



THE UNIVERSITY OF
SYDNEY

**Design of Thermomechanical Processing Routes for
Advanced High Strength Steels: Microstructure and Properties**

A thesis submitted in fulfilment of the requirements for the degree of

Doctor of Philosophy

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The University of Sydney

2023

To my beloved wife, Zahra,

And to my cherished son, Ryan,

Certificate of Originality

I hereby declare 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 also 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.

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June 30th, 2023

Attribution Statement

My thesis does not contain materials I have previously published.

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Abstract

The steel industry has shown increasing interest in developing high-strength steels to meet the automotive industry's weight reduction goals for vehicle safety and emissions reduction targets. Medium manganese (Mn) steels (MMnS) are promising candidates for third-generation advanced high-strength steels (AHSS) due to their superior mechanical properties and potentially lower cost compared to other AHSS. Different heat treatment processes have been developed to tailor the microstructure and mechanical properties of the MMn-AHSS. This study introduces a novel two-stage thermomechanical process (TSTMP) with different cold rolling directions on a range of the MMn-AHSS. Specifically, our TSTMP introduces a second stage cold roll and intercritical anneal (IA). The research compares the effect of the rolling direction, initial dislocation density, initial martensitic microstructure, and alloying with aluminium (Al). The base steel for the study was Fe-0.068C-11.3Mn (wt.%) (FeCMn) and the Al-bearing grade was Fe-0.068C-11.3Mn-1.1Al (wt.%) (FeCMnAl).

Thermodynamic calculations and experiments were used to determine the transformation temperatures of the steels. The evolution of the microstructure was characterised using scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray diffraction (XRD), and atom probe tomography (APT). The mechanical properties were evaluated by conducting hardness and tensile tests.

The calculations and experimental results revealed that Al increases the austenite start (A_s) and austenite finish (A_f) temperatures, and decreases the martensite start temperature (M_s). The average grain size of both the ferrite (α) / martensite (α') and austenite (γ) phases, however, remained unchanged. In addition, the FeCMnAl steel exhibited a higher strain distribution in both the γ and α/α' phases.

Increasing the temperature and time of the IA process during the second stage of the TSTMP resulted in an increase in the volume fraction of the retained γ phase. It also resulted

in a larger grain size, and a lower percentage of the low-angle grain boundaries (LAGB%) in the γ and α/α' phases.

Raising the dislocation density of the initial martensitic microstructure during the single stage thermomechanical process (SSTMP) via cold rolling of as-received FeCMn steel prior to IA led to an 8% increase in the retained γ at room temperature. It is proposed that this increase in initial dislocation density created more γ nucleation sites, resulting in finer γ grains with greater room temperature stability.

The FeCMnAl specimen with an initial deformed martensite microstructure after the IA showed the higher mechanical properties, larger grain sizes, same γ volume fraction, and lower strain distributions than the specimen with the initial quenched and deformed martensite microstructure after the IA.

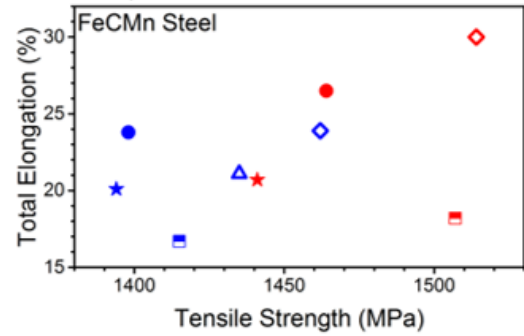
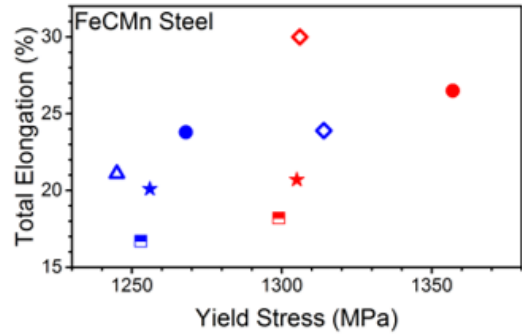
The mechanical properties are summarized in the compilation charts provided below. It is evident that there is significant potential to tailor the thermomechanical process to enhance tensile mechanical properties while achieving the desired balance between strength and ductility. This tailoring is of interest to designers in the auto industry and across other industry sectors.

All the FeCMn samples were annealed at 620 °C for 10 minutes.

■ one-step IA - RD sample
■ one-step IA - TD sample

★ TSTMP, Rolled RD - RD sample
★ TSTMP, Rolled RD - TD sample
● TSTMP, Rolled TD - RD sample
● TSTMP, Rolled TD - TD sample

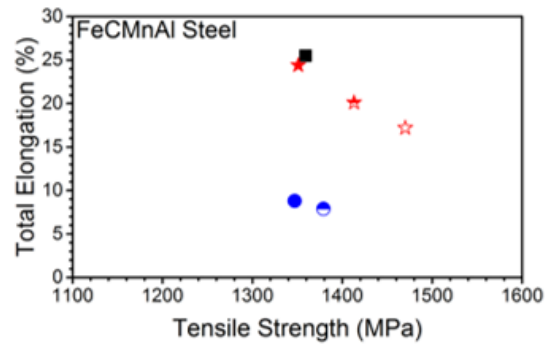
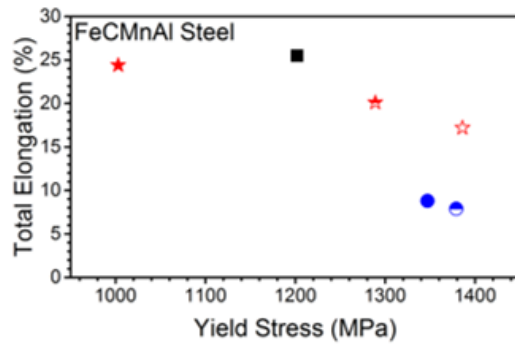
△ SSTMP, Rolled RD - RD sample
◇ SSTMP, Rolled TD - RD sample
◇ SSTMP, Rolled TD - TD sample



FeCMnAl specimens
■ one-step IA

○ TSTMP, 620 °C, 5 min
● TSTMP, 620 °C, 10 min

☆ TSTMP, 650 °C, 3 min
★ TSTMP, 650 °C, 5 min
★ TSTMP, 650 °C, 10 min



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List of Acronyms and Abbreviations

A_f	Complete austenite transformation temperature or A_3
A_s	Austenite nucleation/start temperature or A_1
AHSS	Advanced high strength steel
Al	Aluminium
APT	Atom probe tomography
Ar	Argon
AR	As-received
ART	Austenite reverted transformation
Aust. SS	Austenitic stainless steel
BCC	Body-centred cubic, α and/or α'
BEV	Battery electric vehicle
BH	Bake-hardenable steel
C	Carbon
CMn	carbon-manganese steel
CP	Complex phase steel
DP	Dual phase steel
DSA	Dynamic strain aging
DSBR	Dynamic slip band refinement
DSC	Differential scanning calorimetry
EBSD	Electron back scattered diffraction
EDM	Electrical discharge machining
EDXS	Energy-dispersive X-ray spectroscopy
FCC	Face-centered cubic, austenite, γ
FeCMn	Fe-0.68C-11.3Mn (wt. %) steel
FeCMnAl	Fe-0.68-11.3Mn-1.1Al (wt. %) steel
FQ	Fully quenching
G1	First generation

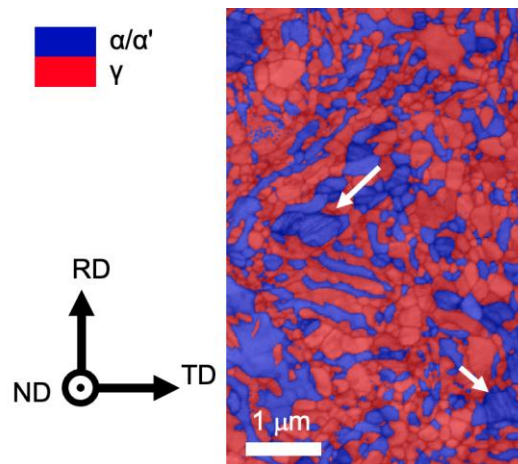
G2	Second generation
G3	Third generation
GND	Geometrically necessary dislocation
HAGB	High angle grain boundary
HCP	Hexagonal close-packed
HDF	Hydrogen-delayed fractures
HF	hot-formed steel
HS-IF	High-strength interstitial-free steel
HSLA	High-strength low alloy
HSS	High strength steel
IA	Intercritical annealing
IF	Interstitial free steel
IPF-Z	Inverse pole figure map in the Z direction
IQ	Interrupt quenching
IQ-map	Image quality map
IS	Isotropic steel
KAM	Kernel average misorientation
KS	Kurdjumov-Sachs
KS OR	Kurdjumov-Sachs orientation
L-IP	Low-weight with induced plasticity steel
LAGB	Low-angle grain boundary
LEAP	Local electrode atom probes
LSS	Low-strength steel
MART	Martensitic steel
MBIP	Micro band induced plasticity
M_f	Martensite finish temperature
min	Minute, Minutes
MMn	Medium manganese
MMnS	Medium manganese steel
M_s	Martensite start temperature
Mn	Manganese
Mo	Molybdenum
ND	Normal direction

NW	Nishiyama-Wassermann
ODF	Orientation distribution function
OM	Optical microscopy
one-step IA	Intercritical annealing of the as-received FeCMn steel at 620 °C or as-received FeCMnAl steel at 650 °C for 10 minutes
P	Phosphorus
PECS	Precision etching coating system
PIPS	Precision ion polishing system
PLC	Portevin-Le Chatelier
PSE	Product of strength and elongation
Q&P	Quenching and partitioning
RD	Rolling direction
Rolled RD	one-step IA sample which was cold rolled in rolling direction
Rolled TD	one-step IA sample which was cold rolled in transverse direction
ROI	Region of interest
Sec	Seconds
SEM	Scanning electron microscopy
Si	Silicon
SIP	Shear band-induced plasticity
SSTMP	Single-stage thermomechanical process
T_{θ}	Cementite precipitation start temperature
TD	Transverse direction
TEM	Transmission electron microscopy
TKD	Transmission Kikuchi diffraction
TMP	Thermomechanical process
TRIP	Transformation induced plasticity
TSTMP	Two-stage thermomechanical process
TWIP	Twinning-induced plasticity
UFG	Ultra fine-grained
UTS	Ultimate tensile strength
WPHT	Without previous heat-treatment
XRD	X-Ray diffraction
YS	Yield strength

α Ferrite with body-centred cubic (BCC) unit cell structure

α' Martensite with body-centred tetragonal (BCT) unit cell structure

α/α' α and α' phases are difficult to discern in SEM-TKD and related techniques because of the similarity of their lattice parameter. Therefore, in this thesis, a nomenclature protocol was adopted that is consistent with that used in the open literature when denoting the occurrence of these phases. In the figure provided below, illustrating the phase map, the areas denoting α/α' are in blue. White arrows have been placed to indicate the locations where some α' grains have formed, while the remaining blue areas represent α -phase grains. Hence, throughout this thesis α' grains marked to separate and distinguish α and α' phases.



γ Austenite with face-centered cubic (FCC) unit cell structure

ε Martensite with hexagonal close-packed (HCP) unit cell structure

PART I

BACKGROUND,

AIMS

&

METHODS

CHAPTER 1 Introduction

Concerns over climate change and air quality are driving the development of new materials that reduce greenhouse gas and other emissions. This is true across many heavy industry sections. For example, the increasing use of high-strength steels in the automotive industry offers advantages, including improved fuel economy and reduced emissions, greater safety, and affordable costs of the vehicles. These advantages stem primarily from the high strength of the steels, introducing design flexibility so that thinner sections can be employed, significantly reducing vehicle weight.

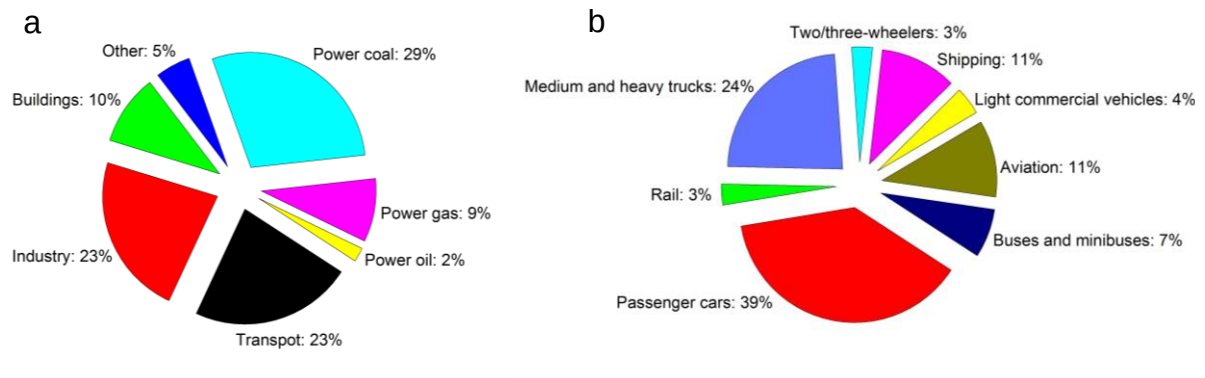


Figure 1.1. Global energy-related CO₂ emission in 2022 (a) by sector and (b) in the transport sector. Adapted from [1, 2].

CO₂ is one of the main greenhouse gases contributing to global warming, and the transportation sector contributes significantly to this problem. As Figure 1.1 indicates approximately 10% of all energy-related CO₂ emissions are attributed to passenger cars alone. Improving vehicle fuel efficiency is one of the most effective methods of reducing greenhouse gas emissions. Steels with high strength and advanced high strength are suitable for these applications. Approximately 3.5 g/km of CO₂ can be reduced with each 100 kg reduction in the weight of a vehicle [3].

Figure 1.2 illustrates the distribution of the vehicle mass among the different vehicle systems. It is evident that about 44% of the vehicle's weight is comprised of the body-in-white, chassis and suspension, which include the formed sheet metal components. In order to reduce the weight of the vehicles, high-strength low alloy (HSLA) and AHSS are used in these systems.

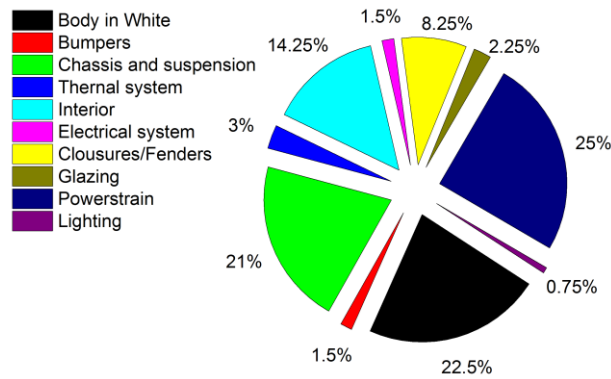


Figure 1.2. Mass distribution in vehicles. Adapted from [4].

It is possible to design lighter and more efficient vehicles with high-strength steels that have both high strength and high ductility. High-strength steels can reduce vehicle weight by up to 25% when compared to traditional steels [5]. This reduction can reduce the cost by 14% [6]. As a result, fuel efficiency can be significantly improved, and CO₂ emissions can be significantly reduced.

In the automotive industry, safety is also a top priority, and high-strength steels demonstrated superior crash test performance over steels [7, 8]. When a crash occurs, high-strength steels are capable of absorbing a greater amount of the impact energy. As a result, passengers are less likely to suffer injuries and lives are saved. The use of high-strength steels in critical areas of the vehicle, such as the A-pillars and the roof significantly enhances vehicle safety.

A lightweight vehicle is also easier to handle and manoeuvre, which can enhance the driving experience and further reduce the risk of an accident. In addition, lightweight vehicles are less likely to damage the road surface, thereby reducing maintenance costs and prolonging the life of the road network.

Figure 1.3 represents the results of what still remains a relatively new design for the body of a battery electric vehicle based on automotive industry standards. Compared to a benchmark

internal combustion engine drives vehicle, this design achieves a mass reduction of 35% in the body structure. In addition, it reduces total lifetime CO₂ emissions by approximately 70%. Dual-phase (DP), transformation induced plasticity (TRIP), twinning-induced plasticity (TWIP), complex phase (CP), and hot-formed (HF) steels comprise the materials portfolio, which achieve giga-Pascal strength levels [9].

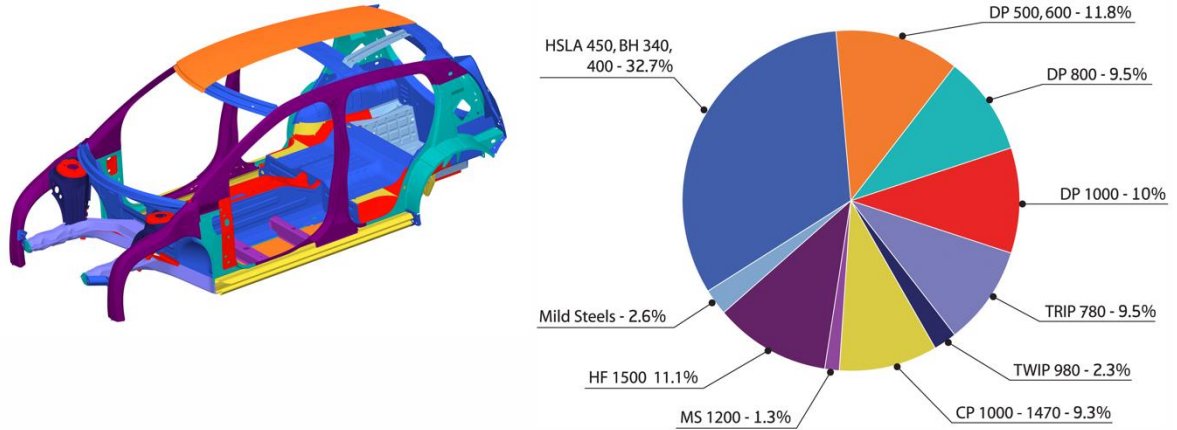


Figure 1.3. The body structure of a battery electric vehicle (BEV). The colours show the type of steels. Taken from [9].

The main objective of this study was to investigate how new thermomechanical processes (TMPs) can impact the properties of two MMn-AHSSs. This thesis is divided into two parts. Part I includes chapters 1-4 and sets the scene for the research with an introduction, literature review, statement of the aims and details of the materials studied and the methods applied. Part II of the thesis includes chapters 5-12 and contains the original new results, discussion, conclusion and suggestion for further work.

This research demonstrates the potential for introducing customisations into the TMP to achieve enhancements in materials properties that enable higher degree of sustainability in our heavy industries.

CHAPTER 2 Literature Survey

2.1 Steels in Automotive Industry

Karl Benz built a combustion engine-powered automobile in 1885 [10]. The early automobiles consisted almost entirely of the wooden structures which gradually gave way to steel and Al sheet metal during the late 19th and early 20th centuries. For the next 100 years, steel dominated the design of the automotive bodies. Mild steel or low carbon (C) steel is the primary steel grade used in these first automobiles. The automotive industry began examining higher-strength steels in the early 1970s, primarily HSLA, in an effort to reduce the mass of their vehicles and optimize fuel efficiency [11].

In the automotive industry, there are approximately 30 steel grades, which are classified by their metallurgical (composition, processing, and microstructure), strength, and formability properties into three categories. The metallurgical properties classification for steels includes low-strength, mild steels, isotropic steels (IS), interstitial free steels (IF), conventional high-strength steels (HSS), bake-hardenable steels (BH), carbon-manganese steels (CMn), HSLA and high-strength interstitial-free steels (HS-IF). and two groups of the AHSSs commonly classified by microstructure at ambient temperature. It is possible to divide advanced high-strength steels into two groups based on this classification. The first group includes TRIP steels, DP steels, CP steels, martensitic steels (MART) and HF steels. The second group includes post-forming heat-treated steels, low-weight with Induced Plasticity steels (L-IP), TWIP steels and austenitic stainless steels (Aust. SS) [11].

Steels can also be classified according to their strength. The following types of the steels are classified in the automotive industry based on this classification: conventional or low-strength steels, HSS and AHSS.

The yield and tensile strengths of the low-strength steels (LSSs) are less than 210 MPa and 270 MPa, respectively. An HSS is defined as having yield and tensile strengths varying

between 210 and 550 MPa and 270 and 700 MPa, respectively. An AHSS has a yield strength greater than 550 MPa and a tensile strength greater than 700 MPa. Figure 2.1 illustrates the schematic of the different types and generations of the steel used in the automotive industry.

The third type of the steel classification is based on mechanical properties such as tensile strength and total elongation. This categorizes steels in the automotive industry into four different groups:

1- Low-carbon and conventional types of the HSSs: It can be seen from Figure 2.1 that this group includes Mild, IF, IS, CMn, BH, HS-IF, and HSLA [6, 12] steels with a single-phase microstructure. As the strength of the steel increases, the ductility of this type of the steel decreases almost linearly [12, 13]. When there is a need to reduce the thickness of the components to reduce the weight of the vehicle, the components do not comply with the safety requirements.

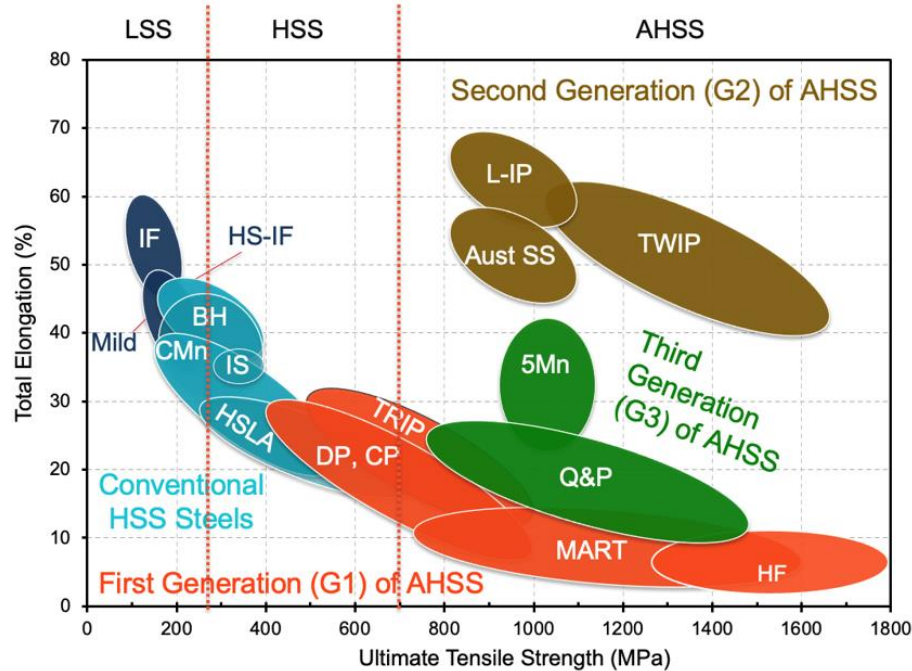


Figure 2.1. The schematic of the different types and generations of the steel used in the automotive industry. Adapted from [14].

2- Advanced high-strength steels – first generation (AHSS – G1): There are several different types of the steels in this type of the category, including DP [13, 15], MART [16, 17], CP [6, 18–20], TRIP [6, 19–21] and HF [22] steels. There are a number of phases in the microstructure of these steels, including ferrite, austenite, bainite and martensite (α'). The tensile strength, ductility and fracture toughness of these steels are below the requirements for effective weight reduction in vehicles [23].

3- Advanced high-strength steels – second generation (AHSS – G2): This group consists of the TWIP steels [24–44], Aust. SS steels [45–47] and L-IP steels [31, 43, 48] with γ phase microstructure at room temperature and large amounts of the γ stabilizer alloying elements, such as Mn, Ni and Cr. Steels of this group are known for their high strength, and high ductility, which makes them very desirable for use in the automotive industry. It is however prohibitively expensive to produce these steels due to the presence of the large amounts of the alloying elements, as well as difficult to process them due to the presence of the large amounts of the γ phase [23]. Furthermore, these steels have shown a poor weldability [49, 50] and are susceptible to hydrogen-delayed fractures (HDF) [26, 28].

4- Advanced high-strength steels – third generation (AHSS – G3): There are MMnSs (3–12 wt.% Mn) [51–53] and TRIP-assisted quenched and partitioned steels (Q&P) [54–56] included in this category. AHSS-G1 has limitations due to insufficient tensile and ductility and AHSS-G2 has limitations due to a large amount of the alloying elements and processing difficulties associated with them. Therefore, AHSS-G3 was designed in an attempt to overcome these limitations by adopting a new approach. An illustration of the microstructure of the different types of the HSS and AHSS can be found in Figure 2.2.

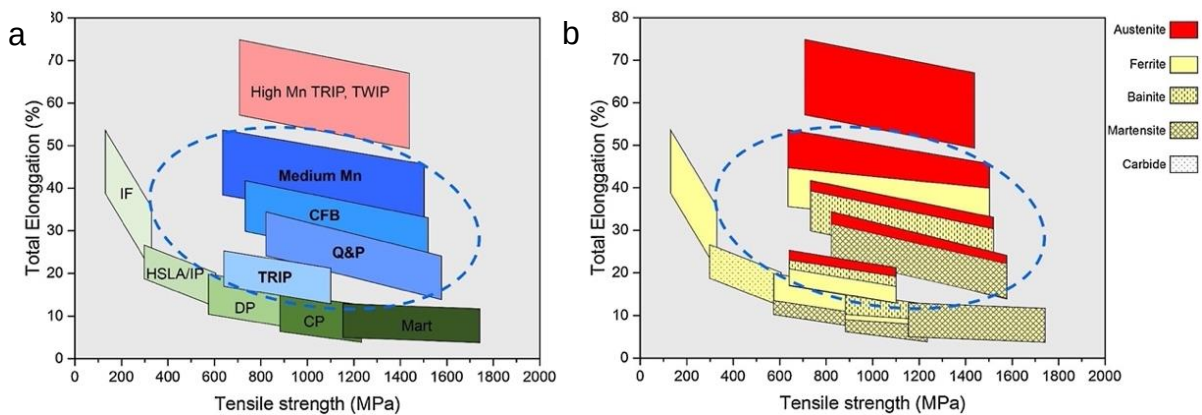


Figure 2.2. The schematic of the different types of the steels in the automotive industry according to (a) the mechanical properties and (b) the corresponding microstructure. Taken from [57].

The first MMnS with ultrafine-grained duplex microstructure was developed by Miller in the 1970s. It has been demonstrated that ultrafine-grained microstructure has a significant effect on the strength of the MMnSs, however, this microstructure exhibits a localised deformation behaviour because strain hardening is absent [58]. Typically, MMnSs contain between 3 to 12 wt.% Mn [59–68], 0.05 to 0.4 wt.% C [59–67], 0 to 8.0 wt.% Al [34, 59, 68–70], and 1.2 to 2.0 wt.% silicon (Si) [63, 67–69].

Several studies have examined the deformation mechanisms [67, 69, 71, 72] and examined the effect of the heat treatment parameters such as annealing temperature [65, 67, 73, 74], heating rate [75] and annealing time [76–78] on the microstructure and mechanical properties. Some of the characteristics of the MMn-AHSSs can be found in Table 2.1.

Table 2.1. Characteristics of the MMn-AHSSs. Taken from [68].

Mn, wt.%	3-7	6-12
heat treatment	Q&P	IA
microstructure after cold rolling	deformed α'	
grain	ultrafine-grained	
microstructure after annealing	γ and α	
deformation mechanism	TRIP	TWIP+TRIP
SFE of γ , mJ/m ²	<10	>20
γ stability	γ composition size	
Mn effect	γ stability	
	SFE	
	hardenability	

2.2 Reverse Austenite Formation (Martensite into Austenite Transformation) during the Intercritical Annealing

The process of forming γ is generally believed to be a combination of the nucleation and growth controlled by diffusion, as supported by various studies [73, 78–84]. The specific nucleation sites for γ can vary based on the initial microstructure of the alloy steel. In steels with an initial microstructure of the ferrite-pearlite, the predominant nucleation sites for γ are at the interphase boundaries between the ferrite grains and pearlite colonies [79, 83]. In steels with athermal martensite, martensite packet or lath boundaries [73, 78, 81, 84] and prior γ grain boundaries [73, 82, 84] have been reported as the nucleation sites for γ . In quenched and tempered steels that consist of the spheroidized carbides and ferrite, austenite generally nucleates at carbide particles which are located at the ferrite grain boundaries [80, 83]. As Garcia et al. [83] pointed out, the carbides in the ferrite grains are not likely to contribute to the nucleation of the γ but rather are more likely to dissolve in the ferrite matrix after nucleation, allowing C to diffuse to the growing γ .

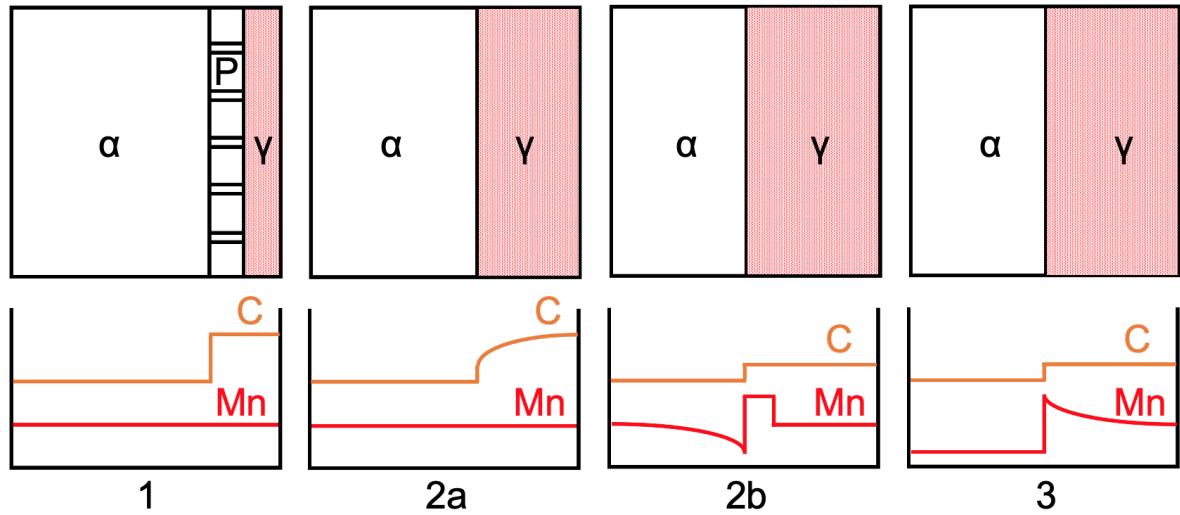


Figure 2.3. Schematic of the γ formation during the IA from a ferrite-pearlite microstructure in three stages: stage 1: pearlite dissolution; stage 2a: C diffusion in γ and γ growth; stage 2b: Mn diffusion in ferrite and γ growth; and stage 3: Mn diffusion in γ and final equilibration. Adapted from [79].

According to Speich et al. [79], and others [59, 80, 82, 85], γ formation in C-Mn steels with a ferrite-perlite microstructure is composed of three distinct steps as shown in Figure 2.3 and explained below:

- Stage 1: The process involves the nucleation of the γ at the interface between the ferrite and pearlite and the subsequent growth of the γ into pearlite until the pearlite dissolves completely. Austenite nucleates instantaneously. Lei et al [80] reported that it is 50 ms for annealing Fe-0.1C-3.4Mn at 740 °C and Speich et. al [79] reported it is 0.12 ms for annealing Fe-0.12-1.5Mn steel at 900 °C. Therefore, it is not considered a rate-limiting factor because there is effectively no practical, discernible barrier to the nucleation step. It is primarily the rate of the C diffusion within the γ between adjacent pearlitic cementite lamellae that determines the rate of the γ growth at this stage, but substitutional elements may also influence the rate of the γ growth. The first step results in the formation of a high-carbon γ . There is, however, no equilibrium between the ferrite and the high-carbon γ .

- Stage 2: The high-carbon γ grows into the ferrite upon reaching partial equilibrium with the ferrite. Diffusion of the C atoms in γ may control γ growth and make it slower (stage 2a) or diffusion of the Mn atoms in the ferrite may control the γ growth (stage 2b).

- Stage 3: At this stage, the diffusion of Mn atom in γ is responsible for the very slow final equilibration of the γ and ferrite. In γ , Mn atom diffuses at a rate three orders of the magnitude slower than in the ferrite [79].

Wei et al [81] studied Fe–0.1C–3Mn–1.5Si steel with athermal martensite microstructure during the IA at 690, 720 and 760 °C. It was found that γ growth has three stages:

- Stage 1: γ grows without partitioning. This stage is controlled by the rapid diffusion of the C atoms in the ferrite which is gradually replaced by the diffusion of the C atoms in γ .
- Stage 2: γ has a relatively slow growth rate. Mn and/or Si diffusion in ferrite controls this stage.
- Stage 3: γ has a very slow growth rate. Mn and/or Si diffusion in the γ controls this stage for the final equilibration condition [81].

There is a phase boundary whenever two crystals with different lattice structures and compositions come into contact. It has been demonstrated that the orientation of the γ and its parent α have a very close relationship [86, 87]. The $(111)\gamma \parallel (110)\alpha$ and $[1\bar{1}0]\gamma \parallel [1\bar{1}1]\alpha$ relationship is known as the Kurdjumov-Sachs (KS) orientation relationship (OR).

2.3 Heat Treatment of the Medium Manganese Steels

Various heat treatment procedures have been developed to increase the strength and ductility of the MMnS and to balance the properties. There are two main heat treatment processes in MMnS: IA and Q&P. The other heat treatment processes being designed by researchers involve repeating and combining the IA and Q&P or combining them with other heat treatment processes such as austenitization. Two of these designed heat treatment processes using the IA and Q&P are explained here.

2.3.1 Intercritical Annealing or Austenite Reverted Transformation

A simple annealing process for MMnS, the IA is also called austenite-reverted-transformation (ART). During IA, the alloy steels are annealed in the area of the FeC equilibrium phase diagram that includes both the γ and ferrite.

Shi et al. [62] announced the introduction of the ART in medium 5Mn steels in 2010. A MMnS with ultrafine-grained γ -ferrite duplex phases achieved excellent strength and ductility

through IA. The amount of the Mn element in steel during this process ranges from 5 to 12 wt.%. A schematic representation of the IA process is shown in Figure 2.4.

This process produces steel with a fully martensitic microstructure. Steel-making processes such as hot-rolling or austenitization and quenching can cause this microstructure. However, there may still be very small amounts of the retained γ in the microstructure. An enhancement of the martensitic microstructure can be achieved by cold rolling. After IA, the final microstructure depends on a number of the parameters, including the martensite condition, the annealing temperature, and the heating and cooling rates. In this process, C and Mn partition from supersaturated martensite into γ , enriching γ with C and Mn.

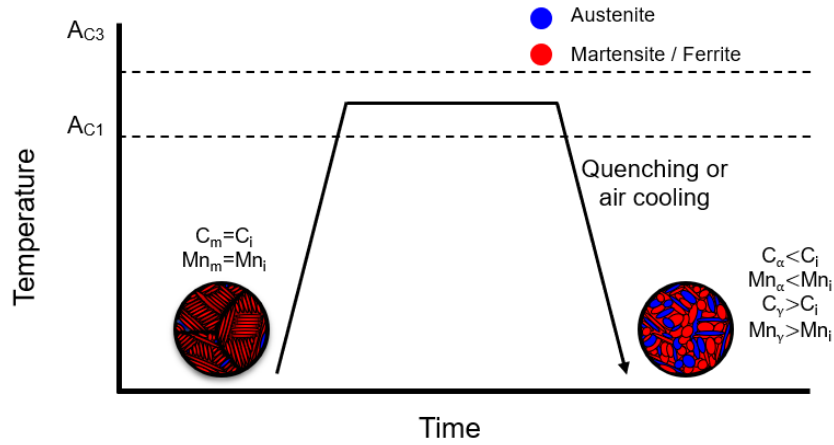


Figure 2.4. Schematic of the IA or ART process for a MMnS. C_i , C_m , C_α and C_γ are C contentment of the initial alloy, martensite, ferrite, and austenite, respectively. Mn_i , Mn_m , Mn_α and Mn_γ are the Mn content of the initial alloy, martensite, ferrite, and γ , respectively.

2.3.2 Quenching and Partitioning

Speer et al. [88] proposed the Q&P in 2003 as a method for producing martensitic steel containing more retained γ . Based on the hypothesis that some alloying elements, such as Si, Al or P, can prevent the formation of the carbides, this procedure has been developed. It has been demonstrated that C partitions from supersaturated α' into γ under para-equilibrium conditions, enriching γ with C [88–90].

As shown in Figure 2.5, Q&P consists of three steps. To achieve a microstructure mixture of partially retained γ and α' , fully γ steel is first quenched to a quenching temperature (QT) between martensite start (M_s) and finish (M_f) temperatures. The second step in the process is partitioning. By holding the steel at partitioning temperature (PT), the C and Mn in the saturated

martensite partition into the retained γ and enrich and increase the chemical stability of the γ . Quenching is the final step.

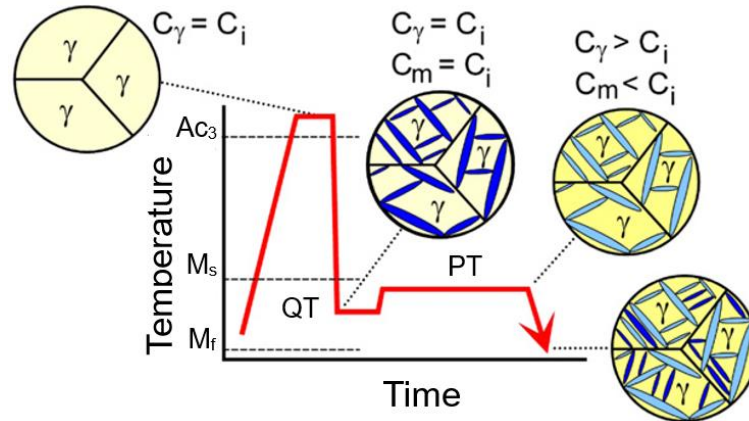


Figure 2.5. Schematic of the quenching and partition heat treatment process. Taken from [56].

2.3.3 Multi-Step Annealing

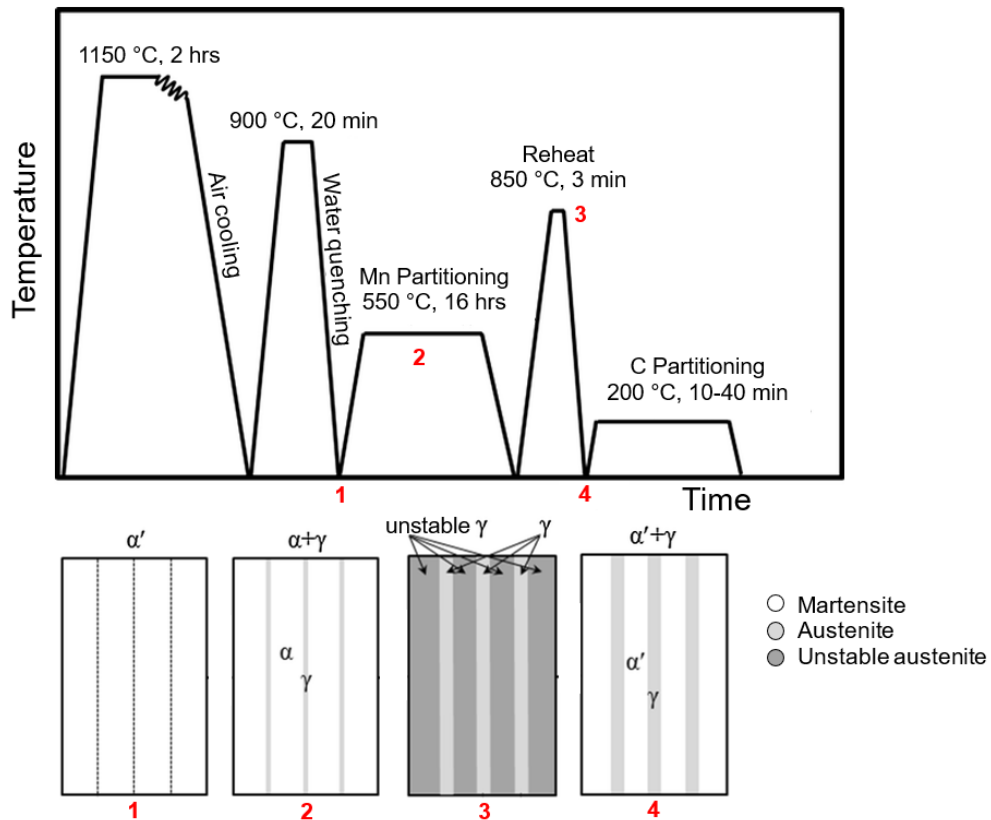


Figure 2.6. Multi-step annealing of the Fe-10.62Mn-2.84Al-0.17C-0.5Mo steel. Adapted from [91].

There have been multi-step annealing treatments applied to the MMnSs, which employ repeated α' phase transformation and γ phase reversion procedures [91, 92]. Figure 2.6 shows the four-step heat treatment of the Fe-10.62Mn-2.84Al-0.17C-0.5Mo steel used by Heo et al. [91]. To form a fully martensitic microstructure, steel was first annealed in the γ single-phase

region (austenitization), followed by quenching. Secondly, Mn was then enriched in γ by an IA process that lasted 16 hours. Following this step, a 3 minutes (min) annealing was performed to form new γ with a lower Mn content. This γ then transformed into α' when quenched. For C partitioning into γ , tempering was the last step. In comparison with one-step intercritical annealed samples, this treatment results in a substantial improvement in the strength of the MMnSs without a significant loss in ductility [92].

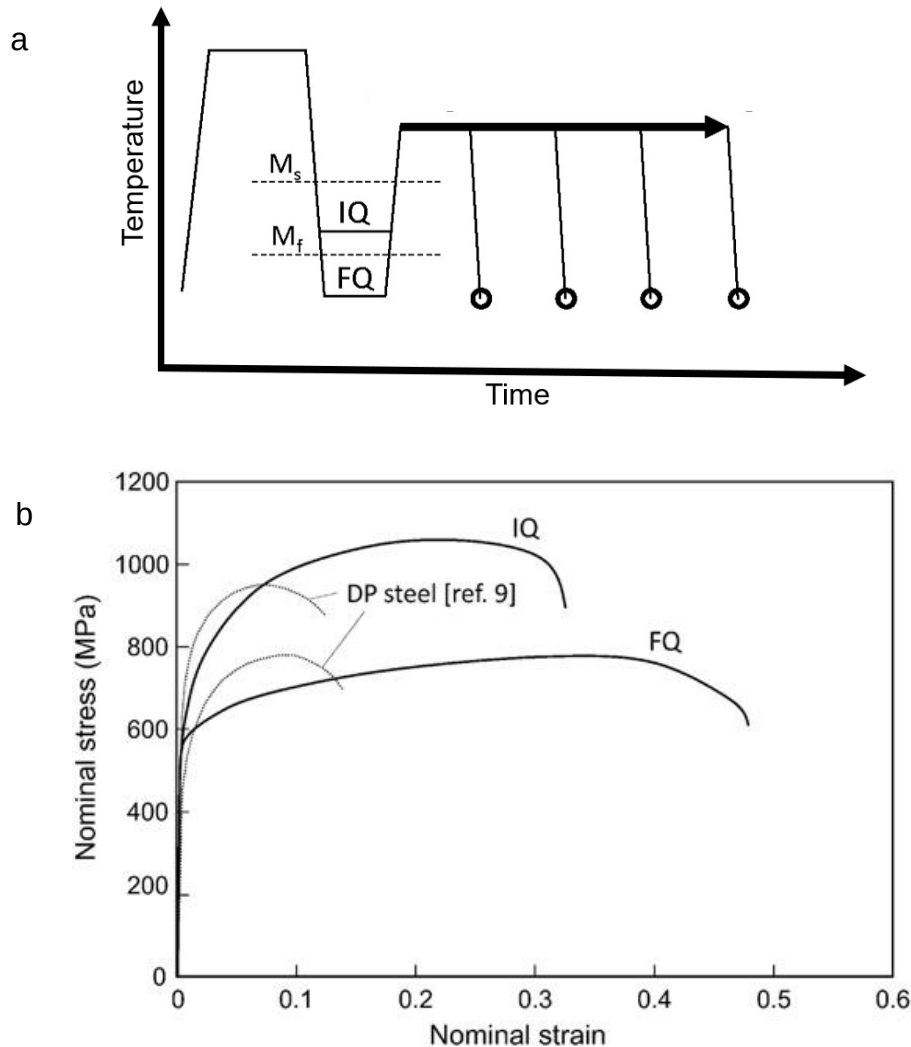


Figure 2.7. (a) Interrupted quenching and IA heat treatment process and (b) stress-strain curve of the IQ and FQ specimens. Taken from [93].

2.3.4 Interrupted Quenching and Intercritical Annealing

The interrupted quenching and the IA process was proposed by Tsuchiyama et al. [93] [2.97] for 0.1C-5Mn-1.2Si steel (Figure 2.7). The first step of the process involves austenitization heat treatment, followed by quenching to a temperature between the M_s and the M_f (interrupted quenching temperature, IQ). There is 20% unstable untransformed γ at this temperature, containing low Mn levels. Following this, an IA and a quenching process are

conducted. The Mn content of this untransformed γ remains low during the IA process due to the low diffusivity of the Mn atom. In addition, as new γ grains form during IA, they are enriched in Mn. During final quenching, untransformed γ from IQ transforms into fresh α' . By comparing the IQ process with the normal intercritical process (fully quenched, FQ), it can be seen that both processes result in almost the same volume fraction of the γ in the microstructure, however, the IQ process has a 20% higher tensile strength and a 40% reduction in elongation.

2.4 Microstructure and Mechanical Properties

A nucleation and diffusion-controlled growth process is generally regarded as responsible for the γ reversion during the IA in MMnSs [73, 78, 94–96]. Austenite nucleates along the primary γ boundaries and the martensite lath, block or packet boundaries [97], leading to the grain refinement [68]. Microstructural characteristics of the MMnSs are ultrafine-grained (UFG) and duplex. Microstructure contains α/α' and γ [60, 77, 98]. There are several parameters that can influence the microstructure of the MMnSs, which will be discussed here.

2.4.1 Effect Initial Martensite Condition

The initial martensite condition affects the final microstructure of the MMnSs after IA. Figure 2.8 shows the fully martensite MMnS before IA.

Initial martensite microstructure could be athermal α' -martensite. This is the result of the hot rolling or austenitization heat treatment followed by quenching. When athermal martensite microstructure is intercritically annealed, the result is an elongated or lath-shaped duplex microstructure [93, 99–103]. The initial deformed α' -martensite is the result of a previous cold rolling process. When an aa deformed martensitic microstructure is intercritically annealed, the grain shape is equiaxed or globular [72, 101–104]. Figure 2.9 shows the effect of the initial martensite conditions on the final duplex microstructures and their mechanical properties.

Austenitization and quenching results in athermal martensite. Han et al [75] examined athermal martensite and following reverse γ under various austenitization temperatures. [75]. Figure 2.10 illustrates their findings. As the austenitization temperature increases it results in:

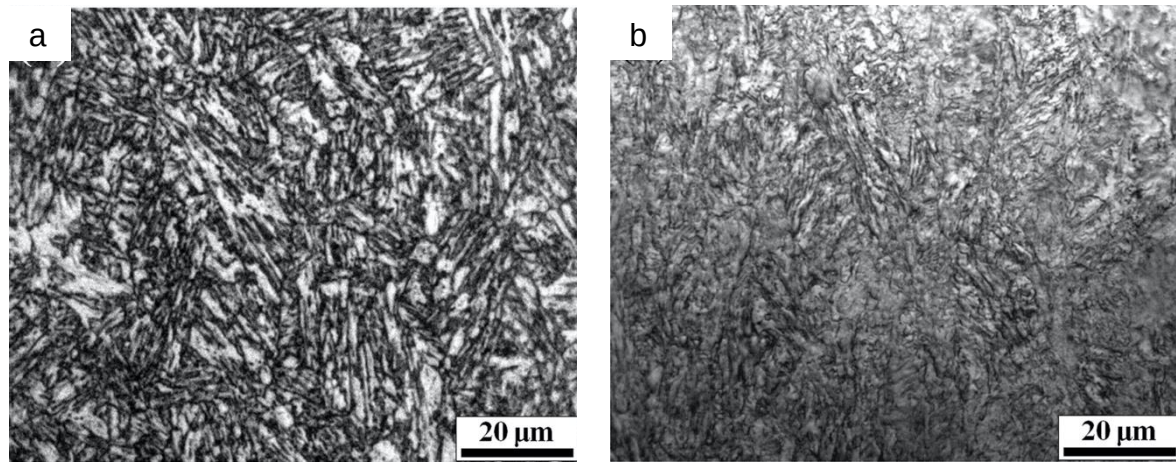


Figure 2.8. Optical microscopy (OM) micrographs of the Fe-9Mn-0.05C steel (a) the athermal α' -martensite for the hot-rolled specimen and (b) the deformed α' -martensite for the cold-rolled specimen. Taken from [64].

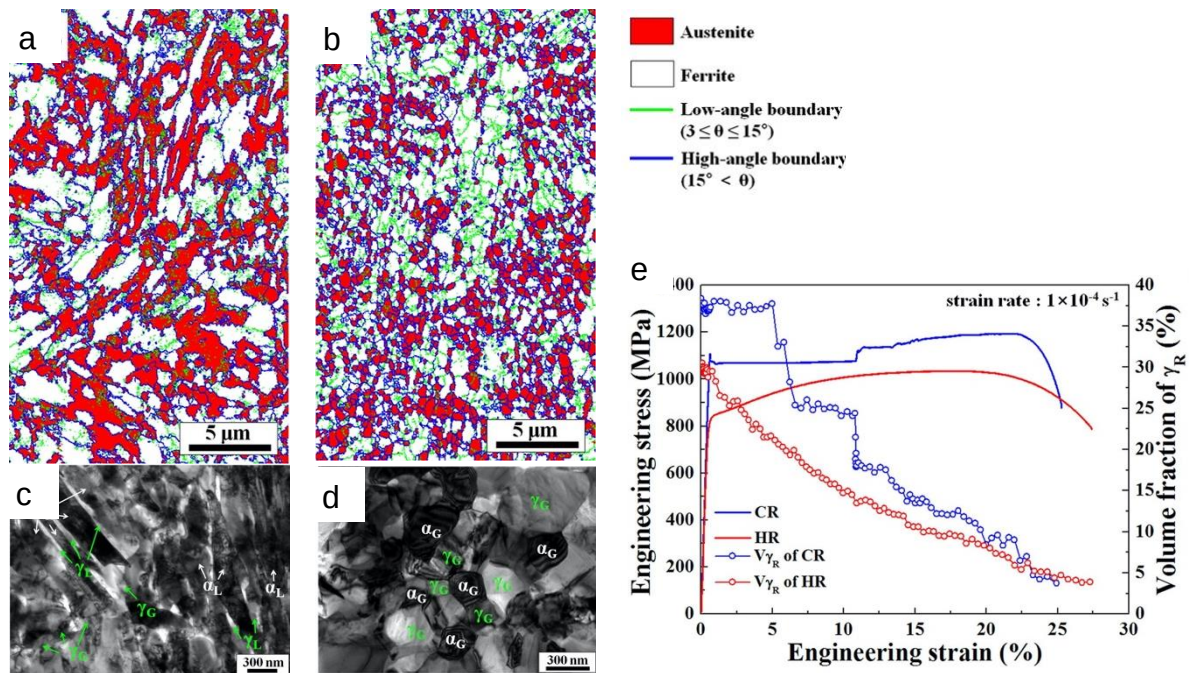


Figure 2.9. Effect initial hot-rolled and cold-rolled martensite condition of the Fe-9Mn-0.05C steel on the microstructure and mechanical properties after the IA for 10 min at 620 °C and 600 °C, respectively. Electron back scattered diffraction (EBSD) phase maps of (a) hot-rolled and (b) cold-rolled specimens after annealing. Bright-field TEM micrographs of the (c) hot-rolled and (d) cold-rolled specimens after annealing. L and G stand for lath-shape and globular-shape grains, respectively. (e) Engineering stress-strain tensile tests for annealed hot-rolled and cold-rolled specimens and variations in the volume fraction of the retained γ during the test. Taken from [64].

- coarsening of the prior γ and an increase in martensite's average block width, packet size, and lath width and as a result reduction of the martensite boundaries,

- reduction of the retained γ volume fraction,
- reduction of the reverse γ nucleation sites which are martensite boundaries,
- reduction in the reverse γ transformation rate,
- reduction of the tensile strength and,
- increase of the total elongation [98].

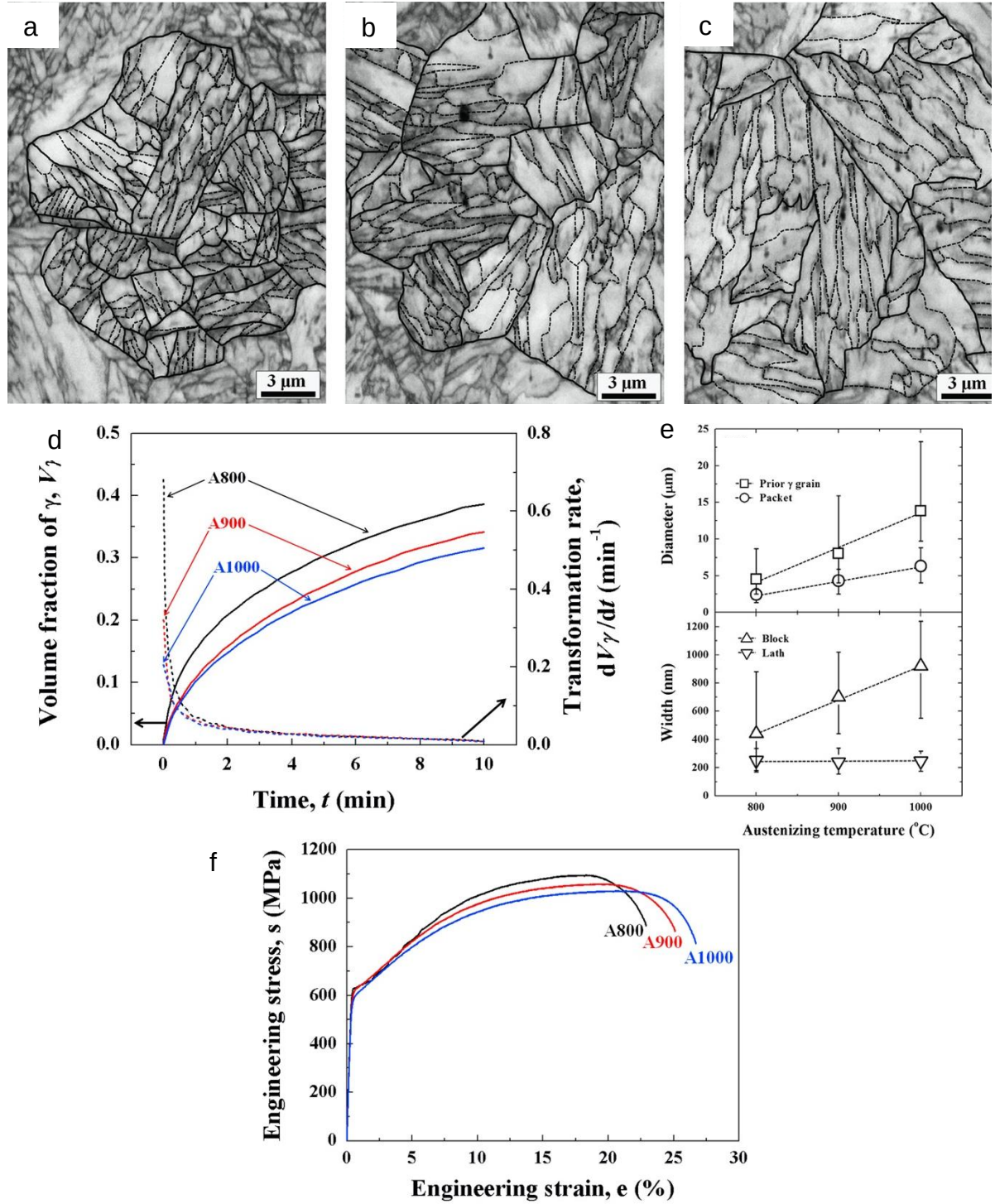


Figure 2.10. EBSD image quality maps of the athermal α' -martensite microstructure after the austenizing Fe-9Mn-0.05C steel at (a) 800 °C, (b) 900 °C and (c) 1000 °C. Solid and dashed lines are packet boundaries and block boundaries, respectively. Effect of the austenitization temperature on (d) reverse γ volume fraction and transformation rate with annealing time, (e) martensite size and width and (f) tensile test results. Taken [98].

2.4.1 Effect of the Intercritical Annealing Temperature

Researchers have investigated the effect of the IA temperatures on MMnS microstructures [67, 74, 95, 105–108]. Using both experimental and mathematical analysis, Gibbs et al. [105] demonstrated that as the annealing temperature increases, the volume fraction

of the retained γ increases and then decreases (Figure 2.11(a)). As the annealing temperature increases, the volume fraction, and the grain size of the γ increase, resulting in a decrease in Mn content in the γ phase [74]. This is shown in Figure 2.11(b) to (d). In light of these factors, reverse γ has a lower stability at room temperature. As a result, reverse γ partially transforms to martensite during quenching, decreasing the volume fraction of the retained γ .

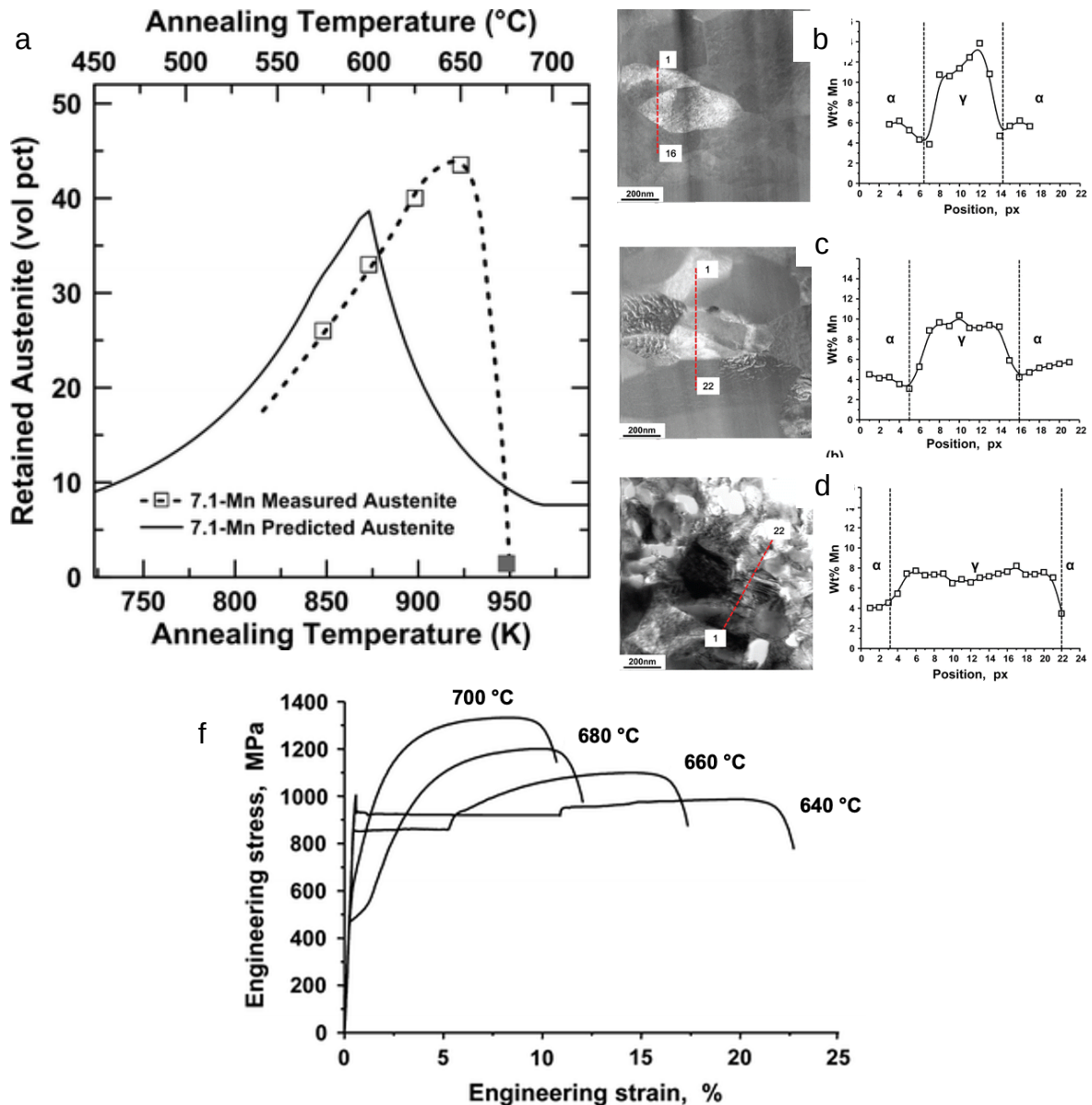


Figure 2.11. Effect annealing temperature on retained γ . (a) experimental (dash line) and calculation (solid line) results of the annealing temperature effect on the volume fraction of the γ for Fe-0.1C-7.1-Mn0.12-Si steel. Taken from [105]. Effect of the annealing at (b) 640 °C, (c) 660 °C and (d) 680 °C for 3 min on the Mn content of the γ and ferrite for Fe-0.05C-6.15 Mn-1.5Si steel. Taken from [74]. (f) Effect of the annealing temperature on tensile test results. Sample annealed for 3 min. Taken from [74].

Figure 2.11(f) illustrates the effects of the IA temperature on the mechanical properties of the Fe-0.05C-1.5Mn steel. As can be seen, annealing temperature had a significant impact on mechanical properties. An increase in annealing temperature reduced the total elongation

and increased the tensile strength. Further, it demonstrates that annealing at lower temperatures results in a yield point phenomenon and Lüders-type deformation bands [74].

2.4.2 Effect of the Intercritical Annealing Time

It has been shown that annealing time has the same effect as the annealing temperature [78, 97, 109]. Increasing the annealing time causes the γ volume fraction to increase and reach a maximum. The volume fraction is then decreased for the same reasons explained for the annealing temperature. Figure 2.12(a) illustrates how the γ volume fraction of the Fe-0.18C-5Mn-0.4Al steel changes during the IA at 650 °C by increasing the annealing time [110]. Mohapatra et. al [111] found that the volume fraction of the retained γ and total elongation increased at first, and then decreased when the annealing time was increased in Fe-0.18-6.78Mn-3.92Al-1.34Si steel (Figure 2.12(b)).

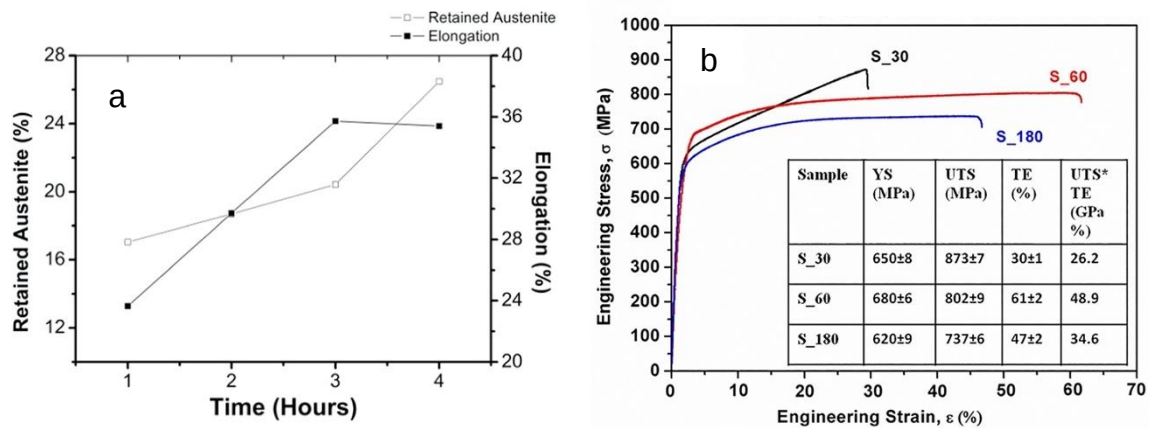


Figure 2.12. Effect of the IA time on (a) retained γ volume fraction and elongation of the Fe-0.18C-5Mn-0.4Al steel annealed at 650 °C taken from [110] and (b) tensile test results of the Fe-0.18-6.78Mn-3.92Al-1.34Si steel specimens annealed at 750 °C for 30 (S_30), 60 (S_60) and 180 (S_180) min. Taken from [111].

2.4.1 Effect of the Heating Rate

The effect of the heating rate on MMnS has been studied [75, 112–114]. As a result of the research conducted by Han and Lee [75], the effect of the heating rate on Fe-(5-9)Mn-0.05C (weight%) steel was examined. Figure 2.13 shows their findings for Fe-9Mn-0.05C steel.

In heating rates below 15 °CSec⁻¹, cementite precipitates at lower temperatures than martensite transformation into γ . In heating rates lower than 15 °CSec⁻¹, it can be observed that T_0 , A_s and A_f increase with an increase in heating rate, which indicates that the mechanism of

the cementite precipitation and martensite transformation into γ is diffusive. It has been shown that when the heating rate is above $15\text{ }^{\circ}\text{CSec}^{-1}$, cementite does not precipitate and A_s and A_f do not change significantly. According to this result, martensite reverse transformation into γ is diffusionless at high heating rates.

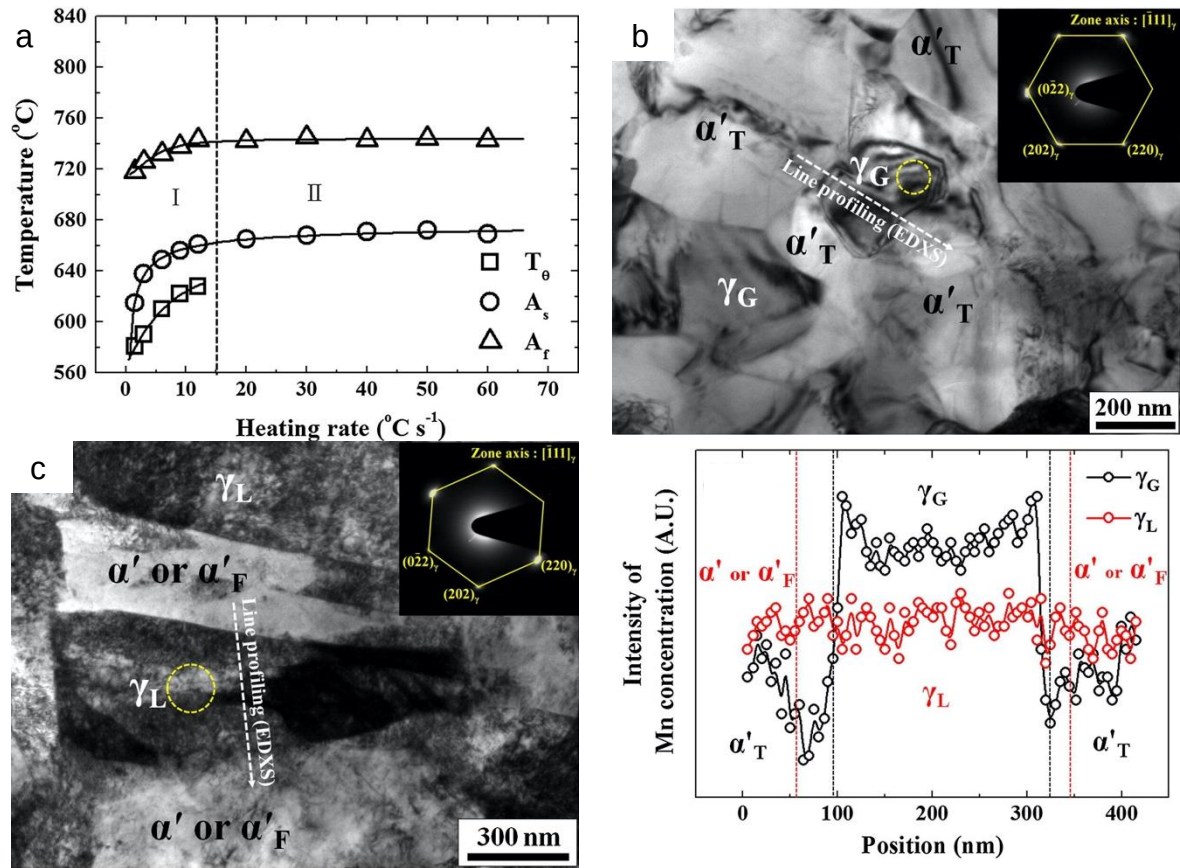


Figure 2.13. The effects of the heating rate on the reverse transformation mechanism of the Fe-9Mn-0.05C steel. (a) effect of the heating rate on T_θ , A_s and A_f which are cementite precipitation start, the reverse γ transformation start and finish temperatures, respectively. (b) and (c) TEM bright field images of the specimens were annealed at (b) 645 °C and (c) 677 °C for 10 seconds (Sec). The heating rate to reach 645 °C was $3\text{ }^{\circ}\text{CSec}^{-1}$ and the heating rate to reach 677 °C was $50\text{ }^{\circ}\text{CSec}^{-1}$. (d) An analysis of the Mn concentration along the white dashed lines in TEM images (b) and (c) using EDXS line profiling. Taken from [75].

Low heating rates result in globular reverse γ grains that have a low dislocation density and a high Mn content as a result of the diffusive transformation process. By contrast, higher heating rates result in reverse γ with lath-shaped grains, a high dislocation density and a low Mn content as a result of the diffusionless transformation.

Hu et al. [115] examined the influence of heating rate on the phase transformation temperature of martensite into γ in Fe-0.1C-5Mn (wt.%) alloy. They employed the differential scanning calorimetry (DSC) to analyse the samples. The researchers observed that as the heating rate increased, the onset, peak, and endset temperatures of the phase transformation exhibited a corresponding increase.

2.4.2 Effect of the Cooling Rate

The effect of the cooling rate on microstructure and mechanical properties of the MMnSs was studied by researchers [113, 114, 116, 117]. Hu et al. [117] found that when Fe-0.11-4.89-1.96 steel is subjected to the same IA treatment, air cooling results in a slightly higher retained γ volume fraction and a lower M_s than water quenching.

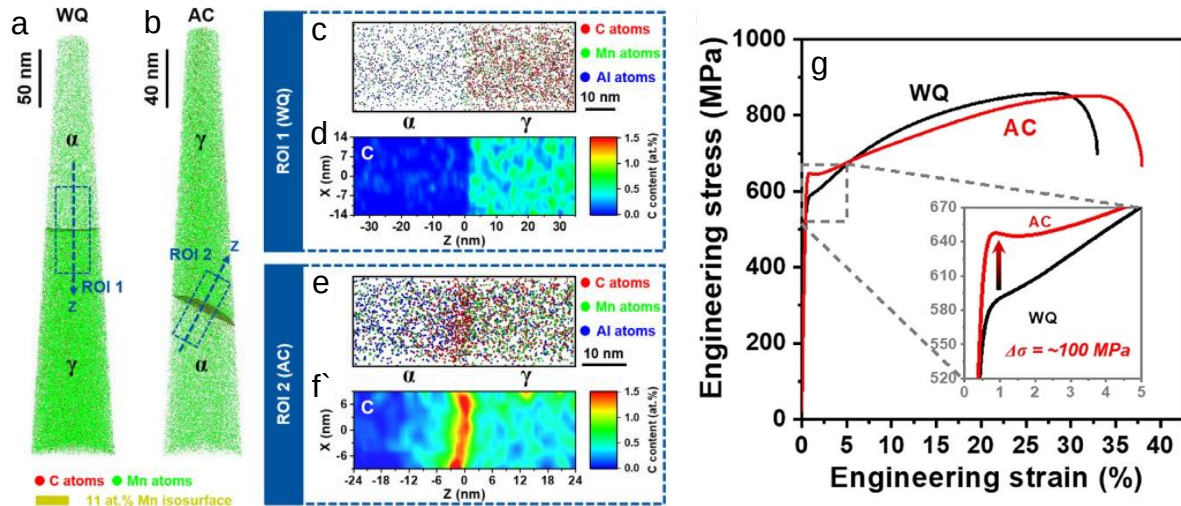


Figure 2.14. Effect of the cooling rate on microstructure and mechanical properties of the Fe-11.7Mn-2.9Al-0.064C steel. APT maps of the Mn and C atoms in (a) the water-quenched (WQ) and (b) the air-cooled (AC) specimens; (c) and (e) C, Mn, and Al atoms distribution in the vicinity of the γ -ferrite phase boundary in the region of interest (ROI) 1 and ROI 2, respectively; (d) and (f) concentration maps of the C atoms in the ROI 1 and ROI 2, respectively. (g) Tensile test results. Taken from [116].

Ma et al. [116] utilised APT technique to study Fe-11.7Mn-2.9Al-0.064C steel. The APT technique enables near-atomic resolution measurements of the atoms' spatial coordinates and elemental identities in volumes spanning hundreds of nanometres [373]. As a result, APT is able to generate three-dimensional images of the solute distribution within the microstructures of the material at atomic resolution. The technique is extremely valuable in the context of the nanoscale metrology investigations. An APT provides the highest three-dimensional compositional information of any microscopy technique (sub-0.3 nm) [374].

Ma et al. [116] found that when Fe-11.7Mn-2.9Al-0.064C steel after the IA was quenched with water, there was no evidence of the C segregation; however, when the sample was cooled with air, there was a significant spike of the C atoms at the boundary, approximately 5 times greater than the nominal C content (0.29 at. %) of the material (Figure 2.14). They showed that during the different cooling procedures, other microstructural characteristics such as grain sizes, phase volume fractions and constituents, and morphologies did not change. Figure 2.14(g) shows that the tensile curve of the water-quenched specimen displays no upper

or lower yield points, and there were no localized deformation bands. Conversely, the air-cooled specimen showed a small ~3 MPa yield stress drop.

According to Furukawa et al. [118], the microstructure of the Fe–5Mn steels with less than 0.1 wt.% C content was not affected by the cooling rate. In contrast, when the steel C content exceeded 0.1 wt.%, the steel microstructure was affected by the cooling rate. The furnace-cooled specimen had a low volume fraction of the retained γ (31.2%) compared to the water-quenched specimen (44.1%). Cementite precipitates along the grain boundaries of the γ , resulting in this condition.

2.5 Austenite Stability

The outstanding mechanical properties of TRIP steels is underpinned by the formation of the metastable γ during processing. This γ is able to transform into martensite under the influence of the external stress. However, the stability of the γ is a critical factor that determines the final microstructure and mechanical properties of the material. In this section, we will review the current knowledge of the factors that affect γ stability in TRIP steel.

2.5.1 Chemical Compositions

A variety of the alloying elements may occupy the interstitial or substitutional positions of the steel depending on their size. As a general rule, any element that tends to expand the γ region in the phase diagrams or increases the chemical free energy difference between γ and martensite stabilizes γ [119]. Alloying nitrogen, C, Mn, and nickel can stabilize γ . A difference in chemical free energy can be estimated for the transformation of the γ into martensite as follows [120]

$$\Delta G^{\gamma \rightarrow \alpha'} = (1 - x)\Delta G_{Fe}^{\gamma \rightarrow \alpha'} + \Delta H^{\gamma \rightarrow \alpha'} \quad 2.1$$

X indicates the proportion of an element (at. %) and ΔH represents the change in enthalpy. From the M_s , it is possible to evaluate the effect of the chemical compositions on γ stability. When the M_s is higher, the γ phase is less stable. In order to predict the M_s^0 , based on γ chemical composition and without considering γ grain size, many formulas have been developed. Some of them are as follows:

$$M_s^0(^{\circ}\text{C}) = 541 - 401[\text{C}] - 36[\text{Mn}] - 10.5[\text{Si}] - 14[\text{Cr}] - 18[\text{Ni}] - 17[\text{Mo}] \quad 2.2 [121]$$

$$M_s^0(^{\circ}\text{C}) = 539 - 423[\text{C}] - 30.4[\text{Mn}] - 17.7[\text{Ni}] - 12.1[\text{Cr}] - 7.5[\text{Mo}] \quad 2.3 [122]$$

$$M_s^0(^{\circ}\text{C}) = 561 - 474[\text{C}] - 33[\text{Mn}] - 17[\text{Cr}] - 17[\text{Ni}] - 21[\text{Mo}] \quad 2.4 [123]$$

$$M_s^0(^{\circ}\text{C}) = 499 - 292[\text{C}] - 32.4[\text{Mn}] - 10.8[\text{Si}] - 22[\text{Cr}] - 16.2[\text{Ni}] - 10.8[\text{Mo}] \quad 2.5 [124]$$

$$M_s^0(^{\circ}\text{C}) = 499 - 324[\text{C}] - 32.4[\text{Mn}] - 10.8[\text{Si}] - 27[\text{Cr}] - 16.2[\text{Ni}] - 10.8[\text{Mo}] - 10.8[\text{W}] \quad 2.6 [125]$$

$$M_s^0(^{\circ}\text{C}) = 499 - 308[\text{C}] - 32.4[\text{Mn}] - 10.8[\text{Si}] - 27[\text{Cr}] - 16.2[\text{Ni}] - 10.8[\text{Mo}] - 10.8[\text{W}] \quad 2.7 [126]$$

$$M_s^0(^{\circ}\text{C}) = 565 - 600(1 - \text{Exp}(-0.96[\text{C}])) - 31[\text{Mn}] - 13[\text{Si}] - 10[\text{Cr}] - 8[\text{Ni}] - 12[\text{Mo}] \quad 2.8 [127]$$

$$M_s^0(^{\circ}\text{C}) = 538 - 350[\text{C}] - 37.7[\text{Mn}] - 37.7[\text{Cr}] - 18.9[\text{Ni}] - 27[\text{Mo}] \quad 2.9 [128]$$

$$M_s^0(^{\circ}\text{C}) = 539 - 423[\text{C}] - 30.4[\text{Mn}] - 7.5[\text{Si}] - 12.1[\text{Cr}] - 17.7[\text{Ni}] - 7.5[\text{Mo}] + 10[\text{Co}] \quad 2.10 [129]$$

$$M_s^0(^{\circ}\text{C}) = 41.7(14.6 - [\text{Cr}]) + 61.1(8.9 - [\text{Ni}]) + 33.3(1.33 - [\text{Mn}]) - 27.8(0.47 - [\text{Si}]) + 1666.7(0.068 - ([\text{C}] + [\text{N}])) - 17.8 \quad 2.11 [130]$$

$$M_s^0(^{\circ}\text{C}) = 531 - 391.2[\text{C}] - 43.3[\text{Mn}] - 16.2[\text{Cr}] - 21.8[\text{Ni}] \quad 2.12 [131]$$

$$M_s^0(^{\circ}\text{C}) = 496(1 - 0.62[\text{C}]) (1 - 0.092[\text{Mn}]) (1 - 0.033[\text{Si}]) (1 - 0.045[\text{Ni}]) (1 - 0.07[\text{Cr}]) (1 - 0.029[\text{Mo}]) (1 - 0.018[\text{W}]) (1 - 0.012[\text{Co}]) \quad 2.13 [132]$$

$$M_s^0(^{\circ}\text{C}) = 520 - 320[\text{C}] - 50[\text{Mn}] - 30[\text{Cr}] - 20([\text{Ni}] + [\text{Mo}]) - 5([\text{Cu}] + [\text{Si}]) \quad 2.14 [133]$$

$$M_s^0(^{\circ}\text{C}) = 512 - 453[\text{C}] - 16.9[\text{Ni}] - 9.5[\text{Mo}] + 217[\text{C}] - 71.5[\text{C}][\text{Mn}] + 15[\text{Cr}] - 67.6[\text{C}][\text{Cr}] \quad 2.15 [122]$$

$$M_s^0(^{\circ}\text{C}) = 692 - 502([\text{C}] + 0.86[\text{N}])^{0.5} - 37[\text{Mn}] - 14[\text{Si}] + 20[\text{Al}] - 11[\text{Cr}] \quad 2.16 [134]$$

$$M_s^0(^{\circ}\text{C}) = 545 - 601.2(1 - \text{Exp}(-0.868[\text{C}])) - 34.4[\text{Mn}] - 13.7[\text{Si}] - 9.2[\text{Cr}] - 17.3[\text{Ni}] - 15.4[\text{Mo}] + 10.8[\text{V}] + 4.7[\text{Co}] - 1.4[\text{Al}] - 16.3[\text{Cu}] - 361[\text{Nb}] - 2.44[\text{Ti}] - 3448[\text{B}] \quad 2.17 [135]$$

where the chemical composition of the element X in weight percent is given by [X]. It is clear from the formulas that Co, Al, and V decrease the γ stability by increasing the M_s temperature, while C, Mn, Si, Cr, Ni, Mo, W, N, Cu and B increase it by decreasing the M_s temperature.

As cementite precipitates in γ during tempering, the γ in bainitic steel will become unstable, which is indirect evidence of the effects of C on γ stability [136]. In 18% Cr-8% Ni steel containing N, γ is more stable than γ in 18% Cr-8% Ni steel containing C [137]. Ni, in addition to being a key component of the Invar alloy [138], stainless steel [139] and maraging steel [140], also contributes to the stabilization of the γ . Using different thermal processing procedures, such as IA [141], quenching and partitioning [142], and bainitic holding [143], it is possible to fully utilize the potential of the Mn and C atoms in stabilizing the γ grains. The effect of Al on M_s temperature can be seen in equations 2.16 and 2.17. According to Equation 2.16, adding Al increases M_s temperature while according to equation 2.17, it decreases M_s temperature. TRIP steels with Al have been found to have a higher γ stability [144]. Phosphorus (P) stabilizes the γ as reported by Jimenez-Melero et al. [144]. The hydrogen embrittlement of the AHSS is frequently observed, however, the hydrogen itself does not substantially affect the γ stability in tensile deformation [145].

2.5.2 Austenite Grain Size

Austenite grains with smaller sizes are generally more stable during deformation or quenching processes [104, 146–150]. Therefore, grain refinement has been used in order to maintain the γ grains at room temperature [109, 151]. There is not much evidence that grain refinement can affect the martensite lath width [152]. Based on the width of the martensite laths in low-carbon steel, which is between 100 nm and 500 nm, it can be expected that grain refinement plays a crucial role in stabilizing the ultrafine γ grain. Several experimental studies have shown that the optimal γ grain size is about 500 nm in the MMnSs [104, 153]. The following equations show that the γ grain size could affect its stability and M_s temperature.

$$M_s^0 - M_s^g = \frac{1}{b} \ln \left[\frac{1}{aV_\gamma} \left\{ \exp \left(-\frac{\ln(1-f_{M_s})}{m} \right) - 1 \right\} + 1 \right] \quad 2.18 [154]$$

and,

$$M_s^0 - M_s^g = Ax_c + BV_\gamma^{-\frac{1}{3}} \quad 2.19 [150]$$

where;

M_s^0 is the martensite transformation-start temperature when V_γ is infinite,

M_s^g is the martensite transformation-start temperature for the finite V_γ ,

b is a constant and equals 0.2689 for α' [154] and 0.19 for ϵ [155],

a is a constant and equals 1 mm^{-3} [154],

V_γ is the average volume of the γ grains,

m is the aspect ratio of the martensite plate which is 0.05 for α' and 0.03 for ϵ [154, 155],

f_{M_s} is 0.01 which is the offset used to determine martensite-start temperatures [154],

A is $423 \text{ wt.}\% / ^\circ\text{C}$ according to Andrew's equation [122],

x_c is C content in wt.% and,

B is the fitting parameter that can be determined based on experimental data.

According to the equations, as γ grain size decreases, M_s^g decreases and γ stability increases.

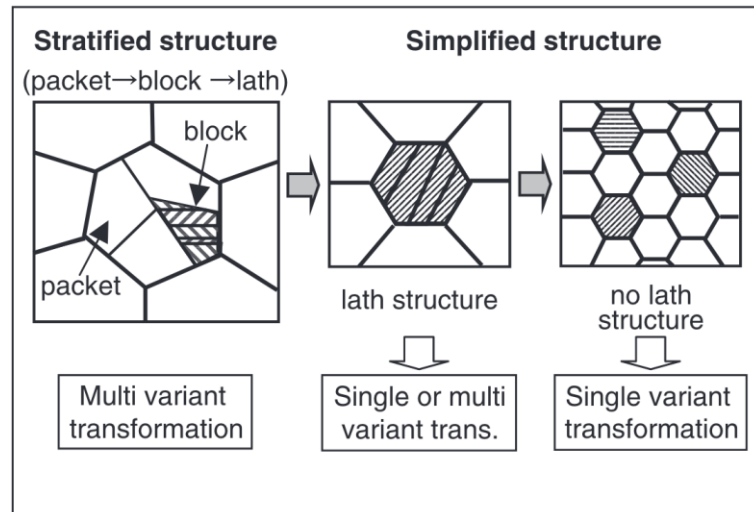


Figure 2.15. Schematic relation between martensitic morphology and γ grain size. Taken from [156].

Figure 2.15 illustrates the relationship between martensite morphology and γ grain size. By refining the grain size of the austenite, martensite's morphology changes from stratified to simplified. Austenite's stability should be impacted greatly by the change from multi variant to single variant transformation [156].

During the transformation of the γ to α' , crystallographic anisotropy is induced as a result of the lattice displacement from γ to α' . The γ into α' transformation (K-S relation) has 24 variants of the lattice displacement, which makes multi variant transformations suitable for

avoiding anisotropy. In order to calculate the amount of the energy required ($\Delta E(\text{total})$) for the martensitic transformation, the following equation can be used.

$$\Delta E(\text{total}) = \Delta E_V + \Delta E_D + \Delta E_B \quad 2.20$$

where;

$$\Delta E_V = 1276.1(x/d)^2 + 562(x/d) \quad 2.21$$

$$\Delta E_D = \tau_y \gamma \quad 2.22$$

$$\Delta E_B = 2e_s/\lambda \quad 2.23$$

ΔE_V is the elastic strain energy for lattice displacement,

ΔE_D is the strain energy required for shear deformation,

ΔE_B is the increment of the boundary energy produced by the formation of the martensite lath,

x is the thickness of the martensite lath,

d is the γ grain size,

τ_y is the shear stress of the pure iron; 100 MPa,

γ is the shear strain; 0.36,

e_s is the boundary energy per unit area; about 0.2 J/m and,

λ is the mean width of the martensite lath; 0.2 mm.

Figure 2.16 illustrates the difference in martensitic transformation energy between single variant mode and multi variant mode using equation 2.20. Compared to multivariate transformations, the transformative energy of the single variants is 17 times greater. Transformation energy is dominated by elastic strain energy, particularly for single variant transformations [156].

According to equation 2.21, a reduction in γ grain size leads to a significant increase in ΔE_V value. It is evident that it is difficult to nucleate martensite lath in γ with grain sizes less than 1 μm .

In contrast, in steels with an added Al content greater than 1 wt.%, Zhu [157] found that M_s temperature increased as the γ grain size decreased. The abnormal phenomenon may be caused by the segregation of Al at prior γ grain boundaries.

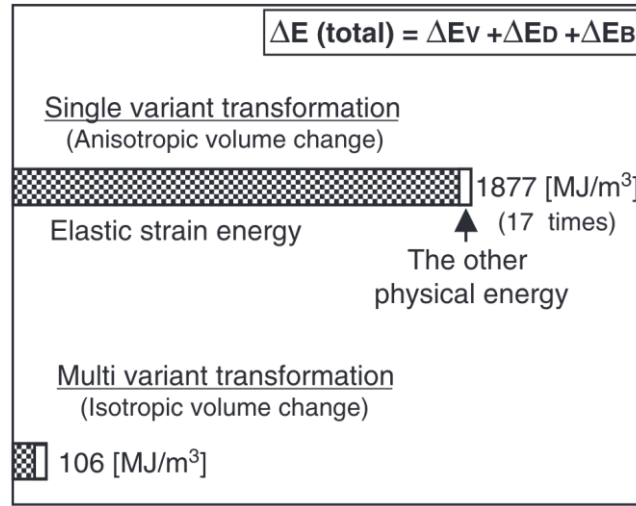


Figure 2.16. Martensitic transformation energy for single variant mode and multi variant mode. Taken from [156].

2.5.3 Dislocations

There is a significant role played by dislocation in the nucleation and growth of the martensite, and, as a result, it has a substantial impact on the γ stability of the steels. There is considerable controversy regarding the effect of the dislocations on γ stability. Figure 2.17 shows an embryo of the martensite in γ formed by the dissociation of two dislocations into partial dislocations and the intersection of the shear lines caused by two arrays of the partial dislocations [158]. It has been reported that dislocation debris could limit the martensite growth [159, 160], and stabilize the γ grains with a large plastic deformations [161]. As the stress driving the interface equals the stress opposing its motion, γ is mechanically stabilized. A critical strain for mechanical stabilization can be calculated using the following equation [159].

$$\tau_T = \tau + \tau_S \Rightarrow b\Delta G = \frac{1}{8\pi(1-\nu)} Gb^{\frac{3}{2}} \left(\frac{\epsilon}{L}\right)^{\frac{1}{2}} + \tau_S b \quad 2.24$$

where;

- τ_T is the stress which is driving the motion of the interface,
- τ is the mean shear stress to force dislocations past each other,
- τ_S is the equivalent shear stress of the solid solution hardening,
- b is Burgers vector,
- ΔG is the chemical free energy change of the austenite into martensite transformation,
- ν is Poisson's ratio,
- G is the shear modulus,
- ϵ is the plastic strain and,
- L is the average distance moved by the dislocations.

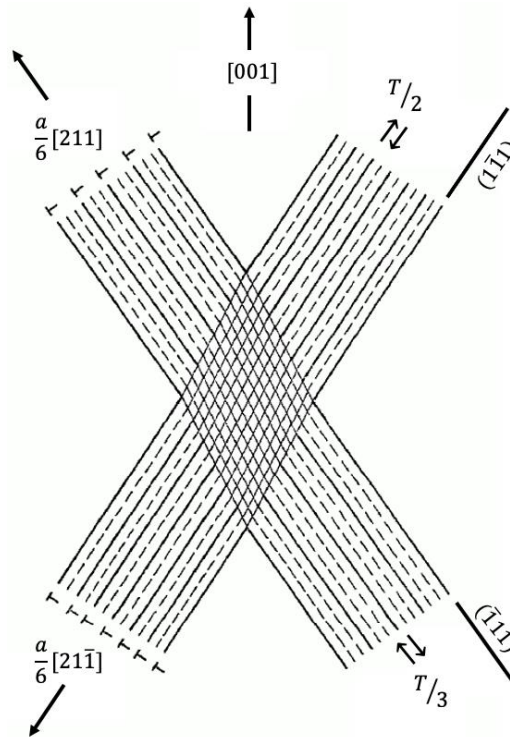


Figure 2.17. Schematic illustration of the intersecting shears due to two arrays of the partial dislocations in γ . This resulted in a doubly faulted intersection with a martensite structure. Adapted from [158].

On the basis of the dislocation amounts, the complex role of the dislocations on γ stability can be explained [162, 163]. Small amounts of the dislocations in γ grains facilitate martensite nucleation, whereas large amounts of the dislocations suppress nucleation [163]. As a result of the small deformation, the potent martensite embryo is located close to the boundaries of the γ grain where the geometrically necessary dislocations (GNDs) are generated [164]. Wang applied severe plastic deformation to austenitic stainless steel and deformed it to a true strain of 6.3. It was found that a true strain greater than 2 is required to mechanically stabilise the steel [165].

2.5.4 Morphology

There are two types of the morphology for γ grains: lath-type and blocky/globular. These morphologies are the consequence of the bainitic transformation [166–168], martensitic transformation [169] or a combination of the different phase transformations [170]. It is also important to note that the morphology is affected by the rolling condition [64], the growth mode [171], and the annealing conditions [172]. It is recognized that athermal α' -martensite transforms into lath-type γ [59, 64, 71, 75, 78, 78, 97, 98, 173–180]. while the deformed α' -martensite, transforms into blocky γ [176, 178, 180, 181].

It has been reported in the literature that γ stability is dependent on its morphology [182–186]. Generally, blocky γ exhibits a lower level of the stability during plastic deformation than lath-type γ [170, 176].

It is important to note that γ grains with different morphologies exhibit a variety of the microstructural characteristics, including grain size, chemical composition, and adjacent phases. It is difficult to discuss how morphology influences γ stability in steels with different phases. In Q&P steel, Xiong found that blocky γ with higher C content and surrounded by ferrite is less stable than lath-type γ with lower C and surrounded by martensite. The lath-type γ found was stable at strains up to 12% [170]. This phenomenon could be explained for several reasons. The first reason could be the martensite shielding effect. In this mechanism, martensite with high yield stress shields the γ during the plastic deformation [176]. The second possible mechanism is that a higher hydrostatic pressure by the residual stress in the lath-type γ could suppress the martensitic transformation [151, 176, 187].

2.5.5 Matrix Strength

There has been considerable research on the effect of the matrix strength on γ stability [176, 188]. In a TRIP-assisted multiphase steel, the γ grains close to the martensite are more stable than those close to the ferrite grain [176]. A tempering process applied to bainitic steel reduces the strength of the bainite by reducing the dislocation density, which in turn destabilizes the γ as compared to bainite steel not tempered. As bainite has a reduced strength, close γ is partitioned with a higher degree of the stress. Additionally, the tempering process may produce a compressive stress field, which may assist in the martensitic transformation [189]. Similarly, γ grains surrounded by a martensite matrix are less stable after tempering [190, 191].

During the transformation process, γ grains located at the boundaries of the ferrite grains transform earlier than those located within the interiors of the ferrite grains [192]. The formation of the GND at grain boundaries accommodates the misfit of the strain at the grain boundary in polycrystalline materials, thus rationalizing this phenomenon [164]. As a result, the γ grain located at the boundary of a ferrite grain is subjected to more deformation than the grain located at the interior of a ferrite grain. Austenite stability is highly dependent upon the morphology of the bainite since the strength of the granular bainite is considerably lower than that of the lath bainite [193]. Mn steels containing Al content of over 3 weight percent

commonly contain delta ferrite. Since delta ferrite is stronger than eutectoid ferrite, the γ grains adjacent to it should be more stable.

A special case of the effect of the matrix strength is the effect of the free surface on γ stability. There are no adjacent phases that oppose the martensitic transformation on the free surface. Observations of the martensitic transformation were conducted following the partial removal of the matrix adjacent to the γ grain in order to investigate the influence of the free surface on the γ stability [194]. A surface martensite forms near the free surface, which is a different type of the martensite from the martensite that is transformed in bulk materials [195]. As a result of a free surface, which indicates zero matrix strength, γ grains are destabilized [196].

2.5.6 Stress State

Shear and dilatational components are involved in the austenite into martensite transformation strain. As a result of the interaction between the applied stress and the shear and dilatational components, different amounts of the mechanical interaction energy are generated. The actual driving force and the mechanical interaction energy (U) can be calculated as follows [187]:

$$\Delta G_{\text{stree}}^{\gamma \rightarrow \alpha} = \Delta G^{\gamma \rightarrow \alpha} + U \quad 2.25$$

$$U = \frac{1}{2} \gamma_0 \sigma_1 \sin 2\theta \pm \frac{1}{2} \varepsilon_0 \sigma_1 (1 + \cos 2\theta) \quad 2.26$$

where;

$\Delta G_{\text{stree}}^{\gamma \rightarrow \alpha}$ is the transformation driving force with the applied stress state,

$\Delta G^{\gamma \rightarrow \alpha}$ is the transformation driving force without the applied stress state,

U is the mechanical interaction energy by an applied stress state,

γ_0 is the shear component of the transformation strain,

σ_1 is the absolute value of the applied stress,

ε_0 is the dilatational component of the transformation strain and,

θ is the angle between the applied stress and the normal direction of the martensite plate.

As shown in Table 2.2, Patel and Cohen calculated the effect of the applied stress on the martensite temperature of two alloys and showed the results are highly consistent with those obtained experimentally. According to the calculation, M_s decreases by hydrostatic pressure and rises by uniaxial tension and uniaxial compression.

Table 2.2. Effect of the applied stress on the M_s [187].

Stress system		Uniaxial tension	Uniaxial compression	Hydrostatic pressure
Alloy (wt.%)		Fe-0.5%C-20%Ni		Fe-30%Ni
$\frac{dM_s}{d\sigma}$ ($^{\circ}\text{C}/10^3\text{psi}$)	Calculated	+1.07	+0.72	-0.38
	Experimental	+1.0	+0.65	-0.57
Change in M_s per 15000 psi ($^{\circ}\text{C}$)	Calculated	+16	+10.6	-5.7
	Experimental	+15	+10	-8.5

A quantitative explanation for the raising of the M_s by compression can be provided by the fact that the shear component of the stress facilitates the transformation more effectively than the compressive normal component of the stress which opposes the transformation. The reason for this is that the displacement under shear is much greater than the displacement under dilatation. In uniaxial tension, both the shear component of the stress and the normal component of the stress facilitate martensitic transformation. Thus, M_s increases more than in the uniaxial compression [187]. In the case of the hydrostatic pressure, both the shear and the normal components of the stress oppose the martensitic transformation, and therefore, the M_s decreases [187, 197].

For Fe–Ni alloys, the linear relationship between the decreasing M_s temperature and the applied hydrostatic pressure stress breaks down when it exceeds 750 MPa [198] which is caused by the Invar effect on martensitic transformation volume expansion [199]. Due to the different thermal expansion ratios of the γ grains embedded in the ferritic matrix, a tensile hydrostatic pressure can be generated on the γ grains. As a result, martensitic transformation may be facilitated [200]. A study found that the maximum and minimum stresses influence the change of the M_s and the intermediate stress has no effect on it [201]. To evaluate the effect of the stress state on γ stability, a more sophisticated self-consistent model is developed based on the orientation relationship [202].

2.5.7 Orientation

There is a correlation between the direction of the loading and the influence of the orientation on γ stability. It has been observed that γ stability depends on orientation during plastic deformation, in which a specific orientation is favoured for martensitic transformations [204–207]. In γ grains with a preferred orientation, martensitic transformation occurs sooner due to a higher resolved shear stress [205, 208, 209].

In plastic deformation, γ grains oriented in a manner that gains a higher mechanical interaction energy have a lower level of the stability. During plastic deformation, grain rotation is one of the mechanisms of the deformation. The grain rotation reduces the misfit caused by the varied Schmidt factors of the grain during the initial plastic deformation [164]. Therefore, γ grains with lower stability are expected to transform into martensite before rotation, while those with higher stability will tend to rotate [203, 210]. As γ grains rotate, Schmidt factors (resolved shear stress) decrease, which reduces the mechanical interaction energy for the martensitic transformation [210]. Stress-induced martensitic transformation [208, 209] or the phenomenological theory of the martensite crystallography [211] can be used to model the effect of the orientation on γ stability. In comparison with the grain size [203] and chemical composition [204, 205], orientation has a less significant effect on γ stability.

Due to the rotation of the γ during the deformation process, it is difficult to assess the effect of the initial orientation on γ stability through macroscopic plastic deformation. The deformation of a single crystal in different directions may, however, provide clarification [212]. In a compression test, a single crystal's critical stress for martensite formation can demonstrate the effect of the orientation on martensitic transformation. Approximately 200 MPa less stress was required to initiate the martensitic transformation in the micropillars [100] than in the micropillars [110]. This difference in γ stability is attributed to the stress required to induce twinning. Only the [100] micropillars contain twins. In [100] micropillars, twins may serve as a site for martensite nucleation, lowering the critical stress for the martensite transformation [213].

2.5.8 Strain Rate

To determine whether strain rate has an effect on γ stability, two factors should be considered, the nucleation of the martensite and the adiabatic heating [214]. At high strain

rates, samples are expected to warm up due to heat dissipation caused by the plastic work [215]. As the strain level increases during the plastic deformation, the adiabatic heating effect becomes more significant [216].

It has been observed that a higher strain rate ($10^3/s$) leads to a greater fraction of the γ transforming into martensite and increases the work hardening (positive strain rate sensitivity) as compared to lower strain rates ($10^{-3}/s$) of the austenitic 304 stainless steel and strains below 0.25 [217–219]. At a high strain rate, more shear bands and their intersections are formed. These intersections may become the potential martensite nucleus, which aids the martensitic transformation [220]. At a high strain rate ($10^3/s$) and strains greater than 0.25, the adiabatic heating significantly suppresses martensitic transformation and reduces the work hardening (negative strain rate sensitivity) [36, 217–219].

Multiphase steel has a more complex relationship between strain rate and γ stability. There was a less martensitic transformation in a typical MMnS at ultralow and high strain rates compared to medium strain rates, possibly as a result of the different strain partitioning between γ and adjacent phases at those different strain rates [221]. According to the results of a high strain rate test, TRIP-assisted multiphase steels containing lath-type γ grains exhibit better mechanical properties than steels containing blocky γ grains [222].

Q&P 980 steel at a high strain rate (175/s) only exhibits positive strain rate sensitivity because the total elongation is less than 10% and the adiabatic heating is not sufficiently high to suppress martensitic transformation [220, 223]. By contrast, Q&P 1500 steel exhibits negative strain rate sensitivity only at high strain rates (103/s) and strains smaller than 0.04 due to the martensite matrix and abnormal TRIP effect [54].

2.6 Strengthening Mechanisms in Steels

It is well known that the presence and mobility of the defects in the crystal structure determine the strength and other mechanical properties of the crystalline metals. Dislocations are the most significant defects in this regard at low temperatures (below about one-third of the melting point), where they serve as plastic strain carriers. As a result, most methods of the strengthening metals are designed to control the movement of the dislocations, which is achieved primarily by introducing obstacles into the movement of the dislocations. Steels at

ambient temperature undergo a variety of the strengthening mechanisms, which are explained here.

2.6.1 Grain Size

It is generally impossible for dislocations to transmit through grain boundaries without an additional increment of the strain energy due to the misorientation between adjacent crystals at grain boundaries. A more severe misorientation can result in a higher threshold of the stress required for transmission than is necessary for the activation of a new dislocation source in the adjacent grain. At the grain boundary, dislocations on a common slip plane tend to pile up until the stress increases sufficiently to cause slip to occur in the next grain. The grain boundaries act as very hard obstacles for dislocation motion in this manner, however, they do not serve as point obstacles like solute atoms or particles. Polycrystals have been experimentally demonstrated to be strengthened by grain boundaries since Hall (Hall 1951; Petch 1953) proposed his relation between yield stress and grain size. The Hall-Petch relation is valid for a wide range of the pure metals and alloys over a wide range of the grain sizes. The Hall-Petch relationship [224] is as follows:

$$\sigma_y = \sigma_0 + kd^{-1/2} \quad 2.27$$

where;

σ_y is the (upper or lower) yield stress or flow stress,

σ_0 is Peierls-Nabarro stress or the friction stress of the overall resistance of the crystal lattice (from solutes and particles but not from dislocations) to the movement of the dislocations,

k is the Hall-Petch constant and,

d is the average grain size.

2.6.2 Work Hardening

As a result of the plastic flow, structural changes are induced, which ultimately increase the difficulty of the subsequent plastic flow; the more a metal is deformed, the stronger it becomes. In polycrystalline metals, work is dissipated during plastic flow due to the motion and interaction of the dislocations. Strain energy is stored through an increase in the length of the dislocation line. This is done through processes such as sequential emission of the newly

formed dislocations for example from the Frank-Read sources and bowing around pinning points and obstacles. An entanglement of the dislocations can result in jogs or sessile kink that acts as pinning point along the line of the dislocation [225].

The Bailey-Hirsch relationship [226] can be used to calculate the enhanced strength as a result of the increased dislocation density and dislocation entanglement as follows.

$$\Delta\sigma_D = \alpha M G b \sqrt{\rho} \quad 2.28$$

where;

- α is constant,
- M is average Taylor factor,
- G is the shear modulus,
- b is the magnitude of the Burger's vector and,
- ρ is dislocation density.

There is a fundamental limit to the $\Delta\sigma_D$ from work hardening when dislocation density reaches the maximum value in each metal.

2.6.3 Solid Solution

An addition of the solute atoms to a pure metal can act as a soft barrier that prevents dislocations from moving through the crystal. Two main ways solutes interact with dislocations are the modulus effect and the size effect. The first effect of the solute atoms is that they alter the local values of the elastic constants, which, in turn, affect the line energy of the dislocations. Those atoms in the matrix that stiffen it repel dislocations, whereas those that make it more compliant attract them. The second effect of the solute atoms is that they can cause distortions in the crystalline lattice when added to a pure metal. The stress fields associated with such distortions can interact with dislocations. The interaction may be attractive or repulsive depending on the mismatch between the size of the solutes and the matrix as well as their position relative to the dislocation core. The overall effect of the solid solution is strengthening, since an additional amount of the stress is required to cause a dislocation to approach or leave the vicinity of an atom of a solute [225].

A solid solution can be strengthened in two ways, depending on the size of the solute atom. Either interstitial solid solution strengthening occurs when the solute atoms are much smaller than the solvent atoms such as C, N, and B solute atoms in Fe solvent, or substitutional solid solution strengthening occurs when atoms of the solutes and solvents are generally similar in size such as Mn, Al, Si, Ni or Cr solute atoms in Fe solvent [119]. It is possible to calculate the critical stress required for dislocations to be unpinned in an interstitial solid solution as follows [227]:

$$\sigma_{\text{crit}} = \frac{0.65}{r_{\text{max}}^2 b^2} \quad 2.29$$

where;

r_{max} is the maximum size of the interstitial atom that can effectively pin the dislocation and,
 b is the Burgers vector.

To relate the shear (or flow) stress and the yield stress to the solute concentration in substitutional solid solutions, Fleischer and Arsenault et al. proposed the following equations respectively [227].

$$\Delta\tau = Gb\varepsilon^{3/2}c^{1/2} \quad 2.30$$

$$\tau = KGb\varepsilon^{4/3}c^{1/2} \quad 2.31$$

where;

τ is the shear (or flow) stress,
 G is the shear modulus,
 b is the Burgers vector,
 ε is the strain and,
 c is the solute concentration.

Individual contributions of the alloying elements to solid-solution strengthening were calculated as follows [34]:

$$\sigma_{\text{ss}} = \sum_j c_j X_j \quad 2.32$$

where;

c_j is the c strengthening effect of the element j in the phase and,

X_j is the concentration of the element j in the phase.

2.6.4 Clustering and Second-Phase (Precipitates and/or Other Particles) Strengthening

Clustering, also known as cluster strengthening, extensively studied in alloys. It refers to the formation of small atomic clusters within the microstructure of alloys, playing a crucial role in shaping their mechanical properties. Researchers [228–230] investigated the clustering phenomenon in alloy steels, specifically examining its effects on the mechanical properties of alloy steels.

Precipitation hardening requires a fine distribution of the second-phase particles in order to achieve effective strengthening. It is possible for precipitates to be coherent with the matrix, and dislocations can cut through those particles. The interaction between the dislocations and the strain fields of the precipitates during this process results in the strengthening of the material as dislocation movement is hindered [231, 232].

The dislocations bend around the particles if the particles become incoherent as a result of the over-aging, leading to Orowan loops. The Orowan mechanism forms a semicircular dislocation curve between two particles during plastic deformation. As a result of yielding, the particles are left with Orowan loops around them. Dislocation motion is significantly hampered by the formation of the Orowan loops. As a result, much work hardening occurs.

When particles are small, dislocations can cut through them at a lower stress than the Orowan bowing stress. It is necessary to apply higher cutting stresses to larger particles. Large particle sizes result in dislocations bending around particles rather than cutting through them. There are fewer particles per unit volume with larger particles, thus flow stresses are lower [227, 233].

Steels may contain precipitates such as carbides, nitrides, and intermetallic compounds. The Ashby-Orowan relationship [231] can be used to calculate the improved strength caused by finely dispersed precipitates:

$$\Delta\sigma_p = 0.84 \left(\frac{1.2Gb}{2\pi L} \right) \ln \left(\frac{d}{2b} \right) \quad 2.33$$

where;

- G is the shear modulus,
- b is the Burgers vector,
- L is the distance between precipitates and,
- d is the diameter of the precipitate.

2.7 Deformation Mechanisms in Steels

There are different plastic deformation mechanisms in steels which depend on factors such as the type of the steel (composition, microstructure), the level of the stress, and the temperature at which the deformation occurs. The main mechanisms include dislocation gliding, twinning, grain boundary sliding, and grain rotation. Additionally, this section explores processes resulting from deformation in steels, such as shear band-induced plasticity (SIP), micro band-induced plasticity (MBIP), dynamic slip band refinement (DSBR), TRIP (Transformation-Induced Plasticity), and dynamic strain aging (DSA).

Stacking fault energy (SFE) strongly influences austenitic steel deformation mechanisms [29, 30, 234–247]. Although dislocation slip can occur at any SFE, it is predominately found at lower degrees of the deformation and in alloys with a high SFE. In steels with SFE less than 18 mJ/m², martensite transformation happens. Twinning occurs when the steel's SFE exceeds 18 mJ/m² but is less than 50–80 mJ/m². Dislocation cells are observed during the deformation of the high-Mn alloys with an SFE of more than 30 - 40 mJ/m². As SFE decreases, the plastic deformation mechanisms change from dislocation glide (slip) to dislocation glide and TWIP and ultimately to dislocation glide and TRIP. [248–251].

2.7.1 Dislocation Gliding

There are two types of the dislocation gliding: planar glide and wavy glide. The planar type operates in low-SFE materials and has low three-dimensional mobility, making cross-slipping difficult. High SFE materials, such as Fe–Mn–Al–C steels, exhibit strong three-dimensional mobility of the dislocations, resulting in easy cross slip. High SFE materials have dense dislocation walls and microbands that exhibit an orientation relationship related to stress.

In contrast, low SFE materials have crystallographic orientations for their domain boundaries and microbands [244, 252, 253].

Ha et al. [254], Herrmann et al [255] and Morris et al [256] found that the superdislocation in ferrite and the planar gliding dislocation in γ perform the majority of the strain hardening during strain hardening of the steel.

