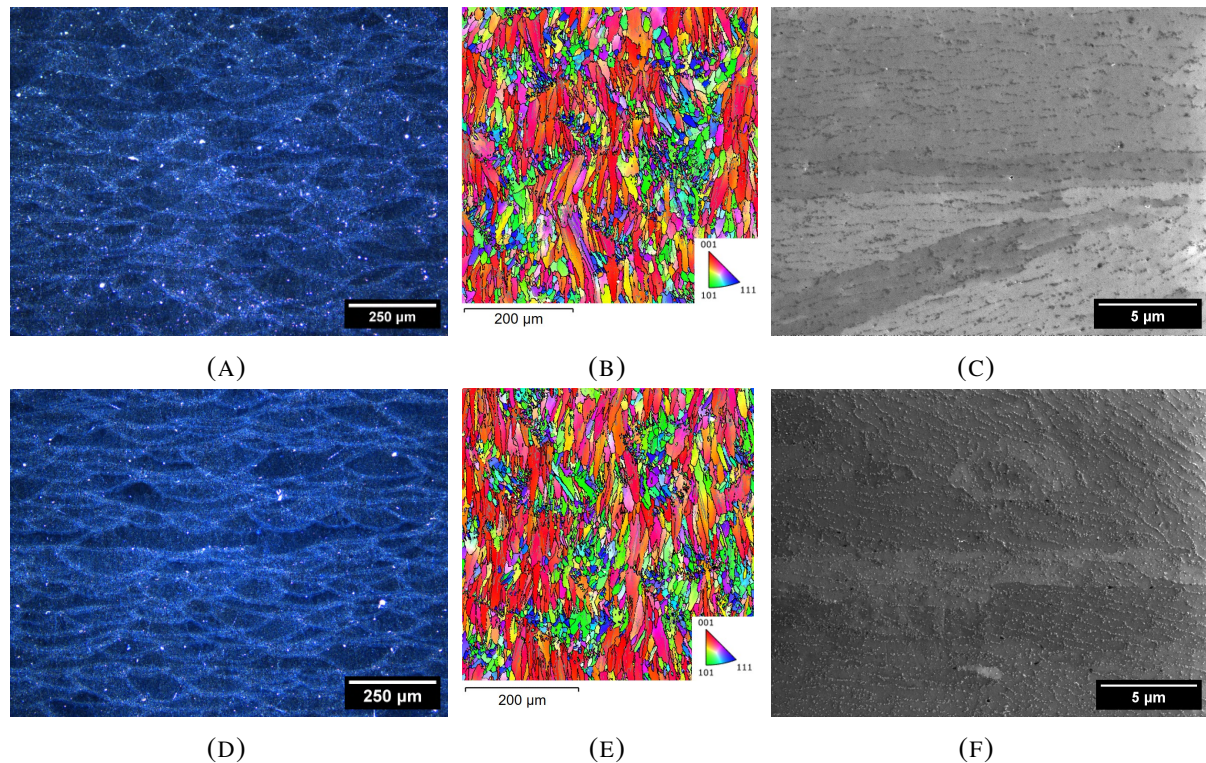


FIGURE 8.37. Comparison of the microstructural features of parts manufactured with powder reused by the refreshing and single batch methods: (a) optical micrograph of R10, (b) IPF map of R10, (c) SEM micrograph of R10, (d) optical micrograph of S10, (e) IPF map of S10, (f) SEM micrograph of S10.

The same is true regarding the grain structure of parts manufactured with reused powders, with neither powder reuse method inducing any change to grain size, shape, or texture. Examples of

these grain structures are displayed in Figure 8.37b and 8.37e. This consistency is observable even given slight changes in chemistry, with changing levels of oxygen and magnesium not affecting solidification behaviour. This suggests that the powder reuse method employed will not influence the grain structure of PBF AlSi7Mg.

This lack of change in the grain structure also results in no changes in the formation of the silicon cell network within these grains. Examples of these cell networks are shown in Figure 8.37c and 8.37f. These cell networks are consistently formed by fine silicon particles that precipitate along the cell boundaries, forming both elongated and equiaxed cells depending upon their location within the melt pool. Although these silicon precipitates are coarsened by the stress relief applied to samples prior to microstructural investigation, given the consistency of larger scale microstructural features and these coarsened precipitates, it is likely that this stress relief does not affect the cell structure differently according to powder reuse state.

It can therefore be seen that the powder reuse method will not have any effect on the microstructure of parts manufactured with reused powders. Both the refreshing and single batch methods can therefore be used in manufacturing applications while allowing for microstructural consistency.

8.5.3 Part Properties

8.5.3.1 Part Relative Density

The relative density of parts manufactured with powders undergoing different powder reuse regimes were seen to be different, as seen in Figure 8.38. The refreshing method saw a decrease in part density with the continual accumulation of foreign particles in the powder batch increasing the presence of keyhole and LOF pores, in contrast to the single batch method where the consistent increase in powder bed density improved part density. This implies that the rapid increase in particle size experienced by the single batch method outpaced any accumulation of foreign particles and spatter that acted to reduce part density, however, later stage reuse would likely see this trend reverse. The differences in initial relative density values likely stem from the different powder properties of the initial powder batches, as mentioned in Section 8.5.1, with the poorer flowability and increased presence of fine particles in the single batch powder resulting in a lower powder bed density and therefore lower relative density.

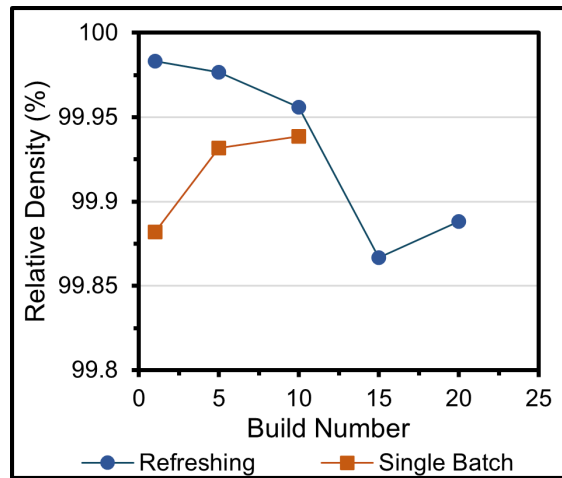


FIGURE 8.38. Relative density as a function of powder reuse by both the refreshing and single batch methods, as measured by micro computed tomography.

Of special concern is the accelerating rate of decrease in part density within the refreshing parts, with this most evident between R10 and R15. While the relative density for R20 shows some recovery, further testing with powder reused for longer times would be beneficial in determining the potential long-term behaviour of this trend.

While the relative density of parts manufactured with powder reused by the single batch method continues to improve, it is noteworthy that it does not reach a level where it is higher than that of parts manufactured with powder reused by the refreshing method. This is due to the initially poor quality of the single batch powder in comparison to the refreshing powder, and so further testing to fifteen manufacturing runs would be beneficial for the single batch material to understand if the trending increase in part density continues, allowing for a comparison with the decrease in relative density experienced between R10 and R15.

Given these changes and their consistency, it is clear that the single batch method shows more desirable behaviour in regard to part density as powder is reused. While this may be influenced by the relatively poor quality of the initial powder used in single batch investigations, the continual reduction in part density for parts manufactured with powders reused by the refreshing method also implies this superiority. Therefore, the single batch method is seen to be a better choice of powder reuse method when considering part density. It should also be noted, however, that each sample tested retained acceptable values of relative density, always being above 99.85%.

8.5.3.2 Surface Roughness

The surface roughness of parts showed good consistency as a function of powder reuse, regardless of the location at which this was measured, as shown in Figure 8.39. The behaviour of surface roughness was also consistent for each sample measured, with the surface roughness of the top of samples being considerably more variable than for those of the front and side. This indicates that the roughness of parts is most strongly influenced by part location and processing parameters, given the top surface of parts was found to be rougher due to its reliance on hatch spacing rather than layer thickness, as for the side and front surfaces. Therefore, the surface roughness of parts can be seen to not be influenced by powder reuse, meaning that both the refreshing and single batch methods can be used without having any detrimental effect on part surface roughness.

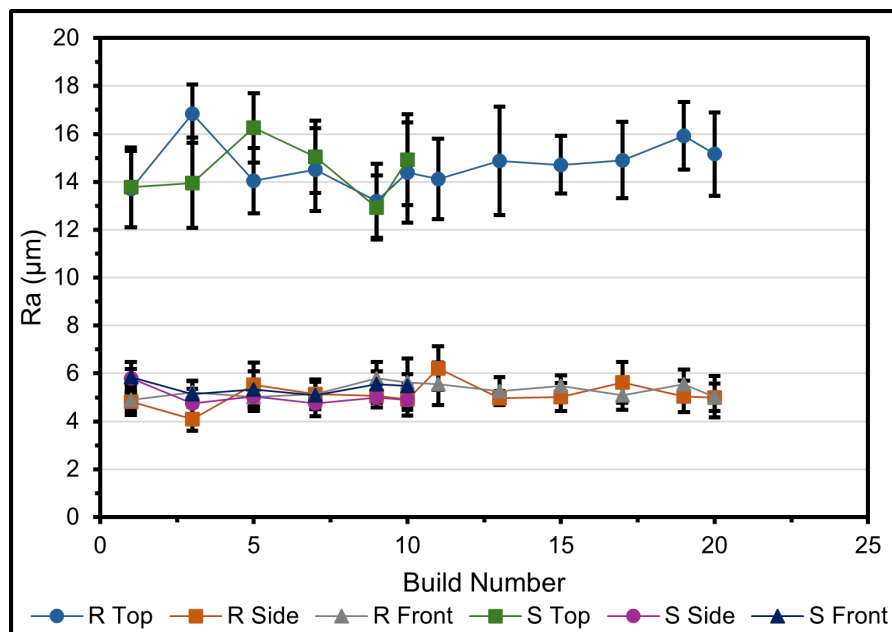


FIGURE 8.39. Part surface roughness as a function of powder reuse for the refreshing and single batch methods. Given sloped surface roughness measurements were not taken for the single batch method, only measurements from cubes are compared here.

8.5.3.3 Part Chemistry

The majority of the chemistry for parts manufactured with powders reused by the refreshing and single batch methods was consistent over all the manufacturing runs tested, with silicon,

iron, and titanium contents all seeing no change. This indicates that powder reuse will not affect the majority of powder chemistry for AlSi7Mg, showing good promise for continual powder reuse.

The oxygen content, as seen in Figure 8.40a, continues to increase as powder is continually reused for both the refreshing and single batch methods. Most noticeable is that the oxygen content of parts match almost identically regardless of the manufacturing run tested, with both powder reuse methods showing identical trends. This is likely due to the similarities in powder oxygen contents, which for the fifth and tenth manufacturing runs were nearly identical for both powder batches.

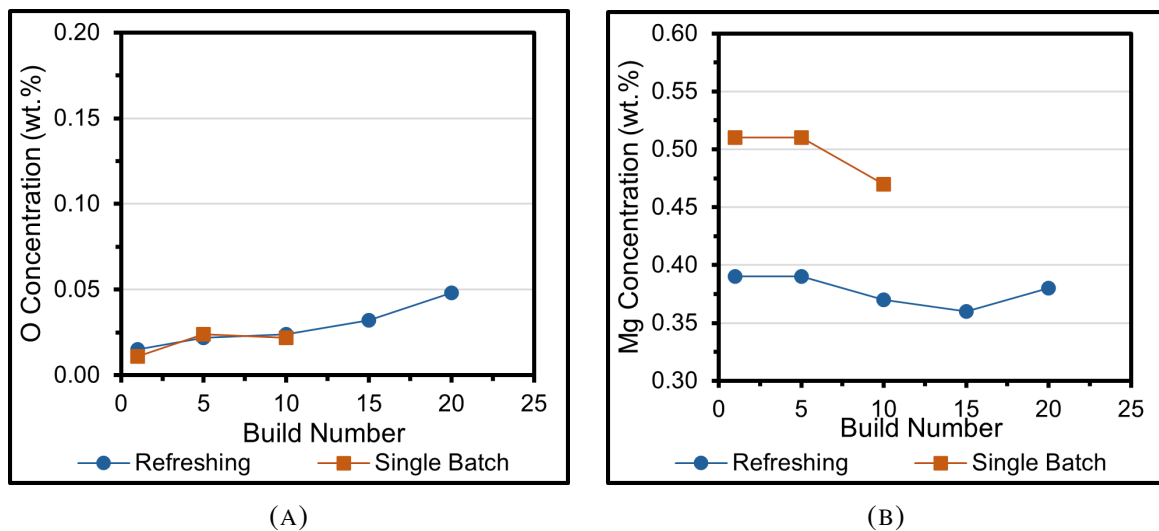


FIGURE 8.40. Part chemistry as a function of powder reuse for the refreshing and single batch methods: (a) oxygen content, (b) magnesium content.

Oxygen accumulates within the powder batch by two mechanisms; the accumulation of spatter within the powder bed, and the exposure of preexisting powder to oxygen while in the build chamber or during powder handling procedures such as sieving. As the volume of parts produced in each manufacturing run was consistent throughout both powder reuse studies, at 18% of the build volume, the relative volumes of spatter generated also remained consistent. Given the total oxygen contents of both powder batches were nearly identical for the manufacturing runs tested and this spatter generation was constant, it follows that the oxidation of powder due to exposure was also consistent for both powder reuse methods employed. This is even though the refreshing powder reuse method continually introduced unused powder to the powder batch, which theoretically has a lower oxygen content than

the powder used in previous manufacturing runs. This suggests that the oxygen pick up of AlSi7Mg is minimal, with the introduction of new powder having minimal effect on the oxygen content of the powder batch as a whole. It should, however, be noted that this result is reliant on careful powder handling procedures that minimise powder exposure to atmosphere, reducing the chance of oxygen pick up.

This consistency in powder oxygen content results in a regular increase in part oxygen content, with minimal reliance on the powder bed density and the corresponding entrapped gasses. The choice of the refreshing or the single batch powder reuse method can therefore be seen to have minimal impact on the trend of part oxygen content as powder is reused. However, both methods result in a gradual increase in part oxygen content which must be monitored, especially when powder is used for a greater number of manufacturing runs than tested in these studies.

The magnesium content of parts is another aspect of concern when reusing powder, with both the refreshing and single batch powder reuse methods resulting in a consistent reduction in part magnesium content, as shown in Figure 8.40b. This is especially pertinent for the single batch method, which shows a rapid reduction in part magnesium content compared to that of the refreshing method. This can again be tied to the powder magnesium content, with Figure 8.41 showing that the trend for magnesium content in parts and powders is identical. This shows that part magnesium must be regularly monitored in order to maintain the magnesium levels required for strengthening during heat treatment. A comparison of these magnesium contents shows that the refreshing powder reuse method shows much greater potential for long term powder reuse than the single batch method, due to the significantly slower reduction in part magnesium content.

Both powder reuse methods also demonstrate a reduction in magnesium content during processing, with part magnesium content consistently lower than that of the powder. This makes magnesium content monitoring an important consideration no matter the powder reuse method used.

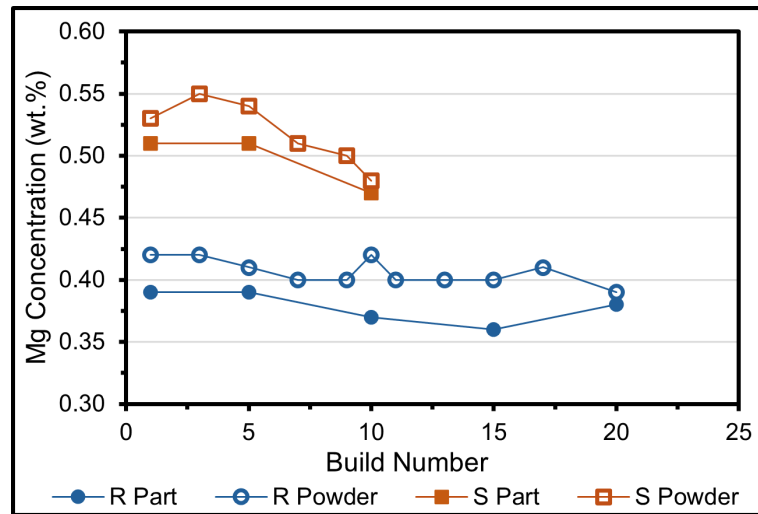


FIGURE 8.41. Part and powder magnesium content as a function of powder reuse for the refreshing and single batch methods.

8.5.4 Mechanical Properties

8.5.4.1 Hardness

The hardness of parts manufactured with reused powders is shown in Figure 8.42. The refreshing powder reuse method can be seen to result in a slight increase in hardness from R1 to R7 before plateauing, with the single batch method resulting in a slight increase in hardness from S1 to S3 before stabilising. The increase in the refreshing sample's hardness is attributable to increased oxygen content, and while this may also be true for the single batch samples, the large error associated with S1 makes the identification of a definitive trend difficult. Given these trends, it is clear that the refreshing reuse method results in a slight improvement in hardness as powder is reused, however, as this occurs only during the initial manufacturing runs, there is no long-term benefit to continuing with this reuse method. While the single batch method does not provide as significant an increase in hardness, the lack of an identifiable trend indicates that part properties are more consistent when using this powder reuse method.

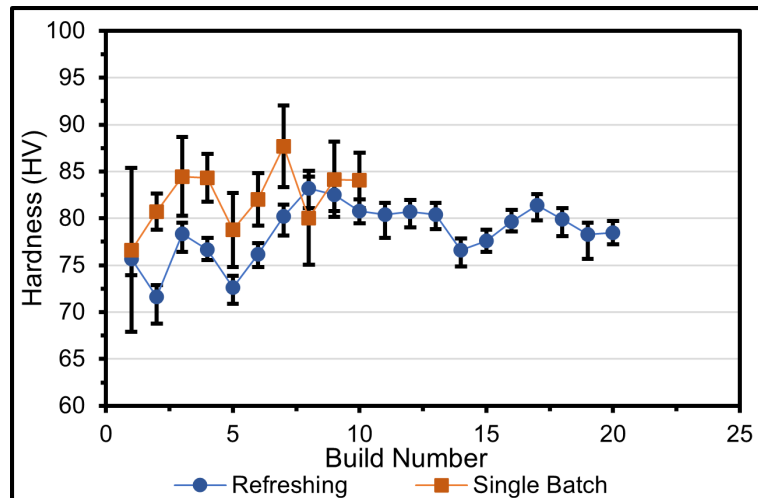


FIGURE 8.42. Hardness as a function of powder reuse for the refreshing and single batch methods.

8.5.4.2 Tensile Properties

Given only large, as-printed tensile samples were tested using powders reused by the single batch method, only these samples are used as a comparison between the two powder reuse methods as seen in Figure 8.43. The trends for each powder reuse method are similar to those seen in the hardness results.

For both the yield and tensile strengths of parts manufactured with powder reused by the refreshing method, there is an initial increase in strength between R1 and R5 before these properties stabilise, attributable to changing oxygen content and porosity as described in Chapter 7. This is in contrast to the single batch method where there is no appreciable change in the yield or tensile strength due to powder reuse. However, given the only minor change present in parts manufactured by the refreshing method and their stabilisation by approximately the fifth manufacturing run, the choice of powder reuse method appears to have minimal influence on the yield and tensile strength of AlSi7Mg.

The ductility of parts also appears to not be influenced by powder reuse, with both the refreshing and single batch powder reuse methods effecting no change on part ductility. This may partly be due to the variability seen within the results, especially the horizontal specimens where residual support structures were seen to significantly reduce the ductility of samples, suggesting that the inherent variation in this property plays a more significant role than powder

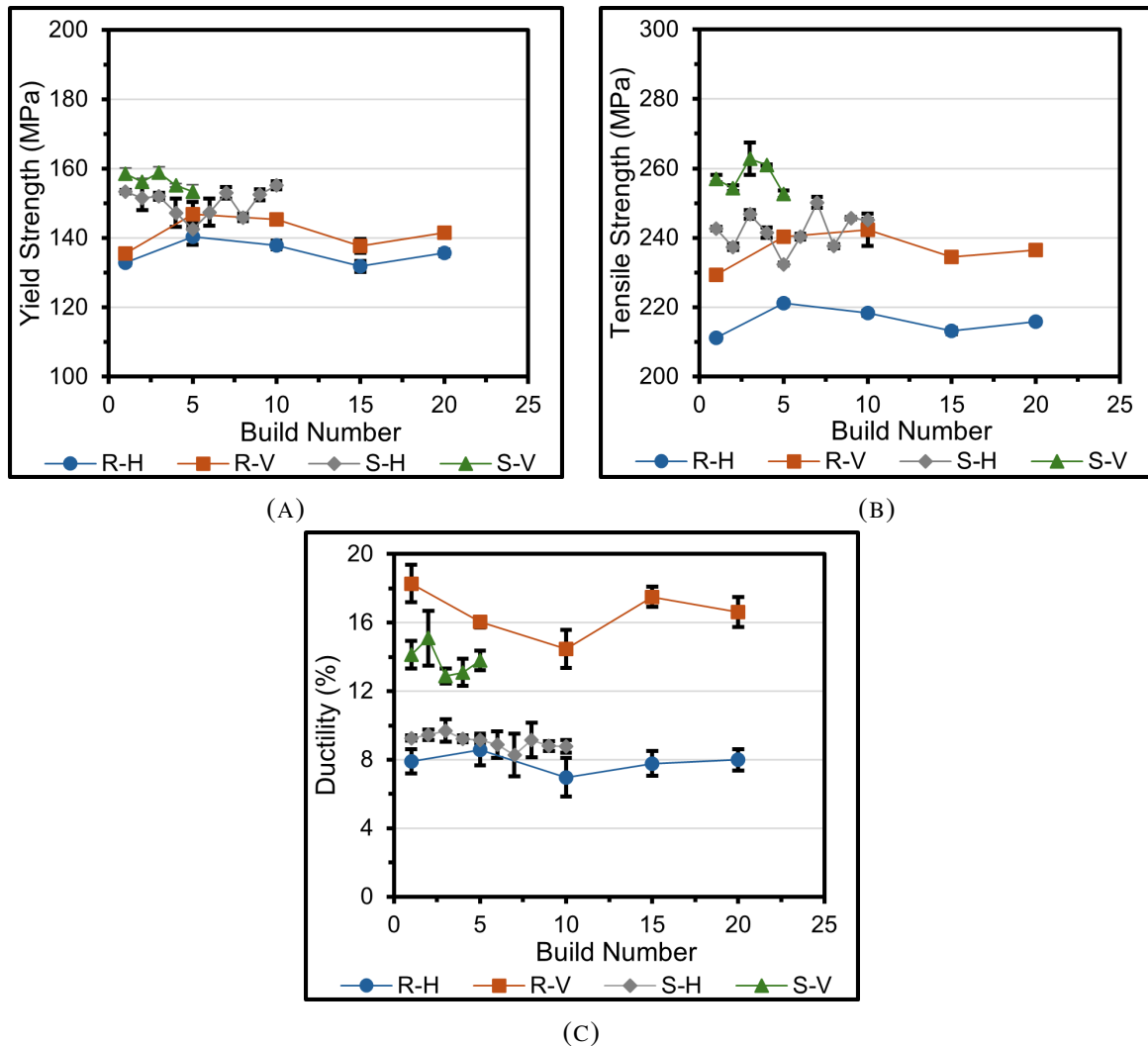


FIGURE 8.43. Tensile properties as a function of powder reuse for the refreshing and single batch methods: (a) yield strength, (b) tensile strength, (c) ductility. Error bars are present on all figures but too small to be seen in some plots. Nomenclature is as follows: R – Refreshing, S – Single batch, H – Horizontal, V – Vertical.

reuse in determining the ductility of parts. Therefore, both the refreshing and single batch powder reuse methods can be seen to both have minimal impact on part ductility.

While these results are true of stress relieved samples, the same may not be true of T6 treated ones. This is as the single batch parts exhibit a significant decrease in magnesium content as powder is reused, which is the element most relied on for strengthening in T6 treated AlSi7Mg samples. This changing presence creates the potential for a reduction in the effectiveness of heat treatment, which would likely become more significant as powder continues to be reused

due to the continual reduction in magnesium content, therefore resulting in reduced yield and tensile strengths. Further testing on heat treated single batch specimens should therefore be conducted to determine the validity of this hypothesis.

8.5.4.3 Flexural Properties

Since only stress relieved samples were tested under bending during single batch powder reuse studies, only these samples, and not T6 treated ones, were used for comparison between the refreshing and single batch powder reuse methods. The yield strength, flexural strength, and ductility of samples tested under bending are shown in Figure 8.44 as a function of powder reuse.

Both the refreshing and single batch powder reuse methods are seen to have no influence on the flexural properties of AlSi7Mg for both strength and ductility. While there is some variation within measurements, with refreshing samples experiencing a slight increase in strength between R1 and R5 and the single batch samples being slightly stronger for S7 and S9, these changes are not large enough over a sufficiently long period of powder reuse to infer a relationship to powder reuse. The reuse of powder can therefore be seen to have no influence on the flexural properties of AlSi7Mg, no matter the powder reuse method used. Heat treatment and part orientation are both seen to have a greater influence on flexural properties than powder reuse.

8.5.4.4 Impact Toughness

The impact toughness of samples shows greater variation than other mechanical properties due to powder reuse, with reductions in the absorbed energy observed in parts manufactured with powders reused by both the refreshing and single batch methods. A comparison of these results is shown in Figure 8.45.

It is evident that the factor most influencing impact toughness is part orientation, with horizontally oriented specimens having impact toughnesses typically 35% greater than those of vertically oriented samples. This suggests that powder age is not the most significant factor influencing the impact toughness of AlSi7Mg.

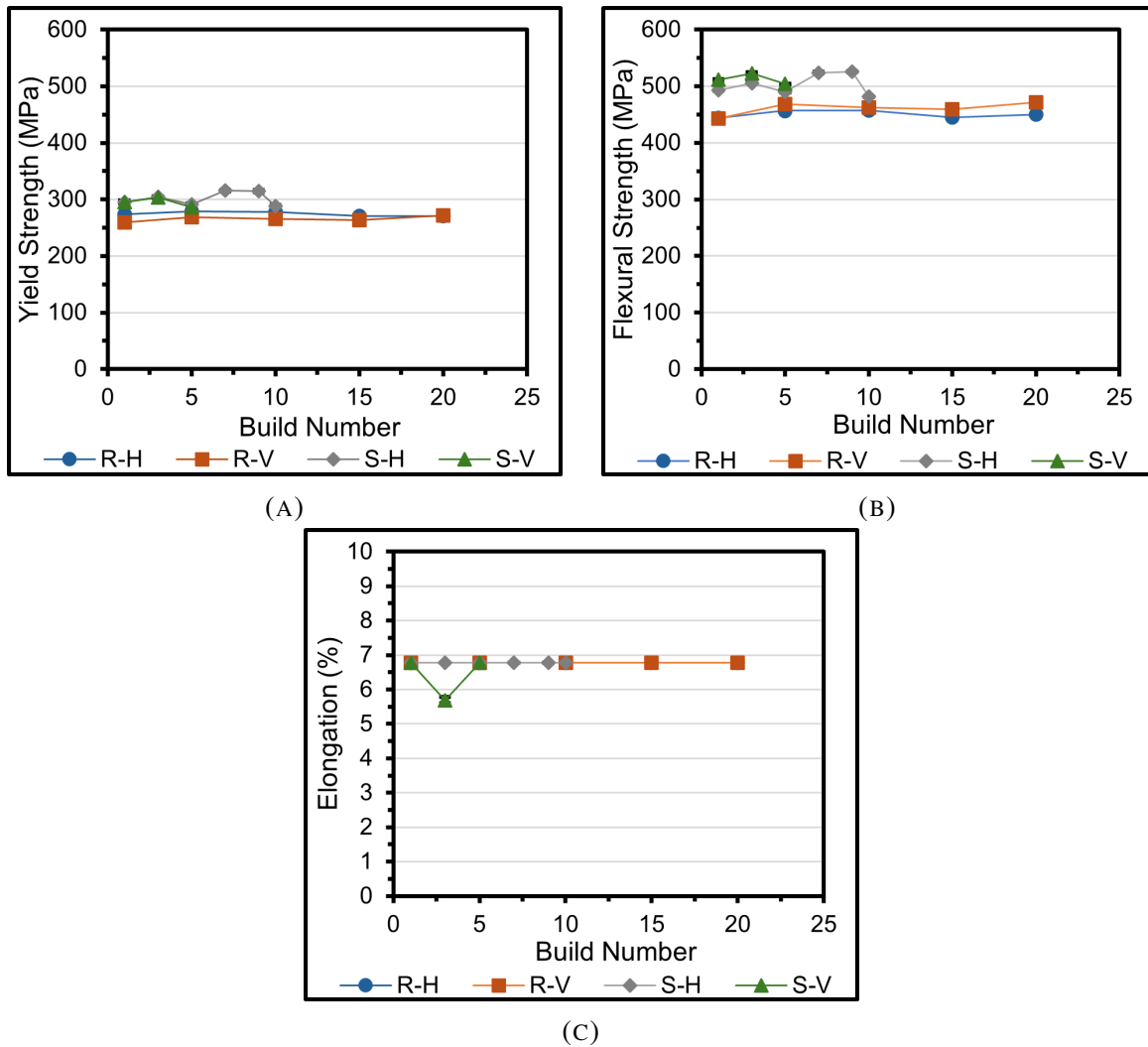


FIGURE 8.44. Flexural properties as a function of powder reuse for the refreshing and single batch methods: (a) yield strength, (b) flexural strength, (c) ductility. Error bars are present on all figures but too small to be seen on most plots. Nomenclature is as follows: R – Refreshing, S – Single batch, H – Horizontal, V – Vertical.

For parts manufactured with powders from both the refreshing and single batch powder reuse method, horizontally oriented samples demonstrate larger decreases in impact toughness than vertically oriented ones. This is as vertically oriented specimens are weakened by the unfavourable orientation of melt pool boundaries, which as seen in Section 8.5.2 remain consistent regardless of the powder reuse method used. Horizontally oriented specimens, however, do not have this unfavourable melt pool orientation. This means their toughness is drawn primarily from other factors, including the oxide dispersion within the microstructure.

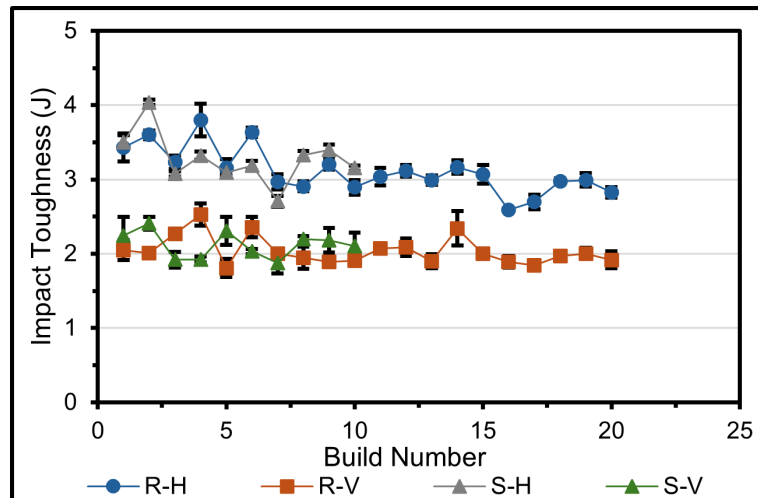


FIGURE 8.45. Impact toughness as a function of powder reuse for the refreshing and single batch methods. Nomenclature is as follows: R – Refreshing, S – Single batch, H – Horizontal, V – Vertical.

Given the oxygen level increases in parts manufactured using reused powders from both the refreshing and single batch methods, this makes horizontally oriented specimens prone to changes in impact toughness as powder is reused. While this trend is also somewhat visible in the vertically oriented samples, it is only slight. Powder reuse can therefore be seen to reduce the impact toughness of AlSi7Mg slightly, with this behaviour most evident in horizontally oriented samples. This behaviour is consistent regardless of the powder reuse method employed.

8.6 Conclusion

These results show that the single batch powder reuse method will influence both powder characteristics and part properties of AlSi7Mg manufactured by PBF.

This is most evidently seen in the powder characteristics, where the continual increase in particle size resulted in a PSD that continuously shifted to the right, first by widening as the increase in D90 value outpaced the increase in D10, before powder sieving limited any further change in the D90 value and the PSD began to narrow. This led to a dramatic improvement in initial powder flowability, which while likely influenced by the initially poor powder quality, continued to improve as powder was reused, resulting in a continually increasing powder bed

density. Powder chemistry remained stable, with the exception of oxygen which showed a slight increase and magnesium which decreased at an accelerating rate.

While changes in powder characteristics were observed, no change to microstructure was found due to powder reuse, with melt pool characteristics, grain size and shape, crystallographic texture, and the cell network remaining consistent. Part density gradually increased as powder was reused due to the improvement in powder bed density, while the surface roughness was stable even given the changes observed in powder size. Part oxygen content increased while magnesium content decreased, which has the potential to significantly influence the mechanical properties of further manufacturing runs given the influences of these elements on the strengthening mechanisms of AlSi7Mg.

The mechanical properties of parts manufactured with powder reused by the single batch method showed remarkable consistency, with hardness, tensile properties, and flexural properties all seeing no significant change as a result of powder reuse. There was, however, a slight decrease in the impact toughness of samples manufactured with reused powder, although other factors such as specimen orientation remained greater influences on this property. These limited changes indicate that the single batch powder reuse method can be successfully used industrially and academically, although chemical monitoring of parts is recommended to ensure oxygen and magnesium contents remain at acceptable levels.

A comparison of the refreshing and single batch powder reuse methods shows that while many powder characteristics and part properties show similar trends, others vary notably. While there were noticeable differences in the initial powder batch characteristics used for the testing of each powder reuse method, a comparison between the two is still viable. It does, however, highlight the importance of testing the properties of the initial powder batch quality before use, as both powder batches met the requirements set forth by the powder manufacturer while displaying, in cases, significantly different properties during testing.

The characteristics of powders reused by the single batch method show significantly greater changes than those of the refreshing method. This is clearest in the particle size, where the D10 value continues to increase for single batch reused powders as no new powder is introduced into the powder batch, in contrast with the refreshing powder reuse method. This results in more significant improvements in powder flowability and powder bed density for

single batch reused powders, meaning this powder reuse method shows good promise for powder batches with initially poor properties. This is, however, at the cost of an accelerated reduction in powder magnesium content, with the continual introduction of unused powder in the refreshing method limiting its loss.

Many aspects of manufactured parts showed no reliance on powder reuse, with part micro-structure, surface roughness, and the majority of part chemistry remaining unchanged. If these issues are of major concern for a PBF manufactured part, then the choice of powder reuse method will not influence the consistency of these properties between manufacturing runs. The same is true of many mechanical properties, with hardness, tensile properties, and flexural properties all showing minimal changes with powder reuse. While the refreshing powder reuse method resulted in a slight increase in the strengths of parts over the first nine manufacturing runs, the small degrees of change and the stabilisation of these properties in later manufacturing runs indicates that these changes are of minimal concern. Therefore, both the refreshing and single batch powder reuse methods show good promise for maintaining initial mechanical properties.

The main differences in part properties due to the powder reuse method used relates to part density and chemistry. The density of parts manufactured with powders reused by the refreshing method show a continual decline in relative density, linked to the increased prevalence of foreign particles and spatter within the powder batch. The acceleration of this decline is of especial concern, although given this density always remains above that specified by the powder manufacturer, this reduction in density may be deemed acceptable. This is in contrast to the single batch method which exhibited a continual increase in part relative density due to changing powder bed density, suggesting that the changes to this powder batch were beneficial. Together, this shows that the single batch powder reuse method will result in improved changes to part density contrary to the refreshing method.

Part chemistry also undergoes significant change due to the use of reused powders. Both the refreshing and single batch powder reuse methods show near identical increases in part oxygen content as powder is reused, suggesting that the powder reuse method does not influence the oxygen content of parts as much as careful powder handling. The magnesium content of parts does, however, differ according to the powder reuse method used, with the single batch method seeing a considerably greater reduction in part magnesium content than the refreshing

method, which also sees some decline. This is likely to influence the effectiveness of heat treatments reliant on magnesium for precipitate growth, meaning that the refreshing powder reuse method is a more suitable choice for applications requiring these heat treatments.

Together, this shows that both the refreshing and single batch powder reuse methods have a variety of impacts on both powder and part properties. The refreshing method provides greater powder longevity than the single batch method, with the topping up of powder allowing for builds of the same volume to be continuously manufactured. This is accompanied, however, by a reduction in part density, and although this has no significant impact on part properties, it should be carefully considered for applications involving fatigue failure, where increased porosity can be detrimental. The single batch method accelerates the changes experienced by the powder batch, showing great potential to improve both the flowability properties of powders as well as part density. This is, however, to the detriment of part magnesium content and so should be employed when heat treatments reliant on magnesium are not used during post processing. Therefore, as the selection of powder reuse method can be seen to influence the final properties of parts manufactured with reused powders, both the refreshing and single batch methods will find use in different applications.

CHAPTER 9

Conclusion

In this thesis, additively manufactured AlSi7Mg produced by powder bed fusion was investigated to determine how part performance can be enhanced and the sustainability of the manufacturing process can be improved. These investigations were carried out using a range of techniques that examined powder characteristics, part properties, microstructural characteristics, and mechanical properties, and for the first time, a comparative study of powder reuse methods is presented.

9.1 Research Summary

The performance of PBF AlSi7Mg has been examined in two main ways: an investigation into some of the manufacturing parameters often not considered during the manufacture of parts, and a thorough microstructural investigation into the heat treatment routes available to this alloy.

In Chapter 5 of this thesis a number of parameters relating to the positioning of parts within the build volume were investigated: the part location on the build plate; the height of parts; and the size, orientation, and surface finish of parts. It was shown that the location of a part on a build plate had no influence on its properties, which showed excellent consistency in their microstructures and mechanical properties. The hardness of as-printed parts was seen to decrease with increasing distance from the build plate with a difference of 18.3 HV over 250 mm, with this effect eliminated by a stress relieving heat treatment. Given the consistency of the grain size and texture, this change was seen to be attributable to differences in the size of the silicon cell network as well as the in-situ precipitation of strengthening precipitates due to the use of a heated build plate. The size, orientation, and surface finish of tensile specimens was also investigated, indicating that larger samples tended to have higher strengths and

ductilities, while machined surfaces also resulted in improved properties. Strain hardening behaviour was also seen to be influenced by the different microstructures of samples according to their machining histories, with the presence of fine grains in as-printed samples improving their performance.

Given the success of heat treatment in homogenising the anisotropic properties of additively manufactured parts, the ageing behaviour of AlSi7Mg was analysed in Chapter 6, with an especial focus on the effects of a prior solution treatment. This showed that ageing is possible only in certain cases, with direct ageing seeing a reduction in hardness while stress relieved samples that were aged showed no change in properties, both as a result of over-ageing. The solution treatment temperature was also shown to strongly influence the ageing behaviour of AlSi7Mg, with solution treatments at temperatures below 500 °C resulting in poor age hardenability, while solution treatments above this temperature showed good age hardenability. This was attributed to changing elemental solute concentrations, as below this temperature magnesium rich iron precipitates formed, reducing the amount of magnesium available for the growth of strengthening phases in the Mg₂Si precipitation sequence. For solution temperatures above 500 °C magnesium was solely in solid solution, promoting the growth of strengthening precipitates and so improving age hardening behaviour.

These chapters provide insight into how the properties of PBF AlSi7Mg can be improved. A correct understanding of the influences of a part's location within the build volume allows for the manipulation of part height, orientation, and size to take advantage of these factors. The improved microstructural understanding during heat treatment presented in Chapter 6 also allows for further optimisation of heat treatment parameters, given the microstructural transformations involved in the solution treatment of these parts is now well understood. Together, these new understandings allow for the production of AlSi7Mg parts that have both consistent and improved properties.

The sustainability of powder bed fusion was also investigated in Chapters 7 and 8 of this thesis, with a focus on the methods of powder reuse. Chapter 7 involved the extensive characterisation of the refreshing powder reuse method, where new powder is added to the powder batch to replace that consumed in the manufacturing process, with characterisation including powders, microstructures, and mechanical properties. This found that slight increases in powder sizes occurred with powder reuse, alongside a reduction in magnesium content. These changes

to powders resulted in an increased part oxygen content and reduced magnesium content, with no observable microstructural changes, resulting in a slight increase in hardness and tensile strengths over the initial manufacturing runs before properties plateaued, with a slight reduction in fatigue life being the most significant change in mechanical properties. T6 treatments were also seen to reduce the effects of powder reuse, indicating that heat treatment may be a viable route to reduce any influences of powder reuse. This showed that the refreshing powder reuse method serves as a useful means of reusing powder while providing a consistent powder batch size, reducing the problems associated with end-of-life powder.

The single batch powder reuse method was then addressed in Chapter 8, using a simplified version of the analysis conducted in Chapter 7. This powder reuse method showed similar results, with particle sizes increasing and magnesium content decreasing, an increased part oxygen and decreasing part magnesium content, and no changes in microstructure. Mechanical properties were more stable, with minimal changes seen in tensile, Charpy, and bending tests. A comparison of the refreshing and single batch powder reuse methods showed that with careful powder handling procedures, both can be readily applied in manufacturing processes with minimal influence on the quality of parts produced. However, this comparison also made it evident that any changes experienced by the powder batch and parts manufactured with reused powders were accelerated by the single batch powder reuse method. While this resulted in a continual improvement in powder flowability, it also led to a rapid reduction in part magnesium content, which is of concern if ageing treatments are to take place on these parts.

The proper investigation of these powder reuse methods, for the first time considering the effects of the methods themselves, allows for the development of improved powder reuse techniques. While it is clear that both the refreshing and single batch powder reuse methods show great potential for practical application, their use cases will likely differ. The refreshing powder reuse method can be used where long-term powder reuse is required, with minimal requirements for powder traceability. Contrastingly, the single batch powder reuse method can be employed either where initial powder properties are poor and need to be improved, or where powder traceability is a key requirement.

9.2 Recommendations for Future Work

While this thesis aimed to increase the understanding of AlSi7Mg parts manufactured by PBF to improve its performance and sustainability, many future avenues of investigation arose as a result of these investigations. Some of the most crucial are presented here.

The effect of build height on the microstructure and properties of parts deserves a full investigation, including an exploration of factors that were beyond the scope of this research such as residual stress distributions. Given mechanical properties have already been seen to change over the build height and many complex parts are becoming increasingly larger as PBF machines increase their production capacities, a full and proper understanding of all the influencing factors and the reasons behind their development would be beneficial. This would then open the way for further research into functionally graded materials where processing parameters can be manipulated at varying stages of production to create materials with properties tailored throughout their geometry according to the requirements of their applications.

A full microstructural characterisation of the surface of parts, in comparison to their centres, would also be a promising extension on the work of Chapter 5. This would investigate the interaction region of the multiple laser parameter sets to understand why the unique microstructures in these locations form, and how they may be manipulated to improve the surface properties of parts. This would be especially helpful for the design of thin-wall parts which are dominated by these surface microstructure regions.

Within Chapter 8 many tests were left uncondacted due to the scope of this thesis. A continuation in this investigation, including, for example, the production of small tensile samples, would allow for a more comprehensive comparison of the single batch and refreshing powder reuse methods. Further work in this field would allow for a greater understanding of the changes experienced by both powders and parts as a result of these reuse methods, providing the potential to greater tailor these powder reuse methods for particular applications.

A further investigation on the effects of the heat treatment of parts manufactured with reused powder would also be worthwhile. Given that in Chapter 5 the anisotropy observable in hardness measurements was eliminated upon heat treatment, with similar effects seen in some

tests conducted in Chapter 7 involving the refreshing powder reuse method, heat treatment shows good promise to limiting the effects of powder reuse. These tests would be especially worth conducting on Charpy and fatigue specimens, as well as all samples from the single batch method, as none of these have as of yet been tested in the heat-treated state. The results from these tests have the potential to limit the influences of powder reuse, increasing the scope of its application.

Powder reuse studies often focus on the production of spatter, with little consideration of other powder particles within the powder batch. It would therefore be worthwhile investigating these other powder types by characterising powders taken from different locations within the build chamber to determine whether heat accumulation within the build chamber changes their properties. This would involve in depth characterisation of these particles to understand if any morphological, chemical, or microstructural changes occur due to their proximity, or lack thereof, to printed parts. A greater understanding of the effects of powder reuse on these particles would allow for better powder management, as well as a greater ability to plan PBF manufacturing runs with the aim of reducing the impact of reuse on powders.

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APPENDIX A

Cell Size Measurement for Section 5.3.4

This appendix contains the images from which cell size measurements were taken for results in Section 5.3.4 of Chapter 5, where measurements of the cell sizes at different distances from the build plate were taken. As per the methodology set out in Section 4.4.2, ten lines were taken from seven images to determine the cell size by averaging the number of intercepts along these lines. To ensure that the cells measured were all taken from the same area of the melt pool, cells were only measured if they were equiaxed in shape. This ensured that all cells measured were from the remelted zone of the melt pool to avoid the effects of different sized cells within different locations in the melt pool.

The following figures and tables display the lines taken on each image, as well as the number of intercepts on each line and the calculations required to determine cell sizes. Each sub-table corresponds to a sub-figure, e.g. Table A.1 (c) refers to the lines drawn in Figure A.1. (c).

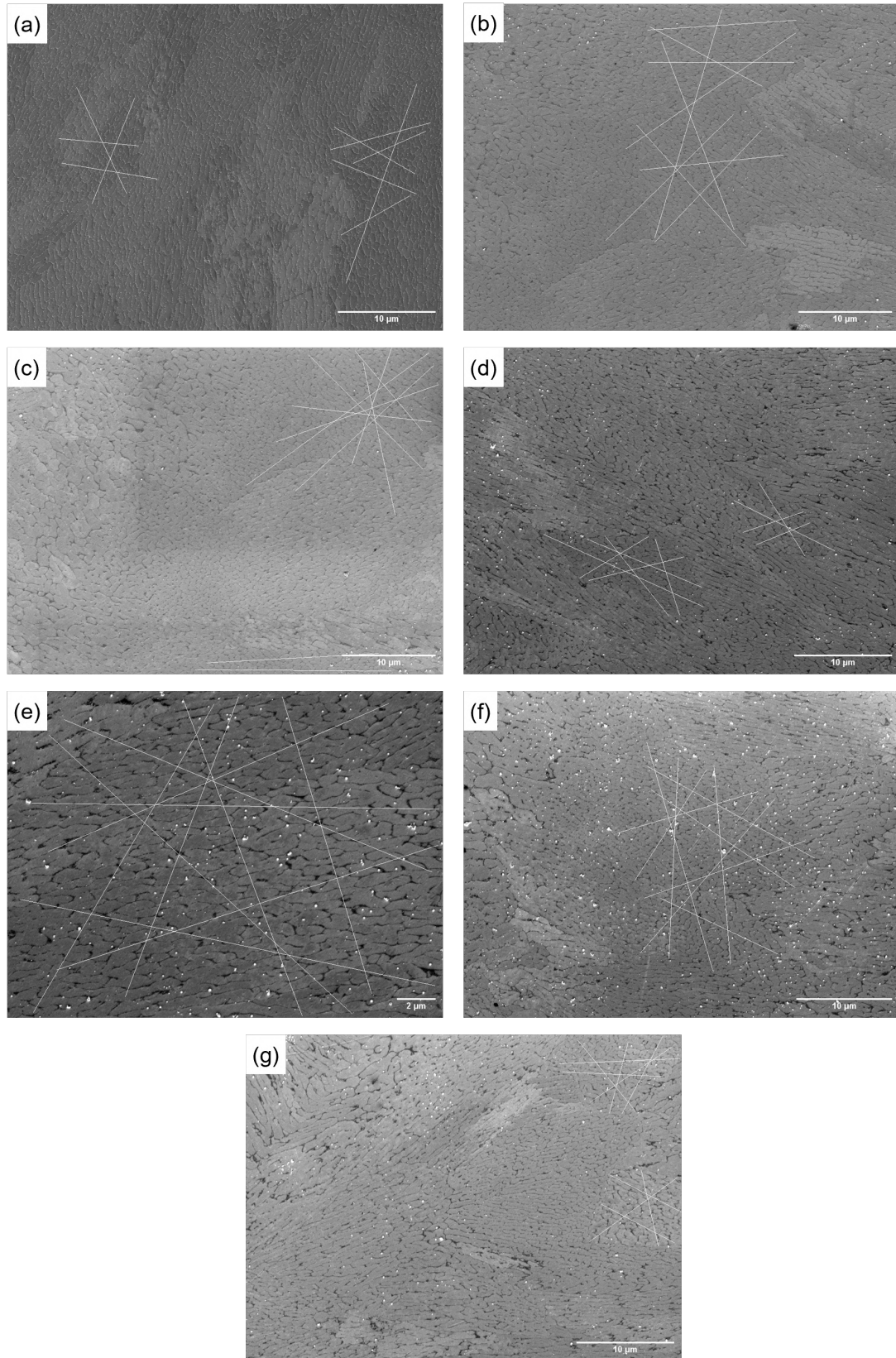


FIGURE A.1. Cell size measurements 10 mm from the build plate.

TABLE A.1. Cell size measurements 10 mm from the build plate.

(A)				(B)			
Line	Length	Intercepts	Average Size	Line	Length	Intercepts	Average Size
1	9.958	22	0.4526	1	16.461	35	0.4703
2	8.733	17	0.5137	2	17.380	28	0.6207
3	8.150	15	0.5433	3	19.092	38	0.5024
4	10.341	19	0.5443	4	15.355	24	0.6398
5	21.779	29	0.7510	5	25.387	51	0.4978
6	12.067	25	0.4827	6	15.545	16	0.9710
7	11.278	16	0.7049	7	13.583	24	0.5670
8	10.629	22	0.4831	8	21.526	40	0.5382
9	8.236	15	0.5491	9	21.950	33	0.6652
10	8.133	13	0.6256	10	15.898	17	0.9352
(C)				(D)			
Line	Length	Intercepts	Average Size	Line	Length	Intercepts	Average Size
1	19.946	39	0.5114	1	17.897	23	0.7781
2	16.577	31	0.5347	2	8.170	17	0.4806
3	20.904	30	0.6968	3	8.124	15	0.5416
4	18.386	27	0.6810	4	10.454	17	0.6149
5	24.079	43	0.5600	5	9.984	18	0.5547
6	14.955	20	0.7478	6	5.868	12	0.4890
7	11.160	16	0.6975	7	8.925	15	0.5950
8	17.223	21	0.8201	8	11.469	14	0.8192
9	13.106	25	0.5242	9	6.516	10	0.6516
10	18.353	31	0.5920	10	12.683	20	0.6342
(E)				(F)			
Line	Length	Intercepts	Average Size	Line	Length	Intercepts	Average Size
1	21.908	22	0.9958	1	24.798	54	0.4592
2	16.539	35	0.4725	2	17.782	24	0.7409
3	21.261	39	0.5452	3	13.659	32	0.4268
4	19.173	19	1.0091	4	18.109	28	0.6468
5	16.019	37	0.4329	5	19.871	25	0.7948
6	20.717	21	0.9865	6	20.872	42	0.4970
7	18.469	30	0.6156	7	15.245	22	0.6930
8	20.553	28	0.7340	8	13.479	19	0.7094
9	17.470	36	0.4853	9	21.419	37	0.5789
10	21.012	17	1.2360	10	20.956	43	0.4873
(G)							
Line	Length	Intercepts	Average Size				
1	9.303	18	0.5168				
2	9.961	17	0.5859				
3	7.467	15	0.4978				
4	12.315	19	0.6482				
5	11.044	15	0.7363				
6	8.460	11	0.7691				
7	7.932	14	0.5666				
8	8.226	10	0.8226				
9	8.946	16	0.5591				
10	10.229	15	0.6819				

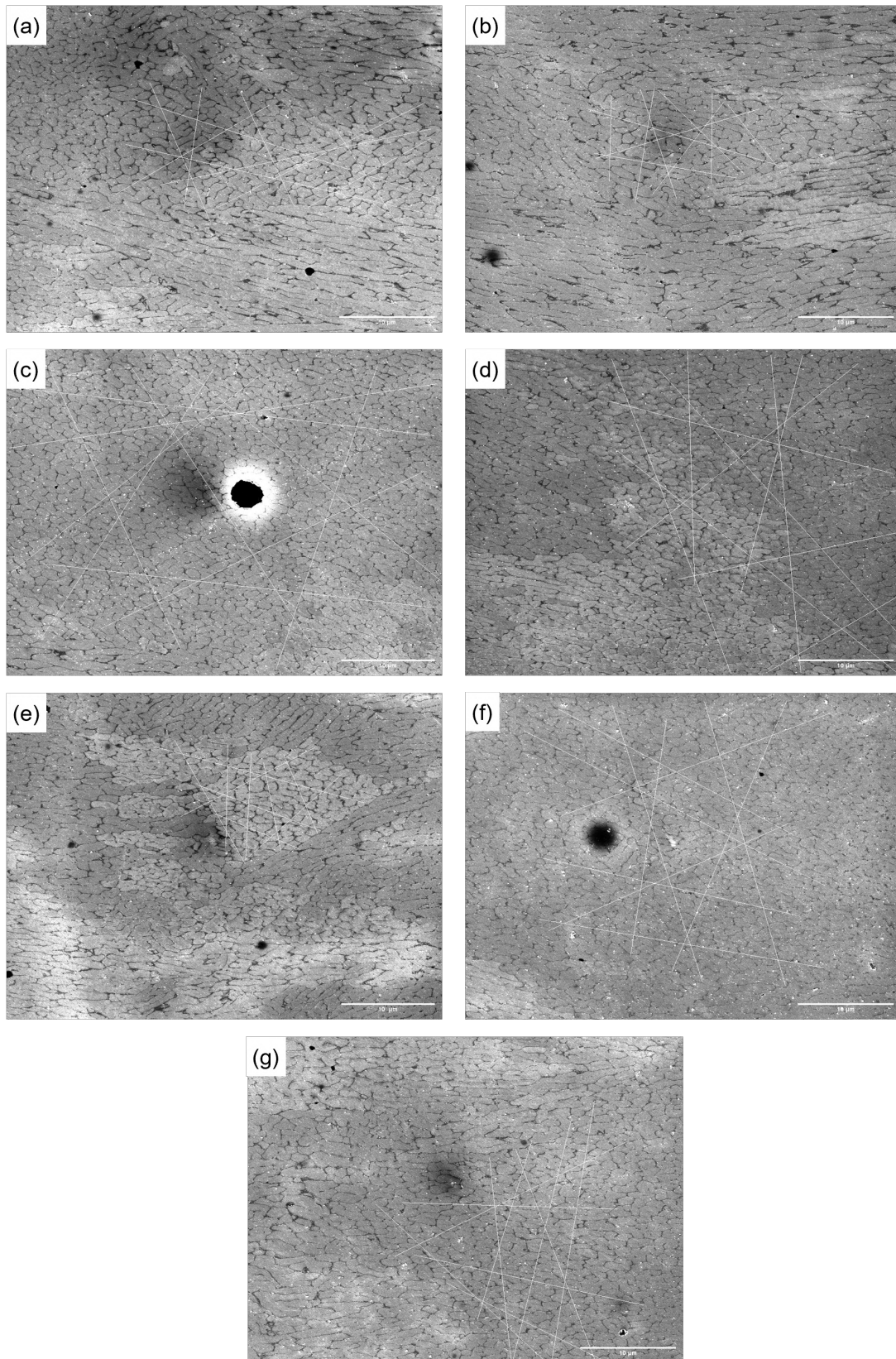


FIGURE A.2. Cell size measurements 130 mm from the build plate.