There are three main parameters that affect the planar glide in FCC metals:

- SFE: A low SFE results in the splitting of the perfect dislocations into Shockley partials, preventing the cross-slipping of the dislocations. Thus, large dissociation widths result in reduced cross-slip frequency, resulting in a planar glide [257].

- stress multiplication effect: If friction stresses are high in a material, the critical resolved shear stress acting on a single dislocation may not be sufficient to overcome the threshold stress necessary for successful gliding. The stress multiplication effect [63] enables dislocations to glide in the same plane collectively [258].

- Short-range order (SRO): Compared to a disordered crystal, a crystal with short-range order (SRO) increases the amount of the stress required for a dislocation to glide [259–261].

2.7.2 Shear Band-Induced Plasticity

This effect occurs due to the formation of the shear bands in γ . Combined with evenly spaced carbides, which are coherent with the matrix, the shear bands prevent the movement of the dislocations. A high rate of the strain hardening is thus caused by this effect, which prevents the formation of a neck in the specimen during the tensile test, resulting in an increased yield strength/tensile strength ratio [29, 262–266].

2.7.3 Micro Band-Induced Plasticity

The microbands are composed of the GNDs that are responsible for the high total dislocation density during deformation and the resulting continuous strain hardening. It was observed that crystallographic microbands and their intersections formed during the deformation [245].

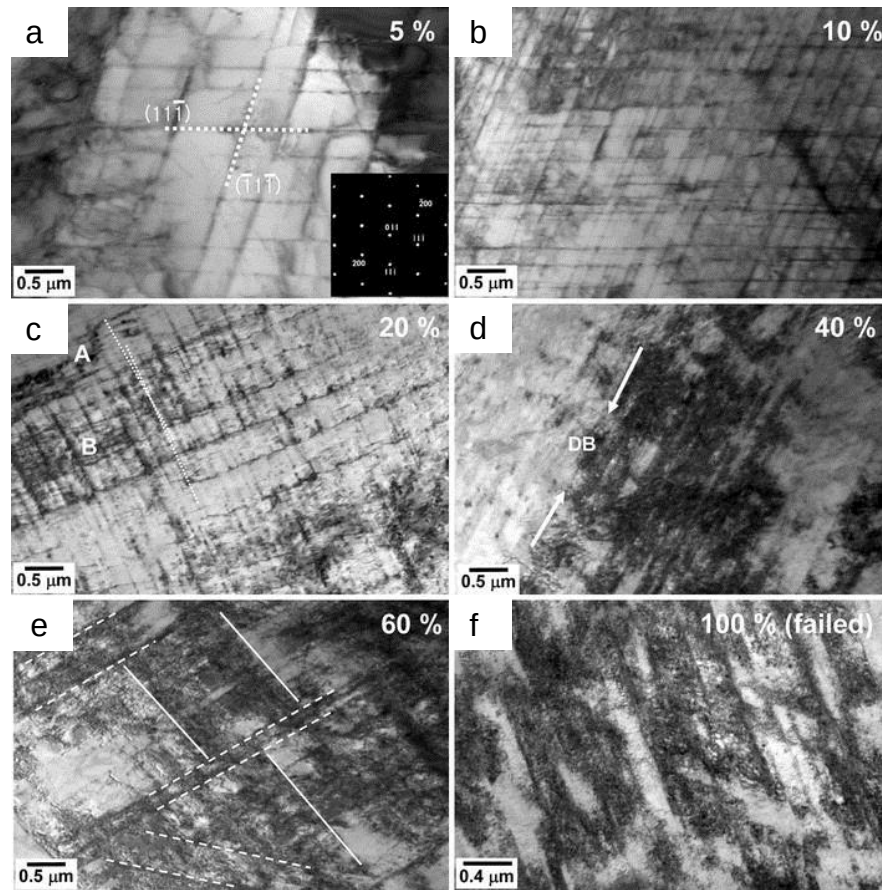


Figure 2.18. Microstructure of the Fe-28Mn-10Al-1C during the plastic deformation. Taken from [247].

Yoo et al [247] found the formation of the domain boundaries (DBs) and microbands (MBs) at high strains in Fe-28Mn-10Al-1C steel (Figure 2.18). The Figure 2.18 shows (a) the $(11\bar{1})$ and $(\bar{1}1\bar{1})$ slip planes are along with the $[011]$ zone with equal spacing dislocation arrays at 5% strain, (b) spacing between slip planes have identical orientation relationship and the space between them decreased at 10% strain. (c) Taylor lattice domains with slight misorientation formed while slip traces faded out ("A") at 20% strain. (d) Since the slip traces were difficult to resolve, single-wall domain boundaries (DBs) were formed instead at 40% strain. (e) There was a dominant intersection between a single wall domain boundary (solid line) and a band structure with two walls (dotted line), leading to the subdivision of the grain into fine Taylor lattice domains at 60% strain. (e) By increasing the number of the intersections between DBs or MBs, the TL domains appeared more subdivided.

2.7.4 Dynamic Slip Band Refinement

The presence of the high slip planarity results in the formation of the very thin slip bands that expand throughout the entire grain. During straining, the slip band structure is refined, which reduces the spacing between the slip bands. This deformation process is therefore called

the dynamic refinement of the slip bands [235]. Figure 2.19 illustrates the schematic of the DSBR deformation mechanism in a grain. Table 2.3 compares the SIP, MBIP and DSBR deformation mechanisms in three Fe-Mn-Al-C alloy steels.

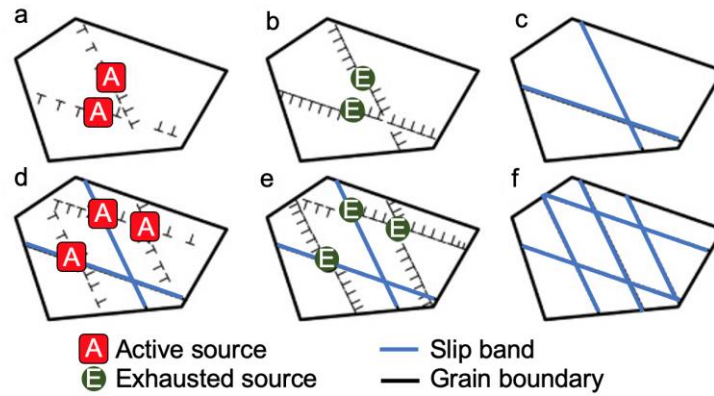


Figure 2.19. Schematic of the DSBR deformation mechanism: (a) and (d) activation of the new dislocation sources, (b) and (e) slip planes consist of the full-up dislocations and (c) and (f) dislocation sources were exhausted due to stress. Adapted from [235].

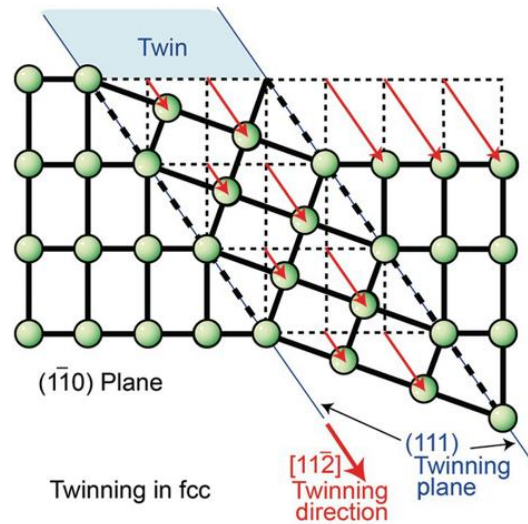
2.7.5 Twinning

Twinning deformation allows crystals to undergo permanent shape changes with minimal volume changes. Twinning consists of the shearing apart a crystal by a fixed magnitude, which is determined by the crystal structure. Twin formation occurs at very high speeds (microseconds) [267]. As shown in Figure 2.20 [268], twinning occurs when the planes of the atoms are moved parallel to a specific plane (twinning plane) so that the lattice is divided into two parts that are symmetrical but oriented in different directions. Through the twin plane, a mirror image forms. Each crystal structure exhibits twinning along a specific crystallographic plane. Table 2.4 shows common twinning systems in metals [267].

Qin [39] observed that, in TWIP steels, the twinning contribution to the overall strain is dependent upon the crystallographic texture and the twin volume fraction. Qin assumed the twinning was the only deformation mode and estimated that the true strain due to twinning was less than 0.15. Mechanical twinning contributes less than 3% to plastic strain in TWIP steel. Deformation twinning can positively affect the mechanical properties of the TWIP steels by influencing the dislocation density [28]. Three models explain how deformation twinning increases tensile ductility and strain hardening: the composite effect [30], the back stress effect [269], and the dynamic Hall-Petch effect [24, 270].

Table 2.3. Comparison of the SIP, MBIP and DSBP deformation mechanisms in three Fe-Mn-Al-C alloy steels.

Deformation mechanism		SIB [271]	MBIP [247]	MBIP [235]
Alloy		Fe-27Mn-12Al-	Fe-28Mn-10Al-	Fe-30.4Mn-8Al-
		0.9C	1.0C	1.2C
Heat treatment	Temperature (°C)	1050	1000	1100
	Time (min)	25	60	120
	Quenched in	Water	Water	Oil
SFE (mJ/m ²)		110	120	85
Strain rate (Sec ⁻¹)		10 ⁻⁴	10 ⁻³	5x10 ⁻⁴
Tensile test	Yield stress (MPa)	875	873	900
	Elongation (%)	58	99	68

**Figure 2.20.** Schematic of the twinning deformation in FCC. Taken from [268].**Table 2.4.** Common twinning systems in metals.

Crystal structure	Twinning direction	Twinning plane
BCC	$\langle 111 \rangle$	$\{112\}$
FCC	$\langle 11\bar{2} \rangle$	$\{111\}$
HCP	$\langle \bar{1}011 \rangle$	$\{10\bar{1}2\}$

2.7.6 Transformation Induced Plasticity

The formation of the strain-induced ϵ -martensite and α' -martensite in γ has been reported in Mn steels [272, 273]. It has been reported that there are two different phase transformations, $\gamma \rightarrow \epsilon \rightarrow \alpha'$ and $\gamma \rightarrow \alpha'$ [274]. Steel's chemical composition, the alloying elements and their distribution within the phases, influence the volume fraction and stability of the retained γ [275]. TRIP deformation is also affected by strain rate and temperature [276]

During cooling, binary austenitic Fe-Mn alloys are partially transformed into ϵ and α' . It has been shown that alloying elements significantly affect the phase stability of the binary Fe-Mn alloys [262, 263]. A correlation exists between the phase transformation and the stacking fault energy of the γ . The $\gamma \rightarrow \epsilon$ phase transformation is favoured when the stacking fault energy is low (equal to or less than 20 mJ/m^2), whereas it is suppressed when the stacking fault energy is high (more than 20 mJ/m^2) [262]. Al increases the SFE of the γ and therefore suppresses the $\gamma \rightarrow \epsilon$ transformation when added to steel [277]. Si, on the other hand, decreases the SFE of the γ and maintains the $\gamma \rightarrow \epsilon$ transformation upon cooling and deformation [278].

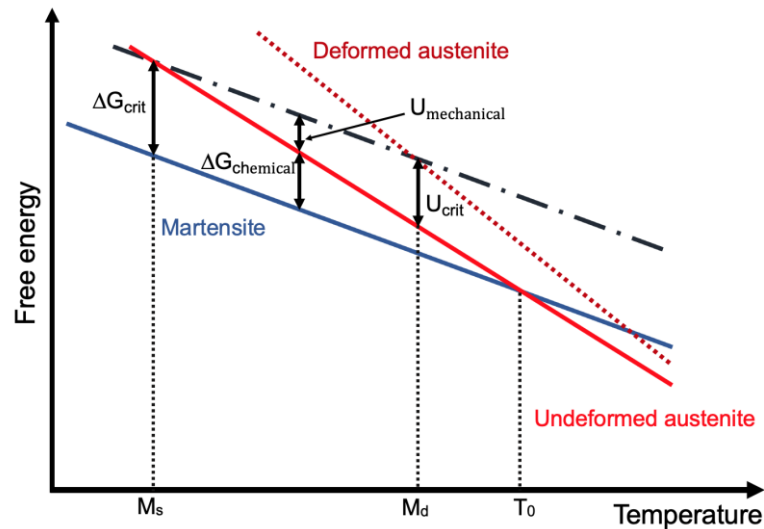


Figure 2.21. Schematic free energy graph of the deformation-induced martensitic transformation. Adapted from [279].

Figure 2.21 shows the Schematic free energy graph of the deformation-induced martensitic transformation. In equilibrium at T_0 , austenite and martensite have the same chemical-free energy. Undercooling below the M_s temperature is necessary to initiate martensitic transformation with the minimum free energy ΔG_{crit} . At M_s temperature, ΔG_{crit} is the difference in chemical free energy ($\Delta G_{\text{chemical}}$) between austenite and martensite phases. It has been found that martensitic transformation can be triggered by supplying additional energy

in the form of the strain energy (U_{mech}) during an inelastic deformation above M_s and below M_d temperatures.

It is not possible to undergo this transformation at temperatures above M_d , since the chemical driving force is insufficient, and the accumulated internal strain energy cannot adequately cover the free energy deficit associated with deformation-induced martensitic transformation, probably due to the recovery effect [279].

M_s^σ temperature is the temperature between M_s and M_d temperatures. At M_s^σ temperature, the required stress for austenite to martensite transformation equals the yield strength. Austenite yields at temperatures below M_s^σ temperature by stress-induced transformations to martensite at pre-existing nucleation sites. As a result of the stress-assisted transformation, the stress for the martensitic transformation is lower than the yield strength, and it is in the elastic region.

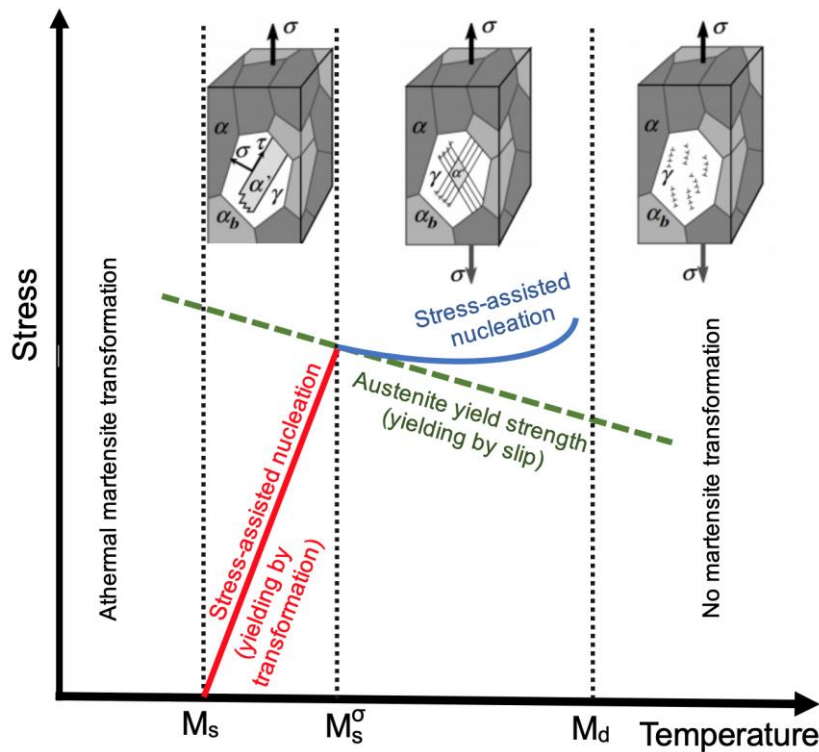


Figure 2.22. Schematic of the austenite to martensite transformation mechanisms. Adapted from [12, 158].

At temperatures between M_s^σ and M_d , martensite nucleation occurs primarily at new nucleation sites formed by slip. This deformation is called strain-induced deformation. The intersection of the strain-induced deformation bands provides additional nucleation sites for austenite to martensite transformation. Figure 2.22 illustrates the mechanism for the transformation of the austenite into martensite.

2.7.7 Dynamic Strain Aging

A DSA effect contributes to the localized plastic deformation and the formation of the deformation bands [33, 280, 281]. Cottrell [282] explained that dislocations interact with interstitial solute atoms in solid solutions (such as C). In the interstitial region of the solute, solute atoms prevent dislocations from moving, which eventually results in the dislocations being pinned. Dislocations can be rapidly unpinned when sufficient amounts of the stress are built up and the pinned dislocations are able to surpass the critical energy barrier to continue moving. Alternate rapid pinning and unpinning of the dislocations is characterised by load drops in the stress-strain curve, called the Portevin Le-Chatelier (PLC) effect.

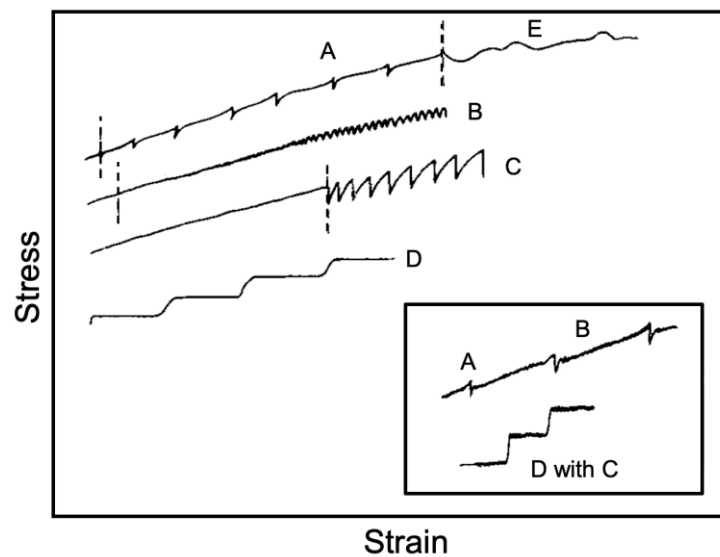


Figure 2.23. Types of the serration. Taken from [280].

Figure 2.23 illustrates 5 types of the serrations caused by DSA, according to Rodriguez [280].

- **Type A:** It occurs when the temperature is low, or there is a high strain rate. These serrations are called 'periodic locking and unlocking serrations'. As a result of the serrations, a deformation band propagates along the specimen's length and reaches the end of it, causing the specimen to deform over time. Repeatedly, this phenomenon occurs.

- **Type B:** When strain rates are low, and temperatures are high, type B serrations can develop from type A serrations. Resulting from the discontinuous propagation of the deformation band, these serrations are seen as fine oscillations in the stress-strain curve.

- **Type C:** These serrations occur at higher temperatures and lower strain rates than A and B. They appear as yield drops below the general levels of the curve.

- **Type D:** Similar to Lüders band, these serrations are plateaus in the strain-stress curve.

- **Type E:** At high strains, type A serrations might change to type E serrations without much work hardening.

DSA has been reported to occur in ferritic steels, at room temperature [283, 284] and as well as at temperatures in the range of 80 °C to 500 °C [285–288], high Mn austenitic steels at room temperature [289, 290], low Mn steels [291, 292] and MMnSs [64, 293–297]. A study by Wang et al. [293] indicated that heterogeneous plastic flow occurs as a result of the differences in dislocation densities in both α and γ phases of the multiphase steels.

2.7.8 Grain Rotation

It was found that during plastic deformation, nanosized grains rotate and coalesce along shear directions, providing a longer axis for dislocations to move [298–301]. As shown in Figure 2.24 there is a short line in each grain representing the orientation of the slip systems with the highest Schmid factors (a). When two nearby grains are subjected to plastic deformation, they may rotate in a manner that brings their orientation closer to one another (b). Consequently, a path for more extended dislocation motion is created due to the elimination of the barrier represented by the boundary between them (c). As a result of this mechanism, localized deformation can take place as well as softening [302].

It has been suggested by Murayama et al. [303] that partial disclination defects in iron may permit the rotation of the nanosized crystals during mechanical milling. Disclinations are line defects characterised by the rotation of the crystalline lattice around their lines [304]. When a disclination dipole moves, the crystal lattice rotates behind the disclinations, resulting in a plastic flow as shown in Figure 2.25.

2.7.9 Grain Boundary Sliding

The grain boundary sliding mechanism occurs when the lattice structure is disordered such as high angle grain boundaries (HAGBs). As a result of this mechanism, relative shearing

occurs along the interlayer that separates neighbouring grains, as well as causing the grains to rotate [267]. A high strain rate hardening ability and a high fraction of the grain boundaries in nano-grained/ultrafine-grained alloys are predicted to result in superplasticity [305]. Superplastic materials have high elongation during plastic deformation ($\geq 100\%$) [306–308].

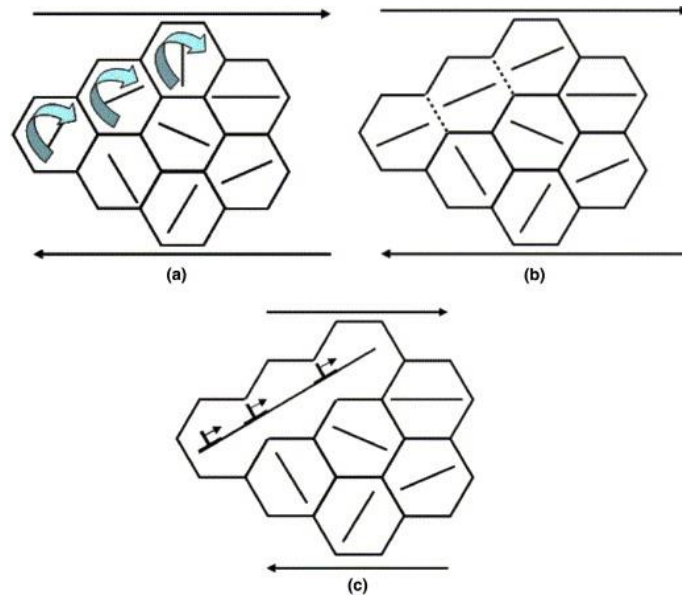


Figure 2.24. The rotation of the neighbouring nanograins during plastic deformation and the creation of the elongated grains as a result of the annihilating grain boundaries. Taken from [302].

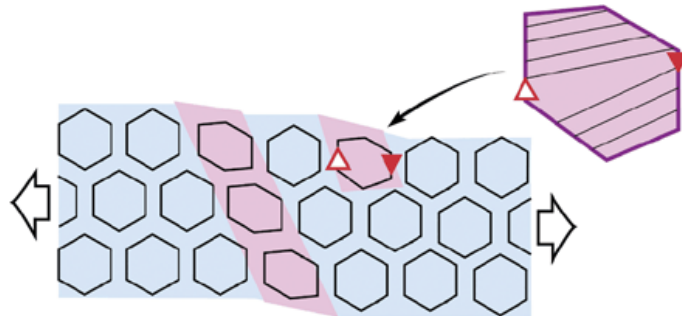


Figure 2.25. Nano grains (hexagons) rotate under mechanical tension via a motion of the dipoles of the disclinations (triangles). Taken from [304].

Misra et al. [309] showed that the ultrafine-grained Fe-0.1C-1.55Mn-0.16Si-0.1V-0.02Al-(0.5–1Ni)-0.018N steel exhibits superplasticity at low temperature controlled by grain boundary sliding and cavitation growth mechanisms. Recently, Liang et al. [310] reported that ultrafine-grained ferritic steel with nano-sized carbides exhibited high strength and plasticity. According to their explanation, grain boundary sliding contributes to the large elongation of the plastic deformations at room temperature.

The phase boundary is the point where two crystals with different lattice structures and compositions meet. The phase boundary sliding can release the stress concentration at the phase

boundary. Phase boundary sliding becomes extremely difficult during deformation due to the cohesive or semi-coherent nature of the interphase boundaries [311–314].

It has been shown that the orientation relationship between the austenite and the parent ferrite is very close to Kurdjumov-Sachs [86, 87]. Kurdjumov-Sachs (KS) orientation relationship (OR) is defined as $(111)_\gamma \parallel (110)_\alpha$ and $[\bar{1}\bar{1}0]_\gamma \parallel [1\bar{1}1]$. This study set a maximum deviation of 10° from the K-S relationship. According to the earlier studies, such a deviation from the KS OR was expected [311, 313–315].

2.8 Influence of the Alloying

A thorough understanding of the actions and interactions of the alloying elements is necessary to control the martensitic transformation during the cooling stages of the continuous annealing. CALPHAD methods (computational calculation of phase diagrams) have been used extensively by the research community to explore the role of various alloying elements in MMn-AHSS [316–322]. Several thermochemical software packages with large thermodynamic databases for this purpose include Thermo-Calc [323], MatCalc Engineering [324], FactSage [325], Pandat [326], CEQCSI [327], OpenCalphad [328], Multi-Phase Equilibrium (MPE) [329] and MTDATA [330]. A summary of the general influences of the usual alloying elements on the transformation behaviour of the DP and TRIP steels can be found in Figure 2.26 [331]. The effect of some alloying elements on steels is briefly discussed here.

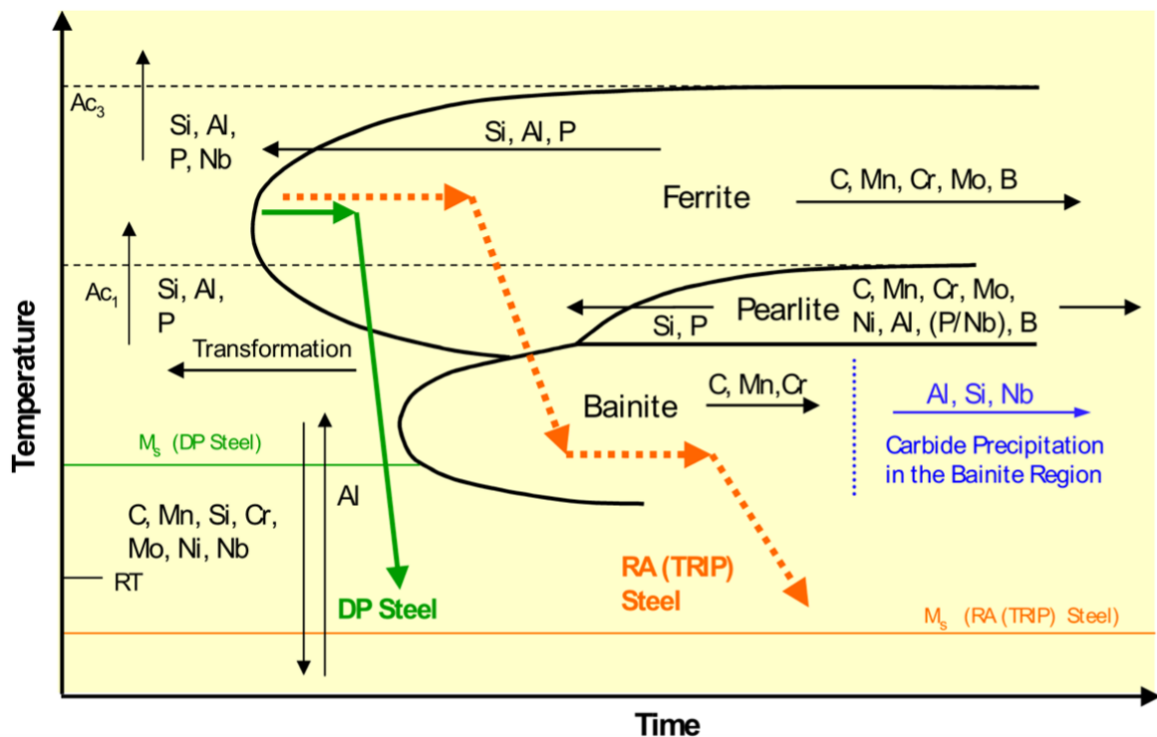


Figure 2.26. Influence of the alloying elements on TTT behaviour. Taken from [331].

- **Mn:** Mn stabilizes γ and reduces the Ac_1 and Ac_3 temperatures [332]. Mn reduces the M_s by 30 °C to 40 °C when 1 wt.% is added to the γ phase [122, 128, 333]. Gibbs [334] and Sun et al. [332] demonstrated that adding 2-3 wt.% Mn content enhances the fraction of the retained γ by about 20%. With increasing Mn concentration, SFE of the γ at room temperature first decreases and then increases [27, 335, 336]. Increasing the steel's SFE changes the deformation mechanism from TRIP to TWIP in Fe-Mn-C systems [337–339]. Evidence also

shows that Mn is prone to segregating into lattice defects such as interfaces, triple junctions, and dislocations [102, 340–343]. The low diffusivity of the Mn atoms has been demonstrated in several publications, particularly within the γ phase [344–346]. This phenomenon has been shown to alter the chemical composition locally, which impacts various mechanical and microstructural properties [102, 340–343, 347].

- **C:** C stabilizes γ and reduces the Ac_1 and Ac_3 temperatures. C solubility is high in steel at higher temperatures, which strengthens the matrix (ferrite/martensite phase) through the interstitial solid solution: for every one weight percent of the C atoms, a hardening effect of a few thousand MPa can be achieved [34, 332]. Higher C content may also reinforce the matrix at room temperature as a result of the precipitation hardening [119]. Also, the increasing C content may result in cementite precipitation, and intergranular fractures along γ boundaries [118]. Further, high C content negatively impacts the weldability of the steels, causing martensite to form after cooling. Additionally, an increase in C content increases the SFE of the γ substantially [348–350], resulting in a change from the TRIP to TWIP deformation mechanism.

Interactions between C atoms and lattice defects like dislocations also profoundly affect discontinuous yielding and the Portevin-Le Chatelier (PLC) phenomenon (DSA) [105, 116, 296, 297, 351]. Even though C plays an important role in many fundamental processes, including static and DSA as well as the deformation twinning, the role of the C atoms on the TWIP steels remains unclear. As a result, a wide range of the advanced methods for microstructure analysis will be required, including measurements of the internal friction [28].

- **Al:** Al acts as a ferrite stabilizer, enlarges the austenite-ferrite dual-phase region in the phase diagram, and allows more control over the microstructure. Al contributes to a reduction of the steel mass density [352, 353]; it is sometimes used up to 10 wt. % in MMnSs [352, 354]. It has been reported that Al increases SFE of the γ by $6.7 \text{ mJ.m}^{-2}.\text{wt.}\%^{-1}$ to $10 \text{ mJ.m}^{-2}.\text{wt.}\%^{-1}$ [249, 355–359]. It has been demonstrated that Al suppresses the formation of ϵ - and α -martensite or martensitic transformations [263, 356, 360]. According to some reports, Al improves the resistance of the TWIP steels to hydrogen embrittlement due to the reduction of the effective diffusivity of the hydrogen atoms within the γ [361, 362]. Al enriches the surface oxides and inhibits the permeation of the hydrogen atoms into the steel [363]. In addition, its high passivity improves corrosion resistance. Al reduces the C diffusivity within the dislocations by increasing the apparent activation energy for the initiation of the PLC effect in

the steels [28, 264]. The use of Al enhances the toughness of the steels when they are exposed to cryogenic temperatures [263, 364, 365].

- **Si:** Si acts as a stabilizer for ferrite. The yield strength of the γ and ferrite increases by adding Si, an effective solution-strengthening element. Si has a complex effect on the SFE of the γ . Based on a thermodynamic model developed by Dumay et al. [366], Si increases SFE in small amounts and decreases it in higher amounts. As a result of the experimental data, the SFE of the γ was shown to decrease with increasing Si concentration [367]. It has been demonstrated that adding Si leads to the formation of the ordered phases within ferrite, such as B_2 and DO_3 [368, 369].

The presence of the Si element suppresses cementite (carbides) formation during heat treatment [119, 370, 371]. Si can be effectively added to Fe-27Mn binary system to refine ϵ plates and to increase toughness [263]

- **Mo:** It has been shown that Mo is a ferrite stabilizer and can form a brittle σ phase [372–374]. 2 to 6 wt.% of the Mo element in conventional austenitic stainless steels increases localized corrosion resistance [375–377]. It has been found that although Mo thermodynamically favours carbide precipitation and it slows its precipitation kinetically [370].

2.9 Principles of the Phase Fraction Measurement by X-Ray Diffraction

An effective non-destructive technique for characterising crystalline materials is XRD. In XRD measurements, the peaks result from constructive interference between a monochromatic beam of the X-rays scattered from each set of the lattice planes of the sample at specific angles. XRD can give information of phases, structures, texture or preferred crystal orientations, crystal defects, strain, crystallinity and average grain size.

The total integrated intensity for all peaks of a particular phase is proportional to its volume fraction. X-ray diffraction patterns can quantify the relative volume fraction of the ferrite and γ for a randomly oriented sample. Assuming that crystalline phases and their grains are oriented randomly, each single diffraction peak (hkl) crystalline plane has an integrated intensity proportional to the phase volume fraction [378].

$$I_{\alpha}^{hkl} = KR_{\alpha}^{hkl}V_{\alpha}/2\mu \quad 2.34$$

Table 2.5. Ferrite or Martensite calculated theoretical intensities, R, using Molybdenum K α radiation (unit-cell dimension $a_0 = 2.8664\text{\AA}$) [378].

hkl	θ	R	hkl	θ	R
110	10.1	1840	600		2.45
200	14.36	288.6	532	49.84	18.14
211	17.68	530	611		9.07
220	20.53	142.5	620	51.63	8.46
310	23.08	172.8	541	53.46	16.01
222	25.43	37.56	622	55.32	7.67
321	27.64	155.6	631	57.22	14.87
400	29.73	14.06	444	59.19	2.43
330	31.73	21.12	543	61.23	14.51
411		42.23	550		3.63
420	33.67	32.8	710		7.25
332	35.55	26.23	640	63.37	7.33
422	37.4	21.51	552	65.64	7.53
431	39.21	36.1	633		7.53
510		18.05	721		15.05
521	42.77	26.94	642	68.08	15.8
440	44.53	5.97	730	70.76	8.55
433	46.29	10.75	651	77.46	24.03
530		10.75	732		24.03
442	48.06	9.81			

where:

$$K = \left(\frac{I_0^4}{m^2 c^4} \right) \times \left(\frac{\lambda_A^3}{32\pi r} \right) \quad 2.35$$

and

$$R_{\alpha}^{hkl} = \frac{|F|^2 p L P e^{-2M}}{v^2} \quad 2.36$$

and for diffractometric analysis

$$LP = \frac{1 + \cos^2 2\theta}{\sin^2 \theta \cos \theta} \quad 2.37$$

and for diffraction by monochromator

$$LP = \frac{1 + \cos \theta^2 2\alpha \cos^2 2\theta}{(\sin^2 \theta \cdot \cos \theta)(1 + \cos^2 2\alpha)} \quad 2.38$$

and if diffraction by the monochromator happens in a plane perpendicular to the plane of the sample diffraction

$$LP = \frac{\cos^2 2\alpha + \cos^2 2\theta}{\sin^2 \theta \cos(1 + \cos^2 2\alpha)} \quad 2.39$$

and

Table 2.6. Austenite calculated theoretical intensities, R, using Molybdenum K α radiation (unit-cell dimension $a_0 = 3.60\text{\AA}$) [378].

hkl	θ	R	hkl	θ	R
111	9.84	1351	642	47.62	20.44
200	11.39	644.3	553	49.3	9.46
220	16.21	376.7	731		18.91
311	19.11	390	800	52.15	2.12
222	19.99	107.9	733	53.9	8.07
400	23.26	42.59	644	54.48	7.96
331	25.48	113.9	820		7.96
420	26.2	100.7	660	56.88	3.81
422	28.92	64.77	822		7.61
333	30.86	16.21	555	58.74	2.49
511		48.64	751		14.92
440	33.94	16.16	662	59.37	7.43
531	35.73	52.42	840	61.99	7.41
442	36.32	24.57	753	64.06	15.03
600		6.14	911		7.51
620	38.63	19.42	842	64.78	15.15
533	40.34	16.65	664	67.81	8
622	40.9	15.88	931	70.32	17.18
444	43.15	4.46	844	75.27	10.68
551	44.82	11.95	755	79.15	13.93
711		11.95	771		13.93
640	45.38	11.56	933		13.93

$$M = \frac{B(\sin^2 \theta)}{\lambda^2} \quad 2.40$$

and

$$B = 8\pi^2(\mu_s)^2 \quad 2.41$$

Where:

I_{α}^{hkl} = the integrated density of the hkl plane in the α -phase,

K = a constant that depends upon the instrumentation geometry and radiation but is independent of the sample.

R_{α}^{hkl} = based on basic principles, R can be calculated. It depends upon the phase composition, crystal structure, the Bragg angle and interplanar spacing. Table 2.5 and Table 2.6 show the R values for the ferrite and γ phases respectively.

V_{α} = the α -phase volume fraction,

μ = the steel's linear absorption coefficient,

I_0 = intensity of the incident beam,

e = charge of the electron

m = mass of the electron

A = cross-sectional area of the incident beam,

c = velocity of the light,

λ = the incident radiation's wavelength,

r = radius of the diffractometer

v = the unit cell's volume,

p = multiplicity factor of the (hkl) reflecting plane,

$|F|^2$ = structure factor times its complex conjugate,

LP = Lorentz Polarization factor,

2α = the diffraction angle of the monochromator crystal

e^{-2M} = Debye-Waller or temperature factor

θ = Bragg angle,

μ_s = the atoms' mean displacement from mean position, in a direction perpendicular to the diffracting plane.

Equation 2.34 applies to ferrite or martensite and austenite. The equation can be written for any pair of the austenite and ferrite hkl peaks:

$$\frac{I_{\alpha}^{hkl}}{I_{\gamma}^{hkl}} = \left(\frac{R_{\alpha}^{hkl}}{R_{\gamma}^{hkl}} \right) \left(\frac{V_{\alpha}}{V_{\gamma}} \right) \quad 2.42$$

when there are two phases in a steel

$$V_{\alpha} + V_{\gamma} = 1 \quad 2.43$$

then

$$V_{\gamma} = \frac{\frac{I_{\gamma}}{R_{\gamma}}}{\frac{I_{\alpha}}{R_{\alpha}} + \frac{I_{\gamma}}{R_{\gamma}}} \quad 2.44$$

If there are numerous peaks of the ferrite or martensite and austenite, the volume fraction of the austenite can be calculated as

$$V_{\gamma} = \left(\frac{1}{p} \sum_{j=1}^p \frac{I_{j\gamma}}{R_{j\gamma}} \right) / \left[\left(\frac{1}{q} \sum_{i=1}^q \frac{I_{i\alpha}}{R_{i\alpha}} \right) + \left(\frac{1}{p} \sum_{j=1}^p \frac{I_{j\gamma}}{R_{j\gamma}} \right) \right] \quad 2.45$$

Although this method was developed for polycrystalline samples with a random orientation, Dickson [22] demonstrated that the technique was equally applicable to materials with a preferred orientation as long as the proper number of the peaks was considered.

CHAPTER 3 Aims of the Study

As is clear from Chapter 2, AHSSs have revolutionised the automotive industry by allowing lightweight vehicles to be designed with lower greenhouse emissions, enhanced fuel efficiency, and improved crashworthiness. In particular, the MMn-AHSSs have emerged as promising materials for automotive applications due to their excellent strength, ductility, and formability. As summarised, there are scientific, technological, and environmental imperatives for research that explores the relationships between the TMP, and the resulting microstructure and properties in MMn-AHSS to fully explore their potential. This was the high-level goal of this thesis.

Of the various TMP routes applied to MMn-AHSSs, chapter 2 reveals a paucity of published research on the combination of cold rolling with annealing. This set the scene for the specific aims of this thesis, which were as follows.

1. To combine CALPHAD methods, empirical calculations of the M_s temperature and DSC experiments to explore the effect of alloying a base Fe-C-Mn AHSS composition with Al on phase composition. Our emphasis was on the effects of Al on phase fractions and transformation temperatures, given the potential of this element to further light-weight the steel and its capacity as a ferrite stabiliser.
2. To investigate the effect of increasing the initial dislocation density ahead of intercritical annealing via an additional cold rolling step to the as-received base FeCMn AHSS. The concept here was to understand the changes in microstructure and properties when there is an enhanced driving force for the $\alpha' \rightarrow \alpha + \gamma$ phase transformation during intercritical annealing.
3. To explore the effect of the nature of the martensite in the initial microstructure prior to intercritical annealing. Specifically, we generated starting microstructures such that

~70% of the martensite was generated by transformation from quenching from the austenite, and ~30% was transformed via deformation of the retained austenite.

4. To assess the impact of the TSTMP on microstructure and mechanical properties. Specifically, we applied a second stage of cold rolling and subsequent intercritical anneal after generating the initial dual phase $\alpha+\gamma$ microstructure. This part of the study also examined the effect of the rolling direction during the second stage. Rolling was applied in both the transverse direction (TD) and in the initial rolling direction (RD). The assessment of the TSTMP was the major work of the thesis.

CHAPTER 4 Experimental Methods

In this thesis, the microstructure and mechanical properties of the alloys were investigated after different TMPs to determine the relationship between the steel microstructure and properties. This chapter describes details of the different thermomechanical routes applied. It also describes the methodology for handling and preparing the samples, and subsequent specimen preparation for the variety of microstructural techniques. The tensile and hardness mechanical tests procedures are also described in detail.

4.1 Materials

The chemical compositions of the alloy steels used in this study are listed in Table 4.1 and Table 4.2. These compositions were determined through Leco Combustion, inductively coupled plasma atomic emission spectroscopy (ICP-AES) and Optical Emission Spectrometer (OES) methods by Spectrometer Service Pty. Ltd.

A standard melting, casting, and rolling technique provided by China Steel Corporation's R&D Department (Taiwan) was used to prepare all the alloy steels in the form of the fully martensitic cold-rolled steel sheets. Alloy steels were prepared by vacuum induction melting and cast into ingots. The ingots were heated at 1100 °C for 1 h, which was followed by a hot-rolling process from 25 mm down to 3.5 mm in thickness with a finishing temperature of 750 °C, after which the steel was cooled in the furnace to room temperature. The light optical microstructure of the FeCMnAl and FeCMn alloy steels is provided in Figure 4.1 after hot rolling.

The microstructure of the hot-rolled steels contained α' and retain γ phases. It can be seen in Figure 4.1 that FeCMnAl steel did show the grain boundary in light optical microscopy image due to adding Al. This final cold rolling step was accomplished by performing over eight passes at room temperature after pickling and removing the oxide layer from the steel sheets. After cold rolling, both alloy steels had a martensitic microstructure. The as-received (AR)

cold-rolled FeCMn steel for this study had a thickness of 1.4 mm, while the AR cold-rolled FeCMnAl steel was available in two different thicknesses of 1.8 mm and 1.0 mm. The rolling history of the FeCMn and FeCMnAl alloy steels is presented in Figure 4.2.

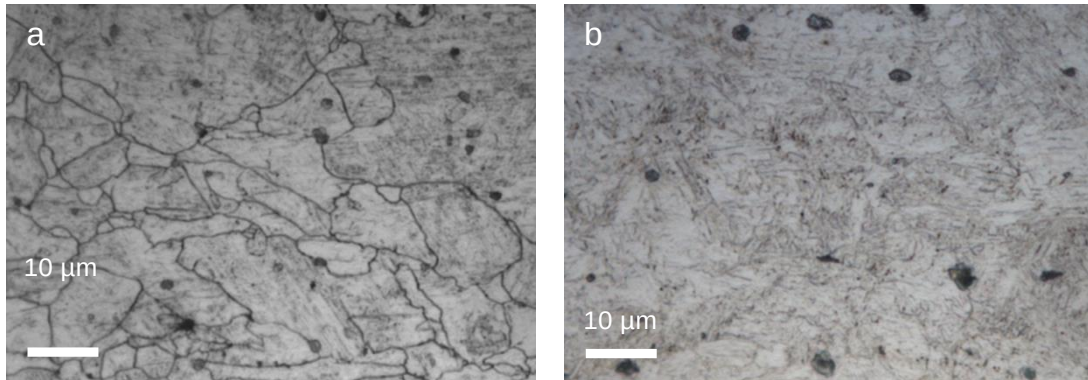


Figure 4.1. Light optical microscopy image of the (a) FeCMn steel and (b) FeCMnAl steel after hot rolling and furnace cooling. The microstructure contains α' and retained γ phases. The grain boundaries of the initial γ phase can be seen in FeCMn steel.

4.2 Thermomechanical Processing

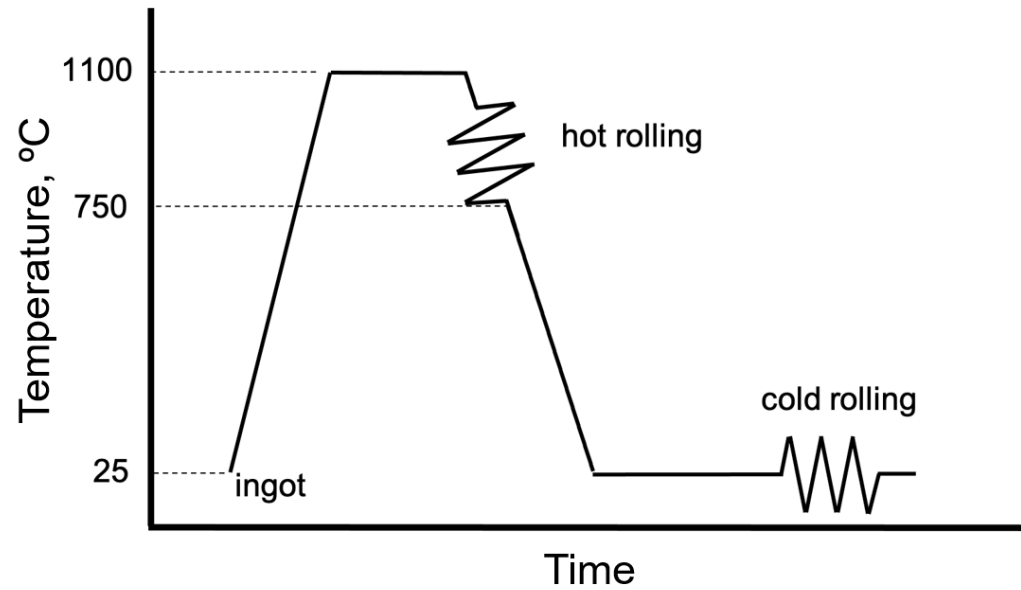
Schematic diagrams of the TMPs applied in this research are described in Figure 4.3 and Figure 4.4.

Table 4.1. FeCMn steel chemical composition.

	Fe	C	Mn	Al	Si	Mo	Cr	P	S	N
wt.%	Bal.	0.068	11.30	0.004	0.33	0.28	0.03	0.01	0.01	0.003
at.%	Bal.	0.31	11.4	0.008	0.67	0.16	0.032	0.018	0.018	0.01

Table 4.2. FeCMnAl steel chemical composition.

	Fe	C	Mn	Al	Si	Mo	Cr	P	S	N
wt.%	Bal.	0.068	11.30	1.10	0.34	0.28	0.03	0.01	0.01	0.003
at.%	Bal.	0.31	11.35	2.24	0.66	0.16	0.032	0.02	0.016	0.01

**Figure 4.2.** The rolling history of the FeCMn steel and FeCMnAl alloy steels.

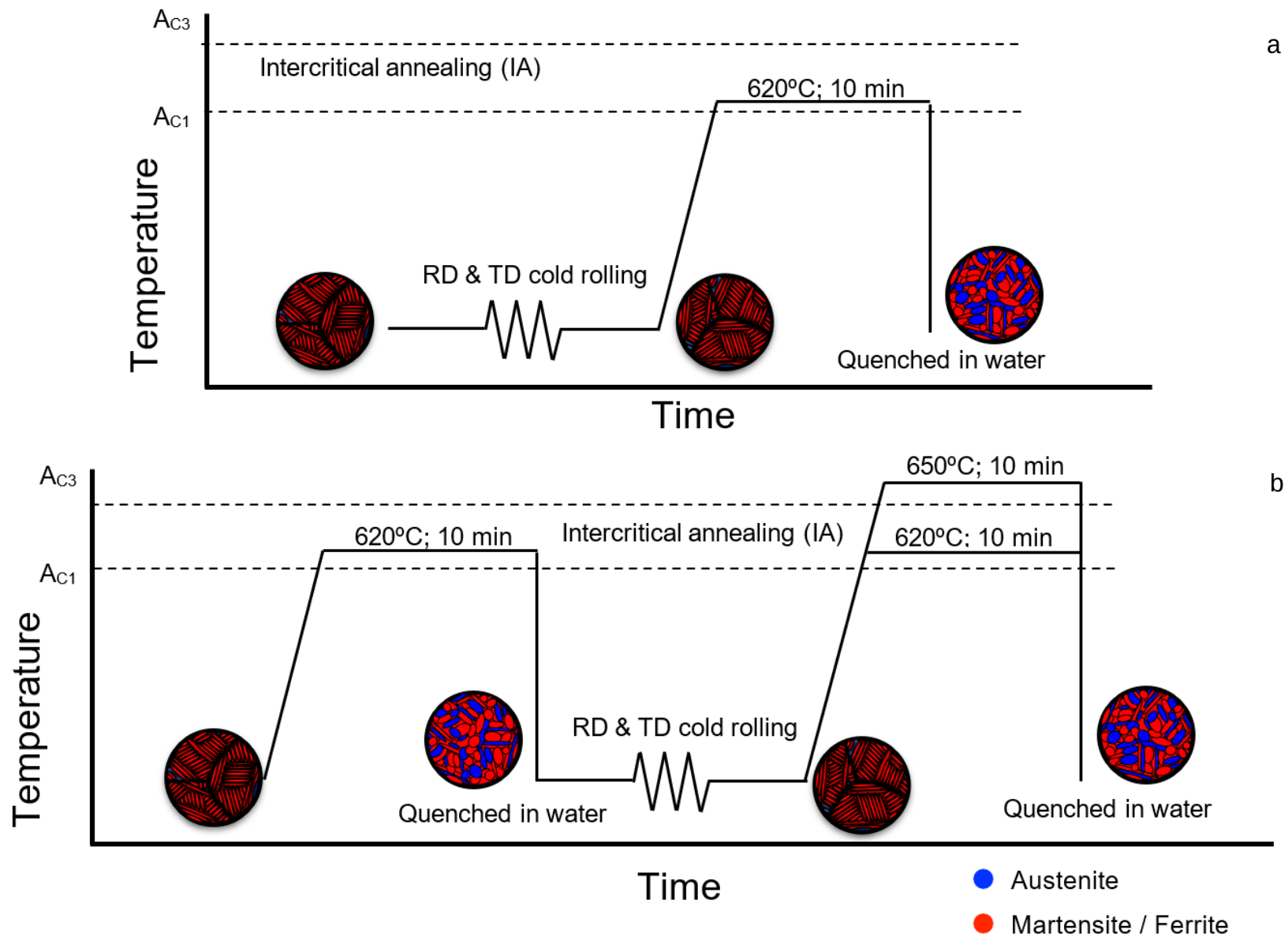


Figure 4.3. Schematic diagram of the (a) SSTMP and (b) TSTMP of the FeCMn steel.

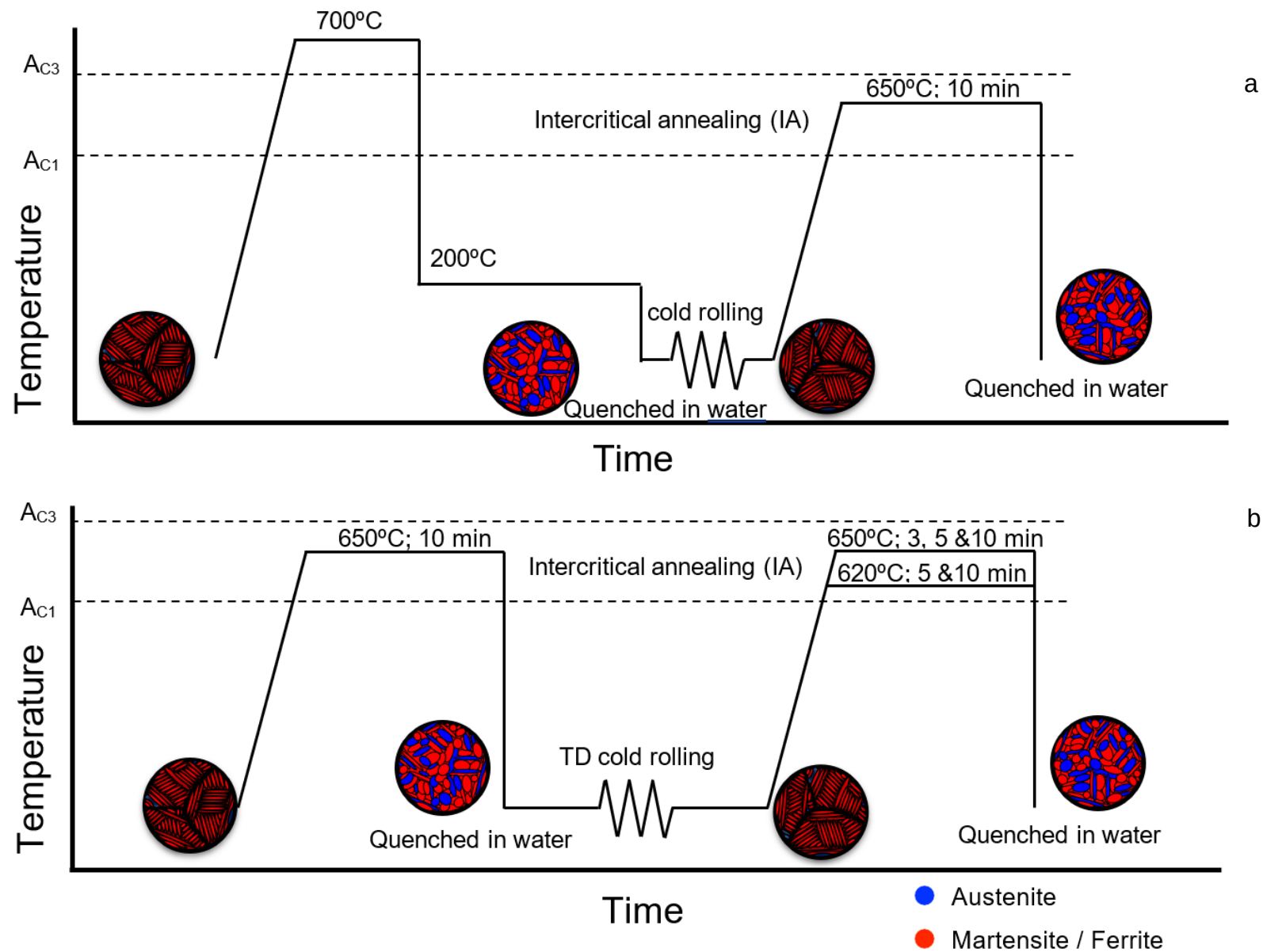


Figure 4.4. Schematic diagram of the (a) TSTMP-1 and (b) TSTMP-2 of the FeCMnAl steel.

4.2.1 Cold Rolling

Different rolling machines were used to decrease the thickness of the samples. These machines were located at the University of Sydney and the University of Wollongong (Figure 4.5).

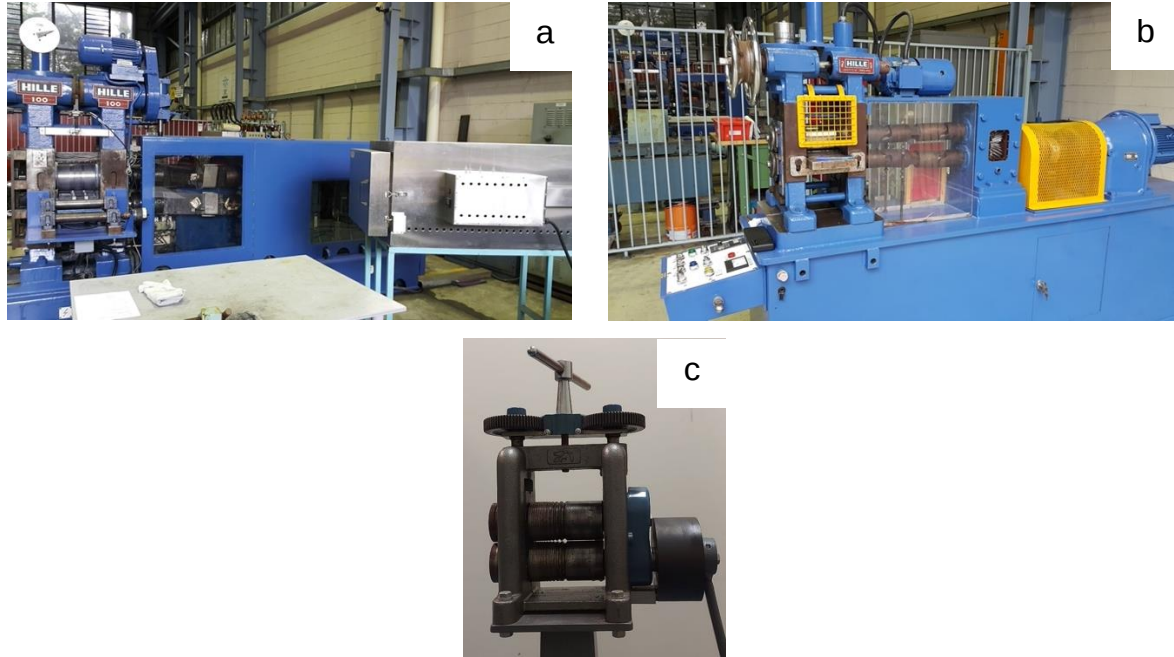


Figure 4.5. Rolling machines were used in this study (a) 100 tonnes rolling machine (b) 25 tonnes rolling machine (c) rolling machine for soft materials or low thickness reduction.

For the second stage of the TMP, two different rolling directions were applied: longitudinal rolling according to the first rolling direction (RD) and transverse rolling according to the first rolling direction (TD) (Figure 4.6).

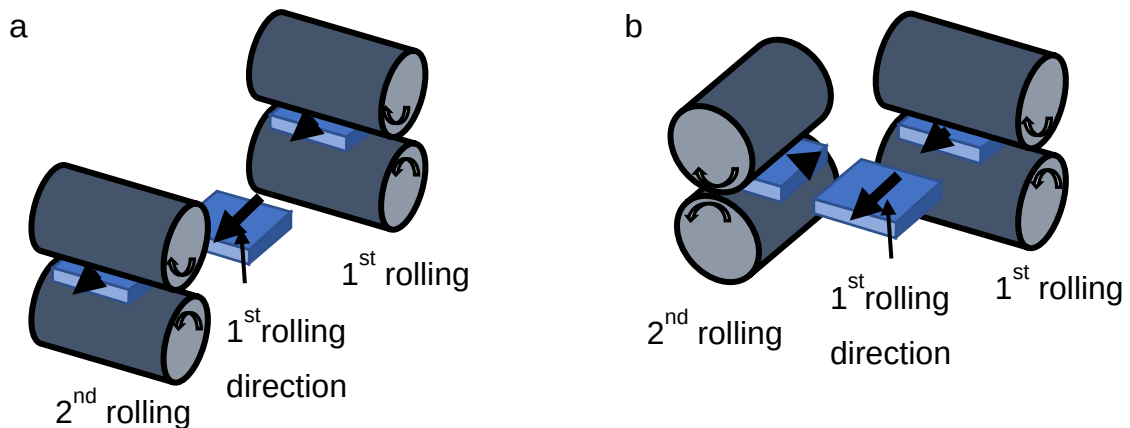


Figure 4.6. (a) RD and (b) TD cold-rolling at the second stage of the TMP.

4.2.2 Heat Treatment

A salt bath was used for the heat treatment of these alloys. The heating rate of the salt bath was 35 ± 3 °C/sec. The samples were wrapped in copper foil before soaking in the salt bath to prevent oxidation and decarburisation. As Figures 4.3 and 4.4 show, the samples were either quenched into the water or oil at 200 °C followed by quenching into water.

Table 4.3. Heat treatment conditions used in this study.

Alloy	Temperature (°C)	Time (min)
FeCMn	620	10
	650	10
FeCMnAl	620	5, 10
	650	3, 5, 10
	700	3

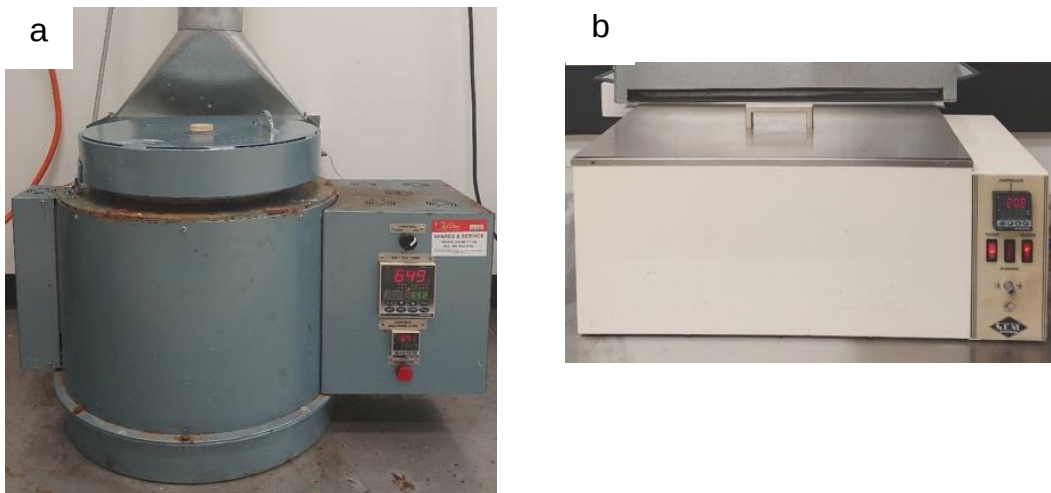


Figure 4.7. Heat treatment furnaces (a) the salt bath furnace for heat treatment at 600 °C - 700 °C (b) the oil bath furnace for quenching to 200 °C.

4.2.3 Differential Scanning Calorimetry

The DSC is a technique for measuring the heat flow in and out of a sample over time or as a function of the temperature while the sample is subjected to controlled temperatures. The method can be utilized to determine material properties such as glass transition temperature, melting point, crystallization, specific heat capacity, cure process, purity, oxidation behaviour, and thermal stability.

DSC was used in this study to find phase transformation temperatures for designing heat treatment of the alloys like α to γ phase transformation temperature. DSC – NETZSCH STA 449 F3 was located at the University of Sydney.



Figure 4.8. DSC – NETZSCH STA 449 F3 at the University of Sydney.

4.3 Sample Preparation

A variety of the samples were prepared following heat treatment or rolling steps, as shown in Figures 4.3 and 4.4. The samples were used to evaluate the effects of the TMPs on the alloys' microstructure and mechanical properties.

4.3.1 Transmission and Scanning Electron Microscopies Specimens

The TEM and SEM-TKD samples were thin, electron-transparent foils. Electropolishing was used to prepare thin foils for using the TEM and TKD methods. These foils were prepared from the middle of the sample thickness.

The first step involved mechanically polishing the specimens to a thickness of approximately 100 μm using sandpaper of 4000 grit. Then, a disc puncher was then used to punch 3 mm discs. The discs were made into electron-transparent thin foils via electrolytic polishing using a Struers TenuPol-5 twin-jet electropolisher with an electrolyte consisting of 6% perchloric acid and 94% acetic acid at a temperature of 15 $^{\circ}\text{C}$ and a voltage of 18 V. Immediately following electropolishing, the thin foil samples were washed with ethanol.

Precision ion polishing system (PIPS) was utilized for precision ion milling of the samples. A Gatan PIPS instrument equipped with a cold stage and an argon (Ar) gas chamber was used for this purpose. The instrument working parameters were 5 °C temperature, 3 keV ion gun beam energy, 2.5 rpm rotation speed, double ion gun modulator and 4° gun angle.

4.3.2 X-Ray Diffraction Specimens

For the preparation of the XRD sample, the specimens were mechanically polished with 320, 500, 1200, and 4000 grit sandpaper. Afterwards, they were electropolished with a 10% perchloric acid and 90% acetic acid solution using a Struers LectroPol-5 electropolishing machine. The polishing was conducted at a voltage of 28 V for one min at room temperature. In electropolishing, a final step was to clean the surfaces with ethanol to remove the electrolyte.

In addition, some samples were ion milled using a precision etching coating system (PECS) to study effect of the ion milling on the XRD results. A Gatan PECS II instrument was used for this purpose. The instrument working parameters were 25 °C temperature, 3 keV ion gun beam energy, 2.5 rpm rotation speed, double ion gun modulator and 4° gun angle.

4.3.3 Atom Probe Tomography Specimens

First, the samples were cut into approximately 500 μm x 500 μm cross sections and were about 15 mm in length (along the RD). Then, the preparation of the atom probe tips was accomplished by using the standard electropolishing technique with two stages, rough and fine polishing.

The rough polishing procedure was performed at room temperature and 20 volts with 25% perchloric acid in glacial acetic acid. After this stage, the needle-shaped specimens had a tip radius of the about two μm . In the next step, an electrolyte containing 2% perchloric acid in n-butoxyethanol was used to perform the fine polishing. After this stage, a needle-shaped specimen would have a tip radius of fewer than 100 nanometers.

4.3.4 Tensile Test Specimens

Engineering stress-strain curves derived from uniaxial tensile testing were used to determine the tensile properties of the alloys. The testing was performed according to ASTM E8 and AS 1391 standards, providing values for the yield strength (YS), ultimate tensile strength (UTS), elongation at fracture and work hardening rate. The test coupons were prepared in the largest size possible for a given experimental workflow and for a given TMP. The size of the test coupons was identical for each TMP.

The tensile test specimens were prepared in two directions: parallel and transverse to the rolling direction. These were designated as the RD and TD samples, respectively. The test coupons were machined using electrical discharge machining (EDM).

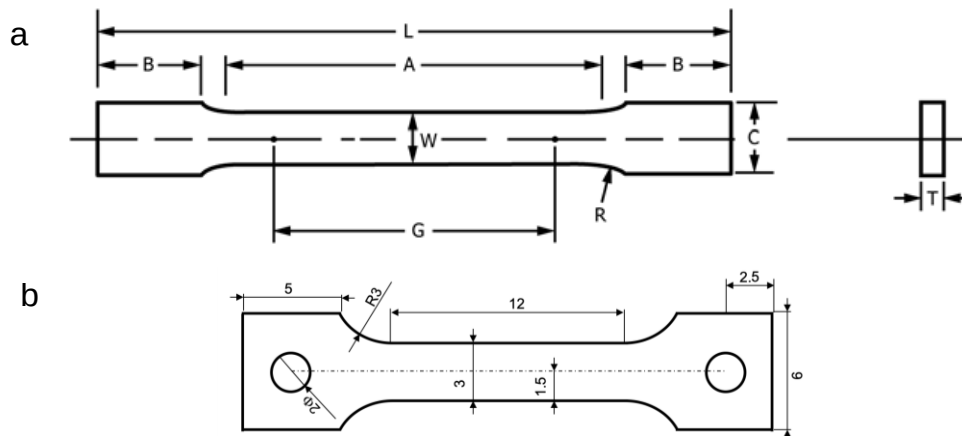


Figure 4.9. Tensile test specimen (a) ASTM E8/E8M – 15a subsize tensile test specimen [379] and AS 1391 - 2007 [380] (b) fabricated at the University of Sydney.

Table 4.4. Tensile test specimen dimensions.

	Dimensions (mm)		
	ASTM E8/E8M	AS 1391 - 2007	Designed
G (Lo): Gauge length	25.0 ± 0.1	12	10
W (b): Width	6.0 ± 0.1	3 ± 0.2	3
T (a): Thickness			
R: Radius of the fillet, min	6	3	3
L (Lt): Overall length, min	100		27
A (Lc): Length of the reduced section, min	32	18	12
B (Gripped ends): Length of the grip section, min	30		5
C: Width of the grip section, approximate	10		6

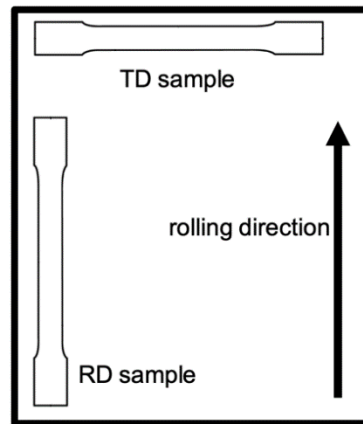


Figure 4.10. The RD and TD tensile test specimens.

4.4 Microstructural Characterisation Methods

Different techniques were employed to characterise the microstructure of the alloy, including SEM-TKD, TEM, XRD and APT. This section presents a detailed explanation of these methods.

4.4.1 Scanning Electron Microscopy - Transmission Kikuchi Diffraction

To evaluate the microstructure of the samples, SEM-TKD was used [381, 382]. In this method, the sample is placed perpendicular to the electron beam, and the EBSD detector is located beneath it, as depicted in Figure 4.11. This configuration enables the acquisition of the diffraction patterns from the bottom of thin foil, reducing the issue of the overlapping grains that can arise when applying TEM to nano and ultrafine structures.

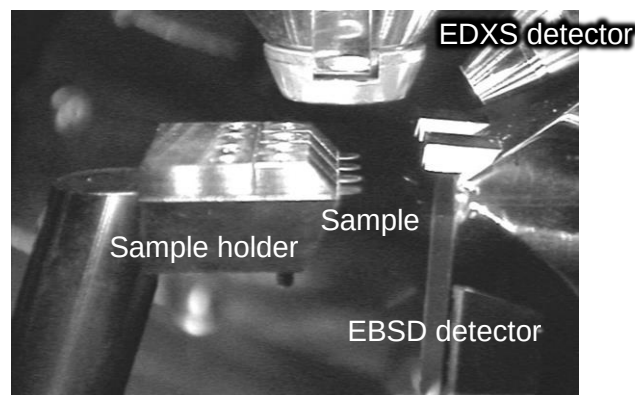


Figure 4.11. SEM-TKD setup.

These patterns are indexed to determine the phase of the material. Due to the development of the emission gun technology and its subsequent increase in spatial resolution,

TKD has three times better spatial resolution than the EBSD. TKD can be used to measure metals and alloys with ultra-fine and nano grain size. [381].

The TKD microstructural characterisations were carried out on a Zeiss ULTRA SEM equipped with an EBSD and an energy-dispersive X-ray spectroscopy (EDXS) detectors. All TKD assessments were performed using high-current mode and the beam energy was 30 kV (Figure 4.12). Oxford Instrument Aztec software was used to process TKD, EBSD and EDXS maps.



Figure 4.12. Zeiss ULTRA plus SEM.

A working distance of approximately 7 mm was used and an aperture size of 120 μm was selected for all TKD experiments. The indexing of individual Kikuchi diffraction patterns up to nine bands were utilized in this study. The γ , α/α' , and HCP reflectors were 42, 43, and 38.

The HKL Channel-5 software and its modules: Tango and Salsa software were used to analyse the TKD data [383–386]. The wild spikes (incorrectly indexed) were removed, and zero solutions (unindexed) were found by cyclically extrapolating from eight to six neighbours [387].

TKD maps were used to analyse microscopic features such as grain size, phase fraction, grain boundary misorientation, internal misorientation, and texture.

4.4.2 Transmission Electron Microscopy

In this study a JEOL JEM-1400 TEM operating at 120 kV with “Gatan in-situ heating double-tilt holder” and FEI Themis-Z Double-corrected S/TEM operating at 300 kV were used to assess the samples.

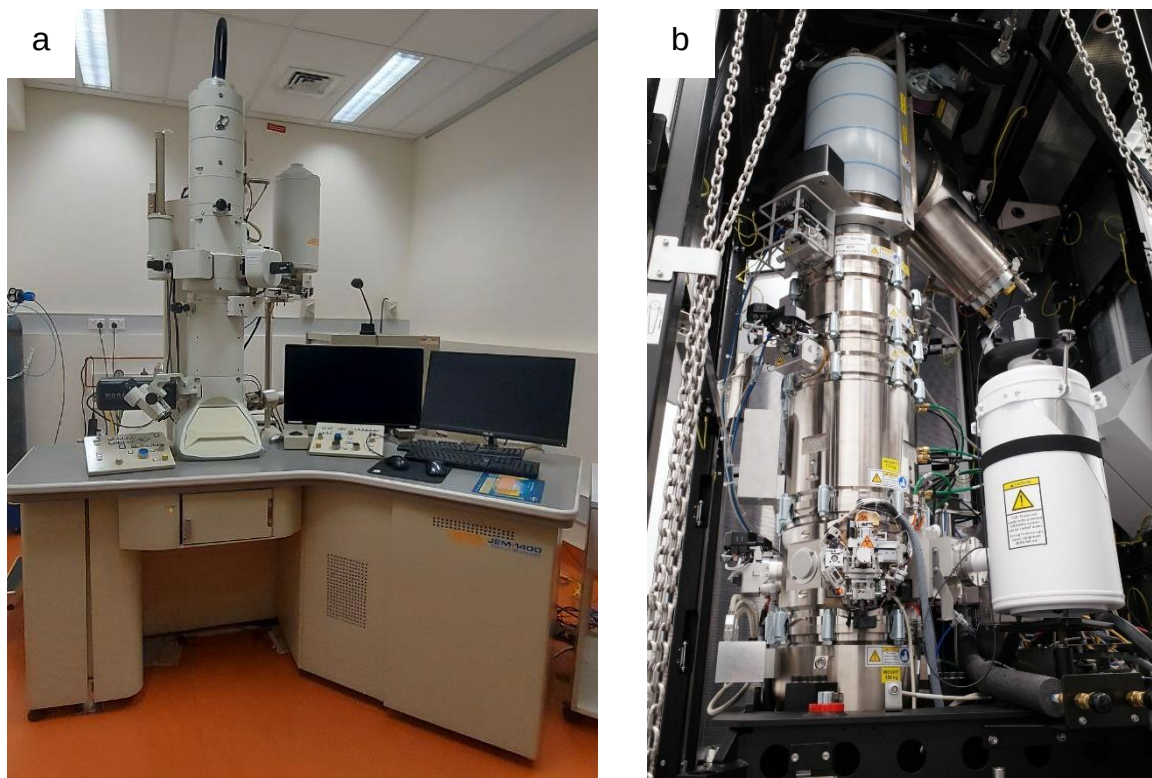


Figure 4.13. (a) JEOL JEM-1400 TEM and (b) FEI Themis-Z Double-corrected S/TEM.

4.4.3 X-Ray Diffraction

In this study, the XRD method was used to calculate the phase fraction of the γ and α/α' . XRD measurements were made with a Stoe Stadi P (RHS) diffractometer equipped with a Molybdenum $k\alpha$ radiation and the wavelength of 0.70930 nm.

In this experiment, the XRD diffractometer generator was operated at a voltage of 50 kV and a current of 40 mA at room temperature. Scans were conducted in the 2θ regions between 19° and 80° . The selection of this specific range was made to ensure that there were enough diffraction peaks of both γ and α/α' , with four γ peaks (111), (200), (220), and (311) and three α peaks (110), (200), and (211), to properly account for the impact of the texture. The scanning step size, θ , was 0.005° , and the dwell time was a minimum of 120 Sec.

During XRD measurements, samples were rotating around the Z-axis (normal direction (ND) of the rolled sheet). Each experiment was repeated at least twice. The results of the each sample were combined, the background was subtracted, and then, it was smoothed using the WinX^{POW} software [388]. The normalised XRD data were presented in the figures included in this study.

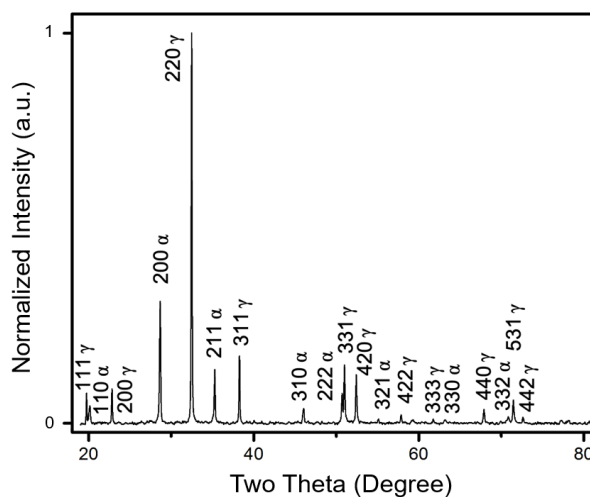


Figure 4.14. XRD peaks position of the γ and α phases.

4.4.4 Atom Probe Tomography

For this research, two Local Electrode Atom Probes (LEAP) in voltage mode were used; LEAP 3000 Si and LEAP 4000X Si.

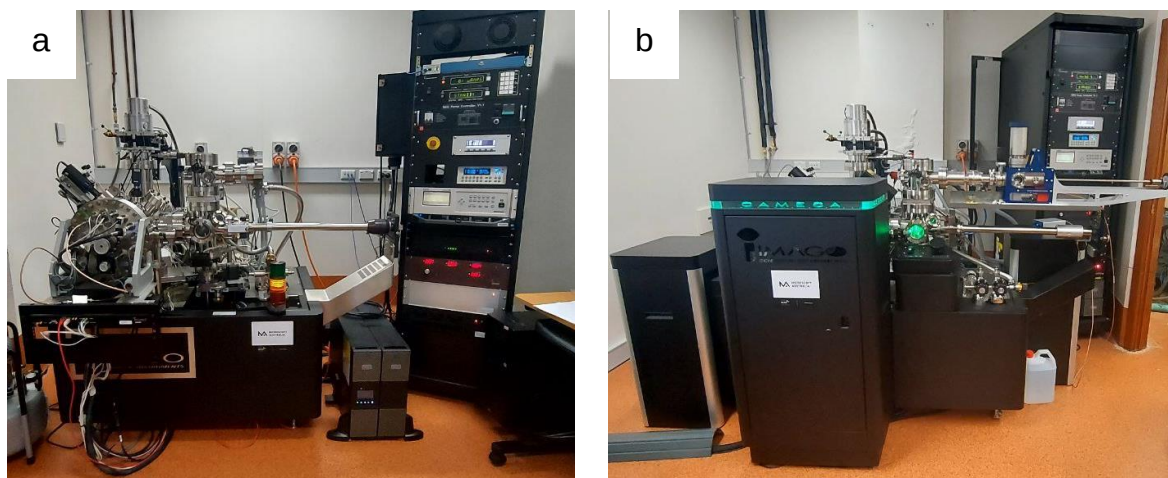


Figure 4.15. (a) LEAP 3000 Si and (b) LEAP 4000 Si.

The APT experiments conducted in this research used specimens maintained at a cryogenic temperature of 40 °K, under a vacuum level of less than 1×10^{-10} Torr. The detection rate of the atoms per pulse was maintained ~ 0.005 , with a pulse fraction of 20% and a voltage pulse repetition rate of 200 kHz.

Upon completion of the data collection through the atom probe, the reconstructions, visualizations, and chemical analyses were carried out utilizing the IVAS and AP Suite

software packages. The mass-to-charge ratio and corresponding ion species identified from the mass spectra obtained during APT experiments are summarised in Table 4.5.

Table 4.5. Ion species and mass-to-charge ratios of the APT experiments.

Mass to charge ratio (Da)	Ion Species	Mass to charge ratio (Da)	Ion Species
6	$^{12}\text{C}^{2+}$	26.5	$^{53}\text{Cr}^{2+}$
9	$^{27}\text{Al}^{3+}$	27	$^{54}\text{Fe}^{2+}$
10.3	$^{31}\text{P}^{3+}$	27.5	$^{55}\text{Mn}^{2+}$
12	$^{12}\text{C}^{+}$	28	$^{56}\text{Fe}^{2+}$
13.5	$^{27}\text{Al}^{2+}$	28.5	$^{57}\text{Fe}^{2+}$
14	$^{28}\text{Si}^{2+}$	29	$^{58}\text{Fe}^{2+}$
14.5	$^{29}\text{Si}^{2+}$	46	$^{92}\text{Mn}^{2+}$
15	$^{30}\text{Si}^{2+}$	47	$^{94}\text{Mn}^{2+}$
15.5	$^{31}\text{P}^{2+}$	47.5	$^{95}\text{Mn}^{2+}$
18	$^{12}\text{C}_3^{2+}$	48	$^{96}\text{Mn}^{2+}$
18.5	$^{56}\text{Fe}^{3+}$	48.5	$^{97}\text{Mn}^{2+}$
24	$^{12}\text{C}_2^{+}$	49	$^{98}\text{Mn}^{2+}$
25	$^{50}\text{Cr}^{2+}$	50	$^{100}\text{Mn}^{2+}$
26	$^{52}\text{Cr}^{2+}$		

4.5 Mechanical Testing Procedures

Uniaxial tensile and hardness tests were conducted to evaluate the mechanical properties of the alloys.

4.5.1 Uniaxial Tensile Test

For the tensile tests, the Instron 3366 with 10 kN load cell at room temperature was utilized. Based on the sample size, two types of the extensometers were used: 10 mm and 25 mm. An engineering strain rate of 0.001 Sec^{-1} was applied to the gauge region of the sample during the tensile test. In order to conduct tensile tests on the small tensile specimens (Figure 4.9(b)), a holder was designed which is shown in Figure 4.16. A minimum of two samples were tested for each heat treatment condition.

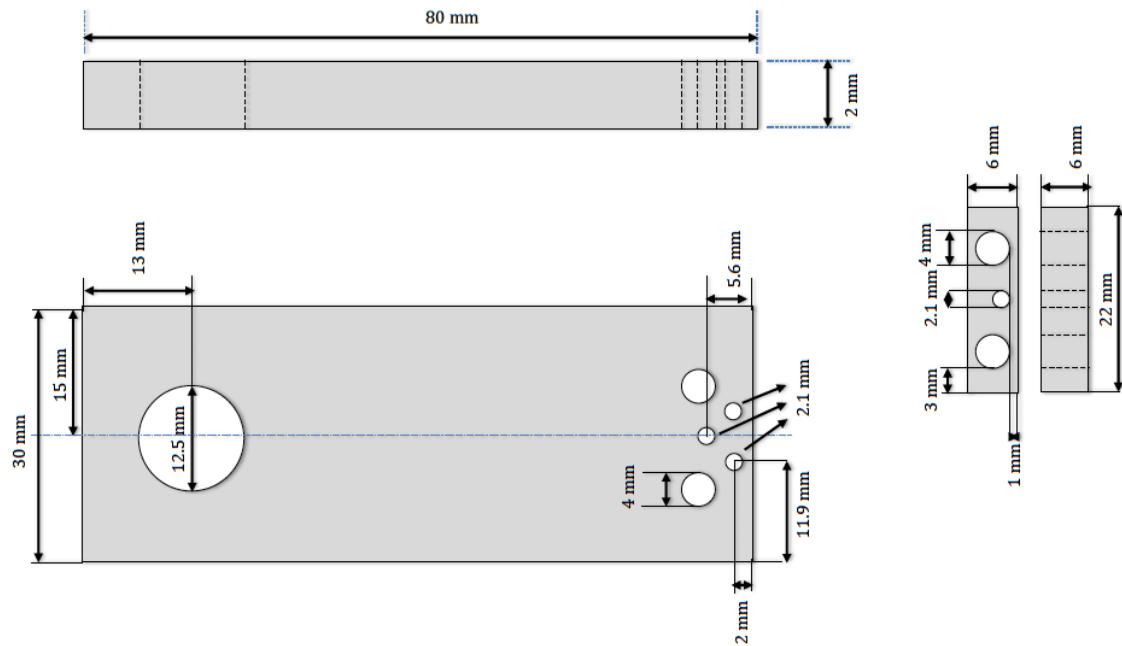


Figure 4.16. The designed holder for testing tensile test specimens.

4.5.2 Hardness Test

To prepare the samples for hardness measurement, their surfaces were mechanically ground using 4000 grit sandpapers.

LECO Macro Vickers Hardness Tester (LV700 AT) was used to measure the hardness of the material under a load of 10 kg and for a dwell time of 15 Sec. A mean value was derived as a result of the minimum 5 vickers hardness measurements. The distance between the identifications and the testing method was in accordance with ASTM E92 – 82 [389].

PART II

RESULTS

&

DISCUSSION

This part encompasses chapters 5 to 9, which present the results, along with chapter 10, dedicated to the in-depth discussion of these outcomes.

CHAPTER 5 Thermodynamics and Differential Scanning Calorimetry Results

5.1 Equilibrium Phase Compositions

Equilibrium phase compositions at various temperatures ranging from 400 to 800 °C were calculated using Thermo-Calc 2019b software with TCFE9 database [390]. The analysis revealed that the main phases present were γ and α . At these temperatures, four different precipitate phases may occur, namely HCP (A3), $M_{23}C_{63}$ (D8₄), M_6C (E9₃) and M_7C_3 (D10₁). The compositions of the FeCMn and FeCMnAl alloys were examined, and the variations in C, Mn, Si, Mo, and Al concentrations in the γ and α phases are summarised in Figure 5.1 and Figure 5.2, respectively.

Upon comparison of the α phase of the FeCMn and FeCMnAl alloys, it was observed that at elevated temperatures, the concentrations of the C, Mo, and Al atoms increased, whereas Si concentration decreased in both alloys. In the case of the FeCMn alloy, the concentration of the Mn decreased with increasing temperature. However, for the FeCMnAl alloy, the concentration of the Mn initially decreased and then increased with increasing temperature.

The C, Mn, Si and Mo have the same trends in the γ phase of the FeCMn and FeCMnAl alloys. By increasing the temperature, the phase fraction of the γ increases and its Mn concentration decreases. Si in γ phases of both alloys increases to the maximum amount at 446 °C and 456 °C and then decreases to the 0.33 and 0.34 wt.% amounts, in FeCMn and FeCMnAl alloys, respectively.

The Al content in the γ phase increases by increasing the temperature to the maximum of 1.1 wt.%. The C content in the γ increases to the maximum amount at 501 and 509 °C then

decreases to 0.68 and 0.66 wt.% in FeCMn and FeCMnAl alloys, respectively. Above these temperatures, the M_7C_3 ($D10_1$) phase was not stable.

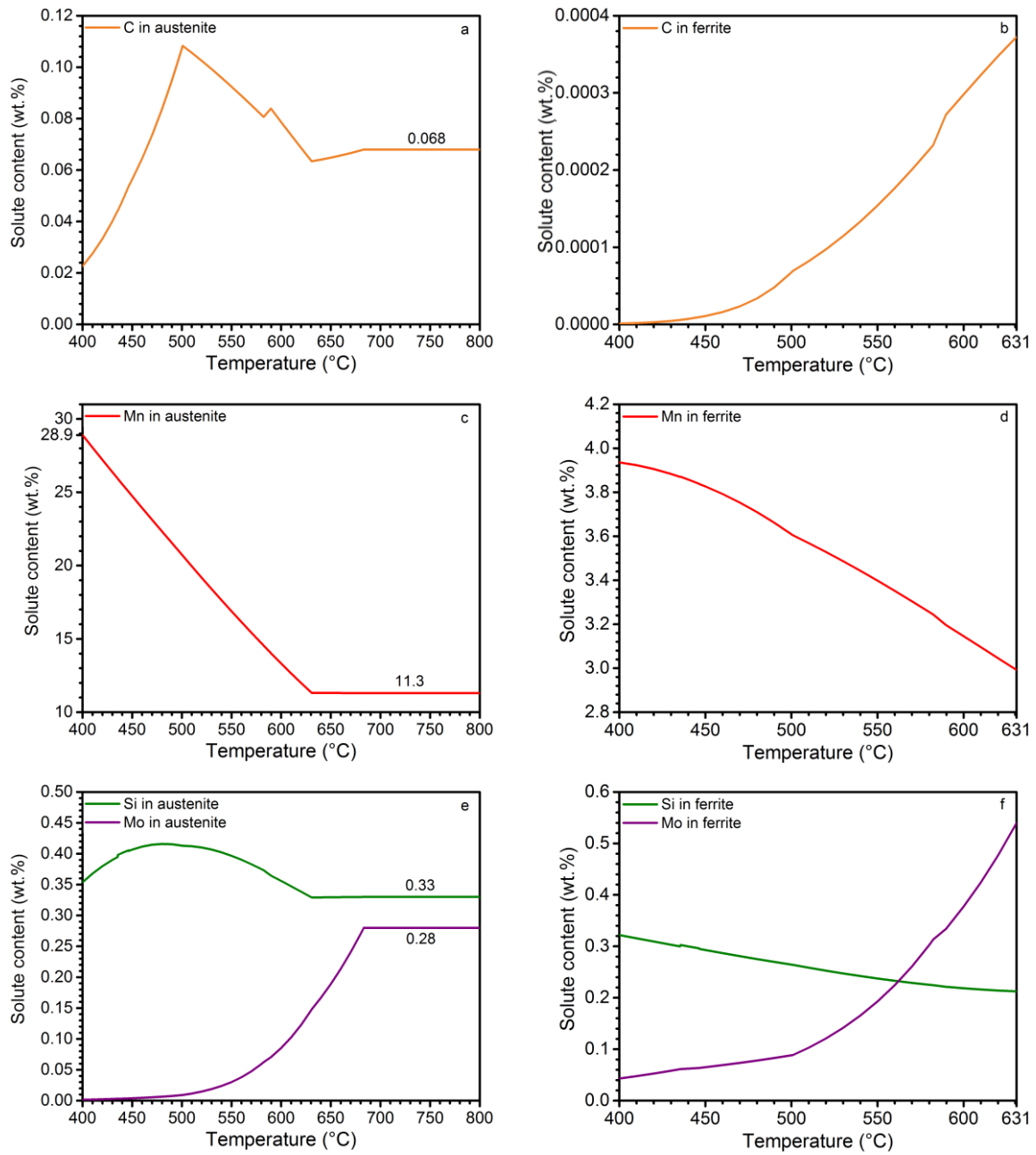


Figure 5.1. Equilibrium phase compositions of the FeCMn steel at different temperatures for the γ phase (a) C, (c) Mn, (e) Mo and Si, and for the α phase (b) C, (d) Mn and (f) Mo and Si, computed by Thermo-Calc 2019b software with TCFE9 database.

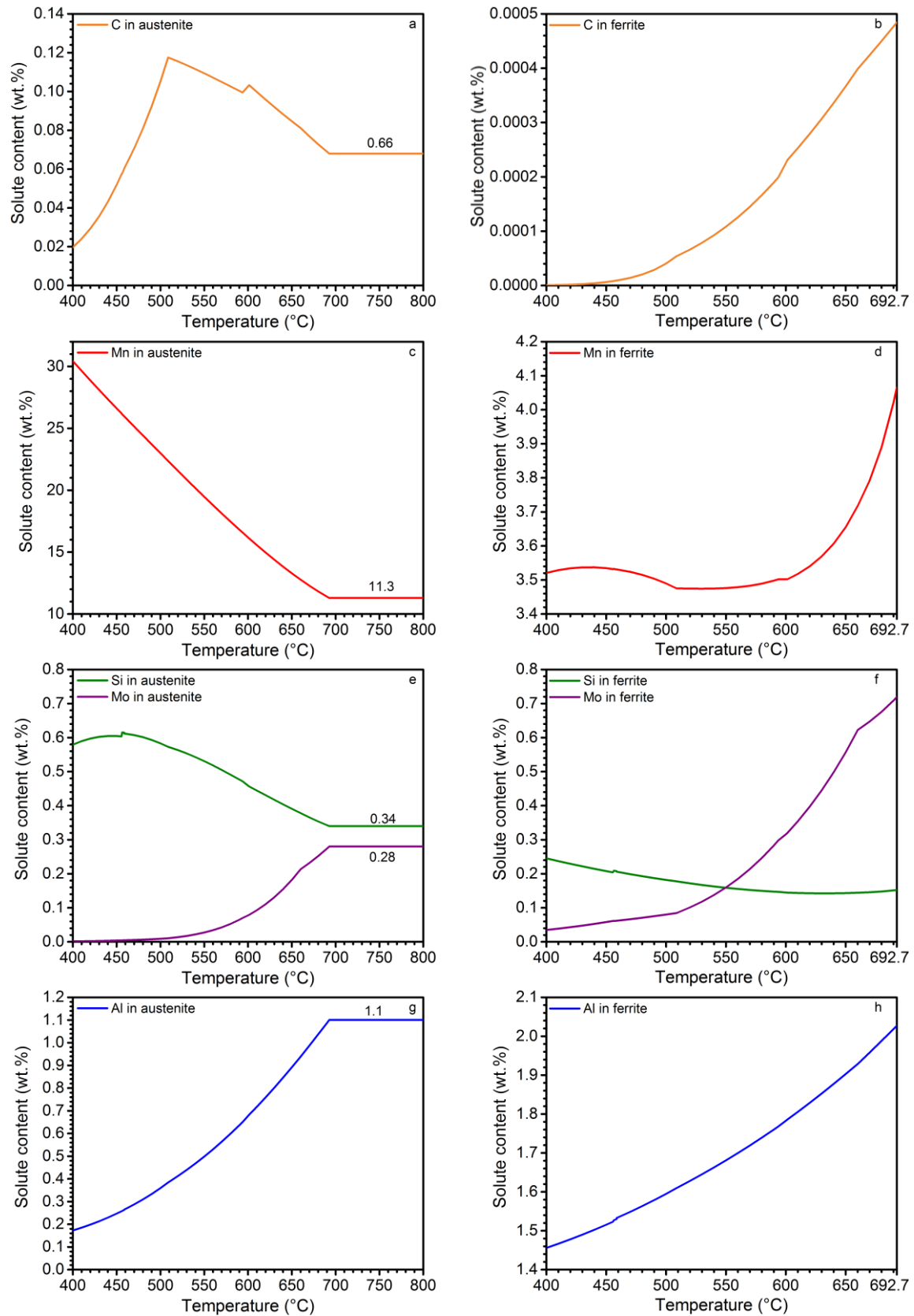


Figure 5.2. Equilibrium phase compositions of the FeCMnAl steel at different temperatures for the γ phase (a) C, (c) Mn, (e) Mo and Si, (g) Al, and for the α phase (b) C, (d) Mn and (f) Mo and Si and (h) Al, computed by Thermo-Calc 2019b software with TCFE9 database.

5.2 Equilibrium Phase Fractions

The equilibrium phase fractions in the FeCMn and FeCMnAl alloys, calculated using Thermo-Calc 2019b software with TCFE9 database, are plotted in Figures 5.3 and 5.4, respectively, for temperatures ranging from 400 to 800 °C. It is evident from these Figures that the addition of Al to the alloy increased the maximum stability of the α phase from 630.7 °C to 692.7 °C.

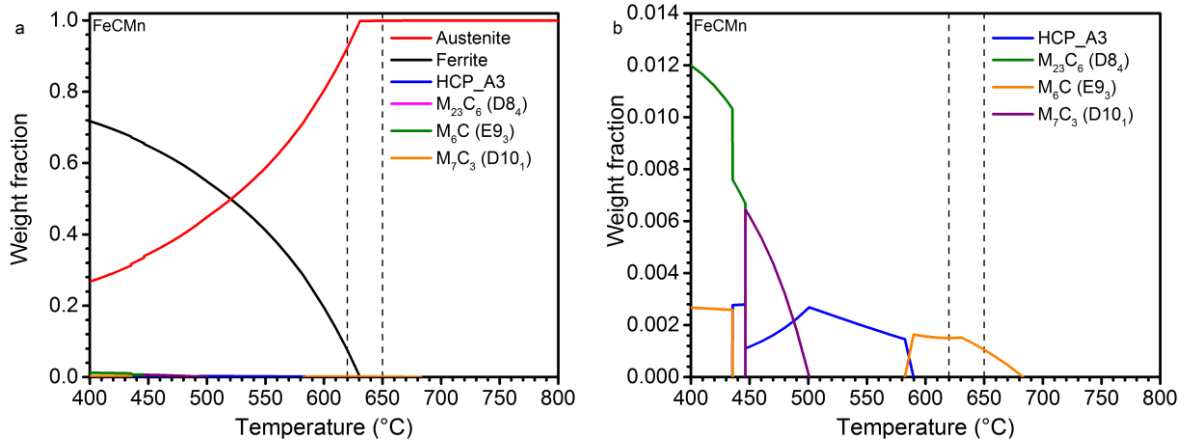


Figure 5.3. The equilibrium phase fractions of the FeCMn alloy steel at different temperatures and (b) zoomed-in view, computed by Thermo-Calc 2019b software with TCFE9 database.

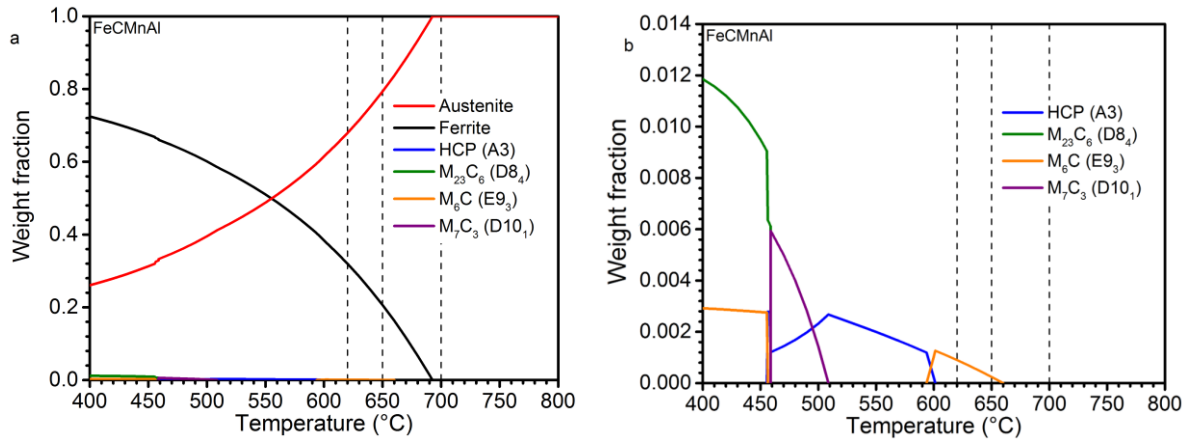


Figure 5.4. (a) The equilibrium phase fractions of the FeCMnAl alloy steel at different temperatures and (b) zoomed-in view, computed by Thermo-Calc 2019b software with TCFE9 database.

Adding Al changes the stability of other phases as well. The M_6C (E9₃) phase is stable below 435 °C and between 582 and 683 °C in FeCMn alloy while by adding Al, it is stable below 456 °C and between 593 and 660 °C in FeCMnAl alloy.

The stability of M_7C_3 ($D10_1$) phase from between 446 and 501 °C to between 458 and 509 °C by adding Al. The $M_{23}C_{63}$ ($D8_4$) phase is stable below 446 °C and 458 °C in FeCMn and FeCMnAl alloys, respectively. The HCP ($A3$) phase is stable between 435 and 590 °C and between 455 and 601 °C in the FeCMn and FeCMnAl alloys, respectively.

Table 5.1. Equilibrium phase fractions of the FeCMn alloy at annealing temperatures.

Temperature, °C	Phase weight fraction, %		
	α	γ	M_6C ($E9_3$)
650	0.0	99.9	0.1
620	7.5	92.3	0.2

Table 5.2. Equilibrium phase fractions of the FeCMnAl alloy at annealing temperatures.

Temperature, °C	Phase weight fraction, %		
	α	γ	M_6C ($E9_3$)
700	0.0	100	0.0
650	20.7	79.3	0.0
620	32.0	67.9	0.1

Table 5.3. Equilibrium phase compositions of the FeCMn alloy steel at 620 °C and 650 °C temperatures.

Temp. °C	Phase	Content (at. %)					
		C	Al	Si	Mn	Mo	Fe
620	α	0.002	0.031	0.426	3.094	0.277	96.170
	γ	0.317	0.006	0.668	12.111	0.0708	86.827
	M_6C ($E9_3$)	14.296	0	1.960	0	50.602	83.744
650	γ	0.300	0.008	0.652	11.439	0.017	49.398
	M_6C ($E9_3$)	14.295	0	1.756	0	50.162	87.584

Table 5.4. Equilibrium phase compositions of the FeCMnAl alloy steel at 620 °C, 650 °C and 700 °C temperatures.

Temp. °C	Phase	Content (at. %)					
		C	Al	Si	Mn	Mo	Fe
620	α	0.001	3.712	0.278	3.529	0.227	92.253
	γ	0.438	1.544	0.842	14.954	0.063	82.159
	M_6C ($E9_3$)	14.297	0	3.778	0	49.482	32.443
650	α	0.002	3.862	0.279	3.643	0.318	91.896
	γ	0.387	1.814	0.764	13.277	0.104	83.654
	M_6C ($E9_3$)	14.296	0	3.326	0	49.250	33.128
700	γ	0.305	2.193	0.651	11.065	85.628	0.158

Table 5.1 and Table 5.2 summarise the equilibrium phase fractions of FeCMn and FeCMnAl alloys at various annealing temperatures. Additionally, Table 5.3 and Table 5.4 provide the equilibrium phase compositions of FeCMn and FeCMnAl alloys, respectively, at different annealing conditions.

5.3 Martensite-Start Temperature Calculations

Calculating the α' -Ms can be conducted by using the equilibrium γ composition. The following equation was used by Barbier in his study [135]:

$$M_s^{\alpha_0}/^{\circ}\text{C} = 545 - 601.2(1 - \exp(-0.868[C])) - 34.4[\text{Mn}] - 13.7[\text{Si}] - 9.2[\text{Cr}] - 17.3[\text{Ni}] - 15.4[\text{Mo}] + 10.8[\text{V}] + 4.7[\text{Co}] - 1.4[\text{Al}] - 16.3[\text{Cu}] - 361[\text{Nb}] - 2.44[\text{Ti}] - 3448[\text{B}]$$

5.1

The chemical composition of alloying element X is [X] in weight percent. This equation was considered for an infinitely large γ grain size. Table 5.5 shows the calculated $M_s^{\alpha_0}$ for the equilibrium γ compositions at different temperatures. Based on the results calculated under equilibrium conditions using Equation 5.1, the FeCMnAl alloy exhibits a significantly lower $M_s^{\alpha_0}$ temperature compared to the FeCMn alloy, attributable to the influence of Al.

Table 5.5. $M_s^{\alpha_0}$ for the equilibrium γ at different temperatures.

Annealing Temperature ($^{\circ}\text{C}$)	$M_s^{\alpha_0}$ ($^{\circ}\text{C}$)	
	FeCMn	FeCMnAl
700		111.3
650	115.4	35.7
620	90.9	-26.6

According to equation 5.2 from Yang et al. [391] it is possible to calculate ε -Ms for the equilibrium γ compositions at different temperatures. Considering an infinitely large grain size for γ , this equation can be applied. Table 5.6 shows the calculated $M_s^{\varepsilon_0}$ temperature for equilibrium γ compositions after annealing at various temperatures. Upon annealing at 650 $^{\circ}\text{C}$, FeCMn and FeCMnAl alloys display very close $M_s^{\varepsilon_0}$ temperatures, whereas FeCMnAl alloy displays eight less $M_s^{\varepsilon_0}$ temperatures. As a result of the annealing at 620 $^{\circ}\text{C}$, FeCMnAl alloy's $M_s^{\varepsilon_0}$ is 23 $^{\circ}\text{C}$ lower than FeCMn alloy's.

$$M_s^{\varepsilon_0}/^{\circ}\text{C} = 303 \pm 8 - (489 \pm 31)[\text{C}] - (9.1 \pm 0.4)[\text{Mn}] - (17.6 \pm 2)[\text{Ni}] - (9.2 \pm 1)[\text{Cr}] + (21.3 \pm 2)[\text{Al}] + (4.1 \pm 1)[\text{Si}] - (19.4 \pm 5)[\text{Mo}] - (1 \pm 1)[\text{Co}] - (41.3 \pm 6)[\text{Cu}] - (50 \pm 18)[\text{Nb}] - (86 \pm 12)[\text{Ti}] - (34 \pm 10)[\text{V}] - (13 \pm 5)[\text{W}] \quad 5.2$$

Table 5.6. $M_s^{\varepsilon_0}$ for the equilibrium γ at different temperatures.

Annealing Temperature, $^{\circ}\text{C}$	$M_s^{\varepsilon_0}$, $^{\circ}\text{C}$	
	FeCMn	FeCMnAl
700		186.3
650	166.1	157.7
620	159.3	136.1

The relationship between γ grain size and M_s^0 temperature is illustrated in equation 5.3 by Yang and Bhadeshia [154].

$$M_s^0 - M_s^g = \frac{1}{b} \ln \left[\frac{1}{aV_\gamma} \left\{ \exp \left(-\frac{\ln(1-f_{M_s})}{m} \right) - 1 \right\} + 1 \right] \quad 5.3$$

Where:

M_s^0 is the martensite transformation-start temperature when V_γ is infinite,

M_s^g is the martensite transformation-start temperature for the finite V_γ ,

b is a constant and equals 0.2689 for α' [154] and 0.19 for ε [155],

a is a constant and equals 1 mm^{-3} [154],

V_γ is the average volume of the γ grains,

m is the aspect ratio of the martensite plate which is 0.05 for α' and 0.03 for ε [154, 155], and

f_{M_s} is 0.01 which is the offset used to determine martensite-start temperatures [154].

Equation 5.3 shows that $M_s^g \rightarrow M_s^0$ as $V_\gamma \rightarrow \infty$. In Figures 5.5 and 5.6, the $M_s^{\alpha'g}$ and $M_s^{\varepsilon g}$ temperatures for equilibrium γ compositions at different annealing temperatures are shown for FeCMn and FeCMnAl alloys, respectively. The Figures demonstrate that decreasing the γ grain size decreases the martensite-start temperature (M_s^g).

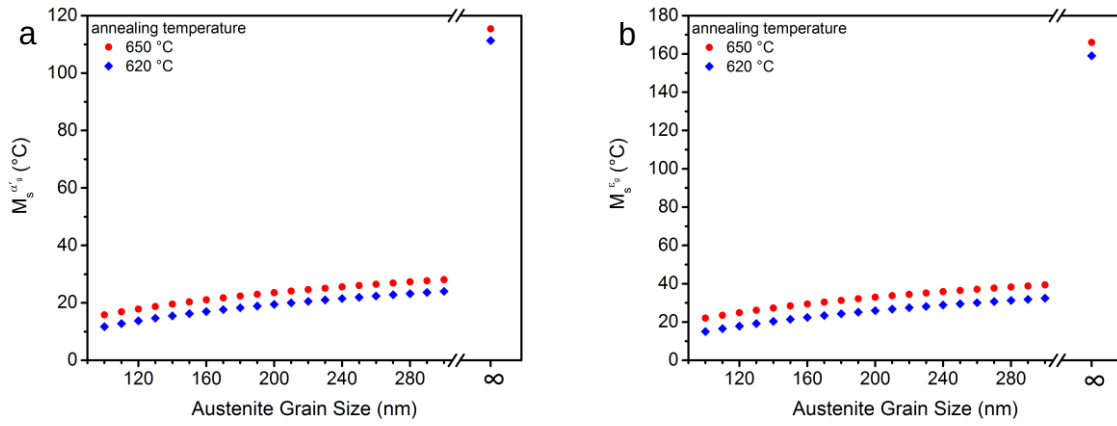


Figure 5.5. Calculated the M_s^g temperature using equilibrium γ composition at different IA temperatures as a function of the grain size (a) α' -martensite and (b) ϵ -martensite for the FeCMn alloy.

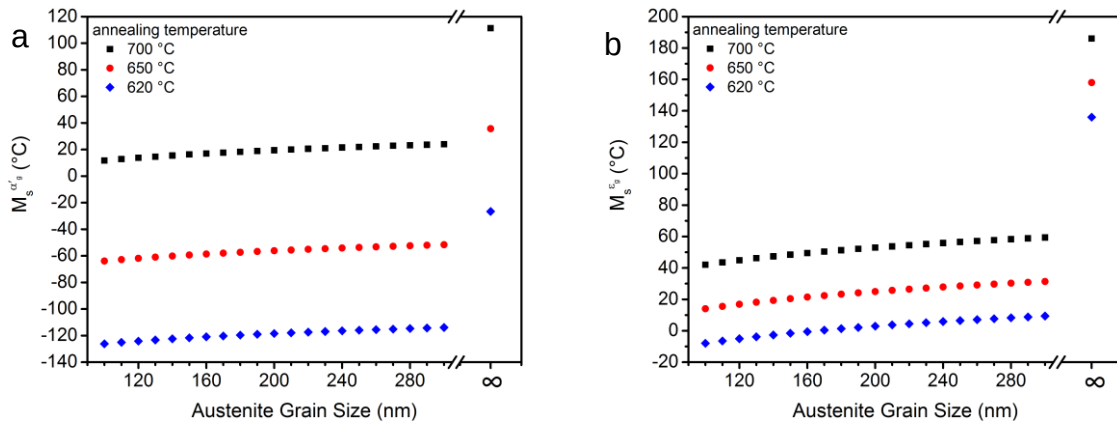


Figure 5.6. Calculated the M_s^g temperature using equilibrium γ composition at different IA temperatures as a function of the grain size (a) α' -martensite and (b) ϵ -martensite for the FeCMnAl alloy.

5.4 Differential Scanning Calorimetry Analysis

Table 5.7 illustrates the sample were analysed by DSC. Figures 5.8 and 5.9 show the DSC curves of the FeCMn and FeCMnAl alloys in different conditions. All the samples were in the cold rolled condition and the heating rate was 10 °C/min. Tables 5.7 and 5.8 illustrate α/α' to γ phase transformation endset and peak temperatures extracted from DSC curves of the FeCMn and FeCMnAl steels, respectively.

All specimens that underwent cold rolling during thermochemical processing exhibited lower peak and endset temperatures than the initial sample, except for the 0.95 mm FeCMnAl specimen. Among the specimens with the same amount of the thickness reduction, the specimen that was cold-rolled in the TD had a lower peak temperature. The second run of the DSC test did not reveal any significant differences between the specimens as shown in Table 5.8 and Table 5.9.

Table 5.7. The FeCMn and FeCMnAl specimens were analysed by the DSC.