TABLE A.2. Cell size measurements 130 mm from the build plate.

(A)				(B)			
Line	Length	Intercepts	Average Size	Line	Length	Intercepts	Average Size
1	33.562	25	1.3425	1	21.081	16	1.3176
2	24.845	23	1.0802	2	11.983	18	0.6657
3	15.844	20	0.7922	3	17.786	16	1.1116
4	18.501	21	0.8810	4	12.031	18	0.6684
5	33.788	28	1.2067	5	9.467	14	0.6762
6	11.526	16	0.7204	6	12.540	13	0.9646
7	14.725	16	0.9203	7	15.110	10	1.5110
8	13.103	17	0.7708	8	8.475	10	0.8475
9	28.860	28	1.0307	9	8.847	11	0.8042
10	12.716	16	0.7948	10	12.135	16	0.7584
(C)				(D)			
Line	Length	Intercepts	Average Size	Line	Length	Intercepts	Average Size
1	32.384	42	0.7710	1	35.165	53	0.6635
2	34.278	43	0.7972	2	31.214	39	0.8004
3	34.244	52	0.6585	3	30.549	29	1.0534
4	42.530	42	1.0126	4	33.345	55	0.6063
5	36.767	43	0.8550	5	37.317	44	0.8481
6	46.138	41	1.1253	6	23.352	28	0.834
7	33.923	46	0.7375	7	25.569	40	0.6392
8	45.742	40	1.1436	8	37.517	40	0.9379
9	35.192	46	0.7650	9	15.734	18	0.8741
10	38.371	40	0.9593	10	24.412	35	0.6975
(E)				(F)			
Line	Length	Intercepts	Average Size	Line	Length	Intercepts	Average Size
1	23.242	21	1.1068	1	30.421	51	0.5965
2	11.334	18	0.6297	2	35.393	35	1.0112
3	13.910	20	0.6955	3	27.553	22	1.2524
4	13.965	15	0.9310	4	30.742	55	0.5589
5	10.957	20	0.5479	5	30.070	35	0.8591
6	9.203	16	0.5752	6	30.816	48	0.6420
7	9.770	17	0.5747	7	30.111	26	1.1581
8	9.902	12	0.8252	8	27.408	28	0.9789
9	16.060	21	0.7648	9	27.930	26	1.0742
10	15.228	23	0.6621	10	25.080	41	0.6117
(G)							
Line	Length	Intercepts	Average Size				
1	24.130	28	0.8618				
2	27.310	30	0.9103				
3	21.321	24	0.8884				
4	27.473	39	0.7044				
5	22.499	19	1.1842				
6	19.557	31	0.6309				
7	17.929	15	1.1953				
8	23.741	32	0.7419				
9	24.523	20	1.2262				
10	26.065	39	0.6683				

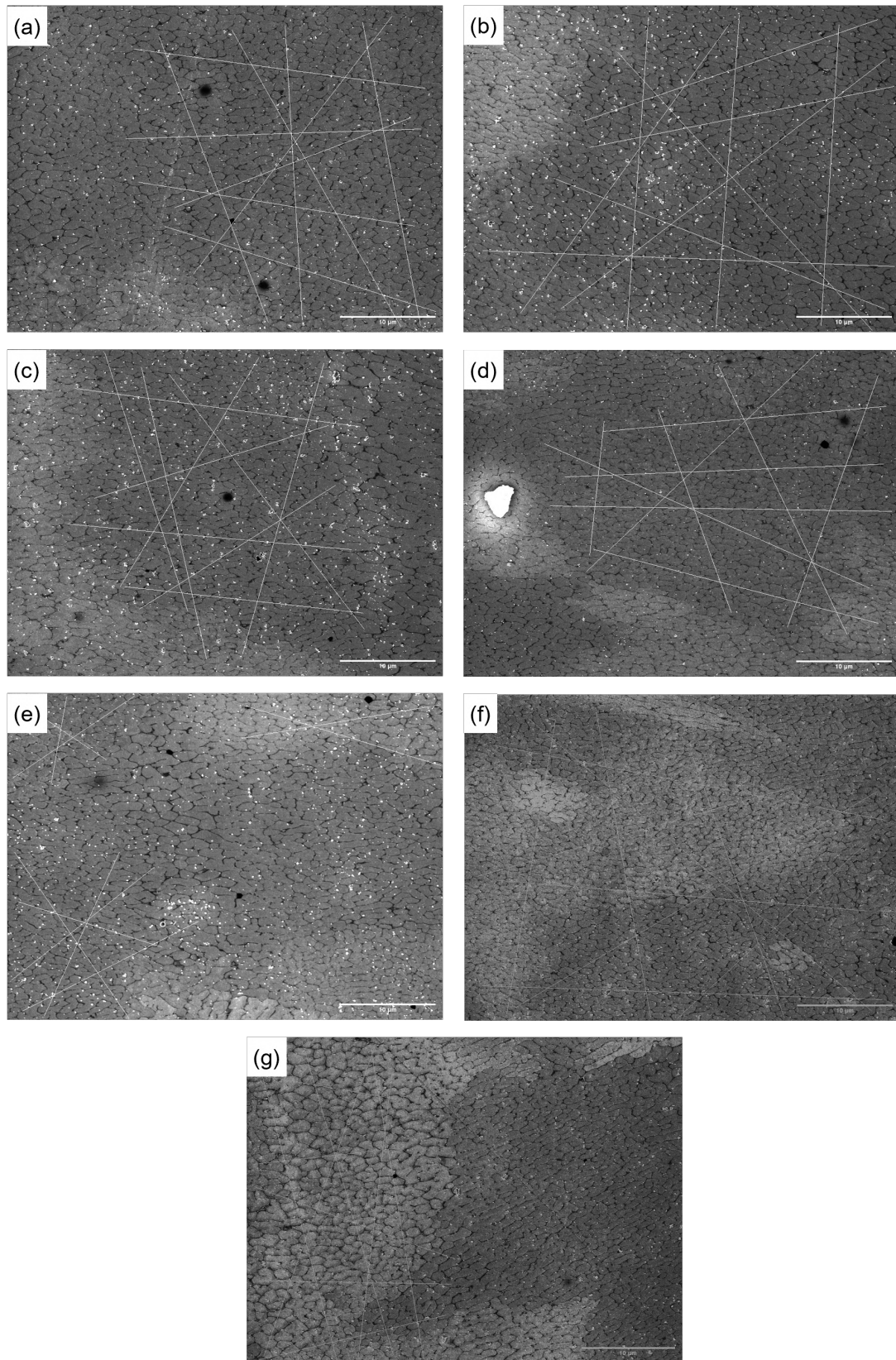


FIGURE A.3. Cell size measurements 250 mm from the build plate.

TABLE A.3. Cell size measurements 250 mm from the build plate.

(A)				(B)			
Line	Length	Intercepts	Average Size	Line	Length	Intercepts	Average Size
1	31.976	39	0.8199	1	37.179	48	0.7746
2	29.162	27	1.0800	2	31.478	41	0.7678
3	35.650	45	0.7922	3	39.353	38	1.0356
4	33.941	35	0.9697	4	43.054	34	1.2663
5	30.807	37	0.8326	5	32.698	34	0.9617
6	30.674	26	1.1798	6	32.407	32	1.0127
7	30.817	24	1.2840	7	32.758	43	0.7618
8	29.674	24	1.2364	8	32.440	32	1.0138
9	32.217	41	0.7858	9	37.172	27	1.3767
10	26.348	23	1.1456	10	42.758	50	0.8552
(C)				(D)			
Line	Length	Intercepts	Average Size	Line	Length	Intercepts	Average Size
1	31.238	46	0.6791	1	37.098	33	1.1242
2	29.764	19	1.5665	2	32.939	43	0.7660
3	31.520	48	0.6567	3	31.143	38	0.8196
4	30.045	19	1.5813	4	29.664	24	1.2360
5	33.540	36	0.9317	5	22.572	34	0.6639
6	27.958	40	0.6990	6	35.685	26	1.3725
7	24.229	25	0.9692	7	14.078	20	0.7039
8	25.599	23	1.1130	8	27.914	39	0.7157
9	28.336	39	0.7266	9	30.715	24	1.2798
10	24.997	17	1.4704	10	33.307	27	1.2336
(E)				(F)			
Line	Length	Intercepts	Average Size	Line	Length	Intercepts	Average Size
1	21.050	14	1.5036	1	47.035	59	0.7972
2	21.468	17	1.2628	2	44.033	48	0.9174
3	20.995	27	0.7776	3	32.231	43	0.7496
4	20.612	20	1.0306	4	39.290	32	1.2278
5	18.984	23	0.8254	5	41.543	37	1.1228
6	18.774	24	0.7823	6	26.825	34	0.7890
7	15.799	13	1.2153	7	28.815	35	0.8233
8	15.273	15	1.0182	8	41.804	33	1.2668
9	12.179	12	1.0149	9	31.126	45	0.6917
10	9.163	15	0.6109	10	34.047	30	1.1349
(G)							
Line	Length	Intercepts	Average Size				
1	34.831	48	0.7257				
2	34.112	36	0.9476				
3	35.676	36	0.9910				
4	34.852	35	0.9958				
5	30.567	34	0.8990				
6	15.584	12	1.2987				
7	15.135	11	1.3760				
8	22.755	19	1.1976				
9	20.680	15	1.3787				
10	21.030	16	1.3144				

APPENDIX B

Statistical Analysis for Powder Reuse Studies

This appendix contains details of the statistical analysis conducted on the results of some of the tests carried out as part of the investigations into powder reuse. An ANOVA analysis was conducted on the results to determine if they were statistically significant. If these results were significant, then a Tukey-Kramer test was carried out to determine which pairs of results were statistically significant from each other.

This appendix contains these results. Firstly, a table presenting the results of the ANOVA analysis is presented, with the p value highlighted as either red or green, red denoting statistical significance, and green denoting no statistical significance. A table containing the results of the Tukey-Kramer test is then presented, with the same colour system used to indicate whether pairs of results are significantly different or not.

B1 Statistical Analysis from the Refreshing Powder Reuse Method

B1.1 Apparent Density

TABLE B.1. ANOVA analysis of apparent density data from the refreshing powder reuse method.

Source	DF	Sum of Square	Mean Square	F Statistic	P-Value
Groups	18	0.003315	0.000184	9.0919	1.52E-12
Error	76	0.00154	2.03E-05		
Total	94	0.004855	5.16E-05		

TABLE B.2. Tukey-Kramer analysis of apparent density data from the refreshing powder reuse method.

(A)

Group	R3	R4	R5	R6	R7	R8	R9	R10	R11
R1	0.031	0.0074	0.01	0.017	0.014	0.018	0.0084	0.011	0.014
R3	0	0.023	0.021	0.014	0.017	0.013	0.022	0.02	0.017
R4	0.023	0	0.0027	0.0095	0.0062	0.01	0.001	0.0037	0.0061
R5	0.021	0.0027	0	0.0068	0.0035	0.0075	0.0017	0.0011	0.0034
R6	0.014	0.0095	0.0068	0	0.0033	0.00067	0.0085	0.0058	0.0034
R7	0.017	0.0062	0.0035	0.0033	0	0.004	0.0052	0.0024	0.000081
R8	0.013	0.01	0.0075	0.00067	0.004	0	0.0092	0.0064	0.0041
R9	0.022	0.001	0.0017	0.0085	0.0052	0.0092	0	0.0027	0.0051
R10	0.02	0.0037	0.0011	0.0058	0.0024	0.0064	0.0027	0	0.0024
R11	0.017	0.0061	0.0034	0.0034	0.000081	0.0041	0.0051	0.0024	0
R12	0.02	0.0034	0.00074	0.0061	0.0028	0.0068	0.0024	0.00032	0.0027
R13	0.012	0.011	0.0084	0.0016	0.0049	0.00092	0.01	0.0073	0.005
R14	0.012	0.012	0.009	0.0022	0.0055	0.0015	0.011	0.0079	0.0056
R15	0.015	0.0081	0.0054	0.0014	0.0019	0.0021	0.0071	0.0043	0.002
R16	0.014	0.0088	0.0062	0.00066	0.0026	0.0013	0.0078	0.0051	0.0027
R17	0.018	0.0048	0.0022	0.0047	0.0014	0.0053	0.0038	0.0011	0.0013
R18	0.017	0.0061	0.0034	0.0034	0.00011	0.0041	0.0051	0.0023	0.000032
R19	0.016	0.007	0.0044	0.0025	0.00086	0.0031	0.006	0.0033	0.00094

(B)

Group	R12	R13	R14	R15	R16	R17	R18	R19	R20
R1	0.011	0.019	0.019	0.015	0.016	0.012	0.013	0.014	0.015
R3	0.02	0.012	0.012	0.015	0.014	0.018	0.017	0.016	0.015
R4	0.0034	0.011	0.012	0.0081	0.0088	0.0048	0.0061	0.007	0.0078
R5	0.00074	0.0084	0.009	0.0054	0.0062	0.0022	0.0034	0.0044	0.0051
R6	0.0061	0.0016	0.0022	0.0014	0.00066	0.0047	0.0034	0.0025	0.0017
R7	0.0028	0.0049	0.0055	0.0019	0.0026	0.0014	0.00011	0.00086	0.0016
R8	0.0068	0.00092	0.0015	0.0021	0.0013	0.0053	0.0041	0.0031	0.0024
R9	0.0024	0.01	0.011	0.0071	0.0078	0.0038	0.0051	0.006	0.0068
R10	0.00032	0.0073	0.0079	0.0043	0.0051	0.0011	0.0023	0.0033	0.0041
R11	0.0027	0.005	0.0056	0.002	0.0027	0.0013	0.000032	0.00094	0.0017
R12	0	0.0077	0.0083	0.0047	0.0054	0.0014	0.0027	0.0036	0.0044
R13	0.0077	0	0.00059	0.003	0.0023	0.0063	0.005	0.004	0.0033
R14	0.0083	0.00059	0	0.0036	0.0028	0.0069	0.0056	0.0046	0.0039
R15	0.0047	0.003	0.0036	0	0.00076	0.0032	0.002	0.001	0.00027
R16	0.0054	0.0023	0.0028	0.00076	0	0.004	0.0028	0.0018	0.001
R17	0.0014	0.0063	0.0069	0.0032	0.004	0	0.0012	0.0022	0.003
R18	0.0027	0.005	0.0056	0.002	0.0028	0.0012	0	0.00097	0.0017
R19	0.0036	0.004	0.0046	0.001	0.0018	0.0022	0.00097	0	0.00076

B1.2 Avalanche Angle

TABLE B.3. ANOVA analysis of avalanche angle data from the refreshing powder reuse method.

Source	DF	Sum of Square	Mean Square	F Statistic	P-Value
Groups	19	115661.61	608.7687	22.5824	2.22E-16
Error	380	10244.07	26.9581		
Total	399	21810.67	54.6633		

TABLE B.4. Tukey-Kramer analysis of avalanche angle data from the refreshing powder reuse method.

(A)

Group	R2	R3	R4	R5	R6	R7	R8	R9	R10
R1	2.64	3.42	7.65	2.47	6.3	6.5	11.86	6.45	13.03
R2	0	0.78	5.01	5.11	3.66	3.86	9.22	3.81	10.39
R3	0.78	0	4.23	5.89	2.88	3.08	8.44	3.03	9.61
R4	5.01	4.23	0	10.12	1.35	1.15	4.21	1.2	5.38
R5	5.11	5.89	10.12	0	8.77	8.98	14.33	8.93	15.51
R6	3.66	2.88	1.35	8.77	0	0.2	5.56	0.15	6.73
R7	3.86	3.08	1.15	8.98	0.2	0	5.36	0.051	6.53
R8	9.22	8.44	4.21	14.33	5.56	5.36	0	5.41	1.17
R9	3.81	3.03	1.2	8.93	0.15	0.051	5.41	0	6.58
R10	10.39	9.61	5.38	15.51	6.73	6.53	1.17	6.58	0
R11	9.92	9.13	4.91	15.03	6.25	6.05	0.69	6.1	0.48
R12	11.51	10.73	6.5	16.62	7.85	7.64	2.29	7.7	1.11
R13	10.65	9.87	5.64	15.76	6.99	6.78	1.43	6.83	0.25
R14	11.61	10.83	6.6	16.72	7.94	7.74	2.38	7.79	1.21
R15	1.18	0.4	3.83	6.29	2.48	2.68	8.04	2.63	9.21
R16	9.41	8.63	4.4	14.52	5.75	5.55	0.19	5.6	0.98
R17	6.82	6.04	1.81	11.93	3.16	2.95	2.4	3	3.58
R18	0.99	0.21	4.02	6.11	2.67	2.87	8.23	2.82	9.4
R19	3.39	4.18	8.4	1.72	7.06	7.26	12.62	7.21	13.79

(B)

Group	R11	R12	R13	R14	R15	R16	R17	R18	R19	R20
R1	12.55	14.15	13.29	14.24	3.82	12.05	9.46	3.63	0.76	0.72
R2	9.92	11.51	10.65	11.61	1.18	9.41	6.82	0.99	3.39	3.36
R3	9.13	10.73	9.87	10.83	0.4	8.63	6.04	0.21	4.18	4.14
R4	4.91	6.5	5.64	6.6	3.83	4.4	1.81	4.02	8.4	8.37
R5	15.03	16.62	15.76	16.72	6.29	14.52	11.93	6.11	1.72	1.75
R6	6.25	7.85	6.99	7.94	2.48	5.75	3.16	2.67	7.06	7.02
R7	6.05	7.64	6.78	7.74	2.68	5.55	2.95	2.87	7.26	7.23
R8	0.69	2.29	1.43	2.38	8.04	0.19	2.4	8.23	12.62	12.58
R9	6.1	7.7	6.83	7.79	2.63	5.6	3	2.82	7.21	7.17
R10	0.48	1.11	0.25	1.21	9.21	0.98	3.58	9.4	13.79	13.75
R11	0	1.59	0.73	1.69	8.73	0.5	3.1	8.92	13.31	13.28
R12	1.59	0	0.86	0.097	10.33	2.1	4.69	10.51	14.9	14.87
R13	0.73	0.86	0	0.96	9.47	1.24	3.83	9.65	14.04	14.01
R14	1.69	0.097	0.96	0	10.42	2.19	4.79	10.61	15	14.97
R15	8.73	10.33	9.47	10.42	0	8.23	5.64	0.19	4.58	4.54
R16	0.5	2.1	1.24	2.19	8.23	0	2.59	8.42	12.81	12.77
R17	3.1	4.69	3.83	4.79	5.64	2.59	0	5.82	10.21	10.18
R18	8.92	10.51	9.65	10.61	0.19	8.42	5.82	0	4.39	4.36
R19	13.31	14.9	14.04	15	4.58	12.81	10.21	4.39	0	0.034

B1.3 Powder Bed Density

TABLE B.5. ANOVA analysis of powder bed density data from the refreshing powder reuse method.

Source	DF	Sum of Square	Mean Square	F Statistic	P-value
Groups	19	0.01556	0.000819	6.8213	4.73E-09
Error	60	0.007205	0.00012		
Total	79	0.02277	0.000288		

TABLE B.6. Tukey-Kramer analysis of powder bed density data from the refreshing powder reuse method.

(A)

Group	R2	R3	R4	R5	R6	R7	R8	R9	R10
R1	0.011	0.012	0.0056	0.021	0.011	0.009	0.015	0.024	0.0092
R2	0	0.00044	0.0057	0.0095	0.00043	0.02	0.026	0.035	0.0021
R3	0.00044	0	0.0061	0.0091	0.00087	0.021	0.027	0.035	0.0025
R4	0.0057	0.0061	0	0.015	0.0052	0.015	0.021	0.029	0.0036
R5	0.0095	0.0091	0.015	0	0.0099	0.03	0.036	0.044	0.012
R6	0.00043	0.00087	0.0052	0.0099	0	0.02	0.026	0.034	0.0017
R7	0.02	0.021	0.015	0.03	0.02	0	0.0059	0.014	0.018
R8	0.026	0.027	0.021	0.036	0.026	0.0059	0	0.0086	0.024
R9	0.035	0.035	0.029	0.044	0.034	0.014	0.0086	0	0.033
R10	0.0021	0.0025	0.0036	0.012	0.0017	0.018	0.024	0.033	0
R11	0.0024	0.0028	0.0033	0.012	0.002	0.018	0.024	0.032	0.00032
R12	0.02	0.021	0.014	0.03	0.02	0.00017	0.0061	0.015	0.018
R13	0.011	0.011	0.0053	0.02	0.011	0.0094	0.015	0.024	0.0089
R14	0.012	0.013	0.0067	0.022	0.012	0.008	0.014	0.022	0.01
R15	0.012	0.012	0.006	0.021	0.011	0.0086	0.015	0.023	0.0096
R16	0.048	0.048	0.042	0.057	0.047	0.027	0.022	0.013	0.046
R17	0.00066	0.00022	0.0063	0.0089	0.0011	0.021	0.027	0.035	0.0027
R18	0.00017	0.00027	0.0058	0.0093	0.0006	0.021	0.026	0.035	0.0023
R19	0.0029	0.0024	0.0085	0.0066	0.0033	0.023	0.029	0.038	0.005

(B)

Group	R11	R12	R13	R14	R15	R16	R17	R18	R19	R20
R1	0.0089	0.0089	0.00035	0.001	0.00041	0.037	0.012	0.011	0.014	0.014
R2	0.0024	0.02	0.011	0.012	0.012	0.048	0.00066	0.00017	0.0029	0.0028
R3	0.0028	0.021	0.011	0.013	0.012	0.048	0.00022	0.00027	0.0024	0.0024
R4	0.0033	0.014	0.0053	0.0067	0.006	0.042	0.0063	0.0058	0.0085	0.0085
R5	0.012	0.03	0.02	0.022	0.021	0.057	0.0089	0.0093	0.0066	0.0067
R6	0.002	0.02	0.011	0.012	0.011	0.047	0.0011	0.0006	0.0033	0.0033
R7	0.018	0.00017	0.0094	0.008	0.0086	0.027	0.021	0.021	0.023	0.023
R8	0.024	0.0061	0.015	0.014	0.015	0.022	0.027	0.026	0.029	0.029
R9	0.032	0.015	0.024	0.022	0.023	0.013	0.035	0.035	0.038	0.038
R10	0.00032	0.018	0.0089	0.01	0.0096	0.046	0.0027	0.0023	0.005	0.0049
R11	0	0.018	0.0085	0.0099	0.0093	0.045	0.0031	0.0026	0.0053	0.0053
R12	0.018	0	0.0092	0.0078	0.0084	0.028	0.021	0.02	0.023	0.023
R13	0.0085	0.0092	0	0.0014	0.00077	0.037	0.012	0.011	0.014	0.014
R14	0.0099	0.0078	0.0014	0	0.00064	0.035	0.013	0.013	0.015	0.015
R15	0.0093	0.0084	0.00077	0.00064	0	0.036	0.012	0.012	0.015	0.015
R16	0.045	0.028	0.037	0.035	0.036	0	0.048	0.048	0.051	0.051
R17	0.0031	0.021	0.012	0.013	0.012	0.048	0	0.00048	0.0022	0.0022
R18	0.0026	0.02	0.011	0.013	0.012	0.048	0.00048	0	0.0027	0.0027
R19	0.0053	0.023	0.014	0.015	0.015	0.051	0.0022	0.0027	0	0.00003

B1.4 Hardness

TABLE B.7. ANOVA analysis of hardness data from the refreshing powder reuse method.

Source	DF	Sum of Square	Mean Square	F Statistic	P-value
Groups	19	1730.475	91.0776	25.0733	2.22E-16
Error	180	653.842	3.6325		
Total	199	2384.317	11.9815		

TABLE B.8. Tukey-Kramer analysis of hardness data from the refreshing powder reuse method.