Alloy	Initial thickness, mm	Heat treatment		Cold rolling (Thickness reduction)		Sample Name
		Temperature, °C	Time, min	Direction	%	
FeCMn	1.4	--	--	--	--	1.4 mm
		--	--	RD	50	0.7 mm, WPHT - Rolled RD
		--	--	TD	20	0.7 mm, WPHT - Rolled TD
		620	10	RD	50	0.7 mm, Rolled RD
		620	10	TD	50	0.7 mm, Rolled TD
FeCMnAl	1.8	--	--	--	--	1.8 mm
		650	10	TD	50	0.9 mm, Rolled TD
	1.0	--	--	--	--	1.0 mm
		700	3	RD	5	0.95 mm, Rolled RD

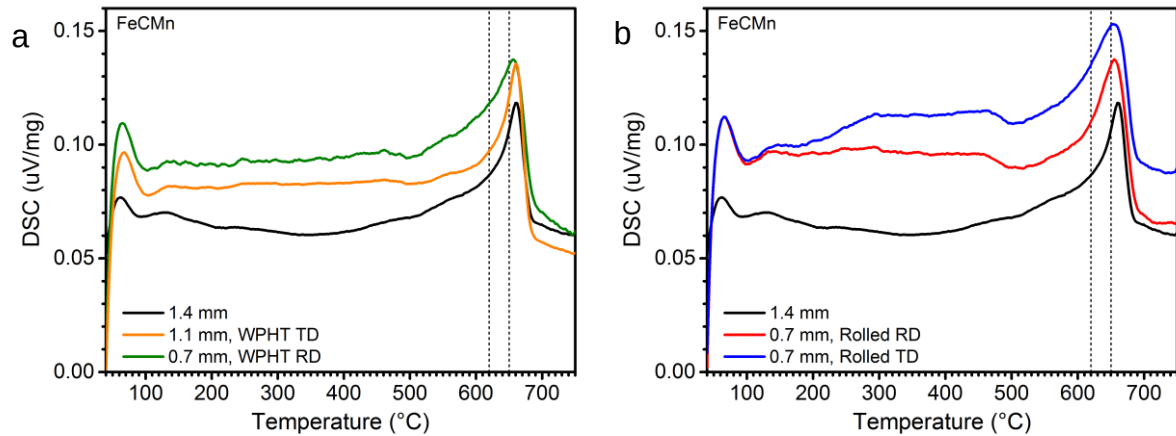


Figure 5.7. DSC curves of the different FeCMn specimens were subjected to the (a) SSTMP (Chapter 6) and (b) TSTMP (Chapter 8). The condition of the each sample was shown in Table 5.7.

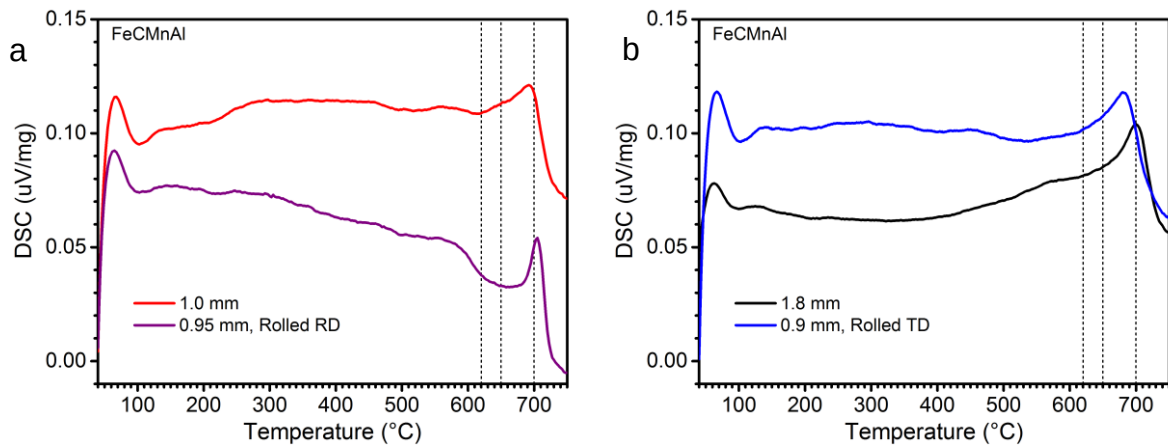


Figure 5.8. DSC curves of the different FeCMnAl specimens (a) with different source of the martensite microstructure (chapter 7) and (b) subjected to the TSTMP (Chapter 9). The condition of the each sample was shown in Table 5.7.

Table 5.8. α/α' to γ phase transformation endset and peak temperatures extracted from DSC curves of the FeCMn alloy specimens.

FeCMn samples	First run		Second run	
	Endset (°C)	Peak (°C)	Endset (°C)	Peak (°C)
1.4 mm	680.6	660.1	694.7	672.8
0.7 mm, Rolled RD	686.6	654.8	692.9	670.9
0.7 mm, Rolled TD	685.8	654.7	690.1	670.0
0.7 mm, WPHT-Rolled RD	680.9	656.6	693.7	672.7
1.1 mm, WPHT- Rolled TD	680.1	658.7	694.1	673.0

Table 5.9. α/α' to γ phase transformation endset and peak temperatures extracted from DSC curves of the FeCMnAl alloy specimens.

FeCMnAl samples	First run		Second run	
	Endset (°C)	Peak (°C)	Endset (°C)	Peak (°C)
1.8 mm	714.1	699.8	749.2	723.1
0.9 mm, Rolled TD	715.3	681.2	749.1	722.7
1.0 mm	721.6	690.8	745.1	724.7
0.95 mm, Rolled RD	722.5	703.9	745.5	723.2

5.5 Summary

In this chapter, we employed CALPHAD methods, empirical calculations of the M_s temperature, and DSC experiments to investigate the impact of alloying a base Fe-C-Mn AHSS composition with Al on phase composition. Our focus centered on assessing how Al influences phase fractions and transformation temperatures, with consideration for its potential to enhance the steel's lightweight properties and its role as a ferrite stabilizer. The results discussed in chapter 10.

CHAPTER 6 Effect of the Initial Dislocation Density on Microstructural Evolution during the Thermomechanical Process of the FeCMn Steel

6.1 Thermomechanical Process Procedure

The AR FeCMn alloy underwent both a one-step I and a SSTMP, as depicted in Figures 6.1(a) and 6.1(b), respectively. The initial microstructure of the AR specimen consisted entirely of α and α' phases. During the one-step IA of the FeCMn steel, the AR specimen was intercritically annealed at 620°C for 10 minutes and subsequently quenched in water.

For the SSTMP of the FeCMn steel, the AR sample underwent cold rolling (CR) to achieve a 50% reduction in thickness along the rolling direction (RD), labelled AR+CR(RD)50%, in one procedure, and a 20% reduction along the transverse direction (TD), labelled AR+CR(TD)20%, in another. Post cold rolling, these samples were annealed in the γ and α dual-phase region, as shown in Figure 6.1 (b), at 620°C for 10 minutes, followed by water quenching.

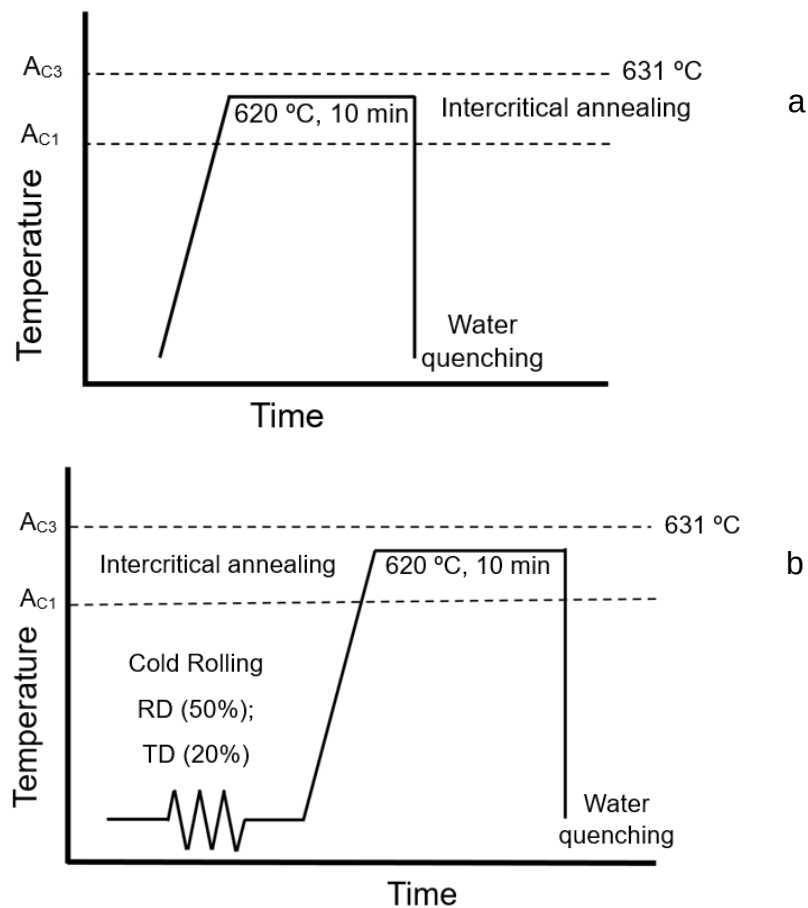


Figure 6.1. The schematic of the (a) one-step IA and (b) SSTMP for the FeCMn alloy.

6.2 Microstructure

The SEM-TKD maps were collected to determine the phase fraction, the average grain size, the boundaries between phases and grains, the strain distribution, and the texture of the phases.

6.2.1 Scanning Electron Microscopy Maps and Phase Volume Fractions

Figure 6.2 illustrates the SEM-TKD, image quality (IQ) phase maps of the AR, AR+CR(RD)50% and AR+CR(TD)20% samples which had close to 100% of the α/α' phases.

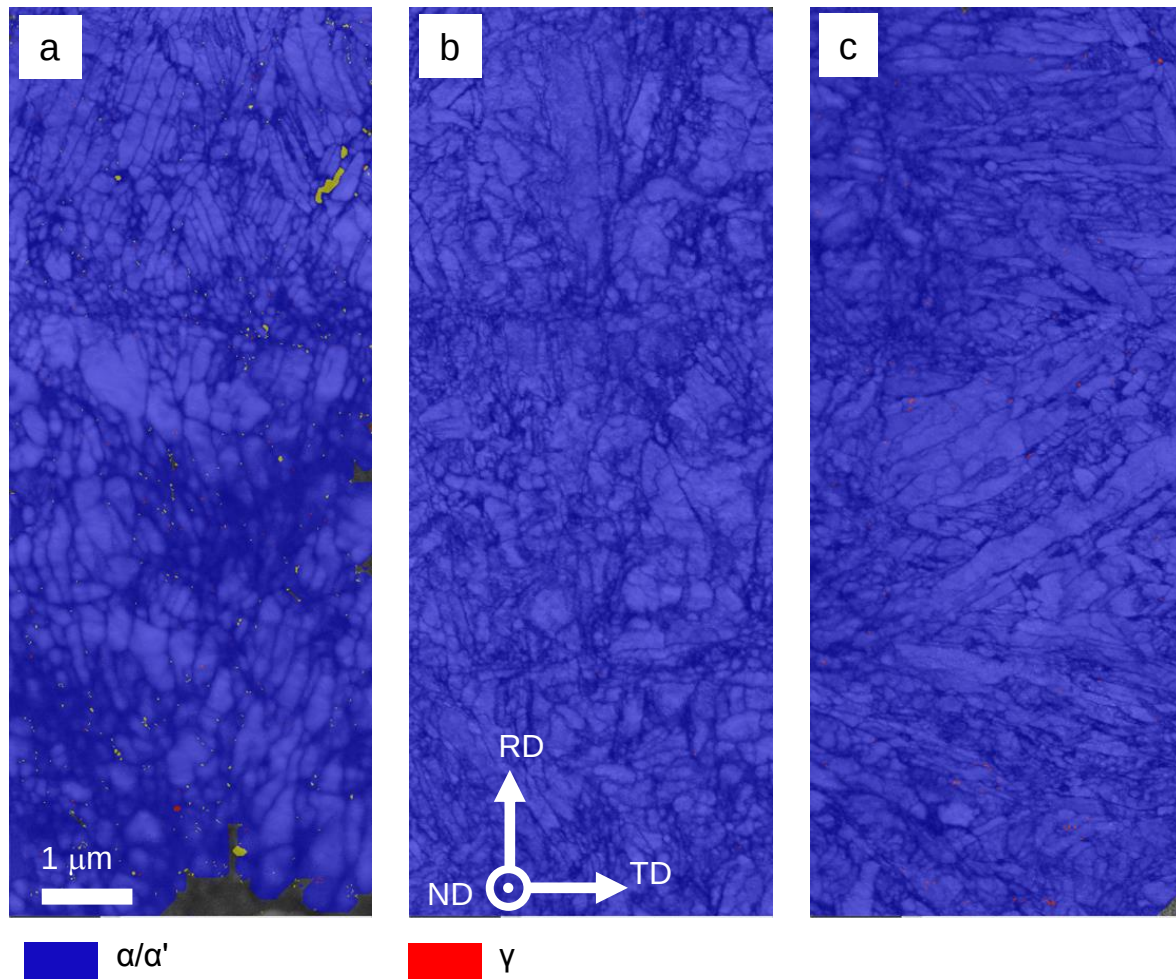


Figure 6.2. SEM-TKD, IQ-phase maps of the FeCMn steel samples (a) AR (b) AR+CR(RD)50% and (c) WPHT AR+CR(TD)20%. On the SEM-TKD maps, blue represents α/α' .

Figure 6.3 illustrates the SEM-TKD, IQ-phase maps of the AR, AR+CR(RD)50% and AR+CR(TD)20% samples after the IA at 620 °C for 10 min (one-step IA, SSTMP(RD) and SSTMP(TD), respectively). The γ phase volume fractions in the SSTMP(RD) and SSTMP(TD) samples were 70.3% and 69.8%, respectively.

Figure 6.4 depicts the effect of the sample condition on the phase volume fraction of the γ and α/α' . Figure 6.4 shows that the one-step IA sample had the γ phase with a volume fraction of the 62%. While in the SSTMP(RD) and SSTMP(TD) samples, these volume fractions were about 8% more.

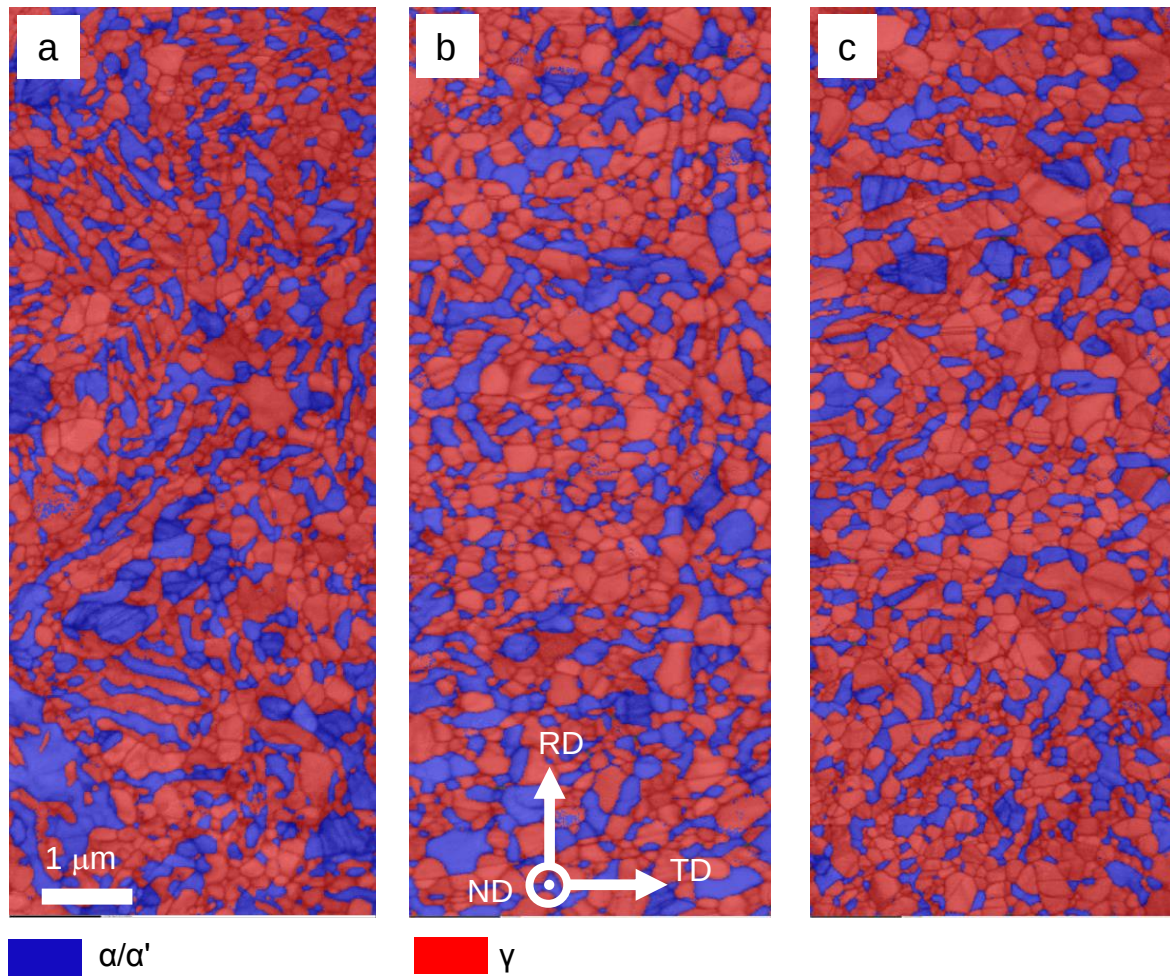


Figure 6.3. SEM-TKD, IQ-phase map FeCMn steel (a) AR, (b) AR+CR(RD)50% and (c) AR+CR(TD)20% after IA at 620 °C for 10 min. On the SEM-TKD maps, red represents γ , and blue represents α/α' .

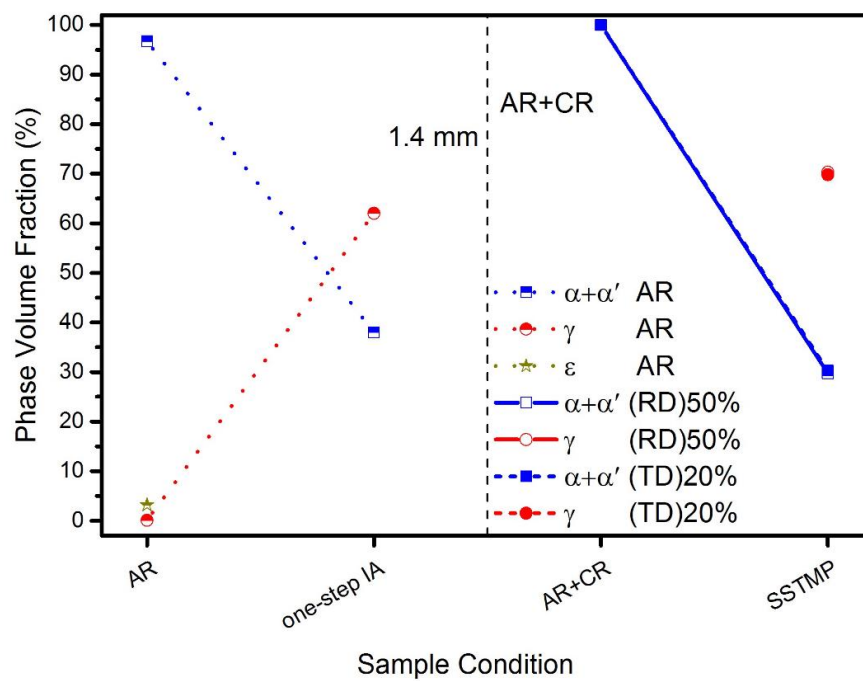


Figure 6.4. Effect of the sample condition on the phase volume fraction of the γ , α/α' , and martensite (ϵ) from SEM-TKD maps.

6.2.2 Grain Size

Figures 6.5 and 6.6 illustrates the effect of the cold rolling in RD and TD on the grain size distribution of the γ and α/α' . These figures do not show the γ phase of the AR+CR(RD)50% and AR+CR(TD)20% samples because the γ phase volume fraction was close to 0%.

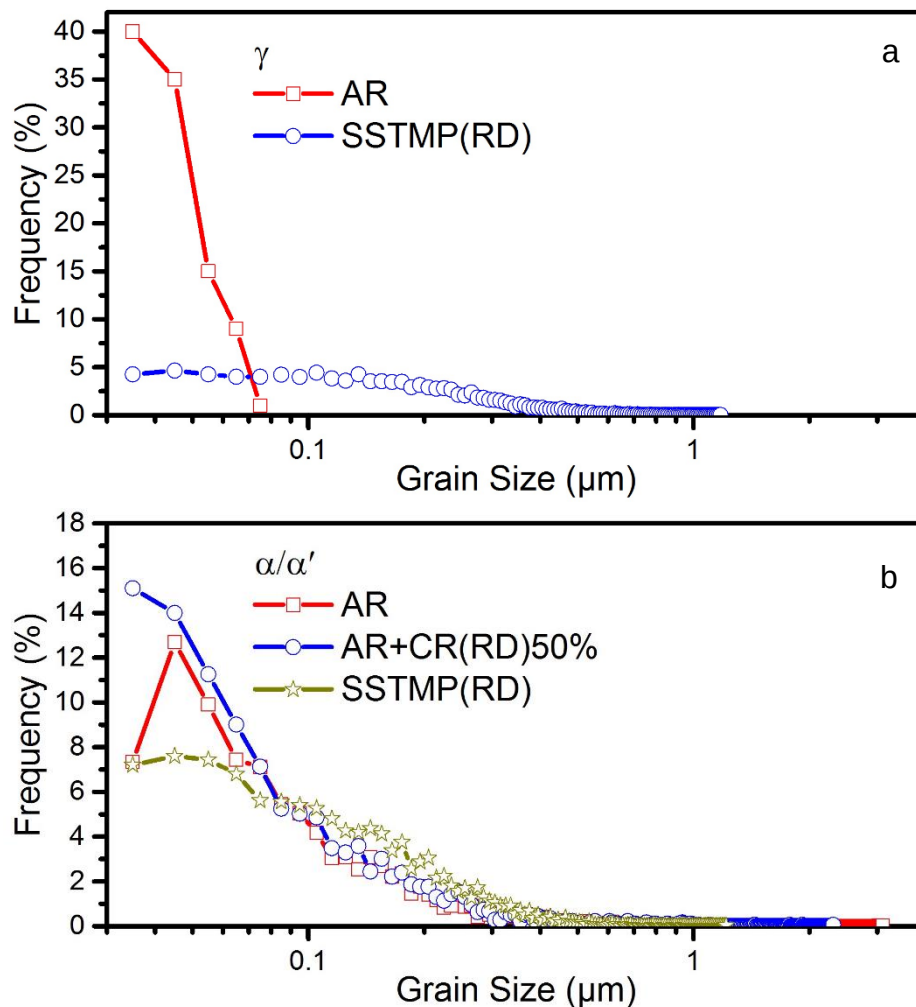


Figure 6.5. Effect of the rolling RD on the grain size distribution of the (a) γ and (b) α/α' from SEM-TKD maps.

As Figures 6.5 (a) and 6.6 (a) depict, in the AR sample, the occurrence of the nano-sized grains (grains ≤ 100 nm) of the γ phase was 100%. The γ grains nucleated and grew during the IA, and it resulted in 29.3% and 33.2% nano-size grains fraction in the SSTMP(RD) and SSTMP(TD), respectively, respectively.

The cold rolling deformed the α/α' grains and increased the dislocation density in them. This phenomenon resulted in the formation of the smaller α/α' grains. Figures 6.5 (b) and 6.6

(b) show that the fraction of the nano-size grains in the AR sample was 55%. The following RD or TD rolling increased the fraction to 66.8% and 76.9% respectively. Despite the fact, the thickness reduction in the RD cold rolling was 50% and, in the TD cold rolling was 20%, the TD cold rolling increased the nano-size grains' fraction more than the RD cold rolling.

During the isothermal annealing (IA), the α grains recrystallized, and the α' grains transformed into smaller γ grains, which subsequently affected the grain size distribution. The fraction of the nano-size grains in α/α' phase was 45.6% and 44.5% in the SSTMP(RD) and SSTMP(TD), respectively.

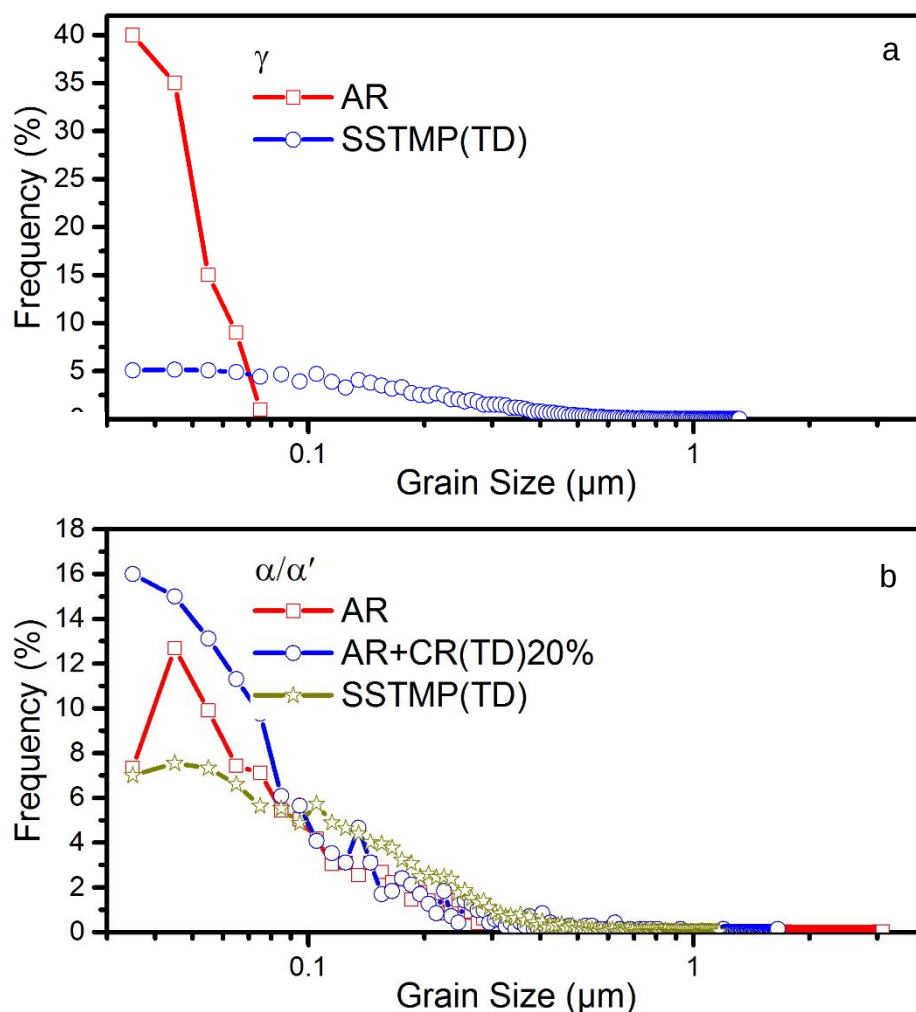


Figure 6.6. Effect of the rolling TD on the grain size distribution of the (a) γ and (b) α/α' from SEM-TKD maps.

Figure 6.7 shows the effect of the sample condition on the nano-size grains frequency of the γ and α/α' phases. It can be seen the α/α' phase in the one-step IA sample had a lower nano-size grains fraction than the SSTMP(RD) and SSTMP(TD) samples. For the γ phase, it was the reverse.

Figure 6.8 shows the effect of the sample condition on the average grain size of the γ and α/α' phases. The average grain size of the γ phase was 207.99 ± 0.03 nm and 211.27 ± 0.03 nm in the SSTMP(RD) and SSTMP(TD) samples, respectively. These amounts were very close to the average grain size of the γ phase in the one-step IA sample (214.52 ± 0.12 nm).

The average grain size of the α/α' phase was 170.74 ± 0.05 nm and 171.09 ± 0.04 nm in the SSTMP(RD) and SSTMP(TD) samples, respectively. These amounts were about 6% less than the average grain size of the γ phase in the one-step IA sample (180.68 ± 0.07).

Figure 6.9 shows the effect of the sample condition on the grains aspect ratio of the γ and α/α' phases. Comparing the aspect ratio of the α/α' grains in the cold rolled samples shows that the aspect ratio did not change in the AR+CR(RD)50% sample while it increased 12% in the AR+CR(TD)20% sample.

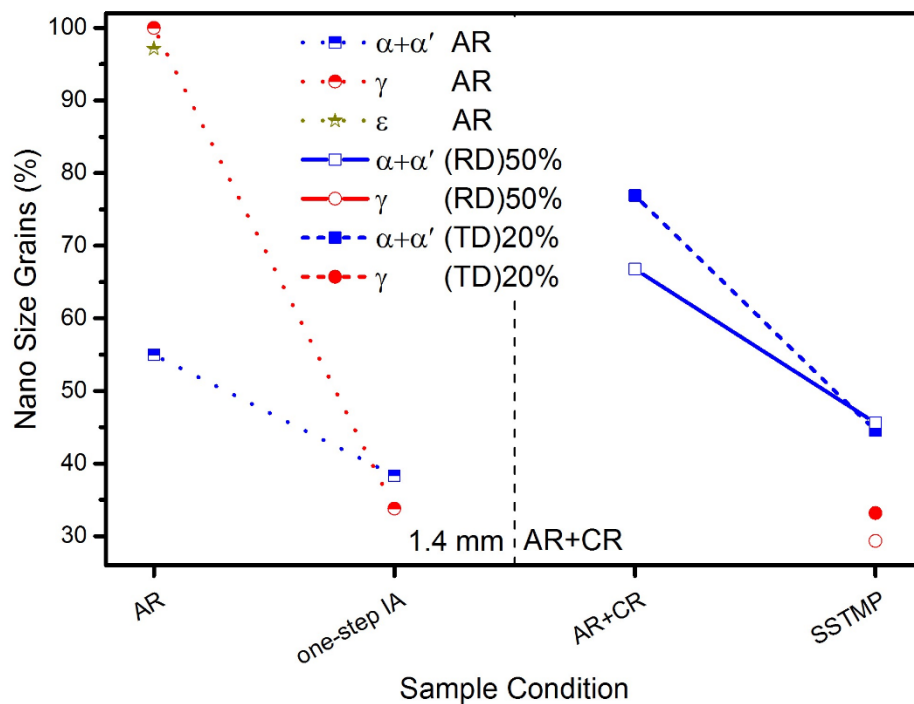


Figure 6.7. Effect of the sample condition on the nano-size grains frequency of the γ , α/α' , and ϵ from SEM-TKD maps.

The aspect ratios of the α/α' grains in the SSTMP(RD) and SSTMP(TD) were 11% and 4% less than the one-step IA sample. The aspect ratio of the γ grains in the SSTMP(RD) sample was as same as the one-step IA sample while in the SSTMP(TD) sample was 6% more than the one-step IA sample.

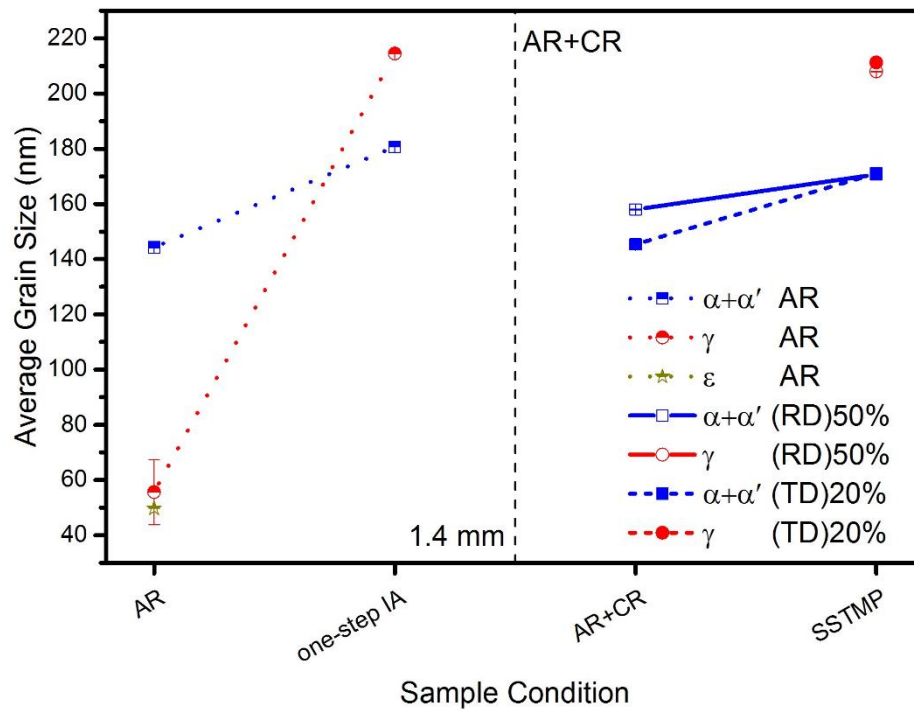


Figure 6.8. Effect of the sample condition on the average grain size of the γ , α/α' , and ϵ from SEM-TKD maps.

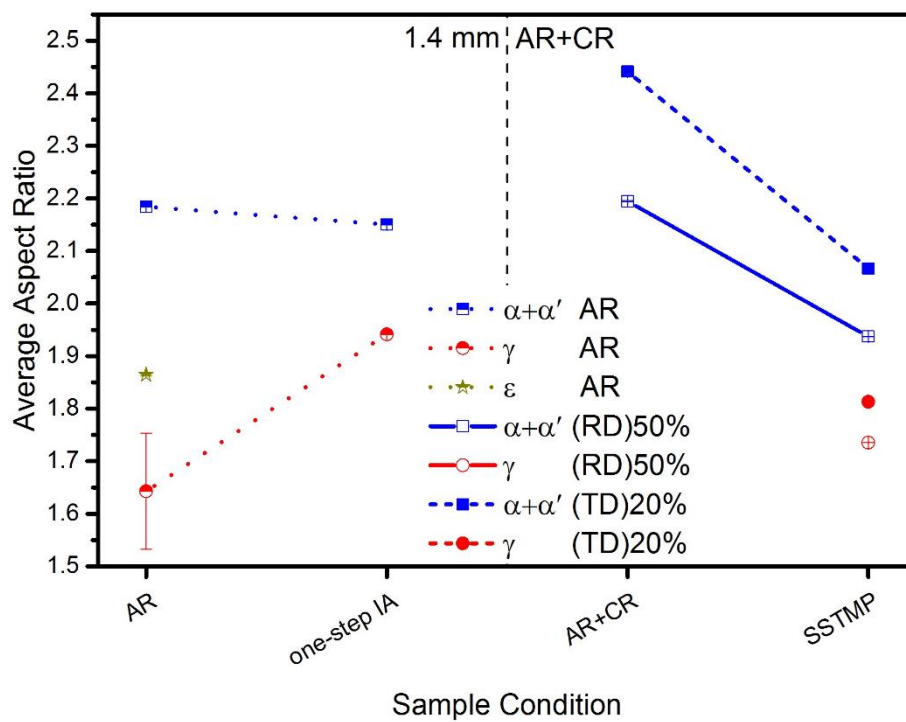


Figure 6.9. Effect of the sample condition on the grain aspect ratio of the γ , α/α' , and ϵ from SEM-TKD maps.

6.2.3 Phase Boundary Orientation Relationship

The phase boundaries between the γ and α/α' phases of the FeCMn steel samples are shown in Figure 6.10. Green lines indicate phases that deviate from the KS OR by less than 10° ; yellow lines indicate phases that deviated by more than 10° .

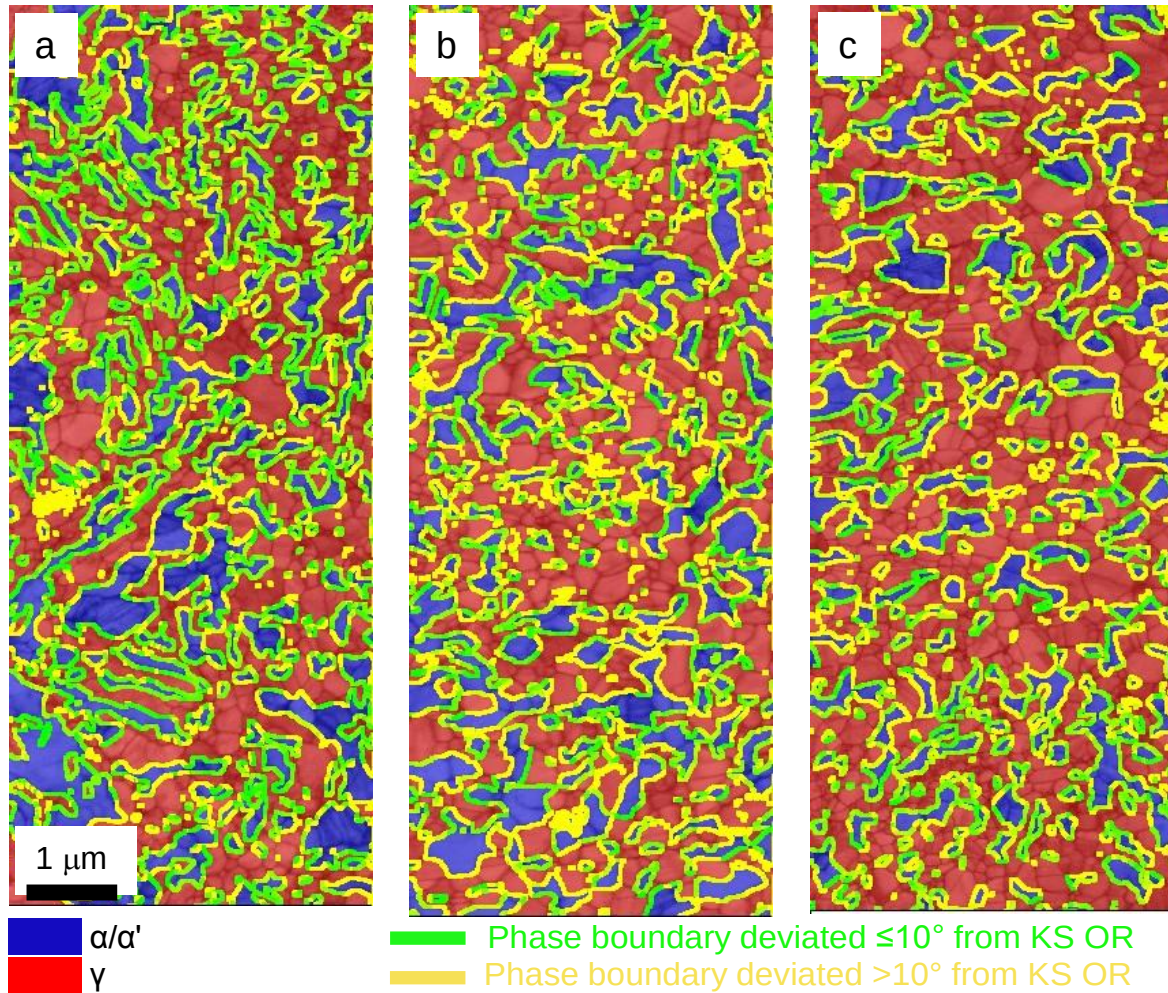


Figure 6.10. SEM-TKD, IQ-phase map FeCMn steel, (a) one-step IA, (b) SSTMP(RD) and (c) SSTMP(TD) samples. The phase boundaries between γ and α/α' phases are shown based on KS OR.

Figure 6.11 illustrates the histogram distribution of the γ and α/α' phases boundaries deviated from the KS orientation relationship. The frequency of the γ and α/α' phase boundaries deviated less than 10° from the KS orientation relationship was less in the SSTMP(RD) and SSTMP(TD) samples in comparison to the one-step IA sample. The frequency of the deviation from KS OR between 41° to 43° was 13.1%, 7.0% and 2.1% for the SSTMP(RD), SSTMP(TD), and one-step IA samples, respectively. There was another peak at 10° in the SSTMP(RD) (7.7%) and the SSTMP(TD) (6.4%).

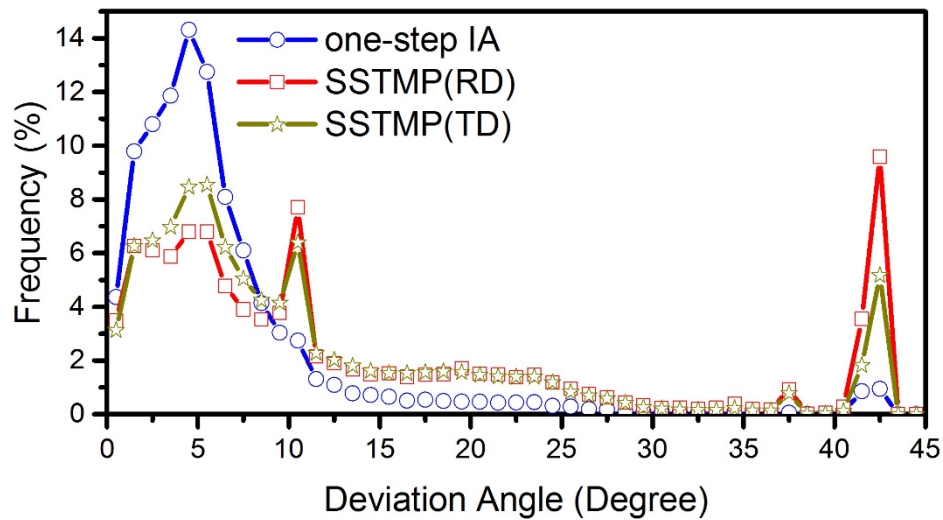


Figure 6.11. Histogram distribution of the the γ and α/α' phases boundaries in FeCMn steel samples deviated from the KS orientation relationship.

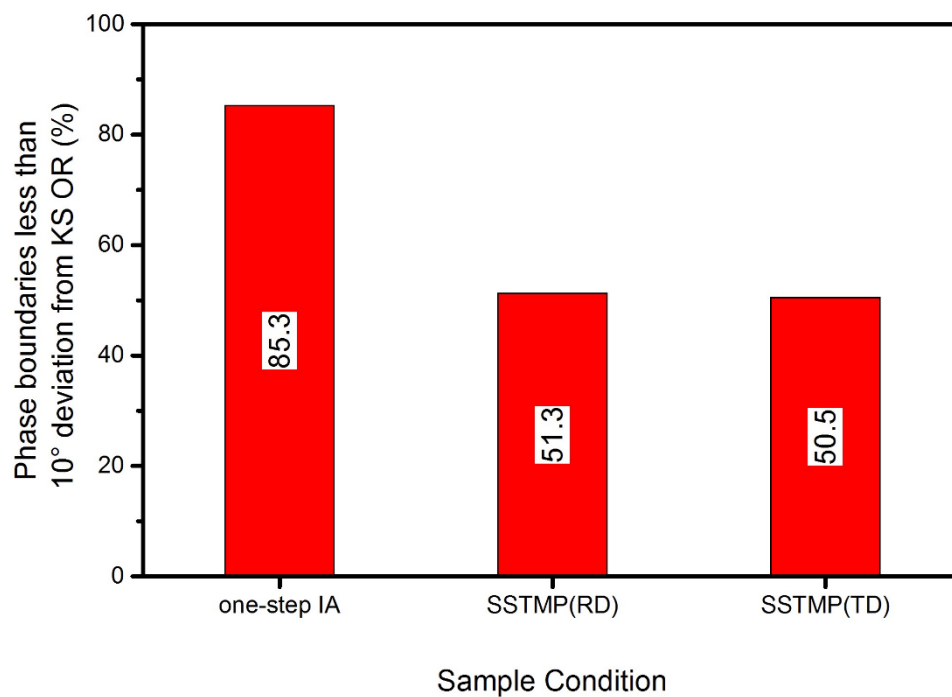


Figure 6.12. The frequency of the phase boundaries between the γ and α/α' with less than 10° deviation from KS OR in FeCMn steel samples.

Figure 6.12 depicts the frequency of the phase boundaries between the γ and α/α' phases with less than 10° deviation from KS. It shows a high percentage of the phase boundaries in the one-step IA sample was close to the KS OR (85.3%) while in the SSTMP(RD) and SSTMP(TD) samples, half of the phase boundaries were close to the KS OR.

6.2.4 Strain Distribution in the Phases

The γ and α/α' strain distributions were assessed qualitatively and quantitatively using the LAGBs and kernel average misorientation (KAM) analyses. They were used as an indicator of the dislocation density within phases.

6.2.4.1 Low-Angle Grain Boundaries

For the purpose of the developing this method, we used a fraction of the grain boundaries with a low angle of less than 15° . The γ and α/α' grain boundaries are illustrated in Appendix 2.

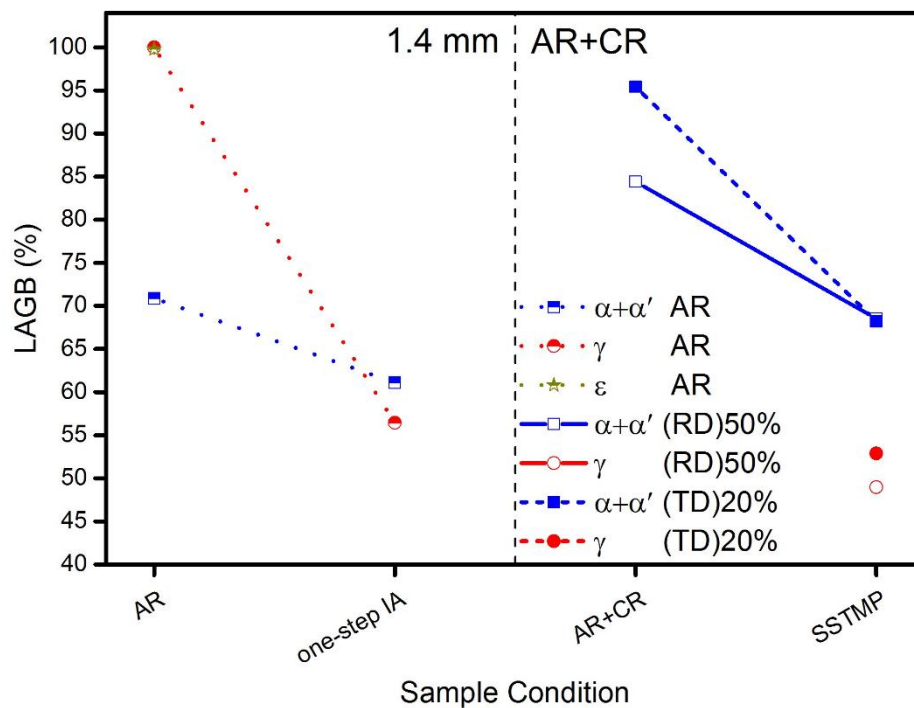


Figure 6.13. Variation of the LAGBs inside the the γ and α/α' phases of the cold-rolled and annealed FeCMn samples.

Figure 6.13 illustrates the variation of the LAGBs inside the γ and α/α' phases of the cold-rolled and annealed FeCMn samples. The fraction of the LAGBs in the α/α' phase was 70.9% for the AR sample. After cold rolling 50% in RD and 20% in TD, the fraction of the LAGBs in the α/α' phase increased to 84.4% and 95.4%, respectively. The highest fraction was for the AR+CR(TD)20% sample.

The fractions of the LAGBs in the α/α' phase were 61.1%, 68.5 and 68.2% for the one-step IA, SSTMP(RD) and SSTMP(TD) samples. The fractions of the LAGBs in the γ phase after the IA are 56.5%, 49.0 and 52.9% for the one-step IA, SSTMP(RD) and SSTMP(TD) samples.

6.2.4.2 Kernel Average Misorientation

By using the KAM method, it was possible to compute the average misorientation around a given measurement point in comparison with a defined set of the nearest neighbours. This study utilized a KAM value of less than 5° . Appendix 2 presents the local misorientation maps as well as their histogram distributions for the FeCMn steel samples.

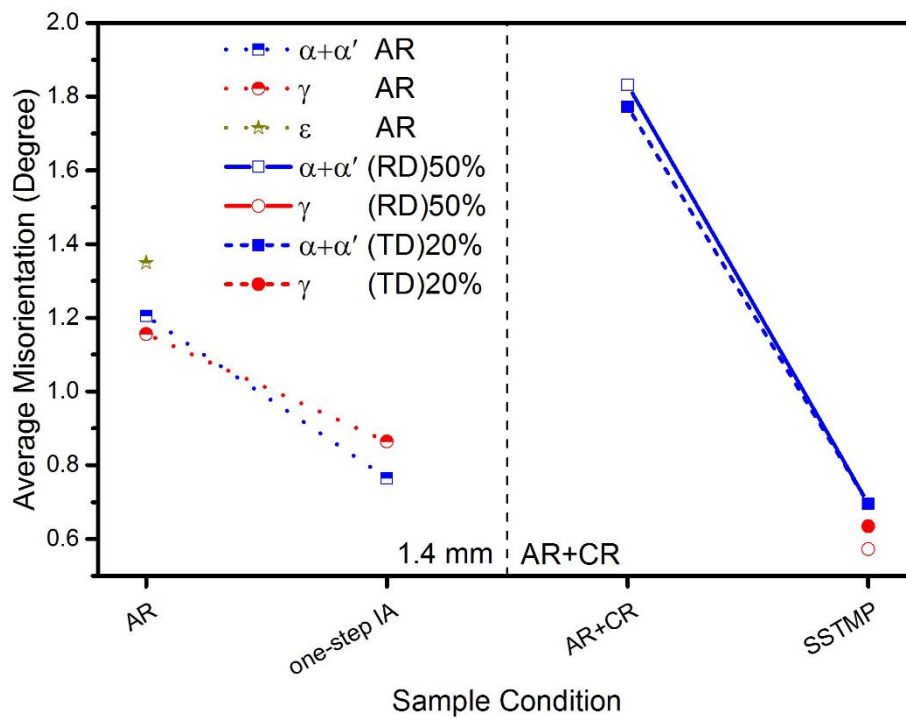


Figure 6.14. The average local misorientation of the γ and α/α' phases in the cold-rolled and annealed FeCMn samples.

Figure 6.14 demonstrates the average local misorientation of the γ and α/α' phases. The average local misorientation of the α/α' phase was 1.56° in the AR sample. After the following RD and TD cold rolling, it increased to 1.83° and 1.77° , respectively. Then, it decreased to 0.69° in SSTMP(RD) and the SSTMP(TD) samples. The figure shows that the one-step IA sample had a higher average local misorientation of the α/α' phase in comparison to the SSTMP(RD) and the SSTMP(TD) samples (10% higher).

The average local misorientation of the γ phase in the SSTMP(RD) and the SSTMP(TD) samples was 0.57° and 0.64° , respectively. As shown in Figure 6.14, the average local misorientation of the one-step sample was 0.86° .

6.2.5 Texture

To assess the texture of the different phases after cold rolling and IA, the SEM-TKD maps were used to conduct the orientation distribution function (ODF) analysis. Figures 6.15 and 6.16 illustrate the common texture components and fibers of the BCC and FCC metals and alloys. Table 6.1 provides Euler angles for the fibers of the BCC metals. For the common fibers and texture components of the FCC metals, Tables 6.2 and 6.3 provide Miller indices and Euler angles.

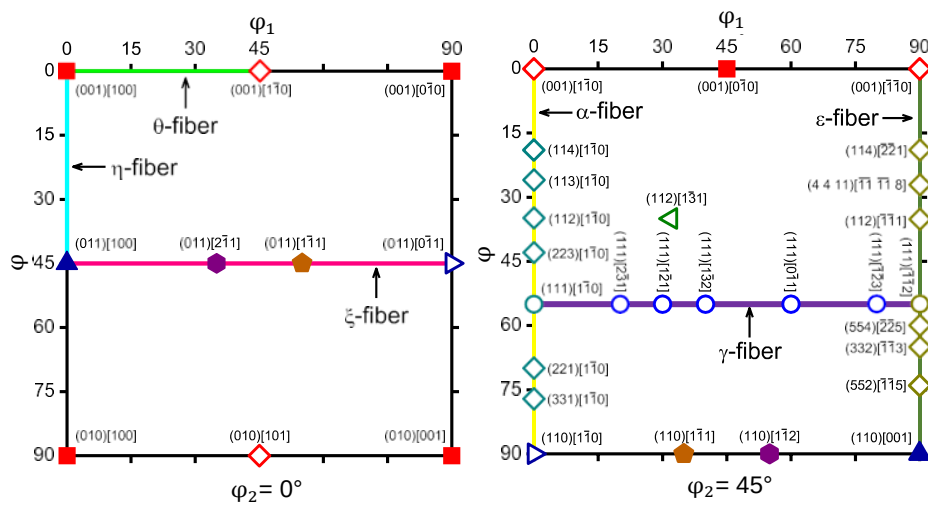


Figure 6.15. Common texture components and fibers of the BCC metals and alloys in $\phi_2 = 0^\circ$ and 45° sections.

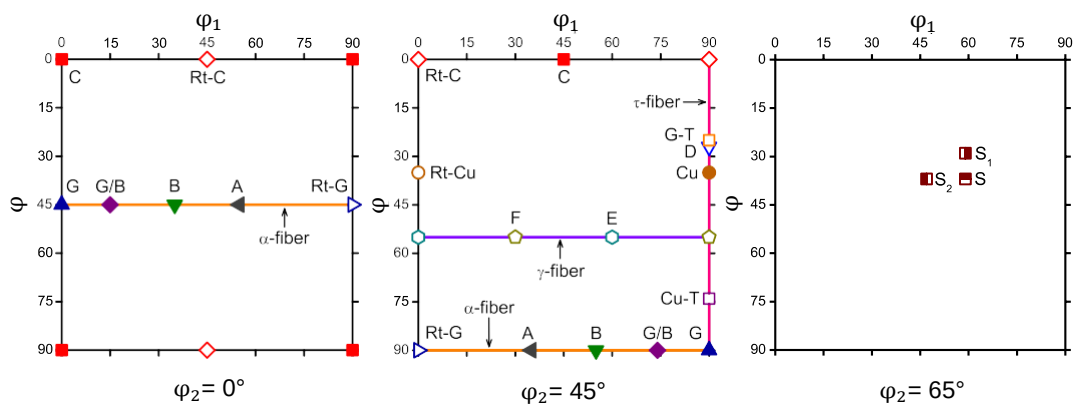


Figure 6.16. Common texture components and fibers of the FCC metals and alloys in $\phi_2 = 0^\circ$, 45° , and 65° sections.

Table 6.1. Euler angles and the axis for common texture fibers in BCC metals and alloys.

Name	Fiber axis	Euler angles, $\varphi_1^\circ, \varphi^\circ, \varphi_2^\circ$
α - fiber	$\langle 011 \rangle \parallel$ RD	$0^\circ 0^\circ 45^\circ$ to $0^\circ 90^\circ 45^\circ$
γ - fiber	$\langle 111 \rangle \parallel$ ND	$0^\circ 55^\circ 45^\circ$ to $90^\circ 55^\circ 45^\circ$
ε - fiber	$\langle 011 \rangle \parallel$ TD	$90^\circ 0^\circ 45^\circ$ to $90^\circ 90^\circ 45^\circ$
β - fiber		$0^\circ 35^\circ 45^\circ$ to $90^\circ 55^\circ 45^\circ$
θ - fiber	$\langle 011 \rangle \parallel$ ND	$0^\circ 0^\circ 0^\circ$ to $45^\circ 0^\circ 0^\circ$
η - fiber	$\langle 001 \rangle \parallel$ RD	$0^\circ 0^\circ 0^\circ$ to $0^\circ 45^\circ 0^\circ$
ξ - fiber	$\langle 011 \rangle \parallel$ ND	$0^\circ 45^\circ 0^\circ$ to $90^\circ 45^\circ 0^\circ$

Table 6.2. Euler angles and the axis for common texture fibers in FCC metals and alloys.

Name	Fiber axis	Euler angles, $\varphi_1^\circ, \varphi^\circ, \varphi_2^\circ$
α - fiber	$\langle 011 \rangle \parallel$ ND	$0^\circ 45^\circ 0^\circ$ to $90^\circ 45^\circ 0^\circ$
γ - fiber	$\langle 111 \rangle \parallel$ ND	$0^\circ 55^\circ 45^\circ$ to $90^\circ 55^\circ 45^\circ$
τ - fiber	$\langle 011 \rangle \parallel$ TD	$90^\circ 0^\circ 45^\circ$ to $90^\circ 90^\circ 45^\circ$
β - fiber		Cu to S to B components

Table 6.3. Miller indices and Euler angles for common texture components in FCC metals.

Texture component	Miller indices	Euler angles, $\varphi_1^\circ, \varphi^\circ, \varphi_2^\circ$
Cube, C	$\{001\} \langle 100 \rangle$	$0^\circ/90^\circ 0^\circ/90^\circ 0^\circ$ and $45^\circ 0^\circ 45^\circ$
Rotated Cube, Rt-C	$\{001\} \langle 110 \rangle$	$45^\circ 0^\circ/90^\circ 0^\circ$ and $0^\circ/90^\circ 0^\circ 45^\circ$
Goss, G	$\{011\} \langle 001 \rangle$	$0^\circ 45^\circ 0^\circ$ and $90^\circ 90^\circ 45^\circ$
G/B	$\{110\} \langle 115 \rangle$	$15^\circ 45^\circ 0^\circ$ and $74^\circ 90^\circ 45^\circ$
Brass, B	$\{011\} \langle 112 \rangle$	$35^\circ 45^\circ 0^\circ$ and $55^\circ 90^\circ 45^\circ$
A	$\{110\} \langle 111 \rangle$	$55^\circ 45^\circ 0^\circ$ and $35^\circ 90^\circ 45^\circ$
Rotated Goss, Rt-G	$\{011\} \langle 011 \rangle$	$90^\circ 45^\circ 0^\circ$ and $0^\circ 90^\circ 45^\circ$
Copper, Cu	$\{112\} \langle 111 \rangle$	$90^\circ 35^\circ 45^\circ$
Rotated Copper, Rt-Cu	$\{112\} \langle 011 \rangle$	$0^\circ 35^\circ 45^\circ$
F	$\{111\} \langle 112 \rangle$	$30^\circ/90^\circ 55^\circ 45^\circ$
E	$\{111\} \langle 110 \rangle$	$0^\circ/60^\circ 55^\circ 45^\circ$
Dillamore/Taylor, D	$\{4\ 4\ 11\} \langle 11\ 11\ 8 \rangle$	$90^\circ 27^\circ 45^\circ$
Goss Twin, G-T	$\{113\} \langle 332 \rangle$	$90^\circ 25^\circ 45^\circ$
Copper Twin, Cu-T	$\{552\} \langle 115 \rangle$	$90^\circ 74^\circ 45^\circ$
S	$\{123\} \langle 634 \rangle$	$59^\circ 37^\circ 63^\circ$
S1	$\{124\} \langle 211 \rangle$	$59^\circ 29^\circ 63^\circ$
S2	$\{123\} \langle 412 \rangle$	$47^\circ 37^\circ 63^\circ$

The α/α' and γ texture in the cold rolled and annealed samples are shown in Figures 8.20 and 8.21. Figure 6.17 illustrates the texture of the α/α' phase in the AR+CR(RD)50% samples, before and after IA. The AR+CR(RD)50% sample had a strong α -fiber and weaker γ -fiber. It

had a strong component at $(0^\circ \ 11^\circ \ 45^\circ)$ ($f(g)=16.4$) and weaker $\{001\}\langle 110\rangle$ at $f(g)=12.8$ and $\{111\}\langle 123\rangle$ at $f(g)=4.2$ times random.

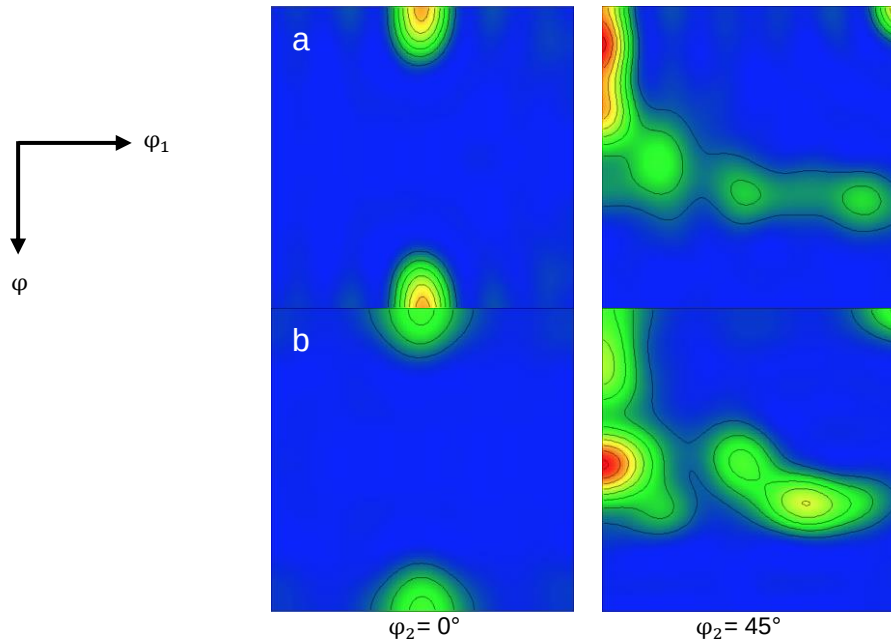


Figure 6.17. The texture of the α/α' phase in FeCMn alloy (a) AR+CR(RD)50% and (b) SSTMP(RD) in $\varphi_2 = 0^\circ$ and 45° ODF sections. Contour levels = $2x$.

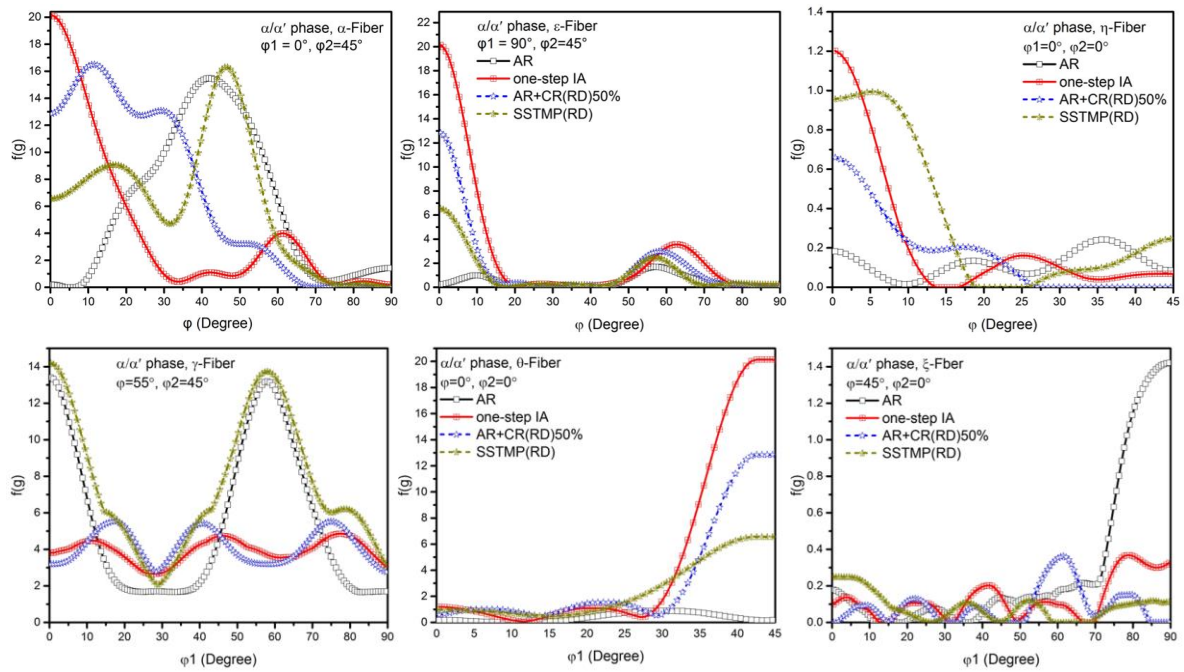


Figure 6.18. The fibers of the α/α' phase in the AR and AR+CR(RD)50% FeCMn steel samples in the cold-rolled and annealed conditions.

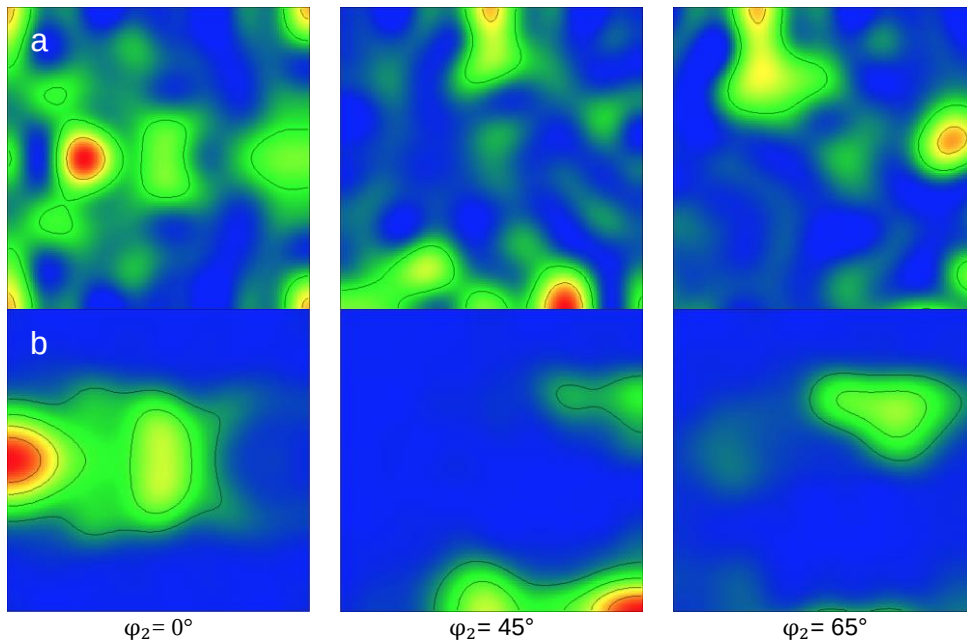


Figure 6.19. The texture of the γ phase in FeCMn alloy (a) AR+CR(RD)50% and (b) SSTMP(RD) in $\phi_2 = 0^\circ$, 45° and 65° ODF sections. Contour levels = $2x$.

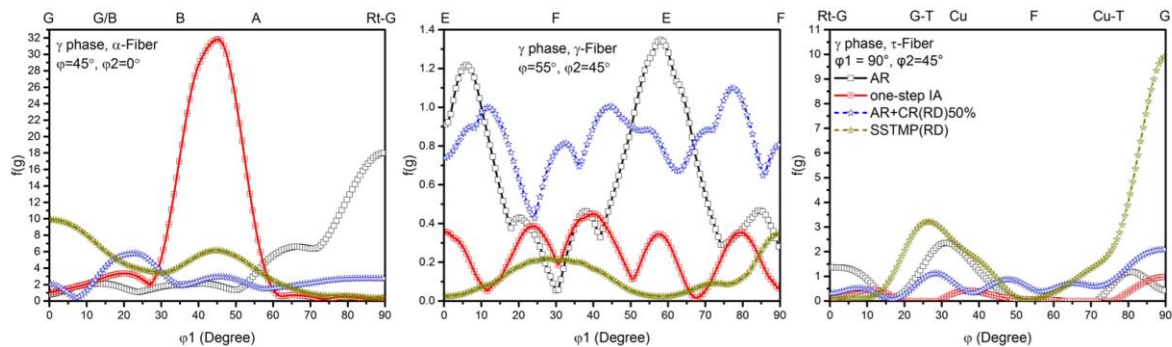


Figure 6.20. The fibers of the γ phase in the AR and AR+CR(RD)50% FeCMn steel samples in the cold-rolled and annealed conditions.

The α/α' phase of the SSTMP(RD) sample had α and γ fibers. It had a strong $\{223\}\langle 110 \rangle$ component at $f(g)=16.3$ times random and weaker $\{111\}\langle 110 \rangle$, $\{114\}\langle 110 \rangle$ and $\{001\}\langle 110 \rangle$ at $f(g)=10.1$, 8.74 and 6.53 times random, respectively.

Figure 6.18 illustrates the fibers of the α/α' phase in the AR and AR+CR(RD)50% before and after IA. The ε -fiber had the same behaviour in the samples except around the $\{001\}\langle 110 \rangle$. This texture component was at $f(g)=0.3$, 12.8 and 6.5 times random for the AR sample, AR+CR(RD)50% and SSTMP(RD) samples, respectively. The γ -fiber of the AR and SSTMP(RD) samples was close similar. It had almost same behaviour in the one-step IA sample and AR+CR(RD)50% samples.

Figure 6.19 illustrates the texture of the γ in the AR_CR(RD)50% samples before and after IA. The AR+CR(RD)50% sample had α , γ and ε fibers and a strong (011)[3 $\bar{2}$ 2], and weaker Cube ($\{001\}<100>$), Rt-G ($\{011\}<011>$) and Goss ($\{011\}<001>$) texture components at $f(g)$ = 5.8, 4.3, 2.8 and 2.1 times random, respectively.

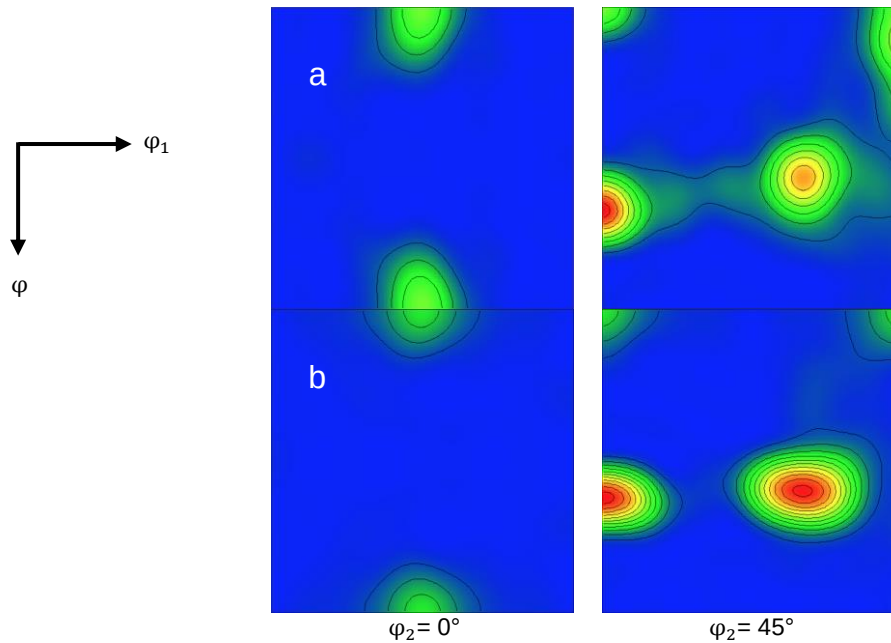


Figure 6.21. The texture of the α/α' phase in FeCMn alloy (a) AR+CR(TD)20% and (b) SSTMP(TD) in $\varphi_2 = 0^\circ$ and 45° ODF sections. Contour levels = 2x.

The γ phase in the SSTMP(RD) had an α -fiber and a strong Goss ($\{011\}<001>$) and weaker (011)[3 $\bar{2}$ 2], S1 ($\{124\}<211>$) and D ($\{4\ 4\ 11\}<11\ 11\ 8>$) texture components at $f(g)$ = 9.91, 5.82, 4.35 and 3.21, respectively. The γ -fiber of the γ phase, which is shown in Figure 6.20, had a low intensity in all the samples, and the lowest was for the SSTMP(RD) sample. The τ -fiber of the SSTMP(RD) sample had the highest intensity.

Figure 6.21 illustrates the texture of the α/α' phase in AR+CR(TD)20% samples before and after the IA. The α/α' phase of the AR+CR(TD)20% sample had a strong γ and weaker ε fibers and a strong E ($\{111\}<110>$) component at $f(g)$ =16.8 times random and a weaker Rt-C ($\{001\}<110>$) at $f(g)$ =7.9 times random.

The α/α' phase of the SSTMP(TD) sample had a strong γ -fiber and a strong E ($\{111\}<110>$) component at $f(g)$ =22.8 times random and a weaker Rt-C ($\{001\}<110>$) at $f(g)$ =7.1 times random.

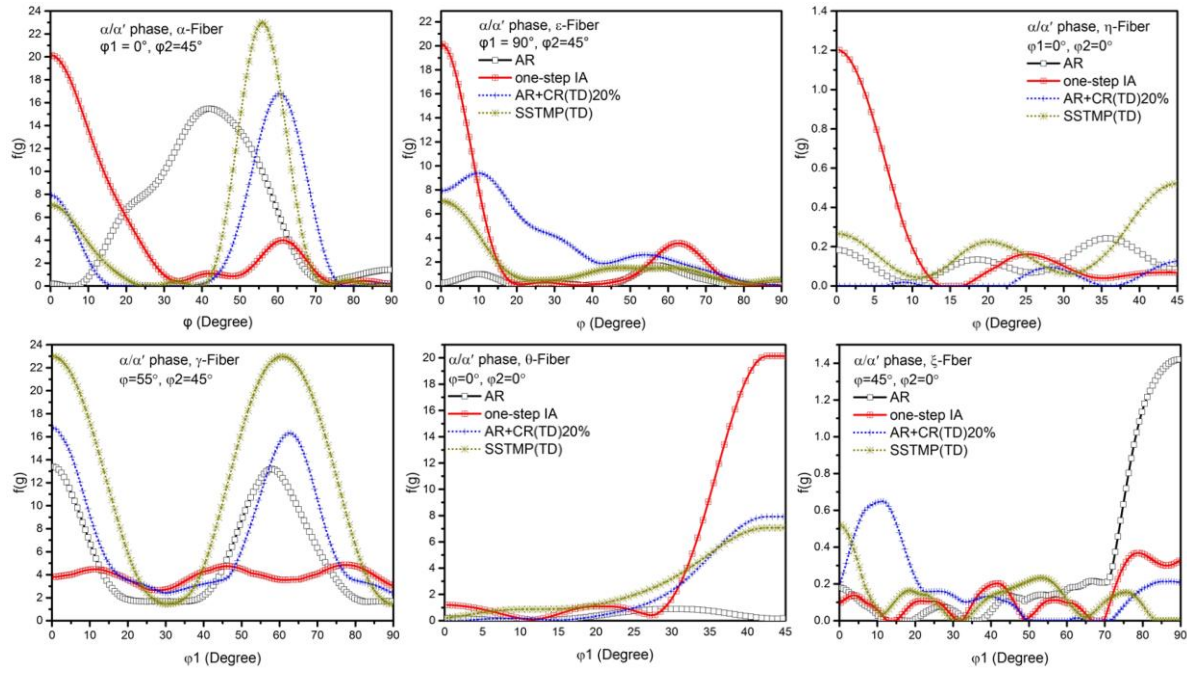


Figure 6.22. The fibers of the α/α' phase in the AR and AR+CR(TD)20% FeCMn steel samples in the cold-rolled and annealed conditions.

Figure 6.22 shows the fibers of the α/α' phase in the AR and AR+CR(TD)20% samples before and after the IA. The SSTMP(TD) had the highest intensity of the γ -fiber for the α/α' phase and the one-step IA sample had the lowest. The AR+CR(TD)20% had almost the same θ -fiber in α/α' phase before and after IA.

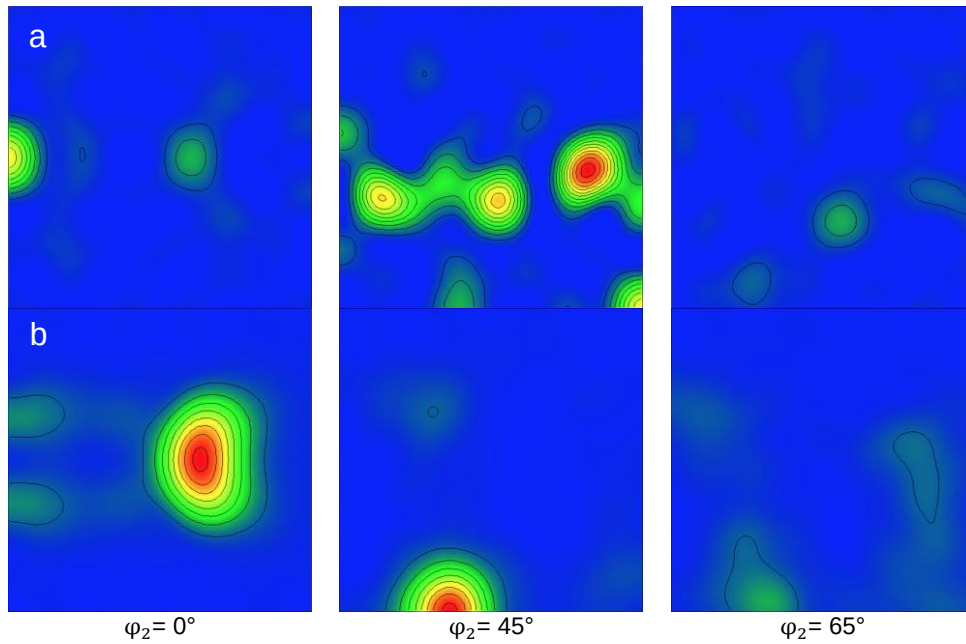


Figure 6.23. The texture of the γ phase in FeCMn alloy (a) AR+CR(TD)20% and (b) SSTMP(TD) in $\phi_2 = 0^\circ, 45^\circ$ and 65° ODF sections. Contour levels = $2x$.

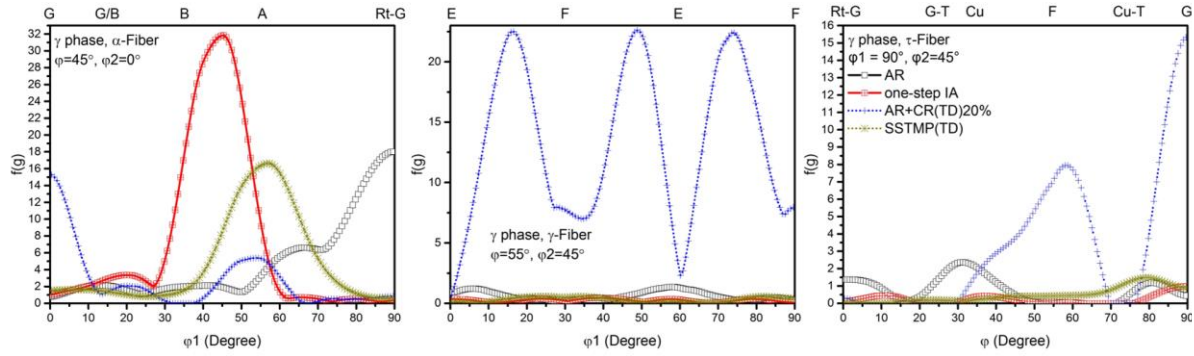


Figure 6.24. The fibers of the γ phase in the 1.4 mm and 1.1 mm WPHT rolled TD FeCMn Steel samples in the cold-rolled and annealed conditions.

The γ phase of the AR+CR(TD)20% sample had a γ -fiber and a strong component at $(74^\circ, 50^\circ, 45^\circ)$ ($f(g)=23$) and weaker $(111)[\bar{2}31]$, Goss ($\{011\}<001>$) and A ($\{110\}<111>$) components at $f(g)=16.2$, 15.3 and 5.32 times random, respectively.

The γ of the SSTMP(TD) sample had the strong A ($\{110\}<111>$) and weaker $\{023\}<100>$ components at $f(g)=16.6$ and 2.78 times random, respectively.

Figure 6.24 shows the fibers in the γ phase in the AR and AR+CR(TD)20% samples before and after the IA. It shows that the SSTMP(TD) did not have γ and τ fibers and its α -fiber was weaker than the annealed 1.4 mm sample.

6.2.6 Summary of the Scanning Electron Microscopy Maps Analyses

The analyses of the SEM-TKD maps of the FeCMn steel before and after the SSTMP summarised in Table 6.4.

Table 6.4. Summary of the SEM-TKD maps of the FeCMn steel after the SSTMP.

Sample Condition	Phase Volume Fraction %		Average Grain Size nm		Nano Size Grains %		KAM Average Misorientation Degree	
	γ	α/α'	γ	α/α'	γ	α/α'	γ	α/α'
1.4 mm - AR	0.1	96.7	56	144	100.0	55.0	1.16	1.21
1.4 mm - one-step IA	62.0	38.0	215	181	33.8	38.3	0.86	0.77
AR+CR(RD)50%		100		158		66.8		1.83
SSTMP(RD)	70.3	29.7	208	171	29.3	45.6	0.57	0.70
AR+CR(TD)20%		100		145		76.9		1.77
SSTMP(TD)	69.8	30.3	211	171	33.2	44.5	0.63	0.70

Sample Condition	Aspect Ratio (No Preferred Direction)		Aspect Ratio (RD/TD)		LAGB %		Phase Boundaries <10° Deviation from KS OR %
	γ	α/α'	γ	α/α'	γ	α/α'	
1.4 mm - AR	1.64	2.18		1.28	100.0	70.9	31.8
1.4 mm - one-step IA	1.94	2.15	1	1	56.5	61.1	85.3
AR+CR(RD)50%		2.19		1.29		84.4	
SSTMP(RD)	1.74	1.94	0.93	0.94	49.0	68.5	51.3
AR+CR(TD)20%		2.44		0.74		95.4	
SSTMP(TD)	1.81	2.07	0.91	0.85	52.9	68.2	50.5

6.3 X-Ray Diffraction

Figure 6.25 displays the XRD findings of all examined samples in this chapter. It is evident that the ratio of the $(211)_\alpha$ peak to the $(200)_\alpha$ peak decreased due to TD cold rolling in both cold-rolled and intercritically annealed samples.

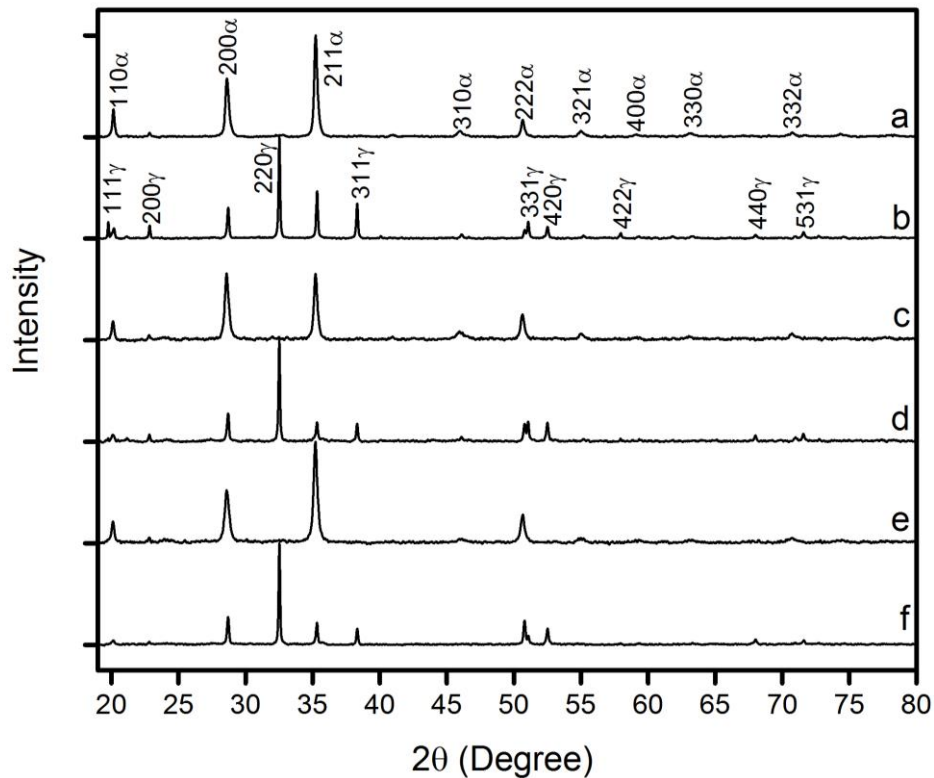


Figure 6.25. X-ray diffraction spectra of the FeCMn steel (a) AR and (b) one-step IA, (c) AR+CR(TD)20% (d) SSTMP(TD) (e) AR+CR(RD)50% and (f) SSTMP(RD) samples.

6.4 Mechanical Properties

6.4.1 Tensile Test Data

Figure 6.26 depicts the engineering stress - engineering strain curve of the FeCMn alloy specimens. The mechanical properties of the samples are summarized in Table 6.5.

Comparing the RD tensile samples of the one-step IA sample with the SSTMP(RD) shows that they had very close yield and tensile strengths but the one-step IA sample had the lower total elongation. This resulted in higher product of strength and elongation (PSE) and the formability of the SSTMP(RD) sample. The PSE is a measure of the energy absorption and formability of a material, calculated by multiplying its ultimate tensile strength (UTS) and total elongation.

The RD tensile sample of the SSTMP(TD) had 4.9% and 3.3% higher yield strength and UTS in comparison to the RD tensile sample of the one-step IA, but they had very close work-hardening properties. The total elongation of the one-step IA sample was 7.2% less than the SSTMP(TD) sample. The TD tensile samples of the SSTMP(TD) and the one-step IA had similar yield strength and UTS but the elongation of the one-step IA sample was 11.8% less.

The results show that the SSTMP(TD) sample had the highest formability especially in the TD direction.

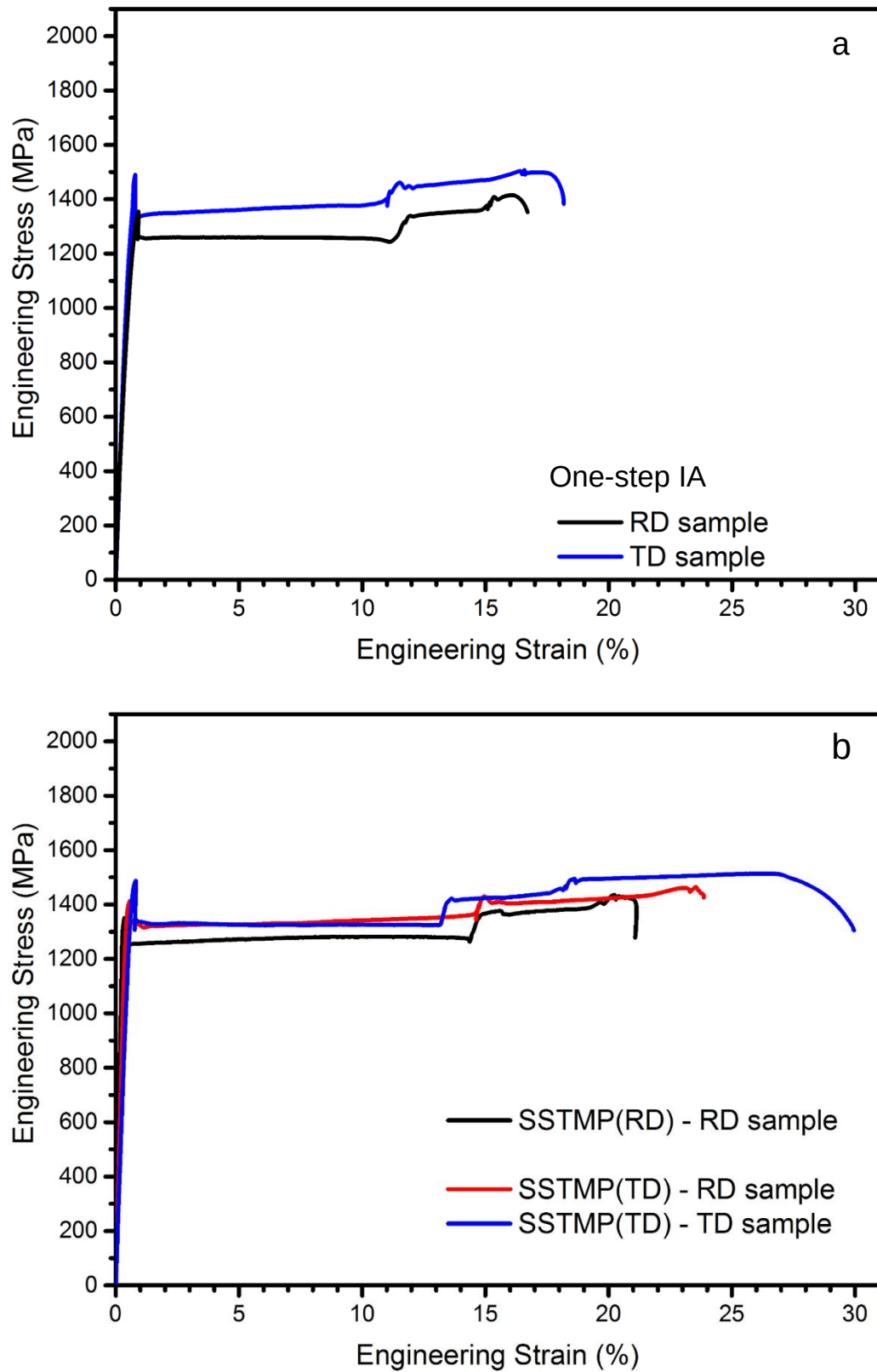


Figure 6.26. Tensile test results of the FeCMn samples after (a) one-step IA and (b) WPHT cold rolling and annealing at 620 °C for 10 min. Tensile experiments were conducted at room temperature and a constant strain rate of 10^{-3} s^{-1} .

6.4.2 Hardness Data

Figure 6.27 shows the hardness of the FeCMn sample after cold rolling and heat treatment conditions. The hardness of the AR was 507.6 ± 3.2 HV. 50% and 20% rolling of this sample in RD and TD directions, increased the hardness to 581.2 ± 9.6 and 525.6 HV, respectively. After the IA of the AR+CR(RD)50% and AR+CR(TD)20% samples, the hardness fell to 458.2 ± 4.2 and 455.2 ± 4.0 Hv, respectively. These amounts were higher than the 442.36 HV which is the hardness of the one-step IA sample.

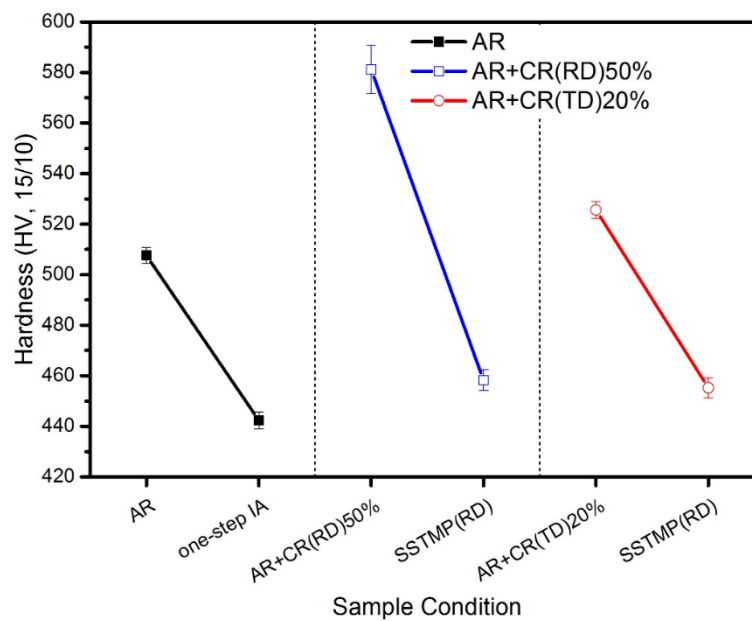


Figure 6.27. Effect of the cold rolling and annealing on the hardness of the FeCMn samples under a load of 10 kg and for a dwell time of 15 Sec.

6.5 Summary

In this chapter, we explored the impact of augmenting the initial dislocation density prior to intercritical annealing by introducing an additional cold rolling step to the as-received base FeCMn AHSS. The objective is to gain insights into the alterations in microstructure and properties that occur when enhancing the driving force for the $\alpha' \rightarrow \alpha + \gamma$ phase transformation during intercritical annealing. The findings indicated that elevating the initial dislocation density prior to IA led to an increase in the γ phase volume fraction and total elongation. The results discussed in chapter 10.

Table 6.5. Room-temperature tensile test data of the 1.4 mm and WPHT FeCMn samples.

	Heat treatment	Rolling Direction	Heat treatment 10 min	Sample direction	Upper Yield Strength MPa	Lower Yield Strength MPa	Tensile Strength MPa	Strength ratio	Total Elongation %	PSE TSxTE MPa.%E
One-step IA	620 °C - 10 min	-	-	RD	1355	1253	1415	0.886	16.7	23631
		-	-	TD	1490	1299	1507	0.862	18.2	27427
SSTMP(RD)	--	RD	620 °C	RD	1350	1245	1435	0.868	21.1	30279
SSTMP(TD)		TD	620 °C	RD	1413	1314	1462	0.899	23.9	34942
			620 °C	TD	1487	1306	1514	0.863	30.0	45420

CHAPTER 7 Effect of the Starting Martensitic Microstructure on the Thermomechanical Process of the FeCMnAl Steel

In this chapter, a comparison is made between a specimen with an initial deformed martensite microstructure and a specimen with initial quenched and deformed martensite microstructure that underwent the IA under the same conditions.

7.1 Thermomechanical Procedure

This chapter examines the effects of two different heat treatment conditions (Figure 7.1) on the microstructure and mechanical properties of the FeCMnAl alloy. In chapter 9, the SEM-TKD maps of the one-step IA heat treatment were presented. Figure 7.1 (a) illustrates the one-step IA of the sample, which was annealed at 650 °C for 10 min, and then quenched in water.

During the TSTMP which is shown in Figure 7.1(b), the specimen had a short austenitization annealing at 700 °C for 3 min, then quenched in oil at 200 °C, and finally quenched in water. Cold rolling was used to reduce the thickness by 5 percent (CR(RD)5%). Lastly, the sample was quenched in water after being annealed at 650 °C for 10 min.

7.2 Microstructure

7.2.1 Scanning Electron Microscopy Maps and Phase Volume Fractions

The SEM-TKD IQ-phase maps of the FeCMnAl sample prior to and following IA at 650 °C for 10 min were presented in Chapter 9 and shown in Figures 9.2 and 9.3, respectively.

Furthermore, Figure 7.2 displays the microstructure after each process of the TSTMP in this chapter. Figure 7.2(a) exhibits the microstructure of the sample after annealing at 700 °C for 3 min, subsequently quenched in oil at 200 °C and ultimately quenched in water. At 700 °C, the sample was entirely in the γ phase, but following quenching, α and α' -laths were observed (indicated by white arrows). The formation of α' could potentially be attributed to quenching or sample preparation. [59, 156].

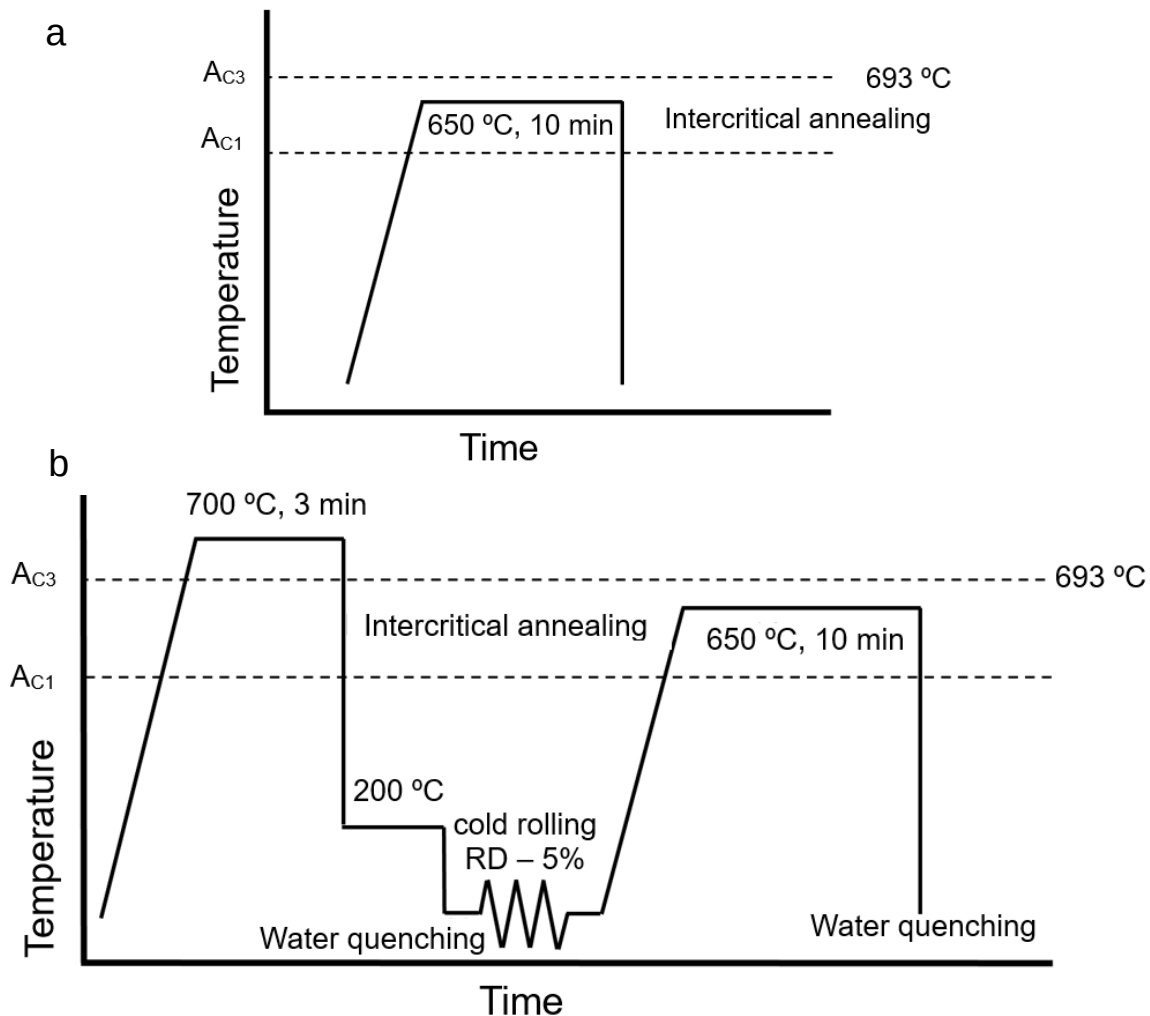


Figure 7.1. The schematic of the (a) one-step IA process (b) TSTMP-1 for the FeCMnAl alloy.

The SEM-TKD IQ-phase map indicates that the volume fraction of the retained γ phase was 1%, whereas the XRD data (Figure 7.3) shows that the volume fraction of the γ phase was 30.9%. The discrepancy between the two methods was attributed to the SEM-TKD sample preparation process. Following annealing at 700 °C and the subsequent quenching, the retained γ grains had the residual stress. In the bulk sample, these grains were surrounded by other grains and were unable to transform into martensite. The SEM-TKD sample was a thin foil,

similar to the TEM sample, and the grains in the sample were more easily capable of undergoing martensitic transformation. In addition, using PIPS during the sample preparation process accelerated the austenite to martensite transformation.

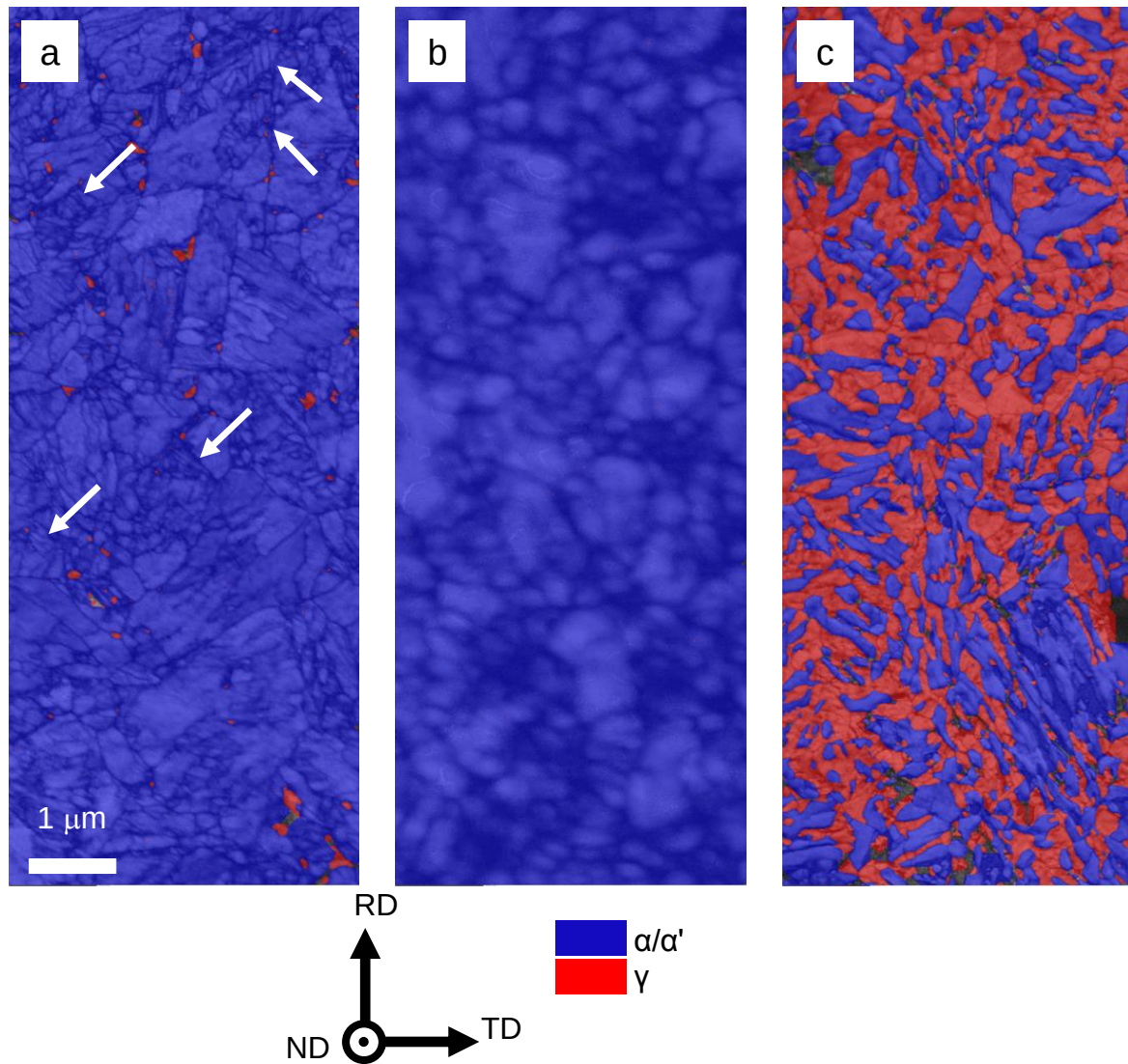


Figure 7.2. FeCMnAl alloy that (a) annealed at 700 °C for 3 min, quenched at 200 °C and then in the water (b) CR(RD)5% and then (c) the IA at 650 °C for 10 min. On the SEM-TKD maps, red represents γ , and blue represents α/α' .

Figure 7.2 (b) illustrates the annealed sample at 700 °C after 5% cold rolling. The phase volume fraction of the retained γ is 0.4% which was close to the XRD result (0.9%). Figure 7.2 (c) shows the IQ-phase map of the annealed and cold-rolled sample after the IA at 650 °C for 10 min. The volume fraction of the retained γ phase was 50.7% while the XRD result shows 63.8%.

Figure 7.3 presents the phase volume fraction of the phases from SEM-TKD and XRD results. The XRD data of the sample after TSTMP was greater than that of the sample after one-step annealing (63.8% and 61%, respectively), whereas the TKD-SEM results exhibited less (50.7% and 59.8%, respectively). This phenomenon indicated that the sample after the TSTMP had more residual stress in the retained γ phase after quenching, which influenced the phase volume fraction during SEM-TKD sample preparation.

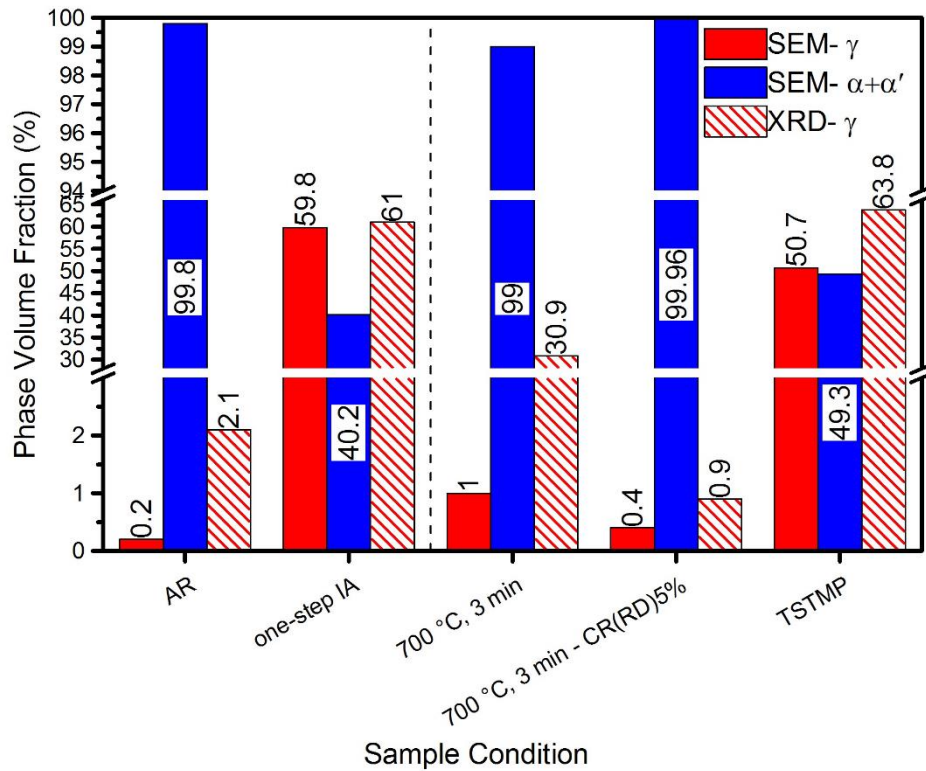


Figure 7.3. The phase volume fraction of the FeCMnAl alloy samples at different conditions using SEM-TKD and XRD.

7.2.2 Grain Size

Figure 7.4 depicts the grain size distributions of the γ and α/α' phases of the FeCMnAl alloy samples after one-step IA and the TSTMP. It could be seen that the TSTMP resulted in the higher frequency of the grains less than 60 nm.

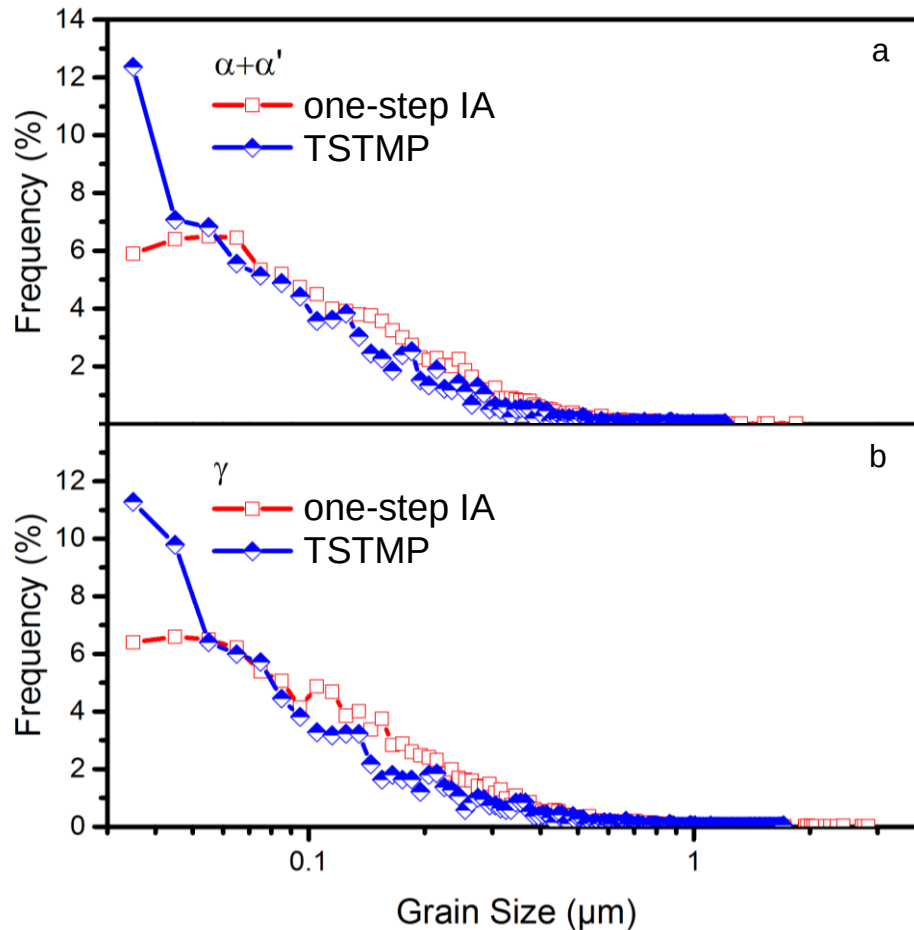


Figure 7.4. Effect of the one-step IA and the TSTMP on the grain size distribution of the (a) α/α' and (b) γ from SEM-TKD maps.

Figure 7.5 shows the effect of the one-step IA and the TSTMP on the nano-size grains volume fraction. It can be seen that the nano-size grains volume fractions of the γ and α/α' phases after one-step IA were 44.4% and 33.1% which were less than the nano-size grains volume fractions of the γ and α/α' phases after the TSTMP, respectively. In addition, the results indicate that for the one-step IA condition, the volume fraction of the γ phase in nano-sized grains was higher than that of the α/α' phase. However, for the TSTMP, the opposite trend was observed.

Figure 7.6 shows the effect of the one-step IA and the TSTMP on the average grain size of the α/α' and γ phases. The average grains size of the α/α' and γ was 194 nm and 218 nm after the one-step IA, respectively. The average grains size of the α/α' and γ was 134 nm and 146 nm after the TSTMP, respectively. It shows that the average grains size of the α/α' and austenite phases after the TSTMP was 31% and 33% less than the one-step IA, respectively.

Figure 7.7 depicts the effect of the one-step IA and the TSTMP on the grains' average aspect ratio in the α/α' and γ phases. The average aspect ratio α/α' was 8% higher than γ after the one-step IA while it was similar for α/α' and γ after the TSTMP.

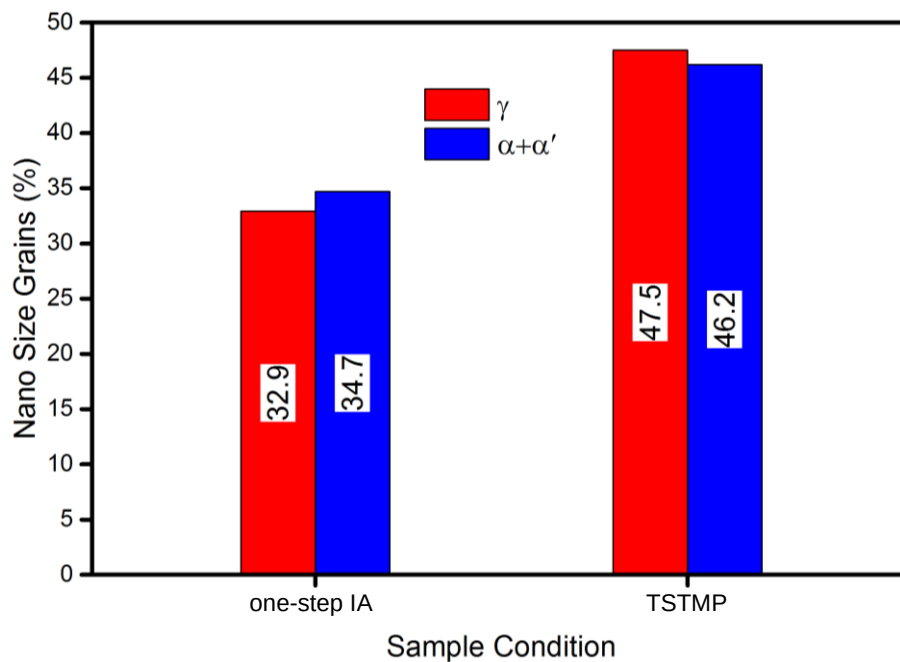


Figure 7.5. Effect of the one-step IA and the TSTMP on the nano-size grains volume fraction of the (a) α/α' and (b) γ phases from SEM-TKD maps.

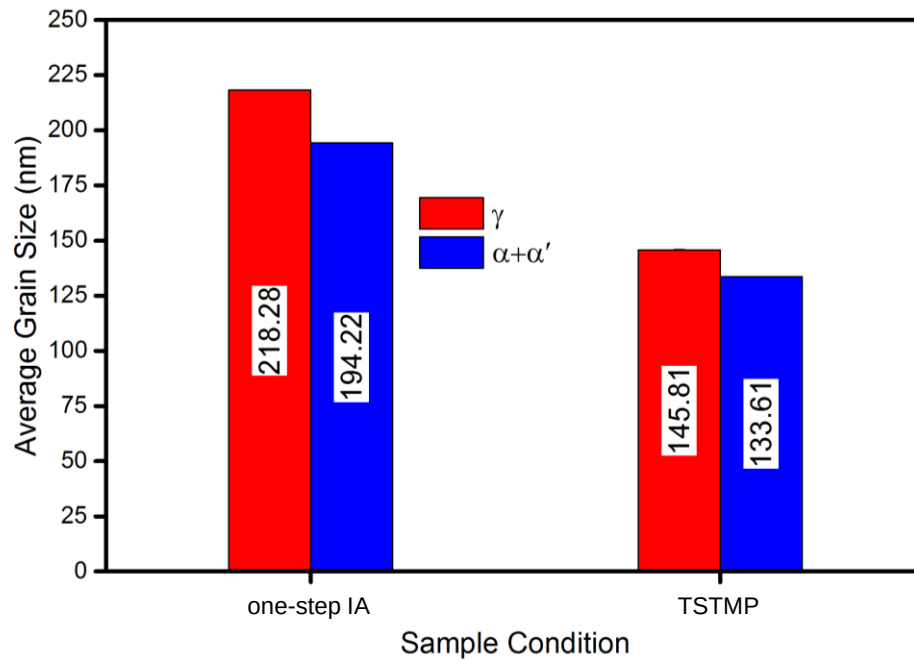


Figure 7.6. Effect of the one-step IA and the TSTMP on the average grain size of the (a) α/α' and (b) γ phases from SEM-TKD maps.

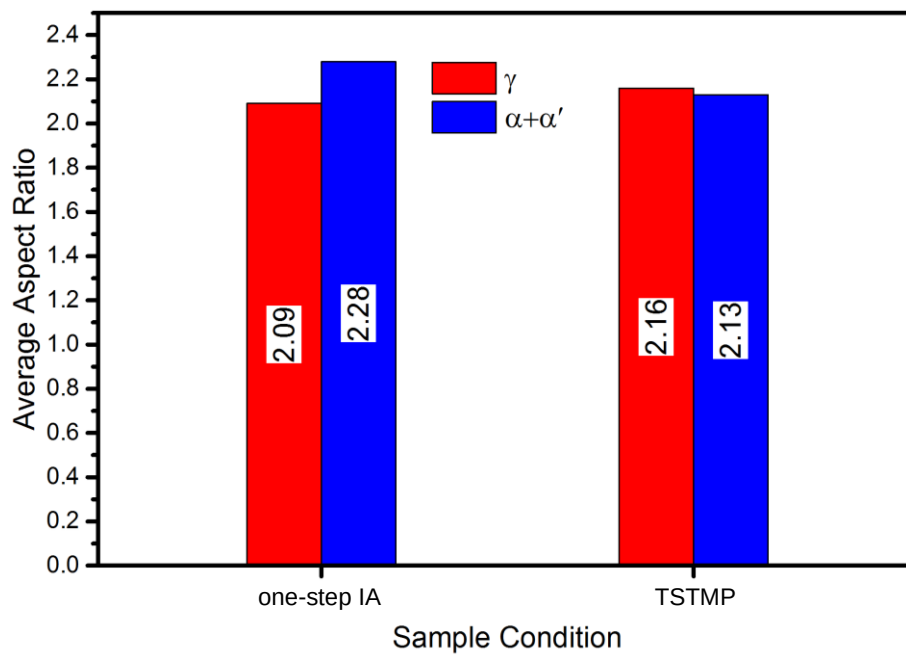


Figure 7.7. Effect of the one-step IA and the TSTMP on the grains' average aspect ratio in the (a) α/α' and (b) γ phases from SEM-TKD maps.

7.2.3 Phase Boundary Orientation Relationship

Figure 7.8 provides the distribution of the phase boundaries between the α/α' and γ phases after the one-step IA and the TSTMP. The green and yellow lines indicate the phase boundaries, respectively, when the phase boundary deviates by less than 10° and more than 10° from KS OR.

Figure 7.9 illustrates the distribution of the α/α' and γ phase boundaries in FeCMnAl steel samples that deviated from the KS orientation relationship after the one-step IA and the TSTMP. High frequency was observed at phase boundaries with 41° to 43° deviation from KS OR. It was 3.9% and 16.7% for the one-step IA and the TSTMP, respectively. After the TSTMP, the sample also displayed a high frequency (10.6%) for phase boundaries with a deviation of 10.5° from the KS OR.

Figure 7.10 depicts the fraction of the phase boundaries between the α/α' and γ with less than 10° deviation from KS OR in FeCMnAl steel samples after one-step IA and the TSTMP. This figure illustrates that the frequency of the phase boundaries with less than 10° deviation from KS OR after the one-step IA is 80.4%, whereas it was 63.7% after the TSTMP (25% less than one-step IA).

The possible reasons for this will be discussed in detail, later. Suffice to mention here that the γ phase partially transformed into α' after annealing at 700°C and quenching to 200°C . Importantly, this TMP might not necessarily produce phase boundaries close to the KS OR, because the cold rolling changed the texture and the grains could rotate. The kinetics of the nucleation and growth of the γ phase might also contribute to this phenomenon.

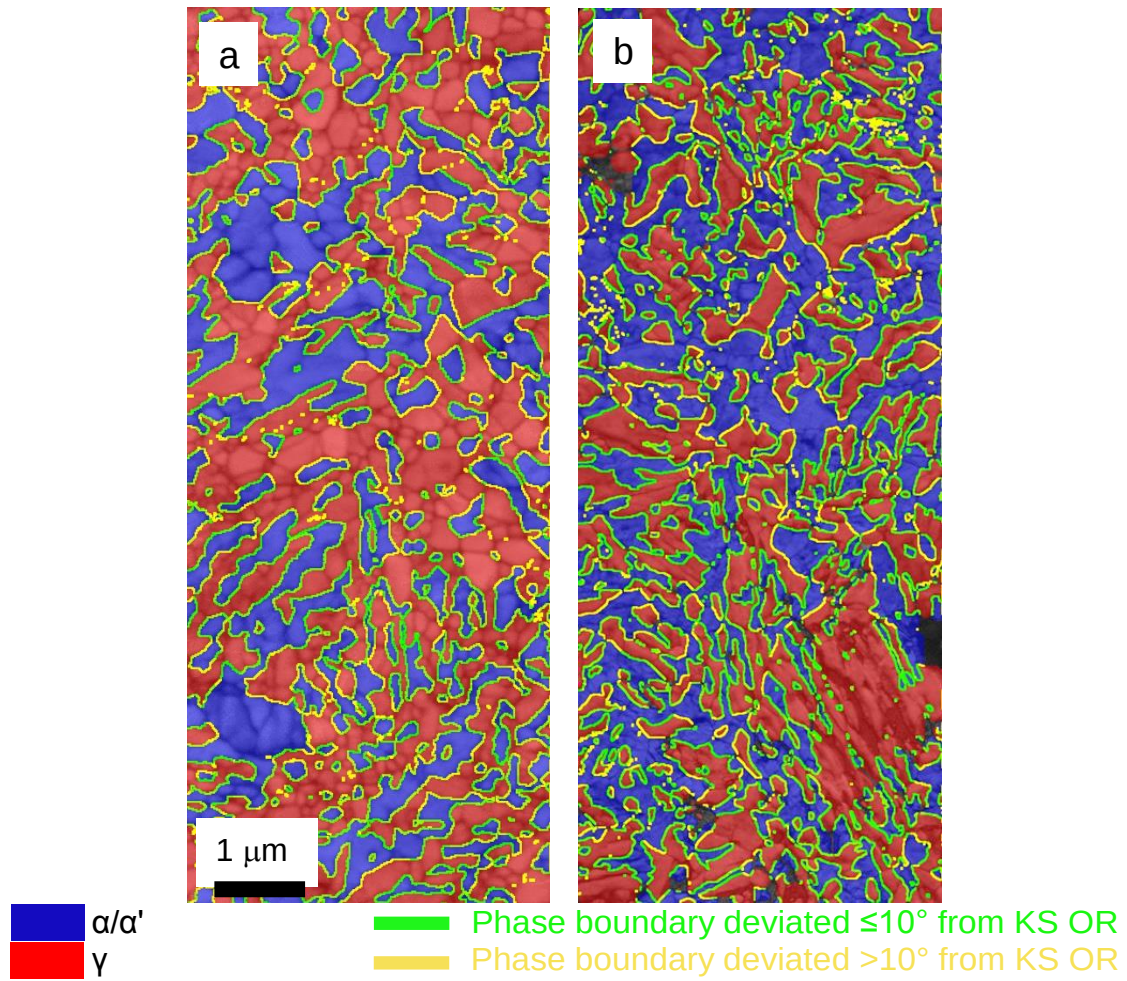


Figure 7.8. SEM-TKD IQ-phase maps of the 1.0 mm FeCMnAl steel after the (a) one-step IA and (b) TSTMP. The phase boundaries between the α/α' and γ phases are shown based on KS OR.

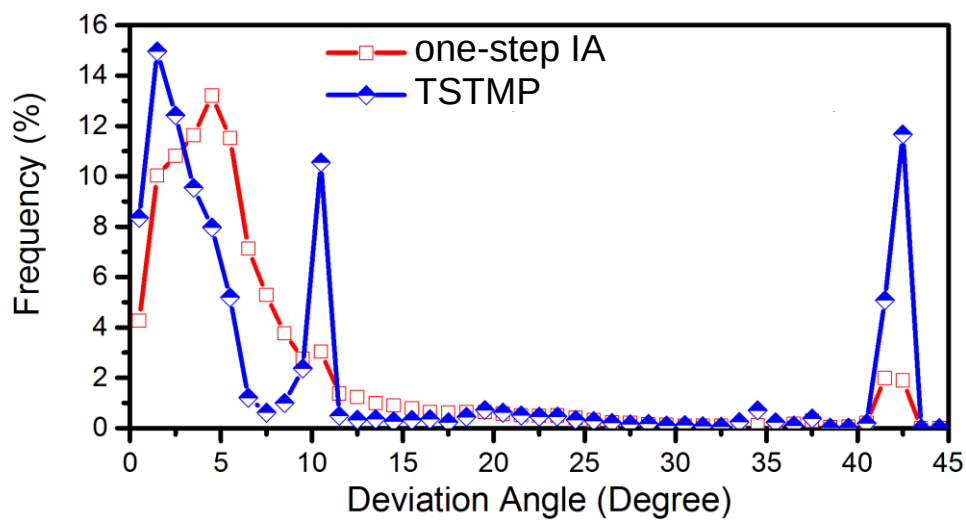


Figure 7.9. The distribution of the α/α' and γ phase boundaries in FeCMnAl steel deviated from the KS orientation relationship after (a) one-step IA and (b) TSTMP.

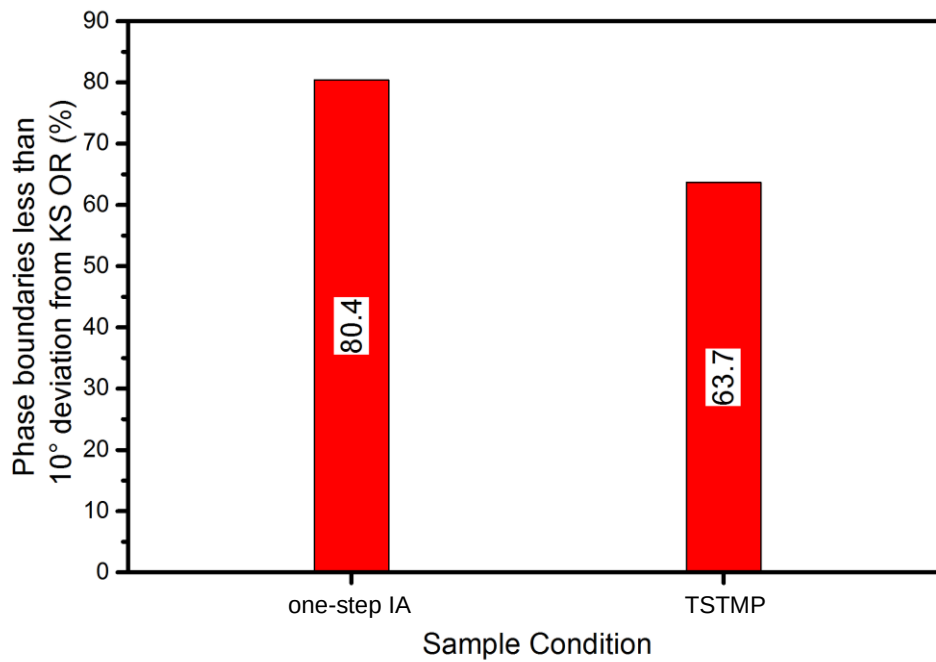


Figure 7.10. The fraction of the phase boundaries between the α/α' and γ with less than 10° deviation from KS OR in FeCMnAl steel samples after one-step IA and TSTMP.

7.2.4 Strain Distribution in the Phases

Two approaches were used to assess the quality and quantity of the strain distribution in the α/α' and γ phases: the LAGBs and KAM analysis. These approaches were used to evaluate the dislocation density inside phases.

7.2.4.1 Low-Angle Grain Boundaries

As a basis for the method, the LAGBs less than 15° were considered. In Appendix 2, the γ and α/α' grain boundaries of the SEM-TKD IQ-maps are shown.

In Figure 7.11, the LAGBs within the γ and α/α' phases of the FeCMnAl steel samples are depicted for both one-step IA and the TSTMP. The LAGBs fractions for the γ and α/α' phases were 77.13% and 71.6%, respectively, after one-step IA, and 82.3% and 89.8% after the TSTMP. The figure indicates that both phases have higher LAGBs after the TSTMP, which means that they had a higher residual stress and dislocation density compared to those after one-step IA.

7.2.4.2 Kernel Average Misorientation

The KAM technique allows one to quantify the average misorientation around a measurement point when compared with a defined set of the nearest neighbours. A KAM value of less than 5° was used in this study.

Figure 7.12 illustrates the average local misorientation of the γ and α/α' phases of the FeCMnAl steel samples after one-step IA and the TSTMP. After one-step IA, the α/α' phase showed an average local misorientation of 0.45° . In the TSTMP, the local misorientation of the α/α' phase increased to 0.63° (a 29% increase). The one-step IA produced a γ phase with an average local misorientation of 0.46° . After the TSTMP, the local misorientation of the γ phase increased by 22% to 0.6° .

Both KAM and LAGB approaches demonstrated an increase in strain distribution after the TSTMP, but KAM showed a greater increase. The results of both approaches showed that after one-step IA, the α/α' phase had a lower strain average than the γ phase, while it was the reverse after the TSTMP.

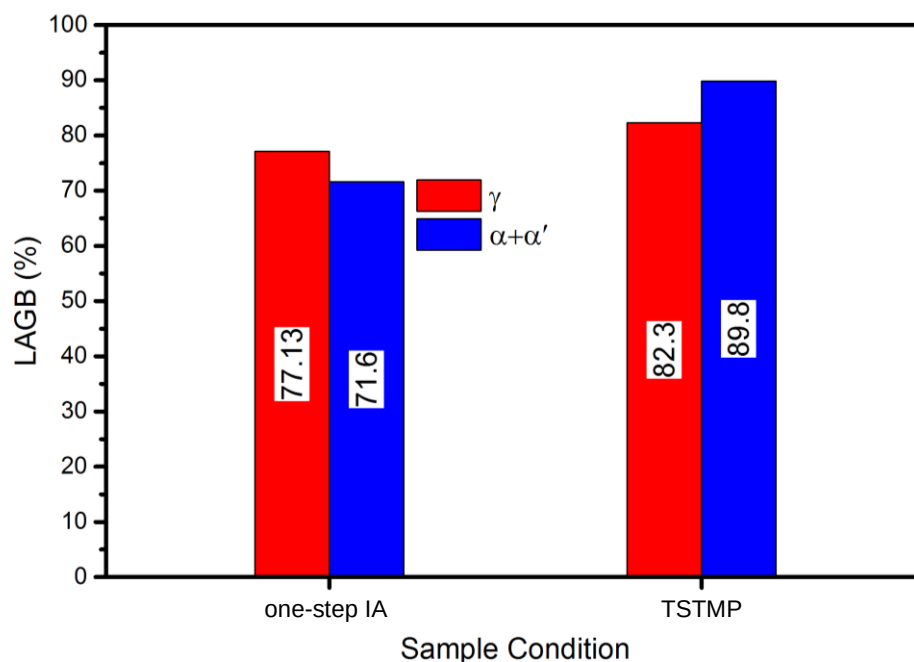


Figure 7.11. Variation of the LAGBs inside the γ and α/α' phases of the FeCMnAl steel samples after the one-step IA and the TSTMP.

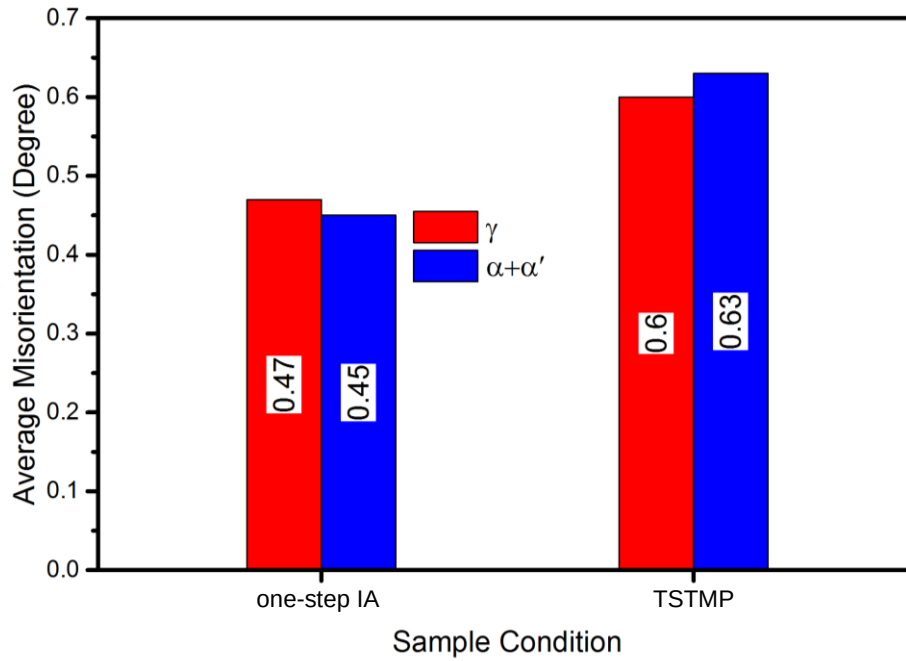


Figure 7.12. The average local misorientation of the γ and α/α' phases of the FeCMnAl steel samples after the one-step IA and the TSTMP.

7.2.5 Texture

The ODF analysis was conducted on the SEM-TKD maps for the purpose of the assessing the texture of the different phases of the samples. The common texture components and fibers of the BCC and FCC metals and alloys were shown in the 6.2.5 section. The process of the rolling BCC metals and alloys develops a fiber texture, as demonstrated by several studies [37, 392–394].

Figure 7.13 illustrates the texture of the α/α' phase in $\varphi_2 = 0^\circ$ and 45° ODF sections in the FeCMnAl Steel samples. Figure 7.13(a) shows that the BCC phase of the AR sample consisted of strong α and ε fibers as well as a weaker γ -fiber. The texture had a $\{001\}\langle 110 \rangle$ component with $f(g) = 12.6$ intensity, which was the result of the deformation of the brass component of the γ phase. As a result of the deformation of the Brass and S components of the γ phase, there was a $(332)[\bar{1}\bar{1}3]$ component ($f(g) = 7.5$) [395, 396].

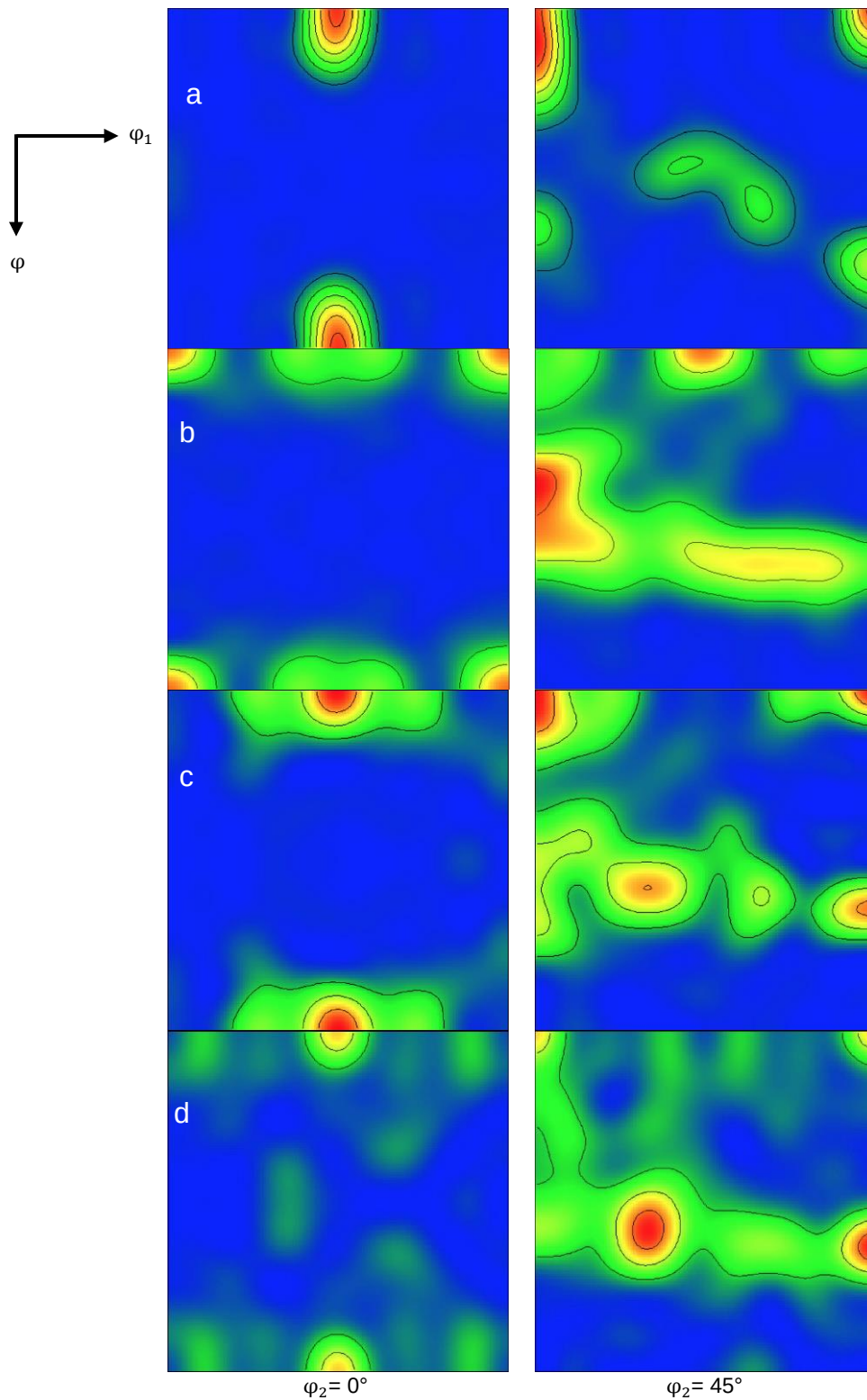


Figure 7.13. The texture of the α/α' phase in $\varphi_2 = 0^\circ$ and 45° ODF sections in the FeCMnAl Steel (a) AR (b) one-step IA, (c) 1.0 mm annealed at 700 °C for 3 min, quenched at 200 °C and rolled 5% in the RD and (d) TSTMP samples. Contour levels = 2x.

Figure 7.13(b) shows that the α/α' phase texture of the one-step IA sample had the α , γ and θ fibers. The maximum intensity could be seen on the $(112)[\bar{1}\bar{1}0]$ component ($f(g) = 6.71$)

whereas the intensity on the (001)[100] component ($f(g) = 5.79$) is weaker. The current results supported both oriented nucleation and selective or oriented growth as mechanisms for recrystallization. $\{001\}\langle 110 \rangle$ and $\{111\}\langle 110 \rangle$ components were texture components within AR sample, and the nucleation in those grains retained the orientation of the former texture during annealing. Consequently, it could be concluded that the oriented nucleation mechanism controlled the texture of those components [397, 398].

Figure 7.13(c) shows the α/α' phase texture of the 1.0 mm sample that was annealed at 700 °C for 3 min, quenched at 200 °C and rolled 5% in the RD. It consisted of the α , γ , ε and θ fibers, the strong Rt-C component ($f(g)=7.75$) and the weaker F ($f(g)=6.4$) and E ($f(g)=4.19$) components.

Figure 7.13 (d) shows the α/α' phase texture of the TSTMP. It consisted of strong γ -fiber and weaker α and ε fibers and has a strong F component ($f(g)=7.05$) and a weaker Rt-C component ($f(g)=5.11$).

The texture of the 1.0 mm sample after annealing at 700 °C for 3 min, quenched at 200 °C was not shown in this figure because the XRD result shows 30.9% of the volume fraction of the γ while the SEM-TKD shows 1%.

Figure 7.14 illustrates the fibers of the α/α' phase in the FeCMnAl Steel samples. It shows how the α/α' phase fibers' intensities changed after the annealing and cold rolling.

Figure 7.15 illustrates the γ phase textures in the one-step IA sample. In Figure 7.15(a), It is found that there were α and τ fibers with maximum intensity on the G component ($f(g) = 24$). In the reverted γ phase, both D and G-T components formed, indicating that twinning of the grain structures with the G texture occurred during the reverse transformation of α' into γ . As indicated by the G component of this sample, partial or complete recrystallization of the γ grains occurred during the IA [25, 399, 400]. In addition, the texture exhibits a strong B ($f(g) = 5.9$).

Figure 7.15(b) shows the γ texture of the TSTMP sample. There was a strong α -fibre in the γ phase with maximum intensity on the brass component ($f(g) = 14.7$). There was a weak component at ($\varphi_1 = 0^\circ$, $\varphi = 30^\circ$ or 60° , $\varphi_2 = 0^\circ$) with the $f(g)=4.59$ time random.

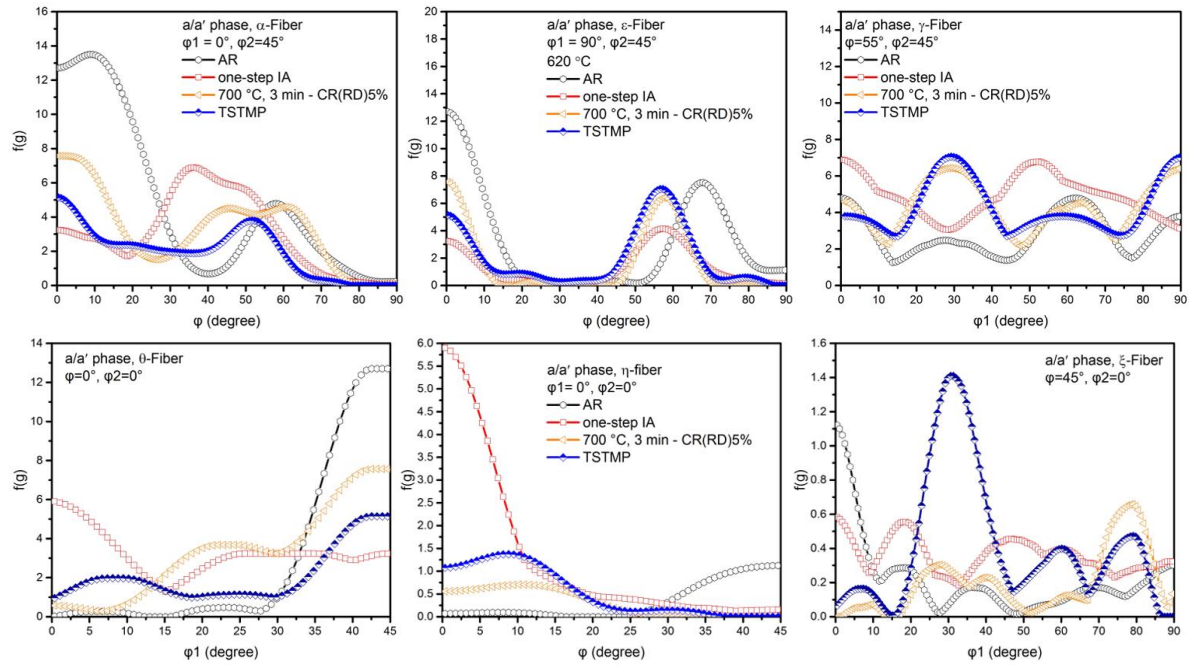


Figure 7.14. The fibers of the α/α' phase in the FeCMnAl steel specimens.

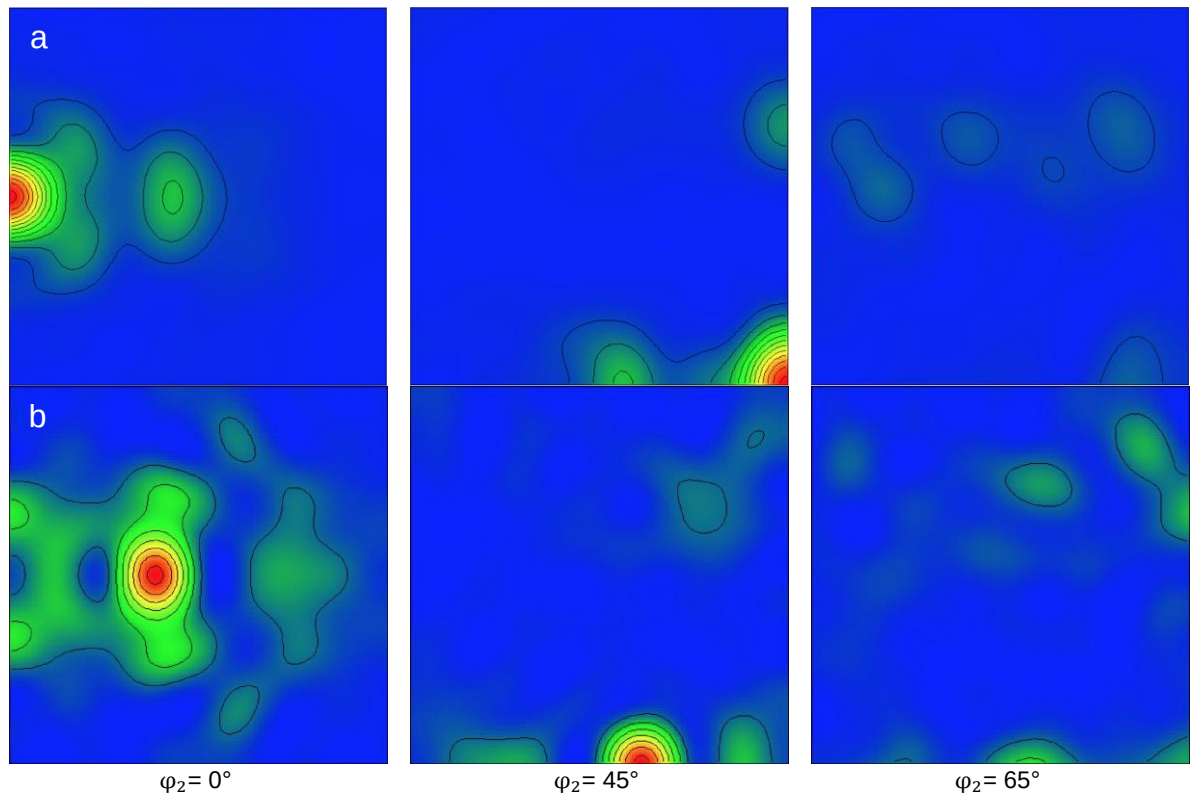


Figure 7.15. The texture of the α/α' phase in $\phi_2 = 0^\circ, 45^\circ$ and 65° ODF sections in the FeCMnAl Steel (a) one-step IA sample and (b) TSTMP sample. Contour levels = $2x$.

Brass component in γ phase was found to result from $\{011\}\langle 110 \rangle$ and $\{332\}\langle 113 \rangle$ components of the α/α' phase [37, 38, 401]. This texture was determined to be the result of the

transformation of the α' into γ . The rapid recovery of the γ in materials with a low SFE resulted in a low driving force for recrystallization.

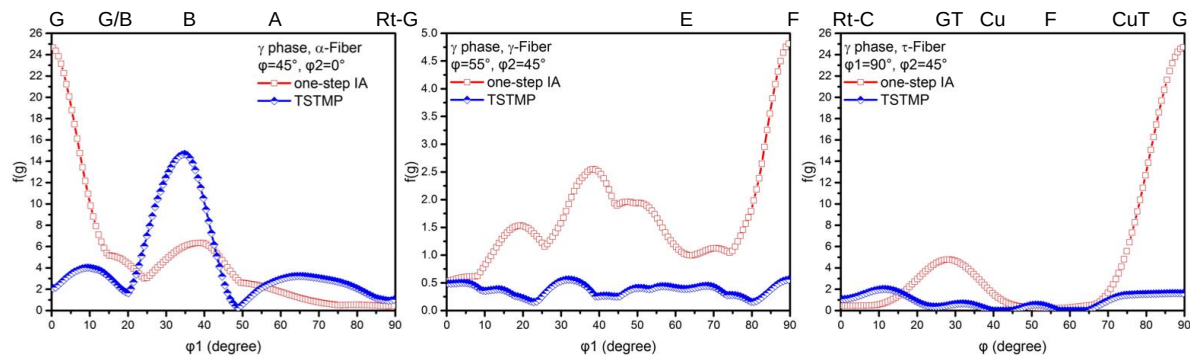


Figure 7.16. The γ phase's fibers of the one-step IA and TSTMP samples.

7.2.6 Summary of the Scanning Electron Microscopy Maps Analyses

The analyses of the SEM-TKD maps of the FeCMnAl steel summarised in Table 7.1.

Table 7.1. Summary of the SEM-TKD maps of the FeCMnAl steel in Chapter 7.

Sample Condition	Phase Volume Fraction %		Average Grain Size nm		Nano Size Grains %		KAM Average Misorientation Degree	
	γ	α/α'	γ	α/α'	γ	α/α'	γ	α/α'
1.0 mm - AR	0.2	99.8						
1.0 mm - One-step IA	59.8	40.2	218	194	32.9	34.7	0.47	0.45
1.0 mm - 700 °C, 3 min	1.0	99.0						
700 °C, 3 min - 5 % CR	0.4	99.6						
700 °C, 3 min - 5 % CR - 650 °C, 10 min	50.7	49.3	145.81	133.61	47.5	46.2	0.60	0.63

Sample Condition	Aspect Ratio (No Preferred Direction)		Aspect Ratio (RD/TD)		LAGB %		Phase Boundaries <10° Deviation from KS OR %
	γ	α/α'	γ	α/α'	γ	α/α'	
1.0 mm - one-step IA	2.09	2.28	1.02	0.98	77.1	71.6	80.4
700 °C, 3 min - 5 % CR - 650 °C, 10 min	2.16	2.13	1.10	1.11	82.3	89.8	63.7

7.3 X-Ray Diffraction

Figure 7.17 illustrates the XRD patterns of the annealed and cold-rolled samples. Figure 7.17(a) shows the AR sample. After annealing the sample at 700 °C for 3 min and quenching to 200 °C (Figure 7.17(b)), the intensity of the γ peaks like $(111)_\gamma$, $(200)_\gamma$, $(220)_\gamma$, $(311)_\gamma$, and $(420)_\gamma$ increased and resulted in 30.9% volume fraction of the γ phase. Annealing for 5 min (Figure 7.17(c)), resulted in a 21.2% of the γ phase volume fraction. Increasing the annealing time to 10 min (Figure 7.17(d)), decreased the volume fraction of the γ phase to 15.7%.

The annealed sample at 700 °C for 3 min was cold-rolled, and thickness was reduced by 5% and 10% (Figures 7.17(e) and (f)). It could be seen that the intensity of the γ peaks decreased further by applying more thickness reduction. The 5% and 10% thickness reduction resulted in 0.73% and 0.43% of the γ phase volume fraction. Figure 7.17(g) shows that the annealing 5% cold-rolled sample at 650 °C for 10 min increased the intensity of the γ phase peaks and its volume fraction to 63.8%.

Table 7.2 summarised the volume fraction of each sample that was calculated from XRD data. The 5% and 10 cold rolled samples analysed twice, once from the surface of the sample and the second time from the middle of the sample. The volume fraction of the γ was calculated, and they were summarised in Table 7.3. It could be seen that the surface and middle of the sample, were representing almost the same volume fraction of the γ .

To investigate the effect of the sample preparation on the XRD results, the annealed 1.0 mm FeCMnAl steel sample at 700 °C for 3 min and quenched at 200 °C was chosen. The sample was prepared with different methods, and the XRD patterns were obtained as shown in Figure 7.18. The results indicated that the intensity of the $(110)_\alpha$ peak was greater in the ground samples. Moreover, the intensity of $(110)_\alpha$ peaks was further increased after ion milling the ground sample for two hours.

The process of grinding caused deformation in the thin layer of the sample surface which led to the transformation of the γ into α' . Electropolishing, on the other hand, removed this deformed layer and restored the surface of the sample. The XRD analysis of the electropolished sample showed that the volume fraction of the γ phase was 21.23%. To investigate the effect of the ion milling on the sample, the electropolished sample was subjected to ion milling for

different durations: 15, 120, and 390 min. The results of the ion milling are presented in Table 7.4, where it could be seen that the volume fraction of the γ increased slightly from 21.23% to 21.53% with increasing ion milling time.

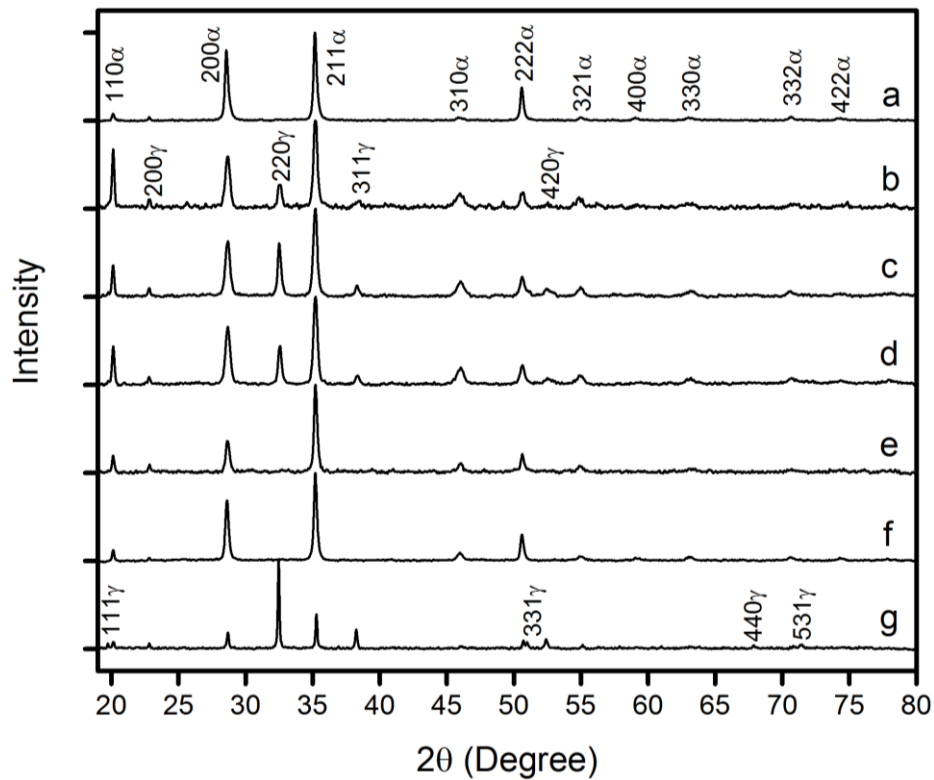


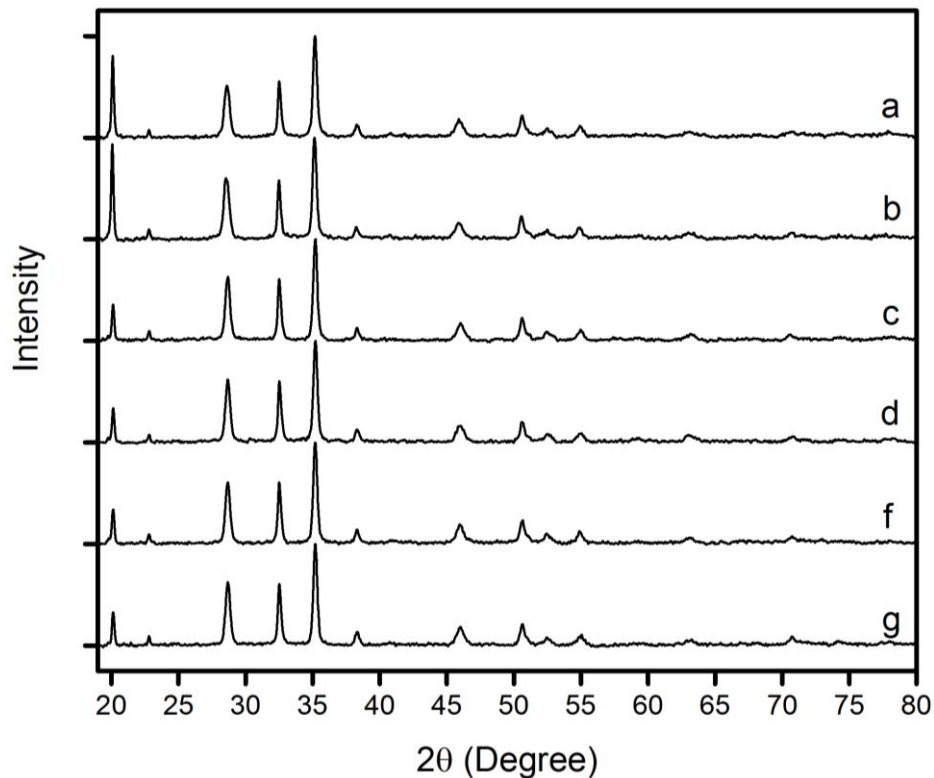
Figure 7.17. X-ray diffraction spectra of the FeCMnAl steel samples; (a) AR sample and the samples were annealed at 700 °C for (b) 3, (c) 5 and (d) 10 min and quenched at 200 °C and then cold rolled (e) 5% and (f) 10% and (g) TSTMP sample.

Table 7.2. Calculated retained γ from XRD results.

		Annealing at 700 °C	Quenched at 200 °C	cold rolled RD	Annealing at 650 °C	Retained γ volume fraction (%)
Initial specimen						2.1
	3 min		X			30.9
	5 min		X			21.2
	10 min		X			15.7
	3 min		X	5%		0.73
	3 min		X	10%		0.43
	3 min		X	5%	10 min	63.8

Table 7.3. Effect of the sampling.

Specimen	Cold-rolling	Surface/Middle	Retained γ volume fraction (%)
Annealed at 700 °C – 3 min, quenched at 200C and cold rolled	5%	surface of the sample	0.73
		middle of the sample	0.92
	10%	surface of the sample	0.43
		middle of the sample	0.43

**Figure 7.18.** Effect of the sample preparation on the X-ray diffraction spectra of the 1.0 mm FeCMnAl steel samples after annealing at 700 °C for 3 min and quenching to 200 °C was (a) ground (b) ground and 120 min PECS, (c) electropolished (d) electropolished and 15 min PECS (e) electropolished and 120 min PECS and (f) electropolished and 390 min PECS.**Table 7.4.** Effect of the sample preparation on the retained γ calculated from the X-ray diffraction spectra of the 1.0 mm FeCMnAl steel samples after annealing at 700 °C for 3 min and quenching to 200 °C.

Sample preparation method	γ volume fraction (%)
Ground	19.74
Ground and 120 min PECS	19.05
Electropolished	21.23
Electropolished and 15 min PECS	21.25
Electropolished and 120 min PECS	21.40
Electropolished and 390 min PECS	21.53

7.4 Mechanical Properties

7.4.1 Tensile Test Data

Figure 7.19 depicts the tensile test results of the one-step IA and TSTMP FeCMnAl samples at room temperature. The mechanical properties of these samples are summarized in Table 7.5. The yield stress of the one-step IA sample was 1104 MPa, which reduced to 1044 MPa in the TSTMP sample. The UTS of the one-step IA sample was 1351 MPa, which was 35 MPa higher than that of the TSTMP sample (1316 MPa). The strength ratio of the TSTMP sample was lower than that of the one-step IA sample, indicating a higher work hardening during plastic deformation. The total elongation of the one-step IA sample was 30.4%, which was 5.7% greater than that of the TSTMP sample. The PSE of the one-step IA sample was 41.1 GPa%, while it was 32.5 GPa% for the TSTMP sample. Therefore, with the exception of the work hardening, all the mechanical properties of the one-step IA sample were higher than those of the TSTMP sample.

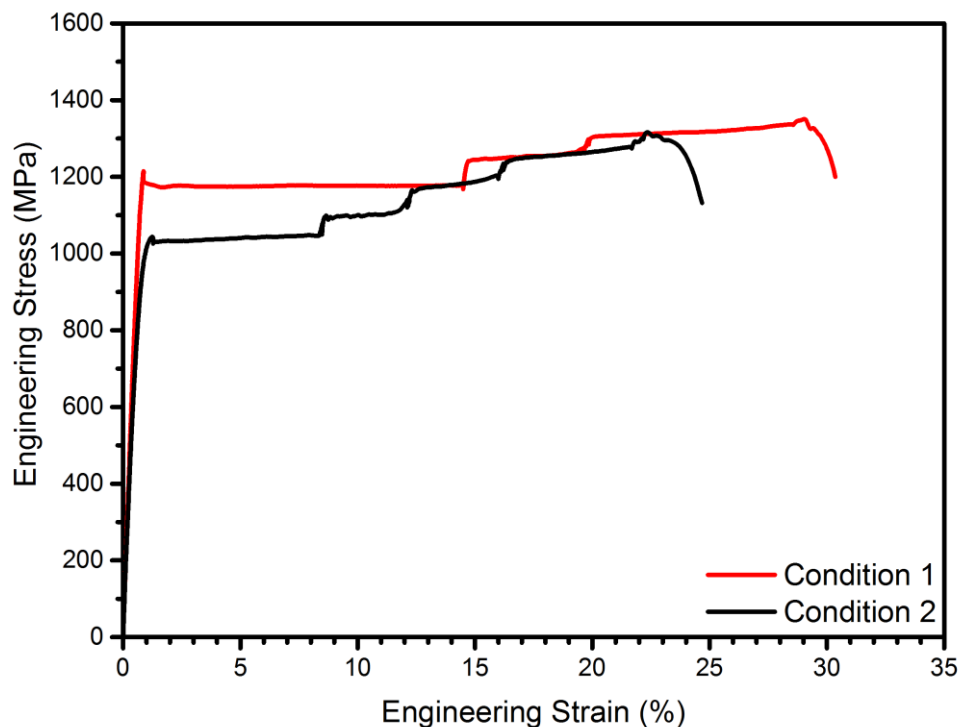


Figure 7.19. Tensile test results of the one-step IA and TSTMP FeCMnAl samples at room temperature. The samples were prepared according to the ASTM E8/E8M and tensile experiments were conducted at a constant strain rate of 10^{-3} sec^{-1} .

Table 7.5. Room-temperature tensile test data of the 1.0 and 0.95 mm FeCMnAl samples.

Condition	Sample name	Heat treatment	Rolling Direction	Heat treatment	Upper Yield Strength MPa	Lower Yield Strength MPa	Tensile Strength MPa	Strength ratio	Total Elongation %	PSE TSxTE MPa.%E
1	One-step IA	650 °C -10 min	-	-	1215	1184	1351	0.876	30.4	41070
2	TSTMP	700 °C -3 min	5% RD	650 °C -10 min	1044	1026	1316	0.780	24.7	32505

7.4.2 Hardness Data

Figure 7.20 depicts the hardness the sample at different conditions. The hardness of the 1.00 mm before annealing was 482.1 ± 1.6 HV. After annealing at 650°C for 10 min the hardness fell to 437.7 ± 6.3 HV.

The 1.00 mm cold rolled sample annealed at 700°C for 3 min and quenched at 200°C which resulted in hardness of 465.5 ± 7.3 HV. The following 5% cold rolling raised the hardness to 542.7 ± 7.5 . The TSTMP sample showed the hardness drop to 415.4 ± 3.7 HV

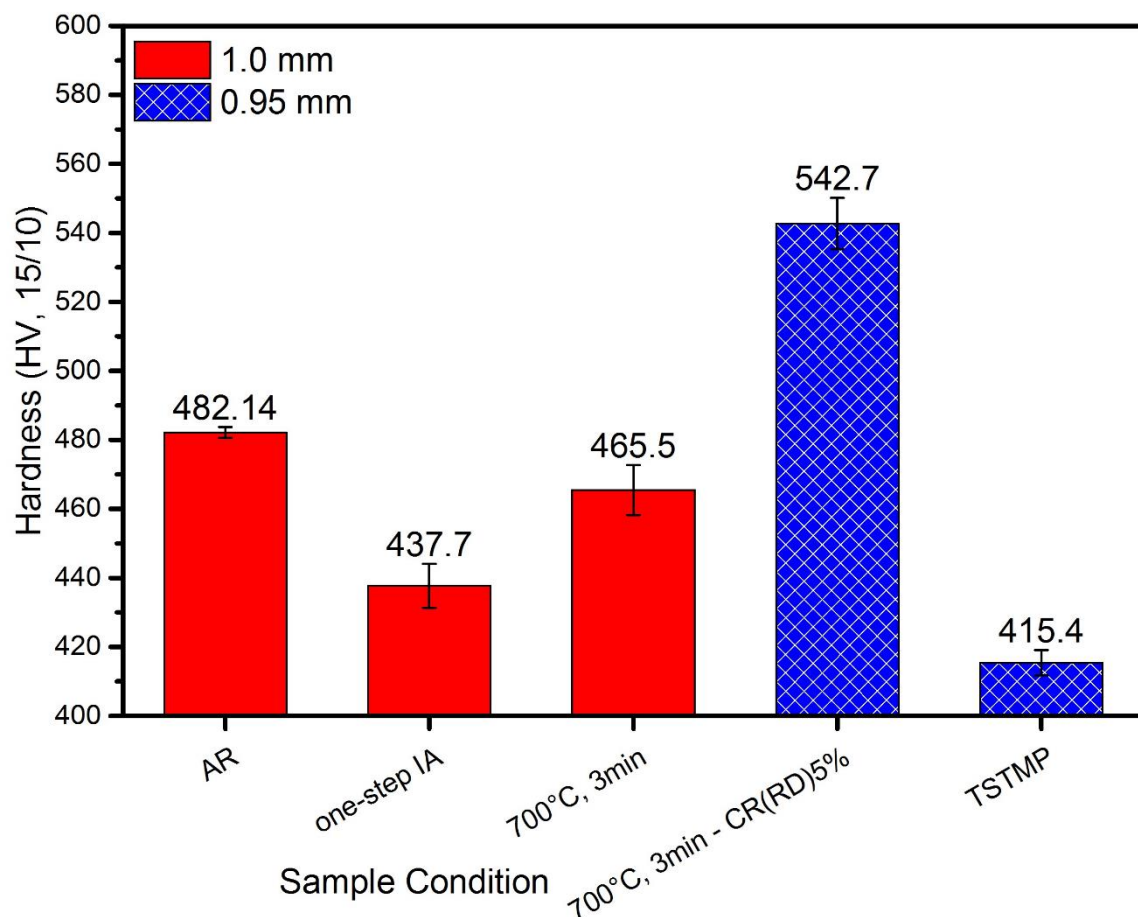


Figure 7.20. Hardness of the FeCMnAl sample after different TMP conditions under a load of 10 kg and for a dwell time of 15 Sec.

7.5 Summary

In this chapter, we thoroughly examined the impact of martensite's characteristics in the initial microstructure before intercritical annealing. To be specific, we engineered initial microstructures in such a way that roughly 70% of the martensite resulted from the transformation during quenching from the austenite phase, while the remaining 30% was produced through the deformation of the retained austenite. It was demonstrated that TSTMP led to a reduction in the average grain sizes of both the γ and α/α' phases, as well as a decrease in the stability of the γ phase, yield stress, UTS, and total elongation when compared to the one-step IA. For an in-depth discussion of the results, refer to Chapter 10.

CHAPTER 8 Effect of the Two-Stage Thermomechanical Process on the FeCMn Steel

8.1 Thermomechanical Procedure

A description of the TSTMP used in this chapter can be found in Figure 8.1. First, AR specimens with α/α' microstructure were intercritically annealed at 620 °C for 10 min and then quenched in water which named the one-step IA. In the next stage, the specimens were cold rolled to a reduction in cross-section of 50% in the RD in one workflow, and 50% in the TD in a second workflow. An IA was performed on the cold rolled samples in the dual phase region (γ and α) at 620 °C for 10 min, followed by water quenching. The cold rolled samples were also annealed at 650 °C for 10 min in a single phase γ region and then quenched in water.

The microstructure and mechanical properties of the FeCMn alloys were studied after the TMP.

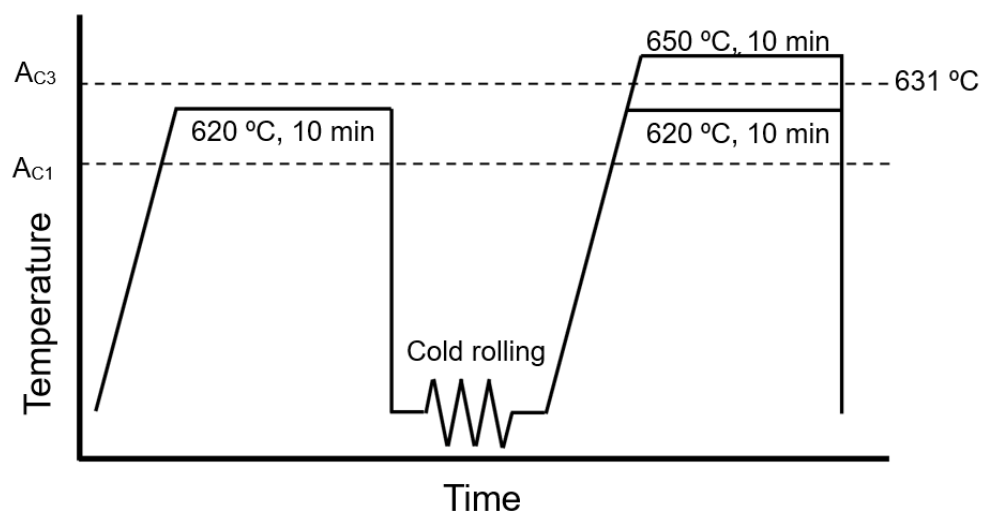


Figure 8.1. The schematic TSTMP of the FeCMn steel.

8.2 Microstructure

In order to collect the SEM-TKD maps, a very large area of the sample was used. These data were analysed in order to determine the phase fraction, the average grain size, the boundaries between phases and grains, the strain distribution, and the texture of the phases. The figures illustrate a selected area.

8.2.1 Scanning Electron Microscopy Maps and Phase Volume Fractions

In this section, the IQ-phase maps of the samples are shown before, at and after 50% cold rolling.

8.2.1.1 Microstructure of the As-received and Annealed FeCMn Steel – 1.4 mm thickness

In Figure 8.2, IQ-phase maps of 1.4 mm FeCMn steel samples have been shown before and after the IA at 620 °C for 10 min. In the AR sample before IA, 96.7% of the sample was composed of the α/α' phase, 3.2% of the ϵ , and 0.1% of the γ .

The IA process transformed α' into γ , resulting in a duplex microstructure composed of the γ and recrystallized α/α' . Austenite grains formed at several locations, including α' lath boundaries, martensite packet boundaries, triple junctions, and parent γ grain boundaries. There were equiaxed γ grains located at packet boundaries, triple junction boundaries, and parent γ boundaries, while lath-type γ grains were found at lath boundaries.

In Figure 8.2, white arrows indicate the locations where some α' -martensite grains have been formed. The formation of α' -martensite might be influenced by a variety of the factors, including (1) quenching the specimens after annealing and (2) transformation of the γ grain to α' -martensite as the SEM-TKD specimens were being prepared.

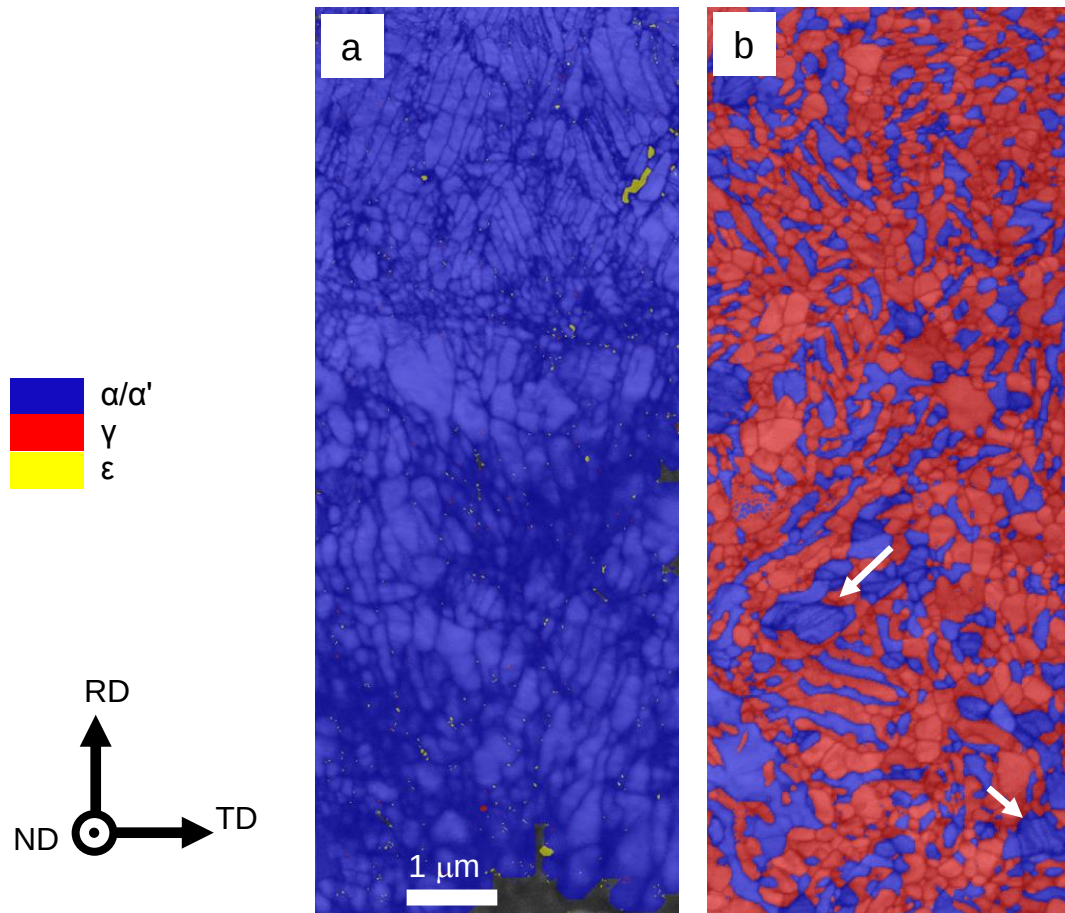


Figure 8.2. SEM-TKD, IQ-phase map 1.4 mm FeCMn steel, (a) the AR and (b) after the IA at 620 °C for 10 min. On the SEM-TKD maps, red represents γ , blue represents α/α' , and yellow represents ϵ . The step size is 10 nm.

8.2.1.2 Microstructure of the Annealed FeCMn Steel after Cold Rolling

After the IA at 620 °C for 10 min, the FeCMn alloy was cold rolled either in the RD or TD to reduce the thickness by 50%. The SEM-TKD, IQ-phase maps of the samples after cold rolling are shown in Figure 8.3. The figure illustrates that the volume fraction of the γ was less than 0.2% and 0.3% after cold rolling in the RD and TD, respectively. Additionally, the sample rolled in the RD had a higher volume fraction of ϵ , as shown in the figure.

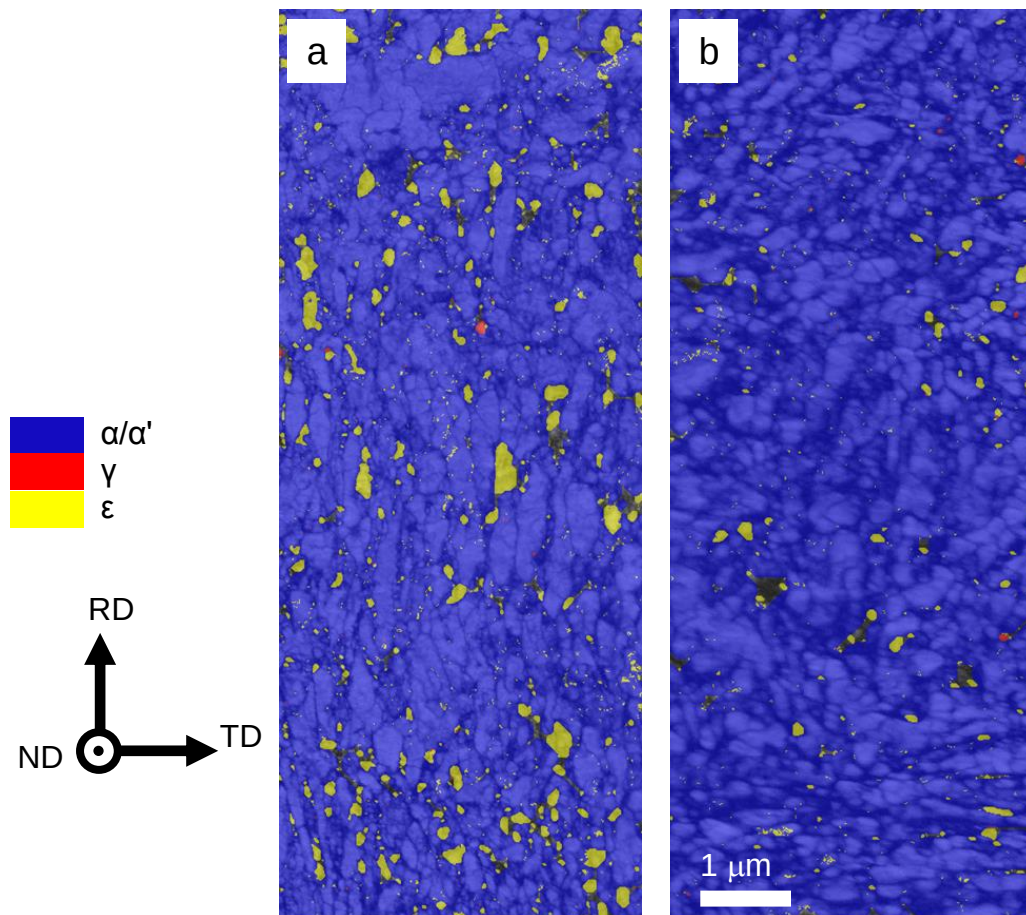


Figure 8.3. SEM-TKD, IQ-phase map FeCMn steel intercritically annealed at 620 °C for 10 min and then (a) rolled in the RD and (b) rolled TD. On the SEM-TKD maps, red represents γ , blue represents α/α' , and yellow represents ϵ . The step size is 10 nm.

8.2.1.3 Microstructure of the FeCMn Steel after Second Intercritical Annealing

Figures 8.4 and 8.5 illustrate the 0.7 mm sample that underwent rolling in the RD and in the TD, followed by the IA at 620 °C (dual phase region) and 650 °C (single phase γ region) for 10 min. The results show that both RD and TD rolled samples had similar volume fraction ratios of the α/α' and retained γ phases after the IA at 620 °C for 10 min.

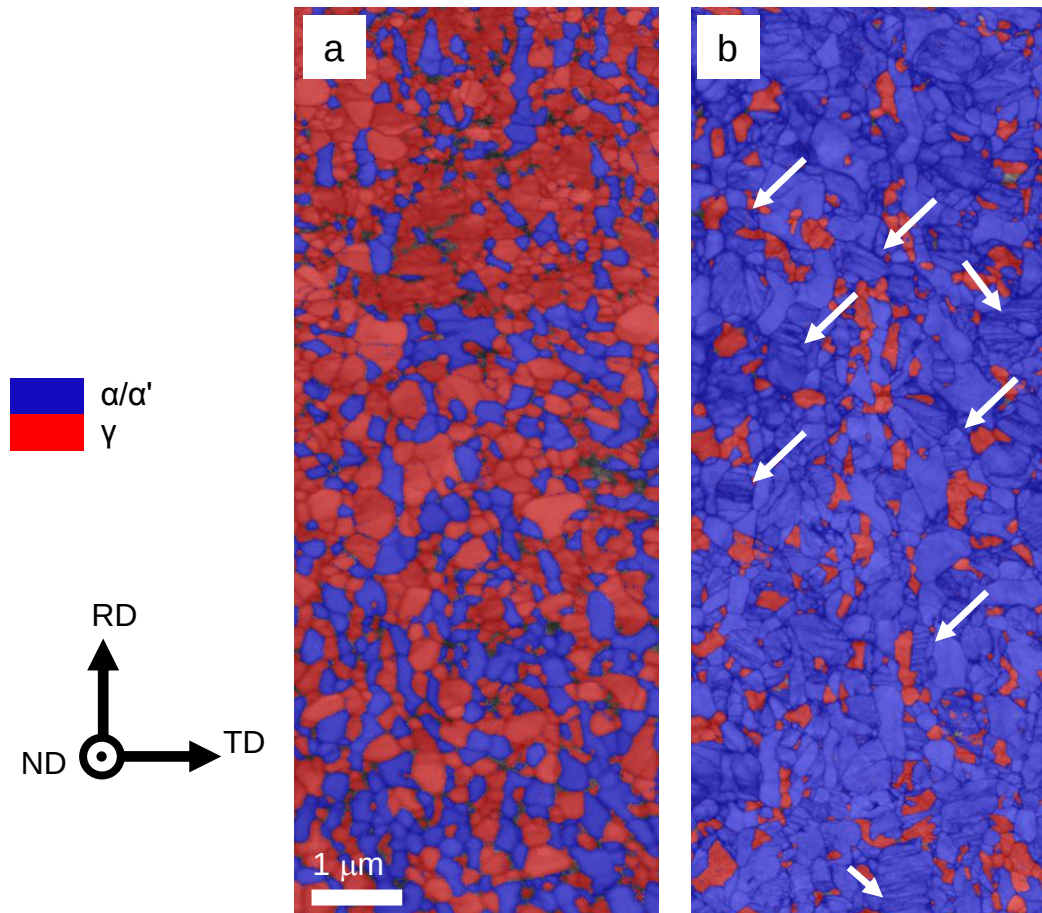


Figure 8.4. SEM-TKD, IQ-phase map FeCMn steel intercritically annealed at 620 °C for 10 min and cold rolled 50% in the RD and then annealed for 10 min at (a) 620 °C and (b) 650 °C. On the SEM-TKD maps, red represents γ , and blue represents α/α' . The step size was 10 nm.

After annealing at 650 °C for 10 min, the entire α/α' and ε phases of the samples transformed into γ , increasing the volume fraction of γ to 100%. As a result, there was a decrease in the average C and Mn contents in the γ grains, leading to reduced stability compared to the γ grains formed during the IA at 620 °C for 10 min. The γ grains with lower Mn content and stability transformed into the α' phase after quenching, as indicated by the white arrows in Figures 8.4 and 8.5 showing the α' lath-shaped grains. Additionally, some γ grains with low stability might transform into martensite during sample preparation using PIPS.

Comparing the RD and TD rolled samples after annealing at 650 °C for 10 min and quenching shows that the TD rolled sample had a higher phase volume fraction of the γ .

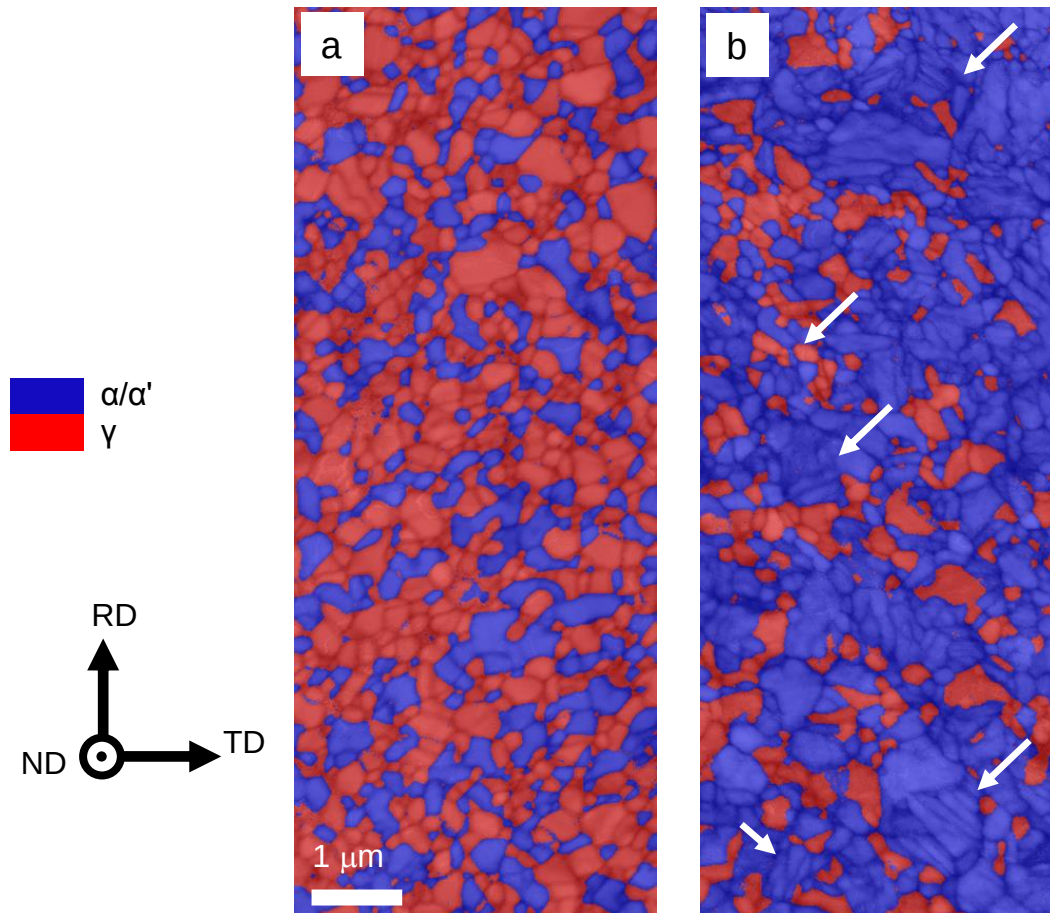


Figure 8.5. SEM-TKD, IQ-phase map FeCMn steel intercritically annealed at 620 °C for 10 min and cold rolled 50% transversely and then annealed for 10 min at (a) 620 °C and (b) 650 °C. On the SEM-TKD maps, red represents γ , and blue represents α/α' . The step size was 10 nm.

Figure 8.6 illustrated the effect of the annealing and the rolling in RD or TD on the phase volume fraction of the γ , α/α' , and ϵ from SEM-TKD maps. The AR sample had 0.1%, 96.7% and 3.2% of the γ , α/α' and ϵ phases, respectively. The one step IA, IA at 620 °C for 10 min, increased the volume fraction of the γ and α/α' phases to 62% and 38%, respectively.

The 0.7 mm cold rolled sample in the RD (Rolled RD) had 0.2%, 93.5% and 6.3% of the γ , α/α' and ϵ phases, respectively. The IA at 620 °C for 10 min, increased the volume fraction of the γ and α/α' phases to 60.1% and 39.9%, respectively. The annealing of the 0.7 mm RD cold rolled sample at 650 °C for 10 min, changed the volume fraction of the γ and α/α' phases to 9.5% and 90.5%, respectively.

The 0.7 mm cold rolled sample in the TD (Rolled TD) had 0.3%, 95.3% and 4.4% of the γ , α/α' and ϵ phases, respectively. The IA at 620 °C for 10 min, increased the volume fraction of the γ and α/α' phases to 61.6% and 38.4%, respectively. The annealing of the 0.7 mm TD

cold rolled sample at 650 °C for 10 min, changed the volume fraction of the γ and α/α' phases to 22.5% and 77.5%, respectively.

As Figure 8.6 shows the γ volume fraction of the TD rolled sample was 2.3 time higher than the RD rolled sample after annealing at 650 °C for 10 min.

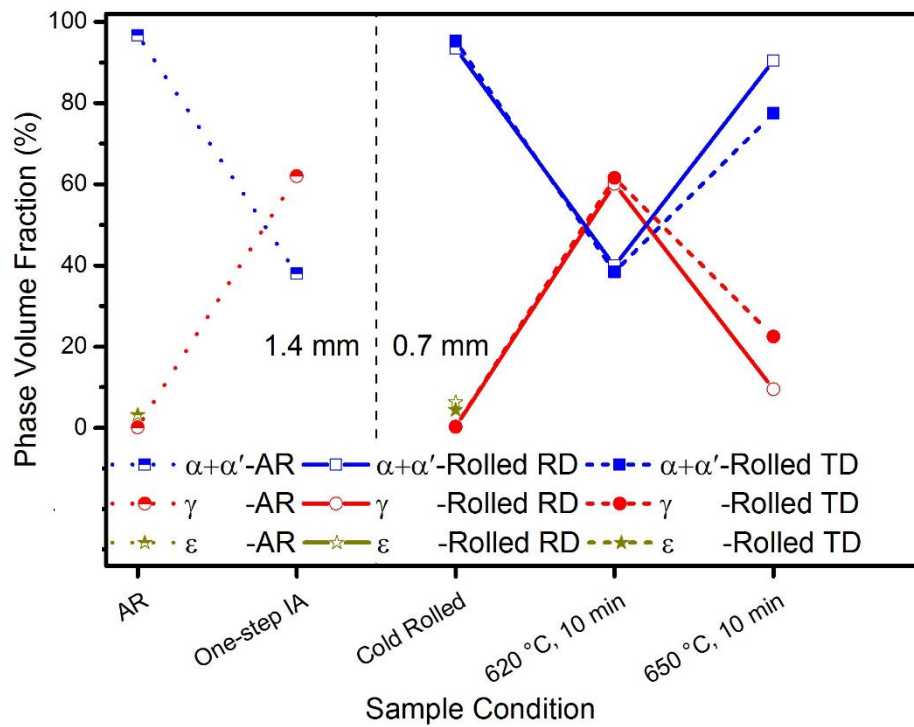


Figure 8.6. Effect of the sample condition on the phase volume fraction of the γ , α/α' , and ϵ phases from SEM-TKD IQ-maps.

8.2.2 Grain Size

Figures 8.7, 8.8 and 8.9 illustrate the Effect of the first annealing at 620 °C, cold rolling in the RD and TD directions and the second annealing at 620 °C and 650 °C on the grain size distribution of the (a) γ , (b) α/α' , and (c) ϵ from SEM-TKD IQ-maps.

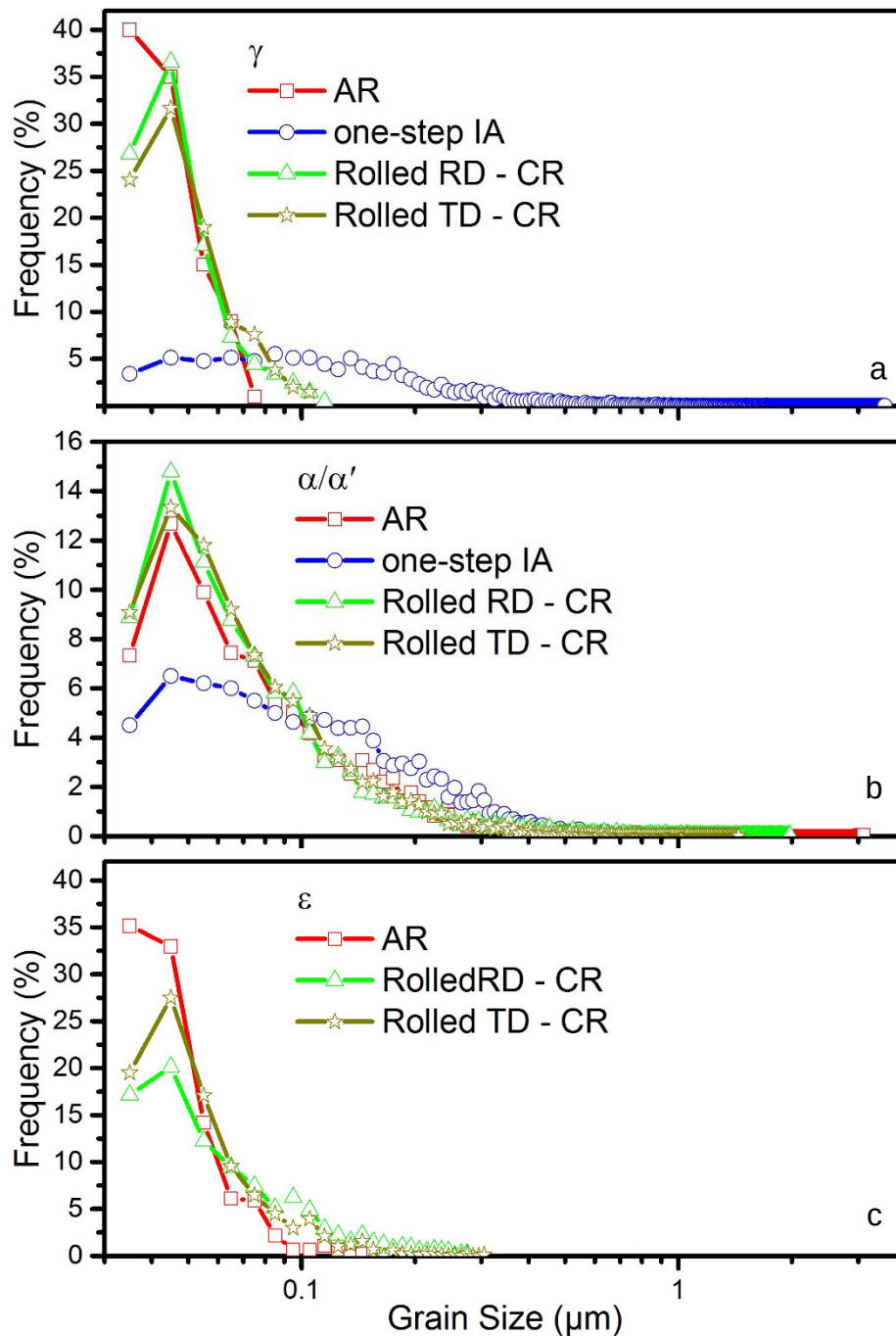


Figure 8.7. Effect of the cold rolling on the grain size distribution of the (a) γ , (b) α/α' , and (c) ϵ from SEM-TKD IQ-maps.

In Figure 8.7 (a), the frequency of the nano-sized γ grains (grains ≤ 100 nm) in the AR sample was 100%. However, after the IA at 620 °C for 10 min, this frequency decreased to 33.8% due to the nucleation and growth of the γ grains, which increased the frequency of the grains larger than 100 nm and decreased the frequency of the grains smaller than 100 nm. Subsequent cold rolling in both RD and TD directions led to the transformation of the large γ grains into α' and ϵ phases. The frequency of the nano-sized grains in the Rolled RD and Rolled TD samples was 98.1% and 96.9%, respectively.

In Figure 8.7 (b), it is shown that the frequency of the nano-sized α/α' grains in the AR sample was 55%. However, after the IA at 620 °C for 10 min, this frequency decreased to 38% due to the recovery, recrystallization, nucleation, and growth of the α/α' grains, resulting in an increase in the frequency of the grains larger than 100 nm. The subsequent cold rolling in RD and TD increased the volume fraction of the lath-shaped α' and deforms the α grains. The frequency of the nano-sized α/α' grains in the Rolled RD and Rolled TD samples was 62.5% and 62.3%, respectively.

In Figure 8.7 (c), the distribution of ϵ grains in the cold rolled samples is presented. The frequency of the nano-sized grains in the AR sample was 97.1%. After cold rolling in RD and TD, the frequency of the nano-sized grains decreased to 77.7% and 87.5%, respectively.

In Figure 8.8, the grain size distribution of the γ and α/α' phases in the 0.7 mm Rolled RD sample is shown before and after the second annealing. The figure indicates that after the IA at 620 °C for 10 min, the frequency of the nano-sized grains in the γ and α/α' phases was lower compared to that after annealing at 650 °C for 10 min.

During the IA at 620 °C for 10 min, γ grains nucleated and grew, which resulted in a decrease of its nano-sized grains' frequency to 32.2%. Similarly, the α/α' grains underwent recovery, recrystallization, nucleation, and growth, leading to an increase in the number of the grains larger than 100 nm. Consequently, the nano-sized grains' frequency of the α/α' after the IA at 620 °C for 10 min was 34%.

After annealing the 0.7 mm Rolled RD sample at 650 °C for 10 min, grains nucleated and grew in the single phase γ region. Upon quenching, the large and low-stable γ grains

transformed into α and lath-shaped α' grains, resulting in an increase in the nano-sized grains' frequency of the γ and α/α' phases to 54.1% and 39.1%, respectively.

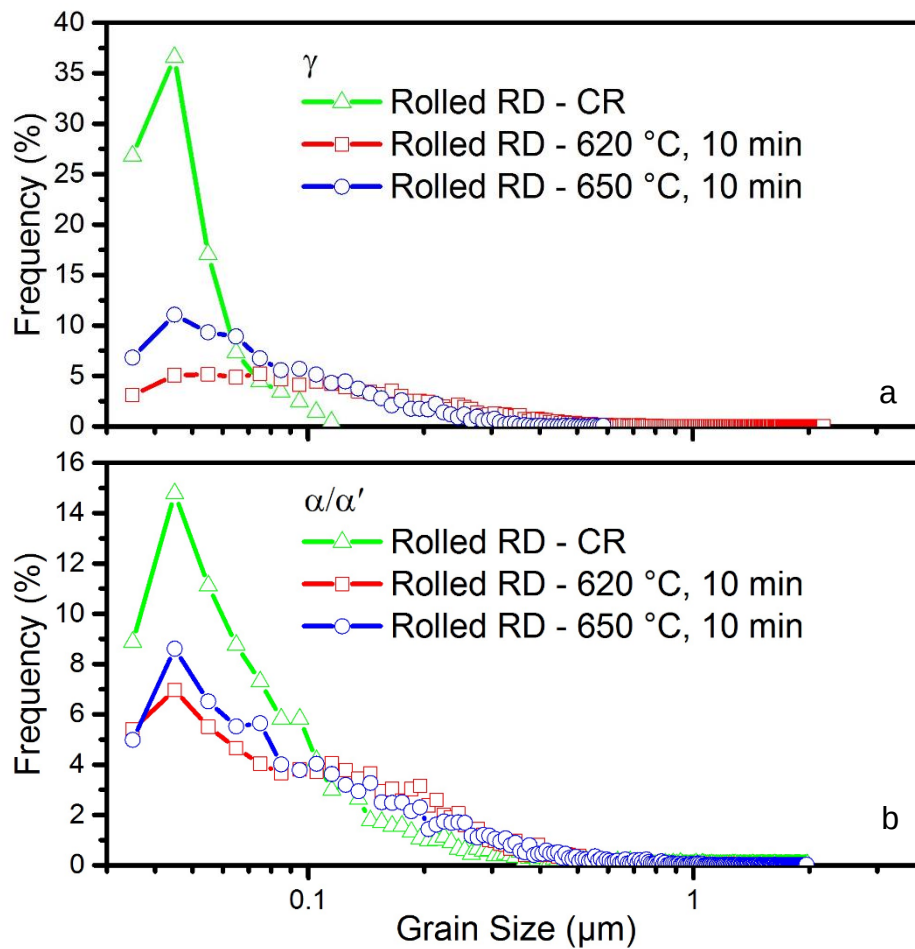


Figure 8.8. Effect of the second annealing of 0.7 mm Rolled RD specimens on the grain size distribution of the (a) γ and (b) α/α' from SEM-TKD IQ-maps.

The grain size distribution of the γ and α/α' phases in the 0.7 mm Rolled TD sample before and after the second annealing is presented in Figure 8.9. Similar to the Rolled RD sample, the frequency of the nano-sized grains in the γ phase was lower in the sample annealed at 620 °C for 10 min (31.6%) compared to the sample annealed at 650 °C for 10 min (44.3%). However, the frequency of the nano-sized grains in the α/α' phase was similar for both samples, with a frequency of 37.7% for the sample annealed at 620 °C and 39.6% for the sample annealed at 650 °C.

The impact of the first annealing at 620 °C, cold rolling in both the RD and TD directions, and the second annealing at 620 °C and 650 °C on the frequency of the nano-sized grains in the γ , α/α' , and ϵ phases is shown in Figure 8.10.

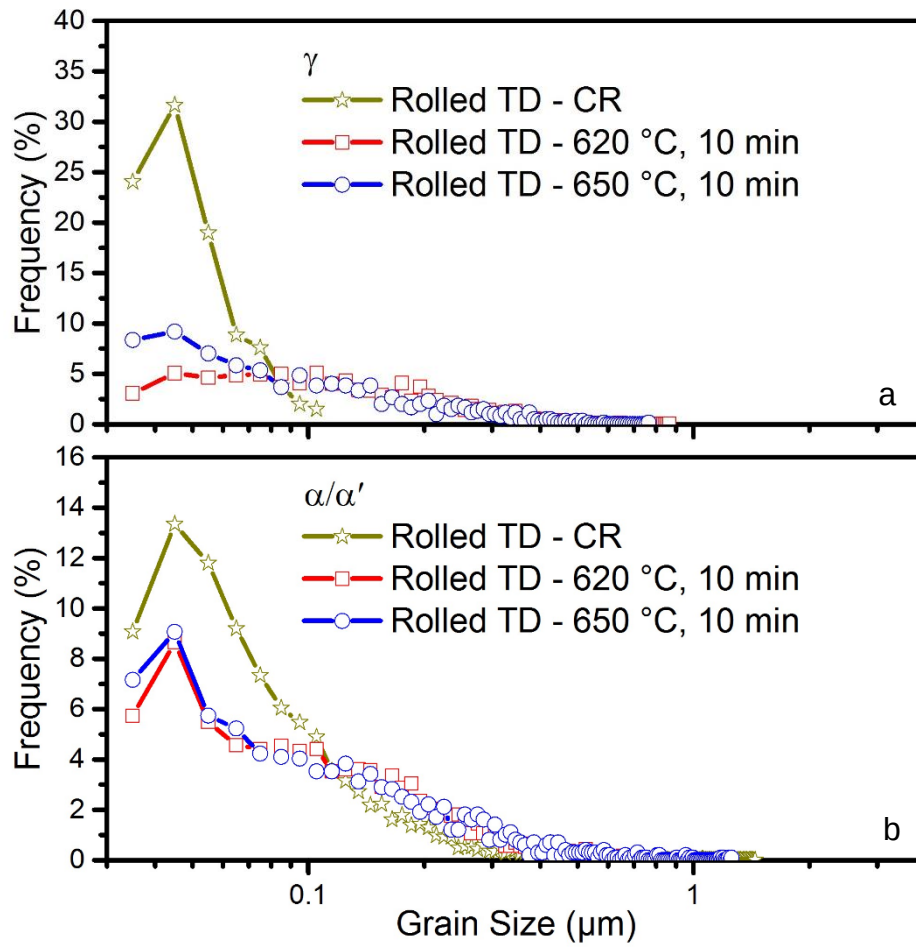


Figure 8.9. Effect of the second annealing of 0.7 mm Rolled TD specimens on the grain size distribution of the (a) γ and (b) α/α' phase from SEM-TKD IQ-maps.

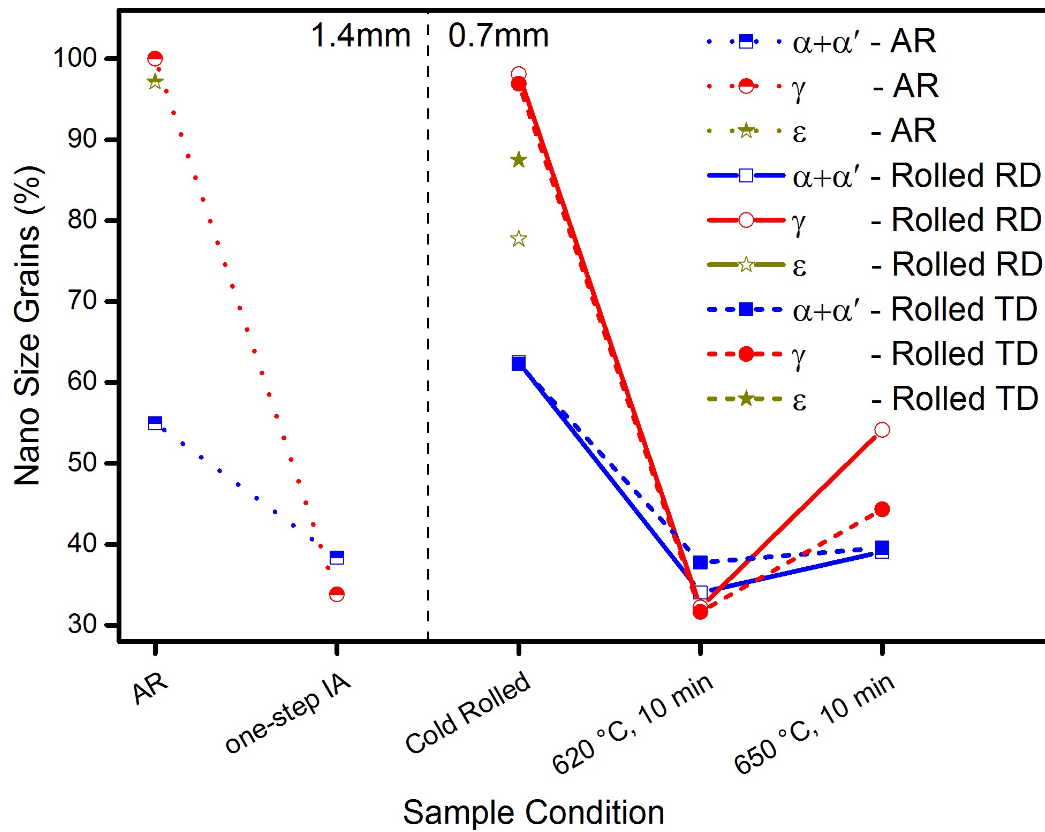


Figure 8.10. Effect of the sample condition on the nano-size grains frequency of the γ , α/α' , and ϵ from SEM-TKD IQ-maps.

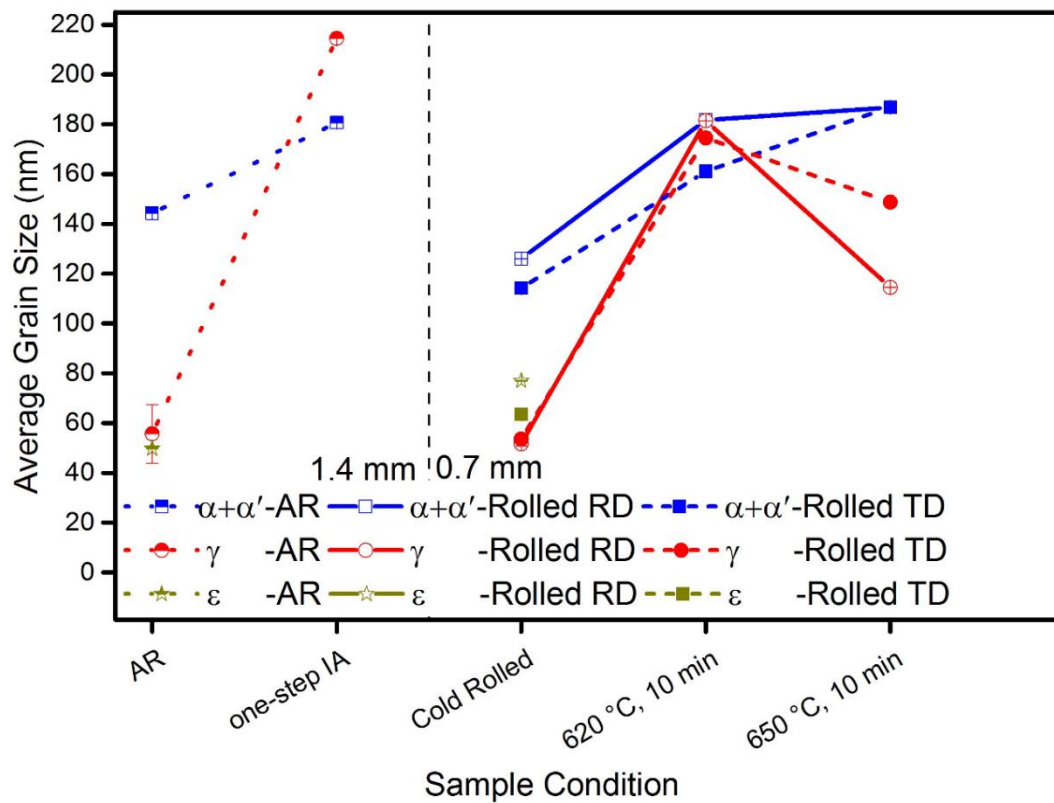


Figure 8.11. Effect of the sample condition on average grain size of the γ , α/α' , and ϵ from SEM-TKD maps.

In Figure 8.11, the impact of the first annealing at 620 °C, cold rolling in the RD and TD directions, and the second annealing at 620 °C and 650 °C on the mean grain size of the γ , α/α' , and ϵ is depicted. The average grain size of the γ , α/α' , and ϵ in the AR sample was 55.7 ± 11.8 nm, 144.3 ± 0.1 nm, and 49.8 ± 0.1 nm, respectively. After the IA at 620 °C for 10 min, the average grain size of the γ and α/α' phase in the 1.4 mm sample increased to 214.5 ± 0.1 nm and 180.7 ± 0.1 nm, respectively.

The α/α' average grain size in the 0.7 mm Rolled RD sample was 126 ± 0.1 nm, while in the 0.7 mm Rolled TD sample, it was 114.2 ± 0.03 nm. After the IA at 620 °C for 10 min, the α/α' average grain size increased to 181.8 ± 0.1 nm and 161.1 ± 0.1 nm in the Rolled RD and Rolled TD samples, respectively. By increasing the annealing temperature to 650 °C, the average grain size in the annealed Rolled RD and Rolled TD samples increased to 186.8 ± 0.01 nm and 187 ± 0.3 nm, respectively. As mentioned, the average α/α' grain size of the 0.7 mm Rolled RD sample was 11% smaller than that of the 0.7 mm Rolled TD sample after the IA at 620 °C. This ratio was similar to the samples' grain sizes before IA.

The average grain size of the γ was very similar between the 0.7 mm Rolled RD and Rolled TD samples in the cold rolled state (51.7 ± 0.9 nm and 53.6 ± 0.4 nm, respectively) and after the IA at 620 °C (181.5 ± 0.1 nm and 174.5 ± 0.1 nm, respectively). However, increasing the annealing temperature to 650 °C led to a decrease in the average grain size of the γ to 114.5 ± 0.1 nm and 148.71 ± 0.4 nm in the annealed 0.7 mm Rolled RD and Rolled TD samples, respectively.

Figure 8.12 illustrates the effect of the first IA at 620 °C, cold rolling in the RD and TD directions and the second annealing at 620 °C and 650 °C on the grains aspect ratio of the γ , α/α' , and ϵ phases.

The aspect ratio of the γ and α/α' phases of the one-step IA sample was 1.95 and 2.15, respectively. The γ and α/α' phases after the RD and TD cold rolling and following annealing had lower grains aspect ratio.

The γ aspect ratio of the Rolled RD and Rolled TD samples after the IA at 620 °C was 1.76 and 1.74 (almost the same), respectively. This ratio increased to 1.90 and 1.86 in the Rolled RD and Rolled TD samples after annealing at 650 °C, respectively.

The α/α' phase aspect ratio of the Rolled RD and Rolled TD samples after the IA at 620 °C was 2.01 and 1.97, respectively. This ratio decreased to 1.90 and 1.80 in the Rolled RD and Rolled TD samples after annealing at 650 °C, respectively.

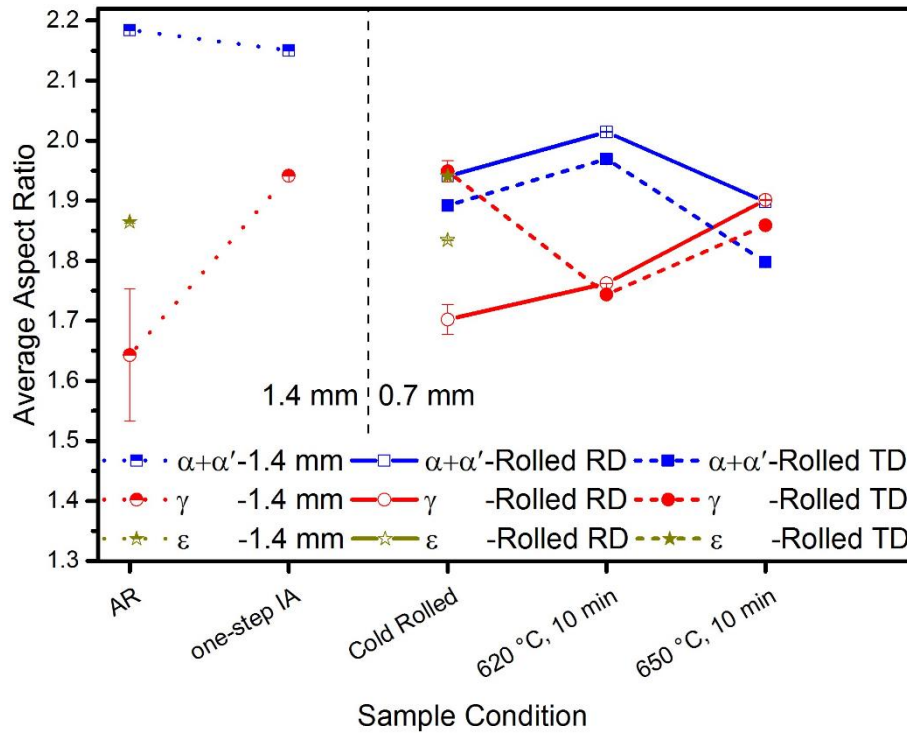


Figure 8.12. Effect of the sample condition on the grain aspect ratio of the γ , α/α' , and ϵ from SEM-TKD IQ-maps.

8.2.3 Phase Boundary Orientation Relationship

In Figures 8.13, 8.14 and 8.15, phase boundaries have been illustrated between the γ and α/α' phases of the FeCMn steel samples. A phase boundary was indicated by a green line if it deviated by less than 10° from the KS OR, while a yellow line if it deviated by more than 10° . These figures used to extract the histogram distribution of the γ and α/α' phases boundaries which is shown in following figures.

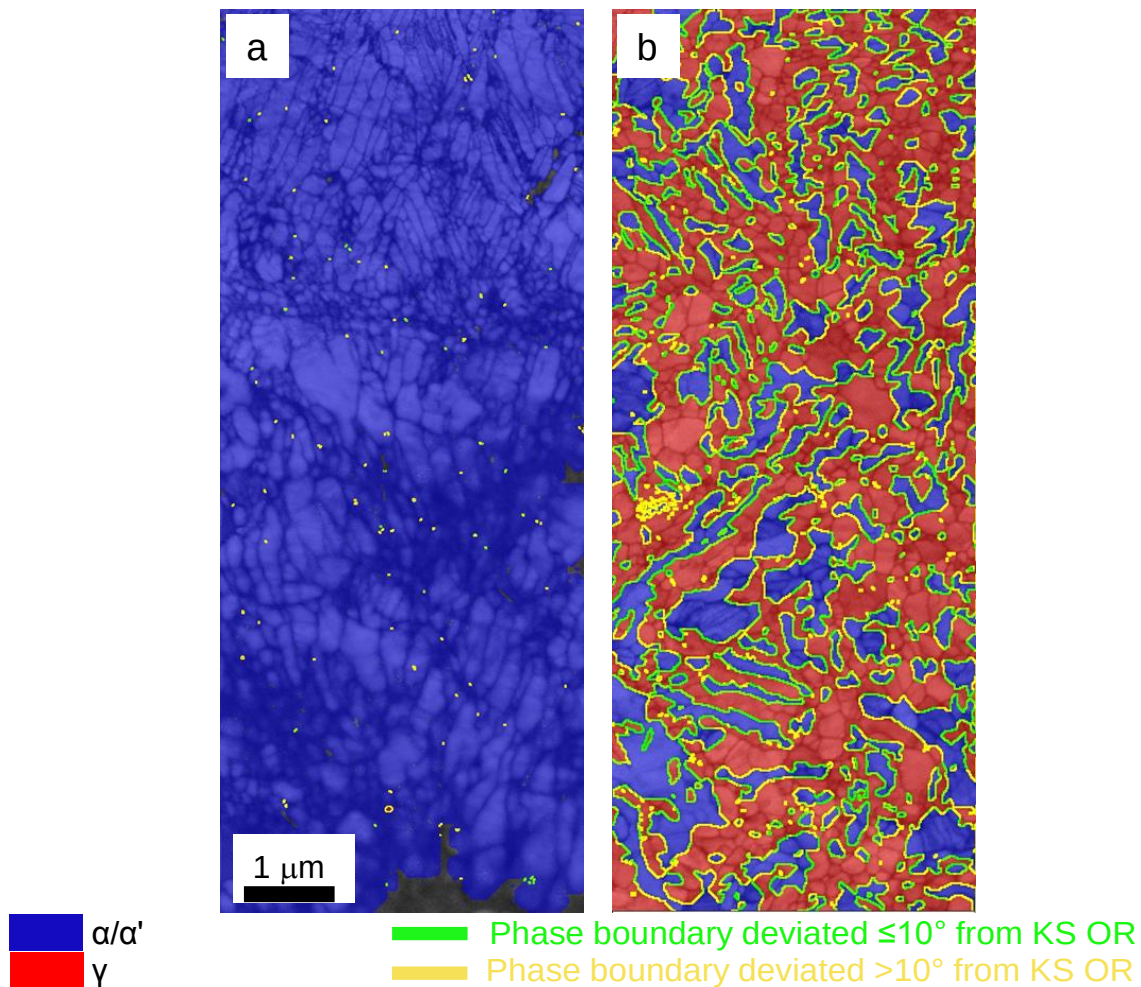


Figure 8.13. SEM-TKD, IQ-phase map 1.4 mm FeCMn steel, (a) the AR and (b) one step IA at 620 °C for 10 min. The phase boundaries between the γ and α/α' phases are shown based on KS OR.

Figure 8.16 illustrates the histogram distribution of the γ and α/α' phases boundaries in the samples that deviated from the KS orientation relationship. Figure 8.16 (a) shows the effect of the IA and the following RD and TD cold rolling on the γ and α/α' phase boundaries' deviation from KS OR in the AR sample. It can be seen in the AR sample; there was 26% frequency of the deviation from KS OR at 35 degrees. This peak disappeared after the IA and appeared after the following RD and TD cold rolling with 5% and 13% amounts, respectively.

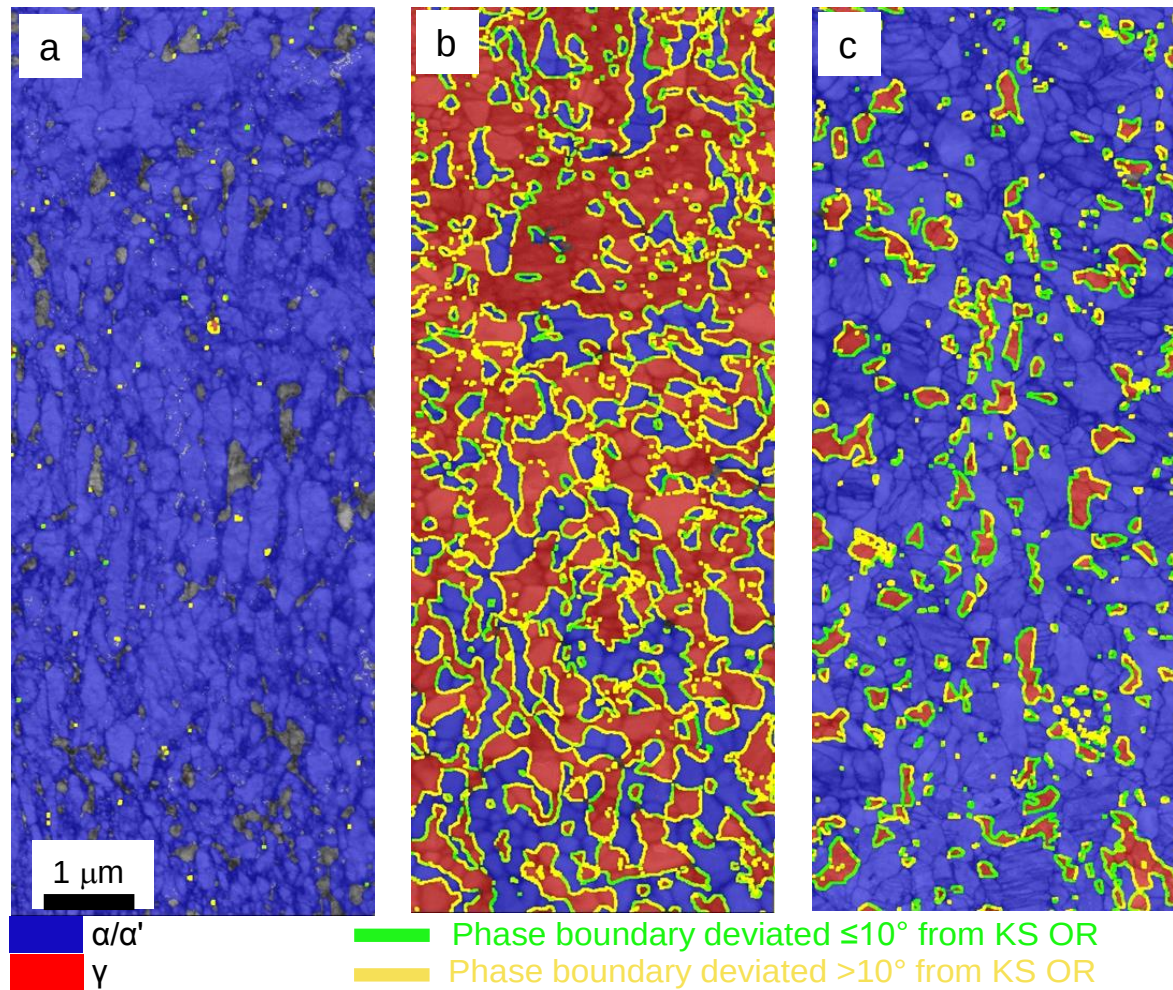


Figure 8.14. SEM-TKD, IQ-phase map the 0.7 mm Rolled RD FeCMn steel samples in the (a) cold rolled condition, annealed for 10 min at (b) 620 °C and (c) 650 °C. The phase boundaries between the γ and α/α' phases are shown based on KS OR.