(A)

Group	R1	R2	R3	R4	R5	R6	R7	R8	R9	R10
R1	4.04	2.65	0.98	3.04	0.47	4.55	7.52	6.86	5.11	0.014
R2	0	6.69	5.02	1	4.51	8.59	11.56	10.9	9.15	0.0028
R3	6.69	0	1.67	5.69	2.18	1.9	4.87	4.21	2.46	0.0024
R4	5.02	1.67	0	4.02	0.51	3.57	6.54	5.88	4.13	0.0085
R5	1	5.69	4.02	0	3.51	7.59	10.56	9.9	8.15	0.0067
R6	4.51	2.18	0.51	3.51	0	4.08	7.05	6.39	4.64	0.0033
R7	8.59	1.9	3.57	7.59	4.08	0	2.97	2.31	0.56	0.023
R8	11.56	4.87	6.54	10.56	7.05	2.97	0	0.66	2.41	0.029
R9	10.9	4.21	5.88	9.9	6.39	2.31	0.66	0	1.75	0.038
R10	9.15	2.46	4.13	8.15	4.64	0.56	2.41	1.75	0	0.0049
R11	8.78	2.09	3.76	7.78	4.27	0.19	2.78	2.12	0.37	0.0053
R12	9.08	2.39	4.06	8.08	4.57	0.49	2.48	1.82	0.07	0.023
R13	8.78	2.09	3.76	7.78	4.27	0.19	2.78	2.12	0.37	0.014
R14	4.99	1.7	0.03	3.99	0.48	3.6	6.57	5.91	4.16	0.015
R15	5.94	0.75	0.92	4.94	1.43	2.65	5.62	4.96	3.21	0.015
R16	8	1.31	2.98	7	3.49	0.59	3.56	2.9	1.15	0.051
R17	9.74	3.05	4.72	8.74	5.23	1.15	1.82	1.16	0.59	0.0022
R18	8.24	1.55	3.22	7.24	3.73	0.35	3.32	2.66	0.91	0.0027
R19	6.64	0.05	1.62	5.64	2.13	1.95	4.92	4.26	2.51	0.000028

(B)

Group	R11	R12	R13	R14	R15	R16	R17	R18	R19	R20
R1	4.74	5.04	4.74	0.95	1.9	3.96	5.7	4.2	2.6	2.79
R2	8.78	9.08	8.78	4.99	5.94	8	9.74	8.24	6.64	6.83
R3	2.09	2.39	2.09	1.7	0.75	1.31	3.05	1.55	0.05	0.14
R4	3.76	4.06	3.76	0.03	0.92	2.98	4.72	3.22	1.62	1.81
R5	7.78	8.08	7.78	3.99	4.94	7	8.74	7.24	5.64	5.83
R6	4.27	4.57	4.27	0.48	1.43	3.49	5.23	3.73	2.13	2.32
R7	0.19	0.49	0.19	3.6	2.65	0.59	1.15	0.35	1.95	1.76
R8	2.78	2.48	2.78	6.57	5.62	3.56	1.82	3.32	4.92	4.73
R9	2.12	1.82	2.12	5.91	4.96	2.9	1.16	2.66	4.26	4.07
R10	0.37	0.07	0.37	4.16	3.21	1.15	0.59	0.91	2.51	2.32
R11	0	0.3	0	3.79	2.84	0.78	0.96	0.54	2.14	1.95
R12	0.3	0	0.3	4.09	3.14	1.08	0.66	0.84	2.44	2.25
R13	0	0.3	0	3.79	2.84	0.78	0.96	0.54	2.14	1.95
R14	3.79	4.09	3.79	0	0.95	3.01	4.75	3.25	1.65	1.84
R15	2.84	3.14	2.84	0.95	0	2.06	3.8	2.3	0.7	0.89
R16	0.78	1.08	0.78	3.01	2.06	0	1.74	0.24	1.36	1.17
R17	0.96	0.66	0.96	4.75	3.8	1.74	0	1.5	3.1	2.91
R18	0.54	0.84	0.54	3.25	2.3	0.24	1.5	0	1.6	1.41
R19	2.14	2.44	2.14	1.65	0.7	1.36	3.1	1.6	0	0.19

B1.5 Charpy Impact Toughness

B1.5.1 Horizontal, As-Printed Specimens

TABLE B.9. ANOVA analysis of horizontal, as-printed Charpy impact toughness data from the refreshing powder reuse method.

Source	DF	Sum of Square	Mean Square	F Statistic	P-value
Groups	19	5.4161	0.2851	17.6121	8.38E-14
Error	40	0.6474	0.01619		
Total	59	6.0635	0.1028		

TABLE B.10. Tukey-Kramer analysis of horizontal, as-printed Charpy impact toughness data from the refreshing powder reuse method.

(A)

Group	R2	R3	R4	R5	R6	R7	R8	R9	R10
R1	0.17	0.2	0.37	0.27	0.2	0.46	0.53	0.23	0.54
R2	0	0.37	0.2	0.44	0.035	0.63	0.7	0.4	0.71
R3	0.37	0	0.57	0.074	0.4	0.26	0.33	0.026	0.34
R4	0.2	0.57	0	0.64	0.16	0.83	0.89	0.59	0.91
R5	0.44	0.074	0.64	0	0.48	0.19	0.25	0.048	0.26
R6	0.035	0.4	0.16	0.48	0	0.67	0.73	0.43	0.74
R7	0.63	0.26	0.83	0.19	0.67	0	0.062	0.24	0.075
R8	0.7	0.33	0.89	0.25	0.73	0.062	0	0.3	0.013
R9	0.4	0.026	0.59	0.048	0.43	0.24	0.3	0	0.31
R10	0.71	0.34	0.91	0.26	0.74	0.075	0.013	0.31	0
R11	0.56	0.19	0.76	0.12	0.6	0.072	0.13	0.17	0.15
R12	0.48	0.11	0.68	0.04	0.52	0.15	0.21	0.088	0.22
R13	0.61	0.24	0.81	0.16	0.64	0.025	0.087	0.21	0.1
R14	0.43	0.065	0.63	0.009	0.47	0.2	0.26	0.039	0.27
R15	0.53	0.16	0.73	0.089	0.57	0.1	0.16	0.14	0.17
R16	1.01	0.64	1.21	0.57	1.05	0.38	0.32	0.62	0.3
R17	0.9	0.53	1.1	0.46	0.94	0.27	0.21	0.51	0.2
R18	0.62	0.25	0.82	0.18	0.66	0.0097	0.072	0.23	0.084
R19	0.61	0.24	0.81	0.16	0.64	0.025	0.087	0.21	0.1

(B)

Group	R11	R12	R13	R14	R15	R16	R17	R18	R19	R20
R1	0.39	0.31	0.44	0.26	0.36	0.84	0.73	0.45	0.44	0.6
R2	0.56	0.48	0.61	0.43	0.53	1.01	0.9	0.62	0.61	0.77
R3	0.19	0.11	0.24	0.065	0.16	0.64	0.53	0.25	0.24	0.4
R4	0.76	0.68	0.81	0.63	0.73	1.21	1.1	0.82	0.81	0.97
R5	0.12	0.04	0.16	0.009	0.089	0.57	0.46	0.18	0.16	0.33
R6	0.6	0.52	0.64	0.47	0.57	1.05	0.94	0.66	0.64	0.81
R7	0.072	0.15	0.025	0.2	0.1	0.38	0.27	0.0097	0.025	0.14
R8	0.13	0.21	0.087	0.26	0.16	0.32	0.21	0.072	0.087	0.079
R9	0.17	0.088	0.21	0.039	0.14	0.62	0.51	0.23	0.21	0.38
R10	0.15	0.22	0.1	0.27	0.17	0.3	0.2	0.084	0.1	0.066
R11	0	0.077	0.047	0.13	0.028	0.45	0.34	0.062	0.047	0.21
R12	0.077	0	0.12	0.049	0.049	0.53	0.42	0.14	0.12	0.29
R13	0.047	0.12	0	0.17	0.075	0.4	0.3	0.016	0	0.17
R14	0.13	0.049	0.17	0	0.098	0.58	0.47	0.19	0.17	0.34
R15	0.028	0.049	0.075	0.098	0	0.48	0.37	0.091	0.075	0.24
R16	0.45	0.53	0.4	0.58	0.48	0	0.11	0.39	0.4	0.24
R17	0.34	0.42	0.3	0.47	0.37	0.11	0	0.28	0.3	0.13
R18	0.062	0.14	0.016	0.19	0.091	0.39	0.28	0	0.016	0.15
R19	0.047	0.12	0	0.17	0.075	0.4	0.3	0.016	0	0.17

B1.5.2 Vertical, As-Printed Specimens

TABLE B.11. ANOVA analysis of vertical, as-printed Charpy impact toughness data from the refreshing powder reuse method.

Source	DF	Sum of Square	Mean Square	F Statistic	P-value
Groups	19	2.0724	0.1091	6.62	2.76E-07
Error	40	0.659	0.01648		
Total	59	2.7314	0.04629		

TABLE B.12. Tukey-Kramer analysis of vertical, as-printed Charpy impact toughness data from the refreshing powder reuse method.

(A)

Group	R2	R3	R4	R5	R6	R7	R8	R9	R10
R1	0.042	0.22	0.48	0.24	0.31	0.046	0.1	0.16	0.14
R2	0	0.26	0.52	0.2	0.35	0.0047	0.061	0.11	0.098
R3	0.26	0	0.26	0.46	0.086	0.27	0.32	0.38	0.36
R4	0.52	0.26	0	0.72	0.17	0.52	0.58	0.63	0.62
R5	0.2	0.46	0.72	0	0.55	0.19	0.14	0.085	0.1
R6	0.35	0.086	0.17	0.55	0	0.35	0.41	0.46	0.45
R7	0.0047	0.27	0.52	0.19	0.35	0	0.057	0.11	0.094
R8	0.061	0.32	0.58	0.14	0.41	0.057	0	0.053	0.037
R9	0.11	0.38	0.63	0.085	0.46	0.11	0.053	0	0.016
R10	0.098	0.36	0.62	0.1	0.45	0.094	0.037	0.016	0
R11	0.062	0.2	0.46	0.26	0.29	0.067	0.12	0.18	0.16
R12	0.082	0.18	0.44	0.28	0.27	0.087	0.14	0.2	0.18
R13	0.11	0.37	0.63	0.09	0.46	0.11	0.048	0.0047	0.011
R14	0.33	0.073	0.18	0.53	0.013	0.34	0.4	0.45	0.43
R15	0.003	0.26	0.52	0.2	0.35	0.0017	0.058	0.11	0.095
R16	0.12	0.38	0.64	0.082	0.47	0.11	0.056	0.0033	0.019
R17	0.17	0.43	0.69	0.032	0.51	0.16	0.11	0.053	0.069
R18	0.037	0.3	0.56	0.16	0.38	0.032	0.025	0.078	0.062
R19	0.003	0.26	0.52	0.2	0.35	0.0017	0.058	0.11	0.095

(B)

Group	R11	R12	R13	R14	R15	R16	R17	R18	R19	R20
R1	0.021	0.04	0.15	0.29	0.045	0.16	0.21	0.078	0.045	0.13
R2	0.062	0.082	0.11	0.33	0.003	0.12	0.17	0.037	0.003	0.09
R3	0.2	0.18	0.37	0.073	0.26	0.38	0.43	0.3	0.26	0.35
R4	0.46	0.44	0.63	0.18	0.52	0.64	0.69	0.56	0.52	0.61
R5	0.26	0.28	0.09	0.53	0.2	0.082	0.032	0.16	0.2	0.11
R6	0.29	0.27	0.46	0.013	0.35	0.47	0.51	0.38	0.35	0.44
R7	0.067	0.087	0.11	0.34	0.0017	0.11	0.16	0.032	0.0017	0.086
R8	0.12	0.14	0.048	0.4	0.058	0.056	0.11	0.025	0.058	0.029
R9	0.18	0.2	0.0047	0.45	0.11	0.0033	0.053	0.078	0.11	0.024
R10	0.16	0.18	0.011	0.43	0.095	0.019	0.069	0.062	0.095	0.008
R11	0	0.02	0.17	0.27	0.065	0.18	0.23	0.099	0.065	0.15
R12	0.02	0	0.19	0.25	0.085	0.2	0.25	0.12	0.085	0.17
R13	0.17	0.19	0	0.44	0.11	0.008	0.057	0.073	0.11	0.019
R14	0.27	0.25	0.44	0	0.34	0.45	0.5	0.37	0.34	0.42
R15	0.065	0.085	0.11	0.34	0	0.11	0.16	0.034	0	0.087
R16	0.18	0.2	0.008	0.45	0.11	0	0.049	0.081	0.11	0.027
R17	0.23	0.25	0.057	0.5	0.16	0.049	0	0.13	0.16	0.077
R18	0.099	0.12	0.073	0.37	0.034	0.081	0.13	0	0.034	0.054
R19	0.065	0.085	0.11	0.34	0	0.11	0.16	0.034	0	0.087

B1.5.3 Horizontal, Machined Specimens

TABLE B.13. ANOVA analysis of horizontal, machined Charpy impact toughness data from the refreshing powder reuse method.

Source	DF	Sum of Square	Mean Square	F Statistic	P-value
Groups	10	3.2417	0.3242	1.4702	0.2159
Error	22	4.8507	0.2205		
Total	32	8.0923	0.2529		

TABLE B.14. Tukey-Kramer analysis of horizontal, machined Charpy impact toughness data from the refreshing powder reuse method.

Group	R3	R5	R7	R9	R11	R13	R15	R17	R19	R20
R1	0.48	0.59	0.54	0.0097	0.41	0.013	0.027	0.31	0.18	0.58
R3	0	0.1	0.055	0.48	0.07	0.47	0.46	0.79	0.66	0.099
R5	0.1	0	0.046	0.58	0.17	0.57	0.56	0.9	0.76	0.0027
R7	0.055	0.046	0	0.53	0.13	0.53	0.51	0.85	0.72	0.043
R9	0.48	0.58	0.53	0	0.4	0.003	0.018	0.32	0.18	0.57
R11	0.07	0.17	0.13	0.4	0	0.4	0.39	0.72	0.59	0.17
R13	0.47	0.57	0.53	0.003	0.4	0	0.015	0.32	0.19	0.57
R15	0.46	0.56	0.51	0.018	0.39	0.015	0	0.34	0.2	0.56
R17	0.79	0.9	0.85	0.32	0.72	0.32	0.34	0	0.13	0.89
R19	0.66	0.76	0.72	0.18	0.59	0.19	0.2	0.13	0	0.76

B1.5.4 Vertical, Machined Specimens

TABLE B.15. ANOVA analysis of vertical, machined Charpy impact toughness data from the refreshing powder reuse method.

Source	DF	Sum of Square	Mean Square	F Statistic	P-value
Groups	10	0.7506	0.07506	3.7564	0.004611
Error	22	0.4396	0.01998		
Total	32	1.1903	0.0372		

TABLE B.16. Tukey-Kramer analysis of vertical, machined Charpy impact toughness data from the refreshing powder reuse method.

Group	R3	R5	R7	R9	R11	R13	R15	R17	R19	R20
R1	0.16	0.11	0.19	0.33	0.2	0.31	0.36	0.61	0.3	0.26
R3	0	0.048	0.033	0.16	0.042	0.15	0.2	0.45	0.14	0.1
R5	0.048	0	0.081	0.21	0.09	0.2	0.25	0.5	0.19	0.15
R7	0.033	0.081	0	0.13	0.0083	0.12	0.16	0.42	0.11	0.07
R9	0.16	0.21	0.13	0	0.12	0.01	0.034	0.29	0.025	0.061
R11	0.042	0.09	0.0083	0.12	0	0.11	0.16	0.41	0.097	0.062
R13	0.15	0.2	0.12	0.01	0.11	0	0.044	0.3	0.015	0.05
R15	0.2	0.25	0.16	0.034	0.16	0.044	0	0.25	0.059	0.094
R17	0.45	0.5	0.42	0.29	0.41	0.3	0.25	0	0.31	0.35
R19	0.14	0.19	0.11	0.025	0.097	0.015	0.059	0.31	0	0.035

B2 Statistical Analysis from the Single Batch Powder Reuse Method

B2.1 Avalanche Angle

TABLE B.17. ANOVA analysis of avalanche angle data from the single batch powder reuse method.

Source	DF	Sum of Square	Mean Square	F Statistic	P-value
Groups	9	1408.697	156.5219	14.6978	1.11E-16
Error	190	2023.376	10.6493		
Total	199	3432.073	17.2466		

TABLE B.18. Tukey-Kramer analysis of avalanche angle data from the single batch powder reuse method.

Group	S2	S3	S4	S5	S6	S7	S8	S9	S10
S1	1.47	2.97	5.96	4.57	5.54	6.86	7.29	7.85	8.26
S2	0	1.5	4.5	3.11	4.08	5.39	5.83	6.38	6.8
S3	1.5	0	2.99	1.6	2.57	3.89	4.32	4.88	5.29
S4	4.5	2.99	0	1.39	0.42	0.9	1.33	1.88	2.3
S5	3.11	1.6	1.39	0	0.97	2.29	2.72	3.27	3.69
S6	4.08	2.57	0.42	0.97	0	1.32	1.75	2.31	2.72
S7	5.39	3.89	0.9	2.29	1.32	0	0.43	0.99	1.4
S8	5.83	4.32	1.33	2.72	1.75	0.43	0	0.55	0.97
S9	6.38	4.88	1.88	3.27	2.31	0.99	0.55	0	0.42

B2.2 Hardness

TABLE B.19. ANOVA analysis of hardness data from the single batch powder reuse method.

Source	DF	Sum of Square	Mean Square	F Statistic	P-value
Groups	9	965.068	107.2298	4.9015	2.39E-05
Error	90	1968.912	21.8768		
Total	99	2933.98	29.6362		

TABLE B.20. Tukey-Kramer analysis of hardness data from the single batch powder reuse method.

Group	S2	S3	S4	S5	S6	S7	S8	S9	S10
S1	4.1	7.85	7.7	2.13	5.41	11.04	3.42	7.53	7.42
S2	0	3.75	3.6	1.97	1.31	6.94	0.68	3.43	3.32
S3	3.75	0	0.15	5.72	2.44	3.19	4.43	0.32	0.43
S4	3.6	0.15	0	5.57	2.29	3.34	4.28	0.17	0.28
S5	1.97	5.72	5.57	0	3.28	8.91	1.29	5.4	5.29
S6	1.31	2.44	2.29	3.28	0	5.63	1.99	2.12	2.01
S7	6.94	3.19	3.34	8.91	5.63	0	7.62	3.51	3.62
S8	0.68	4.43	4.28	1.29	1.99	7.62	0	4.11	4
S9	3.43	0.32	0.17	5.4	2.12	3.51	4.11	0	0.11

B2.3 Charpy Impact Toughness

B2.3.1 Horizontal, As-Printed Specimens

TABLE B.21. ANOVA analysis of horizontal, as-printed Charpy impact toughness data from the single batch powder reuse method.

Source	DF	Sum of Square	Mean Square	F Statistic	P-value
Groups	9	3.2208	0.3579	72.7272	2.61E-13
Error	20	0.09841	0.004921		
Total	29	3.3192	0.1145		

TABLE B.22. Tukey-Kramer analysis of horizontal, as-printed Charpy impact toughness data from the single batch powder reuse method.

Group	S2	S3	S4	S5	S6	S7	S8	S9	S10
S1	0.53	0.43	0.19	0.41	0.32	0.8	0.18	0.11	0.35
S2	0	0.96	0.72	0.95	0.85	1.33	0.71	0.64	0.88
S3	0.96	0	0.25	0.015	0.11	0.37	0.25	0.32	0.078
S4	0.72	0.25	0	0.23	0.13	0.61	0.008	0.079	0.17
S5	0.95	0.015	0.23	0	0.099	0.38	0.24	0.31	0.063
S6	0.85	0.11	0.13	0.099	0	0.48	0.14	0.21	0.036
S7	1.33	0.37	0.61	0.38	0.48	0	0.62	0.69	0.45
S8	0.71	0.25	0.008	0.24	0.14	0.62	0	0.071	0.17
S9	0.64	0.32	0.079	0.31	0.21	0.69	0.071	0	0.25

B2.3.2 Vertical, As-Printed Specimens

TABLE B.23. ANOVA analysis of vertical, as-printed Charpy impact toughness data from the single batch powder reuse method.

Source	DF	Sum of Square	Mean Square	F Statistic	P-value
Groups	9	0.866	0.09622	3.0899	0.01706
Error	20	0.6228	0.03114		
Total	29	1.4888	0.05134		

TABLE B.24. Tukey-Kramer analysis of vertical, as-printed Charpy impact toughness data from the single batch powder reuse method.

Group	S2	S3	S4	S5	S6	S7	S8	S9	S10
S1	0.17	0.32	0.32	0.068	0.21	0.36	0.046	0.06	0.14
S2	0	0.49	0.49	0.1	0.38	0.53	0.22	0.23	0.31
S3	0.49	0	0.0047	0.39	0.11	0.042	0.27	0.26	0.18
S4	0.49	0.0047	0	0.38	0.11	0.047	0.27	0.26	0.18
S5	0.1	0.39	0.38	0	0.28	0.43	0.11	0.13	0.2
S6	0.38	0.11	0.11	0.28	0	0.15	0.16	0.15	0.074
S7	0.53	0.042	0.047	0.43	0.15	0	0.32	0.3	0.23
S8	0.22	0.27	0.27	0.11	0.16	0.32	0	0.014	0.091
S9	0.23	0.26	0.26	0.13	0.15	0.3	0.014	0	0.077

APPENDIX C

XPS Curve Analysis

The following figures show the curve fitting that was carried out on XPS curves during the powder reuse studies in Chapters 7 and 8 of this thesis. Sub-figure (e) of each figure shows the survey spectrum scan taken prior to the measurement of each individual peak.

Also measured were peak locations for Cu, Fe, Mn, Ti, and Zn as they were included as measured elements in the powder suppliers specification sheet. However, none of these elements were present in sufficient quantity on the surface of powders to produce a peak from which curves could be fitted, so they were omitted in analysis.

C1 XPS Curves: Refreshing Powder Reuse Study

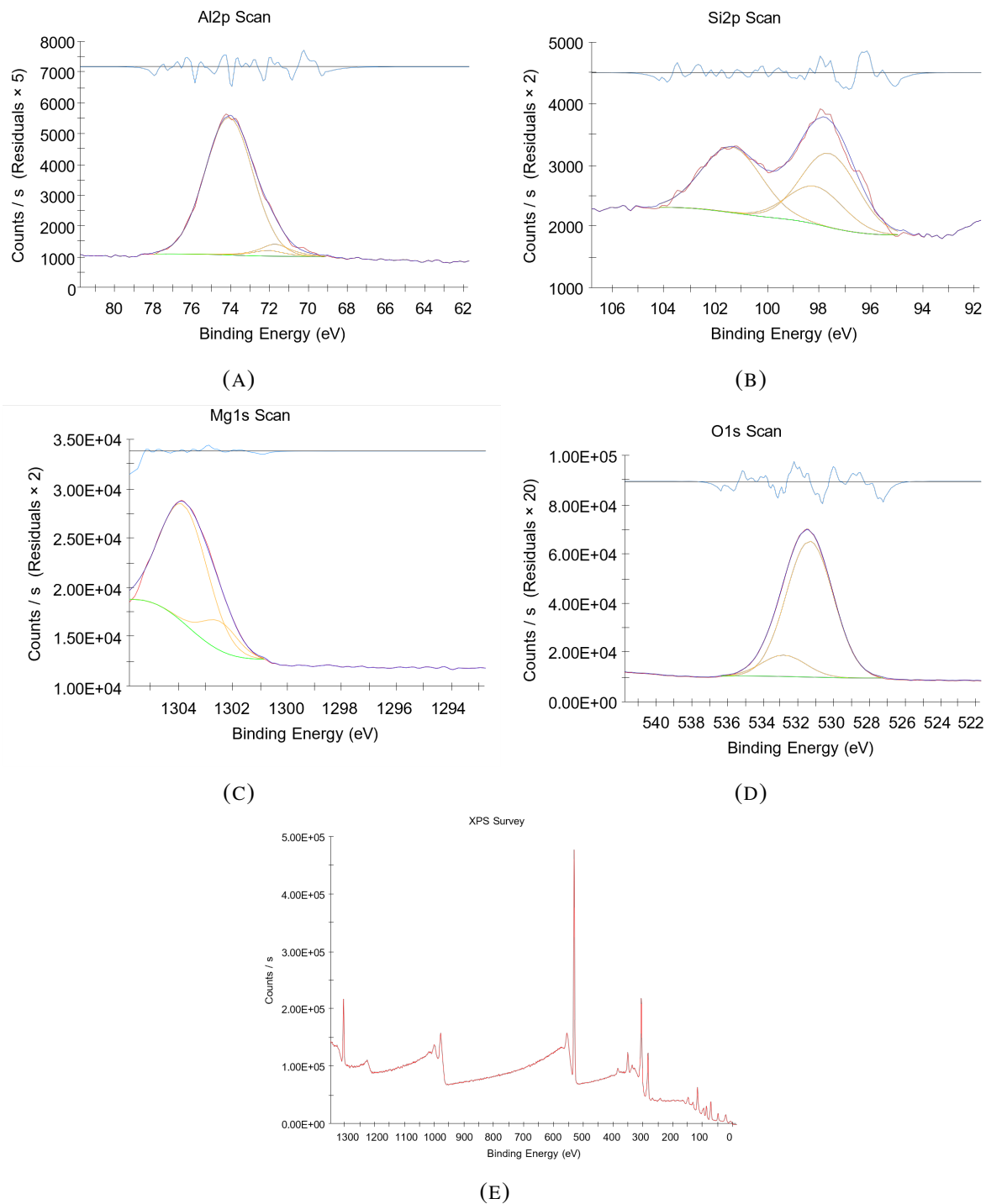


FIGURE C.1. R1 XPS Curves with fitted peaks.