Figure 8.16 (a) shows that in the 1.4 mm and 0.7 mm Rolled RD and Rolled TD samples. There were noticeable peaks at around 19° and 25° that they were disappearing after annealing. The frequency of the deviation from KS OR between 41° to 43° was 2.1% and 1.8% for 1.4 mm before and after annealing, respectively. The amount was 1.8%, 21.3% and 15.7% in the 0.7 mm Rolled RD samples in the cold-rolled, annealed at 620 °C and annealed at 650 °C conditions, respectively. This frequency was 7%, 25.6% and 8.8% in the 0.7 Rolled TD samples in the cold-rolled, annealed at 620 °C and annealed at 650 °C conditions, respectively.

Figure 8.17 illustrates the frequency of the phase boundaries between the γ and α/α' with less than 10° deviation from KS OR. The figures shows that the frequency increased from 31.8% to 85.3% in AR sample after one-step IA. As shown in Figure 8.6, there were 0.2% and 0.3% of the γ phase volume fraction in the 0.7 mm RD and TD cold-rolled samples. As Figure 8.17 shows the phase boundaries frequencies between the γ and α/α' with less than 10°

deviation from KS OR in RD and TD cold-rolled samples were 60.9% and 42%, respectively. It means the γ phase with phase boundaries close to KS OR was resisting more to transform into the α' .

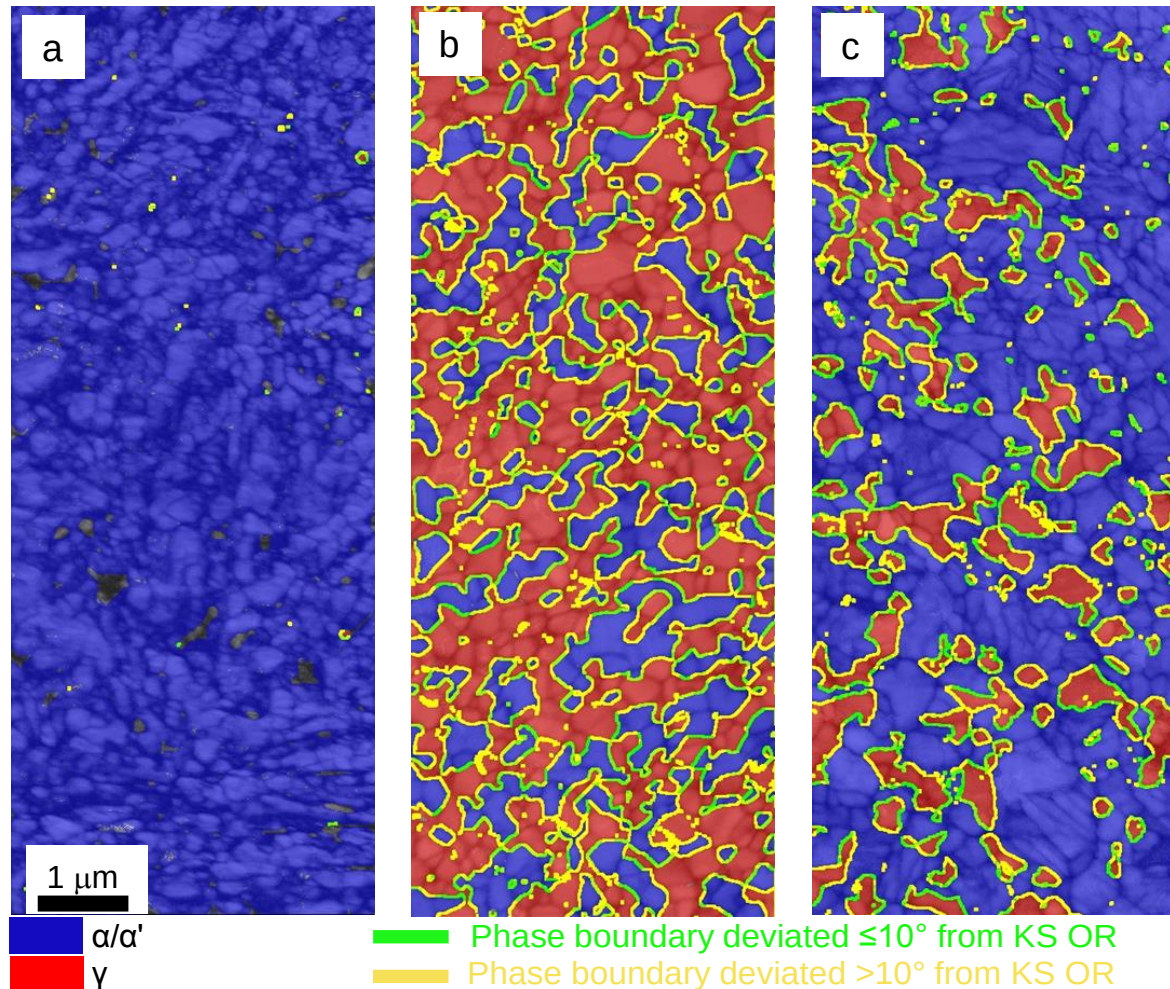


Figure 8.15. SEM-TKD, IQ-phase map the 0.7 mm Rolled TD FeCMn steel samples in the (a) cold-rolled, annealed for 10 min at (b) 620 °C and (c) 650 °C conditions. The phase boundaries between the γ and α/α' phases are shown based on KS OR.

The IA of the 0.7 mm Rolled RD and Rolled TD cold-rolled samples at 620 °C resulted in nucleation and growth of the γ phase and recovery, recrystallization, nucleation and growth of the α phase. The frequency of the phase boundaries with less than 10° deviation from KS OR was 38.6% and 33.3% in the Rolled RD and Rolled TD samples. These amounts were noticeably lower than the one-step IA sample. The frequency of the phase boundaries with less than 10° deviation from KS OR increased to 61.3% and 59.6% after increasing the annealing temperature to 650 °C in the Rolled RD and Rolled TD samples, respectively.

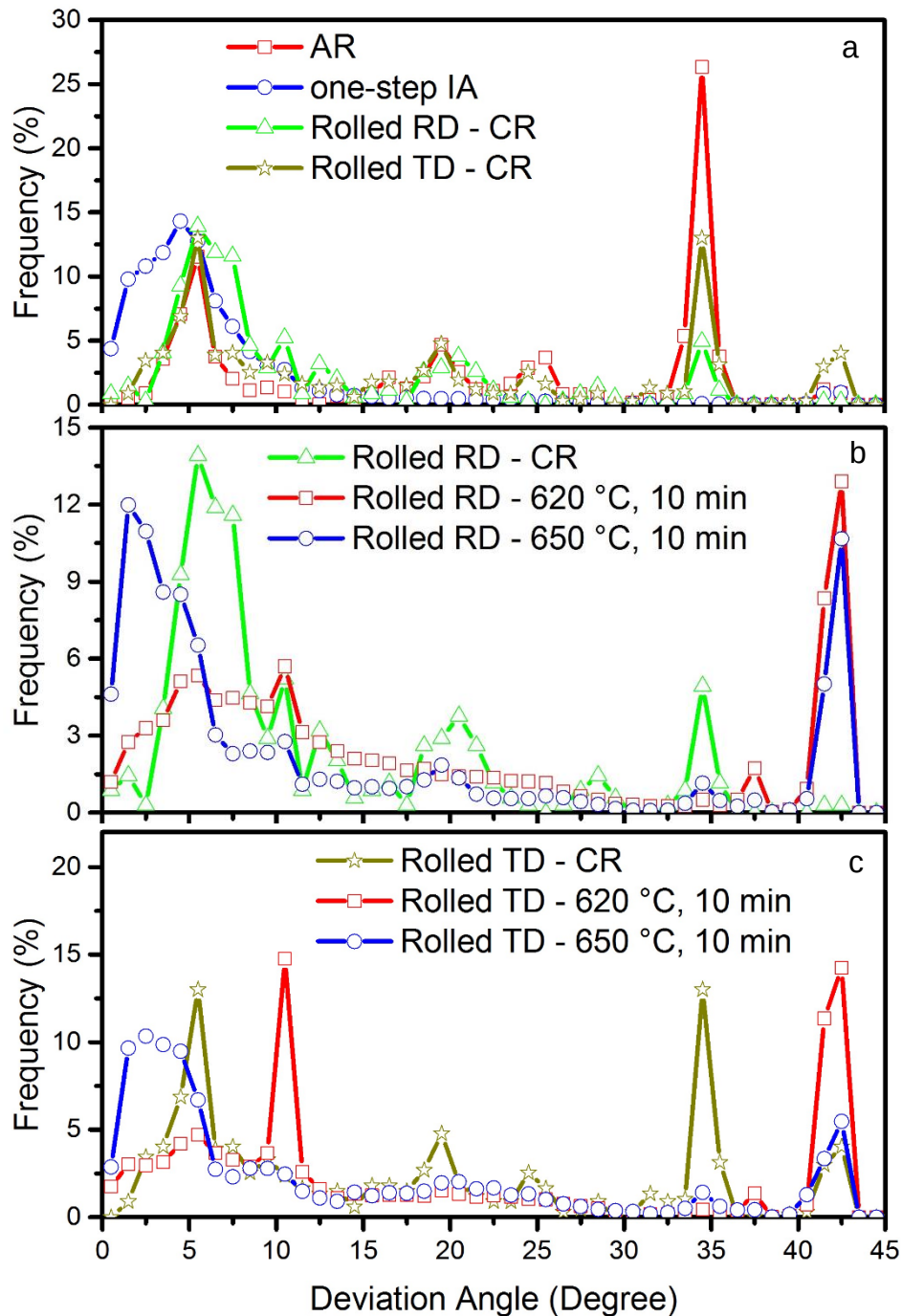


Figure 8.16. Histogram distribution of the γ and α/α' phases boundaries in FeCMn steel samples deviated from the KS orientation relationship (a) effect of the first annealing at 620 °C and the following cold rolling (b) effect of the second annealing on the Rolled RD sample and (c) effect of the second annealing on the Rolled TD sample.

It can be seen in Figure 8.17 that 0.7 mm Rolled RD samples had a higher frequency of the phase boundaries between the γ and α/α' with less than 10° deviation from KS OR in comparison to the 0.7 mm Rolled TD samples before and after annealing. The figure depicts that by increasing the annealing temperature, the Rolled RD and Rolled TD samples' frequency amounts were getting closer.

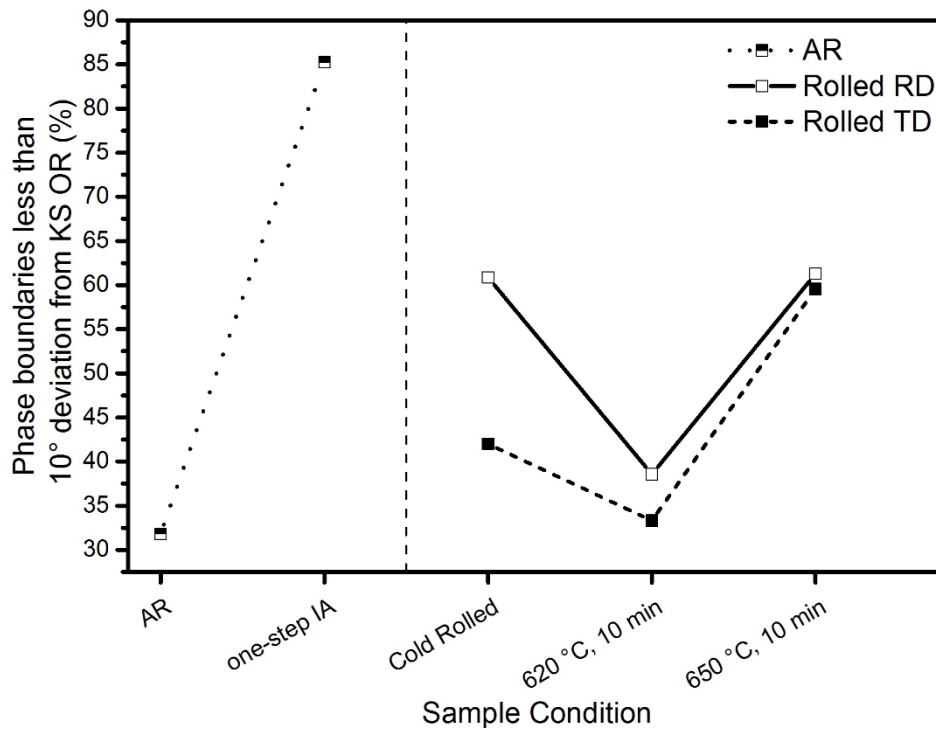


Figure 8.17. The frequency of the phase boundaries between the γ and α/α' with less than 10° deviation from KS OR in FeCMn steel samples.

8.2.4 Strain Distribution in the Phases

The LAGBs and KAM analyses used in order to assess qualitatively and quantitatively the strain distribution in the γ and α/α' . This approach was used as an indicator for evaluating the dislocation density within phases.

8.2.4.1 Low-Angle Grain Boundaries

In this study, a fraction of the grain boundaries with a low angle of less than 15° was used to develop the method. Appendix 2 illustrates the γ and α/α' grain boundaries on the SEM-TKD IQ-maps.

Figure 8.18 illustrates the variation of the LAGBs inside the γ and α/α' phases after cold rolling and annealing. In the 1.4 mm AR sample, the fractions of the LAGBs in the γ and α/α' phases were 100% and 70.9%, respectively. After annealing of the AR sample at 620 °C, the fractions of the LAGBs in the γ and α/α' phases were 56.5% and 61.1%, respectively.

The figure shows that in the γ phase of the 0.7 mm Rolled TD and Rolled RD cold-rolled samples, the fraction of the LAGBs was about 100%. In the α/α' phase of the 0.7 mm Rolled

RD and Rolled TD cold-rolled samples, the fractions of the LAGBs were 77.3% and 78.8%, respectively.

In the 0.7 mm Rolled RD cold-rolled sample after annealing at 620 °C and 650 °C, the fractions of the LAGBs of the γ phase were 52% and 75.1%, respectively. These fractions in the α/α' phase were 52.7% and 56.1% after annealing at 620 °C and 650 °C, respectively.

In the 0.7 mm Rolled TD cold-rolled sample after annealing at 620 °C and 650 °C, the fractions of the LAGBs in the γ phase were 52% and 63.2% and in the α/α' phases were 56.5% and 57%, respectively.

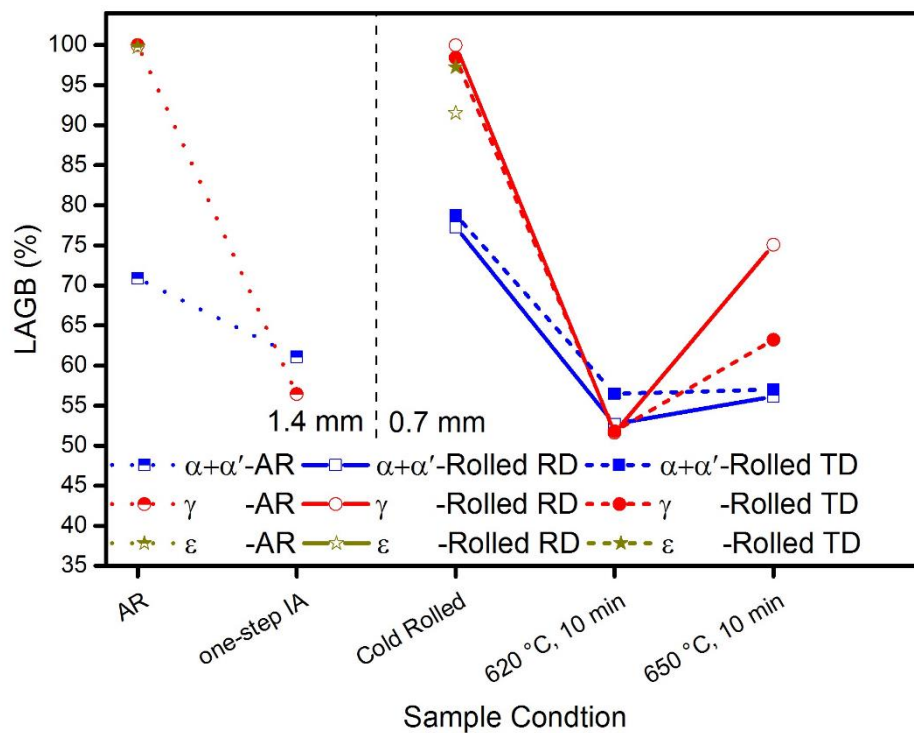


Figure 8.18. Variation of the LAGBs inside the γ and α/α' phases of the FeCMn alloy steel samples after cold rolling and annealing.

It can be seen that increasing the annealing temperature to the γ single-phase region (650 °C) increased the strain distribution in the γ phase, especially in the Rolled RD sample. It might relate to the γ into α/α' transformation.

Due to the recrystallization of the γ and α/α' phases, the growth of the grains, and the transformation of the α' into γ in the sample after the IA, both phases in the sample exhibited a lower strain than the cold rolled samples. This resulted in a decrease in LAGBs and an increase in HAGBs in the phases.

8.2.4.2 Kernel Average Misorientation

The KAM method can be used to quantify the average misorientation around a measurement point in comparison with a defined set of the nearest neighbours. A KAM value of less than 5° was used in this study. In Appendix 2, the local misorientation maps for FeCMn steel samples are presented along with their histogram distributions.

Figure 8.19 illustrates the average local misorientation of the γ and α/α' phases of the samples under different conditions. The average local misorientations of the γ and α/α' phases inside the 1.4 mm AR sample were 1.16° and 1.21° , respectively. These amounts decreased to 0.86° and 0.77° after the IA at 620°C .

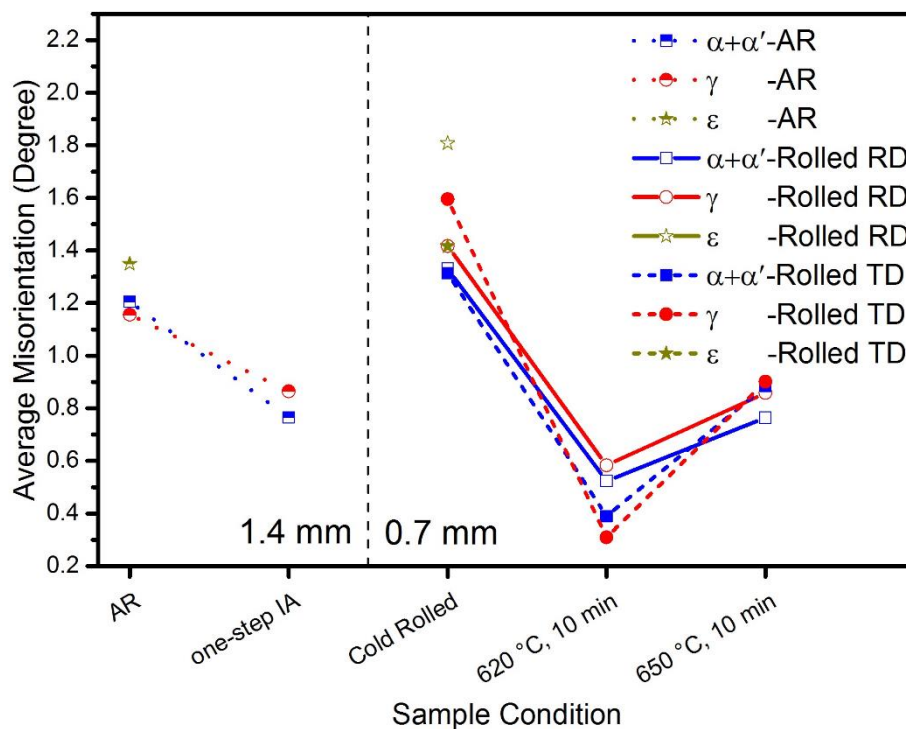


Figure 8.19. The average local misorientation of the γ and α/α' phases of the FeCMn alloy steel samples after cold rolling and annealing.

In the 0.7 mm Rolled RD cold-rolled sample, the average local misorientation of the γ was 1.42° . It decreased to 0.58° and 0.86° after annealing at 620°C and 650°C , respectively. The average local misorientations of the α/α' phase were 1.33° , 0.52° and 0.76° in the Rolled RD cold-rolled, annealed at 620°C and annealed at 650°C samples, respectively.

In the 0.7 mm Rolled TD cold-rolled sample, the average local misorientation of the γ was 1.60° . It decreased to 0.31° and 0.90° after annealing at 620°C and 650°C , respectively.

The average local misorientations of the α/α' phase were 1.31° , 0.39° and 0.88° in the Rolled RD cold-rolled, annealed at 620°C and annealed at 650°C samples, respectively.

Annealing the Rolled RD and Rolled TD samples at 650°C resulted in a higher strain in comparison to the annealing at 620°C because the unstable γ transformed into α' , which generated dislocations in nearby grains of the γ and α [402].

Comparing the KAM approach to the LAGBs approach, it appeared that the KAM approach showed a lower dislocation density ratio (0.7 mm sample to 1.4 mm sample) of the γ phase after annealing.

8.2.5 Texture

On the SEM-TKD IQ-maps, the ODF analysis was performed to assess the texture of the different phases of the samples. A description of the common texture components and fibers of the BCC and FCC metals and alloys was provided in section 6.2.5. After rolling, BCC metals and alloys develop fiber textures [37, 392–394].

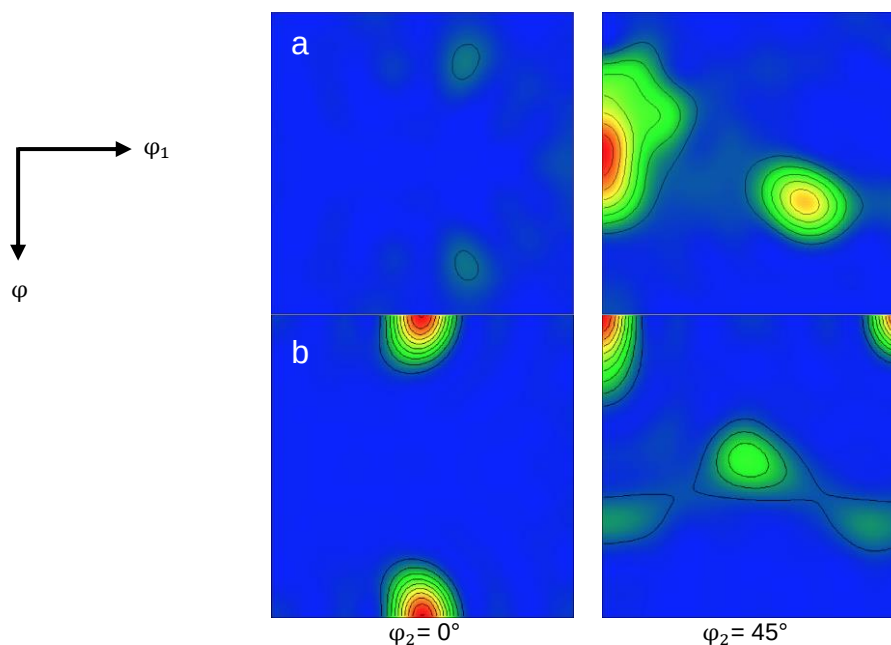


Figure 8.20. The texture of the α/α' phase in the 1.4 mm FeCMn alloy (a) AR and (b) one-step IA at 620°C for 10 min in $\varphi_2 = 0^\circ$ and 45° ODF sections. Contour levels = $2x$.

Figure 8.20 illustrate the texture of the α/α' phase in the 1.4 mm FeCMn alloy in the AR and one-step IA at 620°C for 10 min. The α/α' phase in the AR had the α and γ - fibers and the

strong $(1\bar{1}0)[223]$ texture component with Euler angles of $(0^\circ, 45^\circ, 45^\circ)$ at $f(g)=15.4$ times random. There was a weaker E texture component at $f(g)=11.2$ that resulted from the deformation product of the γ phase Goss texture component [396].

The α/α' phase in the one-step IA sample had the α and γ - fibers. It had a strong Rt-C texture component at $f(g)=20.1$ times random and weaker $\{332\}\langle 113\rangle$ and $\{332\}\langle 110\rangle$ texture components at $f(g)=3.17$ and $f(g)=3.78$ times random, respectively.

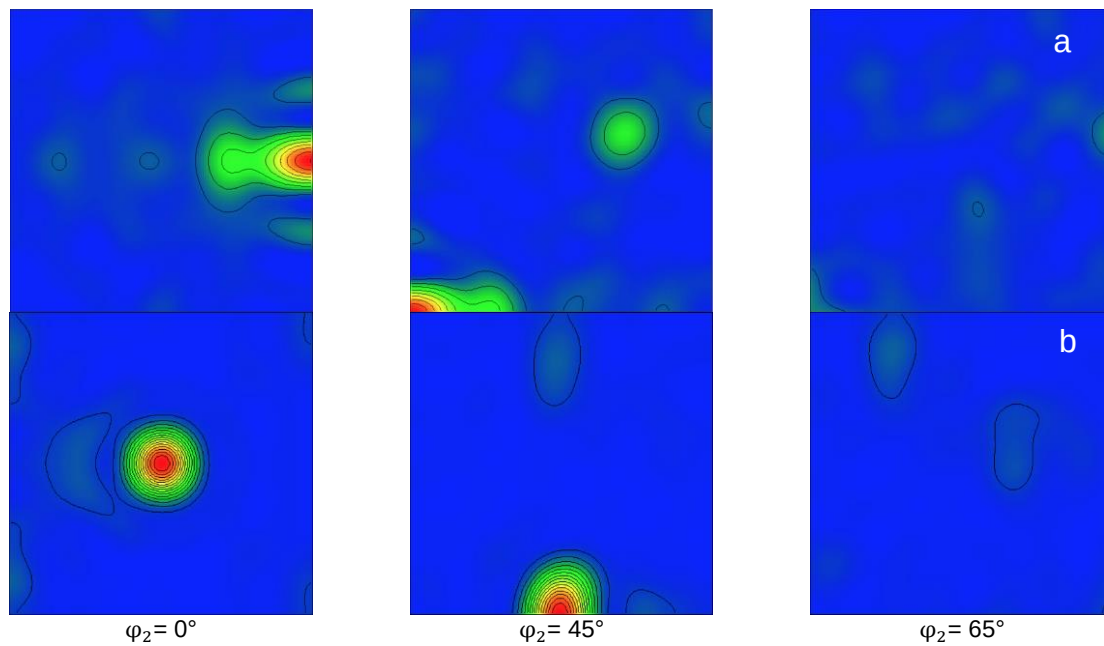


Figure 8.21. The texture of the γ in the 1.4 mm FeCMn alloy (a) AR and (b) one-step IA at 620 °C for 10 min in $\phi_2 = 0^\circ, 45^\circ$ and 65° ODF sections. Contour levels = 2x.

Figure 8.21 illustrates the texture of the γ in the 1.4 mm FeCMn alloy in the AR and after the one-step IA at 620 °C for 10 min conditions. The γ phase of the 1.4 mm AR sample had a strong α -fiber and a strong Rotated Goss (Rt-G) texture component at $f(g)=18$ times random. It had weaker $(010)[2\bar{3}3]$ (at $f(g)=6.53$ times random), $(112)[\bar{1}\bar{3}2]$ (at $f(g)=5.77$ times random), $\{021\}\langle 012\rangle$ (at $f(g)=3.27$ times random), $(332)[1\bar{1}0]$ (at $f(g)=2.35$ times random) and G/B (at $f(g)=2.08$ times random) texture components.

The γ phase of the 1.4 mm one-step IA sample had an α -fiber and a strong $(011)[3\bar{2}2]$ at $f(g)=31.9$ times random that was reported for annealing [403].

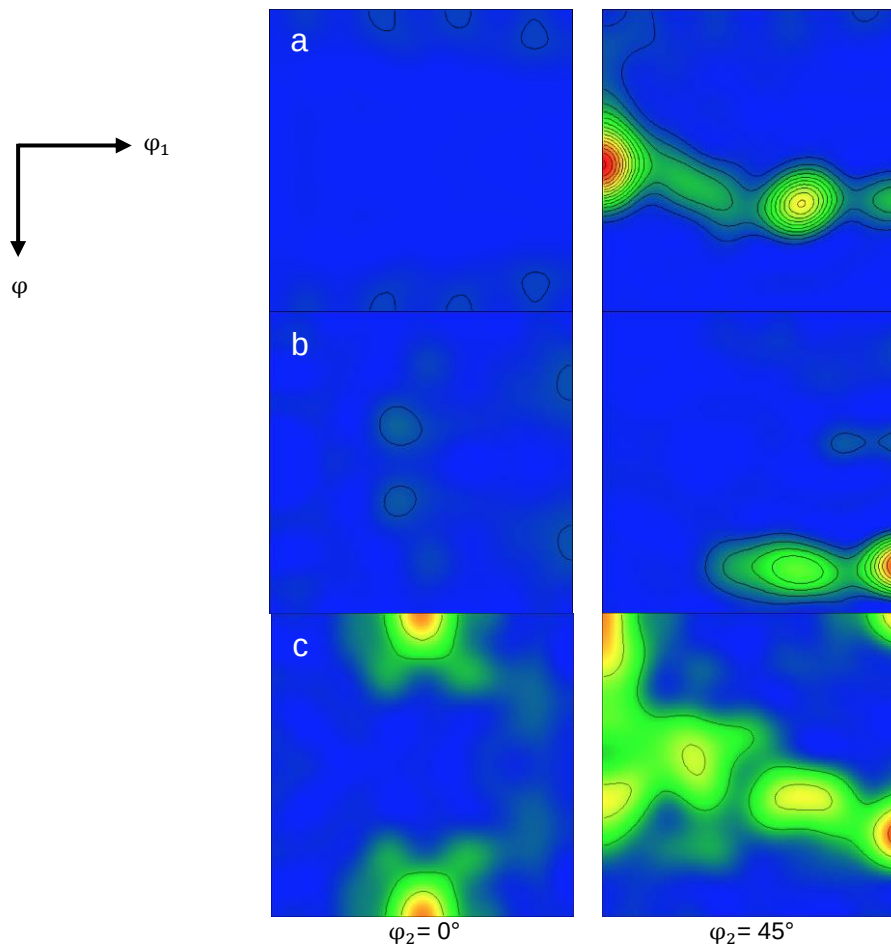


Figure 8.22. The texture of the α/α' phase in the 0.7 mm FeCMn alloy Rolled RD (a) cold-rolled, (b) annealed at 620 °C for 10 min and (c) annealed at 650 °C for 10 min in $\phi_2 = 0^\circ$ and 45° ODF sections. Contour levels = $2x$.

Figure 8.22 illustrates the texture of the α/α' phase in the 0.7 mm FeCMn alloy Rolled RD in cold-rolled and annealed conditions. The 0.7 mm Rolled RD cold-rolled sample had α , γ and weak θ fibers. It had a strong $(332)[0\bar{1}1]$ texture component at $f(g)=29.8$ times random. It had weaker $\{111\}\langle 110\rangle$ and $\{111\}\langle 112\rangle$ texture components at $f(g)=20.1$ and $f(g)=7.65$ times random, respectively which were the transformation texture components from deformed γ phase [395, 396, 404].

Figure 8.22 (b) shows that the α/α' phase of the Rolled RD annealed at 620 °C had a strong copper twin $(552)[\bar{1}\bar{1}5]$ at $f(g)=22.4$ times random and a weaker $(331)[1\bar{2}3]$ at $f(g)=9.95$ times random.

The Rolled RD sample annealed at 650 °C (Figure 8.22(c)) had α , γ and θ fibers. It had a strong $(332)[\bar{1}\bar{1}3]$ at $f(g)=7.11$, and weaker rotated cube, E and $(113)[0\bar{3}1]$ at $f(g)=5.85$, 4.61 and 4.18 times random, respectively.

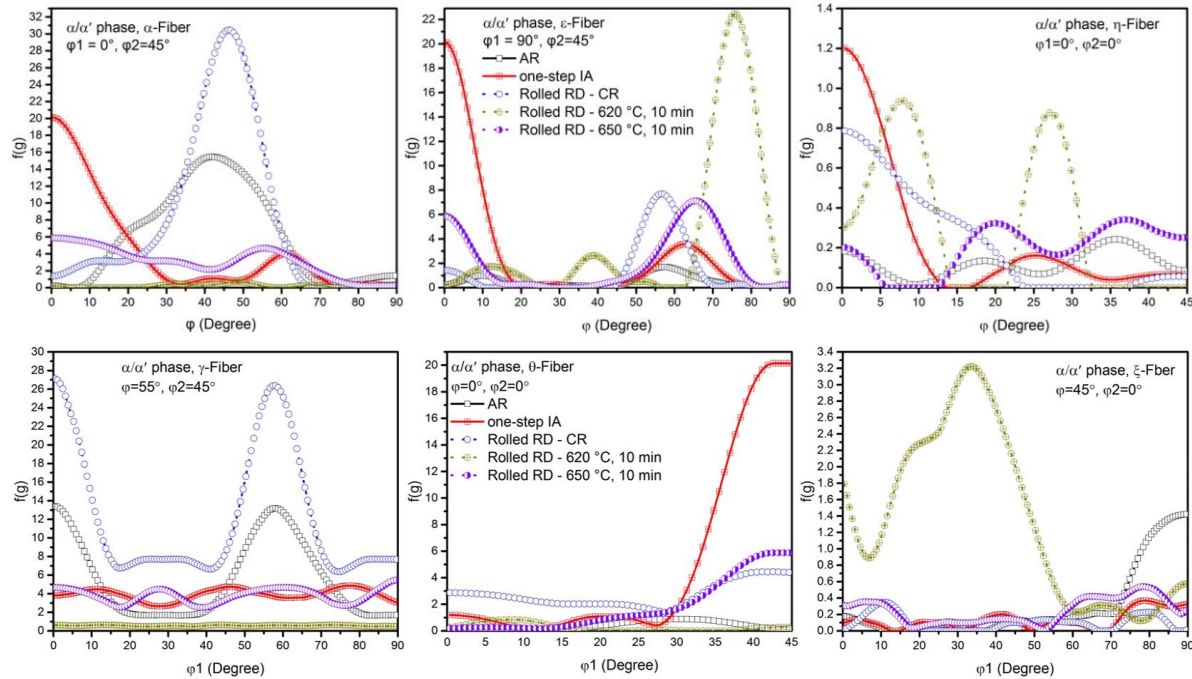


Figure 8.23. The fibers of the α/α' phase in the 1.4 mm and 0.7 mm Rolled RD FeCMn steel samples in the cold-rolled and annealed conditions.

Figure 8.23 presents the α/α' phase fibers in the 1.4 mm and 0.7 mm Rolled RD samples under the cold-rolled and annealed conditions. Both samples exhibited almost the same α and γ fibers behaviour, but the fibers were twice stronger in the 0.7 mm Rolled RD sample. The θ -fiber at 45° indicated that in the AR sample, the cube texture intensity was 0.2 times random, which increased to 20.1 times random in the 1.4 mm one-step IA sample. Subsequent RD rolling decreased the texture intensity to 4.4 times random. The second annealing at 620°C reduced the texture intensity 0.3 times random, and at 650°C , it increased to 5.9 times random. This behaviour was similar for the Rotated Cube (Rt-C) texture.

Figure 8.24 shows that the γ phase in the Rolled RD cold-rolled sample had a strong Brass texture component at $f(g)=24.9$ times random and a weaker texture component at $(10^\circ, 73^\circ, 40^\circ)$ at $f(g)=18.2$ times random. The other weak texture components were $(011)[1\bar{1}5]$ at $f(g)=2.84$ times random and $(100)[2\bar{3}3]$ at $f(g)=3.11$ times random.

The γ phase of the 0.7 mm Rolled TD sample annealed at 620°C had α , γ and τ fibers with a strong Goss texture at $f(g)=29.7$ times random, a weak F texture component at $f(g)=8.01$ times random and a weak texture at $(90^\circ\ 15^\circ\ 45^\circ)$ at $f(g)=6.05$ times random.

The γ phase of the 0.7 mm Rolled TD sample annealed at 650 °C had the α -fiber with a strong S texture component at $f(g)=10.5$ times random and weaker $\{123\}\langle 634 \rangle$ and $(112)[\bar{1}\bar{1}1]$ texture components at $f(g)=5.15$ and 4.74 times random, respectively.

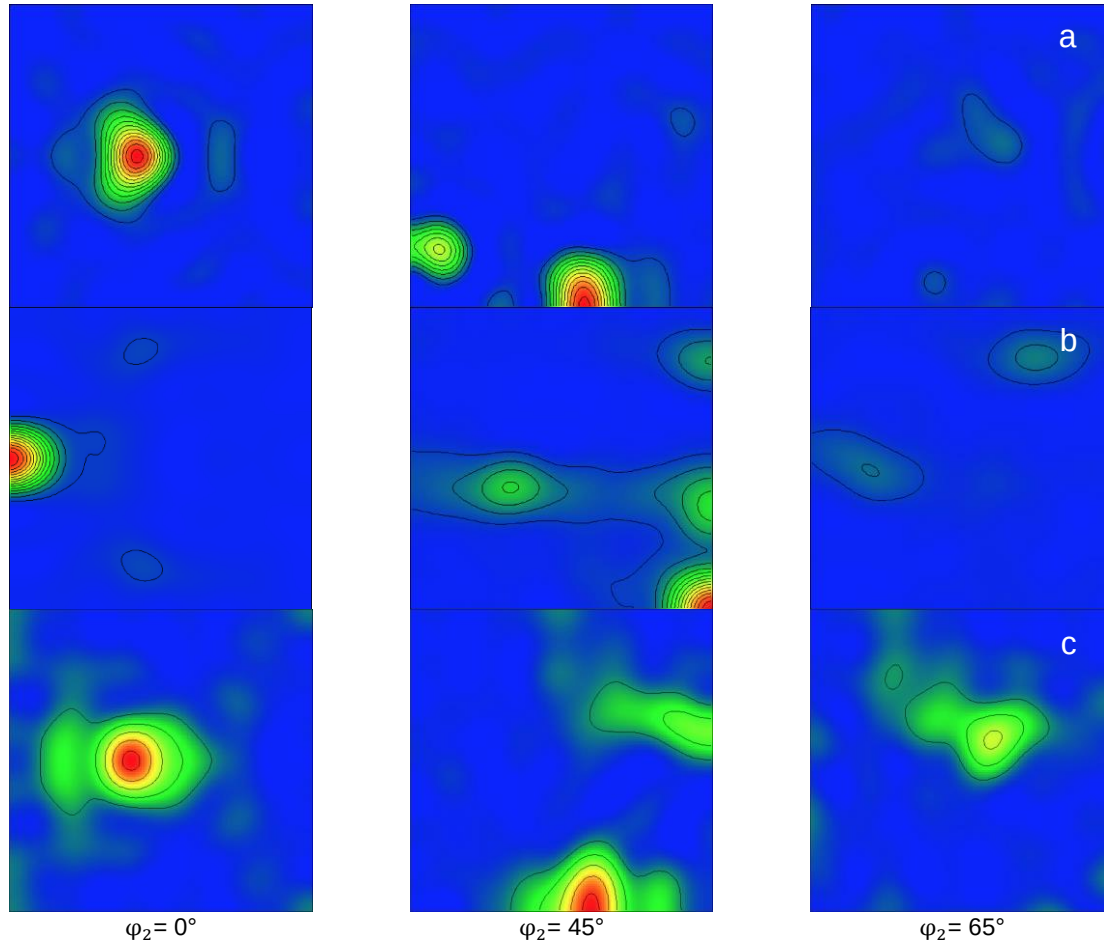


Figure 8.24. The texture of the γ in the 0.7 mm FeCMn alloy Rolled RD (a) cold-rolled, (b) annealed at 620 °C for 10 min and (c) annealed at 650 °C for 10 min in $\varphi_2 = 0^\circ$, 45° and 65° ODF sections. Contour levels = 2x.

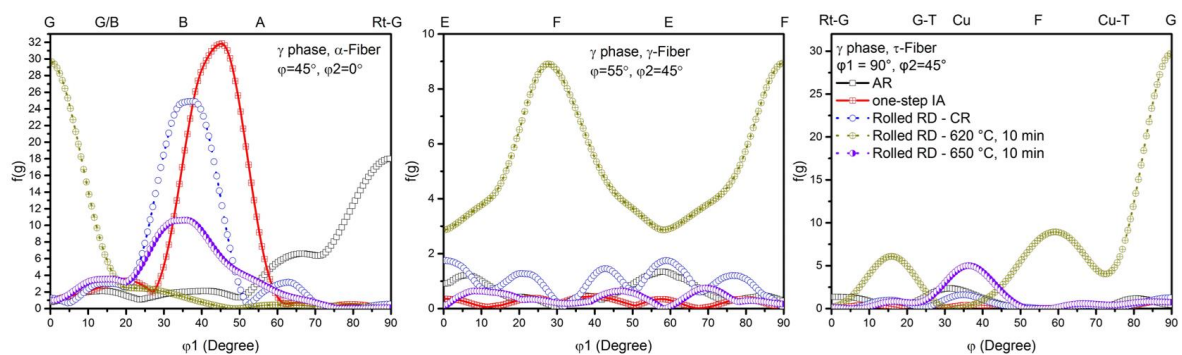


Figure 8.25. The fibers of the γ in the 1.4 mm and 0.7 mm Rolled RD FeCMn Steel sample in the cold-rolled and annealed condition.

Figure 8.25 illustrates the fibers of the γ phase in the 1.4 mm and 0.7 mm Rolled RD FeCMn steel samples in the cold-rolled and annealed conditions. It can be seen that the 0.7 mm Rolled RD that annealed at 620 °C had the strongest γ and τ fibers between the samples.

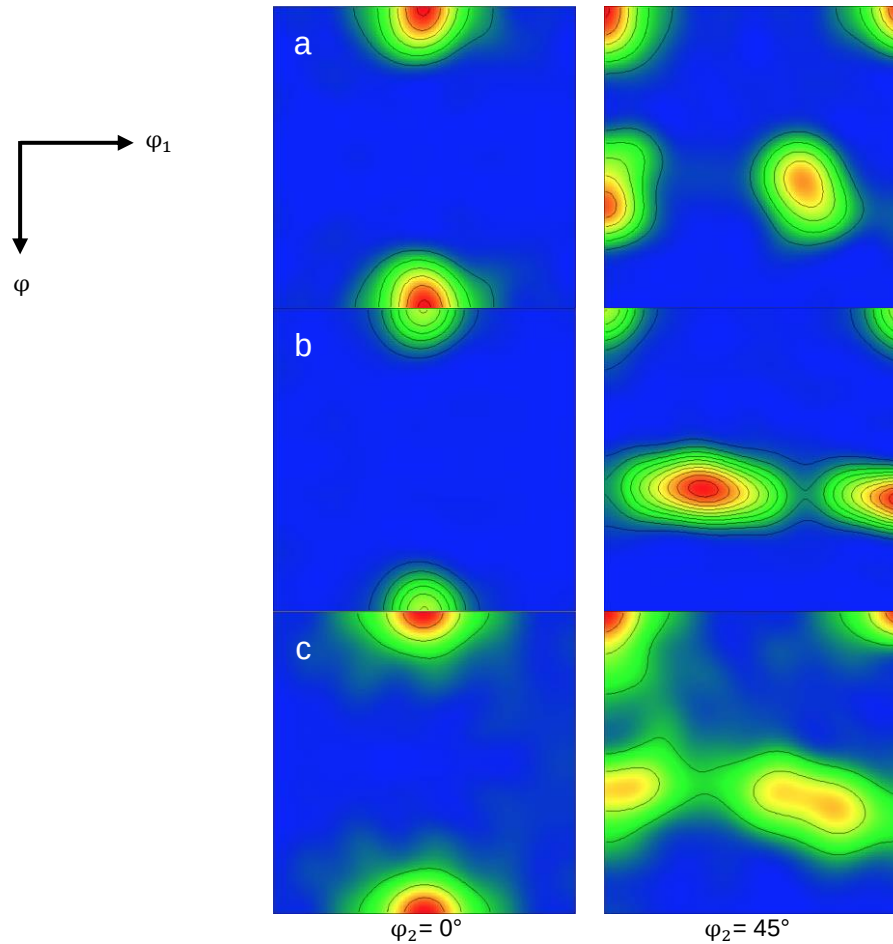


Figure 8.26. The texture of the α/α' phase in the 0.7 mm FeCMn alloy Rolled TD (a) cold-rolled, (b) annealed at 620 °C for 10 min and (c) annealed at 650 °C for 10 min in $\varphi_2 = 0^\circ$ and 45° ODF sections. Contour levels = 2x.

Figure 8.26 illustrates the texture of the α/α' phase in the 0.7 mm Rolled TD in the cold-rolled and annealed conditions. It is clear that the texture of the rolled TD sample was different compared to the Rolled RD samples which was shown in Figure 8.22. The α/α' phase of the 0.7 mm Rolled TD cold-rolled sample (Figure 8.26(a)) had α and γ fibers and a strong Rt-C texture component at $f(g)=12.3$ times random and a weaker E texture component at $f(g)=9.62$ times random. These texture components were derived from γ phase rolling texture components [395, 396, 404].

The α/α' phase of the 0.7 mm Rolled TD sample annealed at 620 °C had a strong γ fiber and a strong F texture component at $f(g)=21.1$ times random, and a weaker Rt-C texture

component at $f(g)=12.1$ times random. These texture components were derived from γ phase rolling texture components.

It was seen that the α/α' phase in the 0.7 mm Rolled RD annealed at 650 °C had α and γ fibers and a strong Rt-C texture component at $f(g)=7.24$ and weaker E and $\{554\}<225>$ at $f(g)=5.03$ and 2.59 times random, respectively.

Results supported the existence of the recrystallization mechanisms based on either oriented nucleation or selective or oriented growth. During the annealing process, the nucleation in these grains preserved the orientation of the former texture, which was presented as $\{001\}<110>$ and $\{111\}<110>$ texture components in the cold rolled martensitic sheets. As a consequence, it can be concluded that the oriented nucleation mechanism controlled the texture of these texture components [397, 398]. Figure 8.27 illustrates the fibers of the α/α' phase in the 1.4 mm and 0.7 mm Rolled TD samples before and after annealing.

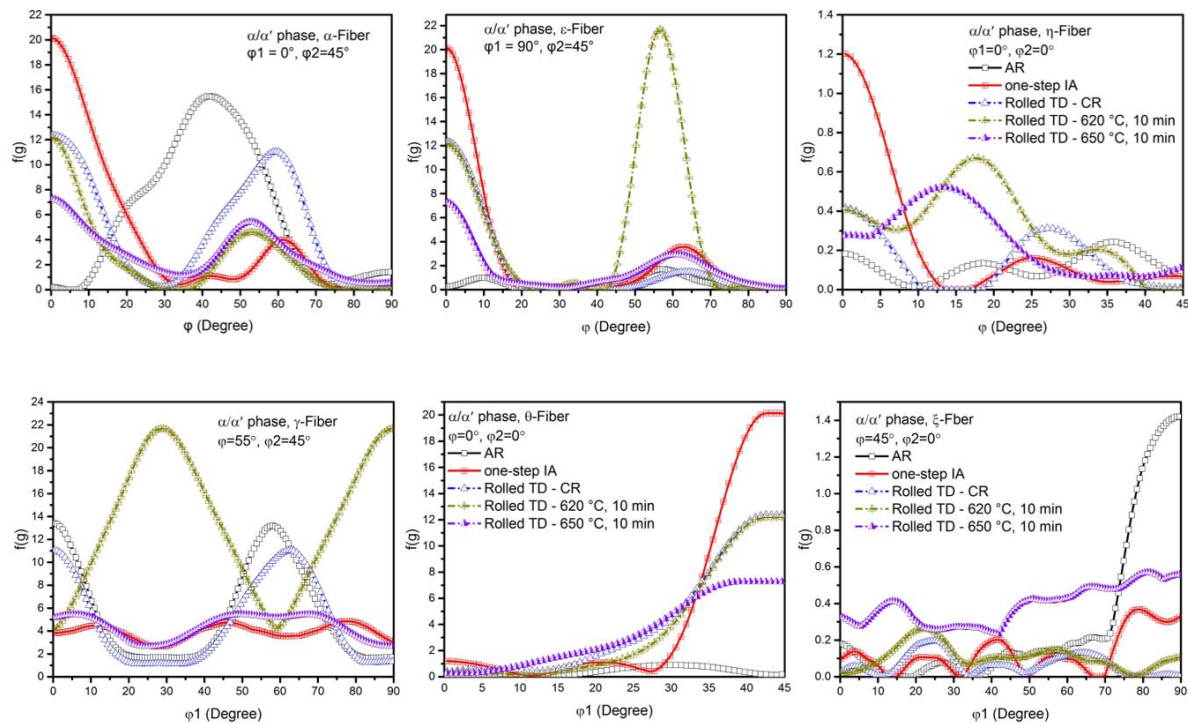


Figure 8.27. The fibers of the α/α' phase in the 1.4 mm and 0.7 mm Rolled TD FeCMn steel sample in the cold-rolled and annealed conditions.

Figure 8.28 shows that γ phase of the 0.7 mm Rolled TD cold-rolled sample had a strong A texture component at $f(g)=26$ times random. The 0.7 mm Rolled RD annealed at 620 °C sample had an α -fiber and a strong Brass texture component at $f(g)=13.5$ times random. There was a demonstration that the B texture component in the γ phase originated from a reverse

transformation of the $\{011\}\langle 110 \rangle$ and $\{332\}\langle 113 \rangle$ texture components of the α/α' phase. A reverse transformation of α' -martensite into γ phase resulted in the Brass texture [37, 38, 401] because of the low driving force of the γ phase recovery and recrystallization [399].

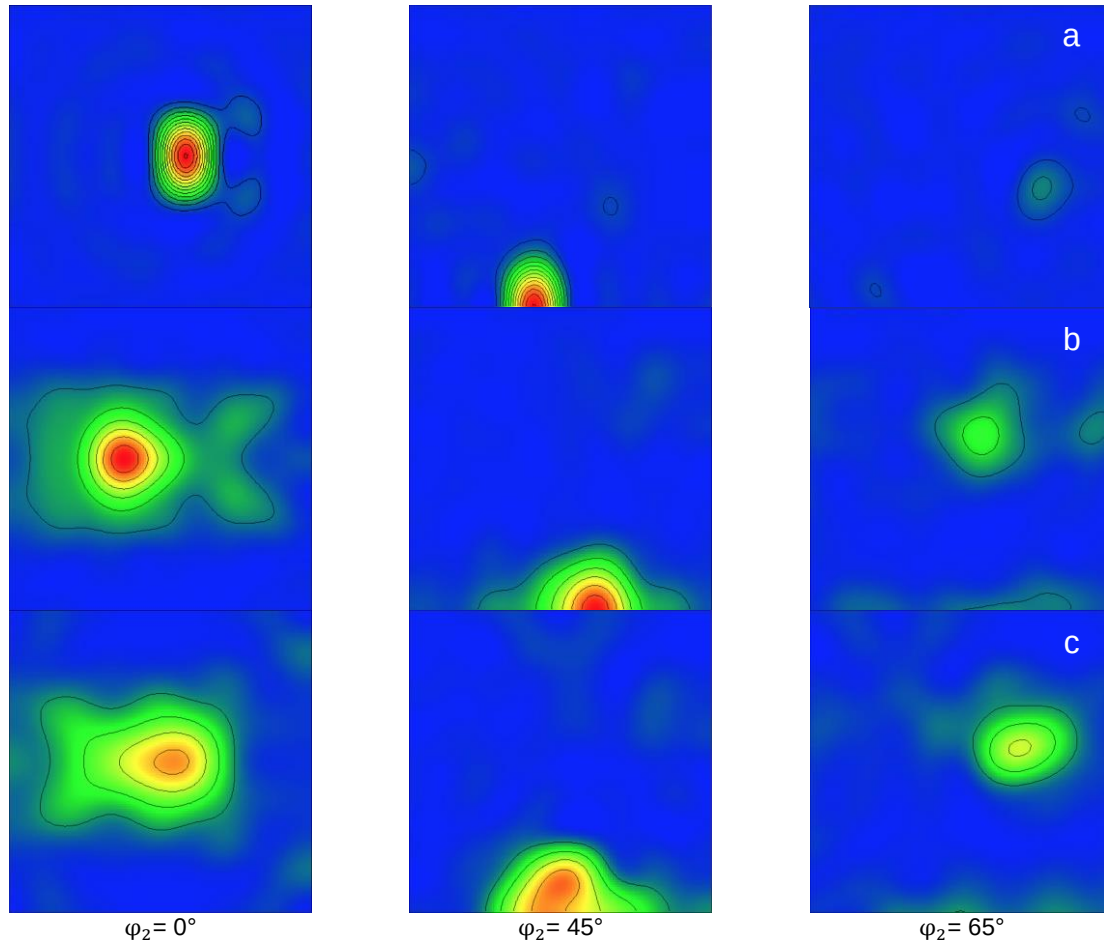


Figure 8.28. The texture of the γ phase in the 0.7 mm FeCMn alloy Rolled TD (a) cold-rolled, (b) annealed at 620 °C for 10 min (b) annealed at 650 °C for 10 min in $\varphi_2 = 0^\circ$, 45° and 65° ODF sections. Contour levels = 2x.

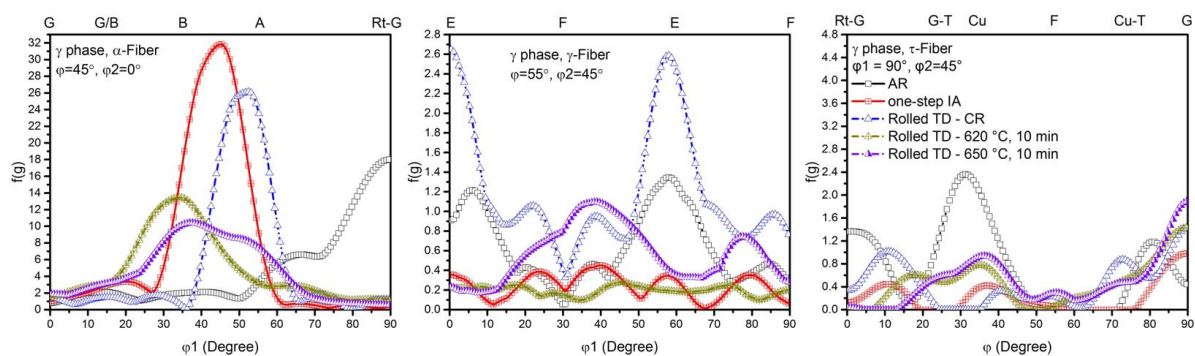


Figure 8.29. The fibers of the γ phase in the 1.4 mm and 0.7 mm Rolled TD FeCMn steel samples in the cold-rolled and annealed condition.

The 0.7 mm Rolled RD annealed at 650 °C sample had a (011)[$3\bar{2}2$] texture component at $f(g)=8.71$ times random and a S texture component at $f(g)=7$ time random. Figure 8.29 illustrates the fibers of the γ phase in the 1.4 mm and 0.7 mm Rolled TD before and after annealing.

8.2.6 Summary of the Scanning Electron Microscopy Maps Analyses

The analyses of the SEM-TKD maps of the FeCMn steel before and after the TSTMP summarised in Table 8.1.

Table 8.1. Summary of the SEM-TKD maps of the FeCMn steel after the TSTMP.

Sample Condition		Phase Volume Fraction %		Average Grain Size nm		Nano Size Grains %		KAM Average Misorientation Degree	
		γ	α/α'	γ	α/α'	γ	α/α'	γ	α/α'
1.4 mm	AR	0.1	96.7	56	144	100.0	55.0	1.16	1.21
	One-step IA	62.0	38.0	215	181	33.8	38.3	0.86	0.77
TSTMP Rolled RD	Cold Rolled	0.2	93.5	52	126	98.1	62.5	1.42	1.33
	620 °C, 10 min	60.1	39.9	181	182	32.2	34.0	0.58	0.52
	650 °C, 10 min	9.5	90.5	115	187	54.1	39.1	0.86	0.76
TSTMP Rolled TD	Cold Rolled	0.3	95.3	54	114	96.9	62.3	1.60	1.31
	620 °C, 10 min	61.6	38.4	175	161	31.6	37.7	0.31	0.39
	650 °C, 10 min	22.5	77.5	149	187	44.3	39.6	0.90	0.88

Sample Condition		Aspect Ratio (No preferred direction)		Average Aspect Ratio (RD/TD)		LAGB %		Phase Boundaries <10° Deviation from KS OR %
		γ	α/α'	γ	α/α'	γ	α/α'	
1.4 mm	AR	1.64	2.18	--	1.28	100.0	70.9	31.8
	One-step IA	1.94	2.15	1	1	56.5	61.1	85.3
TSTMP Rolled RD	Cold Rolled	1.70	1.94	--	1.29	100.0	77.3	60.9
	620 °C, 10 min	1.76	2.01	1.04	1.26	51.6	52.7	38.6
	650 °C, 10 min	1.90	1.90	1.05	0.95	75.1	56.1	61.3
TSTMP Rolled TD	Cold Rolled	1.95	1.89	--	1.04	98.4	78.8	42.0
	620 °C, 10 min	1.74	1.97	0.98	1	51.8	56.5	33.3
	650 °C, 10 min	1.86	1.80	0.96	0.95	63.2	57.0	59.6

8.3 X-Ray Diffraction

The XRD outcomes of all specimens examined in this chapter are exhibited in Figure 8.30. It is noticeable that the TD cold rolling caused a shift of the most intense peak in the cold-rolled or intercritically annealed samples from $(211)_\alpha$ to $(200)_\alpha$ peak. In both samples annealed at 650 °C, the intensity of $(211)_\alpha$ peak was lower than that of $(200)_\alpha$ peak.

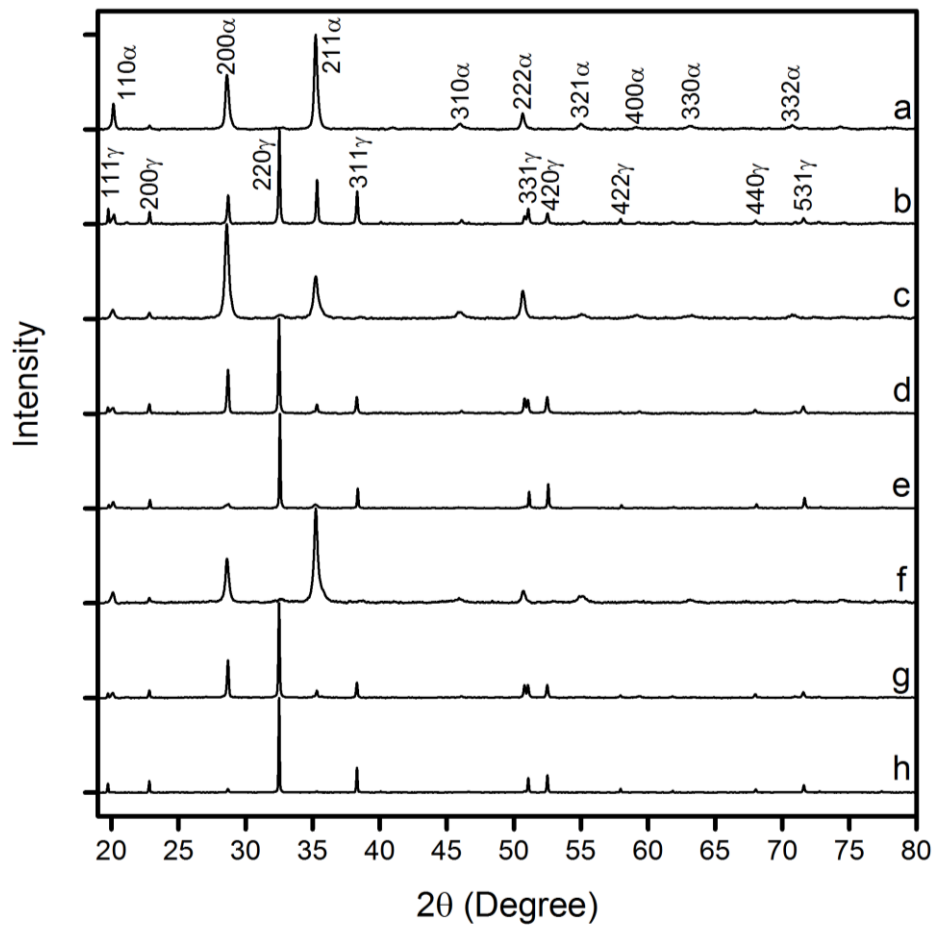


Figure 8.30. X-ray diffraction spectra of the FeCMn steel samples: 1.4 mm (a) AR and (b) one-step IA at 620 °C; 0.7 mm Rolled TD (c) cold-rolled, (d) annealed at 620 °C and (e) annealed at 650 °C; 0.7 mm Rolled RD (f) cold-rolled, (g) annealed at 620 °C and (h) annealed at 650 °C. The annealing time for the samples was 10 min.

8.4 Atom Probe Tomography Results

Using APT, the chemical composition of the γ and α phases was examined. A crystallographic analysis of the phases was not the objective of this study. A high density of the dislocations and small grain sizes of the phases, as well as a high concentration of the alloying elements within the γ phase, made it difficult to obtain data with appropriate poles. It is not possible to reconstruct a crystallographic structure without poles [405]. Figures 8.31 to 8.35 illustrate the atom probe maps of 1.4 mm and 0.7 mm FeCMn steel samples that were annealed at 620 °C and 650 °C for 10 min with alloying elements distributed across the grain boundaries.

A proxigram approach was employed to determine the compositional profiles of the C, Mn, Si and Mo and P atoms across γ and α phases. According to the proxigram approach, solute concentrations were computed along the normal of the iso-concentration surface [405].

Figures 8.31 to 8.35 shows the FeCMn samples after annealing at 620 °C and 650 °C for 10 min. A significant partitioning of the Mn and C atoms observed between the γ and α phases.

Figure 8.31 shows the APT maps of the 1.4 mm sample annealed at 620 °C for 10 min. Comparing the Mn partitioning between the γ and α grains showed that there was not the same pattern for all the phase boundaries. The Mn content in the α and γ grains was about $3.8 \pm 0.2\%$ and $16 \pm 4\%$. There was a Mn compositional spike while there was not any compositional spike in other phase boundaries. These might come from crystallographic orientation relationship between the γ and α at phase boundary that did not study in this thesis.

Figure 8.31 shows that the C content in γ grains was higher than the α grains. It was seen in four out of the eight phase boundaries; the C showed a compositional spike. Another interesting element distribution related to the γ - γ phase boundary. It was seen the Mn and C contents decreased in the grain boundary. This phenomenon might come from two reasons;

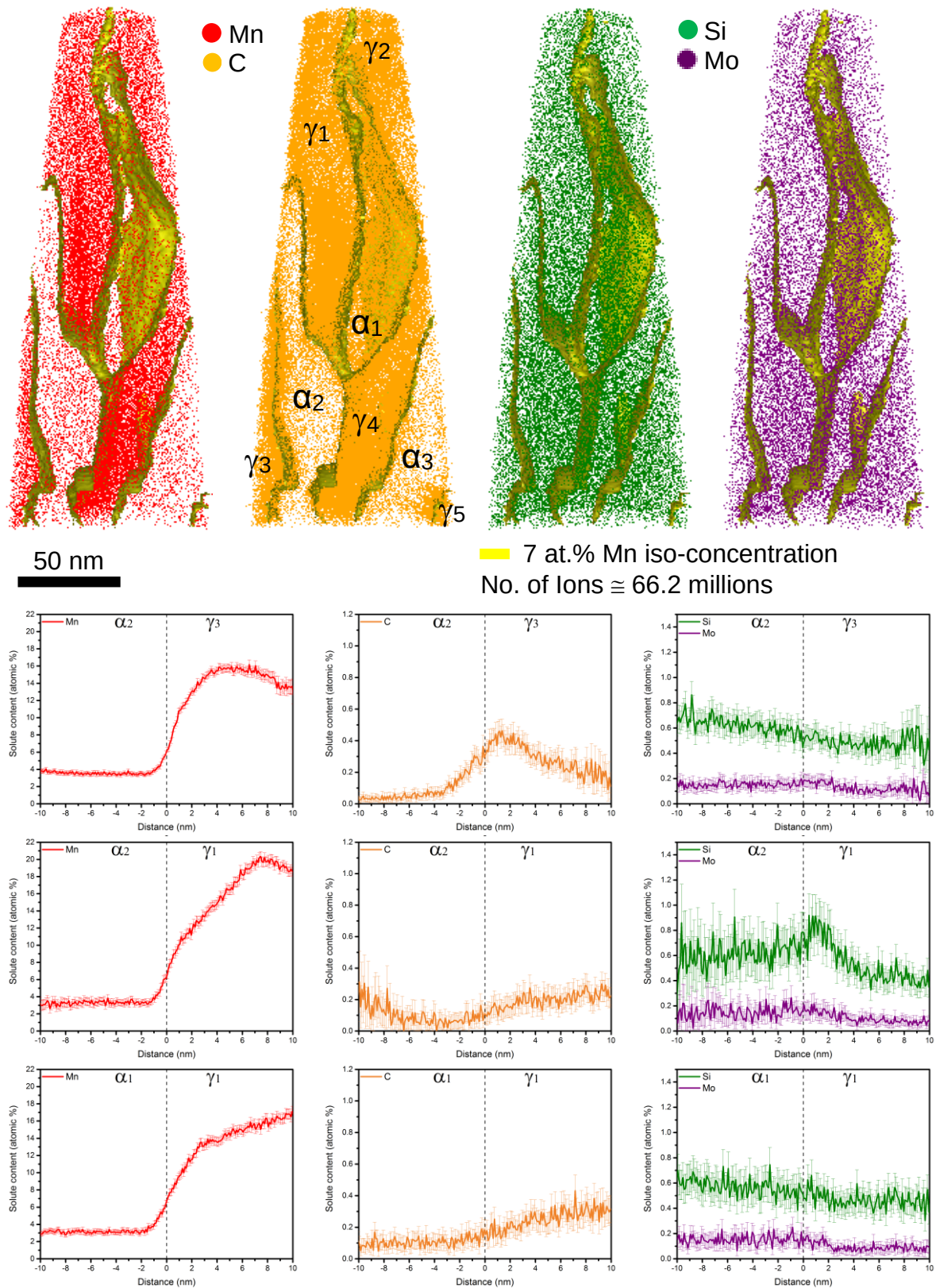
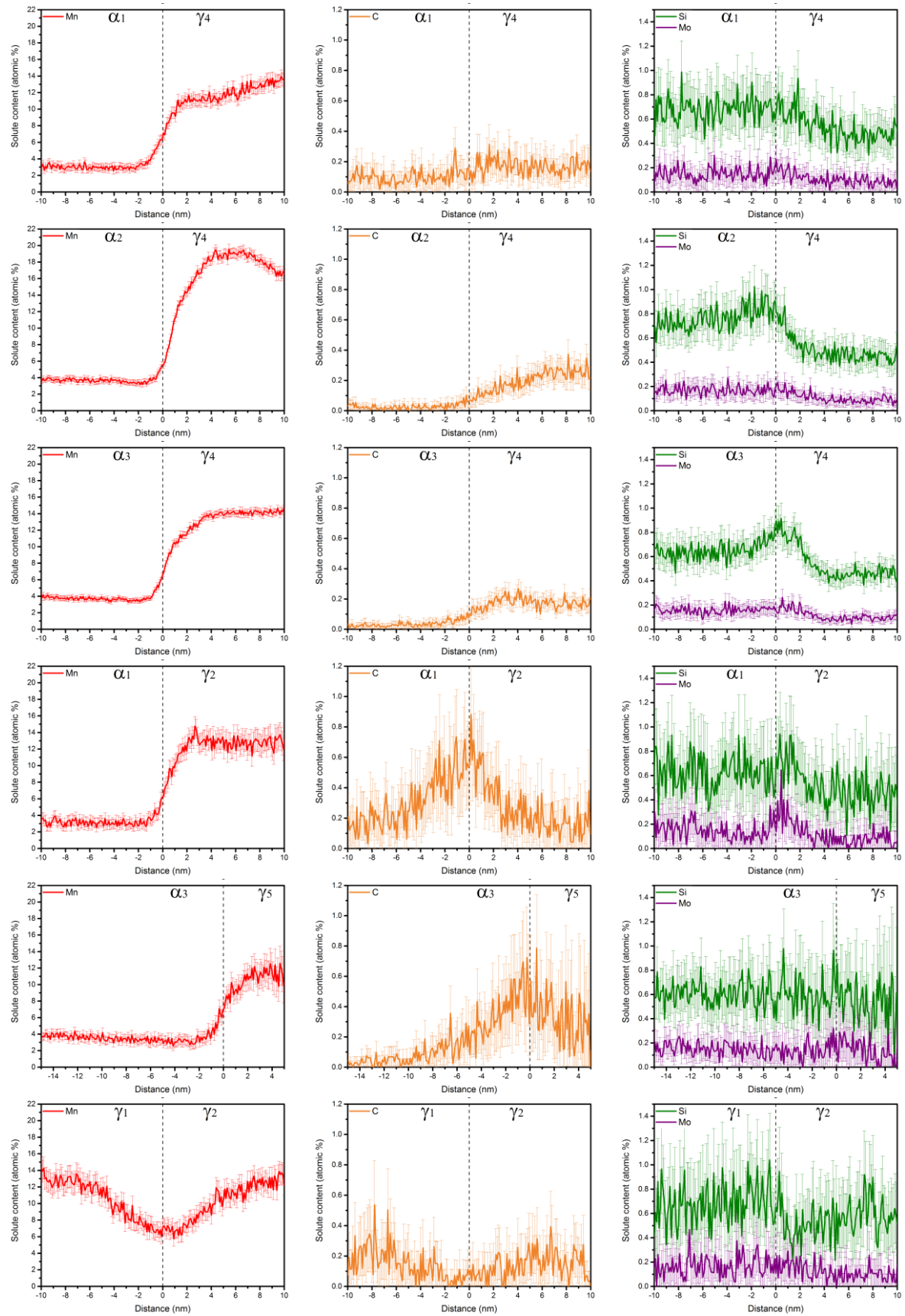


Figure 8.31. Atom probe tomogram of the 1.4 mm FeCMn sample annealed at 620 °C for 10 min along with the compositional profile of the Mn, C, Mo, and Si atoms across the grain boundaries. 7 at. % Mn iso-concentration is shown in the tomogram of the atoms.

Continue **Figure 8.31**

- the small α grain was between two γ grains, and it vanished because of the alloying elements diffusion and γ grains growth, or

- this grain boundary was close to the α_1 and thus γ_1 and γ_2 grain grew towards to the α_1 grain.

Figure 8.32 shows the APT maps of the 0.7 mm Rolled RD sample annealed at 620 °C for 10 min. There were the C, Mo and Si compositional spikes in the phase boundary and close to the γ grain.

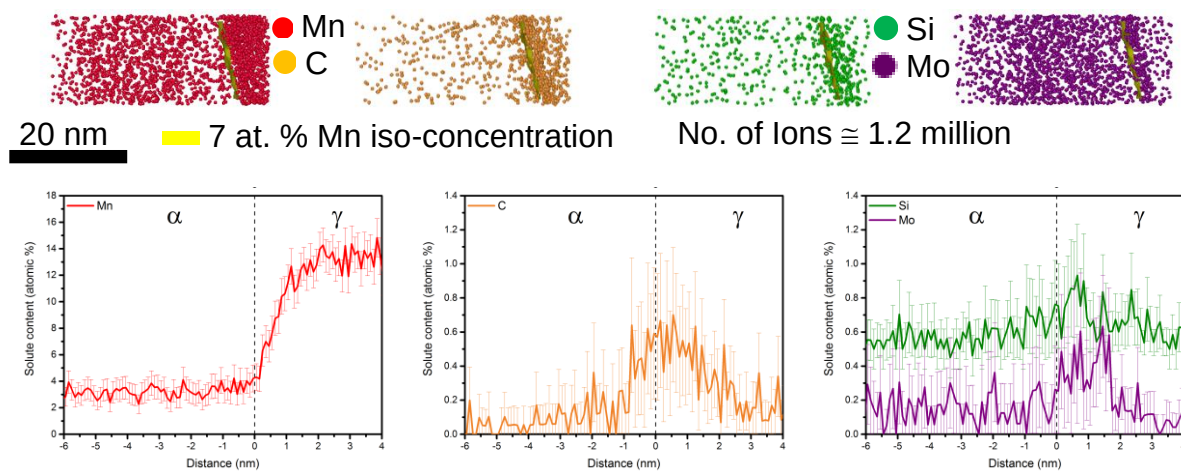


Figure 8.32. Atom probe tomogram of the 0.7 mm FeCMn Rolled RD sample annealed at 620 °C for 10 min along with the compositional profile of the Mn, C, Mo, and Si atoms across the γ - α phase boundary. 7 at. % Mn iso-concentration is shown in the tomogram of the atoms.

Figure 8.33 shows the APT maps of the 0.7 mm Rolled RD sample annealed at 650 °C for 10 min. The figure shows two γ grains and their boundaries. The C, Mo and P concentrations was higher in the grain boundary. The figure also shows that the Mn content was unequal in the γ grains (10.5% and 13%).

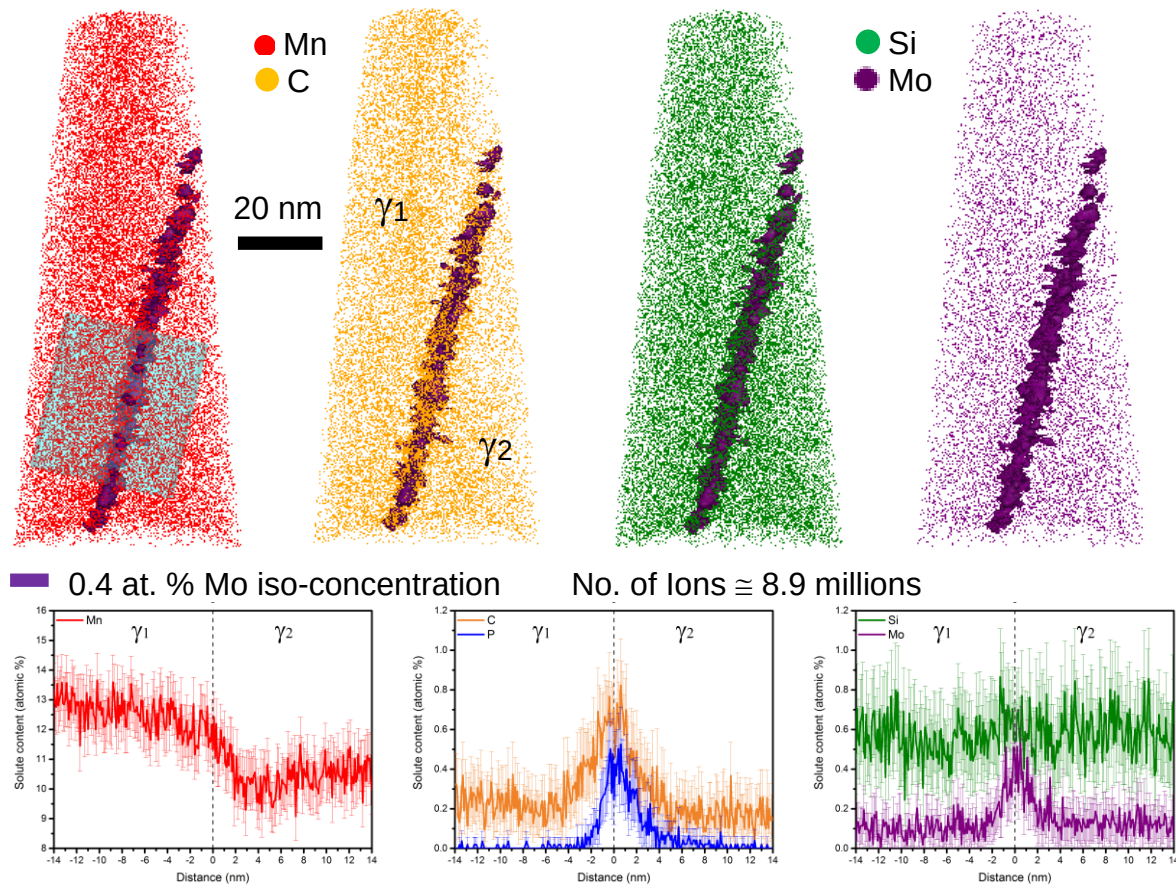


Figure 8.33. Atom probe tomogram of the 0.7 mm FeCMn Rolled RD sample annealed at 650 °C for 10 min along with the compositional profile of the Mn, C, Mo, Si and P atoms across the γ - γ phase boundary. 0.4 at. % Mo iso-concentration is shown in the tomogram of the atoms.

Figure 8.34 shows the APT maps of the 0.7 mm Rolled TD sample annealed at 620 °C for 10 min. There was the Mn atoms compositional spike to 12% in the phase boundary and then it decreased to 10.5% inside the γ grain. As the figure illustrates the C, P, Si and Mo concentrations in the grain boundary were higher than the grains.

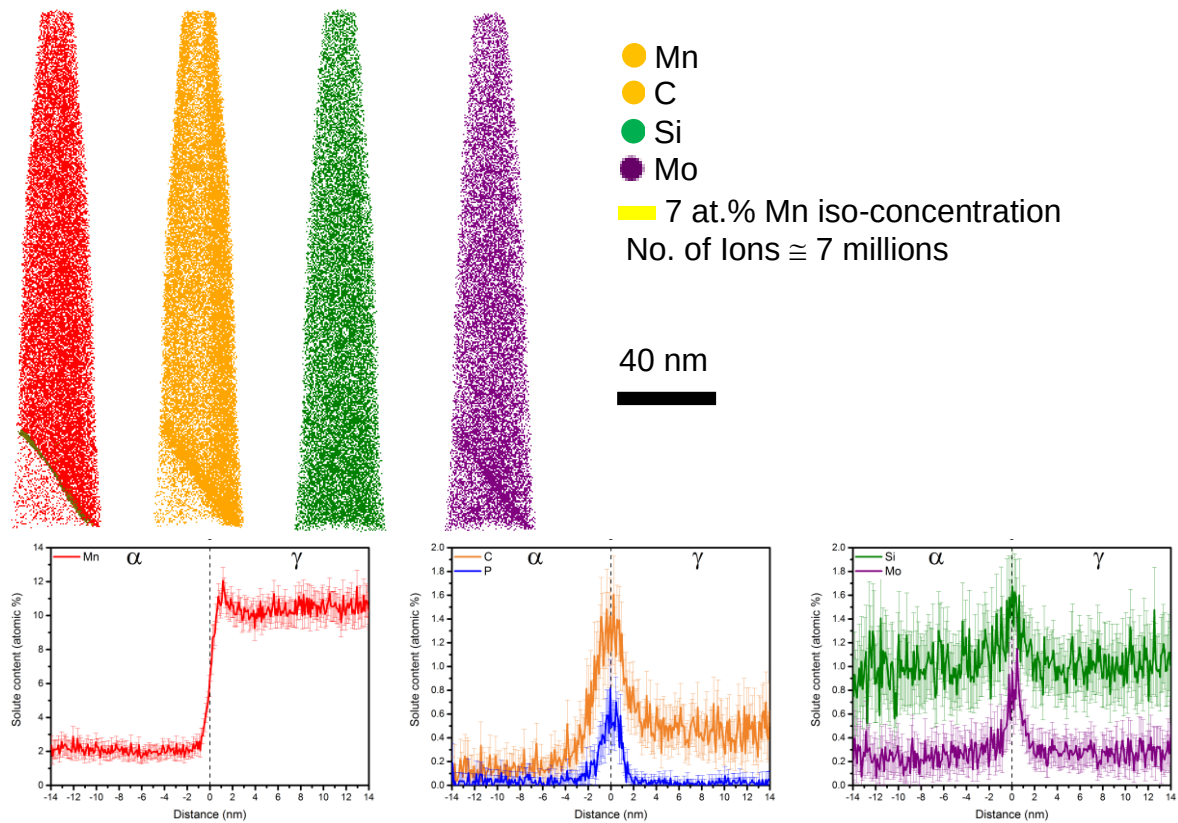


Figure 8.34. Atom probe tomogram of the 0.7 mm FeCMn Rolled TD sample annealed at 620 °C for 10 min along with the compositional profile of the Mn, C, Mo, Si and P atoms across the γ - α phase boundary. 7 at. % Mn iso-concentration is shown in the tomogram of the Mn atoms.

Figure 8.35 shows the APT maps of the 0.7 mm Rolled TD sample annealed at 650 °C for 10 min. At a higher temperature there was not the Mn compositional spike and like previous conditions the C, P, Si and Mo concentrations in the phase boundary was higher than γ and α grains.

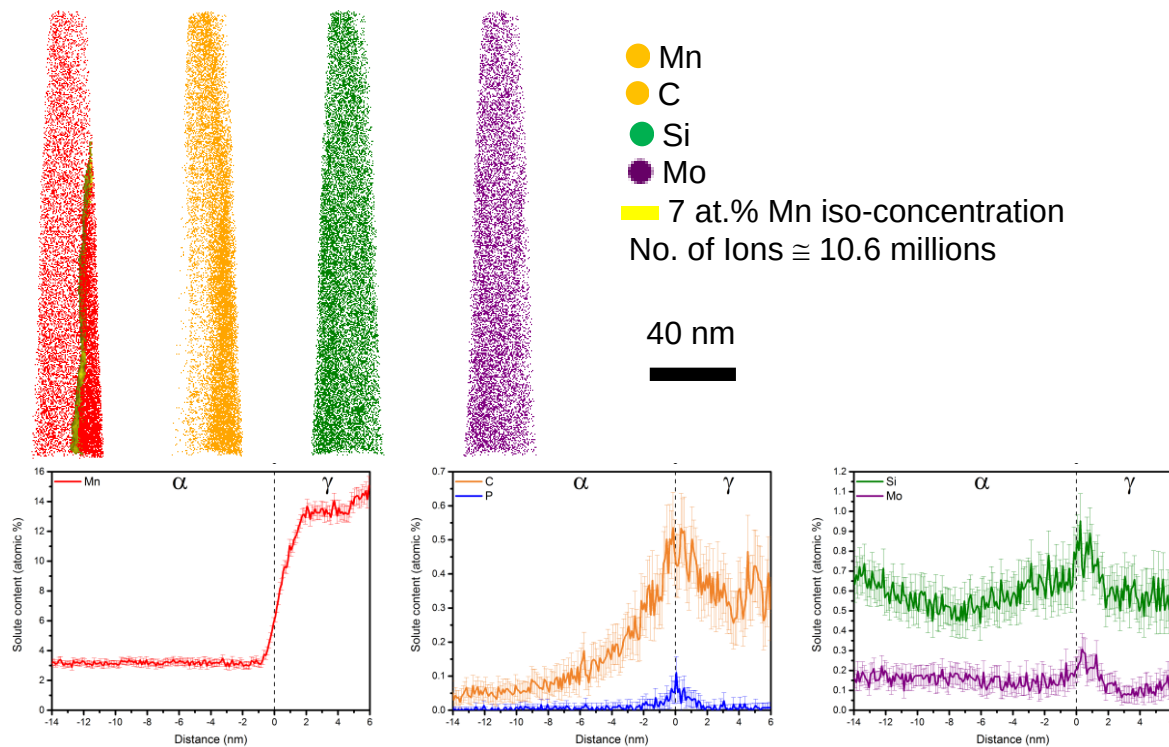


Figure 8.35. Atom probe tomogram of the 0.7 mm FeCMn sample Rolled TD annealed at 650 °C for 10 min along with the compositional profile of the Mn, C, Mo, Si and P atoms across the γ - α phase boundary. 7 at. % Mn iso-concentration is shown in the tomogram of the Mn atoms.

For the α and γ phases, the average concentrations of the alloying elements were measured, and the results are presented under different IA conditions in Figure 8.36. After the annealing at 650 °C, there were insufficient APT data for α grains to calculate the average chemical composition of the α phase. A wide range of the values for each of the annealing conditions was observed based on the atom probe data collected from the γ and α grains with different characteristics. Using these data, the error was calculated with 95% confidence and displayed as a colour band along with the average value for each annealing condition.

Figure 8.36(b) shows that the average Mn content in the 1.4 mm sample after annealing at 620 °C for 10 min was 14.6 ± 1.17 at. %. This amount in the Rolled RD sample after annealing at 620 °C and 650 °C for 10 min decreased to 14.4 ± 1.58 and 12.6 ± 0.64 , respectively. In the Rolled TD sample, after annealing at 620 °C and 650 °C for 10 min, the average Mn content in the γ phase decreased to 13.5 ± 2.48 and 12.1 ± 0.75 , respectively.

The Figure 8.36(b) shows that the influence of the Mn content in the γ was more pronounced at the higher annealing temperatures compared to the lower temperatures. The reduction of the Mn content in the Rolled TD samples was more than the Rolled RD samples after annealing.

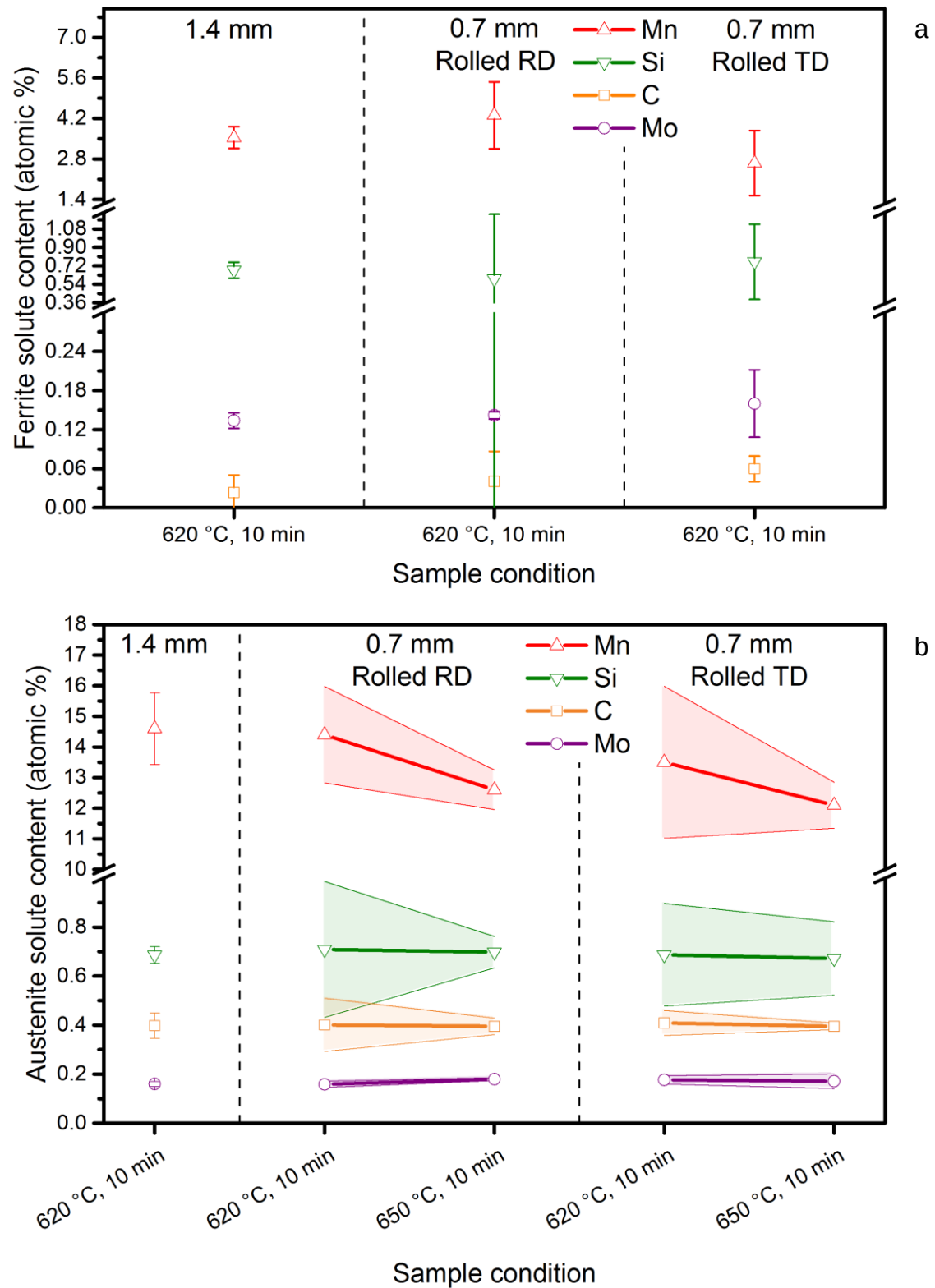


Figure 8.36. Variation in content (at. %) of the solutes in the (a) α and (b) γ phases of 1.4 mm and 0.7 mm FeCMn steel samples annealed at 620 °C and 650 °C for 10 min. The symbols and coloured bands show the average value and the error with 95% confidence, respectively.

The C content was almost constant in all the conditions and was 0.402 ± 0.007 at. %. In the Rolled RD and Rolled TD samples which were annealed at 650 °C the C content was 0.007 and 0.014 at. % , respectively, less than the samples annealed at 620 °C. It is also worth noting, as highlighted in Chapter 5, that the equilibrium carbon content in the γ phase was observed to be lower at 650 °C.

The Si content remains almost constant and was 0.691 ± 0.019 at. % in all the samples. The average Mo content was 0.160 ± 0.010 at. %. in the 1.4 mm sample after annealing at 620 °C for 10 min. This amount in the Rolled RD sample after annealing at 620 °C and 650 °C for 10 min was 0.159 ± 0.013 and 0.180 ± 0.006 , respectively. In the Rolled TD sample, after annealing at 620 °C and 650 °C for 10 min, the average Mo content in the γ phase increased to 0.177 ± 0.017 and 0.171 ± 0.030 , respectively.

8.5 Mechanical Properties

8.5.1 Tensile Test Data

The room temperature engineering stress-strain curves of the FeCMn samples are shown in Figure 8.37. The mechanical properties of the samples are briefed in Table 8.2. According to the results, the rolling and annealing conditions had a significant impact on the deformation characteristics. There were upper and lower yield points and discontinuous yielding (Lüders bands phenomenon) in the tensile test results of the annealed samples.

- Yield Strength

The yield strengths of the 1.4 mm samples annealed at 620 °C for 10 min, in the RD and TD samples were 1253 and 1299 MPa, respectively. The TD tensile sample had a higher yield stress.

After rolling the sample along the RD direction, the yield strength of the RD sample reaches 1900 MPa. After annealing at 620 °C for 10 min, the yield strength was 1256 and 1305 MPa for RD and TD tensile samples, respectively. By increasing the annealing temperature to 650 °C, the yield strength dropped to 582 MPa for the RD sample.

The Rolled TD specimens after annealing at 620 °C for 10 min had the yield strength of 1268 and 1357 MPa for RD and TD tensile samples, respectively. By increasing the annealing temperature to 650 °C, the yield strength dropped to 633 MPa for the RD sample.

The results shows that the 0.7 mm RD and TD tensile samples annealed at 620 °C for 10 min had higher yield strength than the annealed 1.4 mm RD and TD tensile sample..

- Ultimate Tensile Strength

Like the yield strength, the UTS of the TD samples was higher than the RD samples for each annealing condition. The UTS of the 1.4 mm RD and TD samples after annealing at 620 °C for 10 min were 1415 and 1507 MPa, respectively.

After rolling the in RD direction, the UTS of the cold-rolled sample increased to 2024 MPa that was the highest UTS between the samples. The Rolled RD specimens annealed at 620 °C for 10 min showed the UTS of 1394 and 1441 MPa for RD and TD tensile, respectively. This amount in RD tensile sample reached 1512 MPa for the Rolled RD specimen annealed at 650 °C for 10 min.

After cold rolling in TD direction and annealing at 620 °C for 10 min, the RD and TD tensile samples had the UTS of 1398 and 1464 MPa, respectively. the UTS of the RD tensile sample of Rolled TD annealed at 650 °C for 10 min was 1583 MPa which was the second highest UTS amount between the samples.

- Total Elongation

The total elongations of the 0.7 mm Rolled RD and Rolled TD samples annealed at 620 °C for 10 min were higher than the annealed 1.4 mm samples. Like yield strength and the UTS, the TD tensile samples had a higher elongation in comparison to the RD tensile samples after the same annealing conditions. In addition, Rolled TD samples showed a higher elongation in comparison to the Rolled RD samples after the same annealing conditions.

The total elongation of the annealed 1.4 mm at 620 °C for 10 min for the RD and TD tensile samples was 16.7% and 18.2%, respectively. This amount was 1.6% for the Rolled RD cold-rolled sample in the RD tensile sample which was the lowest elongation between the samples as expected.

The total elongation of the Rolled RD samples after annealing for 10 min at 620 °C for both the RD and TD tensile samples was nearly identical at 20.1% and 20.7%, respectively.

The Rolled TD samples after annealing at 620 °C for 10 min showed that the RD tensile sample's total elongation was 23.8% while it increased to 26.5% for the TD tensile sample. The total elongation of the RD tensile sample after annealing at 650 °C for 10 min was 13.8%.

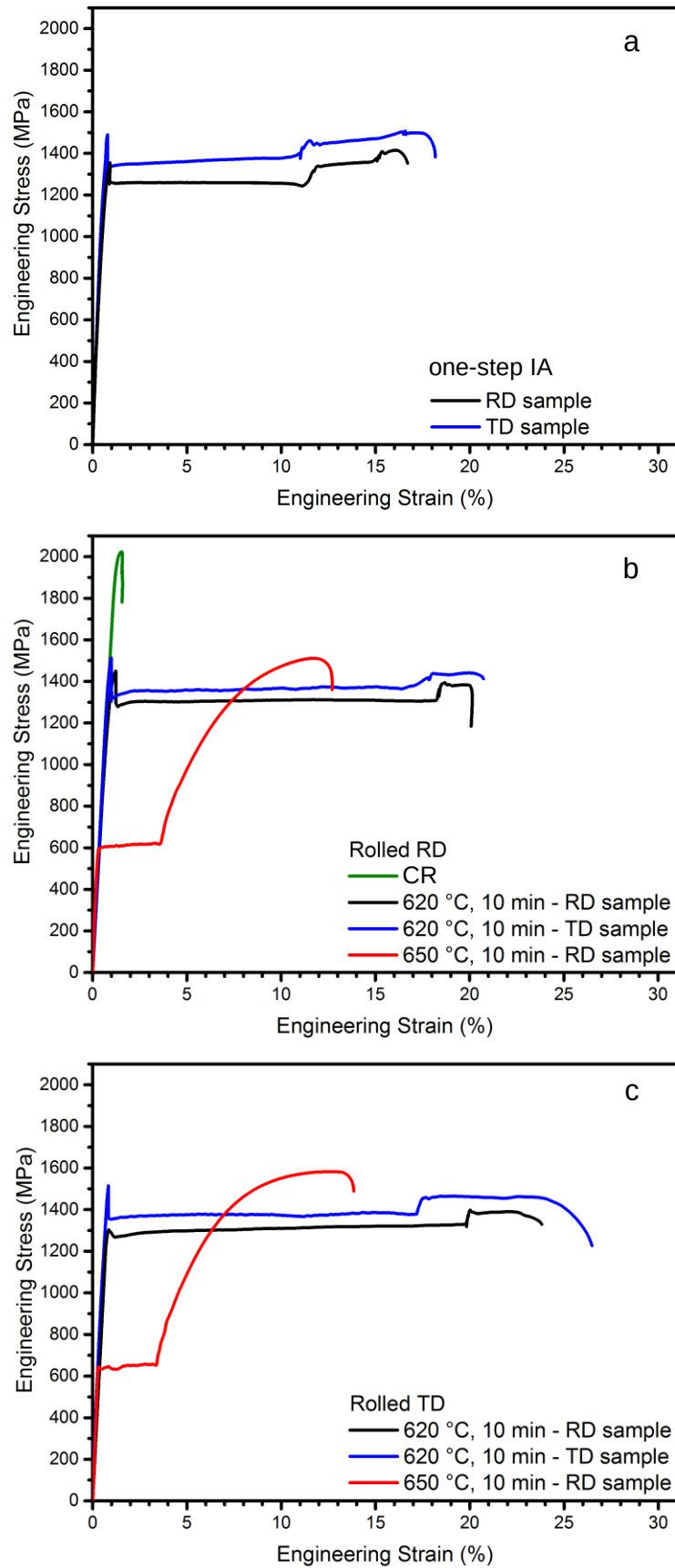


Figure 8.37. Tensile test results (a) one-step IA, (b) Rolled RD and (c) Rolled TD of the FeCMn samples at room temperature. The samples were prepared according to Figure 4.8(b) and tensile experiments were conducted at a constant strain rate of 10^{-3} Sec^{-1}

Table 8.2. Room-temperature tensile test data of the FeCMn samples.

Heat treatment	Rolling Direction %	Heat treatment 10 min	Sample direction	Upper Yield Strength MPa	Lower Yield Strength MPa	Tensile Strength MPa	Strength ratio	Total Elongation %	TSxTE MPa.%E
1.4 mm - 620 °C - 10 min	-	-	RD	1355	1253	1415	0.886	16.7	23631
	-	-	TD	1490	1299	1507	0.862	18.2	27427
	Rolled RD 50%	Cold rolled	RD	--	1900	2024	0.939	1.6	3238
		620 °C	RD	1450	1256	1394	0.901	20.1	28019
			TD	1513	1305	1441	0.906	20.7	29829
		650 °C	RD	596	582	1512	0.385	12.2	18446
	Rolled TD 50%	620 °C	RD	1303	1268	1398	0.907	23.8	33272
			TD	1515	1357	1464	0.927	26.5	38796
		650 °C	RD	644	633	1583	0.400	13.8	21845

- **Strength ratio**

A measure of the work-hardening characteristic of the samples might be obtained by considering the strength ratio (yield strength / ultimate tensile strength). The work-hardening of the 1.4 mm samples were more than 0.7 mm samples after annealing at 620 °C for 10 min. The annealed samples at 650 °C for 10 min had higher work hardening and TRIP effect in comparison to the samples annealed at 620 °C for 10 min. The highest work-hardening was for the Rolled TD sample annealed at 650 °C for 10 min.

- **Product of strength and elongation**

It could be seen the formability of the samples improved after the second annealing at 620 °C for 10 min and reached 33.3 and 38.8 GPa% for the Rolled TD samples. These amounts were 40% more than samples after the first annealing at 620 °C for 10 min.

8.5.2 Hardness Data

Figure 8.38 shows the hardness of the FeCMn samples after different TMP conditions. The hardness of the 1.4 mm AR sample was 507.6 ± 3.2 HV. It decreased to 442.4 ± 3.3 HV after one-step IA at 620 °C for 10 min.

The following RD and TD cold rollings increased the hardness to 556.3 ± 8.9 and 560.6 ± 8.4 HV, respectively. Annealing the Rolled RD and Rolled TD samples resulted in 439.9 ± 3.2 and 438.9 ± 5.5 HV after annealing at 620 °C for 10 min and 349.8 ± 6.6 and 340.6 ± 4.9 HV after annealing at 650 °C for 10 min, respectively.

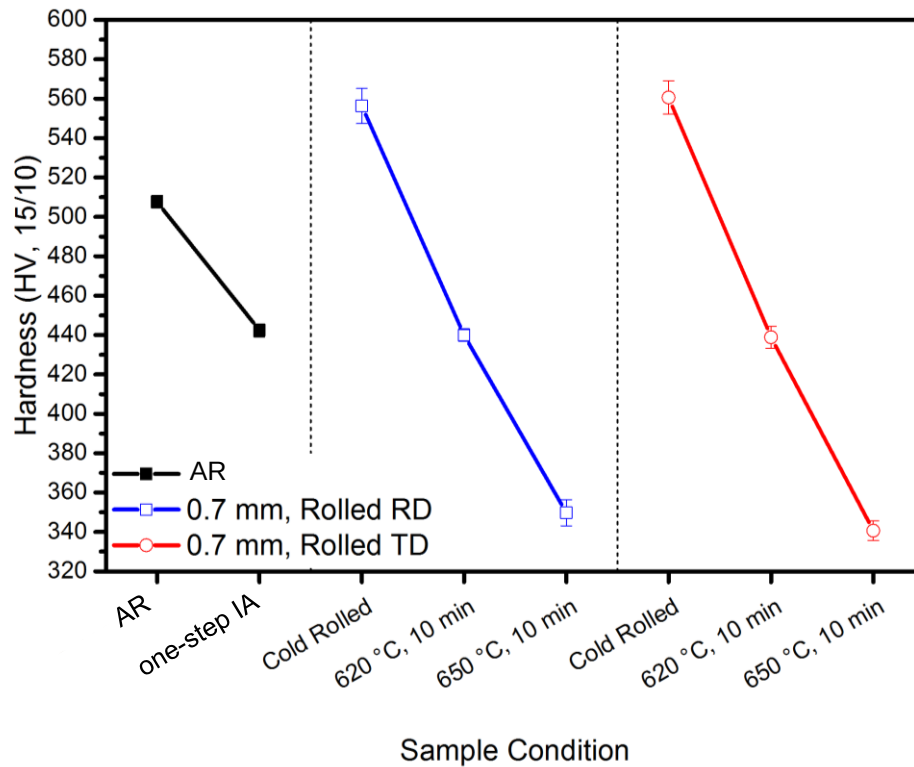


Figure 8.38. Hardness of the FeCMn samples after different TMP conditions under a load of 10 kg and for a dwell time of 15 Sec.

8.6 Summary

In this chapter, we delved into the influence of the TSTMP on the microstructure and mechanical properties of FeCMn steel. To be more precise, we introduced a second stage of cold rolling followed by a subsequent IA process after establishing the initial dual-phase $\alpha+\gamma$ microstructure. This portion of the study also scrutinized the impact of the rolling direction during the second stage, encompassing both the TD and the RD.

It was observed that TSTMP resulted in a reduction in the average grain size of the γ phase and an increase in the total elongation of the samples, particularly in the TD tensile sample. For a comprehensive exploration of the results, refer to the in-depth discussion in Chapter 10.

CHAPTER 9 Effect of the Two-Stage Thermomechanical Process on the FeMnAl Steel

9.1 Thermomechanical Procedure

A description of the TSTMP used in this chapter can be found in Figure 9.1. Initially, the AR specimens with α/α' microstructures underwent IA at 650 °C for 10 minutes and were subsequently quenched in water, a process referred to as the one-step IA. Consequently, in the next stage, the specimens were cold rolled in the TD. However, it's important to note that the results of the FeMn alloy indicated that cold-rolled samples in the TD direction exhibited superior mechanical properties compared to the RD rolling. Therefore, there was no justification for choosing RD rolling, as the primary objective was to enhance mechanical properties through TMPs. The IA was performed on the cold rolled specimens at 620 °C and 650 °C for 3, 5 and 10 min, followed by water quenching. The microstructure and mechanical properties of the FeMnAl alloy were studied after the TMPs.

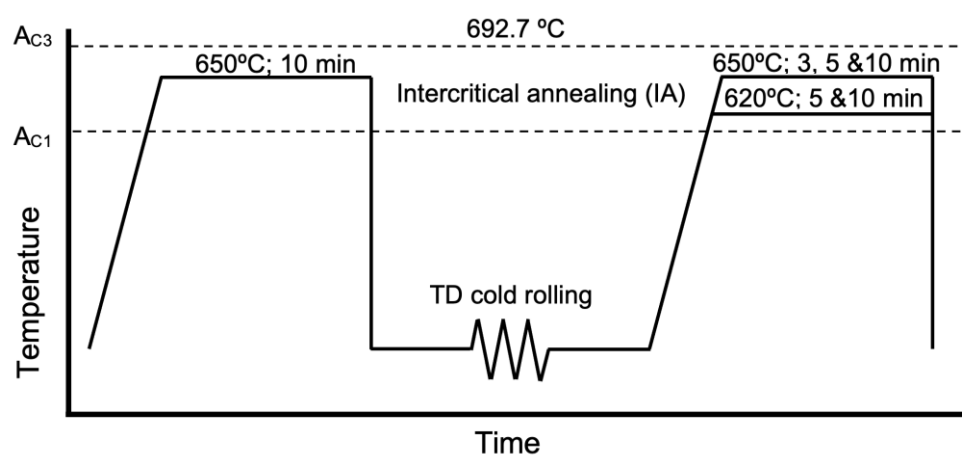


Figure 9.1. The schematic of the TSTMP for the FeMnAl steel.

9.2 Microstructure

9.2.1 Scanning Electron Microscopy Maps and Phase Volume Fractions

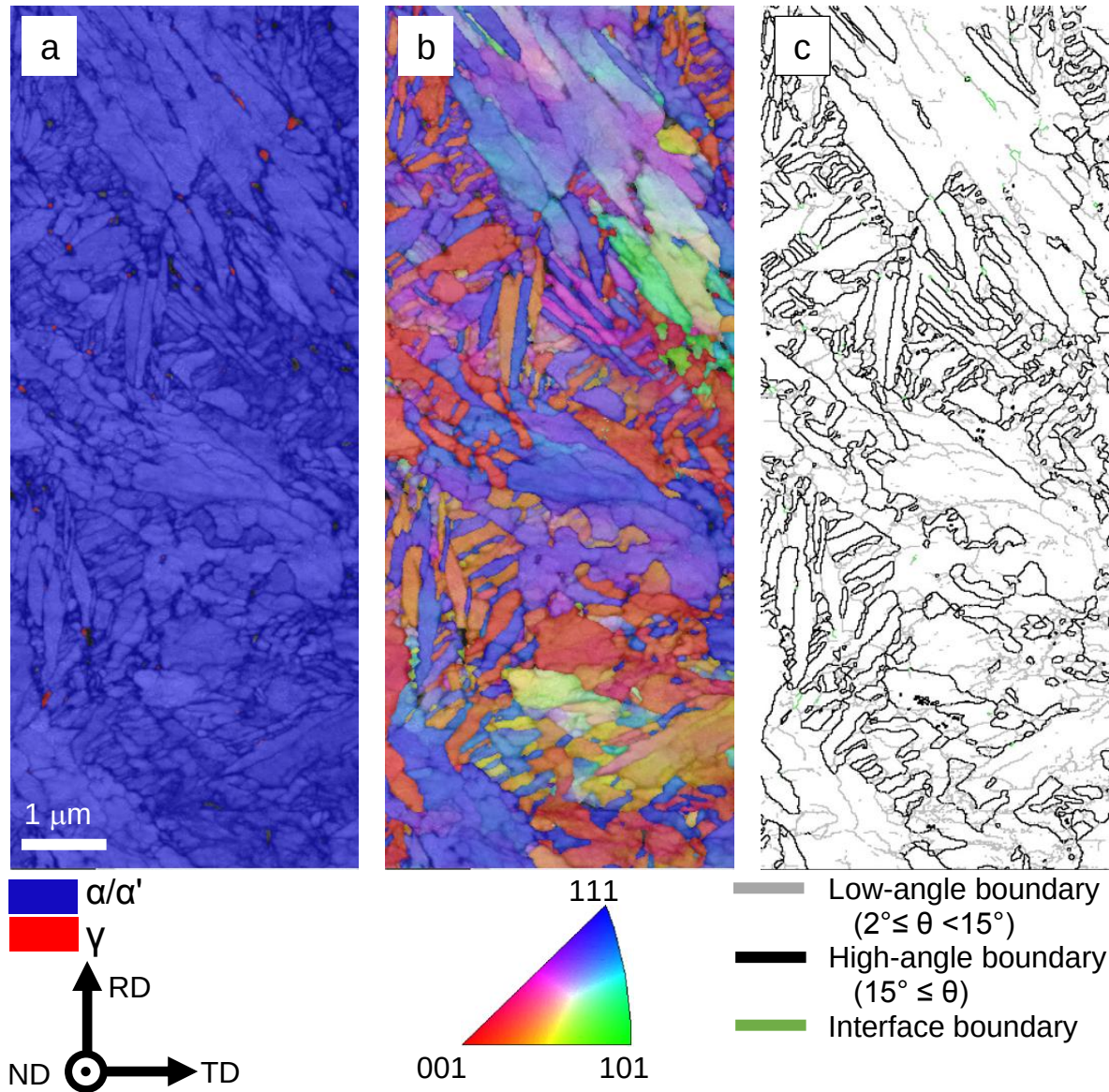


Figure 9.2. SEM-TKD image of the AR FeCMnAl steel, (a) IQ-phase map, (b) IPF-Z map of α/α' , and (c) grains boundary map. On the SEM-TKD maps, red represents γ and blue represents α/α' . The grey and black lines represent low-angle and high-angle boundaries, respectively. The green lines indicate the phase boundaries between the γ and α/α' phases.

A large scanned area was used to extract data from the SEM-TKD IQ-maps like average grain sizes and phase volume fractions. A selected area showed in the figures.