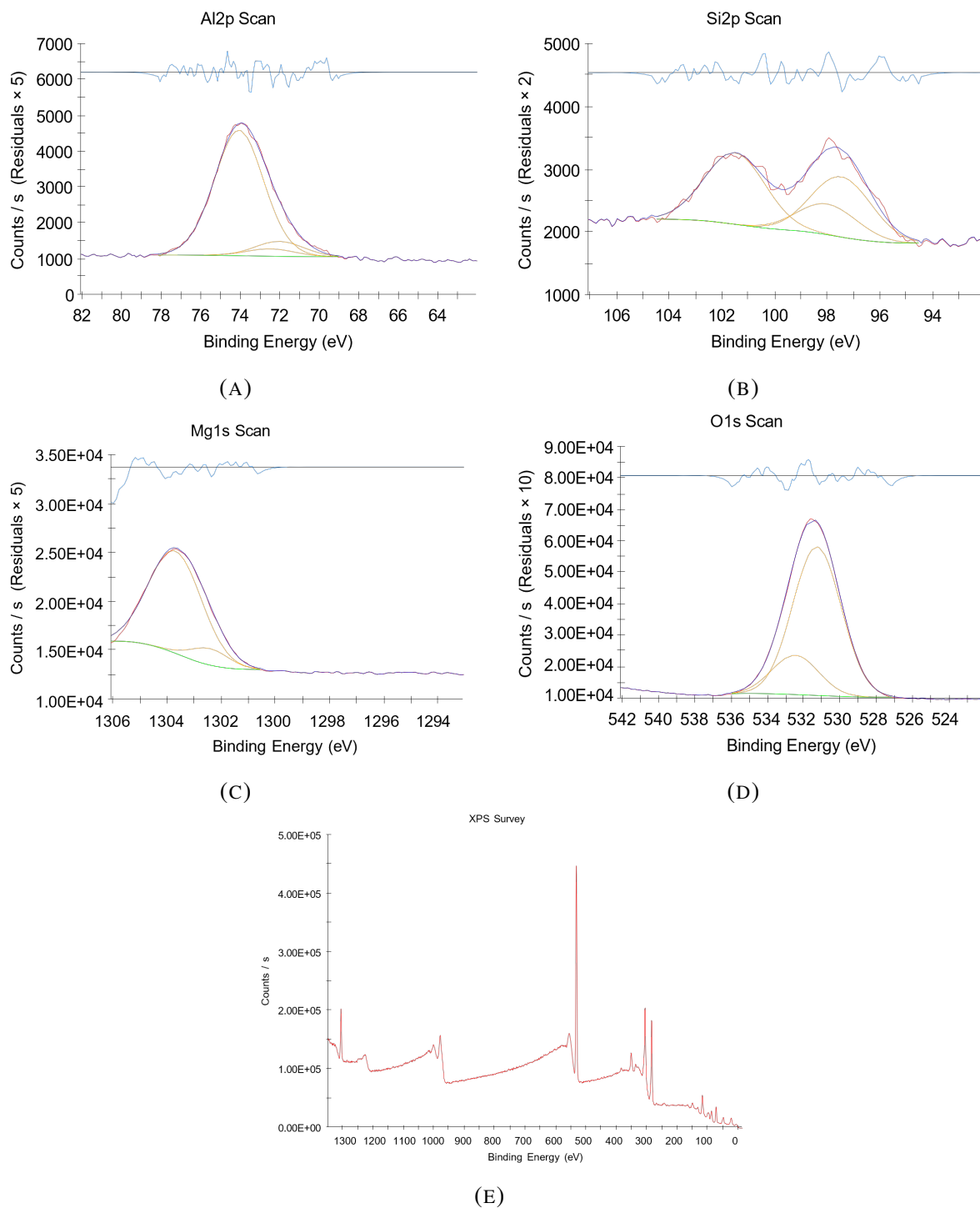


FIGURE C.2. R2 XPS Curves with fitted peaks.

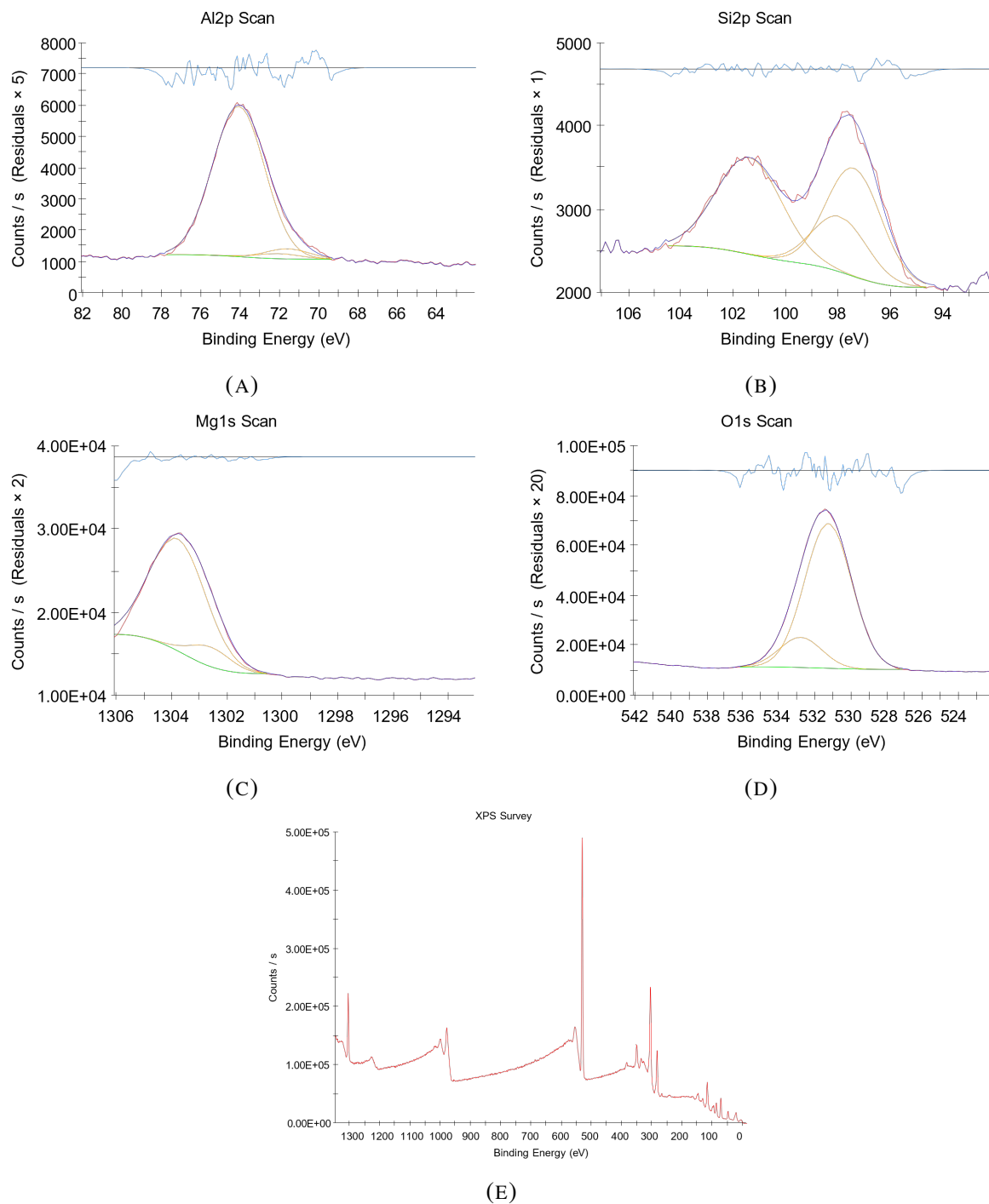


FIGURE C.3. R3 XPS Curves with fitted peaks.

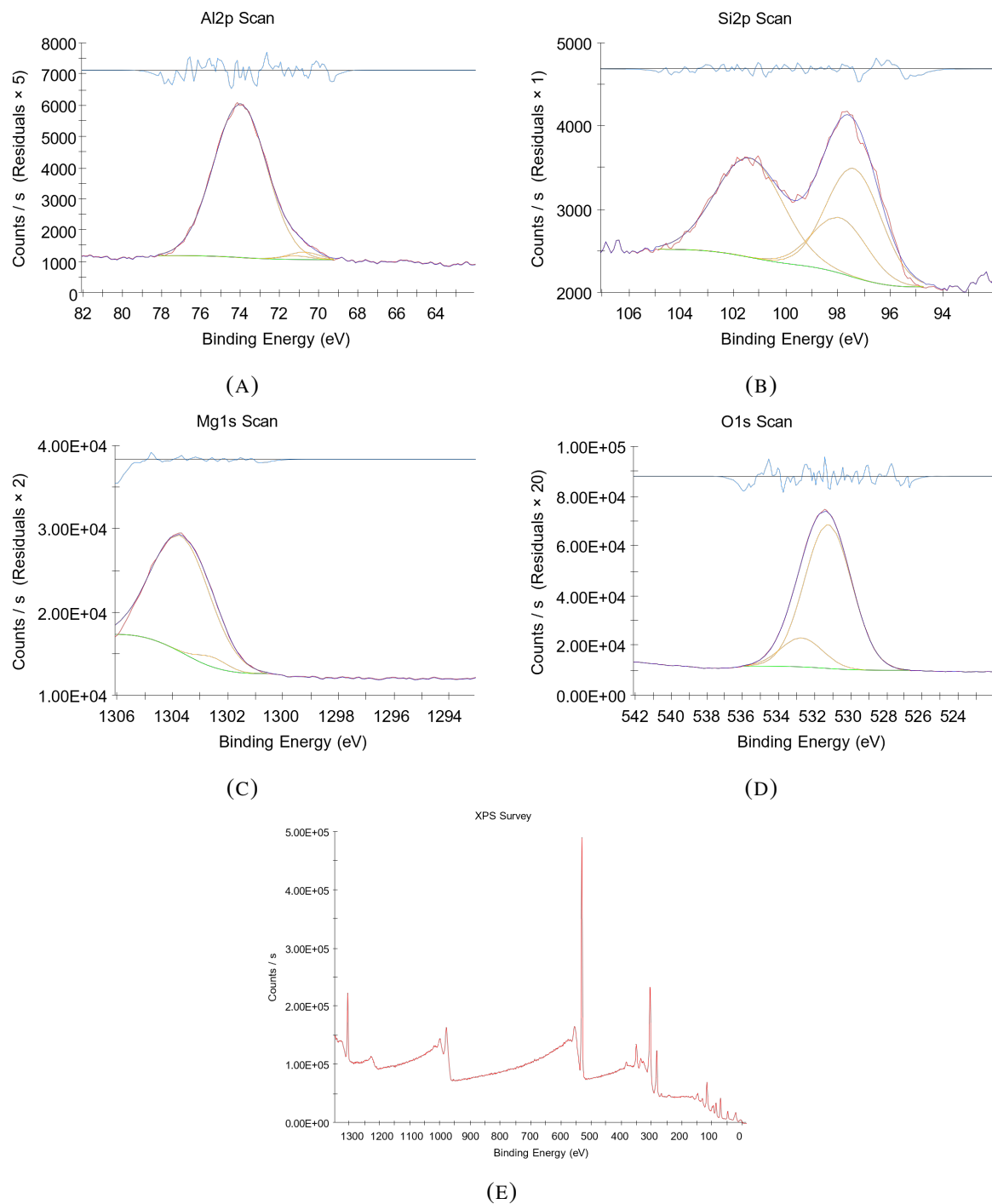


FIGURE C.4. R4 XPS Curves with fitted peaks.

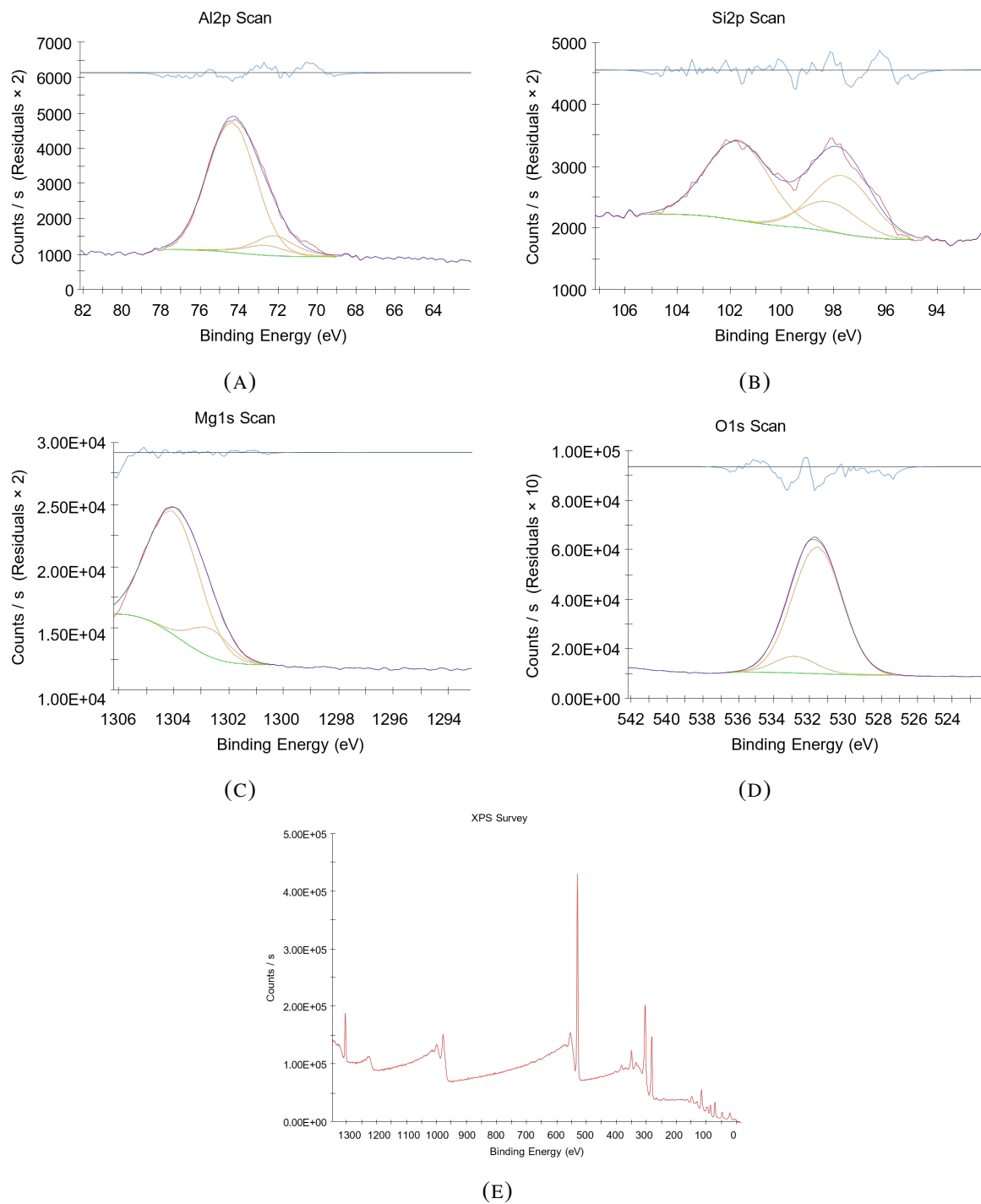


FIGURE C.5. R5 XPS Curves with fitted peaks.

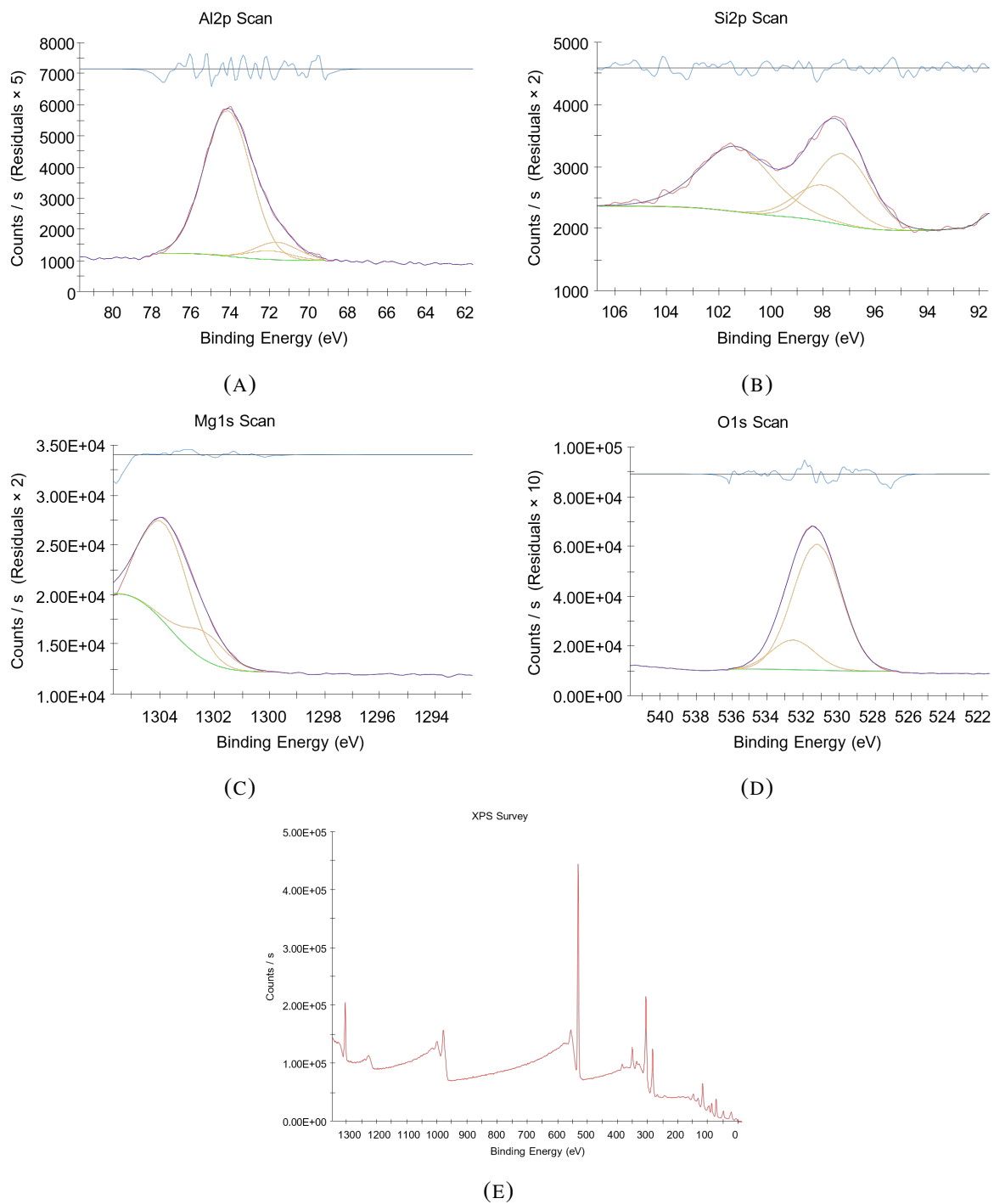


FIGURE C.6. R6 XPS Curves with fitted peaks.

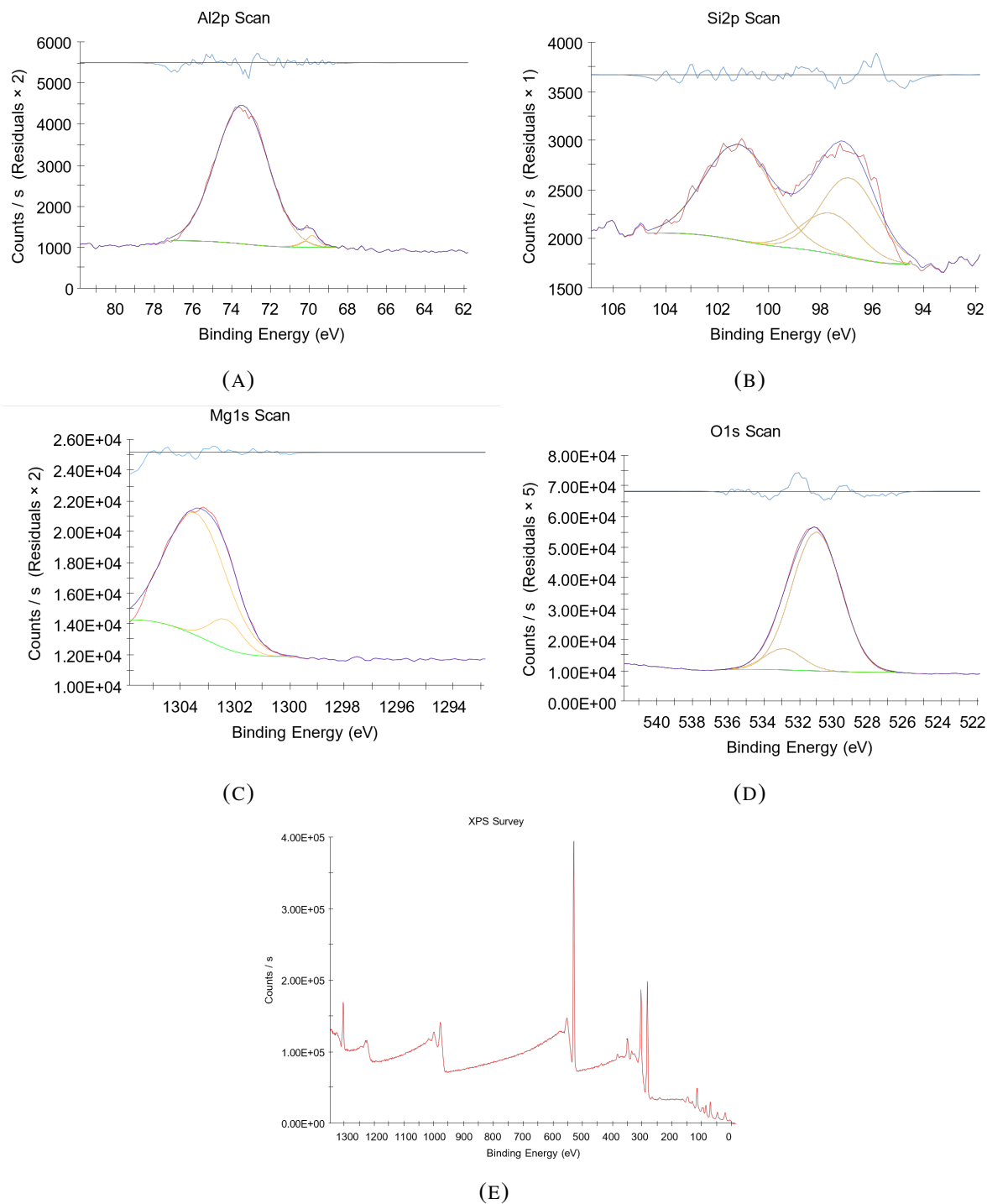


FIGURE C.7. R7 XPS Curves with fitted peaks.

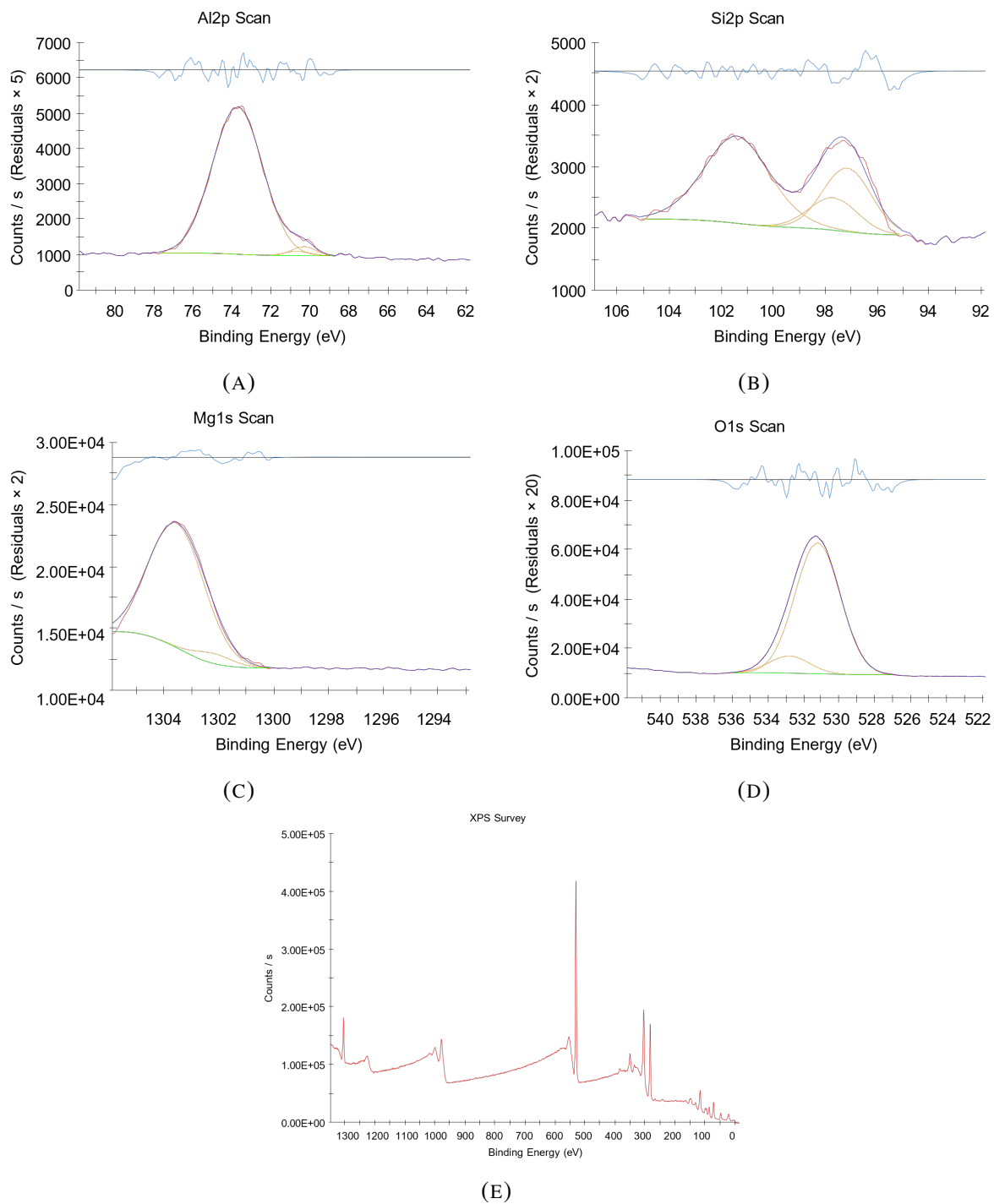


FIGURE C.8. R8 XPS Curves with fitted peaks.

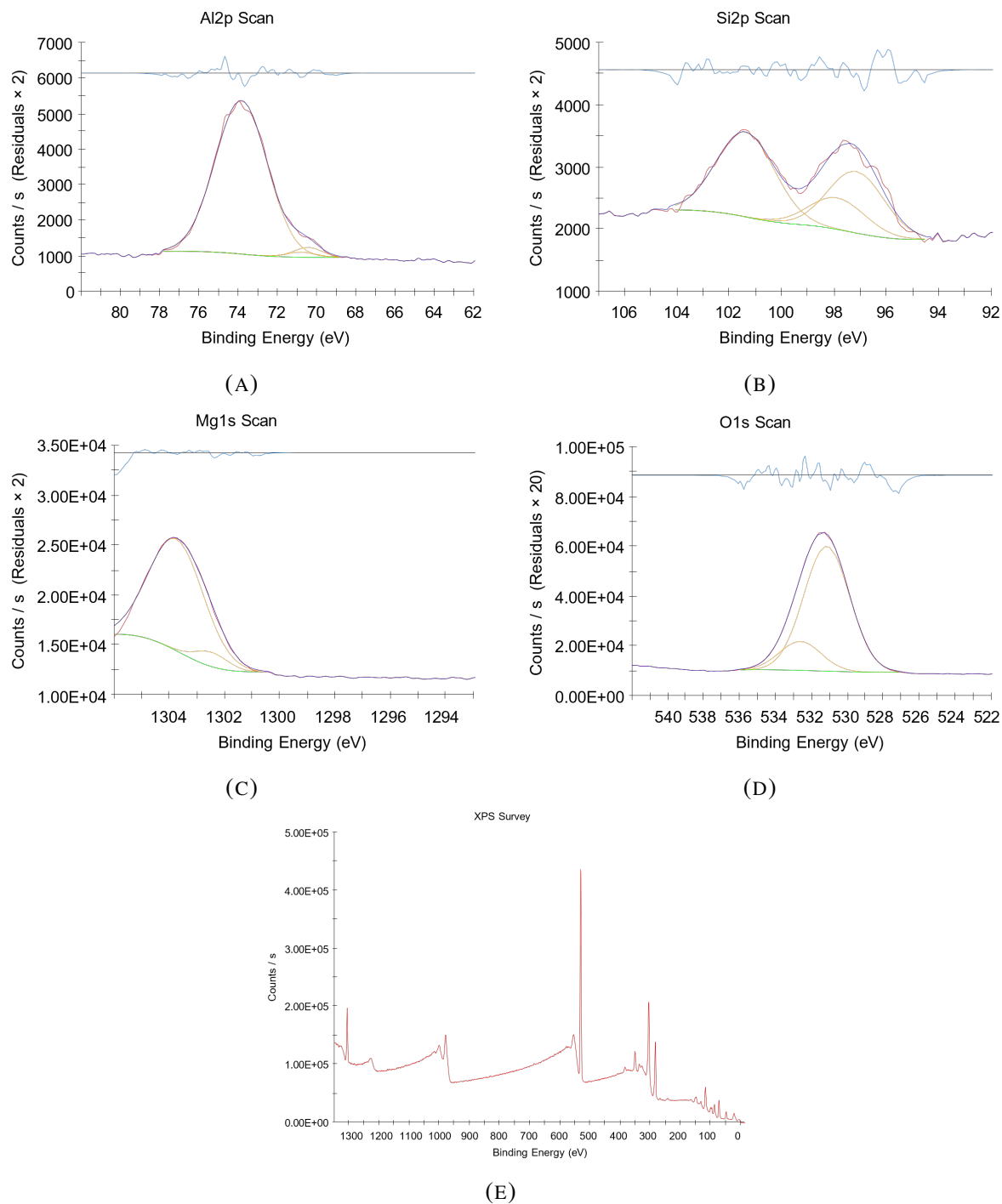


FIGURE C.9. R9 XPS Curves with fitted peaks.

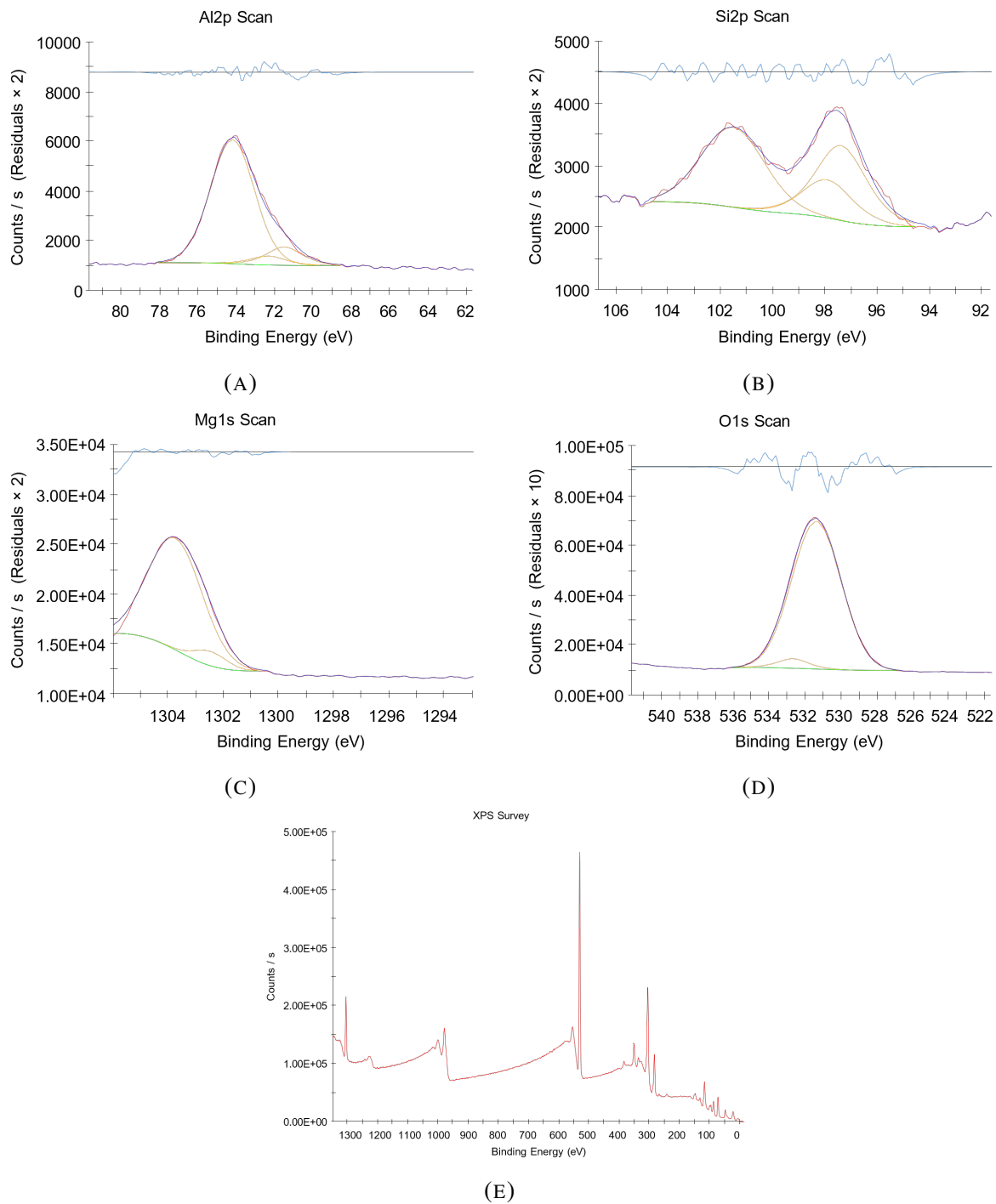


FIGURE C.10. R10 XPS Curves with fitted peaks.

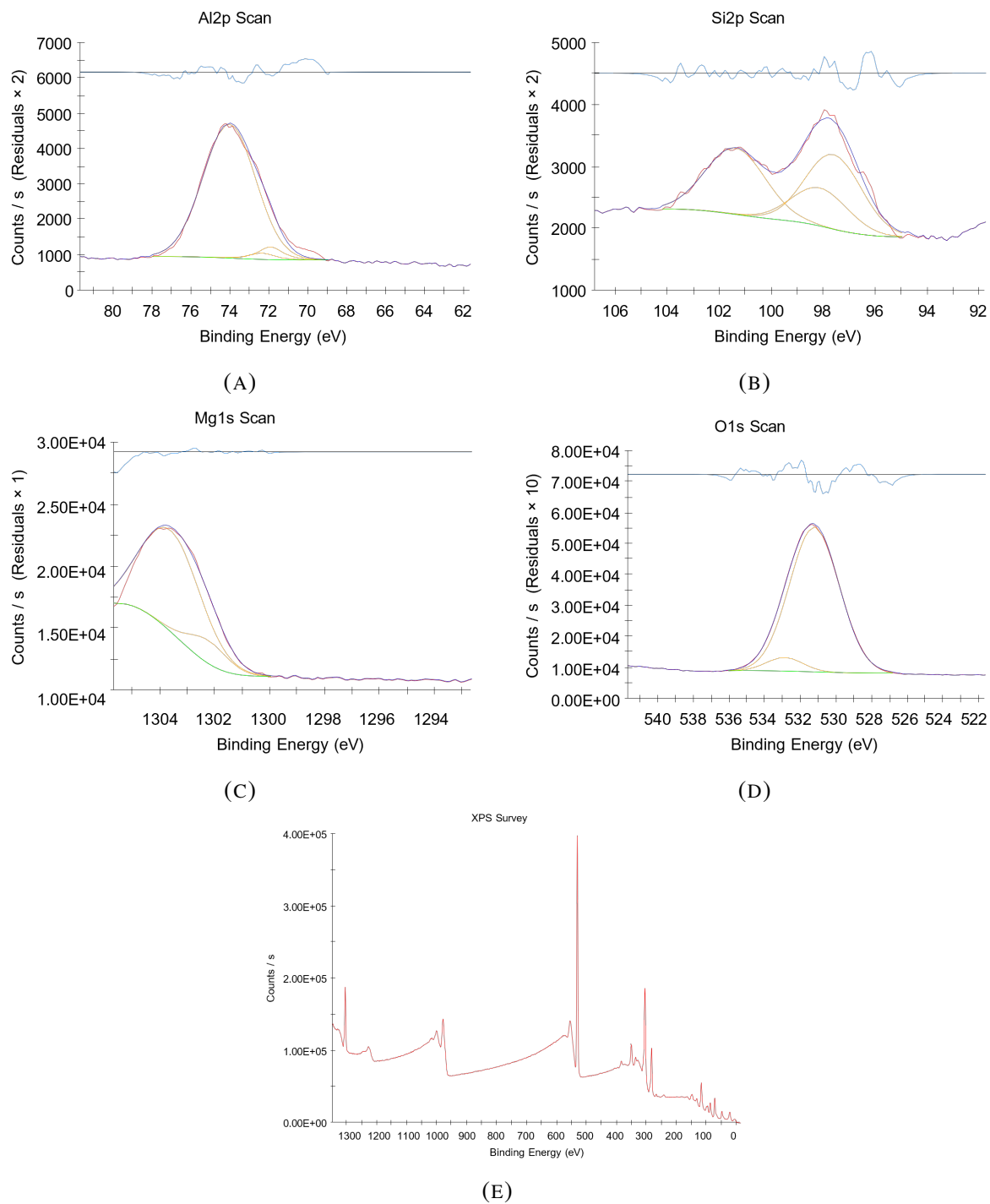


FIGURE C.11. R11 XPS Curves with fitted peaks.

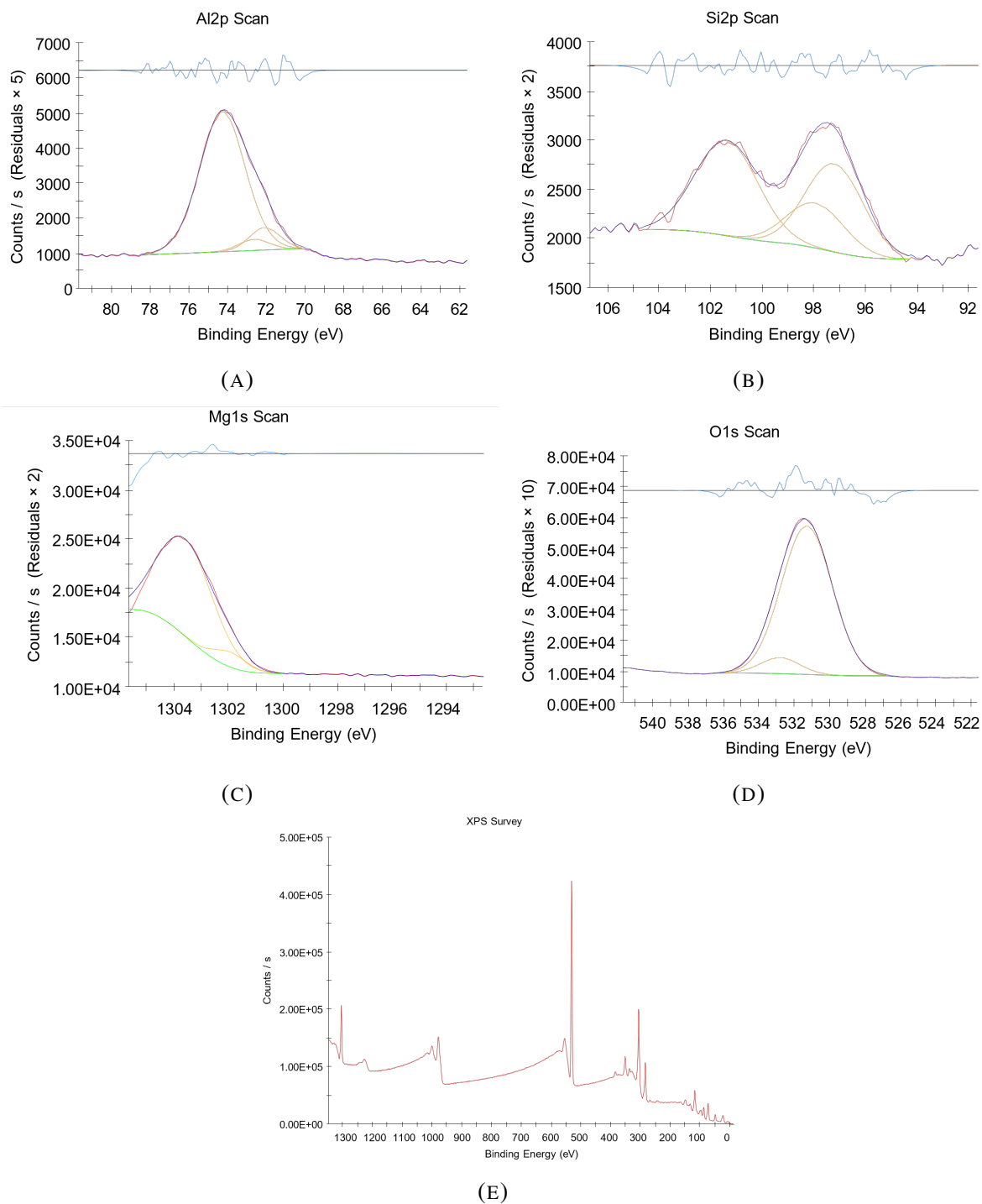


FIGURE C.12. R12 XPS Curves with fitted peaks.

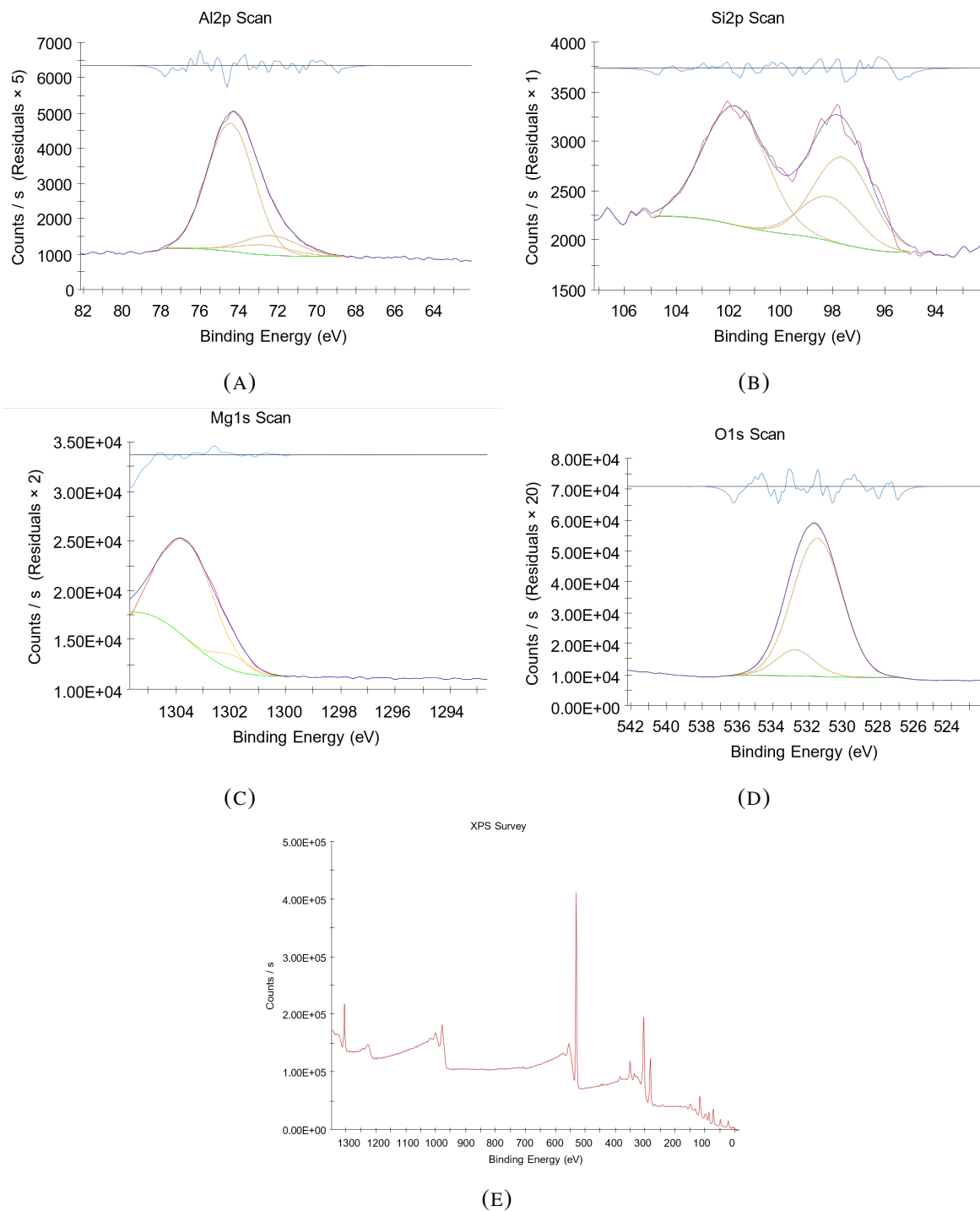


FIGURE C.13. R13 XPS Curves with fitted peaks.

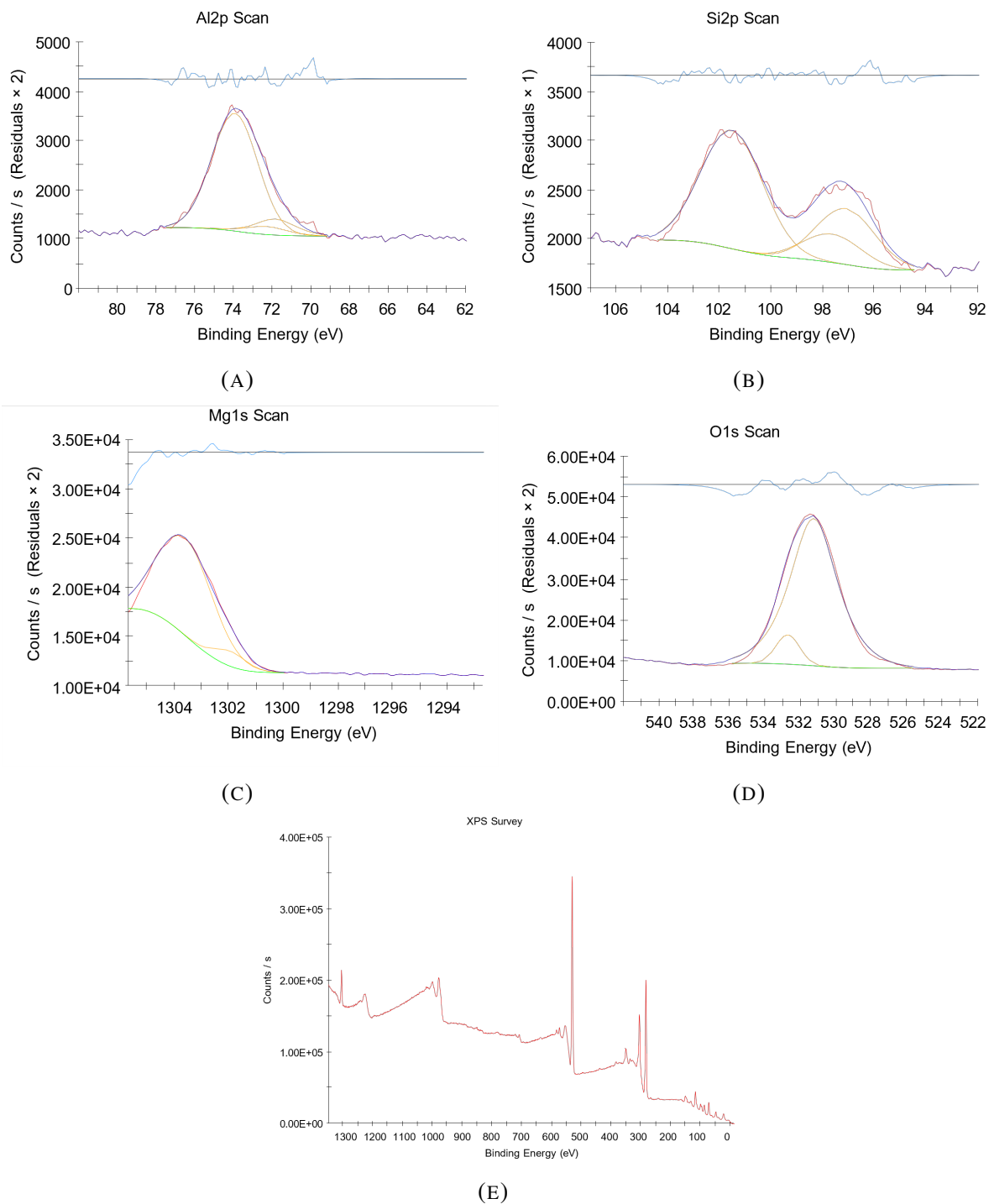


FIGURE C.14. R14 XPS Curves with fitted peaks.

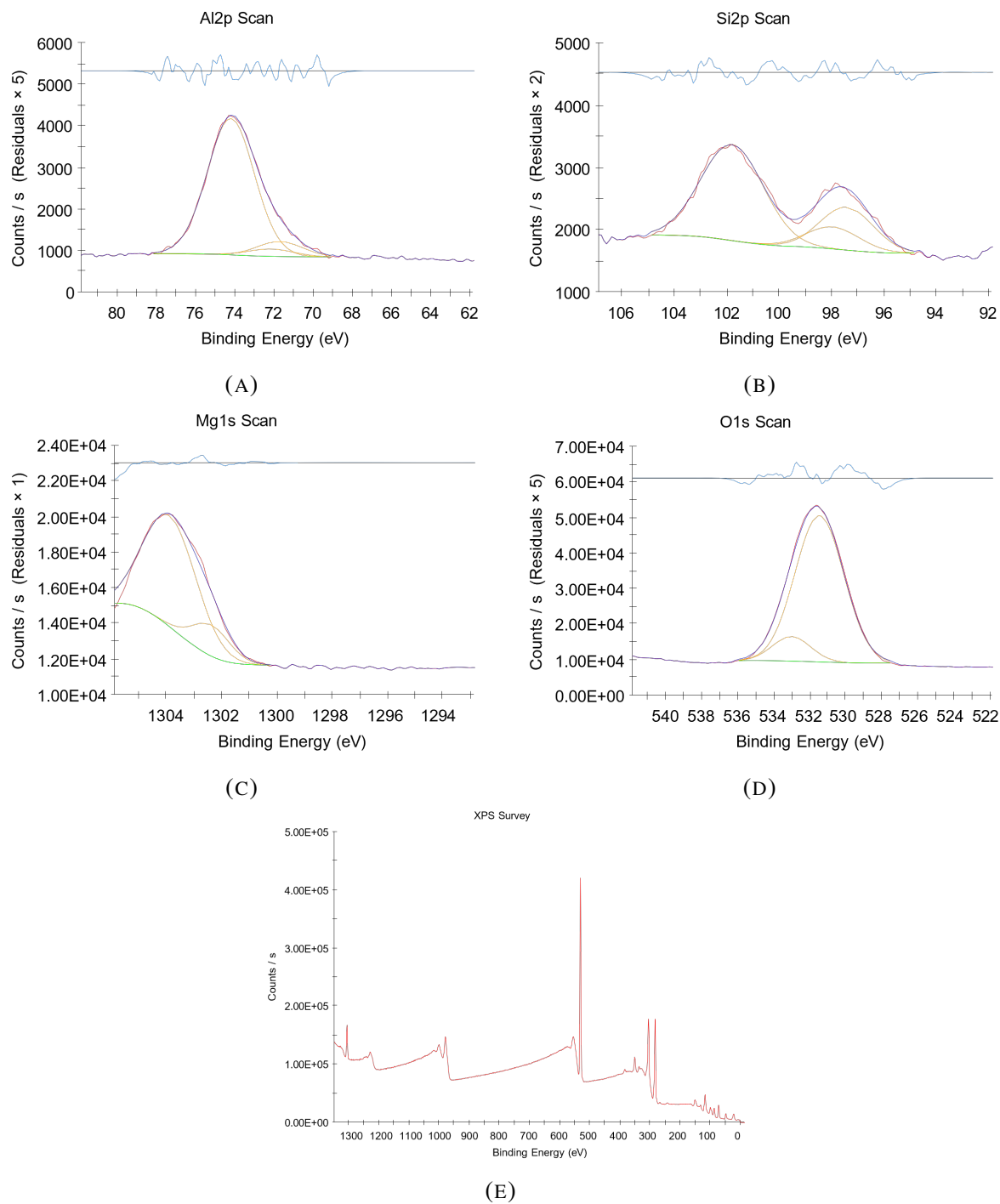


FIGURE C.15. R15 XPS Curves with fitted peaks.