9.2.1.1 Microstructure of the As-received FeCMnAl Steel – 1.8 mm Thickness

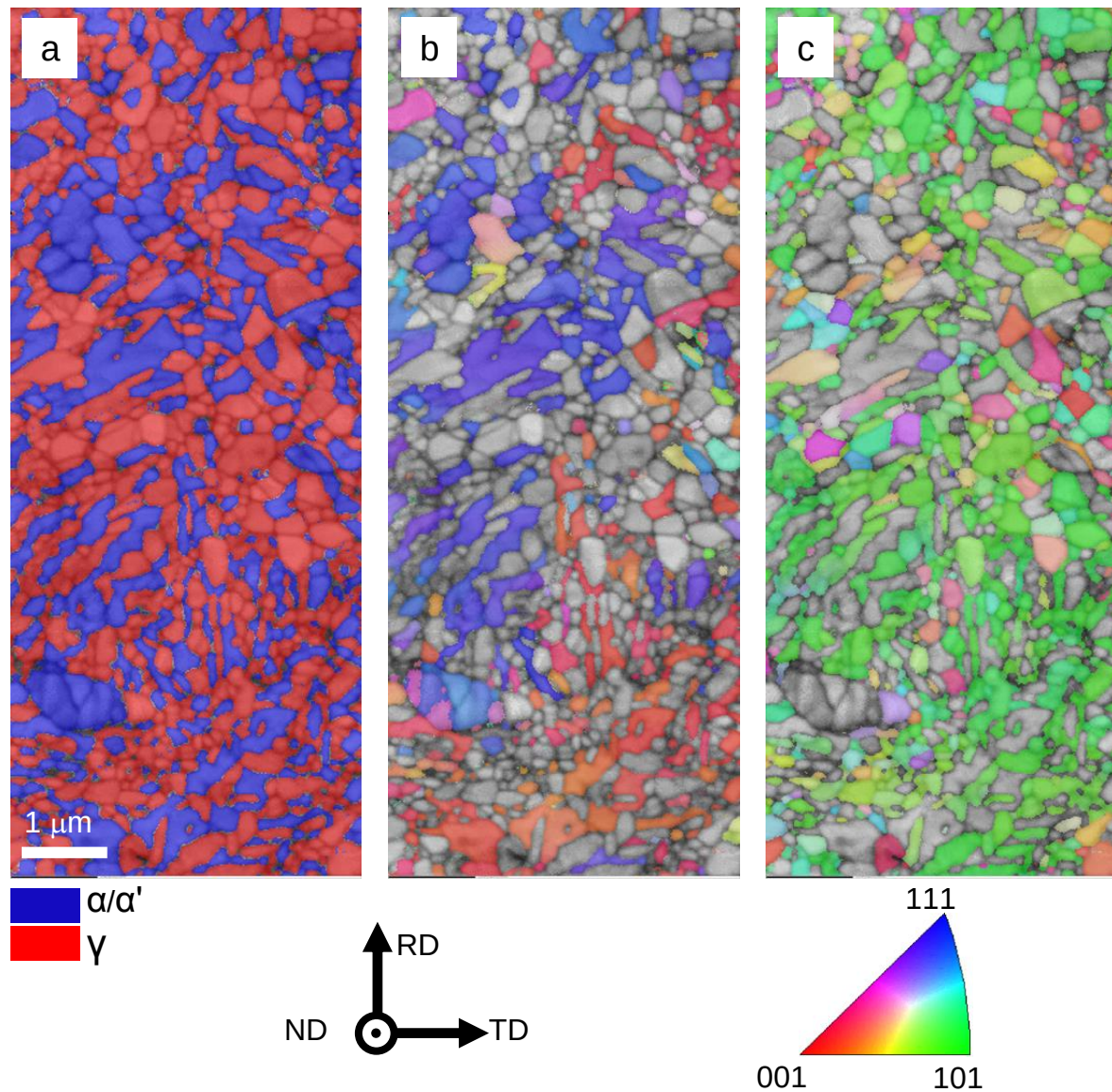


Figure 9.3. SEM-TKD image of the 1.8 mm FeCMnAl steel after the IA at 650 °C for 10 min, (a) IQ-phase map, (b) IPF-Z map of the α/α' , and (c) IPF-Z map of the γ . On the SEM-TKD maps, red represents the γ , and blue represents the α/α' .

In order to examine the microstructure of the cold-rolled FeCMnAl steel in detail, as well as to measure the fraction of the phases and the average grain size, the SEM-TKD technique was employed. Figure 9.2 illustrates the SEM-TKD IQ-phase map, the inverse pole figure (IPF) map in the Z direction and the grain boundary map for cold-rolled FeCMnAl steel. In the SEM-TKD IQ-phase map (Figure 9.2(a)), nano-sized retained γ grains are visible, with an average grain size of 48 nm. On the basis of the large SEM-TKD mapping, the fraction of the retained γ was around 0.5%. The average thickness of the martensite laths was around 244 nm for the

AR FeCMnAl steel. According to the IPF-Z map, the $\{111\}$ plane of the majority of the α/α' grains was parallel to the surface of the specimen.

9.2.1.1 Microstructure of the Annealed FeCMnAl Steel – 1.8 mm Thickness

Figure 9.3 illustrates the IQ-phase map, IPF-Z map of the α/α' , and IPF-Z map of the γ for the 1.8 mm FeCMnAl steel after an IA at 650 °C for 10 min (one-step IA). A large area of the sample, including 4429 of the γ grains and 5034 of the α/α' grains, was scanned with the SEM-TKD technique and a section is presented in Figure 9.3. This approach was used for all the maps in this dissertation, and all data, graphs, numbers and tables were obtained from the large TKD maps.

Figure 9.3 demonstrates that the one-step IA sample had a 60% of the γ phase fraction and an average grain size of 230 nm. The α/α' phase had an average grain size of 201 nm. IPF-Z maps revealed that there was not preferred orientation for α/α' . Also, the $\{101\}$ plane of the majority of the γ grains was parallel to the surface of the specimen. More information will be provided later regarding the texture of each phase.

During the IA, α' transformed into the γ , forming a duplex microstructure containing γ and recrystallized α/α' . There were numerous sites where the γ grains nucleated, such as α' lath boundaries, packet boundaries, triple junctions or boundaries of the parent γ grains. The equiaxed γ grains were found at the packet, triple junctions, and parent γ boundaries; however, the lath-type γ grains were located at the lath boundaries. It resulted in γ grains plexus at the boundaries because of this nucleation mechanism. Due to the high heating rate of the salt bath furnaces (35 °C/s), it was evident that nucleation of the γ grains occurred before the recrystallization of the α grains.

There was a competitive process between the reverse transformation of the γ and the recrystallization of the α/α' . Consequently, the AR FeCMnAl steel with a α/α' structure obtained a duplex microstructure consisting of the equiaxed ultrafine γ and α/α' grains after the IA. Following IA, as seen in Figure 9.4, some α' grains formed. Several factors have been implicated in the formation of α' -martensite, including (1) quenching the annealed specimens and (2) the transformation of the γ grains into α' during the preparation of the specimens [1,2]. Due to the fine size of the reversely transformed γ grains, the laths of α' formed via a single

variant or variants with the same habit plane [1,3]. An example of α' formed following the IA is presented in Figure 9.4.

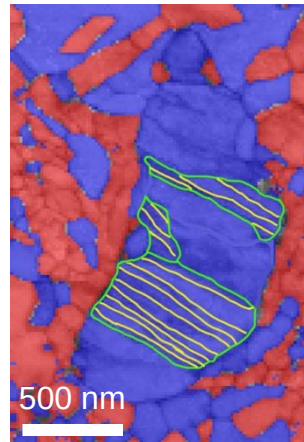


Figure 9.4. α' -martensite formed following the IA at 650 °C for 10 min of the AR FeCMnAl steel. Red and blue indicate the γ and α/α' phases, respectively. Green and yellow lines delineate the boundaries of α' -martensite packets and laths, respectively.

9.2.1.2 Microstructure after Cold Rolling in the Transverse Direction

Following the IA at 650 °C for 10 min, the 1.8 mm FeCMnAl alloy was cold-rolled in TD. As shown in Figure 9.5, thickness reduction and deformation percentage had a significant impact on the microstructure.

Figure 9.5 shows how the γ transformed into α' as more deformation was applied to the sample, resulting in a decrease in the volume fraction of the γ phase.

9.2.1.3 Microstructure of the FeCMnAl Steel after Second Intercritical Annealing

The microstructure evolution after the IA at 620 °C and 650 °C for varying soaking times are illustrated in Figure 9.6 and Figure 9.7. Compared to Figure 9.5(c), it appears that the α' transformed into γ phase during the IA and formed a composite microstructure comprising γ and recrystallized α/α' after both short and long annealing periods. The IA condition again resulted in some α' -martensite grains (marked with white arrows). Figure 9.4 provides an example of this microstructure.

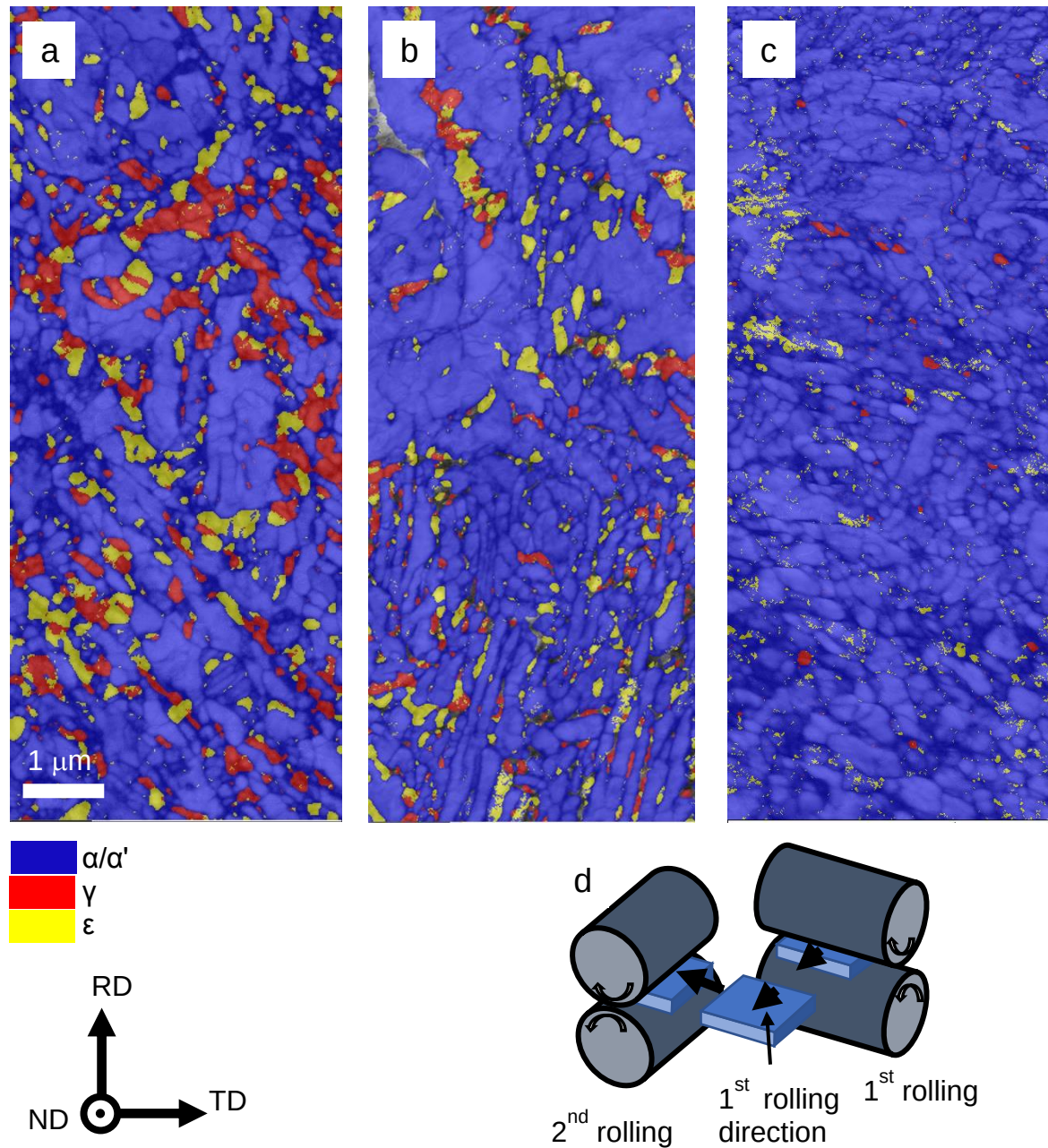


Figure 9.5. Effect of the cold rolling after first IA. SEM-TKD image of the intercritical annealed 1.8 mm FeCMnAl steel at 650 °C for 10 min after (a) 20%, (b) 30%, and (c) 50% cold rolling in TD. On the SEM-TKD maps, red represents the γ , blue represents the α/α' , and yellow represents ϵ .

Figure 9.6 shows that the second IA at 620 °C resulted in 31.6% and 49.9% of the γ phase after 5 and 10 min, respectively. The results of the IA at 650 °C are shown in Figure 9.7. The results showed 52.6%, 55.6%, and 58.4% of the γ phase formation after 3, 5 and 10 min, respectively. Based on these Figures, it can be seen that the temperature and time of the IA had a significant impact on the phase transformation from the α' into the γ . In terms of the γ formation, 3 min of the IA at 650 °C resulted in greater γ phase formation than 10 min of the IA at 620 °C.

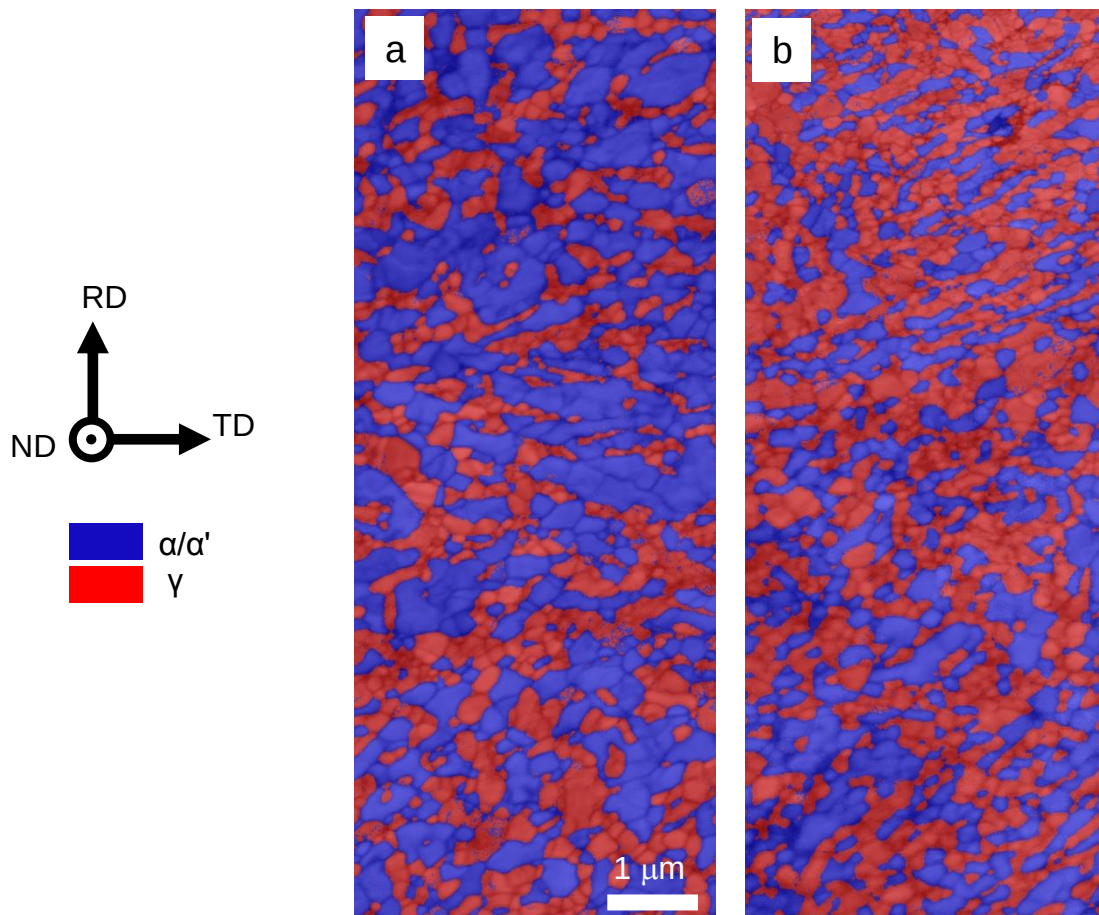


Figure 9.6. SEM-TKD IQ-phase maps of after the second IA at 620 °C for (a) 5 and (b) 10 min. Red represents the γ phase and blue represents the α/α' . The step size was 10 nm.

Figure 9.8 illustrates the volume fractions of each phase in the different samples. The volume fraction of the γ in the sample before cold-rolling was 59.8%. Applying 20%, 30% and 50% thickness reduction with cold-rolling decreased this amount to 8.6%, 4.05% and 0.44%, respectively.

The volume fraction of the ϵ phase after a 20% thickness reduction was 11.1%. As thickness reduced further by 30% and 50%, the volume fraction decreased to 8.23% and 6.81%, respectively. During the cold rolling, the temperature of the samples increased, causing the ϵ phase to partially transformed into α' . As a result, its volume fraction decreased as the thickness was further reduced.

At the second IA, it was observed that a higher annealing temperature led to a greater volume fraction of the γ . Additionally, increasing the annealing time resulted in an increase in the volume fraction of the γ phase. The effect of the time on the increase of the phase volume fraction of the γ was more significant at lower temperatures.

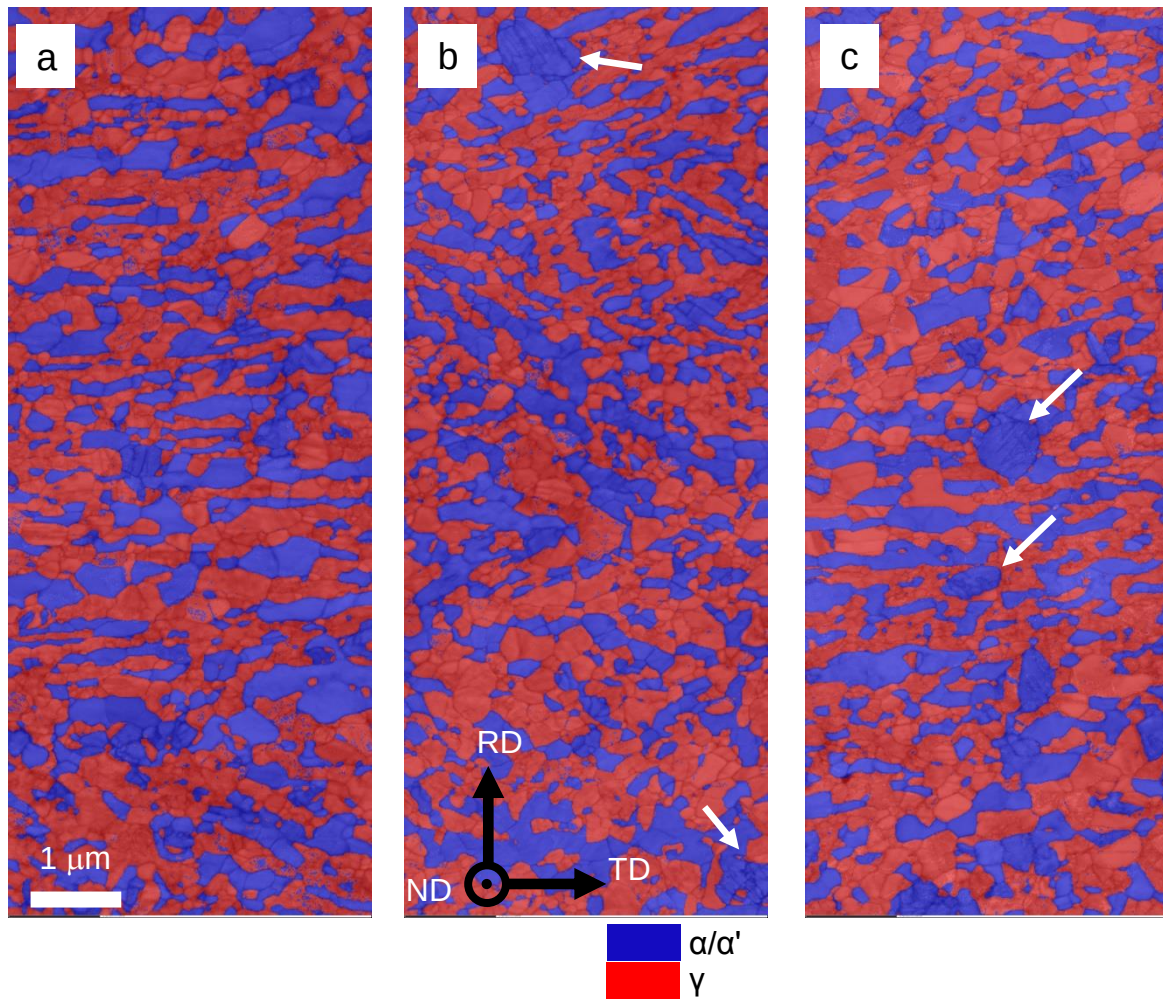


Figure 9.7. SEM-TKD IQ-phase maps after second IA at 650 °C for (a) 3, (b) 5 and (c) 10 min. Red represents the γ phase and blue represents the α/α' . The step size was 10 nm.

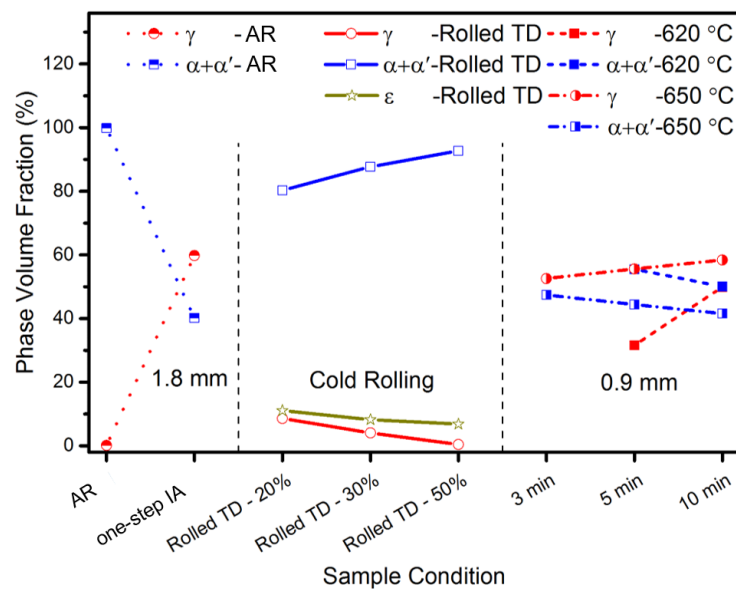


Figure 9.8. Effect of the sample condition on the phase volume fraction of the γ , α/α' , and ϵ from SEM-TKD maps.

9.2.2 Grain Size

9.2.2.1 Effect of the Cold Rolling

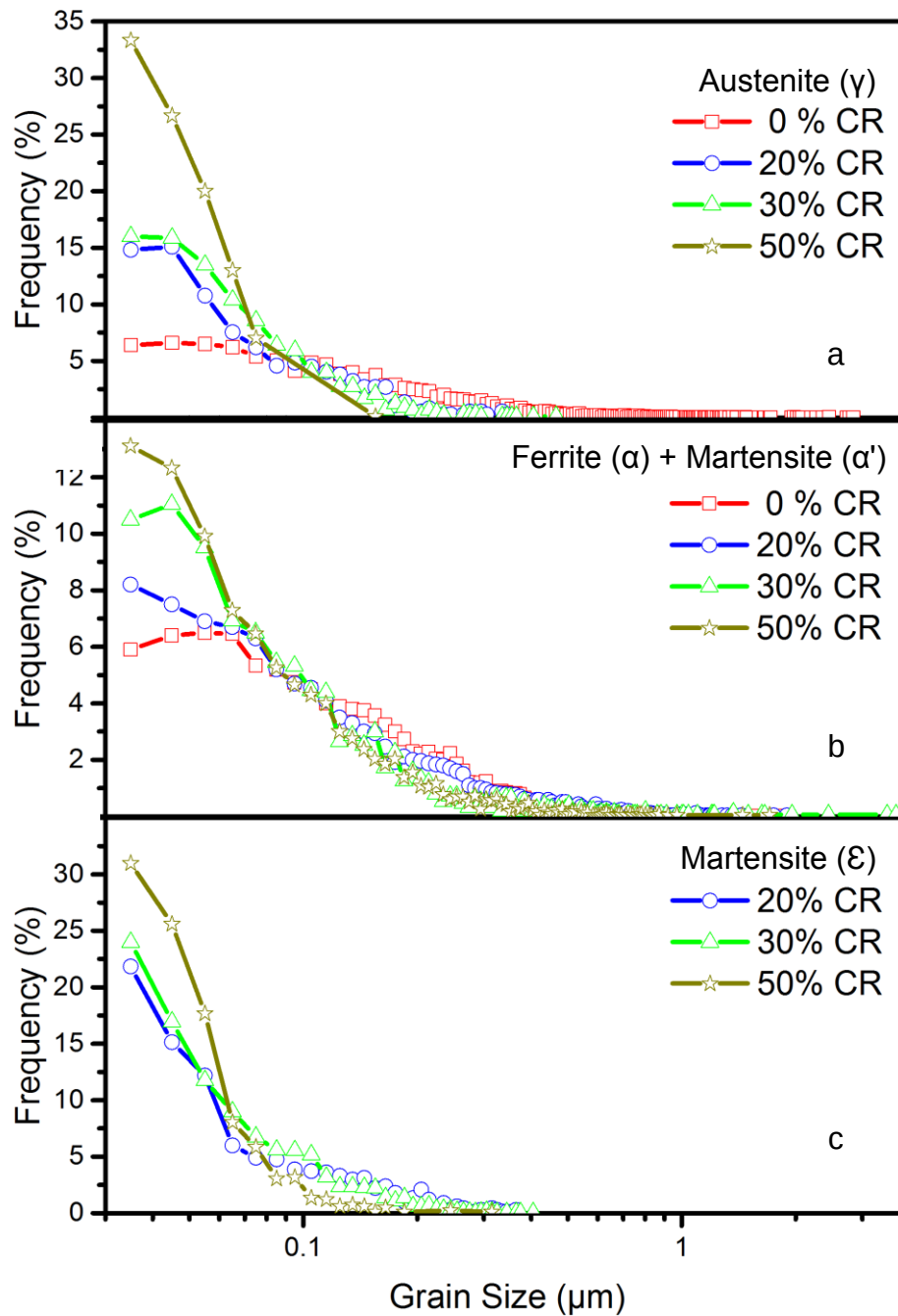


Figure 9.9. Effect of the cold rolling on the grain size distribution of the (a) γ , (b) α/α' , and (c) ϵ from SEM-TKD maps.

The Figure 9.9 depicts the grain size distributions of the γ , α/α' and ϵ phases of the FeCMnAl steel after the IA at 650 °C for 10 min, followed by cold rolling. The 0% CR sample

is the 1.8 mm sample after the IA without cold rolling. According to the figure, cold rolling increased the fraction of the nano-size grains and decreased the fraction of the larger grains.

9.2.2.2 Effect of the Intercritical Annealing

The grain size distribution of the α/α' phase of 0.9 mm FeCMnAl steel after the IA at 620 °C and 650 °C for 3, 5, and 10 min is shown in Figure 9.10. The 0 min sample is a 0.9 mm cold-rolled sample. Figure 9.10 (a) and (b) show that as annealing time increased, the fraction of the nano-sized grains decreased while the fraction of the larger grains increased.

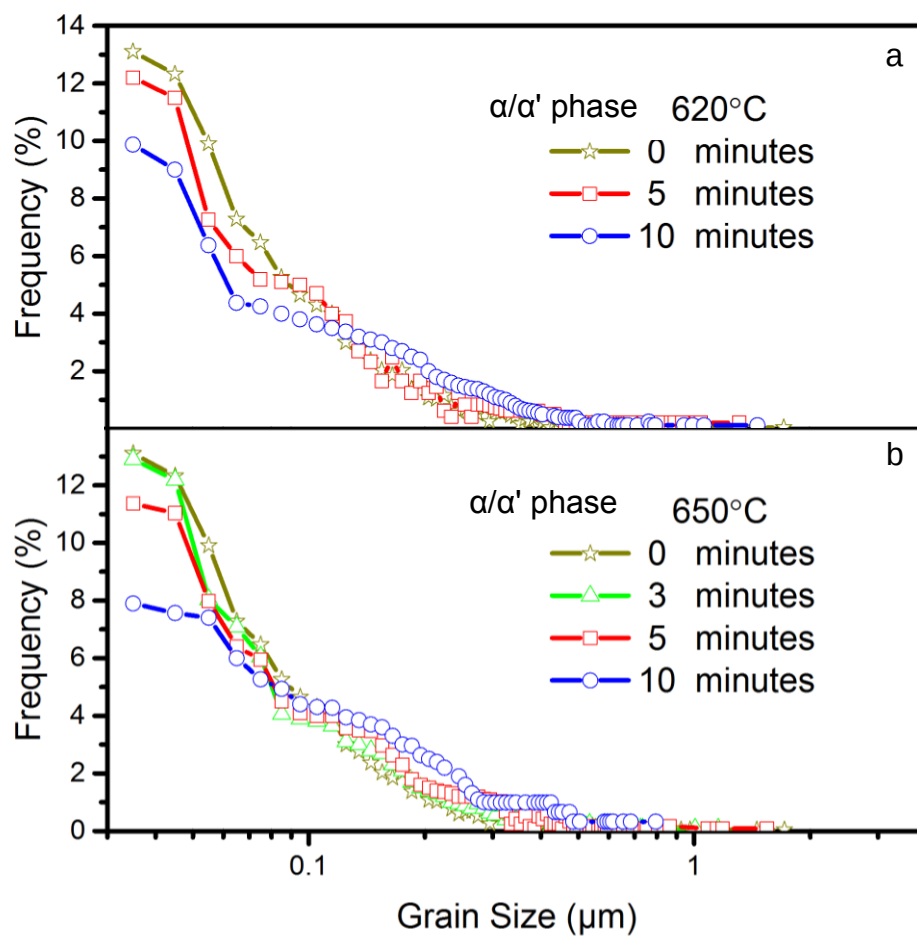


Figure 9.10. Effect of the second IA on grain size distribution of the α/α' phase from SEM-TKD maps at (a) 620 °C and (b) 650 °C.

Figure 9.10 (a) shows the fraction of the nano-sized grains decreased from 53.7% to 42.7% and 34.1% after 0, 5 and 10 min of the IA at 620 °C. In Figure 9.10 (b), the this fraction decreased from 53.7% to 49%, 42.8%, and 33.3% after the IA at 650 °C for 0, 3, 5, and 10 min, respectively.

According to Figure 9.11, the IA at 620 °C and 650 °C resulted in the nucleation and growth of the γ grains, which increased the frequency of the grains larger than 100 nm and decreased the frequency of the grains smaller than 100 nm. Fig. 9.11 (a) illustrates the distribution of the γ grains after the IA at 620 °C for 5 and 10 min, whereas the fraction of the nanosized grains changed from 87.4% to 39% and 40.5% following 0, 5 and 10 min of the IA, respectively. Figure 9.11 (b) shows that the nano-size grains fraction decreased from 87.4% to 42.5%, 37.1% and 32.1% after 0, 3, 5 and 10 min of the IA at 650 °C, respectively.

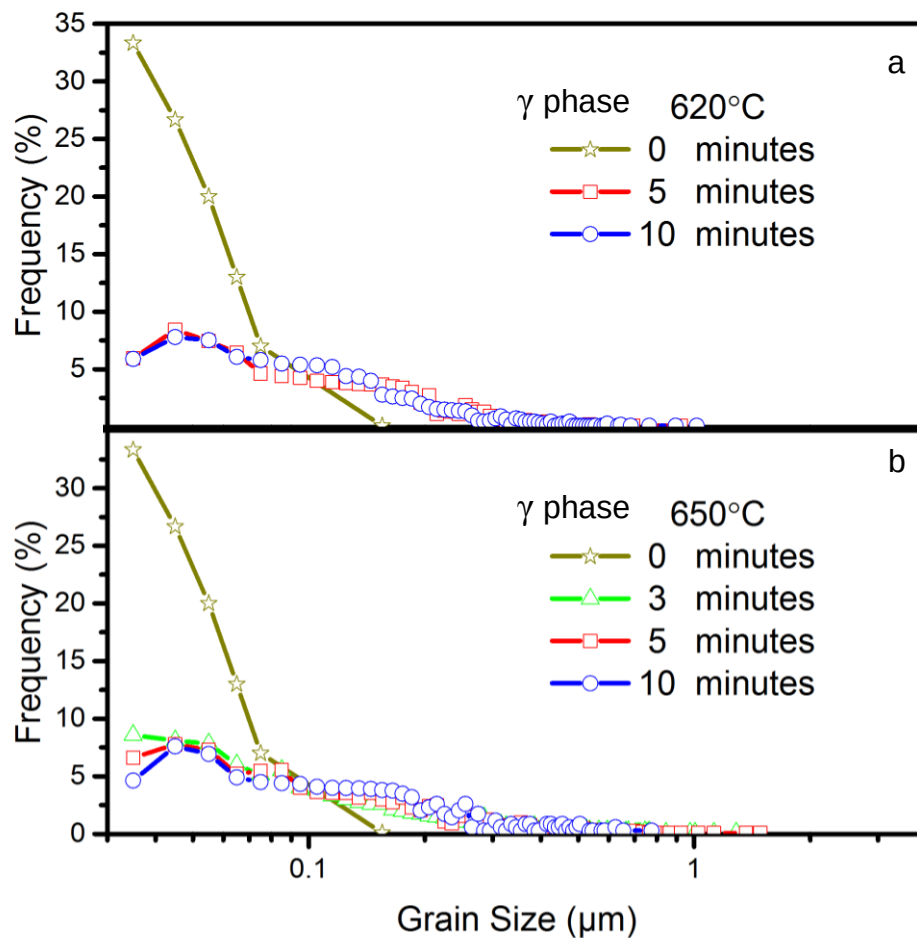


Figure 9.11. Effect of the second IA on grain size distribution of the γ phase from SEM-TKD maps at (a) 620 °C and (b) 650 °C.

As shown in Figure 9.12, the volume fraction of the nanosized grains in the γ and ϵ increased almost linearly. Or, in other words, the larger γ grains transformed into the lath-type α' more rapidly than the smaller grains. As thickness reduced, ϵ grains became smaller as well. In the case of the α/α' phase, the trend was different. As a result of the reducing the thickness from 0% to 30%, there was only a small effect on the volume fraction of the nano-sized grains.

Reducing thickness from 30% to 50% resulted in a 20% increase in the volume fraction of the nano-sized grains. This type of the behaviour can be explained by the fact that, before the 30% reduction in thickness, the majority of the large γ grains transformed into the lath-type α' , and by further cold-rolling, the smaller γ grains transformed into α' .

It can be seen, the α/α' phase had the same nano-size grain volume fraction after the IA at 620 °C and 650 °C and different times. Comparing the volume fraction of the grains less than 50 nm, annealing at 620 °C had a greater volume fraction of the grains in this area than annealing at 650 °C. In addition, Figure 9.12 shows that γ grains grew faster at 650 °C than at 620 °C.

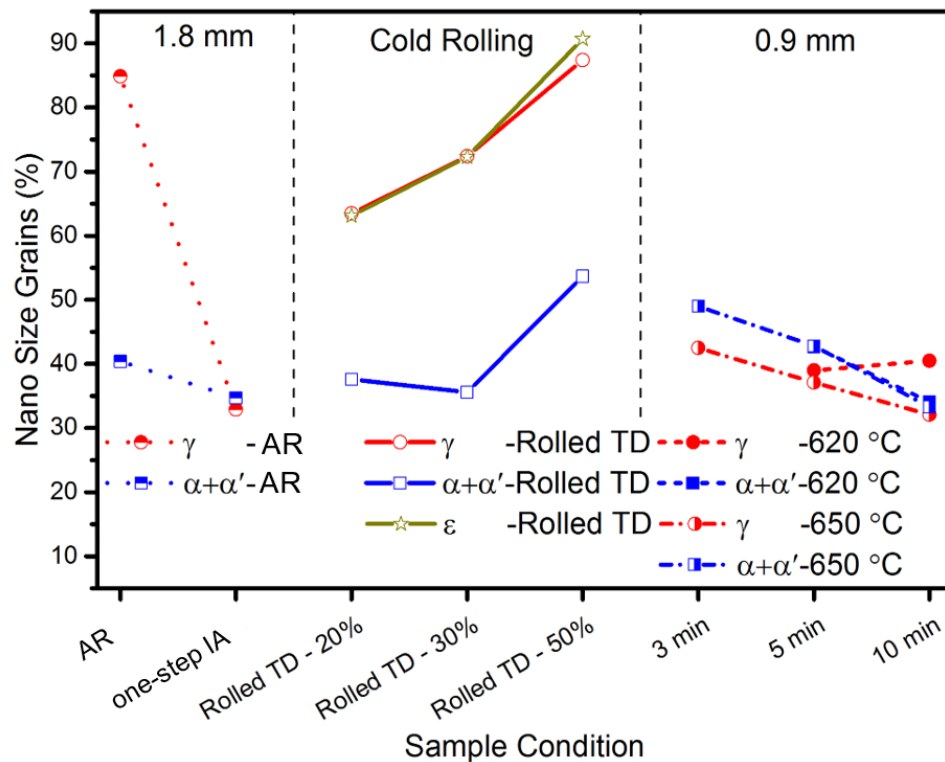


Figure 9.12. Effect of the IA and cold rolling on the nano-size grains volume fraction of the γ , α/α' , and ϵ from SEM-TKD maps.

In Figure 9.13 shows the average grain size of the γ , α/α' , and ϵ phases. The average grain size decreased for the γ phase from 218 nm to 110, 81 and 51 nm, and for the α/α' phase from 194 nm to 188, 157 and 125 nm with a thickness reduction of 0% to 20%, 30% and 50%, respectively.

The average grain size of the γ phase at zero time (50% TD cold-rolled sample) was 51.4 ± 0.9 nm. The γ average grain size increased to 140.5 ± 0.2 and 144.6 ± 0.2 nm after 5 and 10

min of the IA at 620 °C. In addition, the average grain size of the γ increased after the IA at 650 °C for 3, 5 and 10 min to 159.9±0.3, 164.4±0.2 and 171.9±0.8 nm, respectively.

At zero time and before IA, the average grain size of the α/α' phase was 125.3±0.1 nm. After IA for 5 and 10 min at 620 °C, the average grain size of the α/α' phase increased to 150.9±0.7 and 159.2±0.3 nm, respectively. At 650 °C, after 3 min, 5 min, and 10 min of the IA, the average grain size of the α/α' phase increased to 131.9±0.2, 145.3±0.2 and 161.4±0.9 nm, respectively. When the α/α' and the γ phases were compared between 5 and 10 min, it was evident that at 650 °C, the average grain growth rate was twice as high as that at 620 °C.

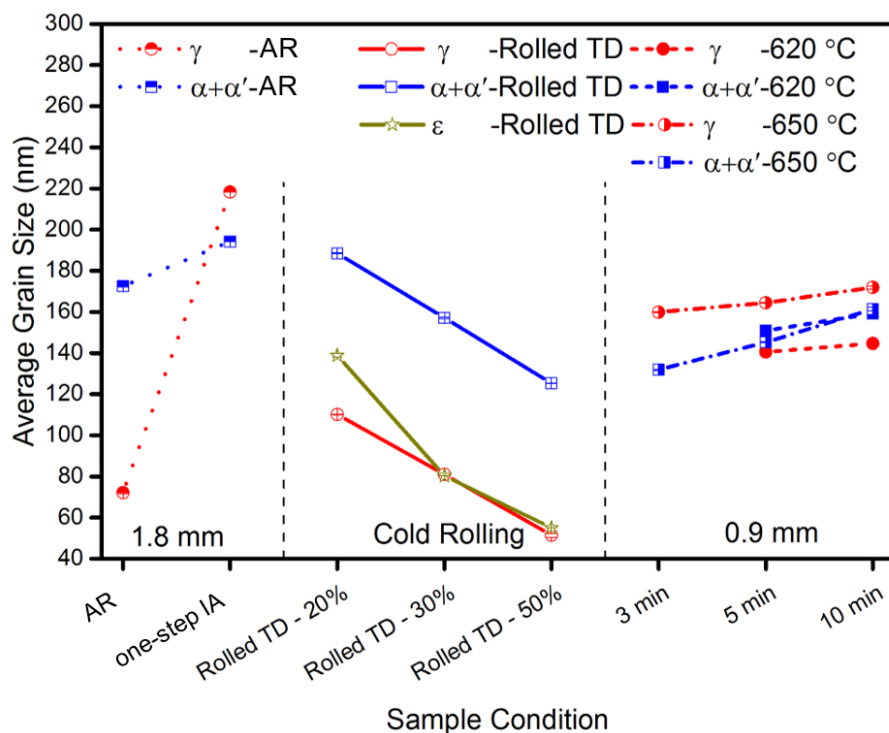


Figure 9.13. Effect of the annealing and cold rolling on the average grain size of the γ , α/α' , and ϵ from SEM-TKD maps.

Figure 9.14 illustrates how the aspect ratios of the γ and α/α' grains increased as the IA process proceeded. As a result of the IA at 620 °C and 650 °C temperatures for 10 min, the aspect ratios of the α/α' grains increased by 13% and 25%, respectively. The IA at 620 °C and 650 °C for 10 min resulted in an increase in the aspect ratio of the γ grains by 9% and 20%, respectively. In other words, γ and α/α' grains exhibited a higher aspect ratio under the IA conditions at higher temperatures.

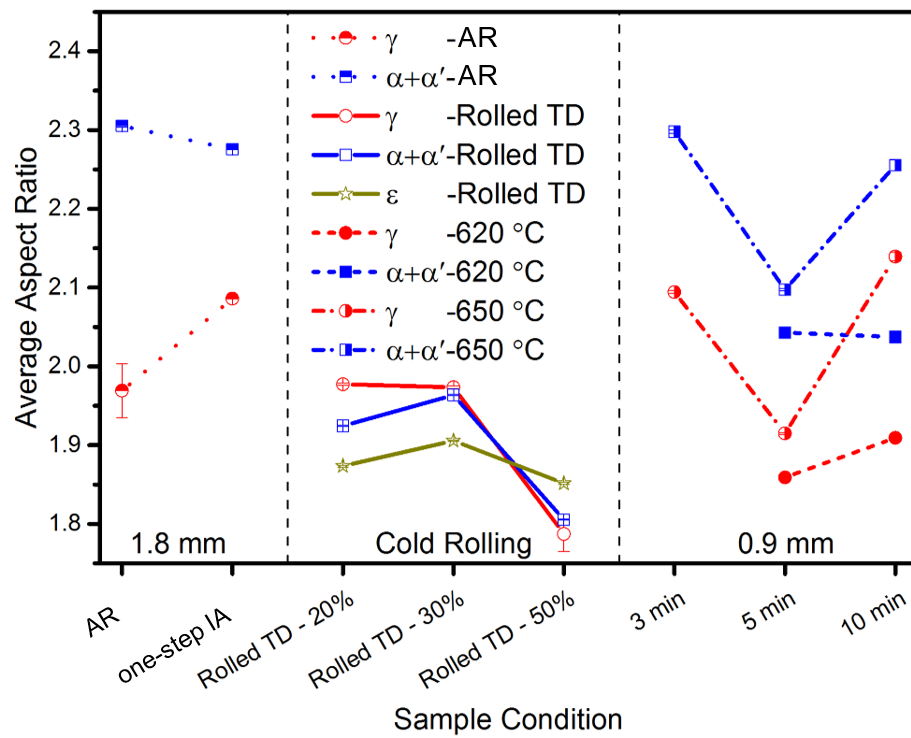


Figure 9.14. Effect of the IA and cold rolling on γ , α/α' and ϵ grains aspect ratio of the FeCMnAl alloy.

9.2.3 Phase Boundary Orientation Relationship

Figures 9.15, 9.16, 9.17 and 9.18 illustrate the phase boundaries, between the γ and α/α' phases of the FeCMnAl steel. The phase boundaries are indicated by the green line and the yellow line, respectively, when the phase boundary deviates by less than 10° and more than 10° from KS OR. These maps were used to extract the histogram distribution of the γ and α/α' phases boundaries.

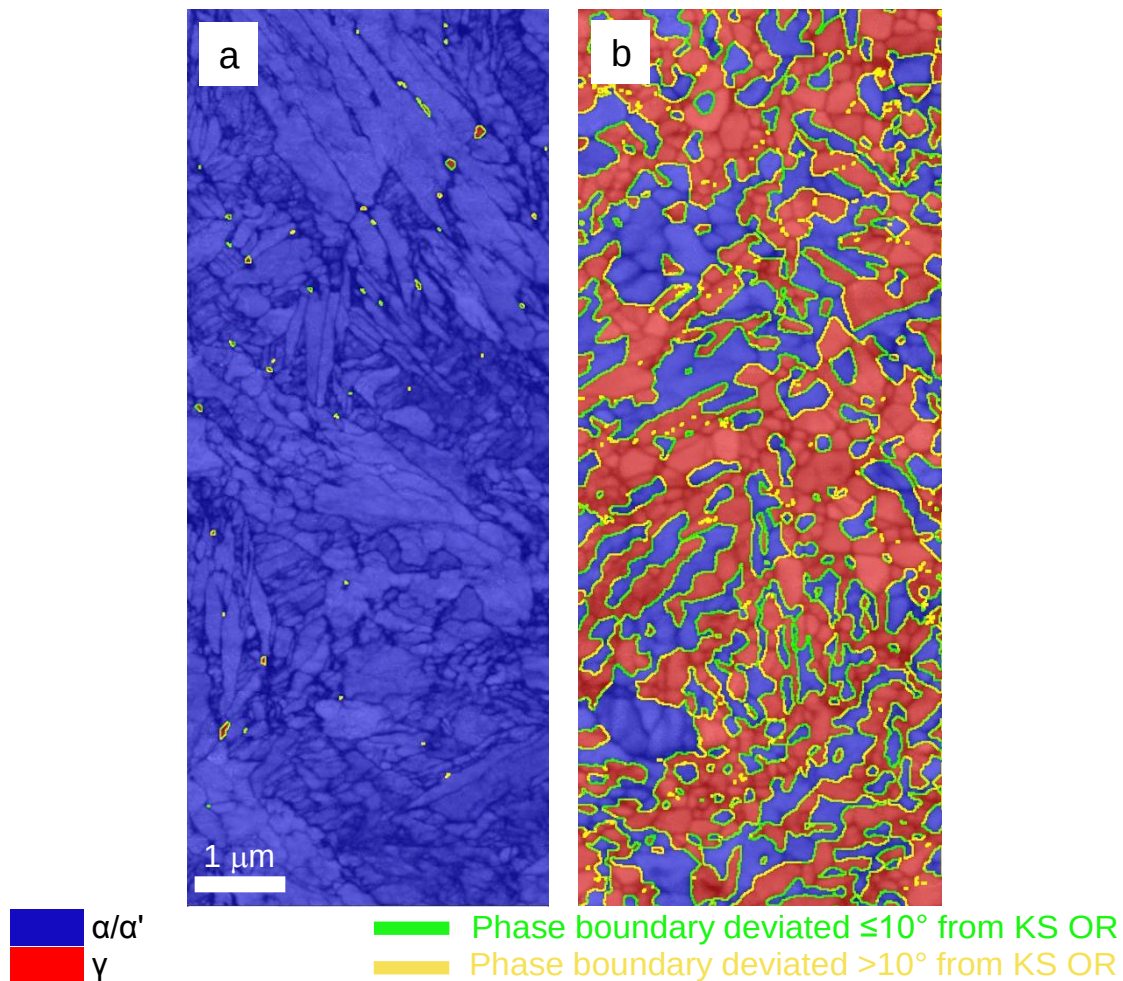


Figure 9.15. SEM-TKD IQ-phase maps of the 1.8 mm FeCMnAl steel (a) AR (b) one-step IA at 650 °C for 10 min. The phase boundaries between the γ and α/α' phases are shown based on KS OR.

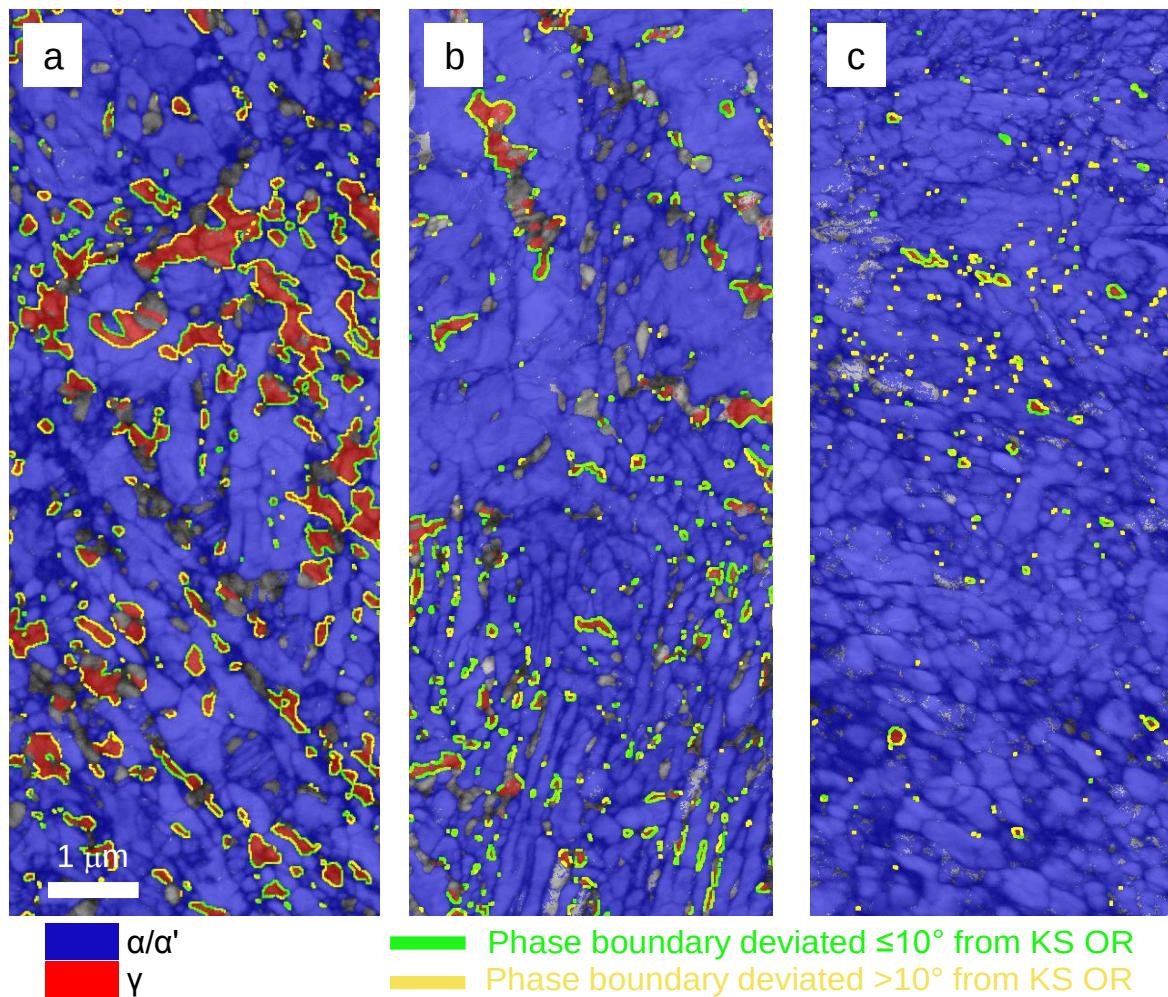


Figure 9.16. The effect of the cold rolling after the first IA on phase boundaries between the γ and α/α' phases based on KS OR after (a) 20%, (b) 30%, and (c) 50% TD cold rolling. On the SEM-TKD maps, red represents the γ , and blue represents the α/α' . ϵ phase was not shown in the maps.

Figure 9.19 illustrates the phase boundary histogram distribution in FeCMnAl steel that differed from KS OR. Figure 9.19 (b) illustrates the effect of the TD cold rolling on phase boundaries. As shown in Figure 9.20, the fraction of the phase boundaries between the γ and α/α' close to the KS OR (with less than 10° deviation) decreased from 80.4% in the one-step IA sample (Figure 9.15) to 79.8% (Figure 9.16 (b)) after 20% TD cold rolling. By further 30% and 50% thickness reduction, the fraction of the γ and α/α' phase boundaries close to KS OR fell to 78.1% and 72.3%, respectively (Figure 9.16 (b) and (c)).

Figure 9.17 shows after the second IA at 620°C for 5 min and 10 min, the fraction of the phase boundaries close to KS OR was 75.3% and 74%, respectively. The fraction did not increase with increasing annealing time, and it remained almost constant.

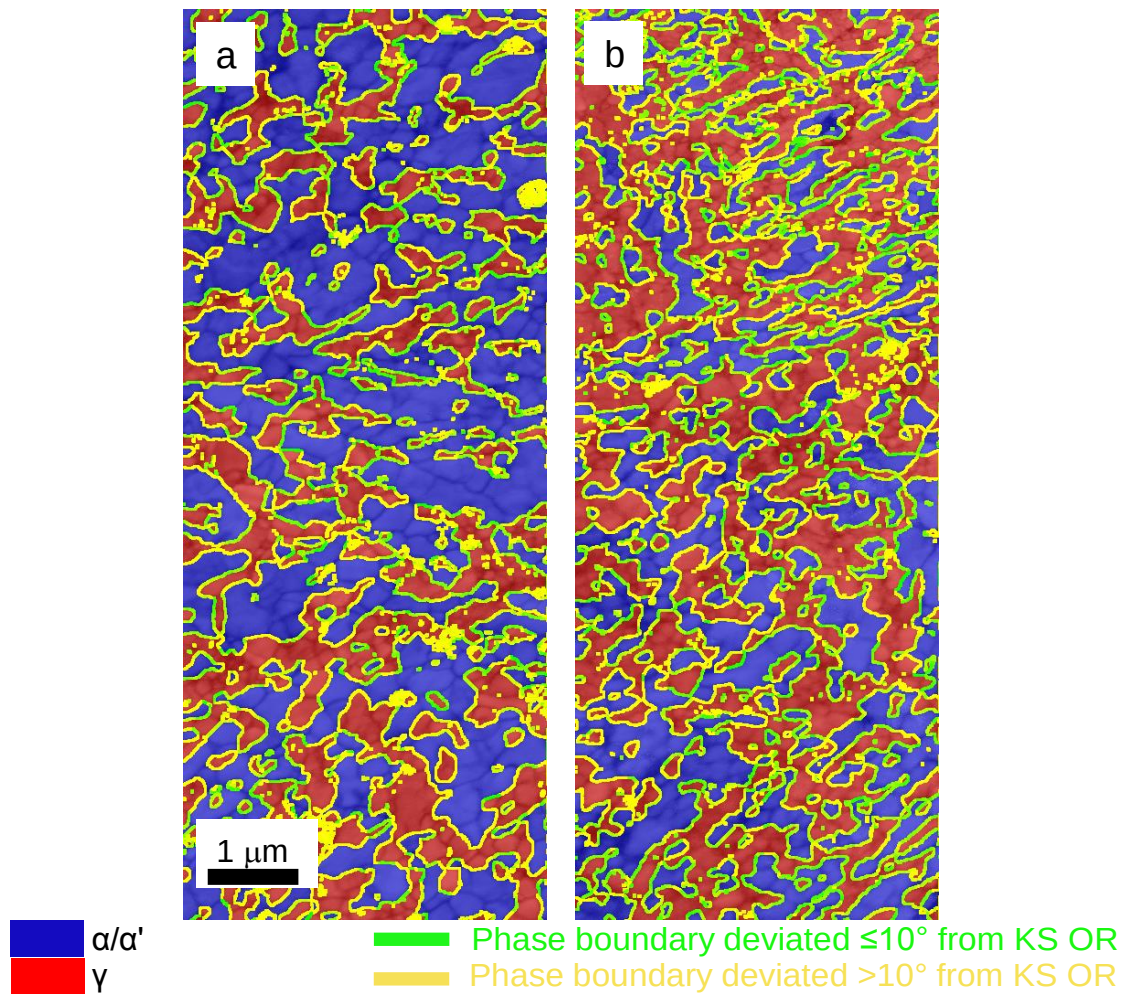


Figure 9.17. SEM-TKD IQ-phase maps of the 0.9 mm FeCMnAl steel samples were annealed at 620 °C for (a) 5 and (b) 10 min. The phase boundaries between the γ and α/α' phases are shown based on KS OR.

Figure 9.19 shows that phase boundaries with 41° to 43° deviation from KS OR, had higher fractions at higher temperatures in the 1.8 mm and 0.9 mm samples after IA. This OR did not appear after cold rolling, indicating that it transformed into α' during the cold rolling process.

Figure 9.20 illustrates the fraction of the phase boundaries between the γ and α/α' with less than 10° deviation from KS OR in FeCMnAl steel samples. The fraction of the phase boundaries near KS OR after the cold rolling and second IA at 620 °C were lower than that for the 1.8 mm one-step IA sample (80.4%). By increasing the temperature of the second IA from 620 °C to 650 °C, this fraction decreased even more. There might be two reasons for this. First, as a result of the cold rolling process, the texture changed and the grains rotated. In addition, it might result from the kinetics of the nucleation and growth of the γ phase. By increasing the

temperature of the IA, the nucleation sites for the γ increased, and the grains grew faster, which affected both the texture as well as the OR between the γ and α/α' phases.

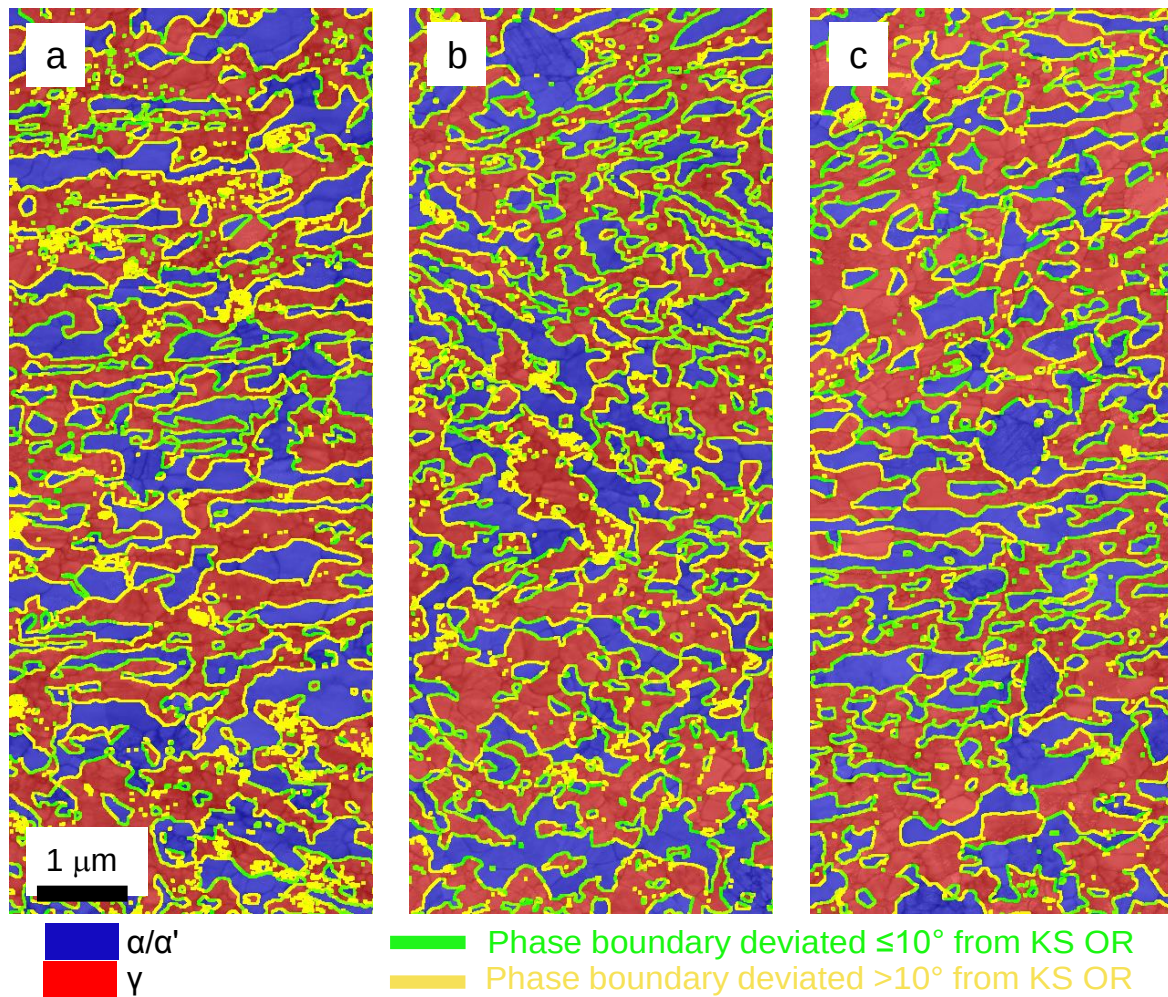


Figure 9.18. SEM-TKD IQ-phase maps of the 0.9 mm FeCMnAl steel samples were annealed at 650 °C for (a) 3, (b) 5 and (c) 10 min. The phase boundaries between the γ and α/α' phases are shown based on KS OR.

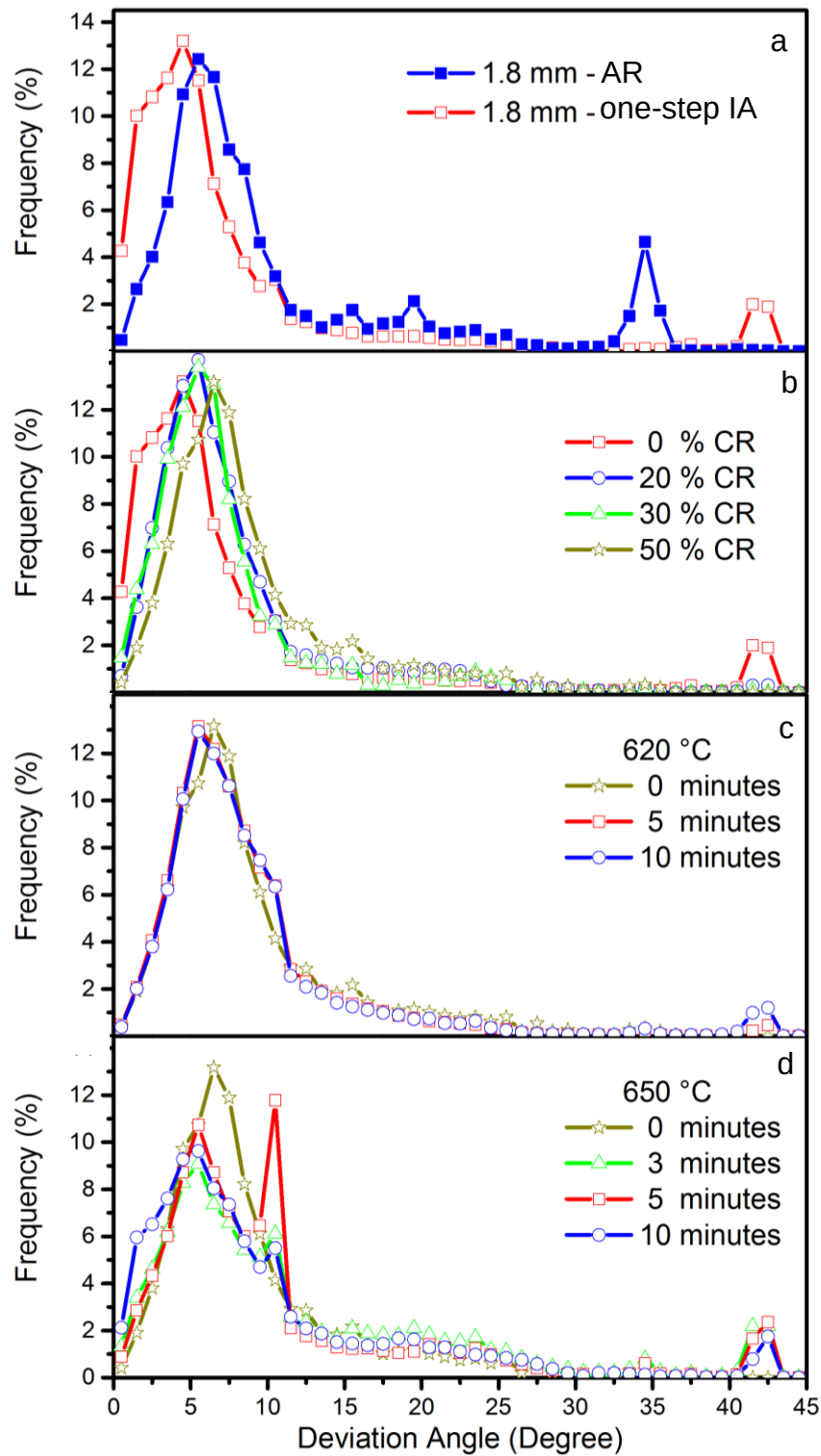


Figure 9.19. Histogram distribution of the γ and α'/α phases boundaries in FeCMnAl steel deviated from KS orientation relationship (a) 1.8 mm AR and one-step IA (b) after cold rolling (c) IA at 620 °C and (d) IA at 650 °C. The 0% CR sample is a 1.8 mm one-step IA sample without cold rolling. The 50% TD cold-rolled sample is the 0 min sample in (c) and (d).

As Figure 9.20 indicates, the phase boundaries close to KS OR after the IA at 650 °C for 3 min was 57.4%, which increased to 61.8% and 67% by increasing annealing time to 5 and 10 min, respectively.

Even though a high percentage of the phase boundaries were close to the KS OR, the question remains as to why not all phase boundaries had a close relationship with the KS OR. The first reason was that all phase boundaries were considered in SEM-TKD maps assessments, while the phase boundary between the one γ grain and one α/α' grain should be close to the KS OR. The second explanation was that the SEM-TKD mapping provided the two-dimensional data of a three-dimensional microstructure. Therefore, an γ grain might have a close relationship with KS OR with a neighbouring α/α' grain in three dimensions on one side.

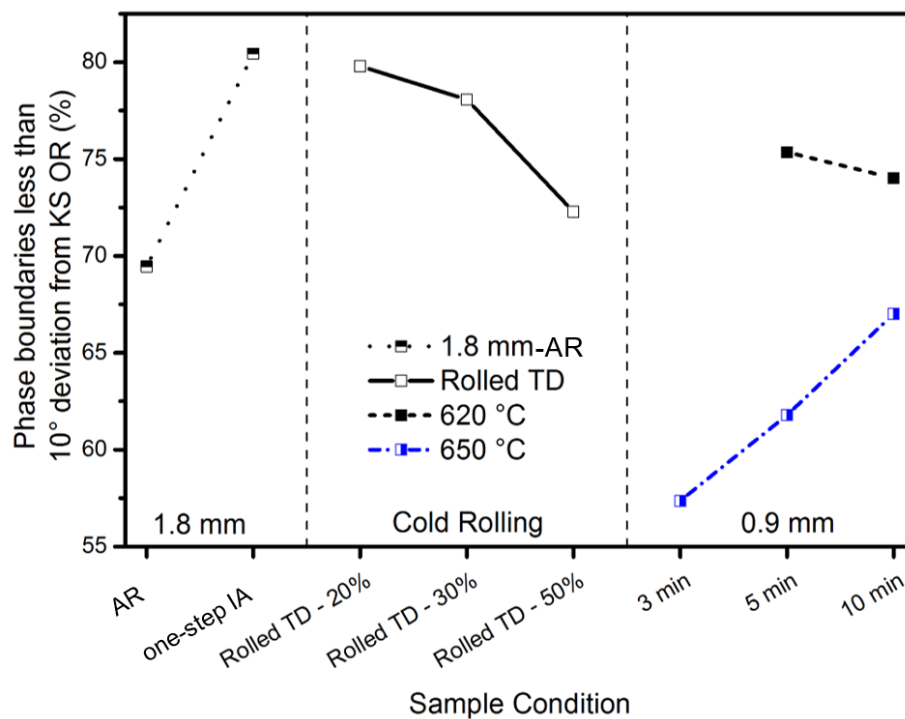


Figure 9.20. The fraction of the phase boundaries between the γ and α/α' with less than 10° deviation from KS OR in FeCMnAl steel samples.

9.2.4 Strain Distribution in the Phases

In order to assess qualitatively and quantitatively the strain distribution in the γ and α/α' , two approaches used: the LAGBs and Kernel average misorientation (KAM) analysis. The dislocation density inside phases evaluated using these approaches as an index.

9.2.4.1 Low-Angle Grain Boundaries

A fraction of the LAGBs, less than 15° , was used as the basis for the method. The γ and α/α' grain boundaries on the SEM-TKD IQ-maps are shown in Appendix 2.

Figure 9.21 illustrates the effect of the cold rolling and IA on the fraction of the LAGBs. In the α/α' phase of the 1.8 mm FeCMnAl one-step IA sample, there was a 71.6% of LAGBs. After a 20% reduction in thickness, the percentage rose to 93.4%. After that, it increased gradually by a further 30% and 50% thickness reduction to 94.5% and 95.1%, respectively. The cold rolling had two effects; first, it partially transformed the γ phase to the α' with the LAGBs; second, it deformed the α/α' phase with the HAGBs to the LAGBs.

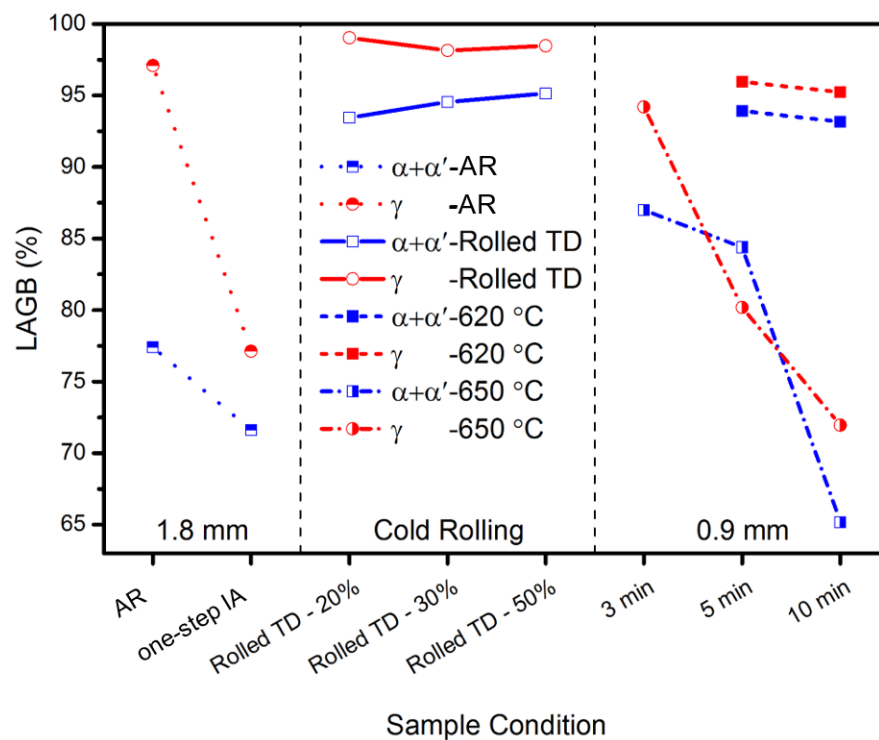


Figure 9.21. Variation of the LAGBs in the γ and α/α' phases of the FeCMnAl steel samples after the cold rolling and IA.

Figure 9.21 shows that the γ phase of the one-step IA sample contained 77.1% of LAGBs. Upon reducing the thickness by 20%, it surged to 99%. It decreased to 89.1% and then increased to 98.5% after further thickness reductions of 30% and 50%, respectively. After 20% thickness reduction, a large amount of the γ transformed into α' , and the retained γ had residual strain. By further thickness reduction to the 30%, the γ phase with a high residual strain transformed to α' , and this might describe the 0.9% drop of the LAGBs. Applying more cold

rolling transformed more γ into α' , which also led to an increase in residual strain in the retained γ phase.

It was shown that the LAGBs within the γ and α/α' phases reduced when the IA process was undertaken, with a faster decrease at higher temperatures. As a result of the IA, three phenomena occurred; first, recrystallization of the γ and α/α' occurred, and this resulted in a reduction of the LAGBs in the phases. Furthermore, the unstable α' transformed into γ phase. As a result, there was a decrease in the amount of the LAGBs in the phases and an increase in the ratio of the HAGBs. Additionally, by increasing the time of the IA, the grain growth increased the ratio of the HAGBs.

9.2.4.2 Kernel Average Misorientation

By using KAM, it is possible to quantify the average misorientation around a measurement point in relation to a defined set of the nearest neighbours. For this study, the KAM values below 5° were used.

Figure 9.22 illustrates the average local misorientation of the γ and α/α' phases inside the FeCMnAl steel samples after the cold rolling and IA. An average local misorientation of 0.45° was determined for the α/α' phase of the 1.8 mm one-step IA sample. The average local misorientation of the α/α' increased to 0.8° , 0.95° , and 1.14° when the thickness was reduced by 20%, 30%, and 50%, respectively. The average local misorientation of the one-step IA (0% CR) sample in the γ phase was 0.47° , as shown in Figure 9.22. By applying thickness reductions of 20%, 30% and 50%, the average local misorientation increased to 0.9° , 1.2° and 1.51° , respectively.

Figure 9.22 shows that the increasing rate of the average local misorientation of the γ was approximately 50% greater than that of the average local misorientation of the α/α' by increasing the thickness reduction. During the cold rolling, the strain increased in γ and α/α' phases; however, the γ phase with high strain partially transformed into α' with lower strain. In this regard, the increasing rate of the γ phase misorientation versus α/α' phase misorientation might be explained.

After the second IA at 620°C for 5 and 10 min, the KAM value of the γ phase decreased from 1.51° to 1.41° and 1° , respectively. A KAM value of the γ at 650°C decreased from 1.51°

to a minimum of 1.03° after 5 min of the IA and then increased with increasing annealing time. The phenomenon is due to the transformation of the unstable γ grains into α' because of quenching or the preparation of the thin foil specimens for the SEM-TKD technique. A study conducted in 2010 has shown that the transformation of the unstable γ into α' led to the generation of the dislocations within the adjacent grains of the γ and α/α' [402]. This might explain why the KAM value in α/α' phase remained nearly constant after the second IA at 620°C and 650°C for more than 5 min despite the recovery and recrystallization of the α/α' phases during the IA.

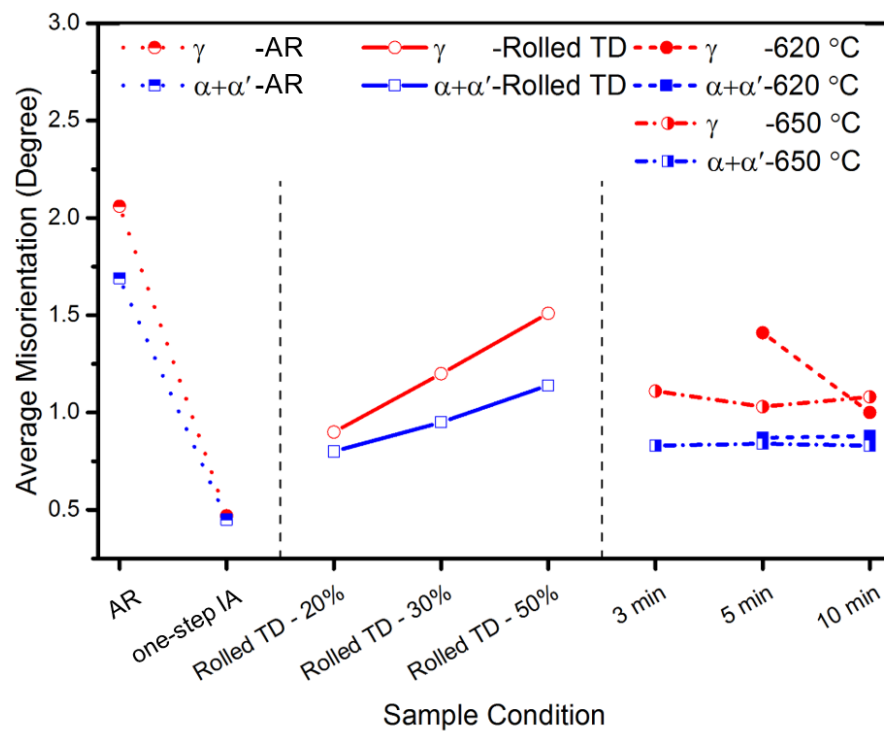


Figure 9.22. The average local misorientation of the γ and α/α' phases of the FeCMnAl steel samples after the cold rolling and IA.

In Appendix 2, local misorientation maps for the FeCMnAl steel samples are presented along with their histogram distributions.

9.2.5 Texture

For the purpose of the assessing the texture of the different phases of the samples, the ODF analysis was performed on the SEM-TKD maps. The 6.2.5 section demonstrated the common texture components and fibers of the BCC and FCC metals and alloys. According to several studies, when BCC metals and alloys are rolled, they develop a fiber texture [35, 363–365].

9.2.5.1 Texture of the As-received FeCMnAl Steel – 1.8 mm Thickness

Figure 9.23 illustrates the texture of the α/α' phase in the 1.8 mm FeCMnAl AR sample. As the figure shows the α/α' phase had strong α -fiber and ε -fiber and a weaker γ -fiber. The texture had a strong $\{001\}\langle 110 \rangle$ component ($f(g) = 12.6$ intensity) that is the deformation product of the Brass component of the γ phase. There was a $(332)[\bar{1}\bar{1}3]$ component ($f(g) = 7.5$) that was the deformation product of the Brass and S components of the γ phase [395, 396].

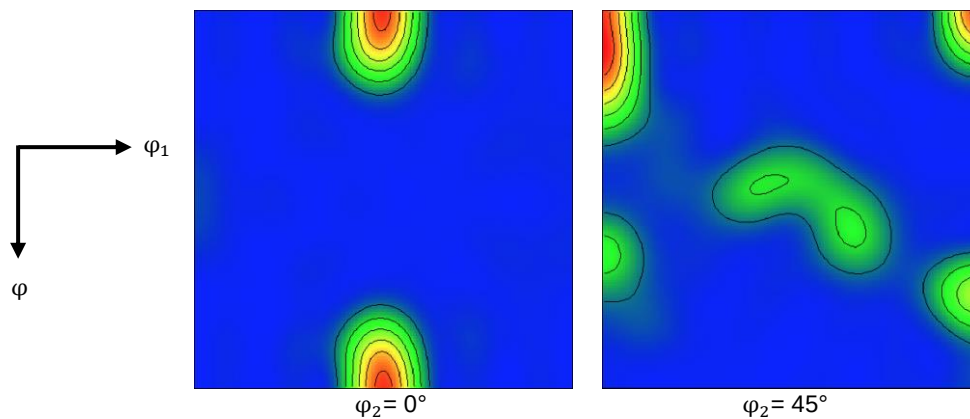


Figure 9.23. The texture of the α/α' phase in the 1.8 mm FeCMnAl AR sample in $\varphi_2 = 0^\circ$ and 45° ODF sections. Contour levels = $2\times$.

Figure 9.24 shows the texture of the retained γ phase in the 1.8 mm FeCMnAl AR sample in $\varphi_2 = 0^\circ, 45^\circ$ and 65° ODF sections. There were α and τ fibers in the texture. A strong S component observed ($f(g) = 12.6$), as well as a weak G component ($f(g) = 8.6$), which were characteristic of the deformed FCC alloys and metals with low stacking fault energy (SFE) [37, 40, 395, 407–412]. There was a texture component at Euler angles of $(46^\circ, 26^\circ, 45^\circ)$ ($\varphi_1^\circ, \varphi^\circ, \varphi_2^\circ$), which was close to $\{114\}\langle 172 \rangle$ orientation and referred to as an X component [25]. The X component was twinned with the B component, emphasizing the importance of the mechanical twinning in the development of a Brass-like texture.

The texture component at Euler angles of (30°, 54°, 15°) was the strongest ($f(g) = 15.7$), and it closely matched the S component.

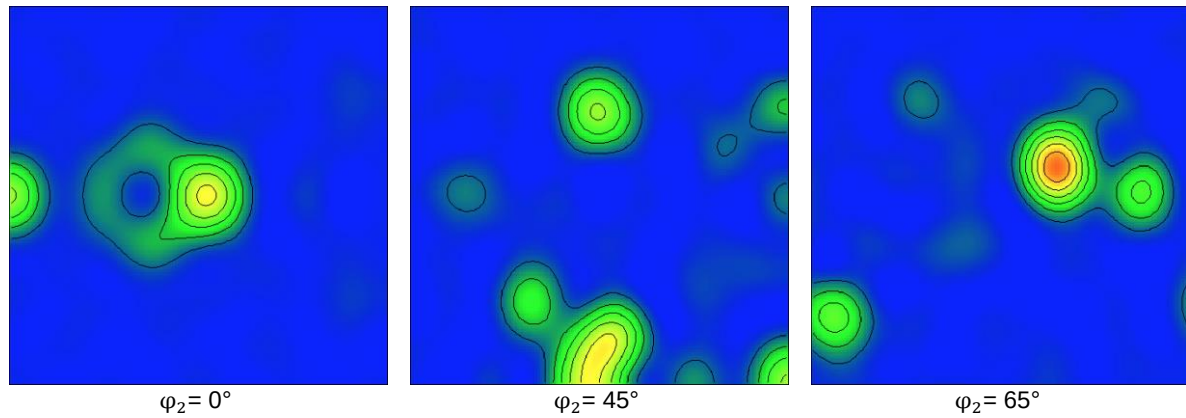


Figure 9.24. The texture of the retained γ in the 1.8 mm AR FeCMnAl steel sample in $\varphi_2 = 0^\circ$, 45° and 65° ODF sections. Contour levels = $2x$.

9.2.5.2 Texture of the 1.8 mm FeCMnAl Steel after Intercritical Annealing at 650 °C for 10 Minutes

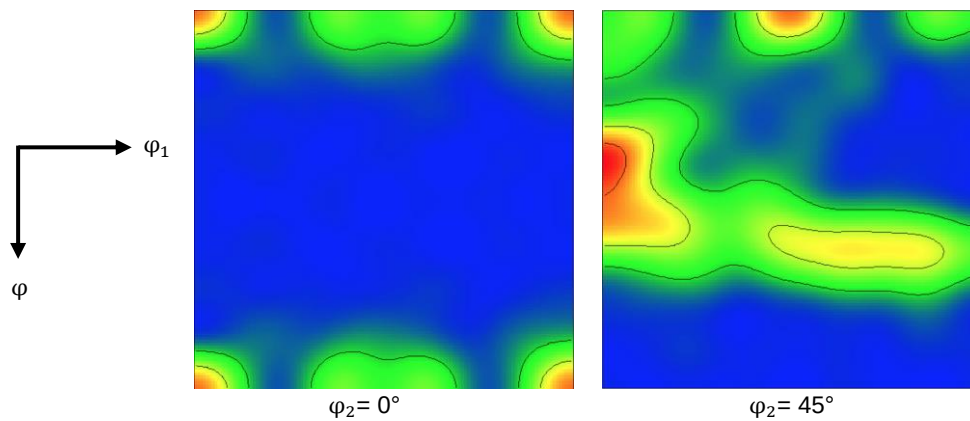


Figure 9.25. The texture of the α/α' phase in the 1.8 mm one-step IA FeCMnAl sample in $\varphi_2 = 0^\circ$ and 45° ODF sections. Contour levels = $2x$.

Figure 9.25 shows the texture of the α/α' phase in the 1.8 mm FeCMnAl one-step IA sample in $\varphi_2 = 0^\circ$ and 45° ODF sections. Figure 9.26 shows the fibers of the α/α' phase in the 1.8 mm sample before and after the IA at 650 °C for 10 min. these figures show the α -fiber, γ -fiber and θ -fiber. It shows the maximum intensity was on (112)[1 $\bar{1}$ 0] ($f(g) = 6.81$) and a weaker intensity on (001)[100] ($f(g) = 5.9$) components.

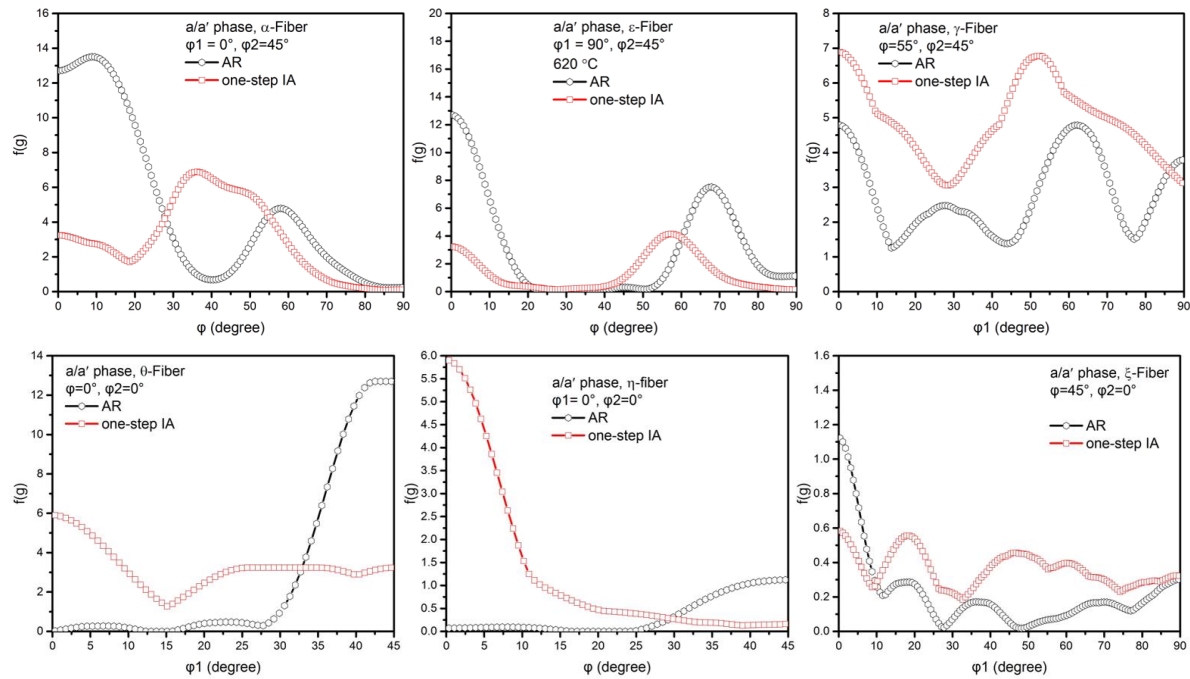


Figure 9.26. The fibers of the α/α' phase in the 1.8 mm AR and one-step IA FeCMnAl steel.

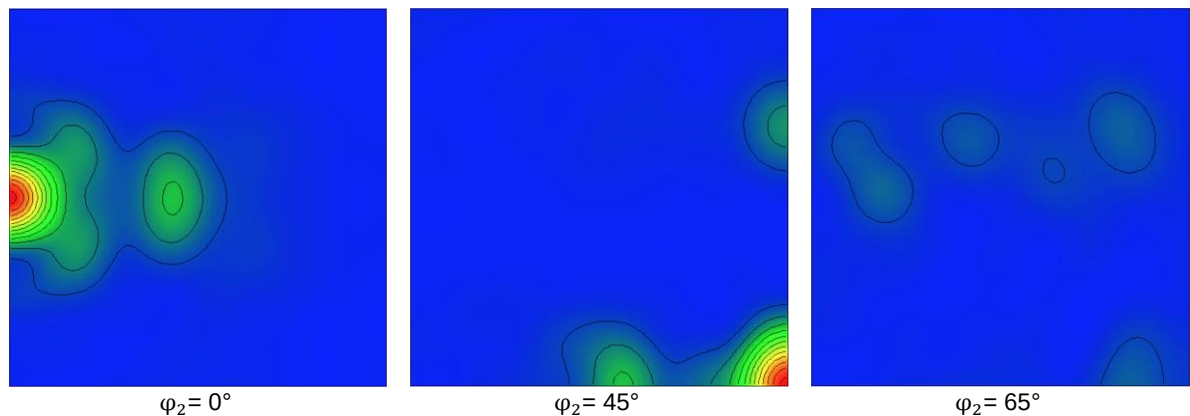


Figure 9.27. The texture of the reverted γ in the 1.8 mm one-step IA FeCMnAl steel sample in $\varphi_2 = 0^\circ$, 45° and 65° ODF sections. Contour levels = $2x$.

Figure 9.27 shows the texture of the reverted γ in the 1.8 mm one-step IA FeCMnAl sample in $\varphi_2 = 0^\circ$, 45° and 65° ODF sections. Figure 9.28 shows the fibers of the γ in 1.8 mm sample before and after the IA at 650°C for 10 min. In the one-step IA sample, there were α and τ -fibers with the maximum intensity on the G component at $f(g) = 24.8$ times random. The formation of the D and G-T components in γ phase indicated that the twinning of the γ grains with G texture occurred during the reverse transformation of α' into γ . The G component in this sample indicated that γ grains partially or fully recrystallized during the IA [25, 399, 400]. Additionally, the texture exhibited a strong B component at $f(g) = 5.9$ times random.

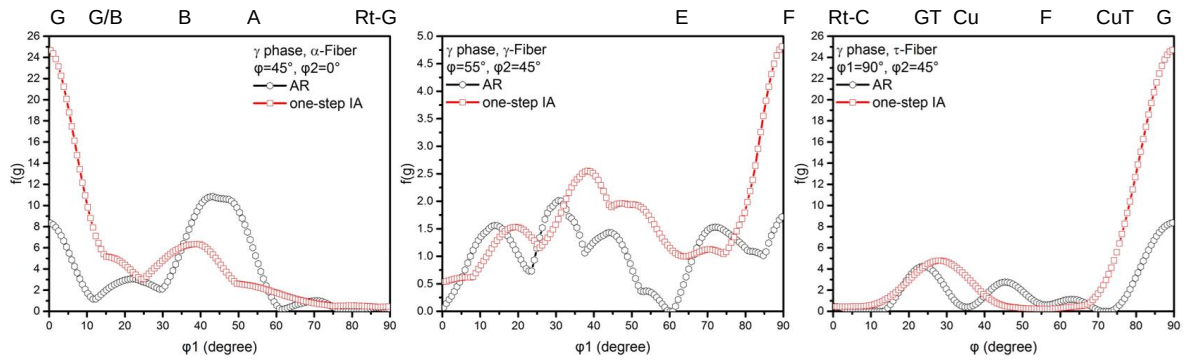


Figure 9.28. The fibers of the γ phase in the 1.8 mm AR and one-step IA FeCMnAl steel samples.

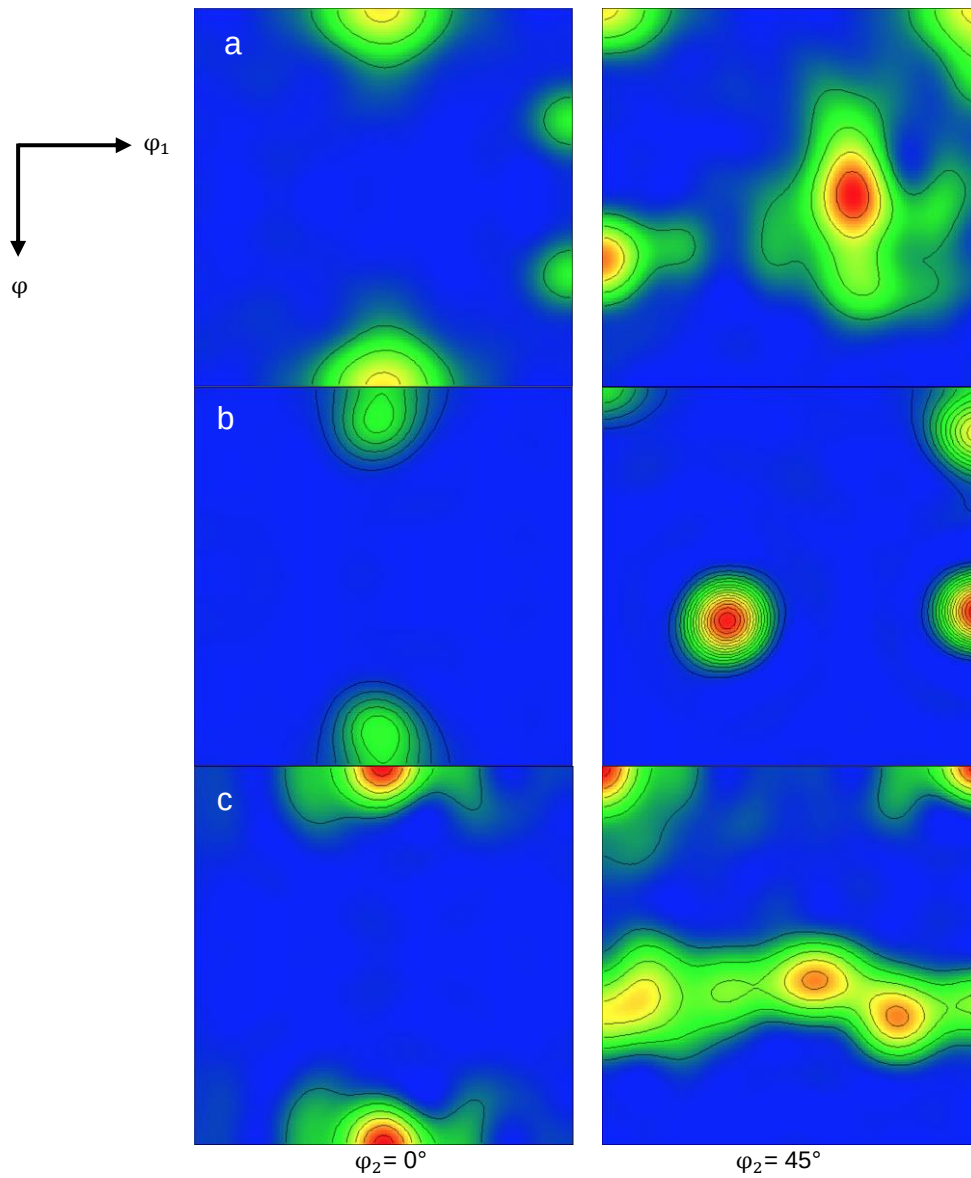


Figure 9.29. The texture of the α/α' phase after (a) 20%, (b) 30%, and (c) 50% TD cold rolling of the 1.one-step IA sample in $\phi_2 = 0^\circ$ and 45° ODF sections. Contour levels = $2x$.

9.2.5.1 Texture of the FeCMnAl Steel after the Cold Rolling

Figure 9.29 and 9.30 show the texture and fiber of the α/α' phase after TD cold rolling of the one-step IA sample in $\varphi_2 = 0$ and 45° ODF sections, respectively. After 20% cold rolling there was a γ -fiber and the maximum intensity was on the $(011)[\bar{1}\bar{1}1]$ component at $f(g) = 9.3$ times random and there were weak $(111)[\bar{1}\bar{1}0]$ and $(001)[\bar{1}\bar{1}0]$ components at $f(g) = 7.7$ and 9.4 times random, respectively.

After 30% cold rolling, the maximum intensity was on the F components ($\{111\}\langle 112 \rangle$) at $f(g) = 31.3$ times random and a weak $(001)[\bar{1}\bar{1}0]$ component was at $f(g) = 9.86$ times random. After 50% cold rolling, the maximum intensity was on the $(011)[\bar{1}\bar{1}1]$ component at $f(g) = 12.7$ times random and weak E and F components were at $f(g) = 7.68$ and 9.34 times random, respectively. The texture had a γ -fiber.

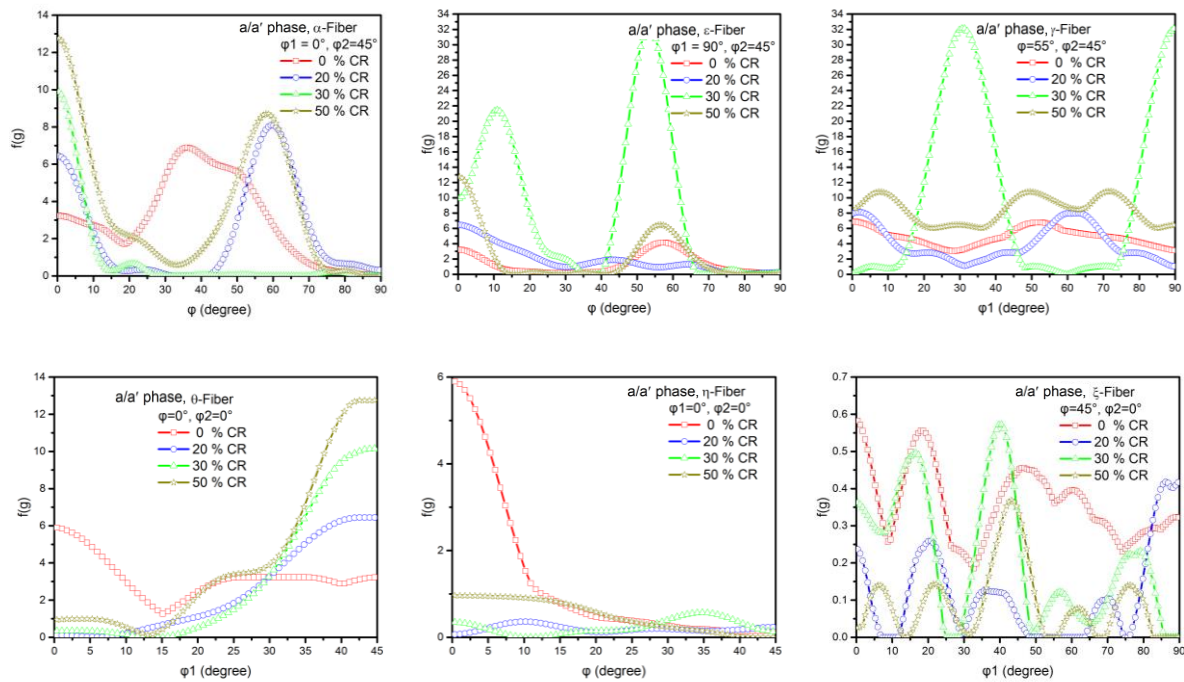


Figure 9.30. The fibers of the α/α' after (a) 20%, (b) 30%, and (c) 50% TD cold rolling of the 1.one-step IA sample. The 0% CR sample is the one-step IA sample.

Figure 9.31 and 9.32 show the texture and the fibers of the γ phase after TD cold rolling of the one-step IA sample, respectively. After 20% cold rolling, the maximum intensity was on the A component at $f(g) = 52.3$ times in the γ phase. After 30% cold rolling, the maximum intensity was on the component with Euler angles $(023)[\bar{0}\bar{3}2](90^\circ, 35^\circ, 0^\circ)$ at $f(g) = 26.7$ times random and weaker A and G/B components were at $f(g) = 17.4$ and 2.9 times random, respectively in γ phase. The texture had an α -fiber.

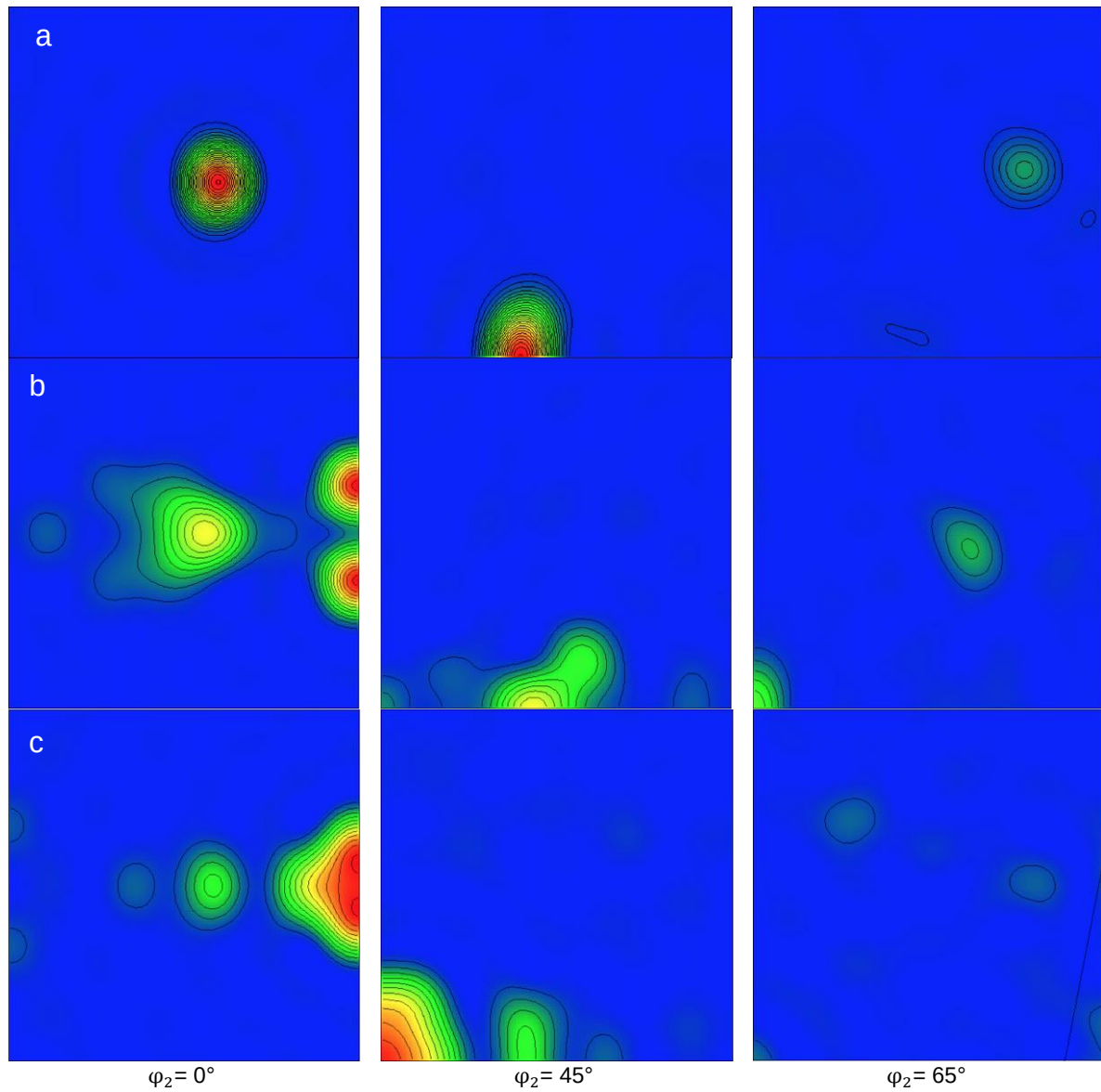


Figure 9.31. The texture of the γ phase after (a) 20%, (b) 30%, and (c) 50% TD cold rolling of the one-step IA sample in $\varphi_2 = 0^\circ$, 45° and 65° ODF sections. Contour levels = $2x$.

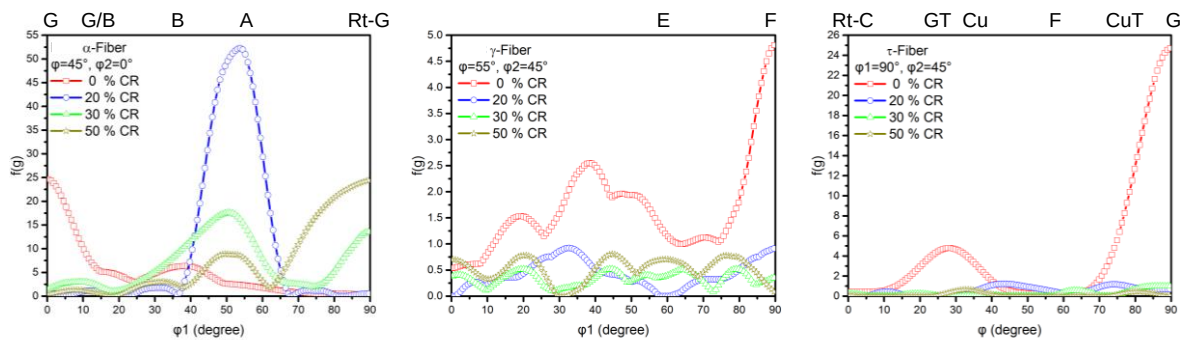


Figure 9.32. The fibers of the γ phase after (a) 20%, (b) 30%, and (c) 50% TD cold rolling of the 1.one-step IA sample. The 0% CR sample is 1.8 mm one-step IA sample.

After 50% cold rolling and in γ phase. the maximum intensity was on the Rt-G component at $f(g) = 24.4$ times random and weaker A and B components were at $f(g) = 8.7$ and 3 times random, respectively. The texture had an α -fiber.

9.2.5.1 Texture of the FeCMnAl Steel after Second Intercritical Annealing

On Figures 9.33 to 9.36, the ODF sections ($\varphi_2 = 0^\circ$ and 45°) and fibers of the α/α' phase after the IA at 620 °C and 650 °C are depicted. After 5 min IA at 620 °C the texture showed a γ -fiber with pronounced intensity at $\{111\}\langle 112 \rangle$ component and weaker $\{001\}\langle 110 \rangle$ and $\{111\}\langle 110 \rangle$ (Figures 9.33(a) and 9.34). Increasing the annealing time to 10 min resulted in strengthening the $\{001\}\langle 110 \rangle$ and $\{111\}\langle 110 \rangle$ components and weakening the $\{111\}\langle 112 \rangle$ component (Figures 9.33b and 9.34 (ϵ -fiber)). After 10 min IA at 620 °C, there were the new weak $\{552\}\langle 115 \rangle$ and $(223)[\bar{2}\bar{2}3]$ ($90^\circ, 45^\circ, 45^\circ$) components (Figure 9.34 (ϵ -fiber)).

Recrystallization mechanisms based on oriented nucleation and selective or oriented growth are both supported by the current results. $\{001\}\langle 110 \rangle$ and $\{111\}\langle 110 \rangle$ components were texture components of the cold rolled martensitic sheets and the nucleation in these grains retained the orientation of the former texture throughout the annealing process. As a result, it was concluded that the texture of these components was controlled by the oriented nucleation mechanism [397, 398].

The intensity of the $\{001\}\langle 110 \rangle$ component did not change after the IA and it was almost as same as the cold rolled sample. The intensity of $\{001\}\langle 100 \rangle$ component decreased after first 5 min IA (from $f(g) = 0.96$ to $f(g) = 0.23$) and then increased to $f(g) = 2.25$ after 10 min IA (Figure 9.34).

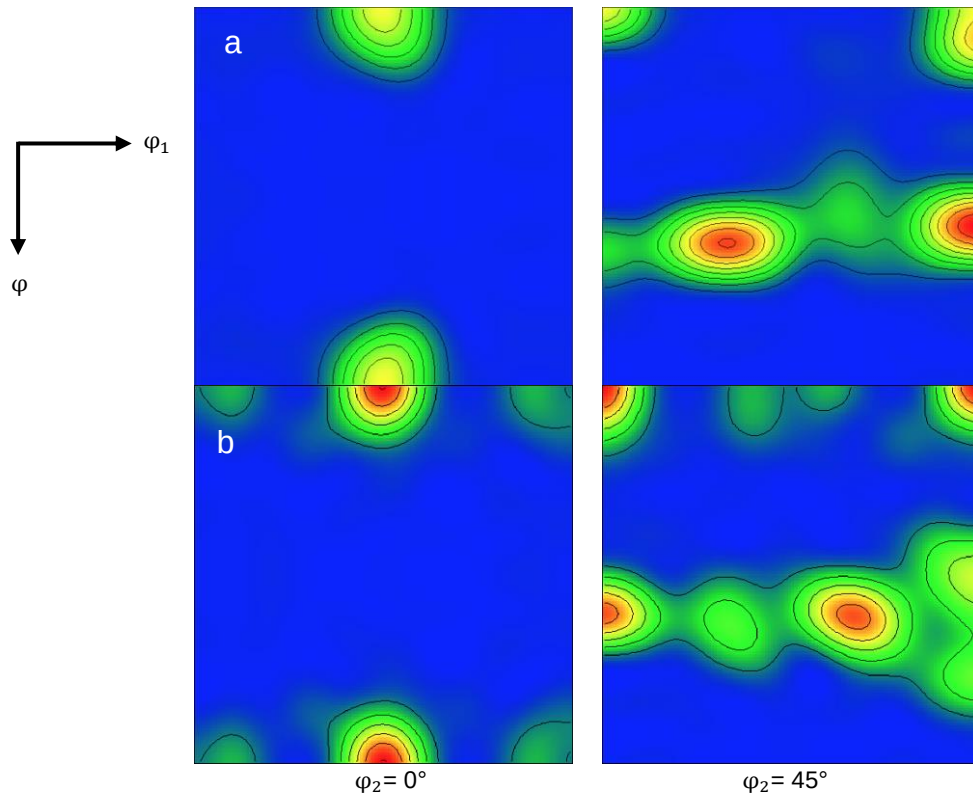


Figure 9.33. The α/α' phase's texture of 0.9 mm sample after the IA at 620 °C for (a) 5 and (b) 10 min in $\varphi_2 = 0^\circ$ and 45° ODF sections. Contour levels = $2x$.

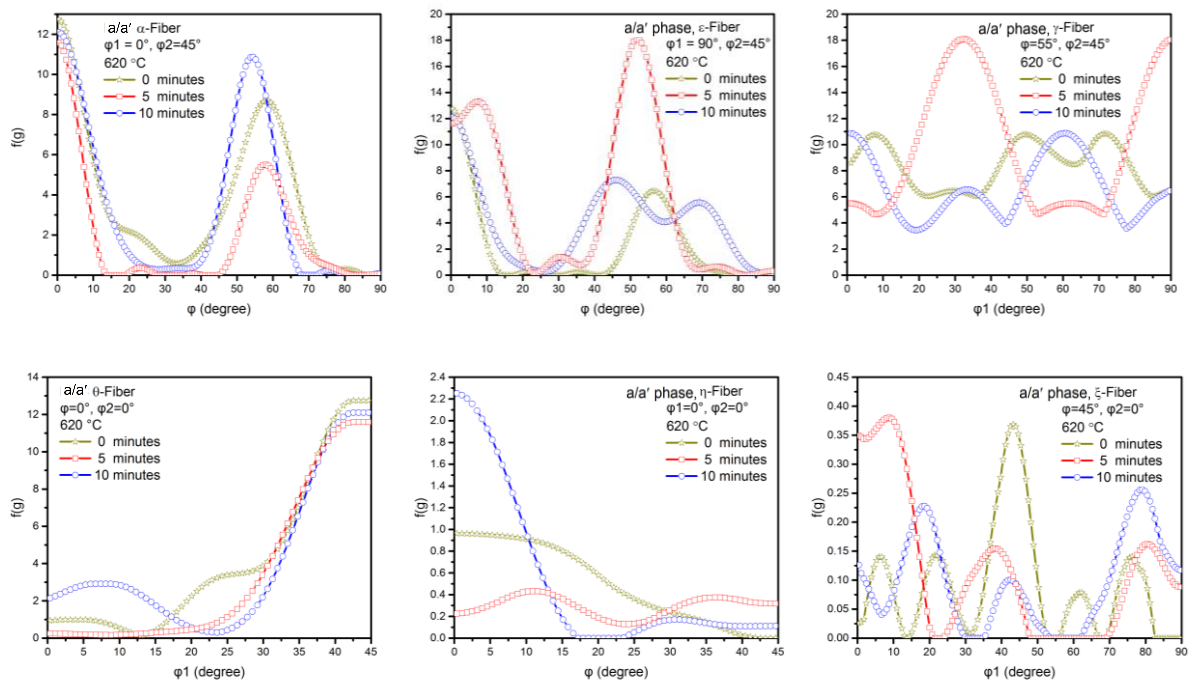


Figure 9.34. The α/α' phase's fibers of the 0.9 mm sample after the IA at 620 °C for 5 and 10 min. The 0 min sample is the 50% cold rolled sample.