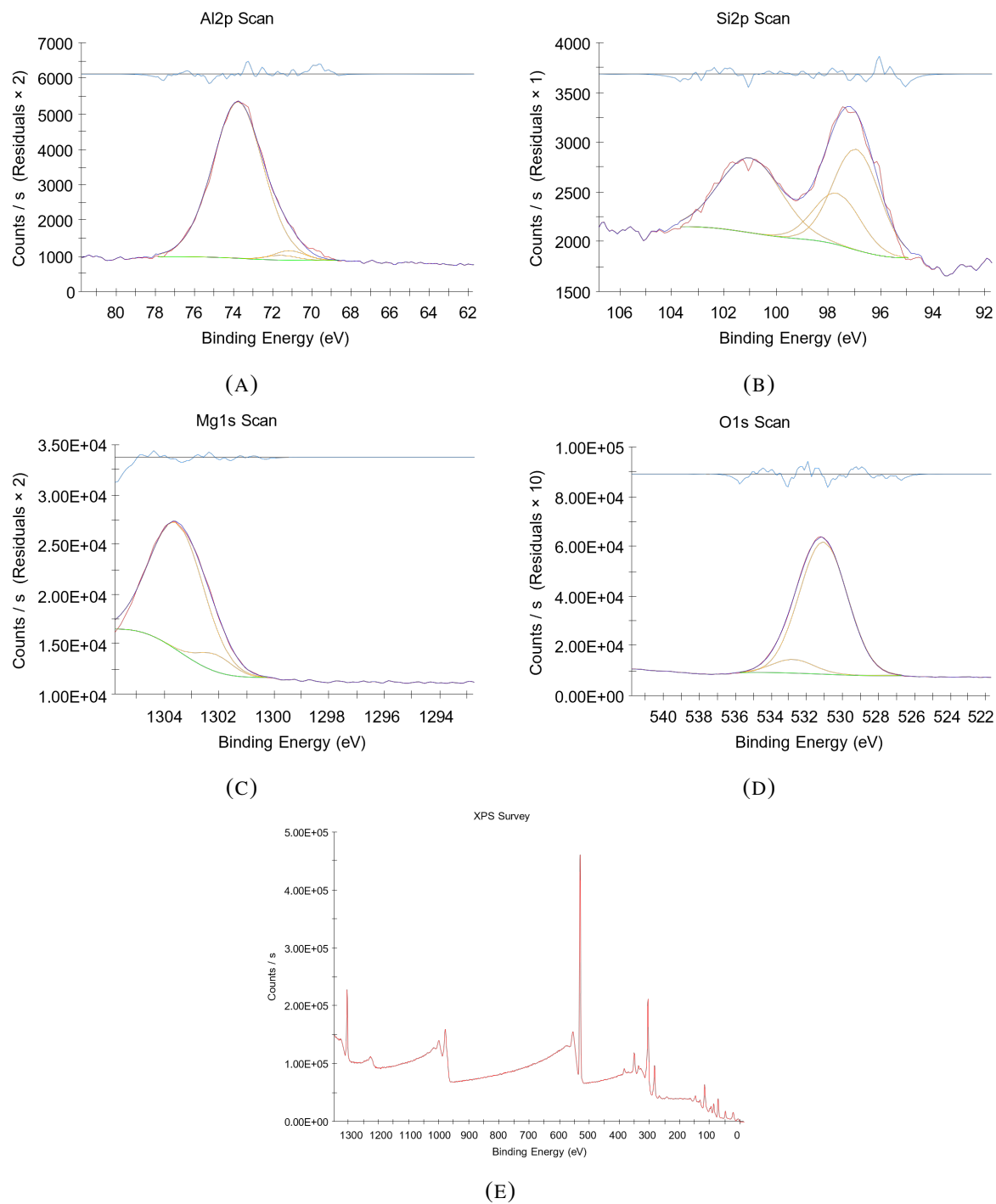


FIGURE C.16. R16 XPS Curves with fitted peaks.

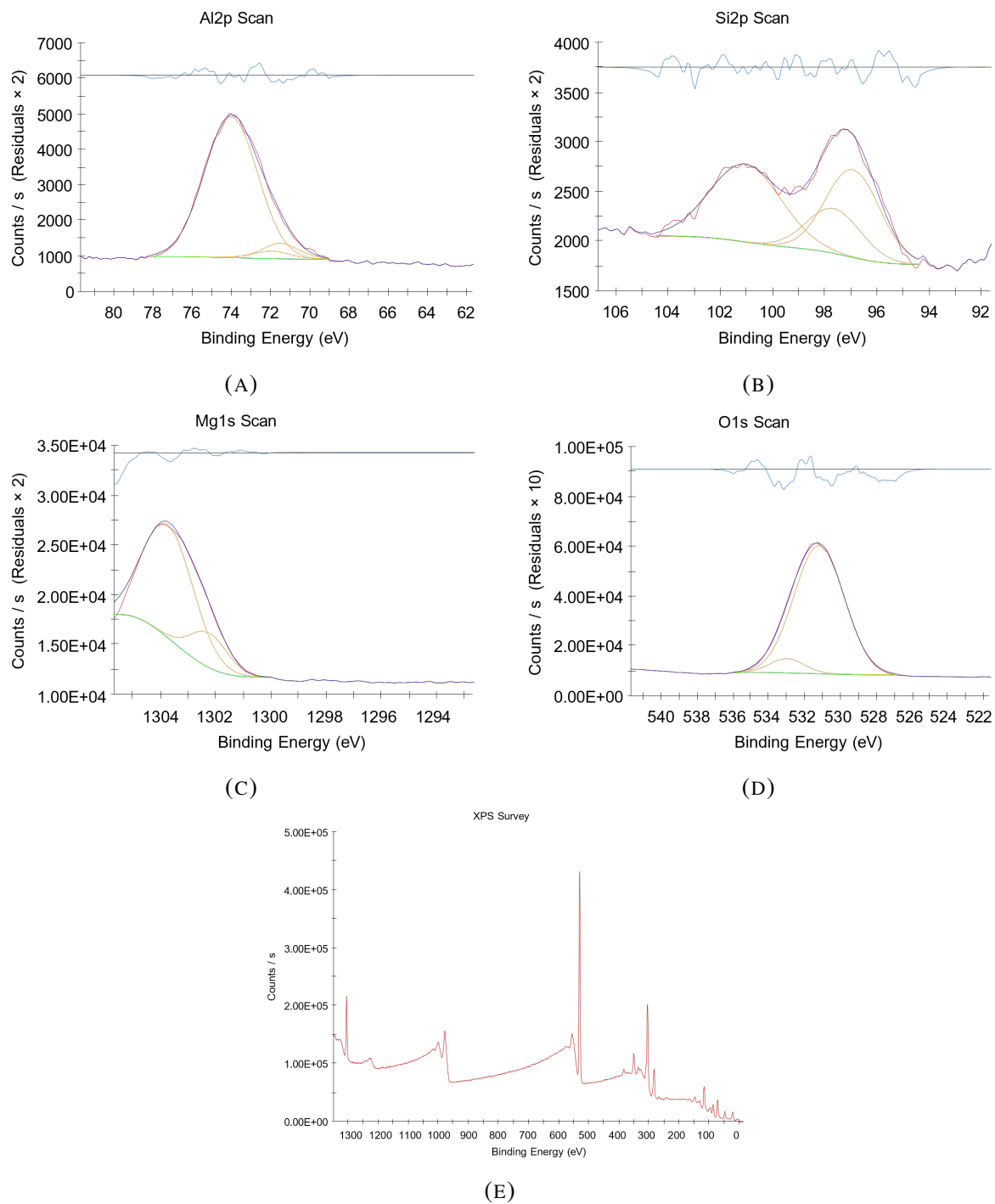


FIGURE C.17. R17 XPS Curves with fitted peaks.

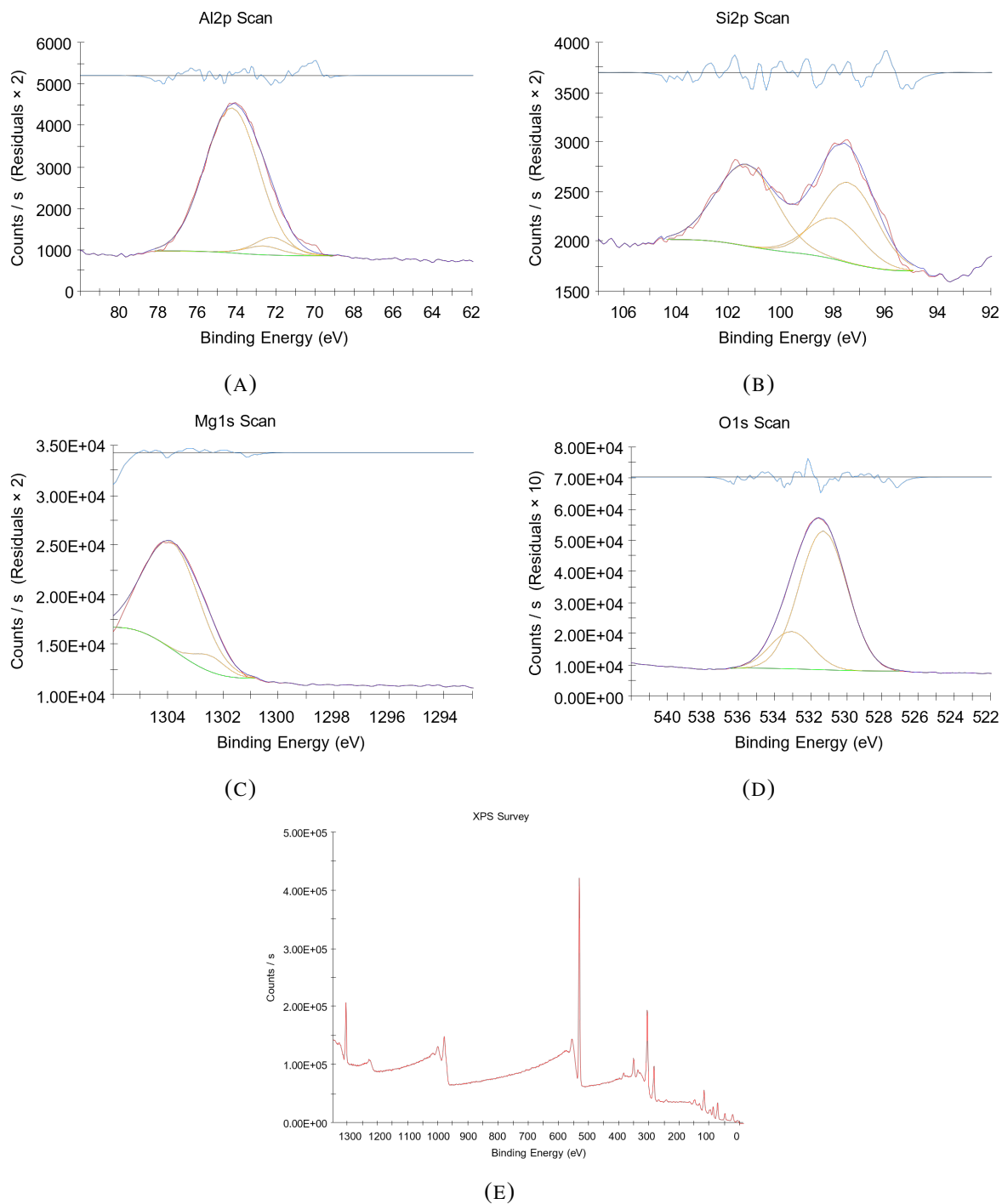


FIGURE C.18. R18 XPS Curves with fitted peaks.

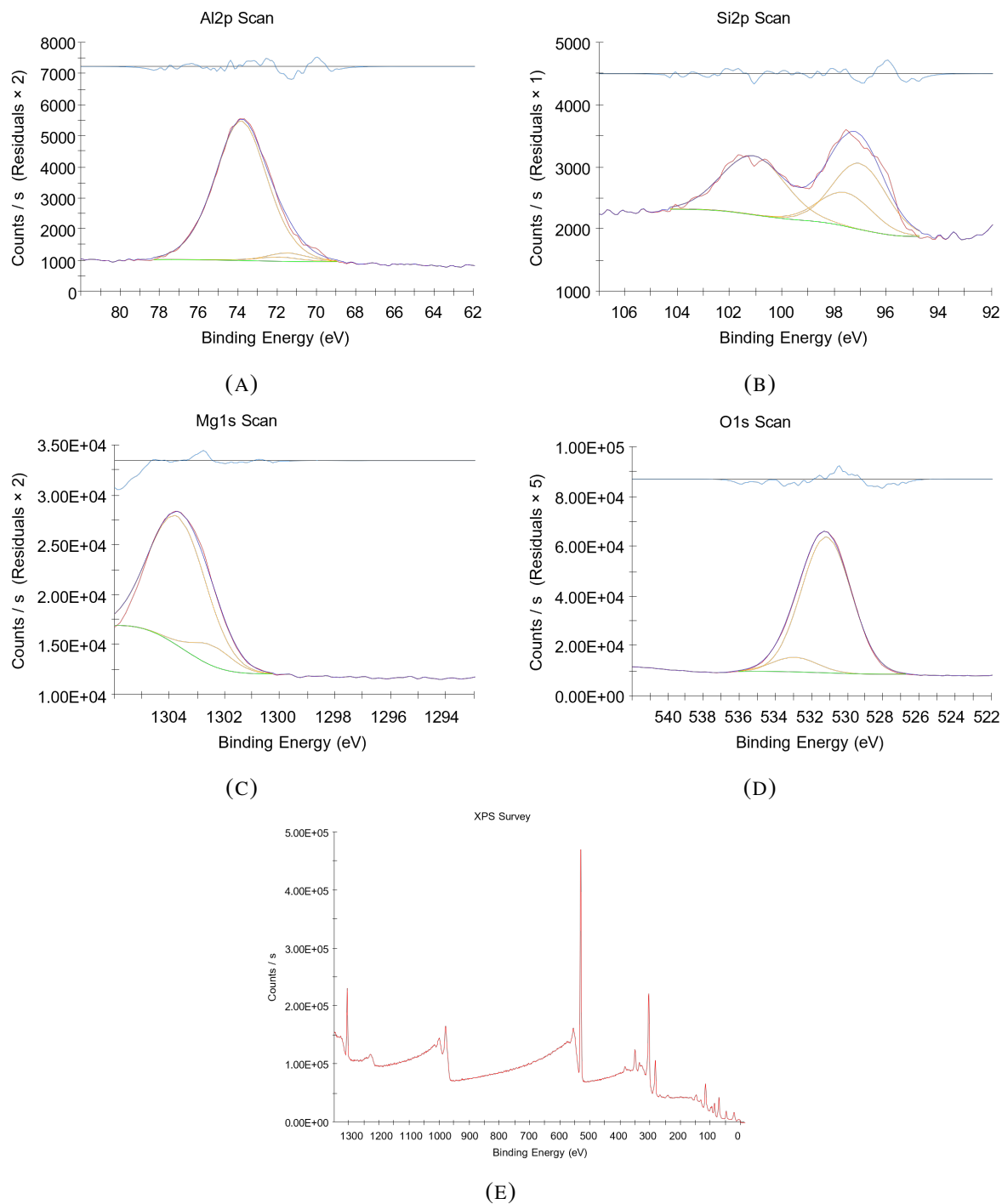


FIGURE C.19. R19 XPS Curves with fitted peaks.

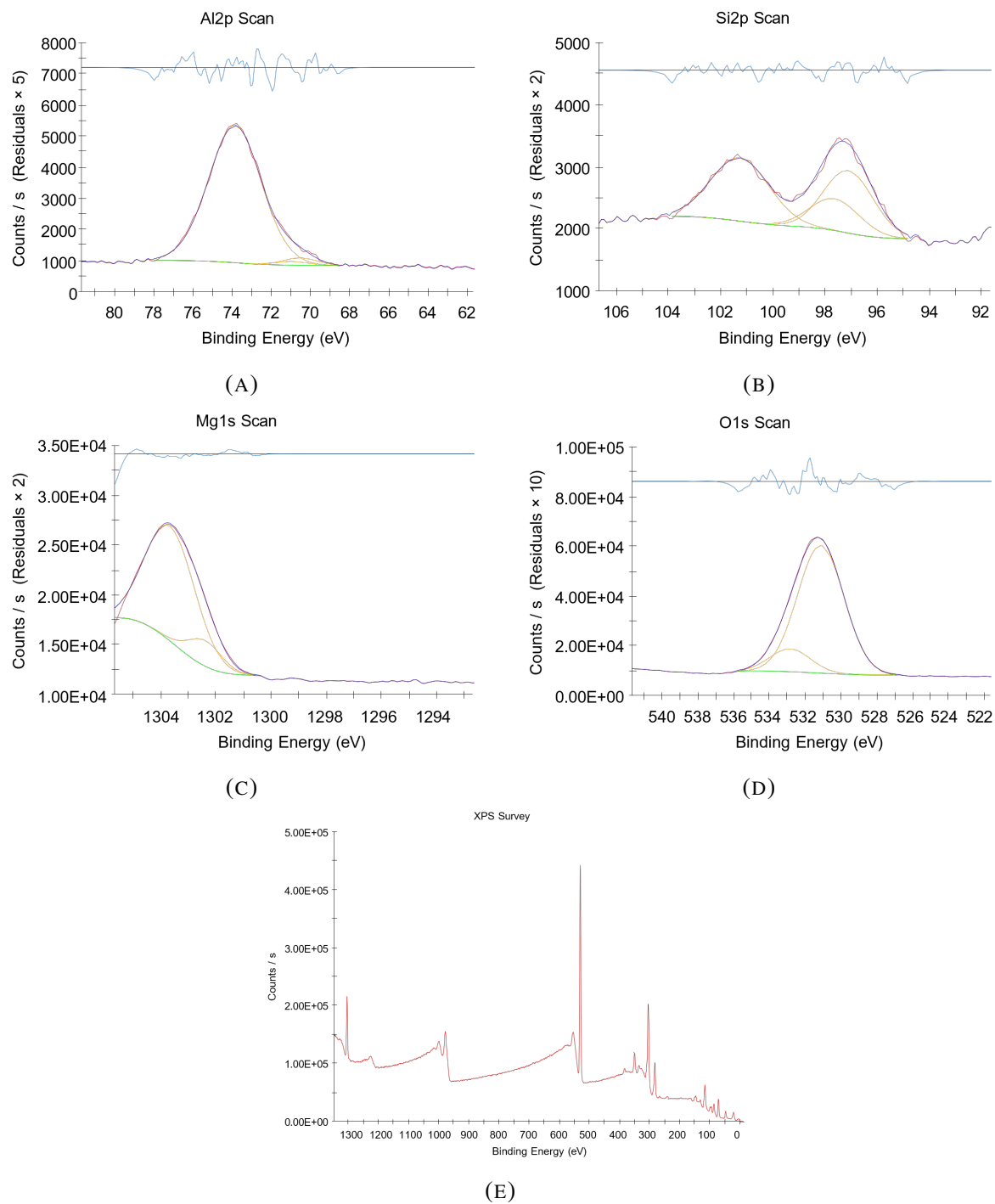


FIGURE C.20. R20 XPS Curves with fitted peaks.

C2 XPS Curves: Single Batch Powder Reuse Study

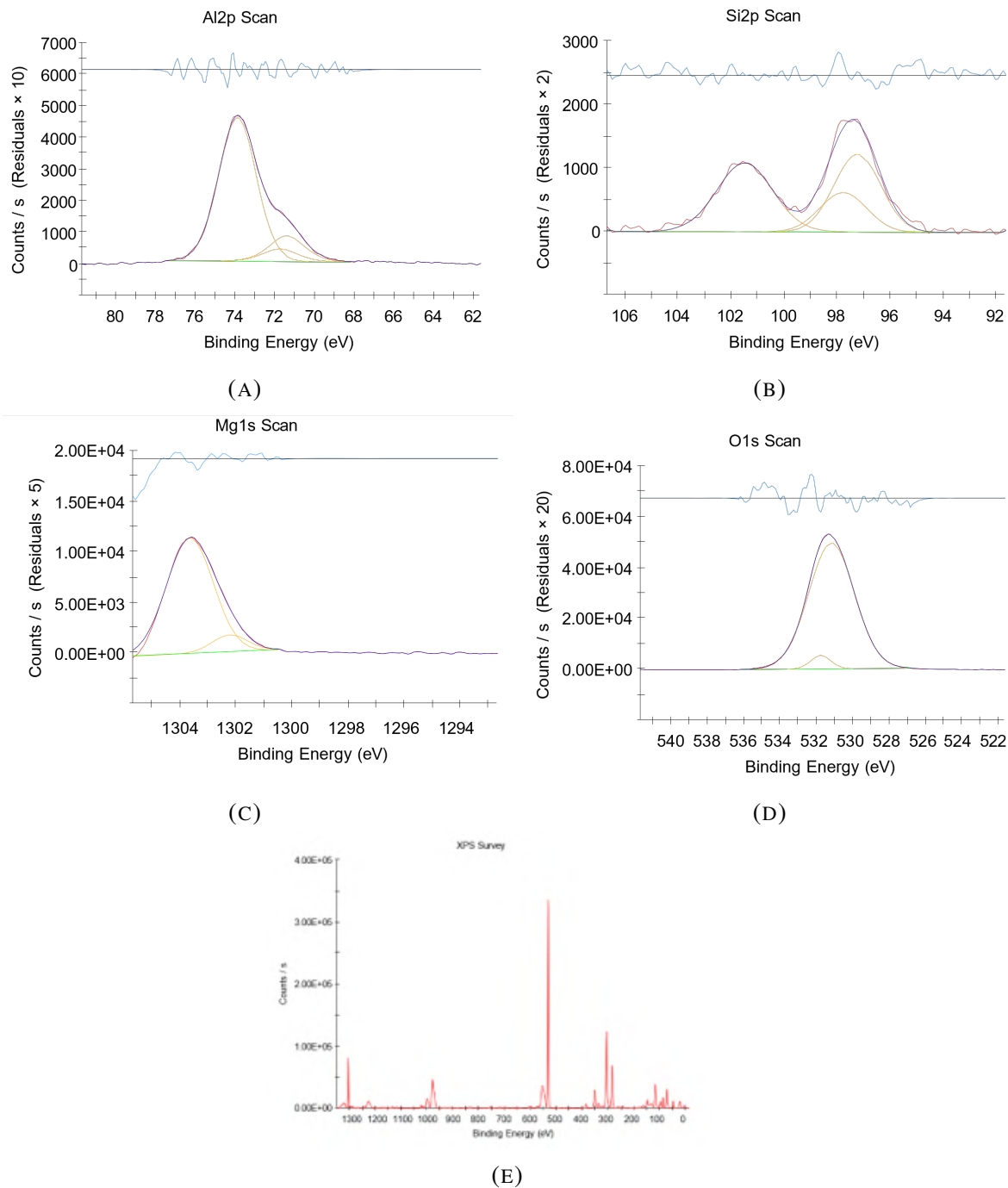


FIGURE C.21. S1 XPS Curves with fitted peaks.

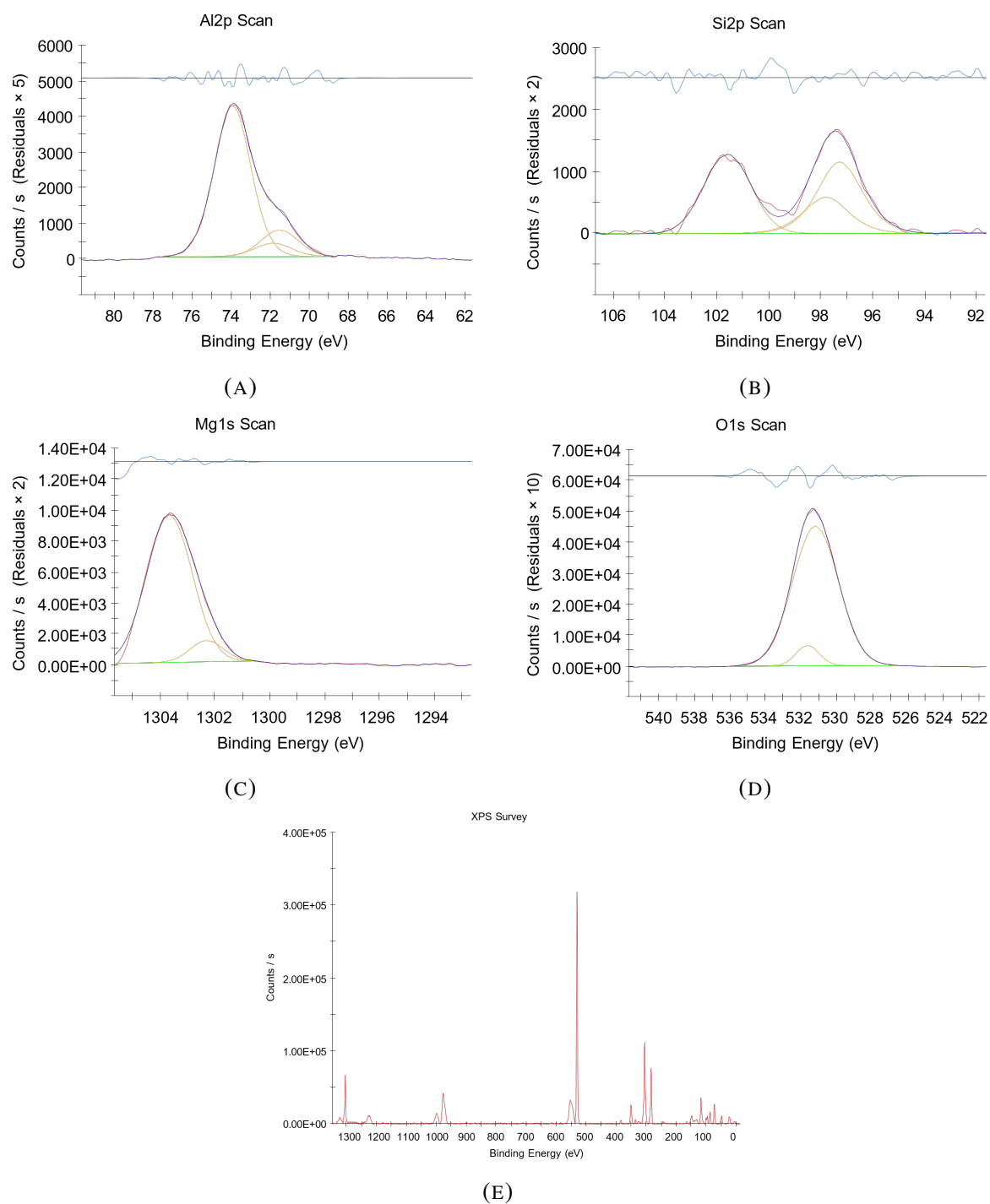


FIGURE C.22. S2 XPS Curves with fitted peaks.