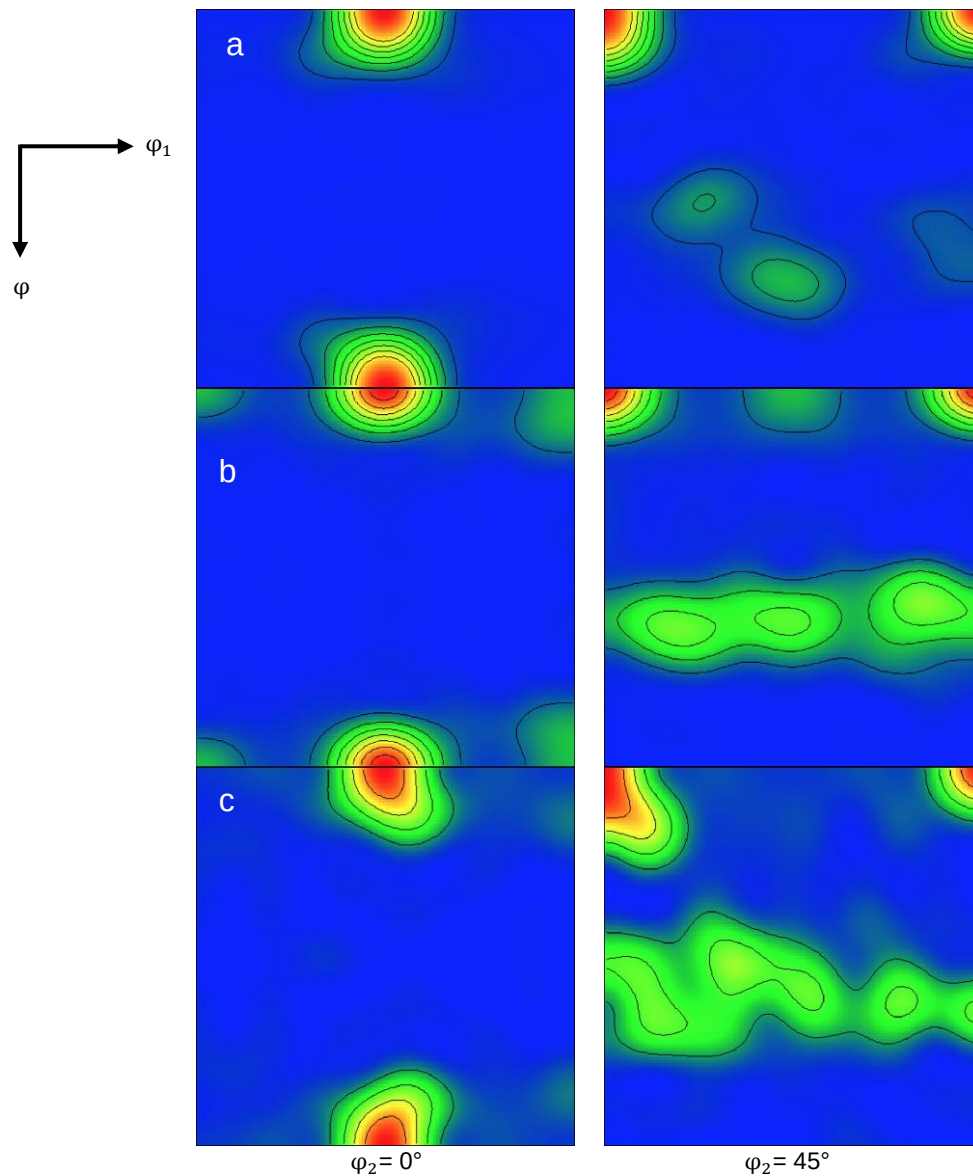


Figure 9.35. The α/α' phase's texture in the 0.9 mm sample after the IA at 650 °C for (a) 3, (b) 5 and (c) 10 min in $\phi_2 = 0^\circ$, 45° and 65° ODF sections. Contour levels = 2x.

However, after the material was subjected to the 50% TD cold rolling and the IA at 620 °C and 650 °C, the intensity of the rotated cube component became stronger than the cube component. Specifically, the intensity of the rotated cube component increased from 12.8 times random in the 50% cold rolled sample to 20.7 times random in the sample was intercritically annealed at 650 °C for 5 min. This increase in intensity could be due to several factors, such as the transformation of other α/α' phase texture components to γ phase during annealing, the recrystallization of some of the other α/α' phase texture components in the rotated cube component, and the growth of the grains with a rotated cube component.

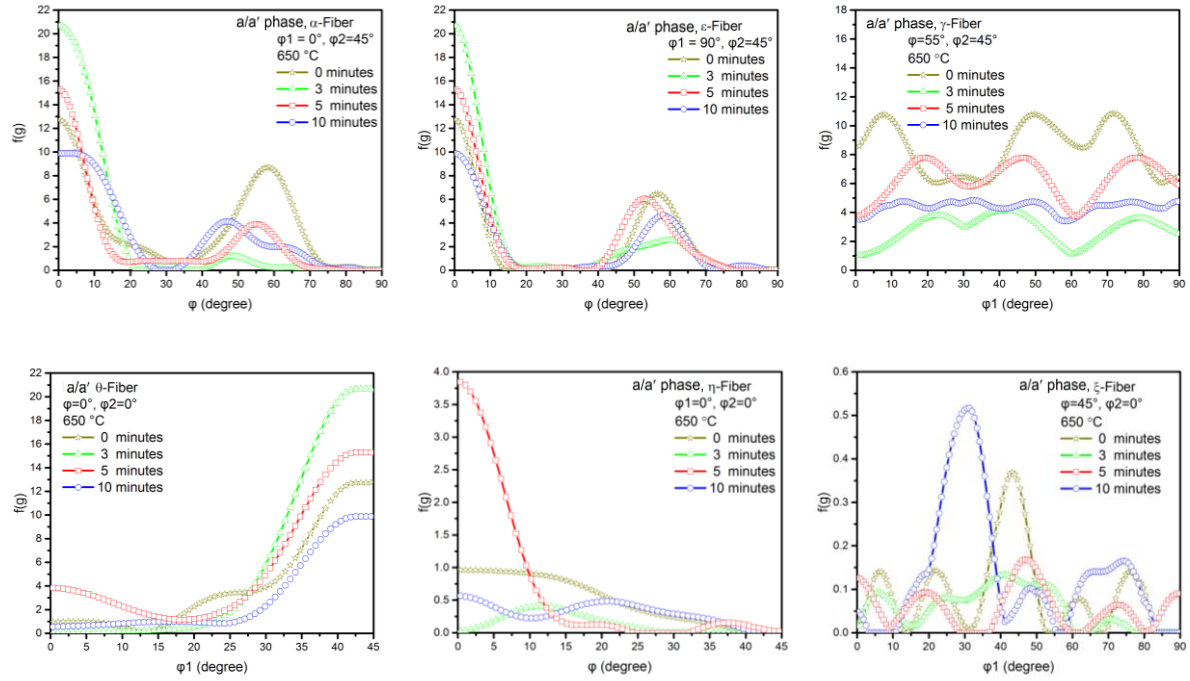


Figure 9.36. The α/α' phase's fibers in 0.9 mm sample after the IA at 650 °C for 3, 5 and 10 min. The 0 min sample is the 50% cold rolled sample.

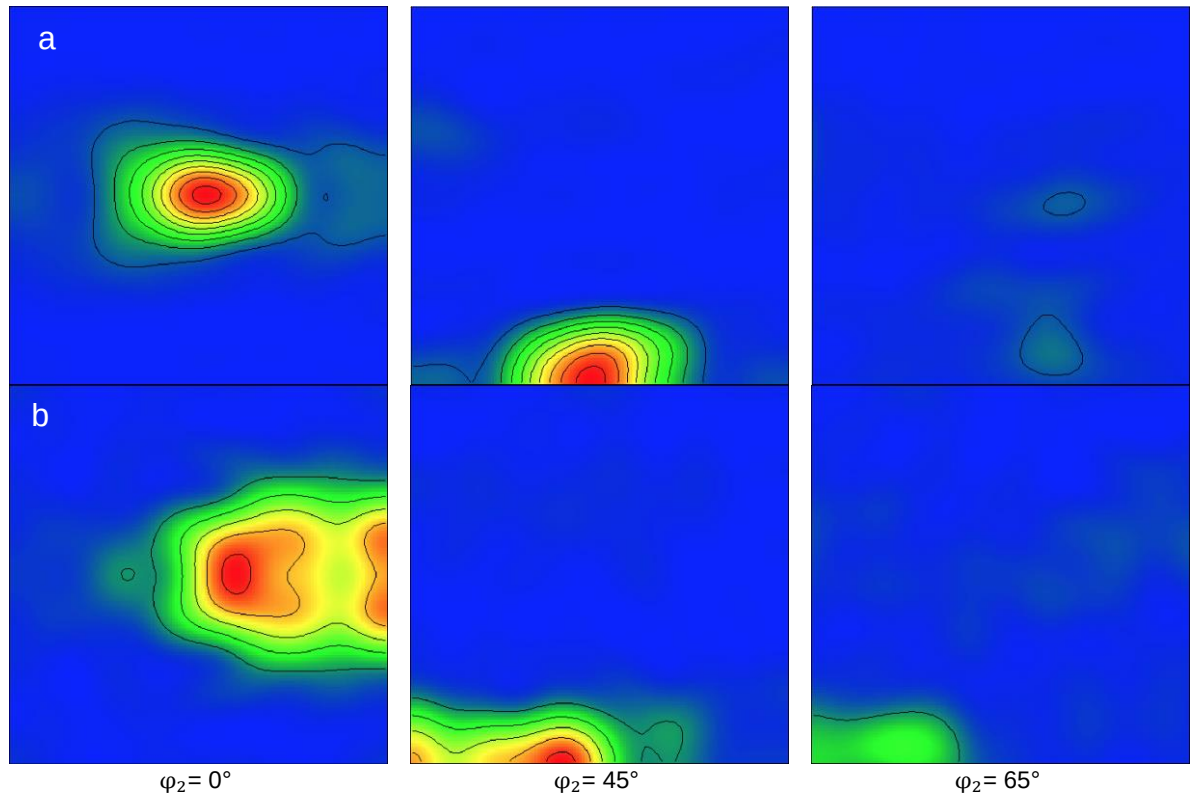


Figure 9.37. The γ phase's texture of 0.9 mm sample after the IA at 620 °C for (a) 5 and (b) 10 min in $\phi_2 = 0^\circ$, 45° and 65° ODF sections. Contour levels = $2x$.

Further analysis revealed that the rotated cube component remained the predominant texture component, albeit weaker, even after increasing the annealing time to 5 and 10 min. Additionally, the formation of the γ -fiber was observed in the material after this time.

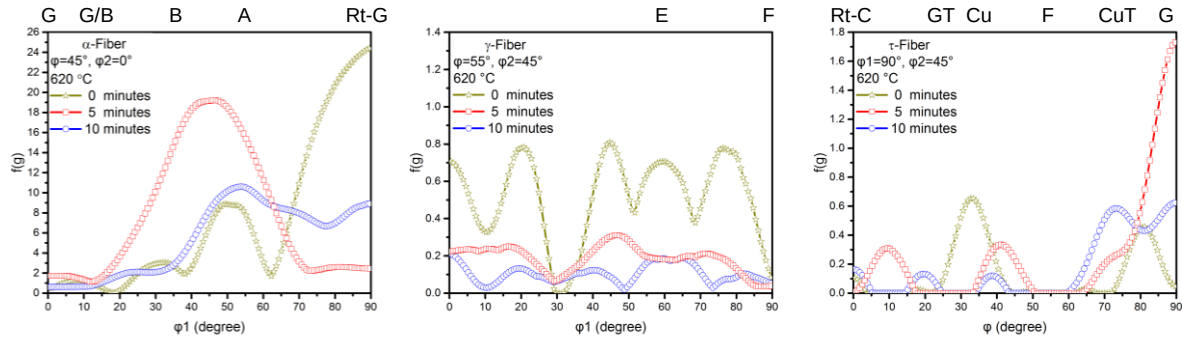


Figure 9.38. The γ phase's fibers of 0.9 mm sample after the IA at 620 °C for 5 and 10 min. The 0 min sample is the 50% cold rolled sample.

Figures 9.37 and 9.38 exhibit the texture and fiber of the γ phase in the 0.9 mm sample after the IA at 620 °C in $\varphi_2 = 0^\circ$, 45° , and 65° ODF sections. All of the samples showed an α -fiber. After the IA at 620 °C for 5 min, there was a high intensity (011)[$32\bar{2}$] component between B and A ($f(g) = 19.2$) in the samples, which was close to the A component that had an intensity of 7.8 and 10.6 in the cold rolled sample and the sample after 10 min IA.

Moreover, the intensity of the Rt-G component is observed to decrease from 24.4 to 2.5 times random after 5 min and increase to 8.9 times random after 10 min of the IA. In addition, the τ -fiber (Figure 9.37) reveals that the intensity of the weak G component increases after 5 min and decreases after 10 min of the annealing. Furthermore, it is evident that the intensity of the weak γ -fiber decreases over time.

Figures 9.39 and 9.40 depict the texture and fiber structures of the γ phase in the 0.9 mm sample after the IA at 650 °C. In all conditions, a strong α -fiber was observed. Additionally, it was evident that after 3 min of the IA, a strong B component with intensity of 17.6 times random was present. It has been demonstrated that the B component in the γ phase came from the reverse transformation of the $\{011\}\langle 110 \rangle$ and $\{332\}\langle 113 \rangle$ components of the α/α' phase [37, 38, 401]. It was observed that the B-type texture resulted from the reverse transformation of the α' into the γ phase. The reason for this was related to the quick recovery of the γ phase in low SFE materials, resulting in a low driving force for recrystallization process [399].

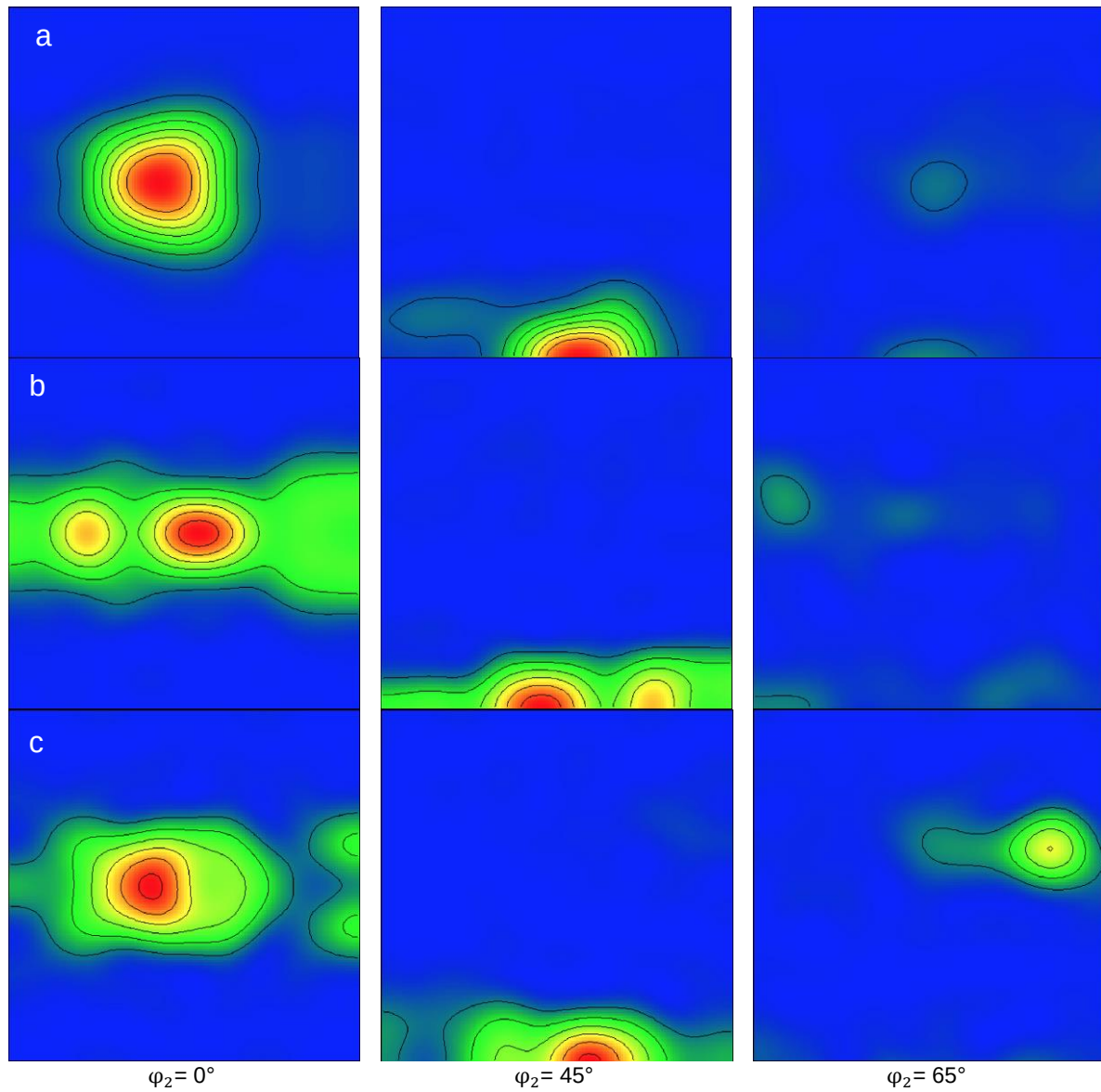


Figure 9.39. The γ phase's texture of 0.9 mm sample after the IA at 650 °C for (a) 3, (b) 5 and (c) 10 min in $\varphi_2 = 0^\circ$, 45° and 65° ODF sections. Contour levels = $2x$.

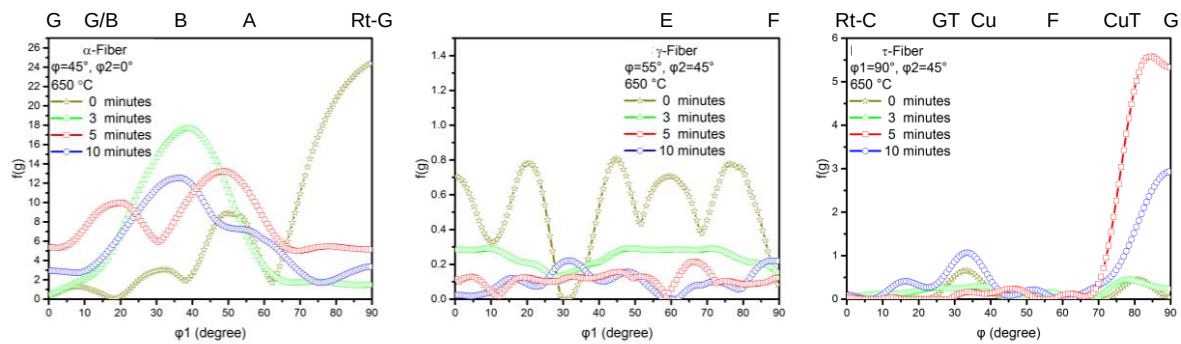


Figure 9.40. The γ phase's fibers of 0.9 mm sample after the IA at 650 °C for 3, 5 and 10 min. The 0 min sample is the 50% cold rolled sample.

Increasing the annealing time to 5 min resulted in the appearance of a strong A component and a weak G/B component, where the G/B and A components had a twin ($\Sigma 9$ - $39^\circ < 110 >$) relationship. On further increasing the annealing time to 10 min, a strong B component and a weak A component were observed. The τ -fiber (Figure 9.40) indicated that the intensity of the G component increased slightly from 0.05 (cold rolled) to 0.23 after 3 min of the annealing. Subsequently, the intensity increased to 5.3 and then decreased to 2.9 after 5 and 10 min of the annealing, respectively.

9.2.6 Summary of the Scanning Electron Microscopy Maps Analyses

The analyses of SEM-TKD maps of the FeCMnAl steel after each process of the TSTMP summarised in Table 9.1.

Table 9.1. Summary of the SEM-TKD maps of the FeCMnAl steel after the TSTMP.

Sample Condition		Phase Volume Fraction %		Average Grain Size nm		Nano Size Grains %		KAM Average Misorientation Degree	
		γ	α/α'	γ	α/α'	γ	α/α'	γ	α/α'
1.8 mm	AR	0.2	99.8	72	173	84.9	40.4	2.06	1.69
	one-step IA	59.8	40.2	218	194	32.9	34.7	0.47	0.45
TD Cold Rolling	CR(TD)20%	8.6	80.3	110	188	63.5	37.6	0.90	0.80
	CR(TD)30%	4.1	87.7	81	157	72.4	35.6	1.20	0.95
	CR(TD)50%	0.4	92.7	51	125	87.4	53.7	1.51	1.14
0.9 mm TSTMP	620 °C, 5 min	31.6	55.5	140	151	39	42.7	1.41	0.87
	620 °C, 10 min	49.9	50.1	145	159	40.5	34.1	1.00	0.88
	650 °C, 3 min	52.6	47.4	160	132	42.5	49	1.11	0.83
	650 °C, 5 min	55.6	44.4	164	145	37.1	42.8	1.03	0.84
	650 °C, 10 min	58.4	41.6	172	161	32.1	33.3	1.08	0.83

Sample Condition		Aspect Ratio (No preferred Direction)		Average Aspect Ratio (RD/TD)		LAGB %		Phase Boundaries <10° Deviation from KS OR %
		γ	α/α'	γ	α/α'	γ	α/α'	
1.8 mm	AR	1.97	2.31		1.44	97.1	77.4	69.5
	One-step IA	2.09	2.28	1.03	1.06	77.1	71.6	80.4
TD Cold rolling	CR(TD)20%	1.98	1.92			99.04	93.5	79.8
	CR(TD)30%	1.97	1.96			98.1	94.6	78.1
	CR(TD)50%	1.79	1.81		1.10	98.5	95.1	72.3
0.9 mm TSTMP	620 °C, 5 min	1.89	2.04	0.98	0.96	96.0	93.9	75.3
	620 °C, 10 min	1.91	2.04	0.94	0.91	95.2	93.2	74.0
	650 °C, 3 min	2.09	2.30	0.95	0.92	94.2	87.0	57.4
	650 °C, 5 min	1.91	2.10	1.03	1.04	80.2	84.4	61.8
	650 °C, 10 min	2.14	2.26	0.94	0.93	72.0	65.2	67.0

9.3 X-Ray Diffraction

XRD patterns obtained from various annealing and rolling conditions of the FeCMnAl alloy steel are shown in Figure 9.41. In cold rolled samples, the α peaks were more intense. The γ peaks became more intense with increased annealing temperature and time due to the development of the γ phase.

$(110)_\alpha$, $(200)_\alpha$, $(211)_\alpha$, $(310)_\alpha$, $(222)_\alpha$, $(321)_\alpha$, $(400)_\alpha$, $(330)_\alpha$, $(332)_\alpha$ and $(422)_\alpha$ were the diffraction peaks of the α phase and $(111)_\gamma$, $(200)_\gamma$, $(220)_\gamma$, $(311)_\gamma$, $(331)_\gamma$, $(420)_\gamma$, $(422)_\gamma$, $(440)_\gamma$ and $(531)_\gamma$ were the diffraction peaks of the γ phase that they were detected.

As a result of the IA, the intensity of the γ peaks increased. Comparing the 1.8 mm AR sample to one-step IA (Figure 9.41 (b)), shows that the α phase partially transformed into the γ . Cold rolled conditions also show a small amount of the γ phase with the $(200)_\gamma$ diffraction peak that was not observed by SEM-TKD.

In the 1.8 mm AR and the one-step IA samples, the intensity of the $(211)_\alpha$ peak was greater than that of the $(200)_\alpha$ peak, whereas, in the second intercritically annealed samples, it was the opposite. The relative intensity of these two peaks changed after the TD cold rolling (Figure 9.41 (c), (d) and (e)).

Almost all the samples after IA, except the 0.9 mm sample after the IA at 650 °C for 3 min, exhibited a greater intensity of the $(111)_\gamma$ peak than that of the $(110)_\alpha$ peak.

A comparison of the $(222)_\alpha$ and $(311)_\gamma$ peaks illustrated the importance of the annealing time and temperature, as well as the effect of the TD cold rolling. For the 0.9 mm sample after IA at 620 °C for 5 and 10 min and at 650 °C for 3 min, the intensity of the $(311)_\gamma$ peak was lower than that of the $(222)_\alpha$ peak. After 10 min of the IA at 650 °C for the 1.8 mm sample, the values were the same. However, the $(311)_\gamma$ peak in the 0.9 mm sample after annealing at 650 °C for 5 min had a higher intensity, and this ratio increased by increasing the annealing time to 10 min at 650 °C. There was a noticeable difference between the behaviour of the 0.9 mm and 1.8 mm samples following the same IA.

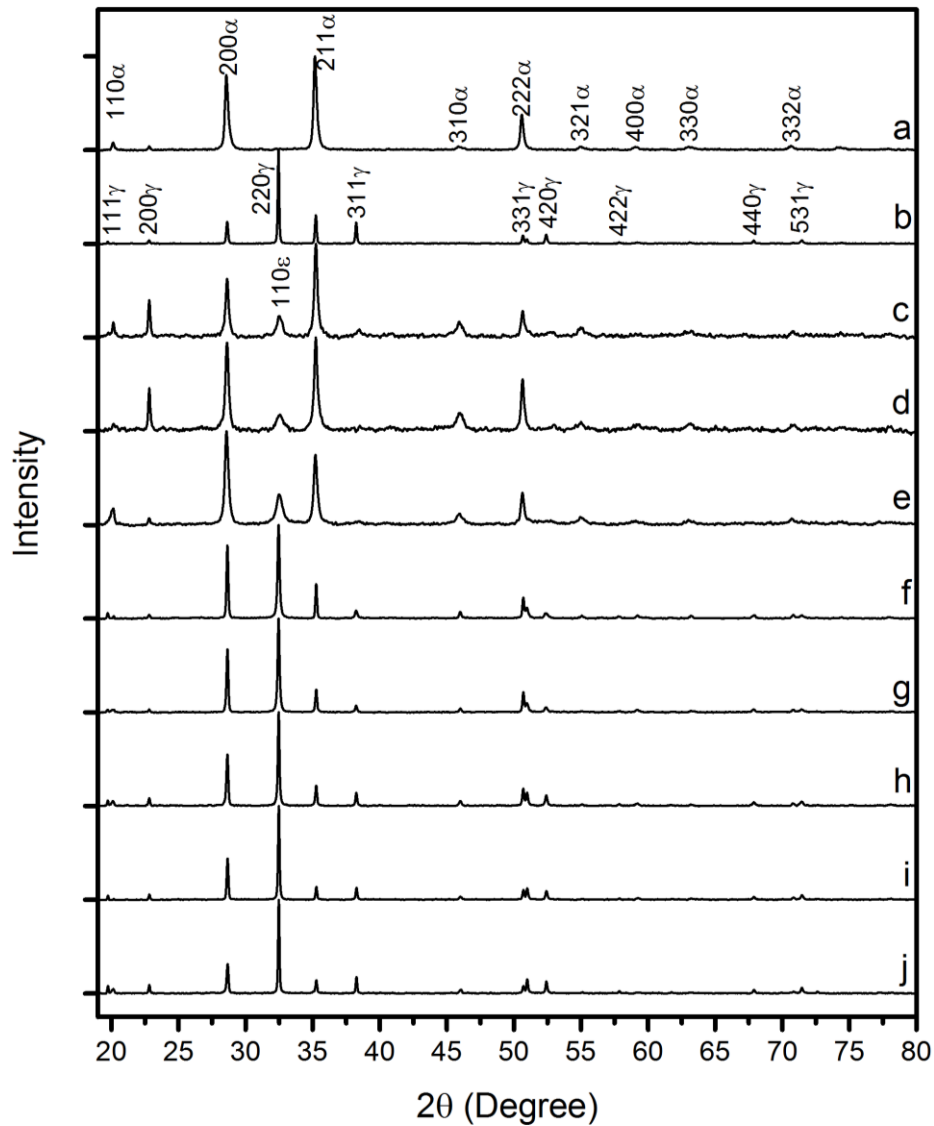


Figure 9.41. X-ray diffraction spectra of the FeCMnAl steel samples (a) 1.8 mm AR (b) 1.8 mm one-step IA, (c) 20% , (d) 30% and (e) 50% TD cold rolled samples. The 0.9 mm FeCMnAl sample after the IA at 620 °C for (f) 5 and (g) 10 min. The 0.9 mm FeCMnAl sample after the IA at 650 °C for (h) 3, (i) 5 and (j) 10 min.

Cold rolled samples showed the $(310)\alpha$ peak, whereas the 0.9 sample showed a stronger peak. This peak was not visible in the annealed samples. Alternatively, the $(400)\gamma$ peak tended to be significant only in annealed samples, with the exception of the 1.8 mm samples.

The intensity of the $(111)\gamma$, $(200)\gamma$ and $(400)\gamma$ peaks increased faster than $(220)\gamma$ and $(311)\gamma$ peaks, which showed preferential orientations of the γ growth.

The XRD results were used to calculate the γ phase fraction, which was found to be higher than that obtained by the SEM method. Figure 9.42 illustrates the difference in the γ

phase fraction between the XRD and SEM methods. Two possible reasons for this discrepancy were:

- 1- the metastable γ phase might have transformed into α' during the sample preparation using the PIPS equipment;
- 2- the SEM-TKD sample was a thin foil similar to the TEM sample, and the edge of the hole used for the analysis contained only a few grains in thickness. Thus, the metastable γ grains with low stability, without surrounding grains, might have easily transformed into α' .

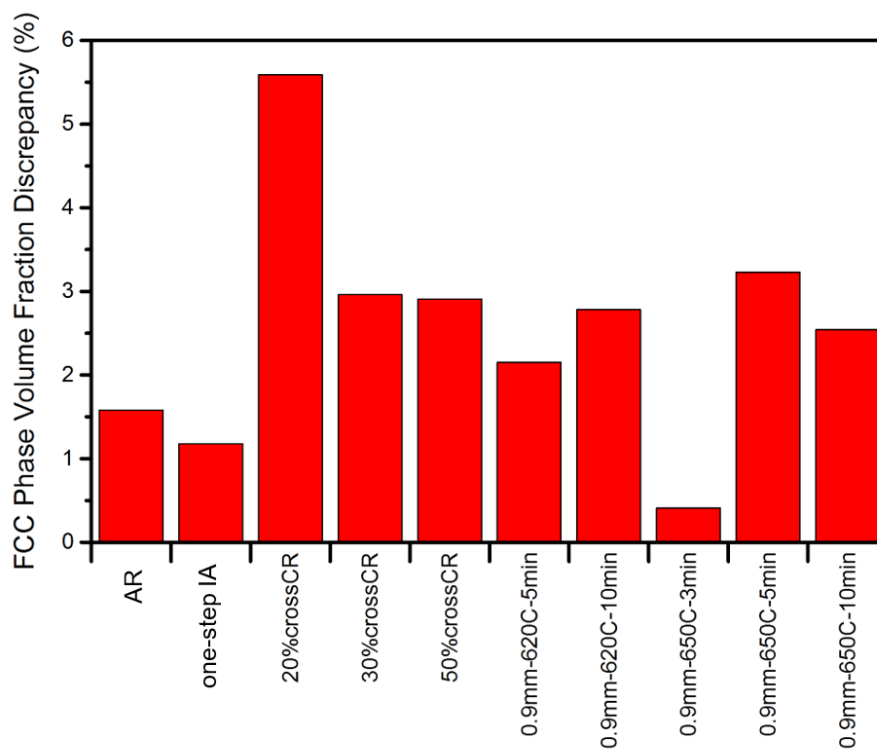


Figure 9.42. The discrepancy of the γ phase fraction between XRD and SEM methods for measuring the γ phase volume fraction.

9.4 Atom Probe Tomography Results

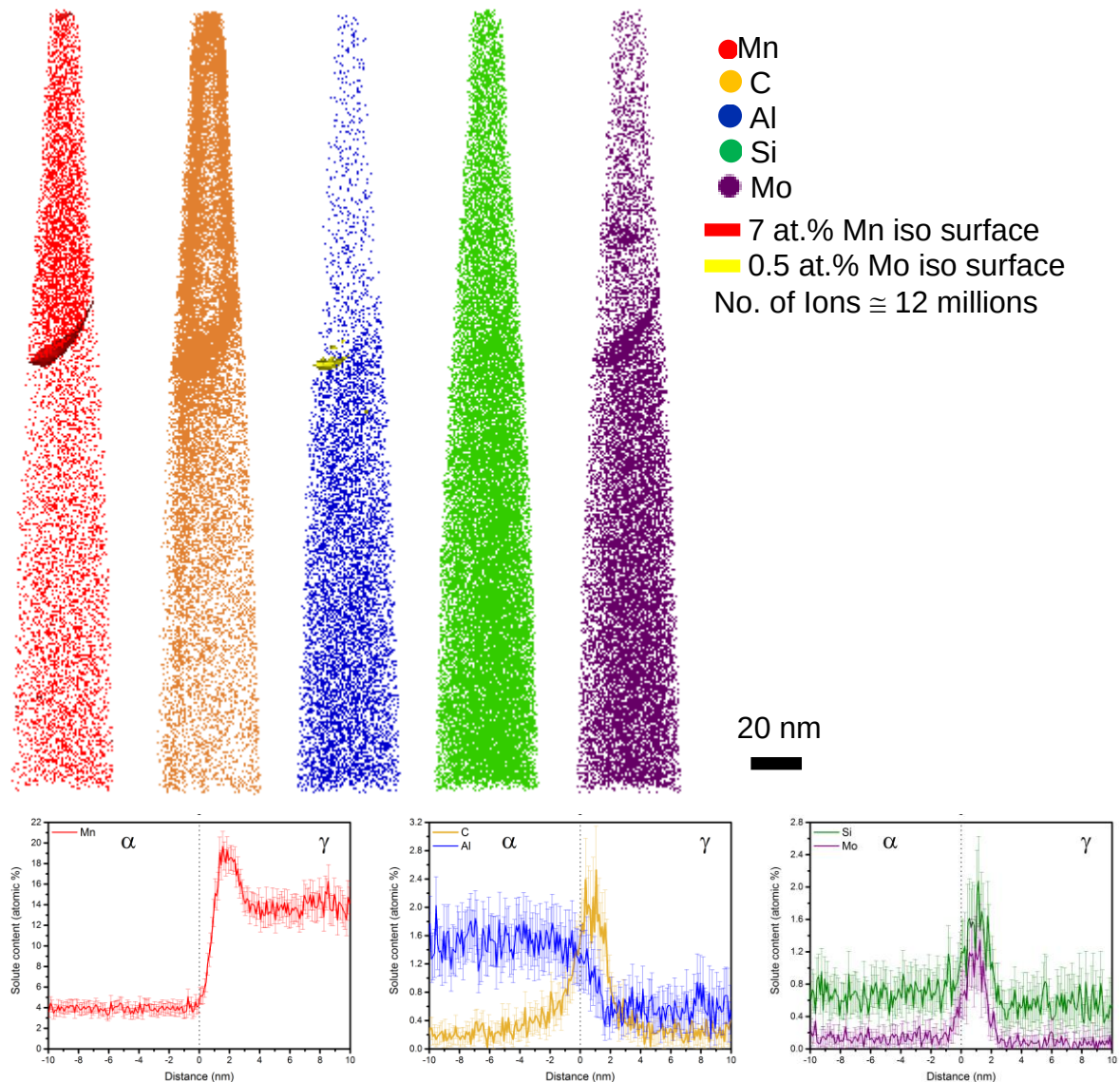


Figure 9.43. Atom probe tomogram of the 1.8 mm FeCMnAl specimen annealed at 650 °C for 5 min along with the compositional profile of the Mn, Al, C, Mo, and Si atoms across the γ - α phase boundary. 7 at. % Mn and 0.5 at. % Mo iso-concentrations are shown in the tomogram of the Mn and Al atoms, respectively.

In this study, the chemical compositions of the γ and α/α' phases and their boundaries were analysed using APT. The purpose of this study was not to evaluate the crystallography of the phases. As a result of the high density of the dislocations, as well as the small grain sizes of the phases (Nano- and Ultrafine-structure), and the high concentration of the alloying elements within the γ phase, it was difficult to obtain data with adequate poles [405]. A crystallographic reconstruction was not possible without poles in the following Figures 9.43 to 9.49. In these Figures, the atom probe maps are shown with alloying elements distributed

across the grain boundaries of 1.8 mm and 0.9 mm FeCMnAl alloy steel samples that were intercritically annealed at 620 °C and 650 °C for 5 and 10 min.

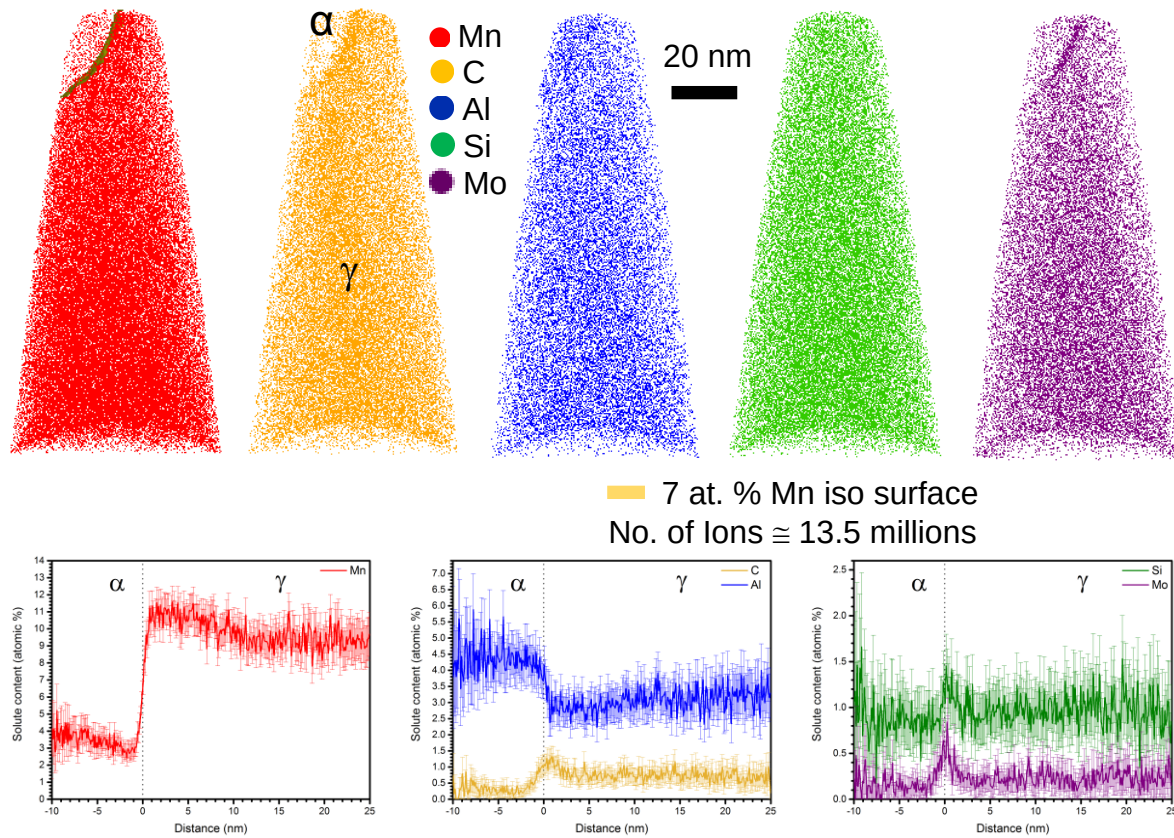


Figure 9.44. Atom probe tomogram of the 1.8 mm FeCMnAl sample annealed at 650 °C for 10 min along with the compositional profile of the Mn, Al, C, Mo, and Si atoms across the γ - α phase boundary. 7 at.% Mn iso-concentration is shown in the tomogram of the Mn atoms.

Using a proxigram approach with a layer width of 0.1 nm on the iso-concentration surfaces, the compositional profile of the C, Mn, Al, Si and Mo atoms was computed across γ and α phases. Proxigrams were used to determine the solute content of the each layer along the normal of the determined iso-concentration surface [405].

The atomic concentration profile of the Mn, Al, C, Mo, and Si atoms across the γ - α phase boundary in the 1.8 mm FeCMnAl sample annealed at 650 °C for 5 min is presented in Figure 9.43 using APT. As shown in the figure, the Mn atomic content gradually increased from approximately 4% in the α grain to about 20% at the γ grain boundary, then it decreased to 14% inside the γ grain. On the other hand, the atomic concentration of the Al decreased from around 1.5% in the α to about 0.5% in the γ . Furthermore, compositional spikes of the C, Si, and Mo atoms were observed at the grain boundary.

In Figure 9.44, it can be seen that the Mn content in α decreased from about 4% to about 3% in the area near the grain boundary. It then increased to about 12% at the γ grain boundary and remained at 9.4% inside the γ grain. The figure also shows the presence of the compositional spikes for C, Si, and Mo, which were similar to those observed in Figure 9.43.

APT maps of the cold-rolled 0.9 mm sample are presented in Figure 9.45, which can be used to represent the 1.8 mm sample after annealing at 650 °C for 10 min because cold-rolling did not change the composition of the grains. A ROI was selected to examine the grain composition, and it was found that the ROI contained one α and three martensite grains.

In Figure 9.45, it can be observed that there was a compositional spike of the Al at the interface of the γ - α . The distribution of the Mo atoms shows a higher concentration of the Mo atoms in the grain boundaries. The iso-surface concentration of 2.5 at. % of the C atoms revealed the presence of two carbides, whose proxigrams were displayed in Figure 9.45. Carbide 2 exhibited a lower Mo content at its centre when compared to its outer side. These carbides were located at the grain boundaries of the α - α' . Comparing Figures 9.44 and 9.45 revealed that the γ grains after annealing under the same conditions exhibited differences in their alloying element content.

In the APT maps of the 0.9 mm sample annealed at 620 °C for 5 min, as presented in Figure 9.46, four distinct ROIs were selected to illustrate the distribution of the alloying elements between the γ and α phase boundaries. Notably, each phase boundary exhibited a distinct pattern of the element distribution. For instance, in ROI 2 and ROI 3, Mn atoms displayed a compositional spike in the area adjacent to the γ . In contrast, no slope or compositional spike was observed for the Mn atomic percentage in ROI 1.

In ROI 2, as demonstrated in Figure 9.46, there was a compositional spike for the Al atoms near the α , while no other elements in the ROIs displayed the similar spikes.

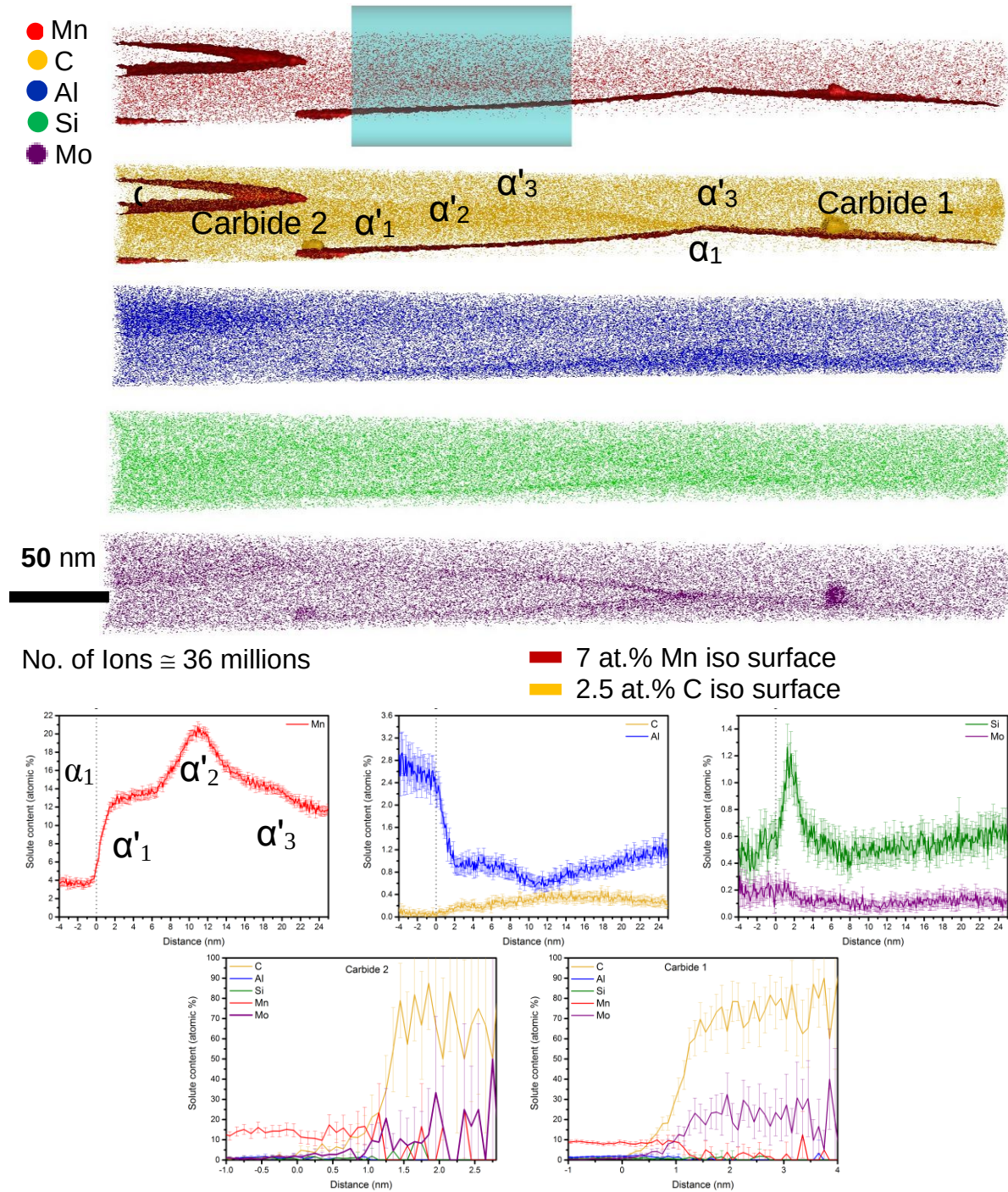


Figure 9.45. Atom probe tomogram of the cold rolled 0.9 mm FeMnAl sample along with the compositional profile of the Mn, Al, C, Mo, and Si atoms across the α - α' phase boundary in the ROI shown in the tomogram of the Mn atoms and the carbides shown in the tomogram of the C atoms. 7 at.% Mn and 2.5 at. % C iso-concentrations are shown in the tomogram of the Mn and C atoms, respectively.

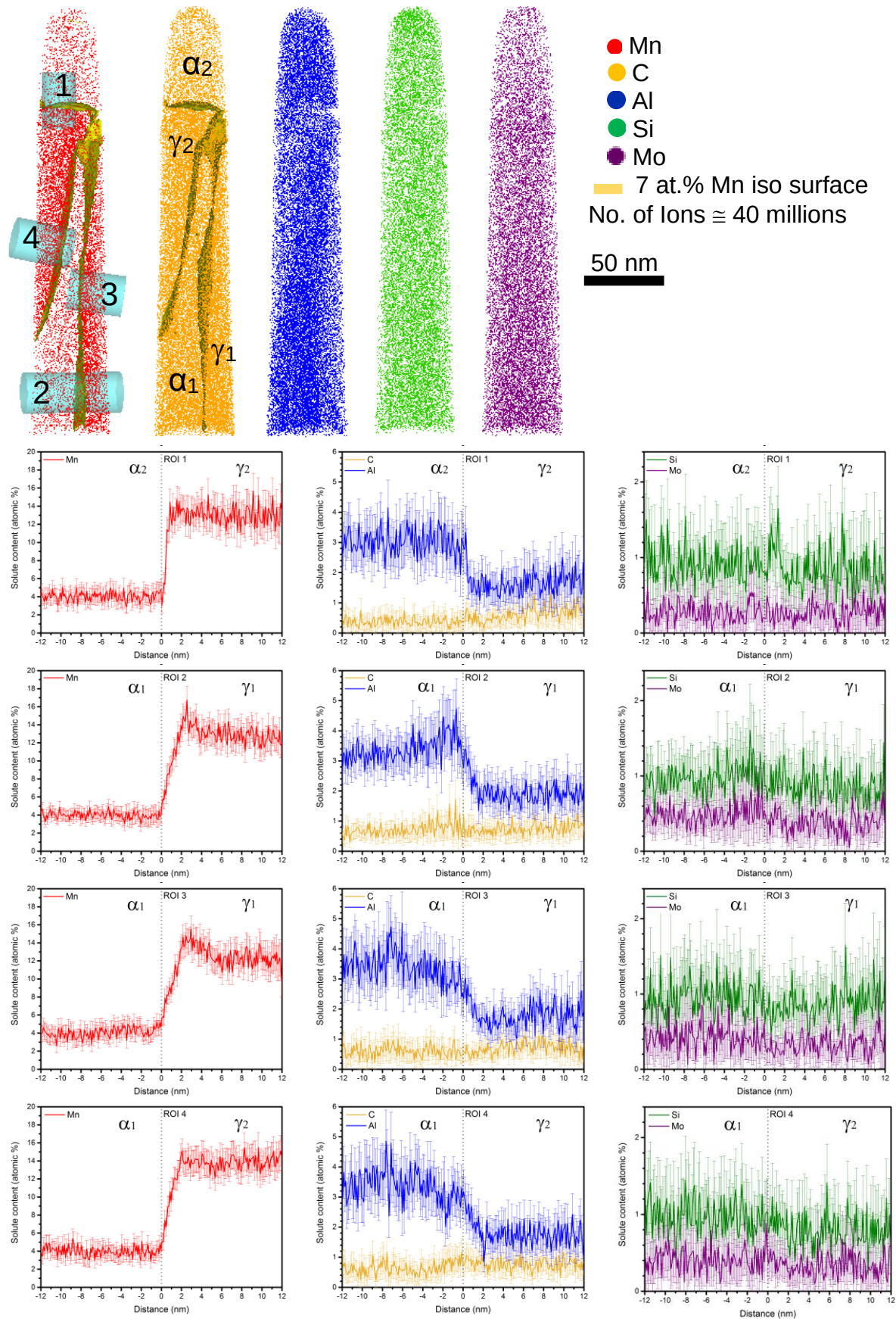


Figure 9.46. Atom probe tomogram of the 0.9 mm FeCMnAl sample annealed at 620 °C for 5 min along with the compositional profile of the Mn, Al, C, Mo, and Si atoms across the γ - α phase boundaries. 7 at. % Mn iso-concentration is shown in the tomogram of the Mn atoms.

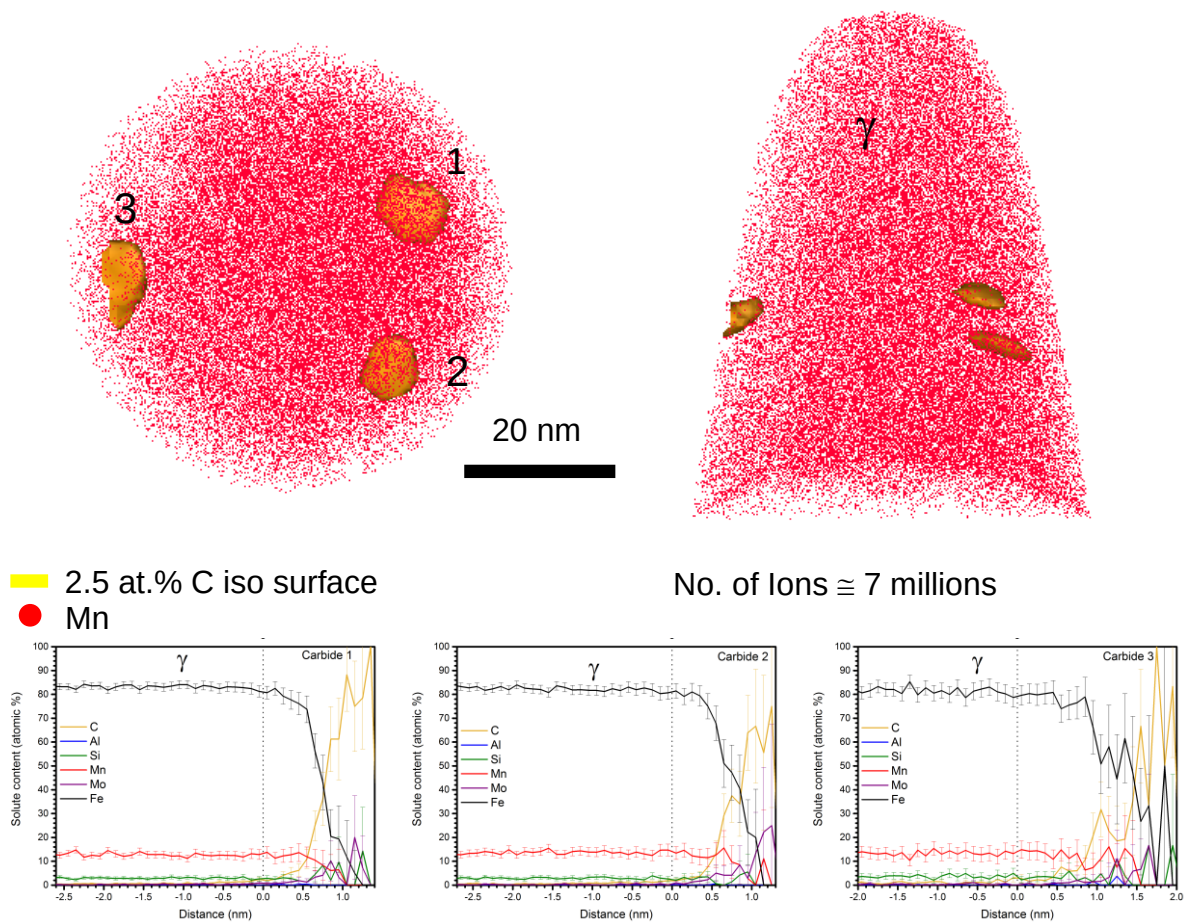


Figure 9.47. Atom probe tomogram of the 0.9 mm FeCMnAl sample annealed at 620 °C for 5 min along with the compositional profile of the Mn, Al, C, Mo, and Si atoms across the austenite-carbide phase boundaries. 2.5 at.% C iso-concentration is shown in the tomogram of the Mn atoms.

Figure 9.47 depicts three carbides present in the γ grain of the 0.9 mm sample annealed at 620 °C for 5 min. These carbides did not have enough atoms to calculate their compositions

In Figure 9.48, the APT maps of the 0.9 mm sample annealed at 620 °C for 10 min are presented, showcasing the distribution of the atoms between the α and γ grains through the selection of three ROIs. Notably, no compositional spikes were observed in the graphs. Moreover, the figure highlighted that the γ grain in ROI 3 exhibited a higher Mn level when compared to the γ grain in ROI 1 and ROI 2.

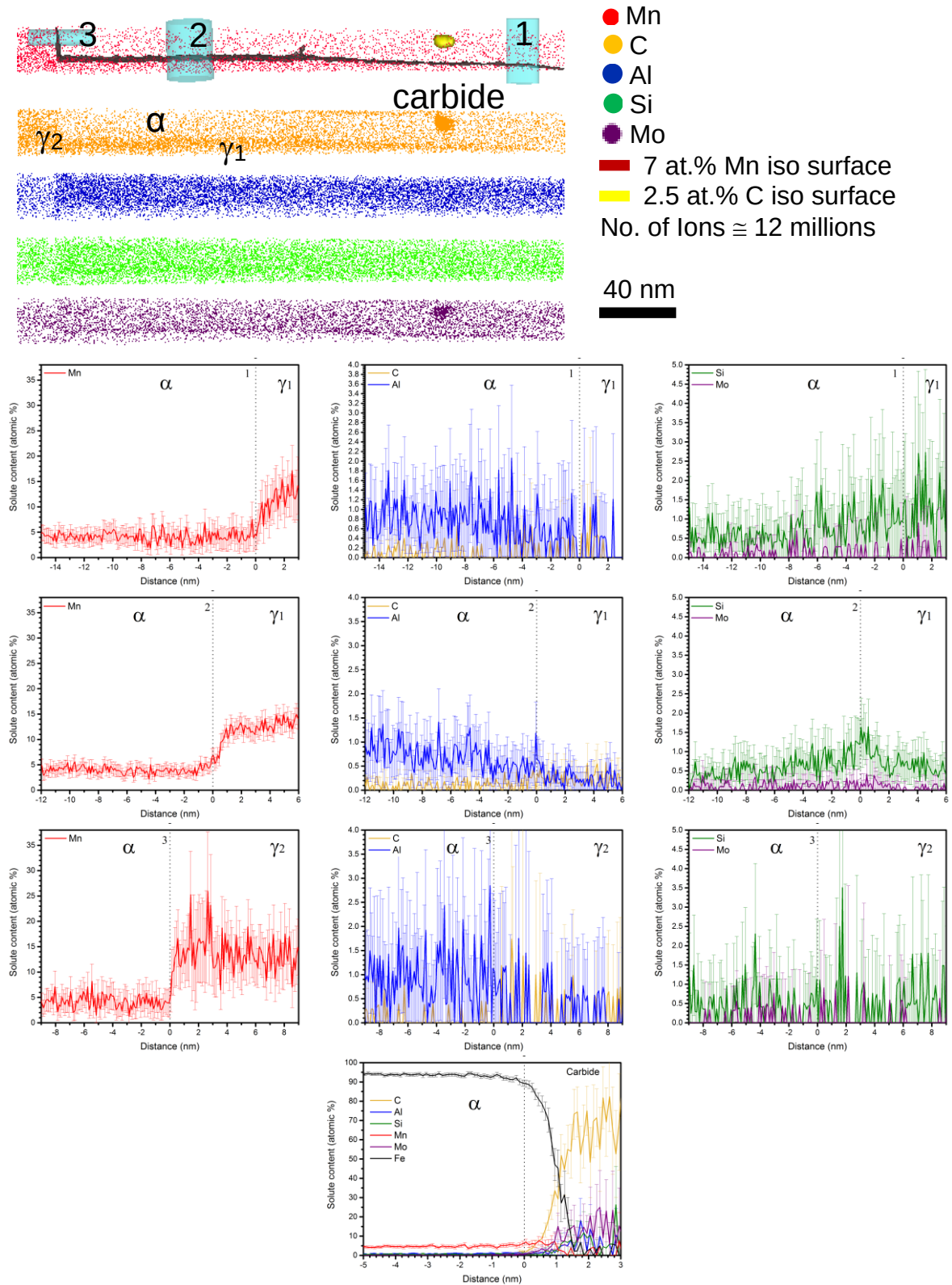


Figure 9.48. Atom probe tomogram of the 0.9 mm FeCMnAl sample annealed at 620 °C for 10 min along with the compositional profile of the Mn, Al, C, Mo, and Si atoms across the martensite- α phase boundaries and the carbides shown in the tomogram of the Mn atoms. 7 at. % Mn and 2.5 at. % C iso-concentrations are shown in the tomogram of the Mn and C atoms, respectively.

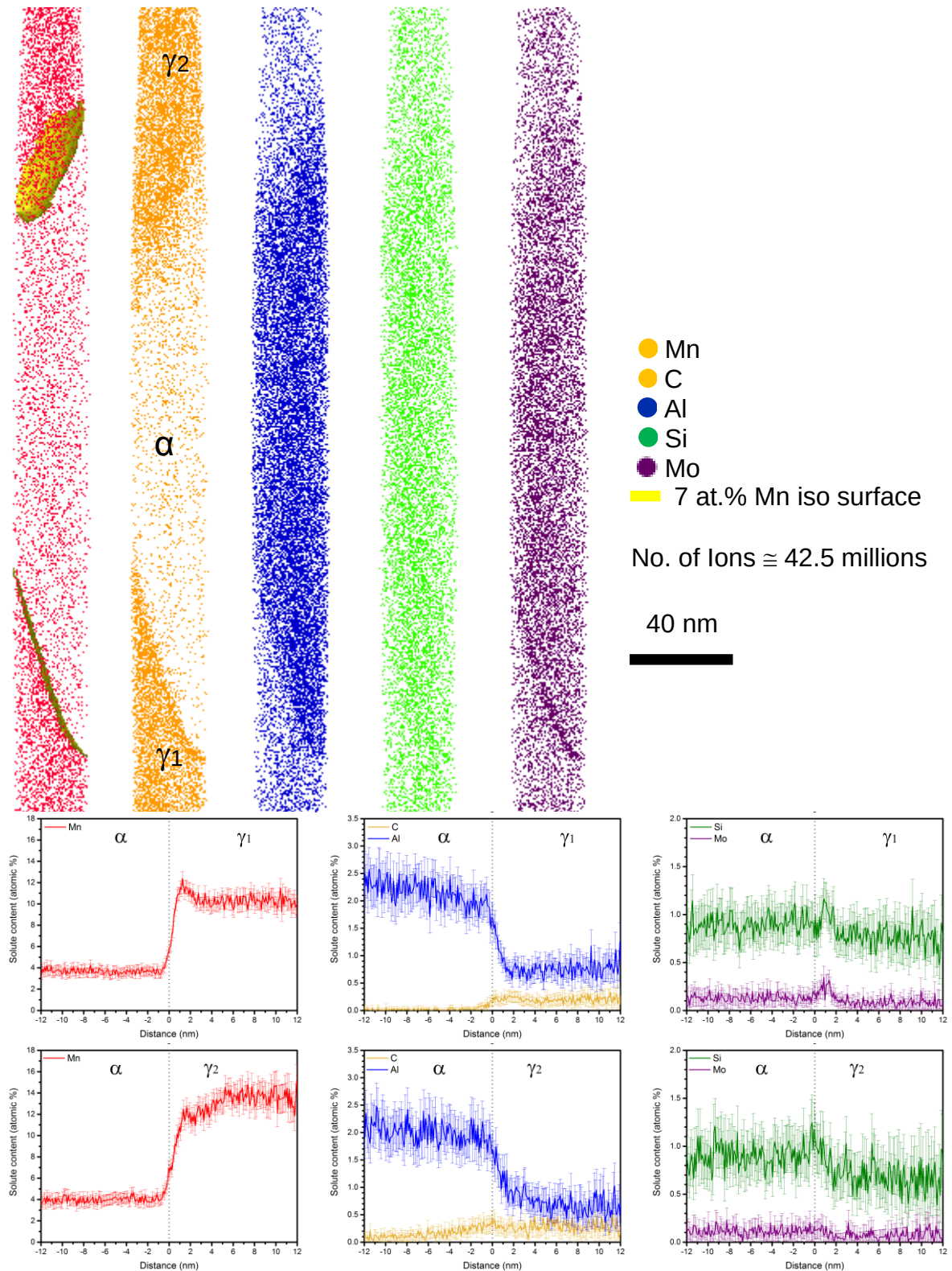


Figure 9.49. Atom probe tomogram of the 0.9 mm FeCMnAl sample annealed at 650 °C for 5 min along with the compositional profile of the Mn, Al, C, Mo, and Si atoms across the γ - α phase boundaries. 7 at. % Mn iso-concentration is shown in the tomogram of the Mn atoms.

A comparison of Figures 9.43, 9.45, 9.47, and 9.48 revealed that carbides could be present in the α grain, γ grain, or at the interface between the α and γ phases.

In Figure 9.49, the APT maps of the 0.9 mm sample annealed at 650 °C for 5 min are presented. The Mn atoms exhibited a compositional spike of 12.5% at the α/γ_1 phase boundary before decreasing to 10.5%. Moreover, the concentration of the Mo and Si atoms was observed to be higher in the α/γ_1 phase boundary, whereas no compositional spike was observed at the α/γ_2 phase boundary. Comparison of the γ_1 and γ_2 grains revealed that the γ_1 grain had a lower Mn content and a higher Al content.

In Figure 9.50, the APT maps of the 0.9 mm sample annealed at 650 °C for 10 min are depicted, indicating the absence of any compositional spike in this sample. A comparison of Figures 9.48 and 9.50 highlighted that the Mn atoms' compositional spike at the α/γ phase boundary disappeared after annealing the 0.9 mm sample at 620 °C and 650 °C for a longer duration (10 min).

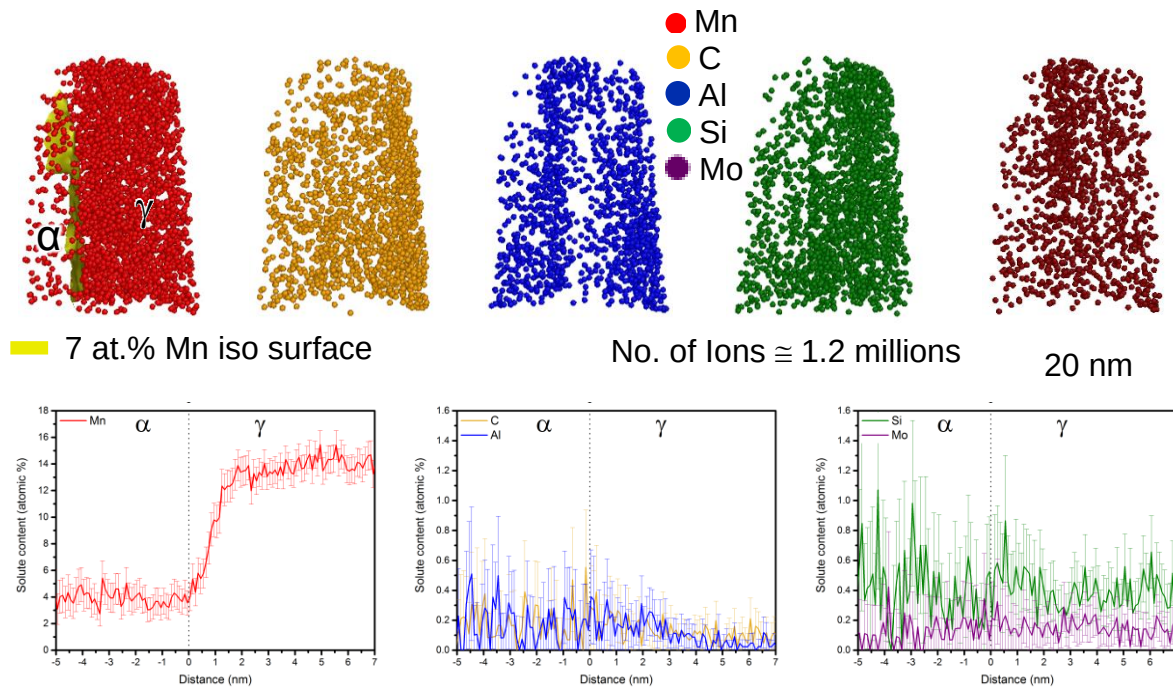


Figure 9.50. Atom probe tomogram of the 0.9 mm FeMnAl sample annealed at 650 °C for 10 min along with the compositional profile of the Mn, Al, C, Mo, and Si atoms across the γ - α phase boundary. 7 at. % Mn iso-concentration is shown in the tomogram of the Mn atoms.

Table 9.2 shows the number of the grains that were used to calculate the average composition of the α or γ phases at different annealing conditions. An analysis of the average concentrations of the alloying elements is presented for both the γ and α phases at different IA conditions in Figure 9.51. The atom probe data collected from the γ and α grains with different characteristics resulted in a range of the values for each of the IA conditions. These data were

used to calculate the error with 95% confidence and were shown as a colour band along with their average value for each annealing condition.

Table 9.2. The number of the γ and α grains to calculate the average phase composition in Figure 9.51.

Condition	Grains numbers	
	γ	α
1.8 mm, 650 °C, 5 min	5	5
1.8 mm, 650 °C, 10 min	17	11
0.9 mm, 620 °C, 5 min	10	10
0.9 mm, 620 °C, 10 min	9	8
0.9 mm, 650 °C, 5 min	14	9
0.9 mm, 650 °C, 10 min	7	11

In Figure 9.51 (a), it is shown that in the 1.8 mm samples, the Mn content in the γ phase decreased from 15.4 ± 1.7 to 15 ± 0.9 at.% by increasing the annealing time from 5 min to 10 min at 650 °C. Further reduction in the Mn content in the γ phase was observed for the 0.9 mm samples annealed at 620 °C, with a decrease to 14.7 ± 0.5 after 5 min and 14.4 ± 1.1 after 10 min. The average Mn content for the 0.9 mm samples annealed at 650 °C for 5 and 10 min was 14.4 ± 1.6 and 13.7 ± 0.6 at.%, respectively. Annealing the 0.9 mm samples at 650 °C resulted in a higher reduction in the Mn content in the γ phase compared to 620 °C for the same annealing time.

According to Figure 9.51 (a), the average Al content in the γ of the 1.8 mm sample decreased from 2.2 ± 0.4 at.% to 2 ± 0.2 at.% after 10 min of the annealing at 650 °C, while in the 0.9 mm sample annealed at 620 °C, it was 2 ± 0.7 at.% and 1.9 ± 0.1 at.% after 5 and 10 min, respectively. For the 0.9 mm sample annealed at 650 °C, the average Al content was 1.9 ± 0.4 and 1.9 ± 0.1 at.% after 5 and 10 min, respectively. The data shows that after 5 min of the annealing at 650 °C, the Al content in the γ phase remained constant.

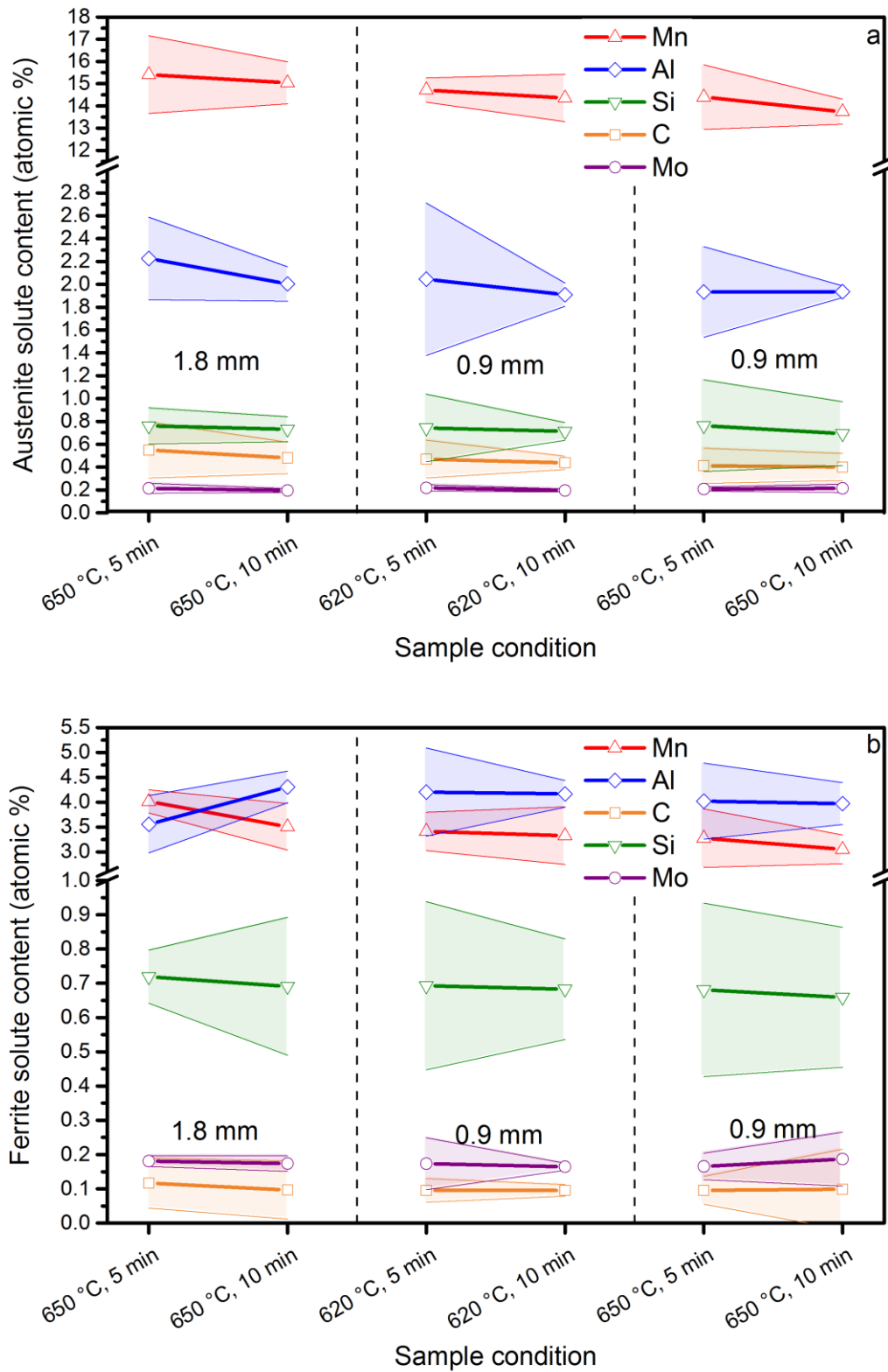


Figure 9.51. Variation in content (at.%) of the solutes in the (a) γ and (b) α phases of 1.8 mm and 0.9 mm FeCMnAl steel samples annealed at 620 °C and 650 °C for 10 min. The symbols and coloured bands show the average value and the error with 95% confidence, respectively.

The average C content of the 1.8 mm samples decreased from 0.54 ± 0.25 to 0.48 ± 0.13 after the IA at 650 °C for 5 and 10 min, respectively. Similarly, the C content of the 0.9 mm

samples decreased from 0.46 ± 0.16 to 0.44 ± 0.05 after the IA at 620 °C for 5 and 10 min, respectively. The IA of the 0.9 mm samples at 650 °C for 5 and 10 min resulted in average C contents of 0.41 ± 0.15 and 0.4 ± 0.11 at. %, respectively. The figure indicates that in all conditions, the average C content decreased over time and decreased further with an increase in annealing temperature.

The γ phase in all annealing conditions exhibited a decrease in average Si content with increasing annealing time, ranging from 0.79 to 0.86 at. %, which remained higher than the initial content of the FeCMnAl alloy (0.66 at. %). On the other hand, the average Mo content in the γ phase remained constant and did not change with the annealing conditions.

In all annealing conditions, the average Mn content decreased with an increase in annealing time, and this decreased is more significant at higher annealing temperatures, as shown in Figure 9.51 (b). For the 1.8 mm sample annealed at 650 °C for 5 min, the average Al content was 3.6 ± 0.6 at. %, which increased to 4.3 ± 0.3 at. % after annealing for 10 min. However, the average Al content remained almost constant after annealing of the 0.9 mm sample at 620 °C and 650 °C.

In 1.8 mm sample, the average C content in α decreased when the annealing time at 650 °C increased from 5 min to 10 min. However, the average C content remained almost constant after the annealing of the 0.9 mm sample at 620 °C and 650 °C.

The average Si content in the α phase after annealing the 1.8 mm sample at 650 °C for 5 min was 0.72 ± 0.08 at. %, which decreased to 0.69 ± 0.2 at. % when annealing time increased to 10 min. For the annealed 0.9 mm sample at 620 °C and 650 °C, there was a reduction in the Si content in the α phase. The average Si content of the 0.9 mm sample after annealing for 10 min at 650 °C was 0.66 ± 0.2 at. %, which was equal to the initial Si content of the FeCMnAl alloy.

The average Mo content in the α phase fluctuated between 0.165 and 0.186 at. %, and there was not discernible pattern based on the annealing conditions.

9.5 Mechanical Properties

9.5.1 Tensile Test Data

Figure 9.52 displays the engineering stress-strain curves of the FeCMnAl samples at room temperature, and the corresponding mechanical properties are presented in Table 9.3. The data highlighted the significant impact of the rolling and annealing conditions on the deformation behaviour of the samples. Notably, the samples annealed at 650 °C exhibited the discontinuous yielding, manifested by the appearance of the Lüders bands. The tensile test results of the annealed samples also showed upper and lower yield points.

The 18Al650C10 sample exhibited a yield stress of 1202 MPa, which increased to 1602 MPa in the 9AlCR sample after rolling in the TD direction. However, the yield point decreased after the second annealing and continued to decrease with increasing annealing time or temperature. The yield stress of the 0.9 mm samples annealed at 620 °C for 5 and 10 min was 1379 and 1347 MPa, respectively. After annealing at 650 °C for 3, 5, and 10 min, the yield stress of the 0.9 mm samples was 1386, 1289, and 1003 MPa, respectively. These results indicated that, except for the 9Al650C10 sample, all samples exhibited higher yield stress than the 18Al650C10 sample after the second IA.

The 18Al650C10 sample had an UTS of 1359 MPa, which increased to 1703 MPa after the cold rolling in the TD in 9AlCR sample. The UTS could not be determined for the 0.9 mm samples after annealing at 620 °C, and the lower yield point was considered as the UTS for these samples. For the 0.9 mm samples annealed at 650 °C for 3, 5 and 10 min, the UTS was 1470, 1413 and 1351 MPa, respectively. Similar to the yield stress, increasing the annealing time decreased the UTS in these samples.

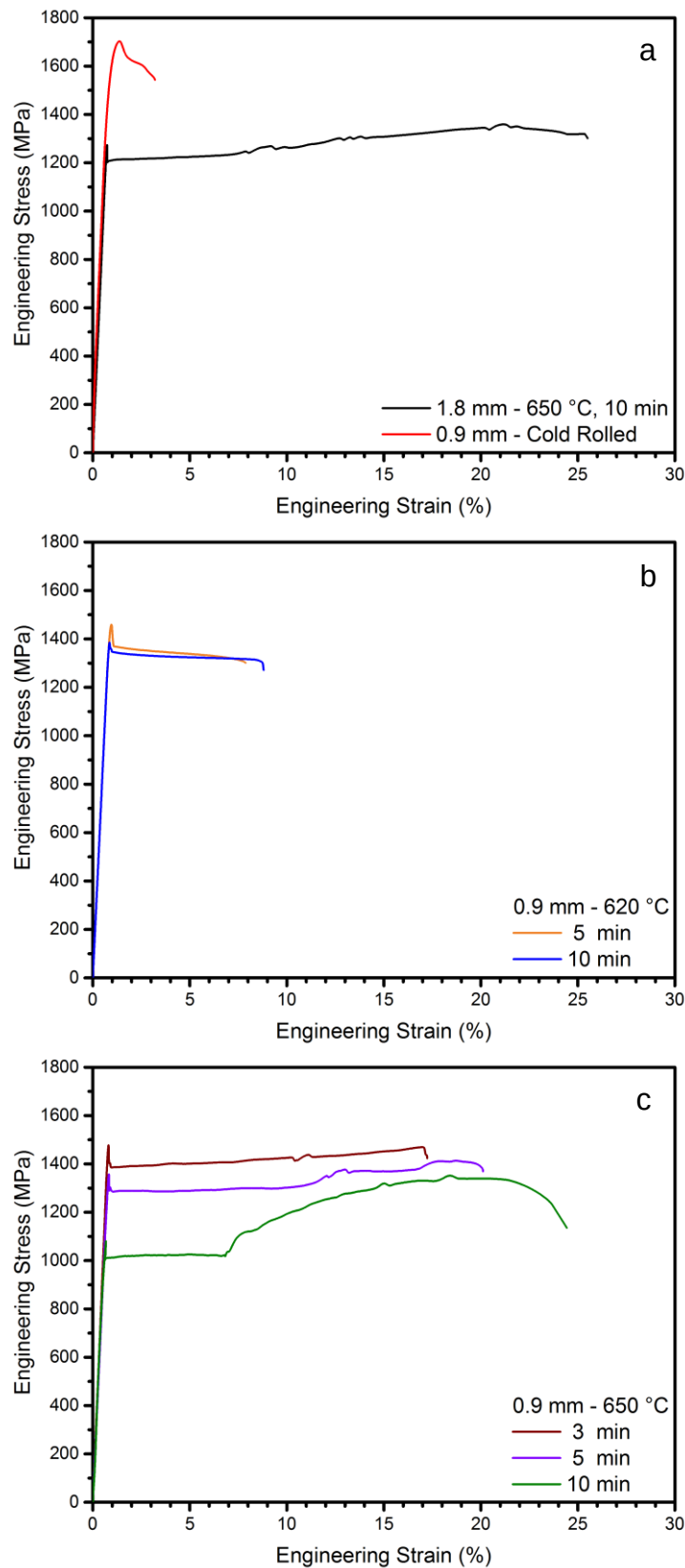


Figure 9.52. Tensile test results of the FeCMnAl samples at room temperature. The samples were prepared according to the AS1391 and tensile experiments were conducted at a constant strain rate of 10^{-3} sec^{-1} .

The ratio of the yield strength to ultimate tensile strength is often used as an indicator of the work-hardening behaviour of the materials. In the FeCMnAl samples, increasing the

annealing time resulted in a decrease in the strength ratio and an increase in work-hardening behaviour. The sample with the lowest strength ratio of 0.74 was the 9Al650C10 sample.

The 18Al650C10 sample had a total elongation of 25.5%, which dropped significantly to 3.2% after cold rolling in the TD, in the 9AlCR sample. After the second annealing, the total elongation of the 0.9 mm sample improved and continued to increase with time. For samples annealed at 620 °C, the total elongation was limited, reaching 7.9% and 8.8% after annealing for 5 and 10 min, respectively. However, for samples annealed at 650 °C, the total elongation was significantly improved, reaching 17.2%, 20.1%, and 24.4% after annealing for 3, 5, and 10 min, respectively.

In this study, the 18Al650C10 sample exhibited the highest PSE value of 34.7 GPa%. After the second IA, the 0.9 mm samples at 650 °C for 3, 5, and 10 min also showed high PSE values of 25.3, 28.4, and 33 GPa%, respectively.

Table 9.3. Room-temperature tensile test data of the FeCMnAl samples.

Sample name	Heat treatment	Rolling Direction	Heat treatment		Upper Yield Strength MPa	Lower Yield Strength MPa	Tensile Strength MPa	Strength ratio	Total Elongation %	PSE TS x TE MPa.%E
			Temperature °C	Time Min						
18Al650C10		-	-	-	1273	1202	1359	0.884	25.5	34655
9AlCR			Cold rolled		-	1602	1703	0.941	3.2	5450
9Al620C5	1.8 mm - 650 °C - 10 min	TD	620	5	1459	1379	1379	1	7.9	10894
9Al620C10				10	1385	1347	1347	1	8.8	11854
9Al650C3			650	3	1477	1386	1470	0.943	17.2	25284
9Al650C5				5	1357	1289	1413	0.912	20.1	28401
9Al650C10				10	1081	1003	1351	0.742	24.4	32964

9.5.2 Hardness Data

The hardness of the FeCMnAl samples at various conditions is presented in Figure 9.53. The 1.8 mm AR sample had a hardness of 454.7 ± 5.9 HV, which decreased to 423.6 ± 3.0 HV after annealing at 650 °C for 10 min. Subsequent 50% cold rolling increased the hardness to 510.9 ± 4.8 HV. After the second IA, the hardness decreased and further reduced with increasing soaking time. The reduction in hardness was more significant after the second IA at 650 °C than at 620 °C for the same soaking time. Except for the 0.9 mm sample annealed at 650 °C for 10 min, which had a hardness of 417.9 ± 2.6 HV, the other 0.9 mm annealed samples exhibited higher hardness than the 1.8 mm annealed sample.

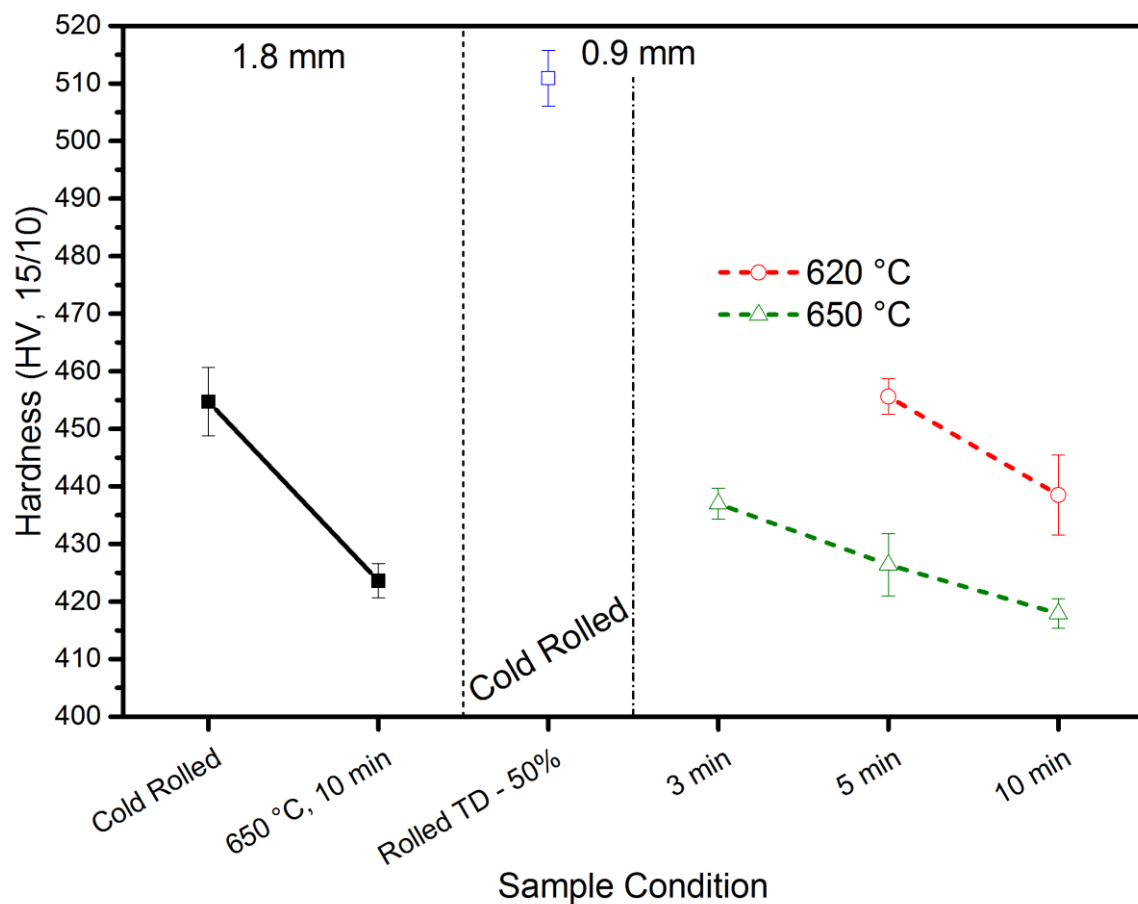


Figure 9.53. Hardness of the FeCMnAl samples after different TMP conditions under a load of 10 kg and for a dwell time of 15 Sec.

9.6 Summary

In this chapter, we investigate the effects of the TSTMP on the microstructure and mechanical properties of FeCMnAl steel. Specifically, we applied transverse cold rolling followed by a subsequent IA step after generating the initial dual-phase α and γ microstructure.

The TSTMP was found to reduce the average grain size of both the α and γ phases. Specifically, TSTMP treatments at 620 °C for 5 minutes and at 650 °C for 3 and 5 minutes led to an increase in both yield stress and UTS compared to the one-step IA condition. For a comprehensive analysis of the results, refer to the detailed discussion in Chapter 10.

CHAPTER 10 Discussion

Recognising the relatively complex array of TMP workflows exercised in this research, the discussion chapter has been separated into sections that address various key phenomenology explored. We firstly present a discussion of the $\alpha' \rightarrow \gamma$ phase transformation that occurs during the IA which is vital to the final microstructure and properties. Next follows a discussion of the solute partitioning during the IA and then follows a discussion of the austenite stability during firstly the IA and subsequently during deformation. This chapter concludes with a discussion of the strengthening mechanisms.

10.1 Martensite to Austenite Phase Transformation

To predict the final mechanical properties and modify the thermo-mechanical process, it is necessary to understand the microstructure formed during the IA processes. At the beginning of the α' to the γ transformation, the γ nucleated at the α' lath and packet boundaries, parent γ boundaries, and triple junctions. As a result of the higher concentrations of the γ stabilizing elements such as C and Mn, and a release of the stress from the host martensite, γ grains nucleate at the boundaries [59, 75, 78, 173, 341–343, 413–415]. As shown in Figures 9.43 and 9.44, there is a high concentration of the C and Mn atoms at grain boundaries. It is possible that these regions will be the preferred sites for the nucleation of the γ during the second IA process.

During the IA, if the ratio of the γ stabilizer content at grain boundaries to the grain is greater than 1, the grain boundaries are the most likely sites for γ nucleation, thermodynamically. Cai et al. [82] found this ratio, 1.1 and 1.25 for C and Mn in their study and according to the study that was published by Raabe et al. [342], this ratio was 2.5 to 5. In this study and Figure 9.44, the segregation ratio for C and Mn elements was 2 and 1.2,

respectively. Figure 9.43 shows a higher segregation ratio due to the formation of the carbide at the grain boundary, which was 6 and 1.5 for C and Mn, respectively.

Diffusionless (displacive) or diffusion-controlled mechanisms may drive the γ reversion. There is a coordinated movement of the atoms in the diffusionless mechanism. The reversed γ , as a result, possesses both a high density of the dislocations and a shape similar to the parent martensite. Furthermore, the reversed γ retains a similar texture to the untransformed γ . In contrast, diffusional-reversed γ is formed by long-range atomic diffusion. It is expected that the reversed γ obtained through diffusional processes will have low dislocation densities and random texture [408, 416].

Studies have demonstrated that increasing annealing temperatures [417], increasing heating rates [74, 75, 146, 418–420] and changing chemical compositions [416, 421] are effective methods of changing the transformation mechanism from the diffusion-controlled to the diffusionless.

In previous studies, it has been demonstrated that the α' to γ transformation is diffusionless (displacive or shear) when the heating rate is greater than 15 °C/Sec [75, 177, 418]. The heating rate used in this study was 35 °C/Sec at both the first and second IA stages. So, diffusionless reversed γ formation occurred at the beginning of the annealing.

Tomimura et al. [416] showed that chemical compositions of the steels and annealing temperatures had a significant impact on the reversion mechanism. By increasing the Ni/Cr ratio in Fe-Cr-Ni austenitic stainless steels, the Gibbs free energy changed between the fcc and bcc structures was heightened, which resulted in the fall-down of the austenitizing temperatures for martensitic shear reversion. In the steel studied here, the increase in the C and Mn composition at the grain boundary to the matrix was similar to this phenomenon.

There have been some studies that have shown that the diffusionless mechanism at the onset of the reverse transformation changed into a diffusive mechanism with the passage of the time [177, 422–424]. In a study by Kaluba et al. [423], rapid heating and quenching periods were applied for a total duration of 100–200 mSec for the heating rates. A lath-type γ was observed to form after rapid heating of the ferrite and cementite microstructures. According to their findings, the lath-type γ exhibited the following microstructural characteristics:

- initially, the product phase had a lath-like appearance,
- at the grain boundaries of the ferrite, direct formation occurred,
- the KS OR with the parent grain of the ferrite and,
- by side-by-side coalescence, the γ sheaves grew laterally.

According to previous figures, the first three features of the lath-type γ were observed in this study.

In a study by Chbihi et al. [424], it was demonstrated that when dual-phase steels are annealed at a high heating rate of 100 °C/Sec, at first, the transformation occurs under para-equilibrium condition, but after 10 Sec, the local equilibrium condition prevails. Due to the fast movement of the interface at the onset of the transformation, the Mn redistribution was not possible at the interface, therefore, para-equilibrium is the dominant mechanism. As the annealing time is extended, the interface speed decreases, which allows the Mn redistribution under local equilibrium conditions to occur. They proposed that the interaction between the Mn and the migrating interface between the α and γ plays a key role in phase transformation. Their findings indicate that the first γ islands appear to form primarily in the deformed Mn-rich α regions, and γ growth is not isotropic, but rather is directed toward the deformed regions.

It was found by Lee et al. [422] that due to the high heating rate of 10 °C/Sec, diffusionless reverse transformation occurs, resulting in lath-shaped γ , which becomes recovered and subdivided into subgrains upon further holding time. The reverse transformation, however, occurs during isothermal holding through a diffusive process without incubation time. As a result, the grains are equiaxed and the γ volume fraction increases.

It is expected that the same phase transformation mechanisms occurred during the IA in this study. The diffusionless α' to γ transformation at the first IA was less than that at the second IA. When the first IA occurred, the diffusionless transformation occurred in areas with high segregation ratios of the C and Mn atoms, which were normally found at the boundaries of the α' grains. In the next phase, the diffusive transformation and recrystallization of the γ grains were carried out. After the first IA at 650 °C for 10 min, the γ phase volume fraction was

59.8%. During the subsequent cold rolling, the γ transformed into α' , which had a higher Mn and C content than the primary martensite. During the second IA, these α' grains with higher partitioned levels of the C and Mn atoms could transform to γ with the diffusionless mechanism and then continued with the diffusive mechanism. The reason for the high volume fraction of the γ after the IA at 650 °C for 3 min (52.6%) was related to this process. Results showed that the diffusive mechanism was slower, and by increasing the soaking time to 10 min the reversed γ volume fraction increased to 58.4%.

In addition to the heating rate, the annealing temperature can affect the diffusionless and diffusive mechanisms [417]. The lower IA temperature resulted in a lower volume fraction of the γ after the IA at 620 °C.

10.1.1 Ferrite Recrystallization

The TRIP-assisted steel's final microstructure and mechanical properties are affected by high heating rates that affect not only the γ reverse transformation but also the α recrystallization. It is possible to distinguish between two types of the interactions between the γ reversion and α recrystallization depending on the heating rate, a weak interaction when heating occurs at a low rate and a strong interaction when heating occurs at a high rate. Strong interaction delays α recrystallization until temperatures above A_{C1} , the point where the γ transformation begins. As a result, both phenomena occur simultaneously, or the γ reverse transformation takes precedence. It is expected that strong interactions would occur during the IA since the heating rate in the present research was high. Literature provides details regarding weak interactions [424–427].

As a result of the high heating rate as well as the slow kinetics, the recrystallization of the deformed α was delayed. For this slow kinetic of the recrystallization, some reasons have been proposed [424, 428–430]. The amount of the internal energy that is stored in the deformed α determines the driving force for recrystallization. The reverse transformation of the γ prior to the recrystallization of the α has two consequences. Firstly, there is a relaxation in the driving force required for α recrystallization, thus preventing the nucleation of the recrystallized α [429]. Secondly, the deformed martensite is able to release the stored elastic energy through the formation of the new γ grains in a favourable orientation [424]. Furthermore, Mn delays the recrystallization of the α grains, probably as a result of its solute drag effect on migration

boundaries [428, 430]. Thus, due to the high Mn content of the alloy in this study, the effects of the Mn atoms are considerable. The ultrafine microstructure of the α phase supports this hypothesis.

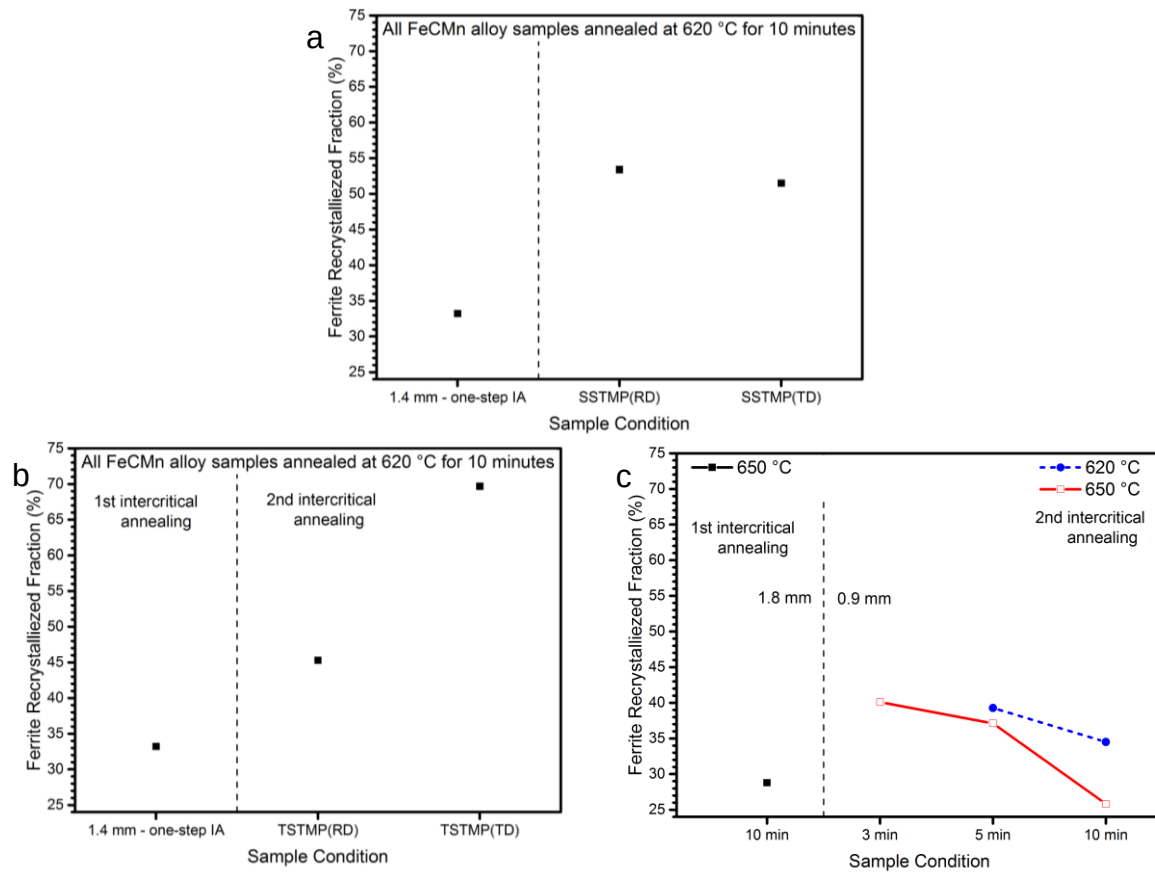


Figure 10.1. A recrystallization fraction after the IA in (a) FeCMn alloy samples after the SSTMP (chapter 6), (b) FeCMn alloy samples after the TSTMP (chapter 8), and (c) FeCMnAl alloy samples after the TSTMP after the IA (chapter 9).

Figure 10.1 illustrates the fraction of the fully recrystallized α grains. The use of a high heating rate, indicating strong interaction, substantially delayed the recrystallization of the α phase. In Figure 10.1(a), it is evident that as the annealing temperature of the IA increased, the fraction of the recrystallized α decreased at the same annealing time. This increase in annealing temperature led to enhanced γ nucleation during the reverse transformation. Since the recrystallization of the α was delayed, there was more stored energy for reverse transformation. Furthermore, a higher annealing temperature and longer annealing times reduced the stability of the γ grains, causing them to transform into martensite either during water quenching or during sample preparation using PIPS.

Figure 10.1a illustrates that the SSTMP(TD) sample had a lower recrystallization fraction in the α phase. The phenomenon could be explained by the lower thickness reduction in AR+CR(TD)20% samples in comparison with AR+CR(RD)50% samples. According to Figure 10.1b, the Rolled TD sample after the second IA exhibited a higher level of the recrystallization in the α phase when compared to the Rolled RD sample.

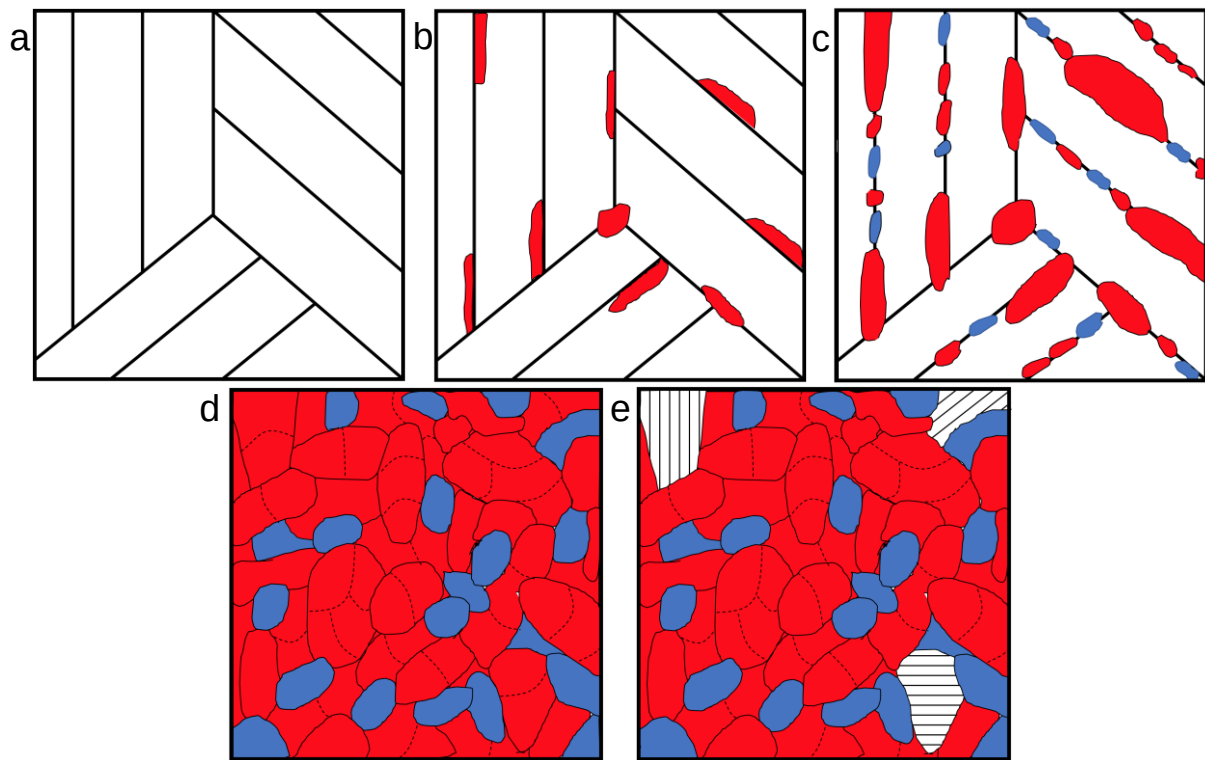


Figure 10.2. Phase transformation during the IA of the cold-rolled FeMnAl and FeMn alloys. The white, blue, and red colours indicate α' , α , and γ phases, respectively.

According to the above explanations and microstructural observations, it might be concluded that cold-rolled steel underwent the following phase transformation steps during the first IA and water quenching (Figure 10.2):

1- The γ grains nucleated at lath, packet, and parent γ boundaries since the concentration of the γ stabilizer elements such as C and Mn were much higher than in the matrix (Figure 10.2b).

2- The nucleation and growth of the γ grains and α recrystallization occurred (Figure 10.2c).

3- The lateral growth of the γ grains was followed by grain subdivision, leading to fine equiaxed γ grains. In this step, α grains underwent more recrystallization and formed the globular grains (Figure 10.2d).

4- Some unstable γ grains transformed into martensite due to the lack of the γ stabilizers and their larger grain size (Figure 10.2e).

The same steps were followed during the second IA, with some differences:

1- There was a greater volume fraction of the α' that transformed into γ during diffusionless transformation (Figure 10.2b) as compared to the first IA. This was a result of the fact that the α' grains have a higher partitioning of the C and Mn atoms.

2- The diffusivity of the interstitial and substitutional contents increased with rising annealing temperature. However, the higher γ phase fraction was mostly due to the change in thermodynamics associated with the phase transformation at higher IA temperatures. By increasing the annealing temperature, more γ nuclei generated in the regions with the lowest concentration of the γ stabilizers (C and Mn). The difference in γ volume fraction after the second IA at 620 °C and 650 °C could be attributed to this phenomenon.

10.1.2 Aluminium Effect on Phase Transformation

The α stabilizers such as Al enlarge the intercritical region and reduce the equilibrium volume fraction of the γ . Through the retardation of the carbide formation, Al increases the stability of the γ against martensitic transformation. H. Xu et al. reported that Al has no effect on the microstructure of the Fe-0.2C-5Mn steel, but can reduce equilibrium γ phase fraction as a result of changing the thermodynamics of the reverse transformation [119, 431, 432]. In this study, comparing the thermomechanical conditions in Table 8.1 and Table 9.1 reveals that the addition of Al in the FeCMnAl alloy did not significantly alter either its microstructure or morphology when compared with FeCMn alloy.

10.2 Solute Partitioning During the Intercritical Annealing

The average solute contents in the γ and α phases are shown in Figures 9.41 and 8.36. In Tables 10.1 and 10.2, the average solute content of the α and γ phases for FeCMn and FeCMnAl are shown after various IA conditions measured by APT.

In the early stages of the reverse transformation, the C atoms partition. According to Speer et al., the partitioning of the C atoms between γ and martensite at 400 °C takes less than 0.1 Sec [433]. Since the C diffusivity in the γ and α phases is higher than that of the substitutional atoms, this process occurs very quickly. The C content in the α phase reached a minimum and decreased slightly for further holding times at all annealing temperatures. In the α/α' , the C diffuses at a much faster rate than in γ [433, 434], which leads to the depletion of the martensite and α phases of the C atoms, resulting in the accumulation of the C atoms at the boundary between the phases. Increasing the holding time reduces the accumulation of the C atoms at the interface by diffusion of the C atoms in γ . In this regard, it appears that the phase boundary between the γ and α does not function as a sink for C atoms [178, 435].

At annealing temperatures of 640 °C to 680 °C, Lee et al. found that C atom partitioning in the γ phase is limited [74]. Based on the atom probe results of the specimens annealed at 620 °C and 650 °C, our results were close to theirs. In this study, the C content of the γ phase in the samples annealed at 620 °C was higher than that the samples annealed at 650 °C. The γ grain growth and a higher γ volume fraction were responsible for this phenomenon.

The results of Yen et al. showed that increasing the annealing temperature from 550 °C to 610 °C, with a heating rate of 3-4 °C/Sec, resulted in a deviation of the calculated solute contents from the results of the APT in low-carbon steel with 11 wt.% Mn micro-alloyed steel [155]. Three factors were identified, all of which were related to the short-time annealing at 610 °C, as follows:

- 1- The formation of the γ grains with widely varying Mn concentrations and a gradient of the Mn content within γ grains.
- 2- It was not possible for V_4C_3 or Mo_2C to form in such a short period of time.

3- The segregation of the C atoms or the precipitation of the cementite during heating could result in accumulating a high amount of the C atoms at the interfaces.

In equilibrium phase calculations, M_6C carbide forms between 582 °C and 683 °C in FeCMn alloy (Figure 5.3) and between 594 °C and 660 °C in FeCMnAl alloy (Figure 5.4). In the APT results, carbides were observed in the FeCMnAl samples after the IA at 620 °C and 650 °C. There may not have been sufficient time for the equilibrium carbide phase to form during IA. This may explain why the γ phase has a higher C solute content following IA. According to Table 10.2, the C content of the γ phase decreased with the increase in annealing time, as the carbide formation time increased.

There has been evidence that a high heat rate inhibits carbide formation [75] and, as a consequence, increases the C concentration in γ . The presence of the higher C concentrations in the γ phase results in the development of the γ grains that are more stable. Further, higher heating rates lead to a smaller γ grain size, resulting in a shorter mean free path for alloying elements.

In accordance with Tables 10.1 and 10.2, the α phase contained a higher C content than equilibrium conditions. As a result of the defects such as dislocations, α grains may contain a higher C content. A second reason, as mentioned before, that there was insufficient time for carbides to be formed equal to the equilibrium fraction because of the short annealing time. As a result, the C content in the α phase also increased.

Table 10.1. Phase compositions of the FeCMn alloy specimens at different conditions. Measured concentrations of the solutes with higher values than calculated equilibrium concentrations are indicated by red colour.

Thickness mm	Rolling	Temp. °C	Phase	Solute Content (at. %)			
				C	Si	Mn	Mo
1.4	-	620	α	0.023±0.027	0.678±0.078	3.54±0.373	0.134±0.012
			γ	0.398±0.051	0.686±0.034	14.6±1.167	0.160±0.011
0.7	RD	620	α	0.0406±0.046	0.593±0.631	4.31±1.154	0.142±0.006
			γ	0.401±0.108	0.709±0.277	14.4±1.576	0.159±0.013
		650	γ	0.395±0.033	0.698±0.064	12.6±0.64	0.180±0.006
	TD	620	α	0.0599±0.020	0.759±0.368	2.66±1.121	0.160±0.052
			γ	0.409±0.051	0.687±0.209	13.5±2.484	0.177±0.017
		650	γ	0.395±0.013	0.672±0.150	12.1±0.751	0.171±0.030

Table 10.2. Phase compositions of the FeCMnAl alloy specimens at different conditions. Measured concentrations of the solutes with higher values than calculated equilibrium concentrations are indicated by red colour.