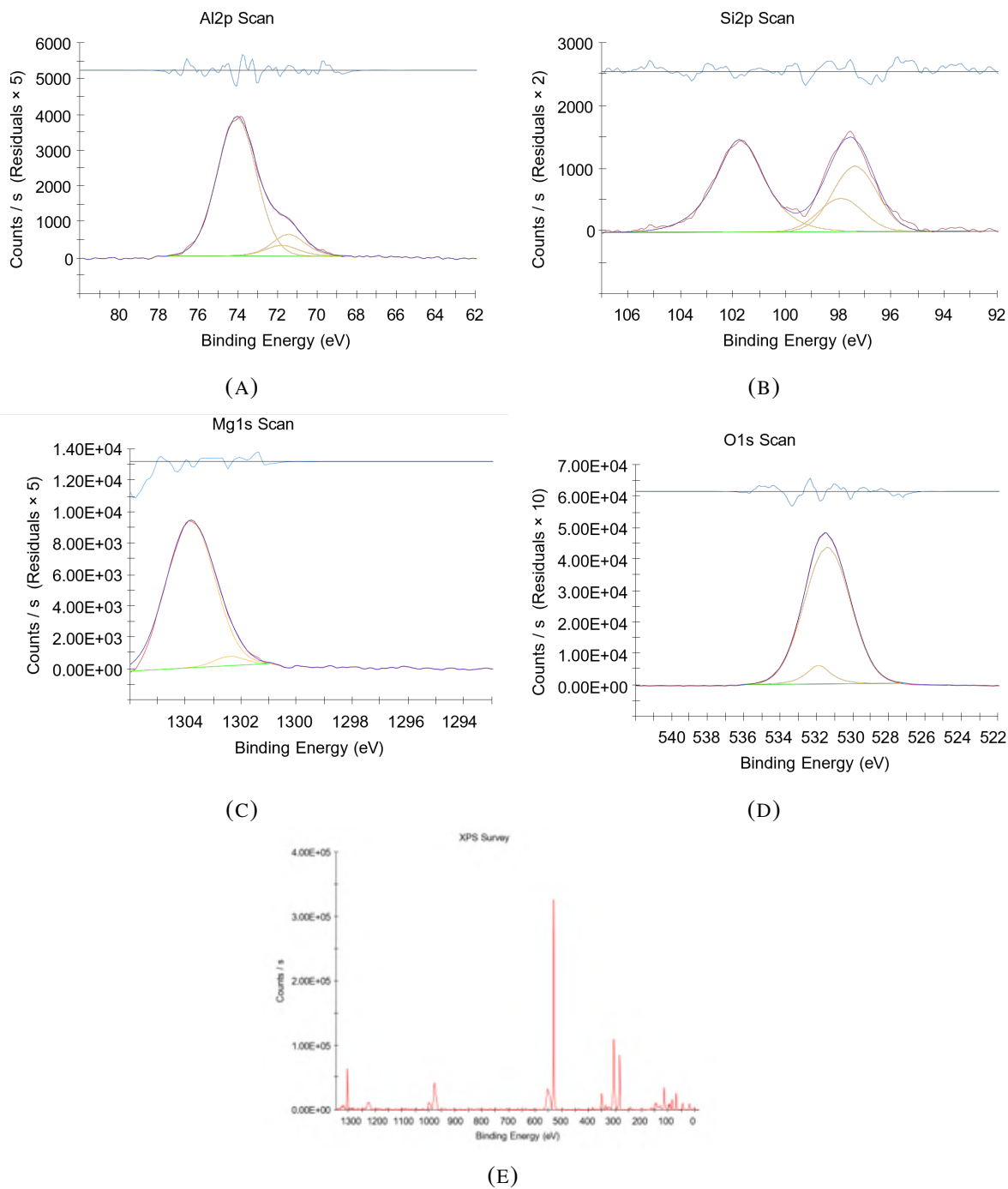


FIGURE C.23. S3 XPS Curves with fitted peaks.

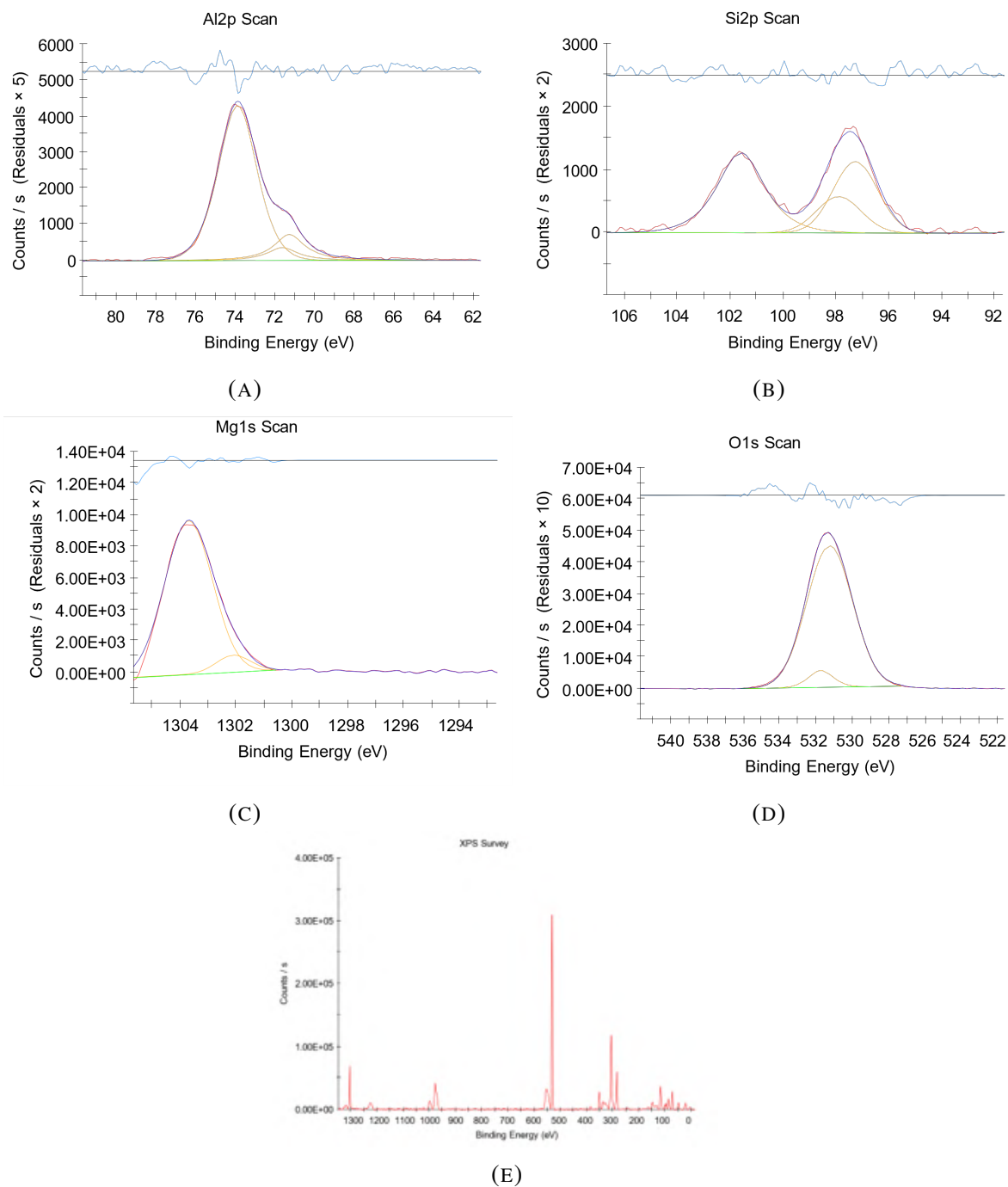


FIGURE C.24. S4 XPS Curves with fitted peaks.

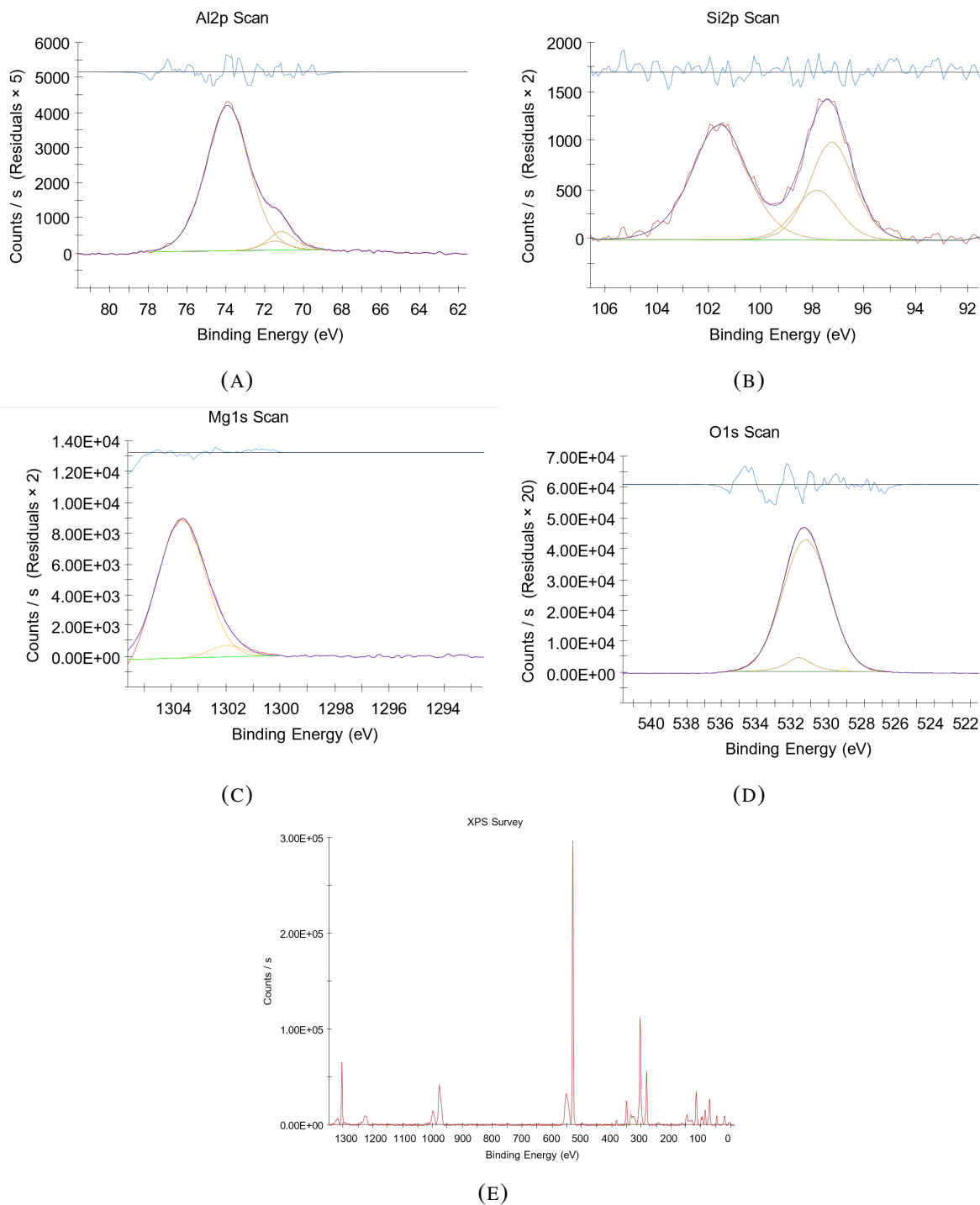


FIGURE C.25. S5 XPS Curves with fitted peaks.

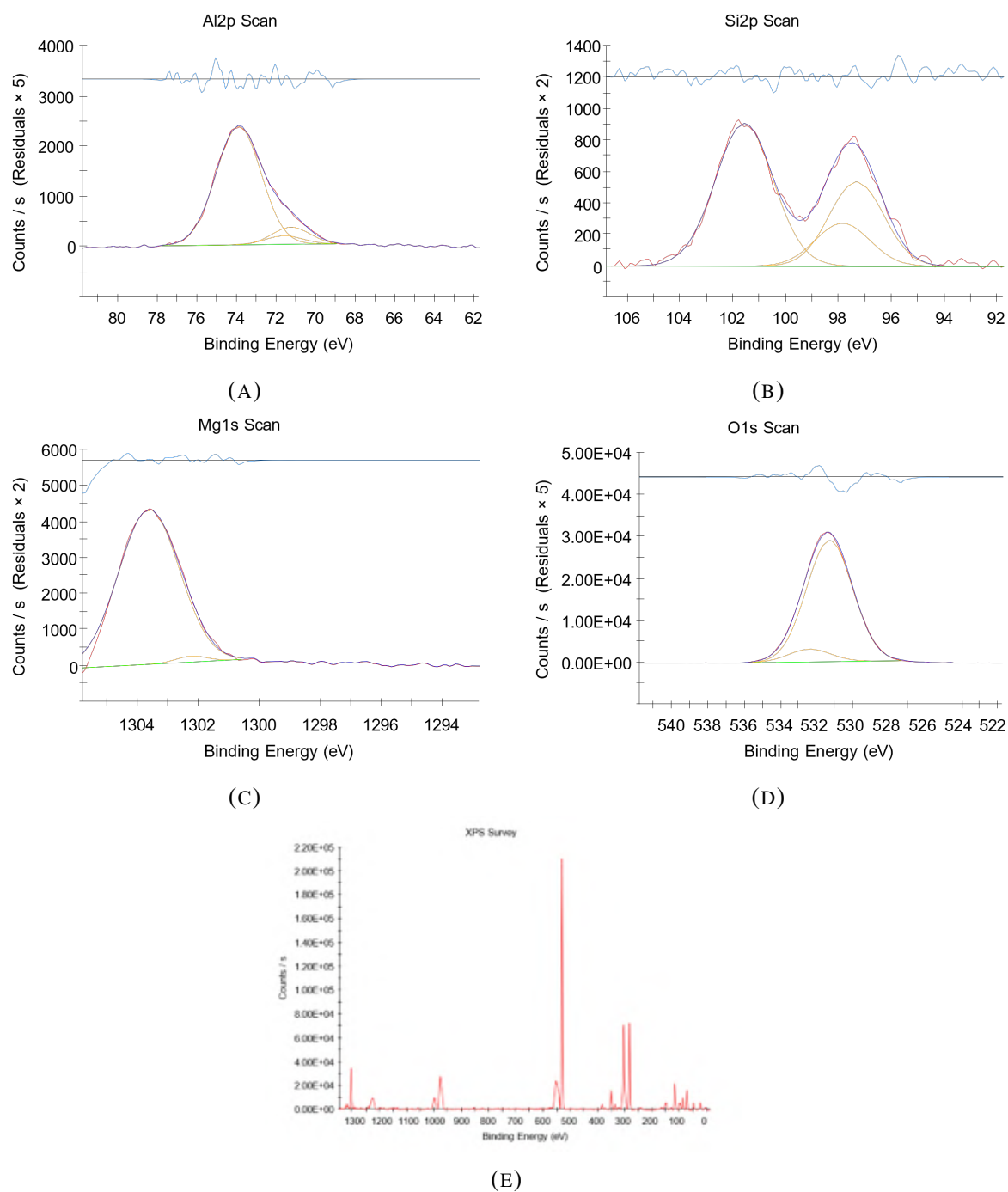


FIGURE C.26. S6 XPS Curves with fitted peaks.

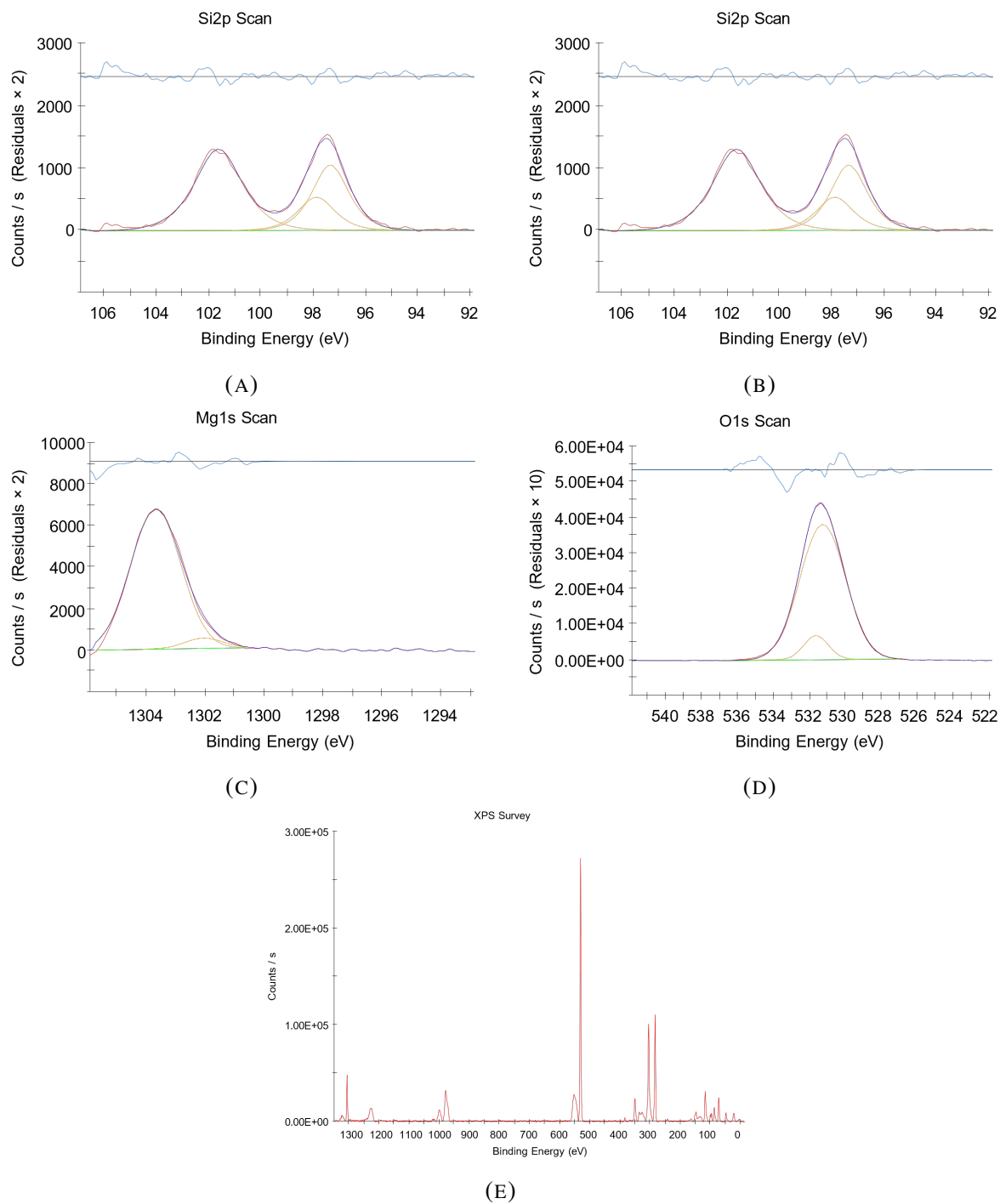


FIGURE C.27. S8 XPS Curves with fitted peaks.

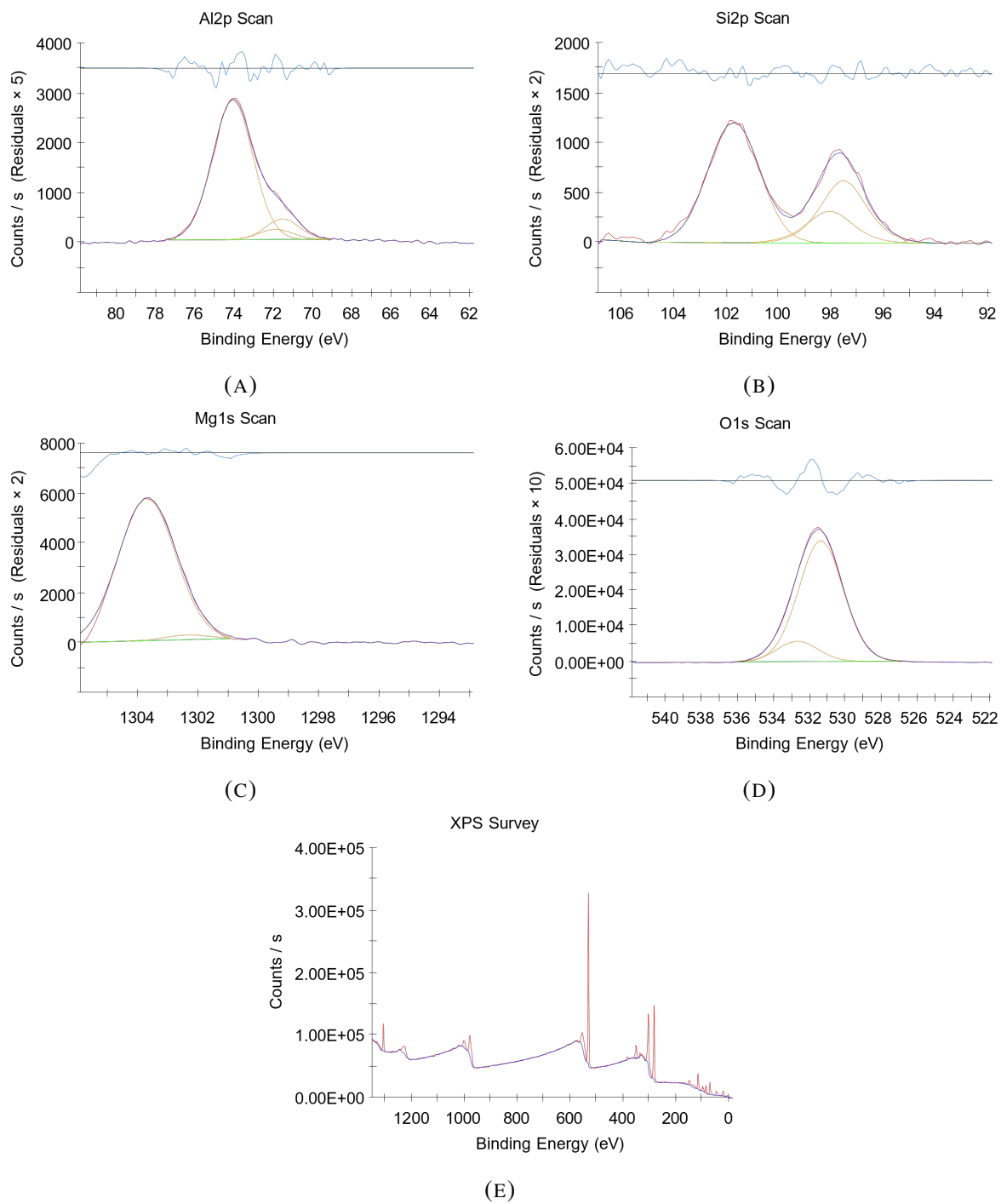


FIGURE C.28. S9 XPS Curves with fitted peaks.

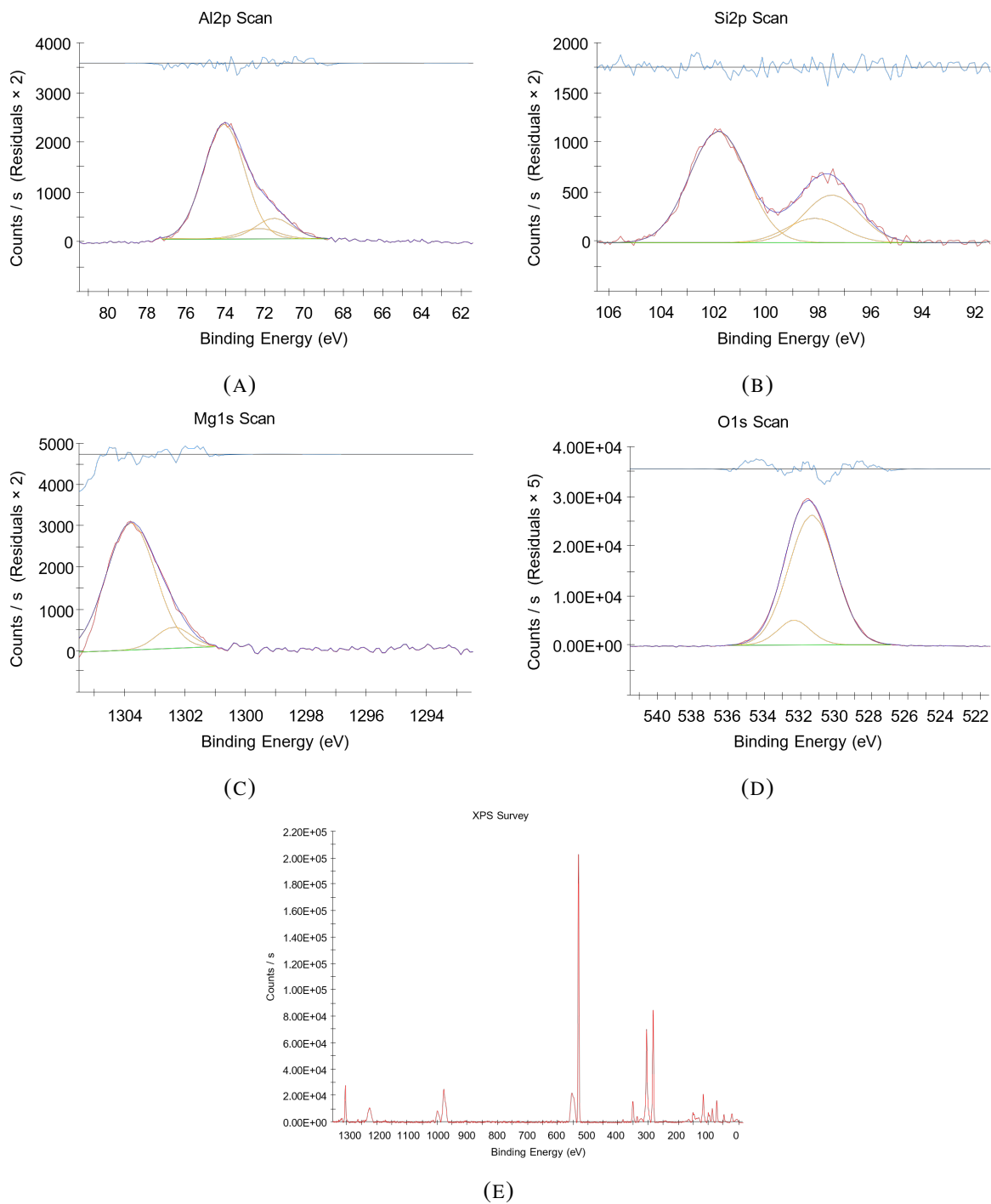


FIGURE C.29. S10 XPS Curves with fitted peaks.