Thickness mm	Temp. °C	Time min	Phase	Solute Content (at. %)				
				C	Al	Si	Mn	Mo
1.8	650	5	α	0.117±0.073	3.557±0.575	0.720±0.078	4.016±0.233	0.181±0.016
			γ	0.549±0.245	2.227±0.361	0.760±0.158	15.412±1.748	0.215±0.044
		10	α	0.097±0.085	4.306±0.318	0.691±0.201	3.506±0.468	0.174±0.023
			γ	0.480±0.138	2.003±0.150	0.732±0.108	15.041±0.947	0.195±0.016
0.9	620	5	α	0.095±0.034	4.204±0.884	0.693±0.245	3.413±0.386	0.173±0.076
			γ	0.470±0.166	2.045±0.667	0.742±0.296	14.716±0.548	0.218±0.028
		10	α	0.095±0.017	4.168±0.270	0.683±0.147	3.329±0.579	0.164±0.010
			γ	0.437±0.058	1.910±0.102	0.713±0.077	14.356±1.064	0.195±0.014
	650	5	α	0.096±0.041	4.022±0.764	0.681±0.253	3.230±0.587	0.165±0.039
			γ	0.412±0.154	1.933±0.397	0.763±0.401	14.397±1.454	0.208±0.018
		10	α	0.099±0.117	3.973±0.419	0.659±0.204	3.053±0.289	0.187±0.079
			γ	0.401±0.118	1.936±0.052	0.692±0.280	13.737±0.563	0.214±0.036

The average Mn concentration in γ and α phases is shown in Tables 10.1 and 10.2. The Mn content in γ was higher than equilibrium because nuclei were formed at locations with high C and Mn concentrations, such as grain boundaries. An increase in annealing time resulted in a decrease in the Mn content in γ until it reached the equilibrium level. However, in this study, the annealing time was not sufficient to reach the calculated equilibrium content. Even though some studies did not find a partitioning of the substitutional elements between the α and γ [178, 435], Mn partitioning has been reported at temperatures as low as around 400 °C to 450 °C [94, 342, 436]. In this study, the γ grains were ultrafine, and this also improved the partitioning of the Mn atoms.

The results in Table 10.2 indicate that Al partitioned into α and its concentration in α and γ phases was higher than that in equilibrium. A possible explanation for this phenomenon could be an overestimation of the peak area of Al in atom probe mass spectrums. As the annealing time increased, it reached close to the equilibrium levels.

Si partitioning into γ was observed to be negligible [81]. As a result of the equilibrium phase calculation, Si was partitioning into the γ phase. After the IA, the Si content in the γ was almost constant and was close to its equilibrium calculated value at various temperatures and

times. In contrast, the Si content of the α was higher than that of the equilibrium. It is possible that some of the α grains did not recrystallize, and their Si content remained high as a result.

According to equilibrium phase conditions, the Mo partitions into α . Based on APT results of the FeCMnAl alloy, the Mo content in α was close to its equilibrium level at 620 °C and about 30% lower at 650 °C. In contrast, the Mo content in γ was three times and two times higher than its equilibrium calculated amount at 620 °C and 650 °C, respectively.

Equation 10.1 can be used to calculate the diffusion coefficient in solids [437]:

$$D = D_0 \cdot \exp(-Q/RT) \quad 10.1$$

where

- D : the diffusion coefficient (m²/Sec)
- D₀: the diffusion coefficient (m²/Sec)
- Q : the activation energy (J/mol)
- T : the absolute temperature (K)
- R $\approx$ 8.31446 J/(mol·°K) : the universal gas constant.

According to Alberry and Haworth [438], the parameters for the diffusion of the Mo atoms in iron are shown in Table 10.3.

Table 10.3. Mo in Iron diffusion constants [438].

Phase	D ₀ , cm ² /s	Q, kJ/mol
Alpha Iron	0.785 (-0.059 , +0.064)	225.5 $\pm$ 4.6
Gamma Iron	0.036 (-0.004 , +0.005)	239.8 $\pm$ 26.4

A calculation of the Mo diffusion coefficient reveals that the ratio of the Mo diffusion in the α phase at 620 °C and 650 °C is 150 and 141 time more than γ phase, respectively. M₆C carbide, on the other hand, forms at these temperatures and contains approximately 70 percent Mo. Therefore, the Mo in α diffuses faster and contributes to the formation of this carbide. In this scenario, it would be predicted that by increasing the annealing time, Mo in the γ phase would diffuse and assist carbide to reach its equilibrium phase fraction, which, in turn, would cause Mo in γ to decrease with time.

For the Si and Mo alloying elements, compositional spikes were observed (Figures 9.43 to 9.50 and Figures 8.31 to 8.35). It was explained that it could occur from segregation [119, 178] or negligible partitioning local equilibrium [434, 439]. In this study, it could not be due to negligible partitioning local equilibrium or partitioning local equilibrium since the solute spikes exceeded their soluble capacities in α and γ . In addition, all the solutes in both phases did not have the same chemical potential. This suggests that the observed spikes were the result of the segregation at the phase boundary. Due to the low compositional level of the Si and Mo atoms, the segregation of these elements could not affect the stability of the γ grains. This was because of the level of these elements which was insufficient to suppress the martensitic transformation. According to the Gibbs energy balance model, they could, however, produce a solute drag effect [440, 441].

10.3 Austenite Stability After Annealing and Quenching

A critical metastable condition should exist for the γ to prevent it from transforming into α' during quenching. During the deformation, the retained γ transforms into the α' through the TRIP effect. According to the previous chapters, large amounts of the reversed γ were stabilised after annealing and quenching and remained at room temperature. During quenching, the following parameters affect γ stability:

- Grain size: The stability of the γ increases with decreasing grain size [109, 150, 154–156, 442–444]
- Chemical stabilization: The γ stabilizer elements, such as C and Mn, are partitioned and enriched in the γ during the annealing process, increasing the stability of the γ [109, 155, 436, 442–444].
- Shortage of the martensite nuclei: According to Wang and Van Der Zwaag [442], an athermal nucleation kinetics needs a nucleus density of the around $2.5 \times 10^{17} \text{ m}^{-3}$ when the γ grain size is 1 μm . A shortage of the nuclei may be responsible for the retention of the γ if the required nucleus density cannot be met.
- Dislocation in γ : Several studies have demonstrated that dislocation debris in the γ phase plays a significant role in the subsequent nucleation of the α' on the cooling to a temperature below M_s [162, 445].

A number of studies have been conducted to determine the effect of the chemical composition on the M_s or the γ stability [122–128, 130, 150, 154, 162, 333, 442, 443, 445–450]. Equations 5.1 and 5.2 proposed by Barbier [135] and Yang et al. [154] and APT results from the current study (Tables 10.1 and 10.2) can be used to calculate the α' -martensite ($M_s^{\alpha'0}$) and ε -martensite ($M_s^{\varepsilon0}$) transformation-start temperatures. In Figure 10.3, the calculated $M_s^{\varepsilon0}$ and $M_s^{\alpha'0}$ for the FeCMn and FeCMnAl alloys are shown with an open symbol.

According to chapter 5, the γ grain size influences γ stability. Equation 5.3, the calculated $M_s^{\varepsilon0}$ and $M_s^{\alpha'0}$ temperatures, and γ average grain size (Figures 9.13 and 8.11 for FeCMnAl and FeCMn alloys, respectively) were used to calculate the alpha-martensite ($M_s^{\alpha'g}$) and epsilon-martensite ($M_s^{\varepsilong}$). These results were shown in Figure 10.3 with a solid symbol.

It can be seen from Figure 10.3 that the experimental results (SEM-TKD and XRD) and theoretical results were consistent when the effect of the γ grain size was taken into consideration in the calculations. These results demonstrate that the γ grain size had a substantial influence on reducing the $M_s^{\alpha'g}$ and $M_s^{\varepsilong}$ temperatures and increasing its stability during quenching. According to Lee et al. [109], γ grains are stable at room temperature only if the average size of the grains is less than 500 nm in low carbon 6Mn TRIP steel.

It is shown in Figure 10.3 that increasing the annealing temperature or time decreased the stability of the γ . Increasing the annealing temperature and time resulted in lower values of the C and Mn content due to the dilution of the γ grains associated with increased γ phase volume fractions.

A study by Takaki et al. [156] examined the influence of the grain refinement on martensitic transformation in a Fe-16%Cr-10%Ni metastable austenitic stainless alloy steel. As a result of calculating the physical energy required for martensitic transformation in both single-variant mode and multi-variant mode, the mechanism of the γ stabilization caused by grain refinement has been clarified. According to their results:

- In order to suppress martensitic transformation from the γ to α' structure, grain refinement of the γ to 1 μ m or less is very effective.

- The transformation energy for single-variant transformation and multi-variant transformation was calculated at 1877 MJ/m³ and 106 MJ/m³, respectively. The elastic strain energy is clearly dominant in transformation energy, particularly for single variant transformations.
- There is a great deal of the chemical driving force required in order for a single variant martensite to nucleate inside an ultrafine grain γ with a grain size of 1 μ m or less.
- As a result of the ultra grain refinement, multi-variant transformation is unlikely to occur in the γ phase, and this results in the suppression of the martensitic transformation in metastable γ .

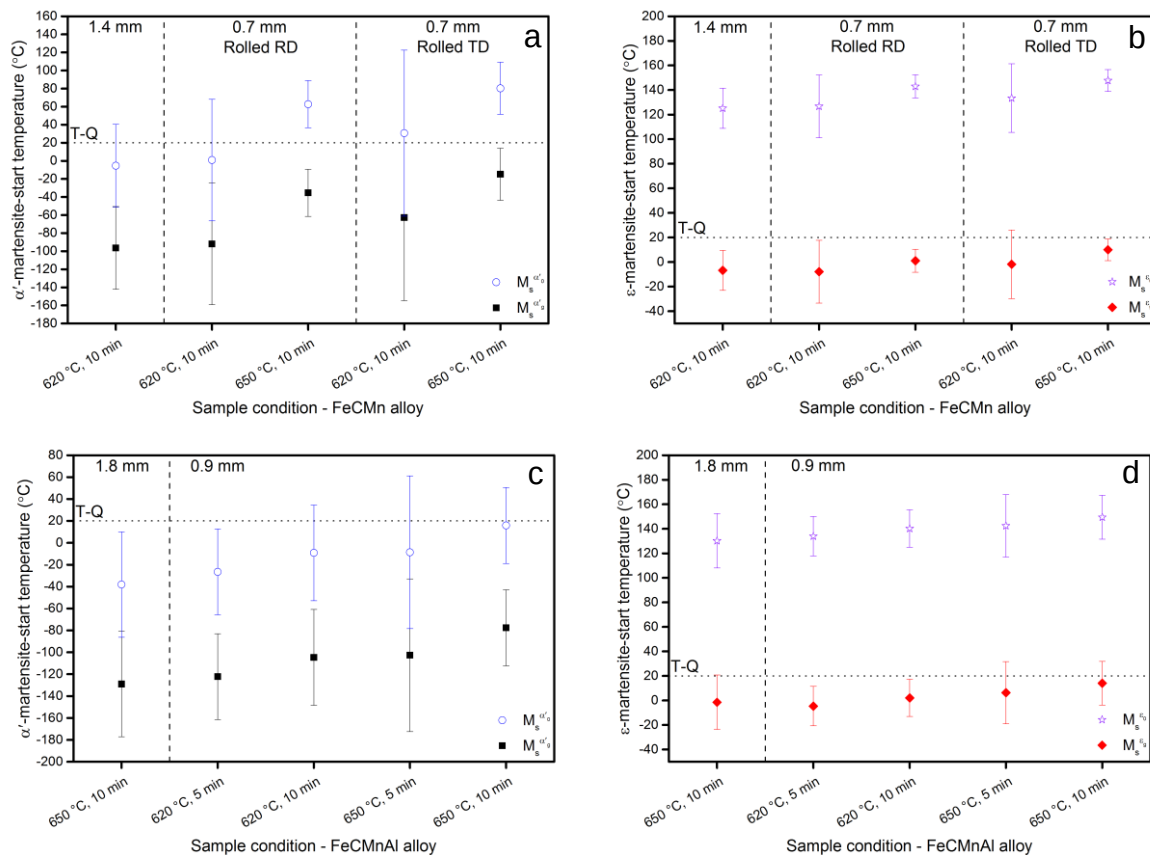


Figure 10.3. Calculated martensite start temperatures ($M_s^{\alpha_0}$, $M_s^{\alpha_g}$, $M_s^{\epsilon_0}$ and $M_s^{\epsilon_g}$) for the FeCMn steel shown in (a) and (b) and for the FeCMnAl steel shown in (c) and (d). $M_s^{\epsilon_0}$ and $M_s^{\alpha_0}$ are shown with an open symbol and $M_s^{\alpha_g}$ and $M_s^{\epsilon_g}$ are shown with a solid symbol.

Due to the lower concentration of the γ stabilizing elements in the outer layer of the reversed γ , Wang et al. [451] concluded that this layer transforms into martensite easier than the centre. Thus, larger γ grains are more stable as they contain a smaller percentage of the outer layer regions. In addition, Wang et al. [452] suggested that small grains are less stable in TRIP-maraging steels. It is observed that large γ grains are mechanically stabilized through the

development of the mechanical twinning. Compared to the larger grains, small grains are less prone to mechanical twinning, resulting in less twinning within the grains. Consequently, smaller grains are less stable when deformed.

The study of Yen et al. [155] showed that when the γ grain size is 300 nm or smaller, the transformation would take place via a single variant with nucleation occurring at the γ grain boundaries. Consequently, based on both mechanisms discussed, it can be inferred that both mechanisms are responsible for stabilizing the γ grains in this study.

10.3.1 Quantitative Evaluation of the Mechanical Stability of the Austenite

This study utilized Equation 10.2, which was developed by Sugimoto et al. [453] and used by others [104, 174, 454, 455] to determine the stability of the γ grains.

$$k_s = \frac{\ln(f_{\gamma 0}) - \ln(f_{\gamma \varepsilon})}{\varepsilon} \quad 10.2$$

Where $f_{\gamma 0}$ is the fraction of the γ after annealing, $f_{\gamma \varepsilon}$ is the γ fraction at the strain ε , and k_s is an indicator of the mechanical stability of the γ . There are several factors that influence k_s , including chemical composition, temperature of the test, strain rate, and texture. It was demonstrated by Sherif et al. [456] that the composition and the temperature of a test can affect the Gibbs free energy of the γ to martensite transformation and, therefore, the amount of k_s . As k_s value decreases, retained γ becomes more resistant to strain-induced transformation or retained γ becomes mechanically more stable.

The γ stability of the FeCMn and FeCMnAl samples was calculated in this study by using the γ fraction after annealing measured by SEM-TKD ($f_{\gamma 0}$), total elongation of the sample under each condition (ε) and the γ fraction at fracture point of tensile testing ($f_{\gamma \varepsilon}$). The k_s calculated amounts are shown in Figure 10.4. There was a good match between these results and mechanical properties. It can be seen the samples with higher elongation had higher γ stability (lower k_s).

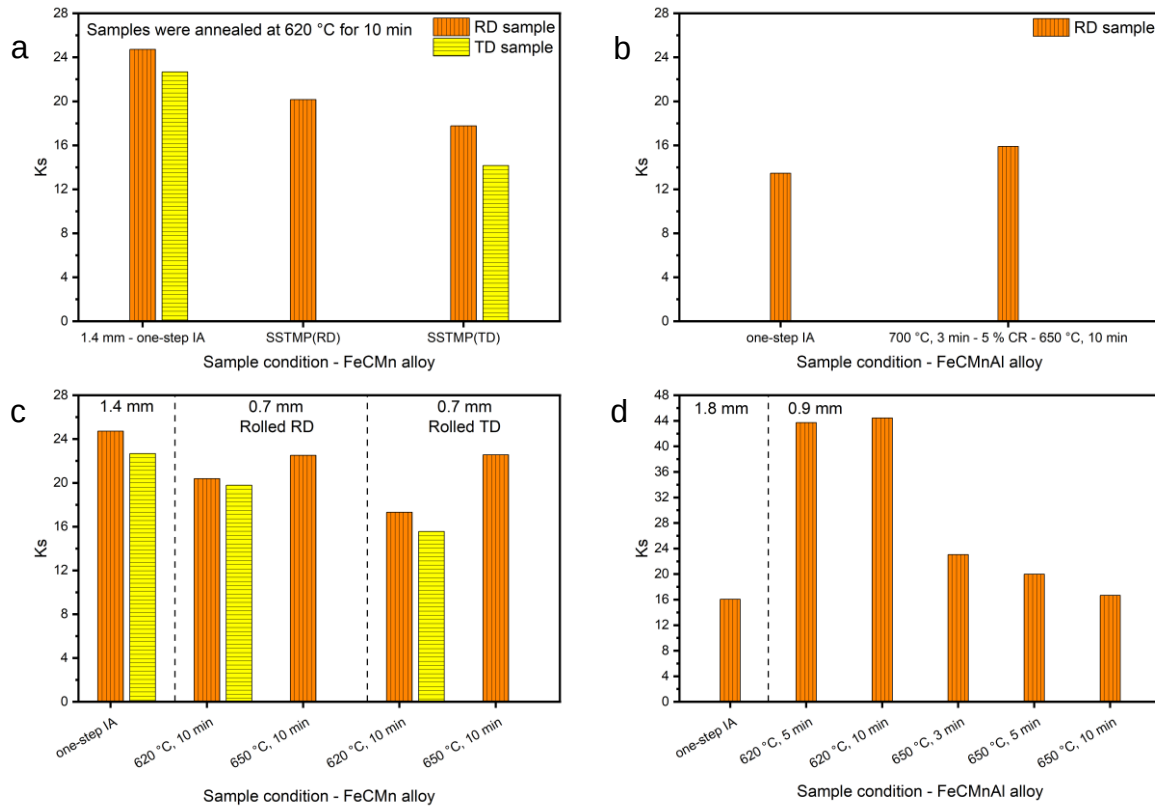


Figure 10.4. The plots of the k_s parameter for the samples of (a) the FeCMn steel after the SSTMP (chapter 6), (b) the FeCMnAl steel with different initial source of martensite microstructure (chapter 7), (c) the FeCMn steel after the TSTMP (chapter 8) and (d) the FeCMnAl steel after the TSTMP (chapter 9).

The lower k_s shows the higher austenite stability. The results of the calculated k_s shown in Figure 10.4 indicate that the Rolled TD FeCMn samples had the lower k_s values and therefore the higher austenite stability. A more stable austenite phase is less likely to transform into martensite during deformation. This retention of austenite can lead to improved mechanical properties like higher ductility, better formability, and increased strength. According to Table 6.5 and Table 8.2, the Rolled TD FeCMn samples had better mechanical properties than the Rolled RD FeCMn samples, which confirmed the lower k_s (the higher austenite stability) for the Rolled TD samples (Figure 10.4 a and c). The different textures along the tensile test direction could explain this. It has been demonstrated by Davut and Zaefferer [457] that γ grains with Brass and Goss texture components transform into martensite more easily than those with different orientations. As a result of changing the direction of the tensile sample from RD to TD, the texture of the sample along the direction of tensile testing changed and it was more difficult for γ grains to transform into martensite. As a result, the γ stability increased.

10.4 Austenite Stability during the Mechanical Deformation

As explained in chapter 2, there are parameters that affect the γ stability during the TRIP effect. The purpose of this sector is to discuss the effects of these parameters. In terms of the stability during mechanical deformation, γ grain size and composition play significant roles.

10.4.1 Grain Size and Morphology

The γ grain size has been identified as a significant parameter influencing the stability of the γ [109, 150, 154, 156, 442–444, 457, 458]. According to Figures 6.7, 7.6, 8.11 and 9.13, the average γ grain size ranged from 115 to 214 nm for FeCMn alloy and from 146 to 218 nm for FeCMnAl alloy depending on the annealing temperature. By reviewing Figures 6.26, 7.19, 8.37 and 9.52, it can be concluded that the size of the γ grain cannot be considered the main parameter influencing the stability of the γ . The γ phase exhibited a wide range of the grain sizes in the annealed samples. The FeCMn alloy samples exhibited grain sizes between 35 and 3500 nm and, the FeCMnAl alloy samples showed grain sizes between 35 and 2800 nm. So, it is reasonable to expect that the annealed specimens contain an array of the γ grains with different stability (assuming a similar chemical composition). Smaller grain sizes, however, could result in shorter mean diffusion paths for alloying contents and increase the distribution of the γ stabilizers inside the finer grain sizes. The combination of these two factors resulted in a range of the γ stability. The degree of the stability decreased with an increase in the size of the γ grain.

Figure 10.5 shows the average grain size, phase fraction, γ grain distribution and SEM-TKD IQ-phase maps of the samples that have been annealed at 650 °C for 10 min following interrupted tensile tests for various strains. It is evident that as strain increased, the fraction of the fine grains increased while the average size of the γ grains decreased. On the basis of the results of Figure 10.5, it can be determined that finer grains of the γ exhibited the higher mechanical stability.

Based on the line intercept method, Figure 10.6 illustrates the grain ratio in the final RD to the TD. If we consider the first RD of the sample for recalculating this ratio for γ grains, it is evident that the γ grains are elongated in all samples along the first RD except for the WPHT-Rolled-RD sample, which was annealed at 620 °C for 10 min. As the annealing temperature or

time increases, this ratio increases. In the WHPT-Rolled-TD FeCMn sample that has been annealed for 10 min at 620 °C, the increase is significant. As shown in Figure 8.37, this may explain the highest mechanical properties of the TD specimen.

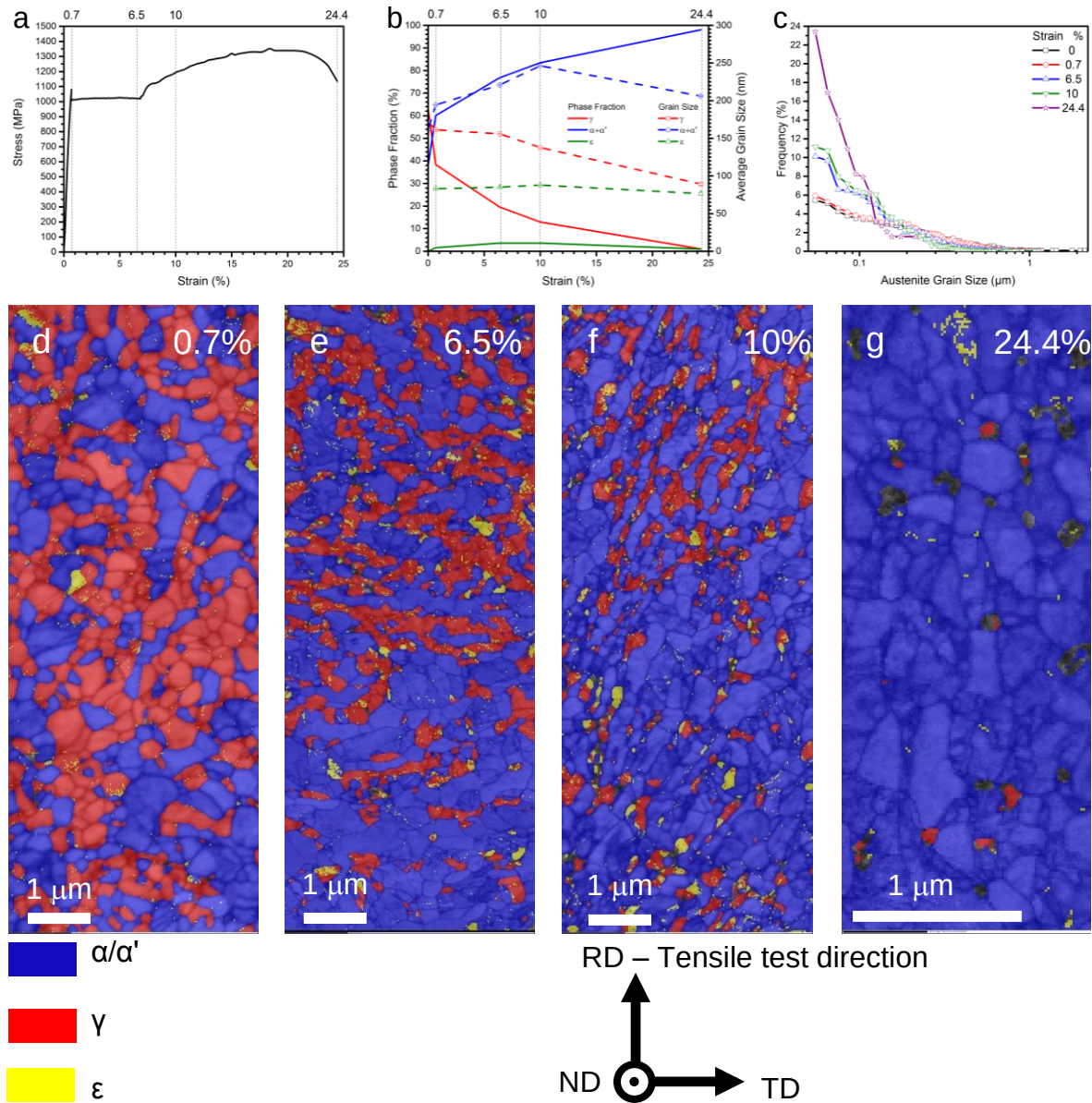


Figure 10.5. (a) Interrupted tensile test for measuring (b) average grain size and phase volume fraction of the phases, (c) grain size distribution of the γ phase, (d), (e), (f) and (g) SEM-TKD IQ-maps of the specimens after the strain of 0.7, 6.5, 10 and 24.4%. The 0.9 mm FeCMnAl alloy samples were annealed at 650 °C for 10 min before the tensile test.

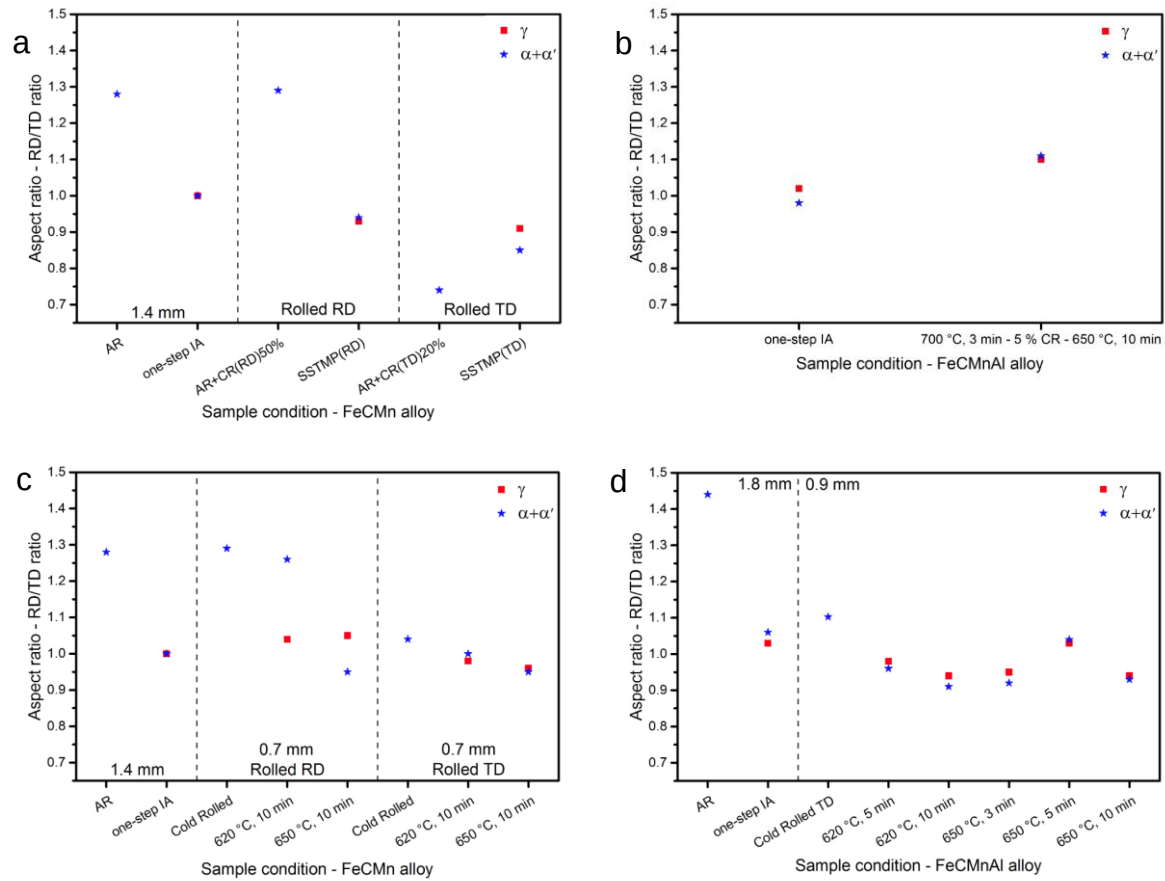


Figure 10.6. The ratio of the grains in the RD to the TD has been calculated using the line intercept method in Channel 5 software for the samples of (a) the FeCMn steel after the SSTMP (chapter 6), (b) the FeCMnAl steel with different initial source of martensite microstructure (chapter 7), (c) the FeCMn steel after the TSTMP (chapter 8) and (d) the FeCMn steel after the TSTMP (chapter 9).

Several studies have demonstrated that the morphology of the γ grains influences their stability [101, 170, 176, 178, 181, 444, 457]. When the volume fraction of the γ grains is the same, lath-shaped grains have a higher interface-to-volume ratio than globular-shaped grains. As a result, γ grains with a globular shape have fewer absorption paths for γ stabilizers. Additionally, the path for the diffusion of the alloying contents is smaller in the grains of the γ with lath shapes. The high alloying elements and stress transfer from the matrix to the γ have been found to be responsible for the higher mechanical stability in lath-shaped γ grain [170, 176, 178, 181].

As shown in SEM-TKD IQ-phase maps (Figures 10.5c to g, the morphology of the γ grains affected its stability during the mechanical deformation. A large proportion of the lath shape γ grains did not undergo transformation until high strains (Figure 10.5c), whereas most of the globular shape γ grains underwent the transformation into α' -martensite.

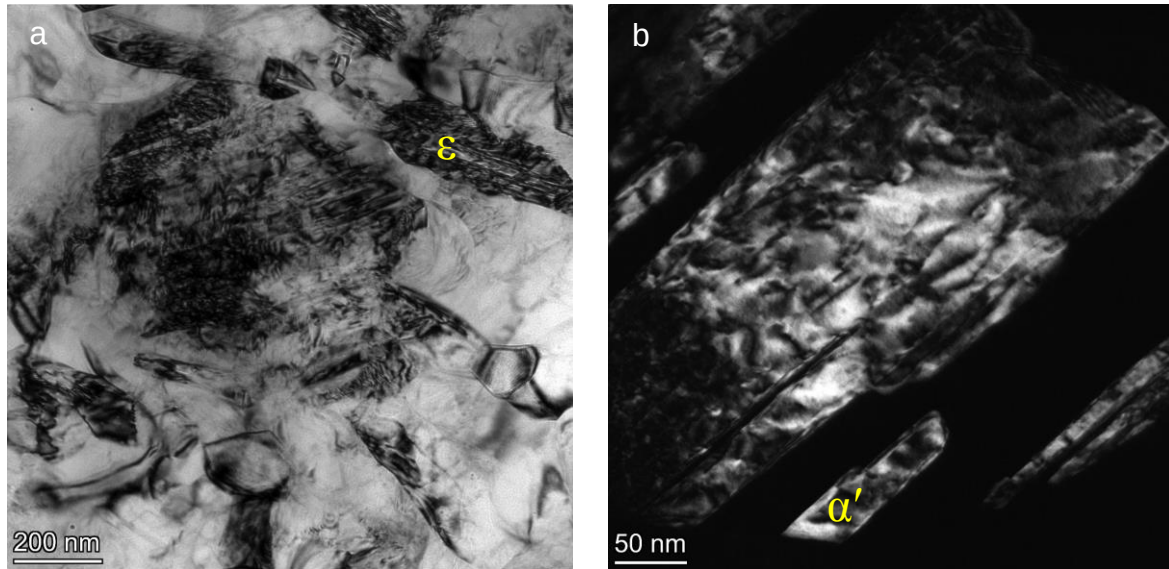


Figure 10.7. TEM bright field image of ϵ transformed from lath-shaped γ grain and (b) TEM dark field image of α' transformed from globular-shaped γ of the 0.9 mm FeCMnAl sample which was annealed at 650 °C for 10 min after 0.7% and 6.5% strains, respectively.

As can be seen in Figure 10.7, the TEM images of the 0.9 mm FeCMnAl specimen which was annealed at 650 °C for 10 min after 0.7% strain showed more information regarding morphology's influence on γ stability. According to Figure 10.7, γ grains with lath shapes were transformed into ϵ -martensite, while γ grains with globular shapes were transformed into α' -martensite. In this way, it can be demonstrated that the morphology of the γ has a significant effect on the grain stability of the γ and, in turn, on the mechanical properties of the alloy, through the delay or prevention of the TRIP effect.

10.4.2 Chemical Composition

A key factor affecting the stability of the γ during mechanical deformation is its composition. It has also been demonstrated by other researchers that the chemical composition of the γ influences its stability. Previously, we saw that increasing the annealing time and temperature resulted in an increase in the γ fraction, which consequently resulted in a decrease in the presence of the γ stabilizers such as Mn and C. APT and tensile test results demonstrate that the γ stability diminishes with decreasing γ stabilizer content [62, 74, 109, 155, 173, 436, 442–444, 454, 455, 459].

10.4.3 Dislocation Density

The dislocation density inside γ grains has been reported as a mechanism for mechanically stabilizing γ grains [162, 445]. Additionally, plastic strain is capable of introducing dislocation debris, dislocation cells, or deformation twins, which inhibits the transformation of the γ to α' [159, 456]. As a result, the γ grains become more stable during the tensile plastic deformation.

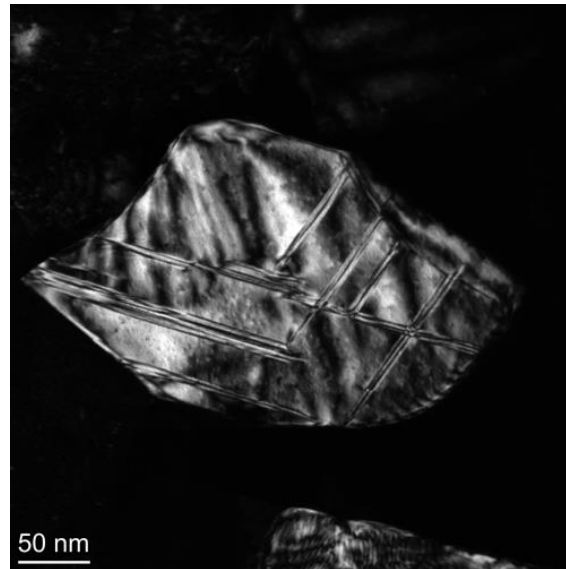


Figure 10.8. TEM dark field image of the twinning in the γ grains after the 0.7% strain.

Figure 10.8 shows the twinning in the γ grains after 0.7% strain. In the stainless steel 15Cr-9Mn-Ni-Cu, Kisko et al. [458] found numerous extended dislocations at or adjacent to grain boundaries. In ultrafine grains (100 to 500 nm), Misra et al. [460] found that twinning facilitated the formation of α' -martensite while, at grain sizes of 500 to 1000 nanometers, α' -martensite was found to occur predominantly at slip bands with a high dislocation tangle density. The findings of Laio et al. [461] indicates that partial dislocation emission occurs at grain boundaries that lead to twinning in nanocrystalline alloys. This study displayed ϵ -martensite and extended dislocations associated with grain boundaries in Figure 10.5d to e.

The results indicate that annealing the FeCMnAl sample at 700 °C and the FeCMn alloy sample at 650 °C for 10 min resulted in lower γ phase fractions than annealing them at 650 °C and 620 °C for 10 min, respectively (Figures 7.3 and 8.6). This is due to difference in the γ composition and grain size after annealing, which altered the stability of the γ during quenching. As shown in the KAM analysis (Figures 7.12 and 8.19), these samples had higher

dislocation densities near their γ boundaries. Dislocation debris near γ boundaries appeared to enhance the kinetics of the γ transformation into martensite, as α' -martensite and ε -martensite could form more rapidly (Figures 7.19 and 8.37).

The diffusionless formation of the γ grains occurred at the onset of the reverse transformation due to the high heating rate. As a result, γ grains, especially in samples that annealed at lower temperatures or for a shorter period of the time, inherited many dislocations from the martensite that deformed by the cold rolling during the SSTMP and TSTMP. By increasing the annealing time, the dislocation density in γ grains decreased as a result of the recrystallization.

10.4.4 Location of the Austenite Grain

Several studies have shown that γ grain location can influence its stability [176, 192, 210, 462, 463]. A study by Jacques et al. [176] demonstrated that γ grains isolated inside α/α' grains did not transform until the onset of the necking, while grains at the boundary underwent faster transformations. A study by Song et al. [192] found that γ grains transformed into martensite in this order: first, γ grains located at the boundaries of the α/α' grains, second, γ grains located within α/α' grains, and lastly γ grains situated at the boundaries of the martensite grains. According to this explanation, the discrepancy between the order of the transformation at different locations could be attributed to the stress condition of the γ grains in different surroundings during deformation as well as the stress levels transformed within the grains.

In the sample with 24.4% strain, a grain of the γ shielded by martensite grains is shown in Figure 10.9 that did not transform into martensite. Based on the results of Jacques et al. [463], it was shown that reducing the hydrostatic stress to the equivalent stress ratio decreased the transformation rate of the γ to martensite. Tirumalasetty et al. [210] and Li et al. [462] observed that γ grains at triple junctions and twinned γ grains were easily transformed. In contrast, γ grains at boundaries and those isolated in α/α' grains underwent transformation at large stresses only. Apparently, it was related to the resistance of the γ grains to rotation. The retained γ embedded completely in a α/α' , in contrast to the lath shape γ distributed between martensite, could resist a larger rotation angle, which would enable it to contribute more to ductility [462].

10.4.5 Surrounding Matrix

The stability of the γ grains is affected by their surrounding matrix during the deformation [176, 193, 464–466]. Stress partitioning between phases depends on the properties of the constituents, and a smaller stress within γ grains could delay γ transformation. A matrix containing hard martensite grains may alter the stress level partitioning between the phases as described by Jacques et al. [176]. In this respect, stronger matrixes, which contained larger martensitic grains, resulted in lower mean stress levels in γ grains, thus postponing martensitic transformation onset [465].

According to Timokhina et al. [193], the γ grains were more mechanically stable when the bainitic ferrite laths were refined to increase the rigidity of the bainite. Figures 9.53, 7.20, 8.38 and 6.27 indicated that samples annealed at higher temperatures or for a longer period of the time had a lower hardness. The surrounding matrix of the γ grains which was tempered martensite and recrystallized α/α' was softer in these samples. As a consequence, the stability of the γ grains should be reduced as high levels of the stress were transferred from the matrix into the γ grain. By combining a micromechanical model such as elastoplastic behaviour [467] and Eshelby's inclusion theory [468], one could get a comprehensive picture of how different constituents in the microstructure affect the γ stability.

10.4.6 Texture

According to Davut and Zaefferer's finding [457], the γ phase with Brass and Goss texture components transform into martensite much easier than γ grains with different orientations. Based on crystallographic slip theory [469–471], it appears that γ grains typically rotate either in an $\langle 100 \rangle$. It can be seen in this study (Figure 10.10) that grains rotated in a $\langle 011 \rangle$ direction with a Brass component texture to an A component texture. In an austenitic stainless steel, Hilkhuijsen et al. [208] studied the effect of the γ phase texture on martensitic transformation. They found that the intensity of the Copper, A, and Goss texture components diminished after 70% of the γ phase transformation. However, in this study, the intensity of the Brass texture component was found to decrease and the retained γ phase near the fracture area had a strong Goss and A texture component with a weaker F texture component. Only grains without a tendency to undergo transformation were present in the austenitic texture close to the fracture area, confirming that transformation was orientation-dependent [208].

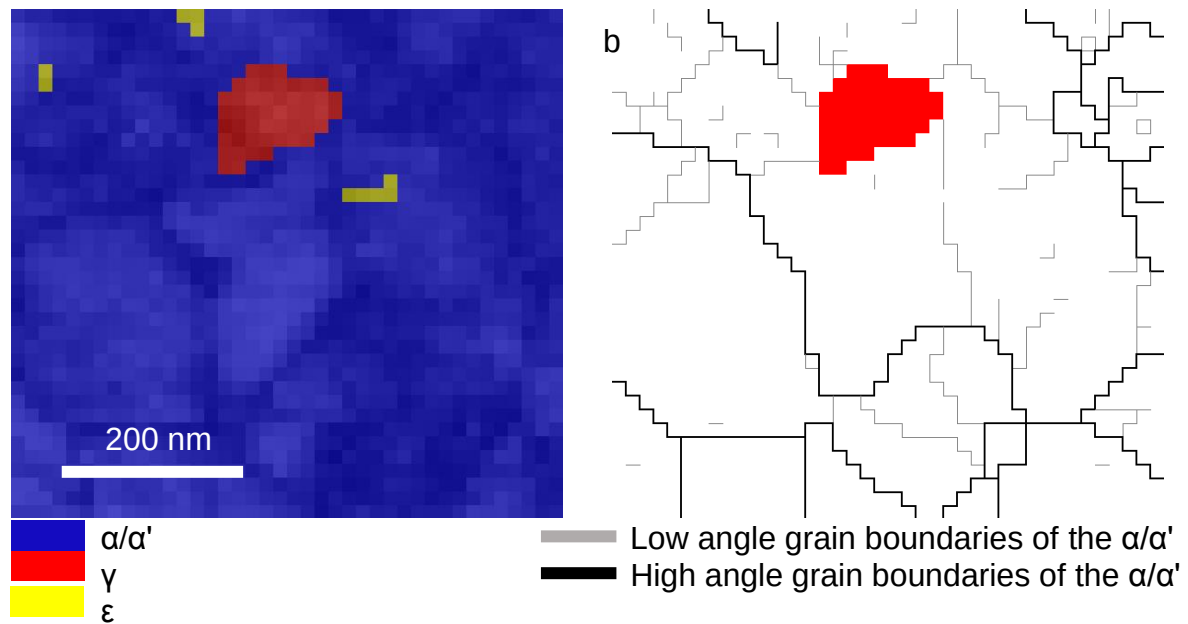


Figure 10.9. A grain of the γ phase is embedded by α/α' grains. (a) SEM-TKD IQ-map and (b) grain boundary map of the α/α' phase after the strain of 24.4% and close to the fracture area. The 0.9 mm FeCMnAl alloy sample was annealed at 650 °C for 10 min before the tensile test.

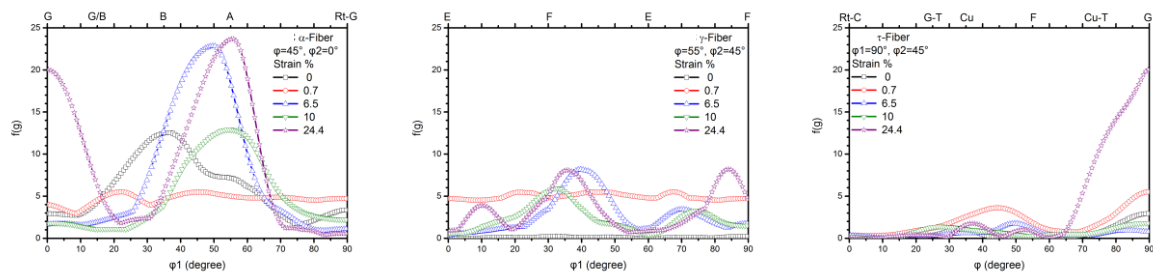


Figure 10.10. The γ phase's fibers 0.9 mm FeCMnAl alloy samples were annealed at 650 °C for 10 min during the tensile test.

10.4.7 Austenite and Matrix Orientation Relationship

Wang et al. [451] found that the Nishiyama-Wassermann (NW) or KS OR between the γ phase and matrix facilitates the penetration of the active slip systems of the γ phase through the phase boundary to the adjacent martensite. As a result, the stress at the phase boundaries decreases and the mechanical stability of the γ phase increases during the deformation process. As the samples have a high fraction of the KS OR, this factor was not a significant parameter for γ phase stability in this study.

10.4.8 Effect of Al in the FeCMnAl Steel on the Stability of the Austenite during the Mechanical Deformation

Al has been known to act as a α stabilizer [119] as well as shown in chapter 5 to decrease the phase fraction of the γ . This was done by raising both the A_s and A_f [431, 432]. It has been demonstrated that Al inhibits carbide formation in α , and increases the C content of the γ , thus increasing γ stability [419, 431].

Furthermore, Al increases the driving force of the martensitic transformation from a thermodynamic standpoint, which results in a lower degree of the γ stability. Theoretically, however, Al increases the stacking fault energy of the γ according to thermodynamic calculations [249, 366]. In closed-packed planes, stacking faults are formed by the movement of the Shockly partial dislocations caused by dislocation dissociation [472, 473]. As a result of the overlapping of the stacking faults, the nucleation of the martensite plates appears to happen at shear bands [158, 473] or γ grain boundaries [155]. Changing the stacking fault energy has a direct effect on the stacking fault width and, therefore, on the nucleation sites for martensite. Al enhances the stability of the γ against martensitic transformation by increasing the stacking fault energy in γ and reducing the potential nucleation sites for martensite.

10.4.9 Austenite Deformation Mechanism

It was found that the grain boundaries and the γ/α interfaces were effective nucleation sites for ϵ -martensite or twinning because of the high level of the grain refinement. These findings differed from those associated with intragranular nucleation models, such as the super-stacking fault model [474], the stair-rod model [475], the pile-up model [476, 477] and the spiral model [478]. As reported in Figures 10.7 and 10.8, ϵ -martensite and twinning primarily formed at grain boundaries or interfaces. Stress-controlled nucleation of α' occurred at grain boundaries during the deformation [479]. In ultrafine and nano-grained stainless steels, Misra found that faults were also emitted from grain boundaries [480]. A study on FeCMnV found that small γ grain sizes led to lamellar faults [155]. It was observed that grain refinement could result in a single variant lamellar twin structure [155, 156, 481]. Stress-controlled fault formation in ultrafine grain γ was proposed by Yen et al. [155]. There was a need for a higher level of the local stress to cause other fault systems to be triggered since the fault system often needed to propagate across existing lamellar faults, ϵ -martensite or twin structures.

Based on the fact that the boundaries were the dominant nucleation sites and the size of the γ grains was ultrafine in this study, it could be concluded that the deformation mechanism is similar to that suggested by Yen et al. [155] (Figure 10.11)

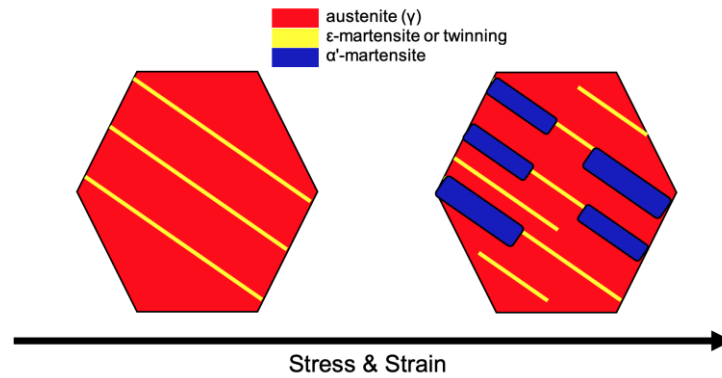


Figure 10.11. Schematic diagram of the ultrafine γ grain deformation to martensite.

10.5 Strengthening Mechanisms

It has been found that the α/α' phase dominates the yield strength of the multiphase steels (ferrite + austenite + martensite or bainite) [444, 459, 482]. The yield strength of the α phase in duplex steels with very low γ grain stability is greater than the stress necessary to induce γ phase transformation under stress. Therefore, the γ phase determines the yield strength [158, 455, 459]. It is important to note that yield strength is dependent on grain size, precipitation, chemical composition and dislocation density. Accordingly, equations 10.2 to 10.5 was used to calculate the yield strength of the α/α' and γ phases.

$$\sigma_Y = \sigma_0 + \sigma_{SS} + \sigma_G + \sigma_D + \sigma_P \quad 10.3$$

where σ_0 , σ_{SS} , σ_G , σ_D and σ_P are friction stress (Peierls-Nabarro stress), solid solution strengthening stress, grain boundary strengthening stress, dislocation strengthening stress, and precipitation hardening stress respectively. Due to a lack of the data about precipitates, σ_P was not calculated in this study.

Depending on the individual contributions of the alloying elements, the solid solution strengthening will be calculated as follows:

$$\sigma_{ss} = \sum_j c_j X_j \quad 10.4$$

where c and X represent, respectively, the strengthening effect and concentration of the element j in the α or γ phase. APT results used for this purpose.

Table 10.4. The parameters for calculating yield stress.

Parameter, Unit	α	γ	Reference
σ_0 , MPa	57	97	[27, 34]
K , MPa/ $\sqrt{\mu\text{m}}$	172	449	
M	2.95	3	[71, 483]
G , GPa	80	75	
b , Å	2.48	2.5	
c_j , MPa/wt. %	C	4670	[27, 34]
	Mn	43.9	
	Al	85.5	
	Si	41	
	Mo	60	
		33	[484, 485]

Based on the grain sizes and the Hall-Petch relation (equation 10.4), the grain boundary strengthening can be determined [224].

$$\sigma_G = K/\sqrt{d} \quad 10.5$$

where K is the Hall-Petch parameter and d is the average grain size of the γ or α/α' phase. The average grain size of the each phase was determined by the SEM-TKD method.

In order to calculate the dislocation strengthening, the Bailey-Hirsch relationship was used [226].

$$\sigma_D = \alpha M G b \sqrt{\rho} \quad 10.6$$

Where α is 0.4, M is Taylor factor, G is shear modulus, b is Burgers vector of the dislocation and ρ is dislocation density. Based on the XRD results and the Williamson-Hall method [486], dislocation densities were calculated for each sample and phase.

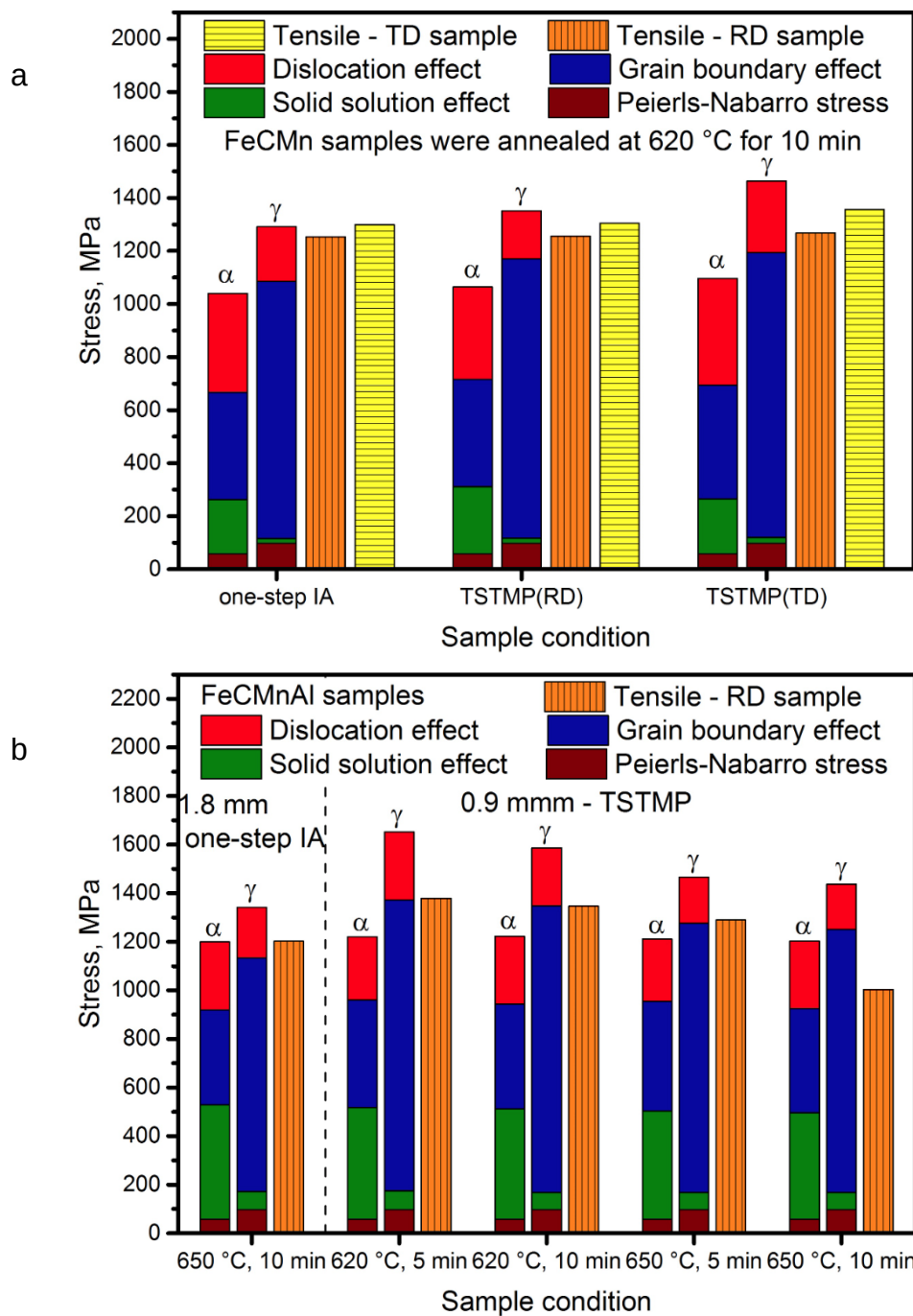


Figure 10.12. The effect of the different strengthening mechanisms on the yield strength of (a) FeCMn steel samples annealed at 620 °C for 10 min in α/α' and γ phases. In addition, yield stress values for the experimental RD and TD tensile samples are presented and (b) FeCMnAl steel samples annealed at 620 °C and 650 °C for 5 and 10 min in α/α' and γ phases. In addition, yield stress values for the experimental RD tensile samples are presented.

According to Figure 10.12, different mechanisms contributed to the yield strength of the FeCMn and FeCMnAl alloy specimens annealed at different conditions. The γ phase yield strength was largely attributed to the ultrafine structure, followed by the strengthening of the dislocations. In contrast, in the α/α' phase, all mechanisms, with the exception of the Peierls

stress, demonstrated significant contributions to yield strength. In contrast to γ phase, the effect of the solid solution strengthening in α/α' was significantly greater. In comparison with the γ phase, the α/α' phase had a much lower grain size strengthening. It could be seen that in all annealing conditions, the yield strength of the α/α' phase was lower than that of the γ grains. Therefore, the α/α' phase was the controlling factor in the yielding behaviour of the current FeCMn and FeCMnAl duplex steels.

According to the calculation and Figure 10.12, the yield strength of the 0.9 mm FeCMnAl sample annealed for 10 min at 650 °C was much greater than the experimental value. This might be attributed to the very low stability of the γ grains under this condition. At this annealing condition, the calculated yield strength of the γ phase was higher than that of the α/α' phase. It suggested that during the water quenching or PIPS step, a high density of the partial dislocations facilitated the nucleation of the ϵ -martensite and α' -martensite. Consequently, the stress-assisted transformation of the γ was achieved at lower stress than the yield strength of the ferrite phase in this sample.

Figures 10.13 to 10.16 show the work hardening rate of the FeCMn and FeCMnAl specimens annealed at various temperatures and for a variety of the holding times. The work hardening rate graphs were slightly smoothed to avoid complications. Even though different samples presented different work hardening rate curves and some of them were very similar, three stages could be considered for all of them, as shown in Figure 10.14(b).

- Stage I: Stage I shows a rapid decrease in work hardening rate, probably due to the rapid dynamic recovery of the dislocations [239, 487]. As grain size reduced to ultrafine scale, dislocation mean free paths were limited to grain boundaries rather than dislocation cells. Thus, conventional mechanisms for generating dislocations and annihilating dislocations were altered. Due to this, ultrafine grains had a reduced capacity to store dislocations or an enhanced dynamic recovery rate, resulting in a lower work hardening capacity [487, 488]. Therefore, plastic instability occurred at an early stage in the plastic deformation process of the ultrafine grain structures. It resulted in a low level of the uniformity and total elongation. There are several factors that could affect this stage, including the γ and α/α' grain size, as well as the γ stability in the specimens.

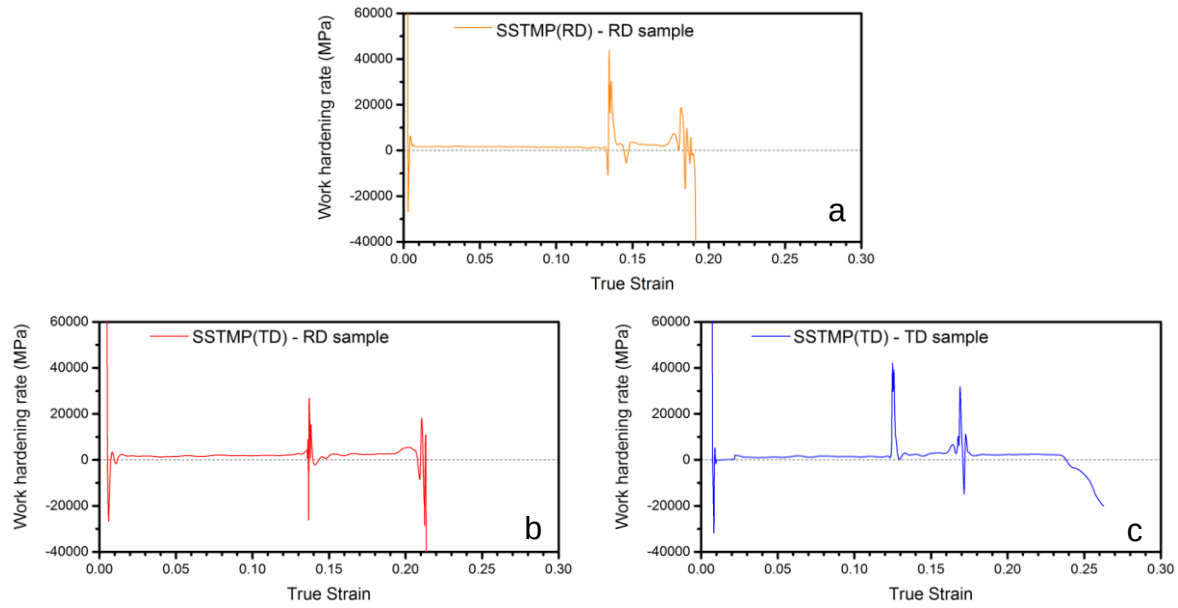


Figure 10.13. Work hardening rate ($d\sigma/d\varepsilon$) of the FeCMn samples after the SSTMP. The sample rolled in the (a) RD, (b) and (c) TD and IA at 620 °C for 10 min and strained to failure. (a) and (b) are the RD tensile samples and (c) is the TD tensile sample. (Chapter 6).

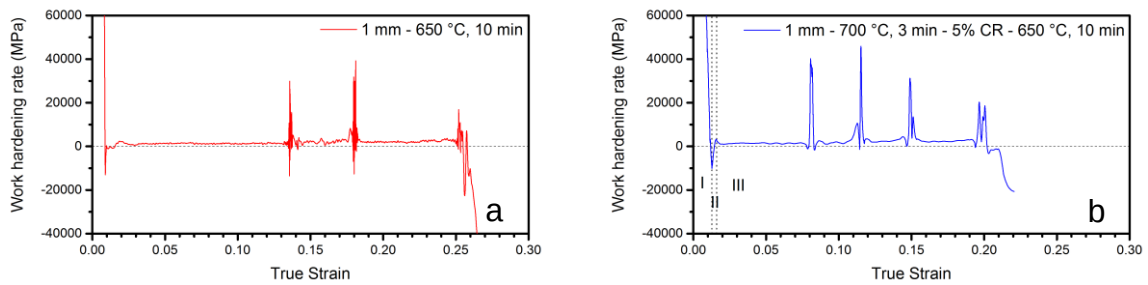


Figure 10.14. Work hardening rate ($d\sigma/d\varepsilon$) of the 1.0 mm FeCMnAl samples. (a) The 1.0 mm FeCMnAl sample intercritically annealed at 650 °C for 10 min and (b) the 1.0 mm FeCMnAl sample annealed at 700 °C for 3 min, followed by the cold rolling and 5% thickness and then intercritically annealed at 650 °C for 10 min. (Chapter 7).

- Stage II: The TRIP effect occurred in stage II of the TRIP-assisted steels, which resulted in an increase in the work hardening rate [59, 62, 104, 174]. Through localized work hardening, the TRIP effect delayed the onset of the localized necking. At stage II, the stability of the γ phase plays a significant role. The work hardening rate increased sharply as the γ stability decreased.

- Stage III: As a result of the yield point phenomenon, all the samples showed almost steady work hardening after Stage II. The yield point resulted from localized deformations in Lüders bands. Numerous materials have been reported to have yield points in ultrafine-grain

size conditions, including interstitial free steel (IF) [489], austenitic stainless steel [490], Al [489], titanium [491], and copper [492].

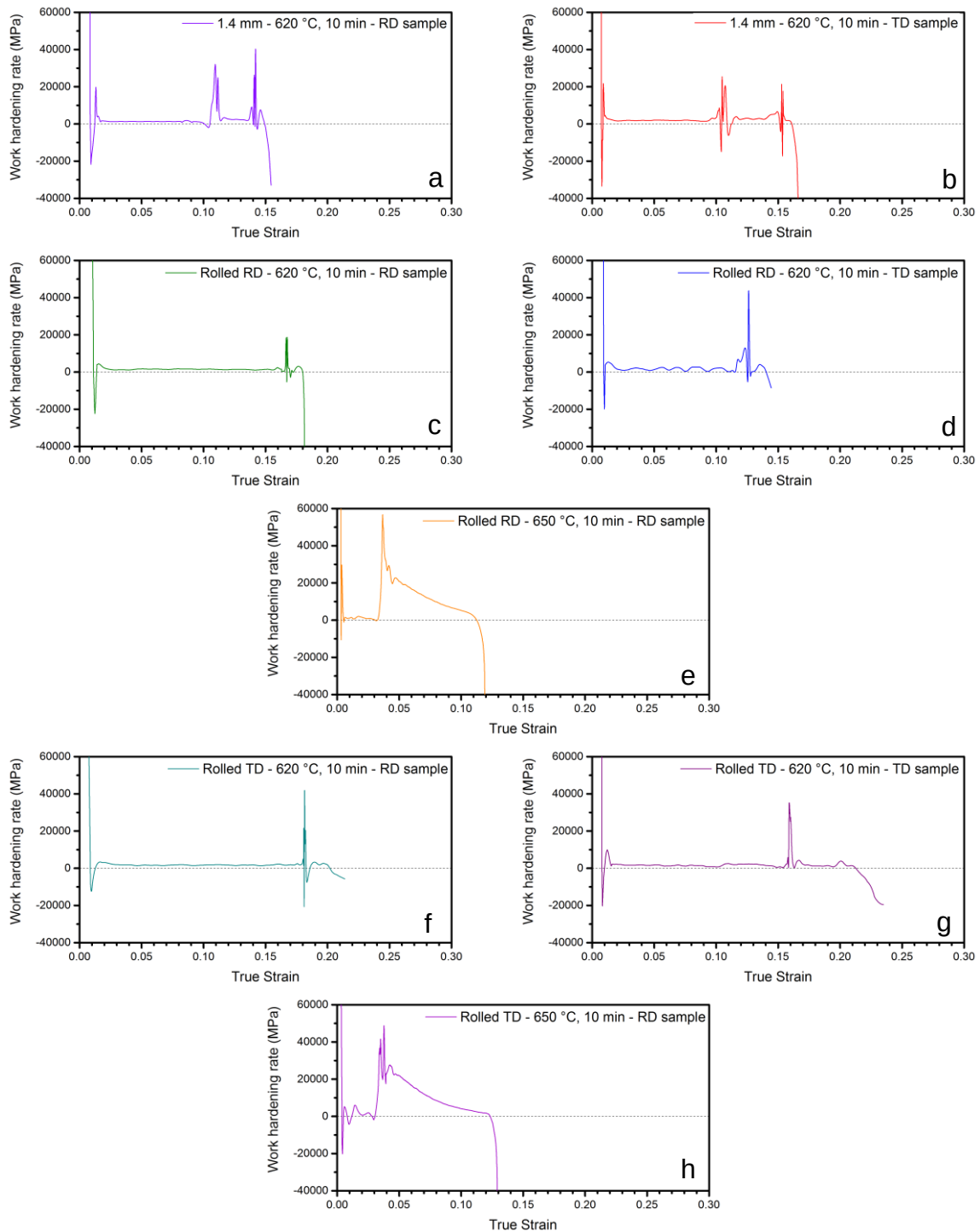


Figure 10.15. Work hardening rate ($d\sigma/d\varepsilon$) of the FeCMn specimens (a), (b) the 1.4 mm rolled RD, (f), (g) and (h) the 0.7 mm rolled TD which annealed at 620 °C or 650 °C for 10 min and strained to failure. (b), (d) and (g) are the TD tensile samples and the remaining results are for the RD tensile samples. (Chapter 8).

In an ultrafine grained MMnS, Sun et al. [351] demonstrated that γ/α' interfaces were preferential sites for nucleating dislocations, causing a sudden increase of the mobile dislocations, leading to Lüders banding. In MMnS, Lüders banding was reported to result from ultrafine-grained structures [351, 493].

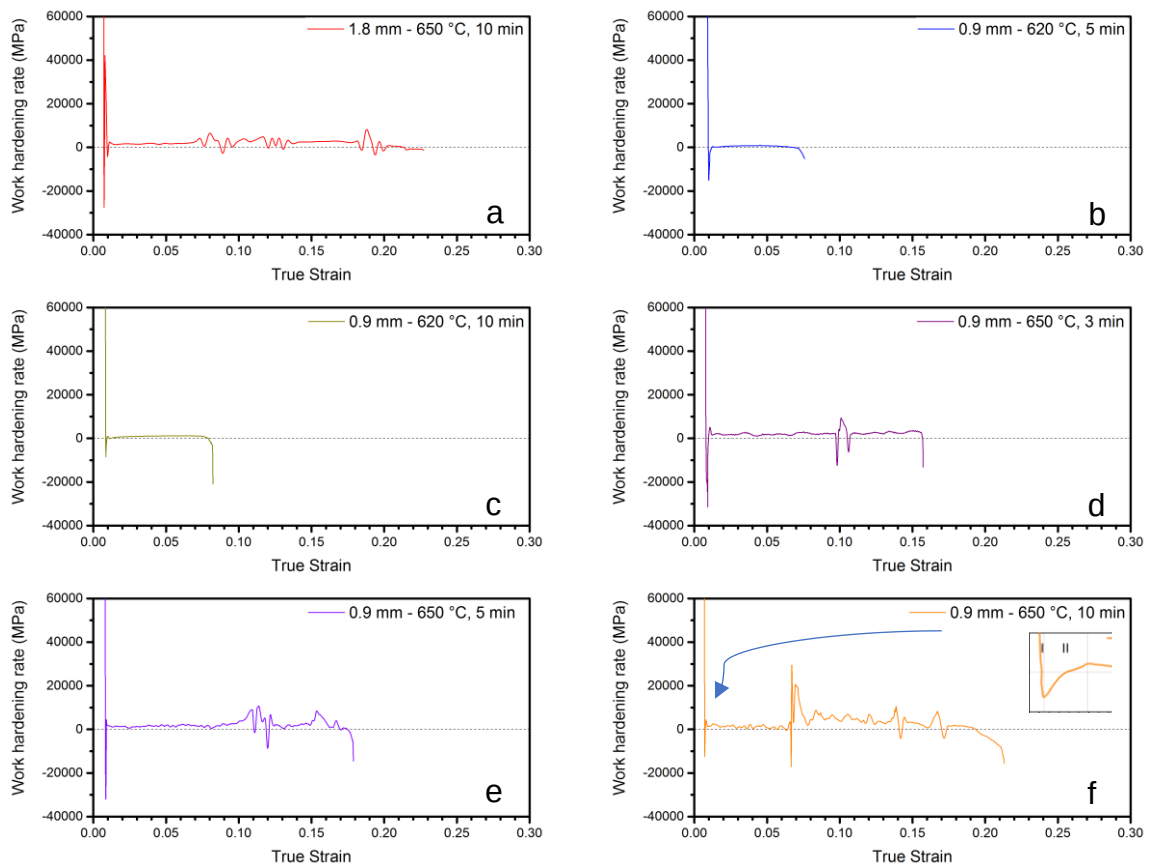


Figure 10.16. Work hardening rate ($d\sigma/d\varepsilon$) of the FeCMnAl alloy specimens for tensile test results in the RD. (a) The 1.8 mm sample intercritically annealed at 650 °C for 10 min. (b) and (c) are the 0.9 mm samples that were intercritically annealed at 620 °C for 5 and 10 min, respectively. (d), (e) and (f) are the 0.9 mm samples that intercritically annealed at 650 °C for 3, 5 and 10 min, respectively (samples of Chapter 9).

There is evidence to suggest that the yield point strain decreases with an increase in grain size [78,147]. Increasing the grain size reduces the velocity of the propagation of the Lüders bands as the grain boundary acts as a barrier against them [101, 493]. There was no correlation between grain size and strain of the yield point phenomenon in this study. Cai et al. [494] also, concluded that the ultra-fine grained or globular microstructure is not an important factor in yield point elongation.

The stability of the γ grains may also affect the yield point elongation. Several studies have demonstrated that the TRIP effect occurs in Lüders bands [9,78,148,149]. The increased stability of the γ results in a slower TRIP effect, which in turn reduces the velocity of the Lüders

band propagation, and consequently, increases the elongation caused by Lüders band propagation. Considering Figure 10.4(b), the stability of the 1.0 mm FeCMnAl annealed at 650 °C for 10 min was significantly higher than that of the 1.0 mm FeCMnAl sample annealed at 700 °C for 3 min, with 5% thickness reduction, followed by the IA at 650 °C for 10 min. As shown in Figure 10.14, higher stability resulted in higher Lüders band elongation.

Researchers have reported that the higher IA temperatures resulted in an increase in strain-induced martensite phase fraction in MMnSs [332, 494, 495]. Increasing the IA temperature causes the γ to become leaner in Mn and C, thereby reducing its mechanical stability, which increases the strain hardening [321]. It is, therefore, reasonable to conclude that the reduced mechanical stability of the γ was responsible for the lower Lüders elongation observed in Figures 10.15(e) and 10.15(h). There was a higher strain hardening rate in these specimens due to a lower γ stability.

Cai et al. [494] demonstrated that α grains with γ fiber have a high capacity for dislocation storage, which also contributed to an increase in dislocation density and work hardening exponent and a decrease in yield point elongation. The α/α' grain textures in this study indicated that specimens with stronger gamma fibers had lower yield point elongations.

In Fe-0.05 pct C-6.15 pct Mn-1.5 pct Si (in mass pct) TRIP steel, Lee et al. [74] observed three Lüders bands. The specimens shown in Figures 10.14(a) and 10.14(b) had the highest numbers of the formed Lüders bands among those studied here, with three and four Lüders bands, respectively. Some other specimens had two formed Lüders bands.

There was a notable fluctuation in the work hardening rate curves, which was associated with the serrations on the stress-strain graphs. This is known as the Portevin Le-Chatelier effect (PLC). Plastic deformation is localized into PLC bands as a result of a non-steady plastic flow [496–498]. DSA is responsible for localized plastic deformation and the PLC bands [33, 280, 281].

Cottrell [282] presented the first theory describing the DSA effect in solid solutions, in which dislocations interact with interstitial solute atoms (such as C and N). As a result of the interstitial solute atoms, dislocations cannot move, and ultimately become trapped in the regions occupied by solute atoms. A rapid unpinning of the dislocations occurs when sufficient

stress is applied and pinned dislocations can overcome the critical energy barrier that prevents them from continuing their movement. During this rapid pinning and unpinning of the dislocations, the stress drops in the stress-strain curve, creating the PLC effect. FeCMn and FeCMnAl alloys exhibited this mechanism, except FeCMnAl alloy samples after the IA at 620 °C. The discontinuities TRIP effect dominated in the FeCMnAl alloy samples intercritically annealed at 650 °C.

CHAPTER 11 Summary

Throughout the duration of the PhD program, advanced microscopy techniques and mechanical assessments were utilized to design TMPs for two MMn TRIP-assisted steels, resulting in several significant findings, as outlined below.

Addressing Aim 1: *To combine CALPHAD methods, empirical calculations of the M_s temperature and DSC experiments to explore the effect of alloying a base Fe-C-Mn AHSS composition with Al on phase composition. Our emphasis was on the effects of Al on phase fractions and transformation temperatures, given the potential of this element to further light-weight the steel and its capacity as a ferrite stabiliser.*

- Both thermodynamic calculations and experimental results indicated that Al increased both the A_s and A_f temperatures and decreased the M_s temperatures.

- When comparing the FeCMn steel with the TSTMP at 620 °C and the FeCMnAl steel with the TSTMP at 650 °C for 10 min, the following observations were made:

- 1- The FeCMn steel had a lower proportion of LAGB in both the γ and the α/α' phases.
- 2- The average grain size for both the α/α' and γ phases was almost the same for both steels, but the aspect ratios were lower in FeCMn steel.
- 3- The FeCMnAl steel had a higher reduction in grain size for both the γ and α/α' phases after the TSTMP compared to one-step IA of the AR steel.
- 4- Both steels had almost the same γ phase volume fraction after the TSTMP conditions explained above.

Addressing Aim 2: *To investigate the effect of increasing the initial dislocation density ahead of intercritical annealing via an additional cold rolling step to the as-received base FeCMn AHSS. The concept here was to understand the changes in microstructure and*

properties when there is an enhanced driving force for the $\alpha' \rightarrow \alpha + \gamma$ phase transformation during intercritical annealing.

- Cold rolling of the AR FeCMn steel with initial fully ferritic/martensitic microstructure to a reduction in cross-section of 50% in the RD in one workflow, and 20% in the TD in a second workflow increased the dislocation density in the specimens.

- According to the XRD data analysis, the dislocation density increased 58% in the AR+CR(RD)50% specimen and 42% in the AR+CR(TD)20% specimen.

- According to the LAGB analysis, the strain distribution increased 19% in the AR+CR(RD)50% specimen and 35% in the AR+CR(TD)20% specimen.

- According to the KAM analysis, the strain distribution increased 17% in the AR+CR(RD)50% specimen and 13% in the AR+CR(TD)20% specimen.

- The DSC test results revealed that the peak temperature of the martensite to the γ transformation decreased with an increase in the dislocation density in the initial martensitic/ferritic macrostructure. This reduction in temperature became more significant as the dislocation density in the microstructure increased further. However, the endset temperature remained almost constant.

- The SSTMP resulted in almost the same yield stress and tensile strength in comparison to the one-step IA specimen. The SSTMP increased the total elongation of the RD tensile sample of the RD cold rolled specimen by 26%, the RD tensile sample of the TD cold rolled specimen by 43%, and the TD tensile sample of the TD cold rolled specimen by 65%. The TD tensile sample of the TD cold rolled specimen after the IA resulted in yield stress of 1306 MPa, tensile strength of 1514 MPa, total elongation of 30%, and PSE of 45.4 GP.%E.

- The SSTMP resulted in a higher amount of the retained γ at room temperature, with an approximately 8% increase. This may be attributed to the fact that increasing the dislocation density created more γ nucleation sites, leading to the formation of the smaller γ grains with higher stability at room temperature.

- The SSTMP resulted in 3-7 nm smaller average grain size of the γ and 10 nm smaller average grain size of the α/α' in comparison to the specimen after the one-step IA.

- The SSTMP resulted in a lower strain distribution in the γ phase compared to the one-step IA, as evidenced by KAM and LAGB analyses.

- The SSTMP resulted in lower aspect ratios of the α/α' and γ grains, or in other words, the specimens exhibited the granular-shaped γ and α/α' grains.

Addressing Aim 3: *To explore the effect of the nature of the martensite in the initial microstructure prior to intercritical annealing. Specifically, we generated starting microstructures such that ~70% of the martensite was generated by transformation from quenching from the austenite, and ~30% was transformed via deformation of the retained austenite.*

- The specimen with the initial deformed martensite microstructure and the specimen with the quenched and deformed martensite microstructure after the IA at 650 °C for 10 min exhibited the yield stresses of 1184 MPa and 1026 MPa, the tensile strengths of 1351 MPa and 1316 MPa, and the total elongations of 30.4% and 24.7%, respectively. The lower mechanical properties for the second specimen might be due to the austenitization annealing process, which resulted in the lower Mn content in the γ phase and therefore reduced the stability of the γ . The IA at a shorter time less than 10 min may lead to the better mechanical properties.

- DSC results showed that the peak and endset temperature of the martensite into the γ transformation increased in the specimen with the quenched and deformed martensite microstructure compared to the specimen with the deformed martensite microstructure. This increase in the temperatures could be due to the austenitization annealing or the longer time of the IA in the specimen with the quenched and deformed martensite microstructure, which resulted in a decrease in the average Mn and C content in the γ phase.

- After the IA, the sample with the deformed martensite microstructure resulted in the γ and α/α' phases with the average grain size of the 218 nm and 194 nm, respectively. While, in the specimen with the quenched and deformed martensite microstructure after the IA, the γ and α/α' phases had the average grain size of the 146 nm and 134 nm, respectively.

- The specimen with the deformed martensite microstructures after the IA exhibited the 60% volume fraction of the γ phase, which was almost as same as the specimen with the initial quenched and deformed microstructure.

- The specimens with the initial quenched and deformed martensite microstructure had the higher dislocation density in both γ and α/α' phases after the IA, as indicated by the KAM analysis and LAGB%.

- The aspect ratio of the γ and α/α' grains was not affected by the source of the martensite.

- The ratio of the grain size along the RD to the TD was one for both γ and α/α' grains in the intercritically annealed specimen with the initial deformed martensite microstructure, while it was 1.1 for both the γ and α/α' grains in the intercritically annealed specimen with the initial quenched and deformed martensite microstructure. This difference could be due to the 5% cold rolling before the IA process.

- It would be beneficial to investigate the impact of the annealing time on the specimen with the quenched and deformed microstructure, as it was not explored in this study.

Addressing Aim 4: *To assess the impact of the TSTMP on microstructure and mechanical properties. Specifically, we applied a second stage of cold rolling and subsequent intercritical anneal after generating the initial dual phase $\alpha+\gamma$ microstructure. This part of the study also examined the effect of the rolling direction during the second stage. Rolling was applied in both the transverse direction (TD) and in the initial rolling direction (RD). The assessment of the TSTMP was the major work of the thesis.*

- The yield stress of all FeCMn steel and FeCMnAl steel specimens, except for the FeCMnAl steel that underwent the second IA treatment at 650 °C for 10 min, were observed to exceed 1250 MPa after the TSTMP. These yield stress values were higher compared to the specimens after the first IA. Additionally, in the FeCMn steel, the yield stress of the TD tensile specimens was found to be higher than the RD tensile specimens.

- The FeCMn steel exhibited a tensile strength exceeding 1390 MPa, while the FeCMnAl steel exhibited a tensile strength exceeding 1350 MPa after undergoing the TSTMP.

Furthermore, in the FeCMn steel, the TD tensile specimens displayed the higher tensile strength compared to the RD tensile specimens.

- Following the TSTMP, all FeCMn steel specimens, including both RD and TD tensile samples, exhibited the total elongation of over 20%, which was a higher value than the specimens after the first IA treatment.

- Upon analysis of the strengthening mechanisms, it was determined that the yield strength was primarily influenced by the α/α' phase.

- For FeCMn steel, when the second IA of the TSTMP was as same as the first IA, the phase volume fraction of the two specimens were almost similar.

- Increasing the temperature of the second IA, caused an increase in the volume fraction of the retained γ phase, a larger average grain size of the γ , and a lower percentage of the LAGB.

- After the first IA of the FeCMn steel, the average grain size of the γ was 215 nm, and after the second IA, it decreased to 181 nm. The average grain size of the α/α' phase remained unchanged at 181 nm.

- When the temperature and time of the first IA and the second IA were same, the second IA resulted in lower LAGB.

- In this study, all the annealed specimens exhibited an average aspect ratio of the γ grains greater than 1.7. This could be attributed to the high heating rate utilized, which prompted the nucleation of the γ phase via the diffusionless mechanism that subsequently transitioned to the diffusive mechanism during the γ grain growth. The influence of the annealing temperature on this diffusionless nucleation mechanism was found to increase with rising the temperature.

- After the first IA, the ratio of the grain sizes in the RD to the TD was found to be one for both the γ and α/α' grains, indicating a random distribution of the lath-shaped γ grains in the microstructure. However, the cold rolling in the RD led to an increase in this ratio.

- The use of the TSTMP resulted in a greater deviation of the γ and α/α' phase boundaries from the KS OR, indicating a significant effect on the microstructure of the specimens.

-The utilization of the APT revealed the partitioning of the Mn and C atoms into the γ phase, and the Al into the α phase. However, the spikes in the C, P, Si and Mo distributions were observed due to their segregation and the carbide formation at the phase boundaries.

- The APT results indicated that the distribution of the Mn atoms within the α grains was almost uniform. However, it was observed that the Mn distribution gradually changes along some of the γ grains, which may disappear with increasing the annealing time and allowing for the Mn atoms diffusion.

- APT results revealed that the average Mn content of the γ grains varied. However, increasing the annealing time led to the reduction in the deviation of the Mn content in each grain from the average Mn content of the γ grains.

- APT results indicated that increasing the temperature and time of the IA led to the reduction in the average Mn and C content in γ grains. This might be attributed to the increase in the volume fraction of the γ phase.

- APT results demonstrated the presence of the Mo carbides in the α grain, γ grain, or at the interface between the α and the γ phases.

- The work-hardening process could be divided into three stages. In Stage I, there was a significant decline in work hardening rate, possibly due to the rapid dynamic recovery of the dislocations. In Stage II, the TRIP effect took place, which led to an increase in the work hardening rate. Finally, in Stage III, the yield point phenomenon occurred along with the localized deformations in Lüders bands and PLC bands. Three Lüders bands were formed in some of the specimens.

- After subjecting the FeCMn steel to the TSTMP at 620 °C, the TD rolled specimens exhibited the highest total elongation of 23.8% and 26.5%, in the RD and the TD tensile samples respectively, which was 42% higher than the specimens after the first IA, while maintaining the similar yield stress and tensile strength. In this specimen, the PSE was

measured to be 38.8 GPa.%E and 33.3 GPa.%E in the TD and the RD tensile samples, respectively, which was 40% higher than the specimens after the first IA.

- In the FeCMnAl steel, the specimen subjected to the TSTMP and the TD cold rolling showed a 7% increase in the yield stress and the 4% increase in the tensile strength, along with the 20% reduction in the total elongation, after the second IA at 650 °C for 5 min, compared to the specimen subjected to the first IA at 650 °C for 10 min. However, the specimen subjected to the TSTMP and the TD cold rolling, followed by the second IA at 650 °C for 10 min, resulted in the yield stress of 1003 MPa, which was 16% lower, but almost the same tensile strength and total elongation, as the specimen after the first IA at 650 °C for 10 min. These findings suggested that the IA at 650 °C and the time between 5 to 10 min might result in the highest mechanical properties, which were not investigated in this study.

- The yield stresses of the phases calculated for the FeCMn steel after the TSTMP at 620 °C. In Rolled TD specimen, it was 1097 MPa and 1464 MPa for the α/α' and γ phases, respectively. For the Rolled RD specimen, it was calculated 1064 MPa and 1351 MPa for the α/α' and γ phases, respectively.

- The peak and endset temperatures of the α/α' into γ transformation were lower in the specimens that underwent the cold rolling in the TD with the same amount of the thickness reduction as those cold-rolled in the RD. This could be attributed to the higher dislocation density and the increased number of the γ nucleation sites resulting from the cold rolling in the TD.

- In the FeCMn steel, the TD cold rolling resulted in the slightly higher γ volume fraction (about 1%) in comparison to the RD rolled specimens after the second stage of the TSTMP.

- In the FeCMn steel, after the austenitization annealing at the second stage, the Rolled TD specimen exhibited the retained γ volume fraction of 22.5% at room temperature, which was significantly higher compared to the Rolled RD specimen that only exhibited 9.5%.

- In FeCMn steel, both RD and TD cold rolling during the TSTMP resulted in the smaller γ grain size (175 nm). However, while the RD cold rolling did not show any reduction in the α/α' grain size, the TD cold rolling decreased the α/α' grain size by about 10% to 161 nm.

- In the FeCMn steel, the austenitization annealing of the TD rolled specimen during the second stage resulted in the formation of the γ and α/α' phases with the average grain size of 149 nm and 187 nm, respectively. In comparison, these grain sizes were 115 nm and 187 nm for the RD rolled specimen. The γ and α/α' grain sizes in both conditions were smaller than the grain sizes after the first IA.

- In the FeCMn steel, after the TSTMP, the aspect ratio of the annealed Rolled TD specimen was similar to that of the annealed Rolled RD cold rolled specimen.

- The TD cold rolling resulted in the decrease in the ratio of the grain sizes in the RD to those in the TD, indicating that more grains were aligned with the RD of the initial sample. In this calculation, the sizes of the grains along both the RD and TD were taken into account.

- In the FeCMn steel, the LAGB% after the second IA at 620 °C was same for both phases of the RD and TD cold-rolled specimens but this amount after the annealing at 650 °C was about 12% lower in the γ phase for the TD cold-rolled specimen.

- In the TSTMP, the TD rolled specimens showed more deviation of the γ and α/α' phase boundaries from the KS OR.

- The XRD results indicated that the AR specimens with the martensite microstructure had the highest intensity of $(211)_\alpha$ peak, which remains the highest after the IA or the RD cold-rolling. However, the TD cold-rolling led to the decrease in the intensity of $(211)_\alpha$ peak and the peak with the highest intensity changed to $(200)_\alpha$ peak.

- The APT analysis of the annealed specimens showed that the γ phase in the Rolled-RD samples had the higher Mn content compared to the Rolled-TD samples.

CHAPTER 12 Future Work

The study presented findings on the effect of a TSTMP, which involved altering the initial microstructure and incorporating Al into the MMn-AHSSs. However, several areas for future research have been identified:

1. One area for future research is to investigate the physical and mechanical properties of the copper-based alloys that contain AHSSs duplex microstructures as precipitates, with varying shapes and distributions of these precipitates. During the PhD candidature, three such alloys were designed and cast, and it is also worthwhile to explore the effects of the TMPs on the size and distribution of these precipitates in the microstructure.
2. Examining the impact of the adding copper to MMnS, considering the shape and distribution of the copper precipitates and how they affect the physical and mechanical properties of the AHSSs.
3. Investigating the potential benefits of the adding small amounts of the Hf and Zr atoms to AHSSs, as these elements have been found to significantly increase hardness when added to iron binary alloys. However, it is crucial to take into account that Hf and Zr tend to form carbides, so the amounts of the C, Hf, and Zr atoms in these alloys must be carefully considered.
4. Exploring how the mechanical properties of the AHSSs could be modified by the short time of the annealing at the third stage of the TMP process, given that austenitization annealing during the second stage was found to result in lower yield stress and lower total elongation.
5. Studying the mechanical properties of the AHSSs under tensile test with different strain rates or other mechanical tests in the room or high temperatures, as the specimens in this study were only investigated at room temperature with tensile and hardness tests.
6. Investigating the effect of the adding alloying elements on hydrogen embrittlement in AHSSs.

7. Designing high and medium Mn steels specifically for additive manufacturing applications.
8. An exploratory study using transmission electron microscopy could be undertaken to assess the relative fraction and distribution of the martensitic and ferritic phases in the final microstructures.

CHAPTER 13 References

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Appendix 1 : Scanning Electron Microscopy Maps of Specimens

In this appendix from right to left (a) the IQ-phase map, (b) the IPF-Z of the austenite phase, (c) the IPF-Z of the ferrite phase and (d) the recrystallization map of the specimens are shown. The legends of the maps are as follows.

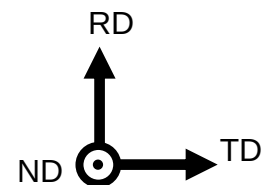
- IQ-phase map



α/α'



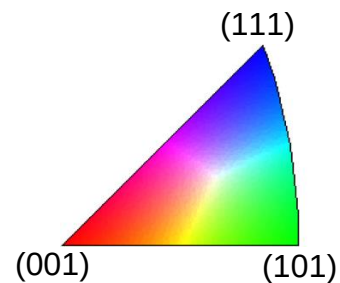
γ



- IPF-Z map

— Low angle grain boundary ($2^\circ < \theta \leq 10^\circ$)

— High angle grain boundary ($15^\circ < \theta$)



- Recrystallization map



Recrystallized



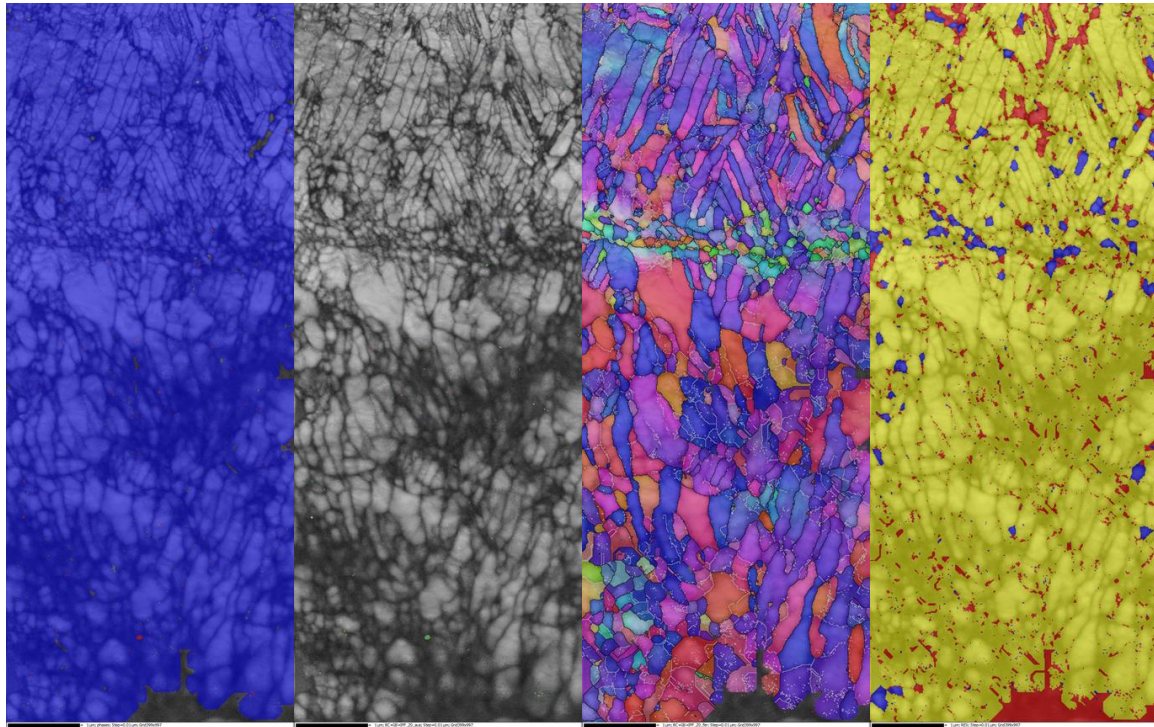
Deformed



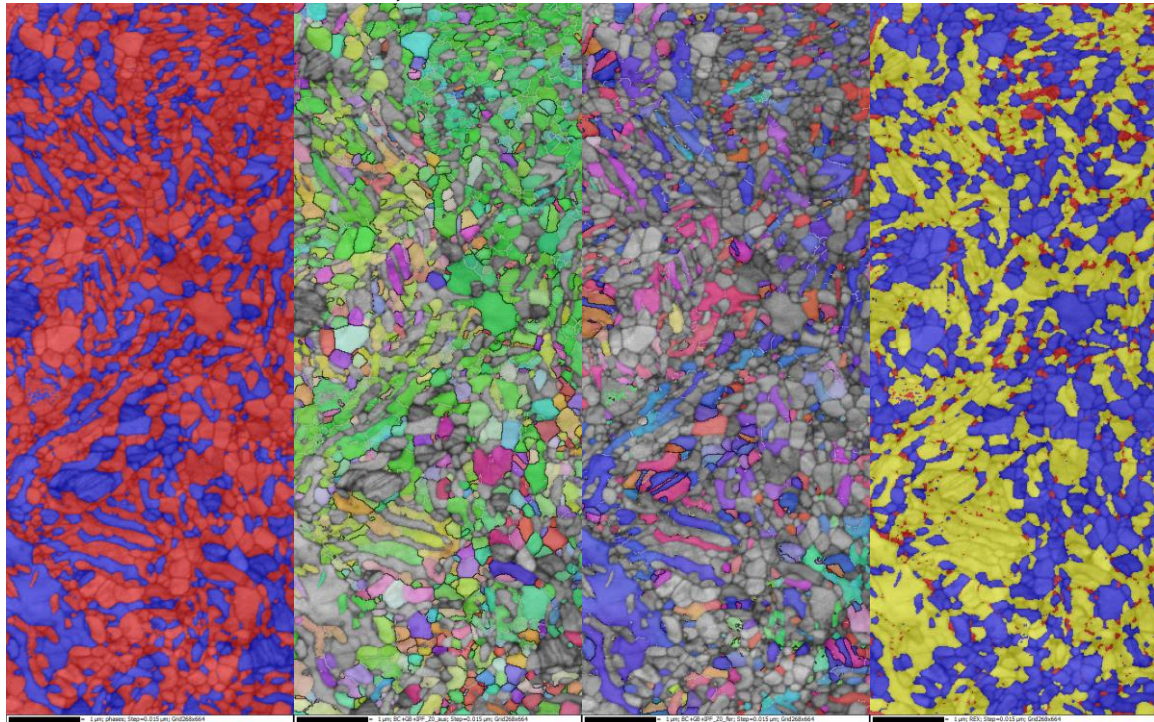
Substructured

A1.1 FeCMn Steel Specimens

FeCMn steel - 1.4 mm – Cold Rolled

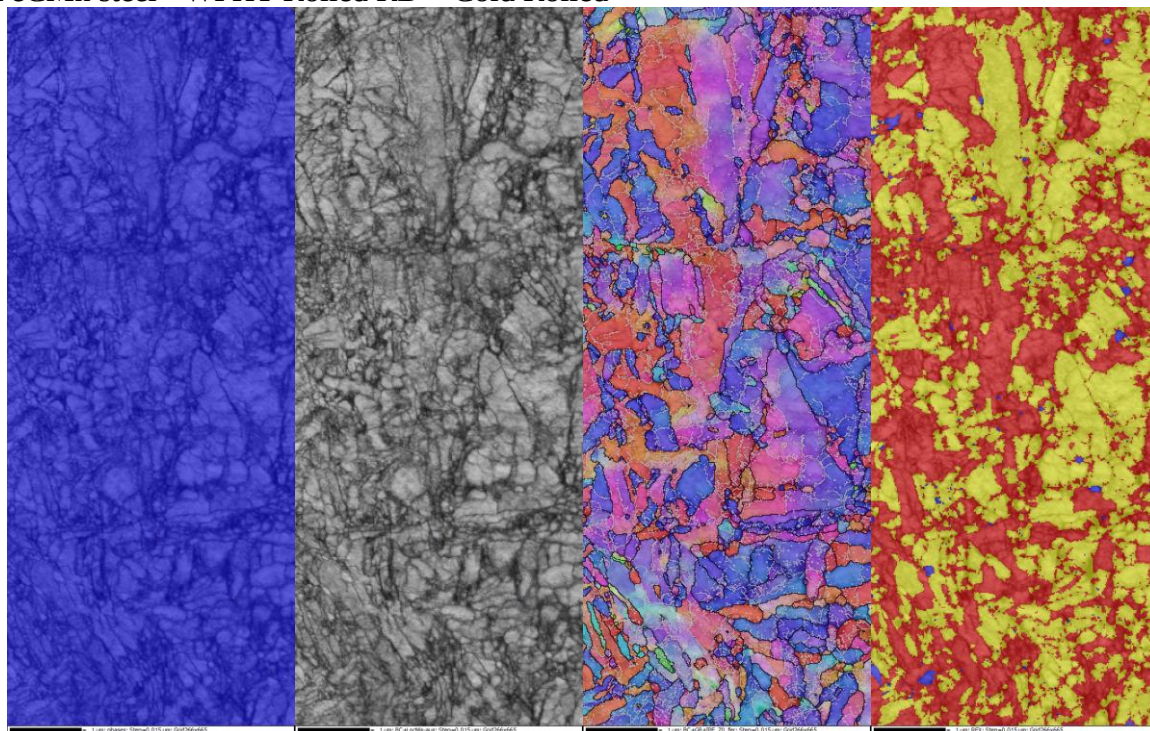


FeCMn steel - 1.4 mm - 620 °C, 10 min

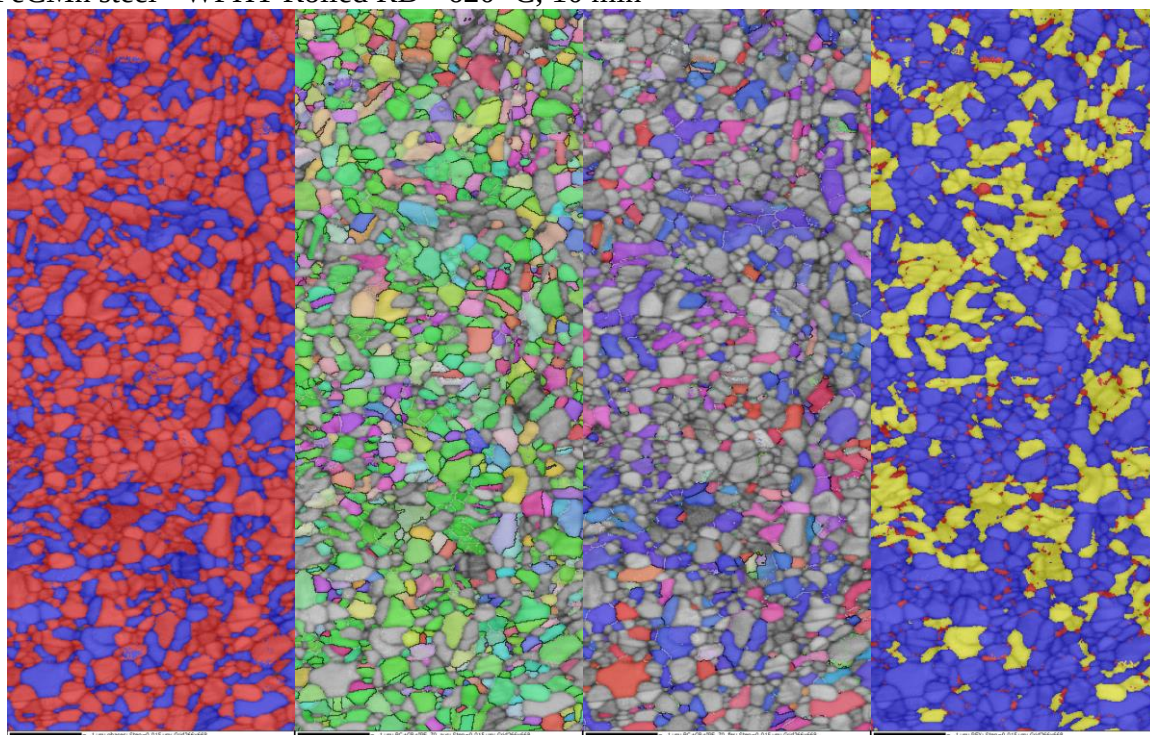


Appendix 1: Scanning Electron Microscopy Maps of Specimens

FeCMn steel - WPHT Rolled RD – Cold Rolled

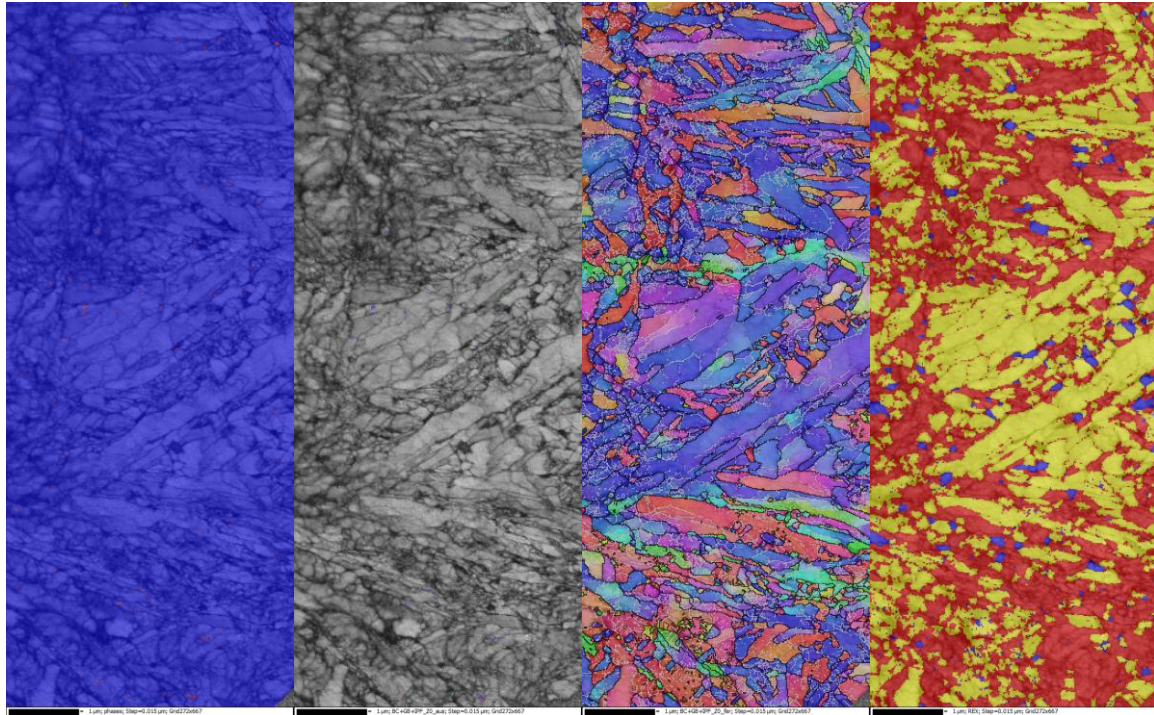


FeCMn steel - WPHT Rolled RD - 620 °C, 10 min

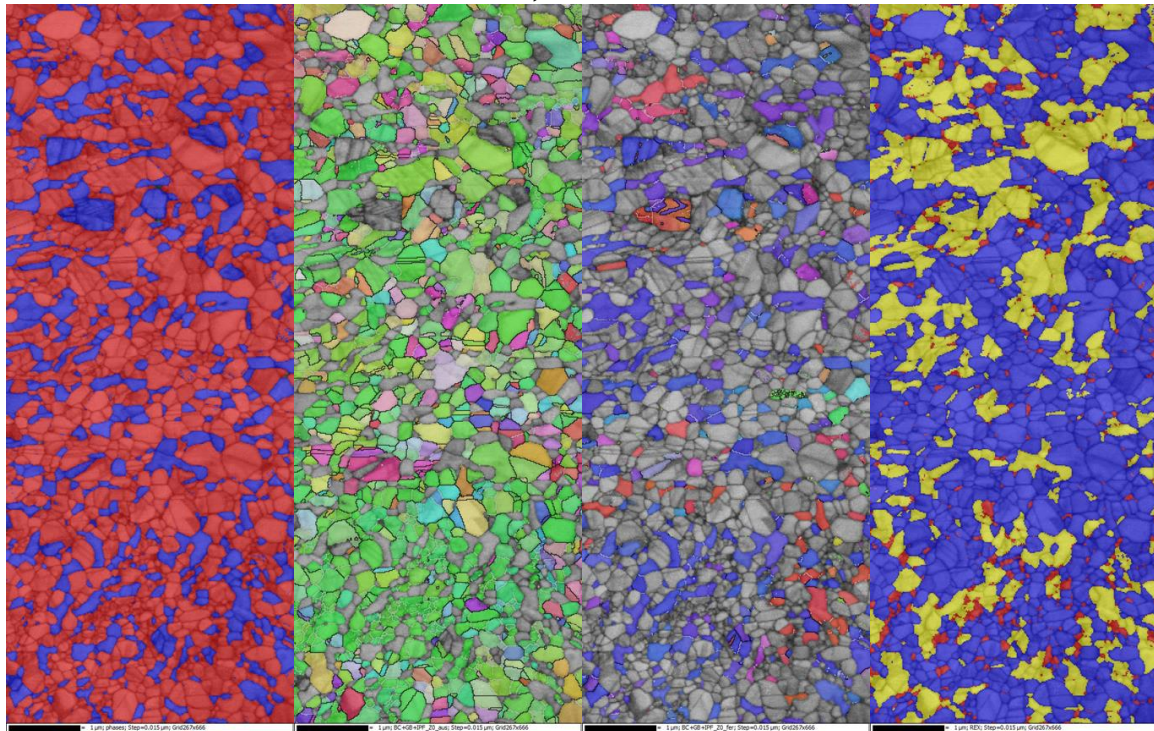


Appendix 1: Scanning Electron Microscopy Maps of Specimens

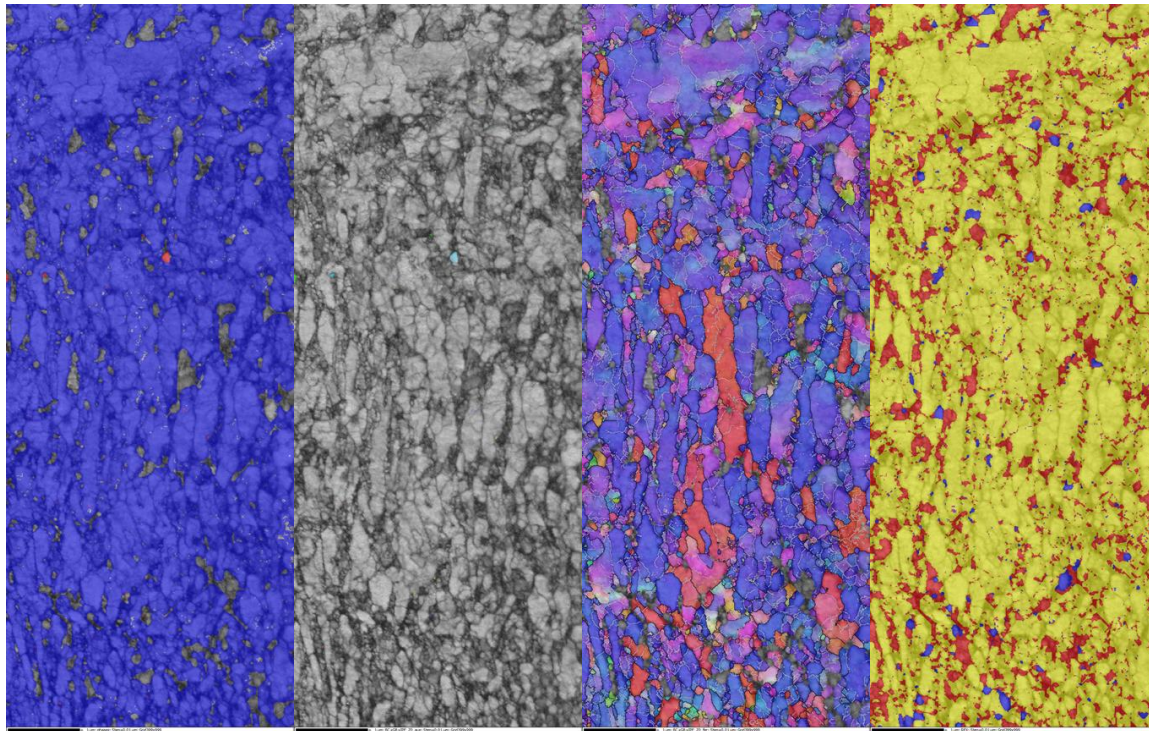
FeCMn steel - WPHT Rolled TD – Cold Rolled



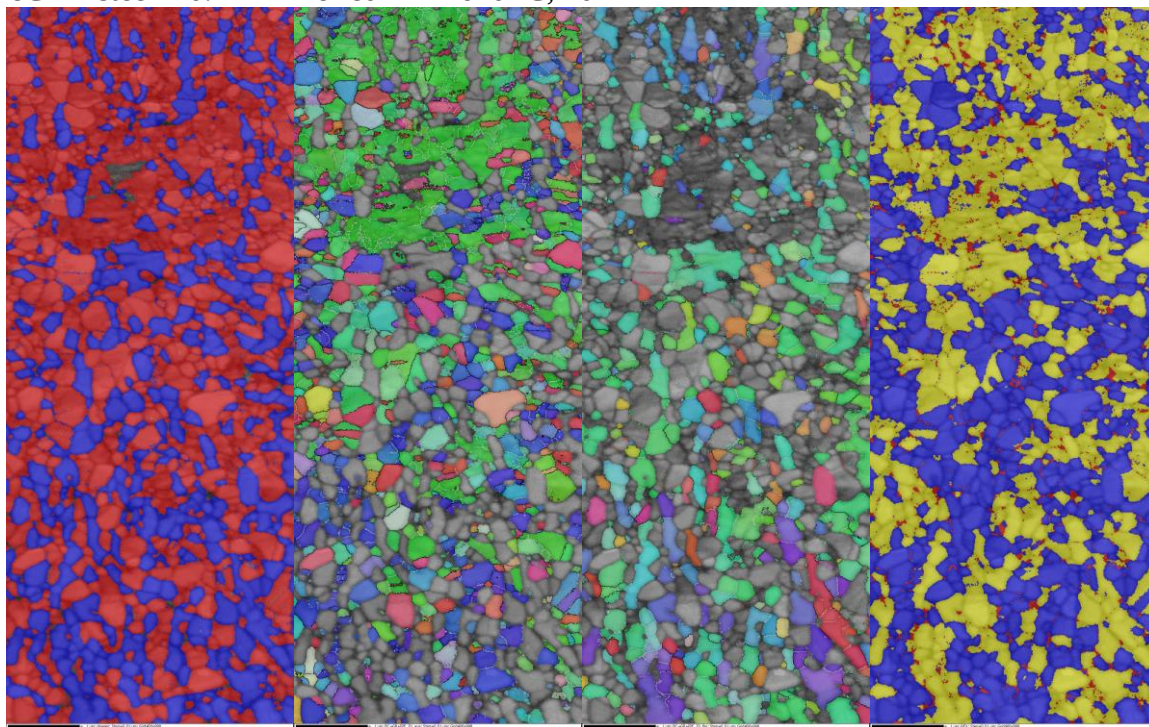
FeCMn steel - WPHT Rolled TD - 620 °C, 10 min



FeCMn steel – 0.7 mm Rolled RD – Cold Rolled

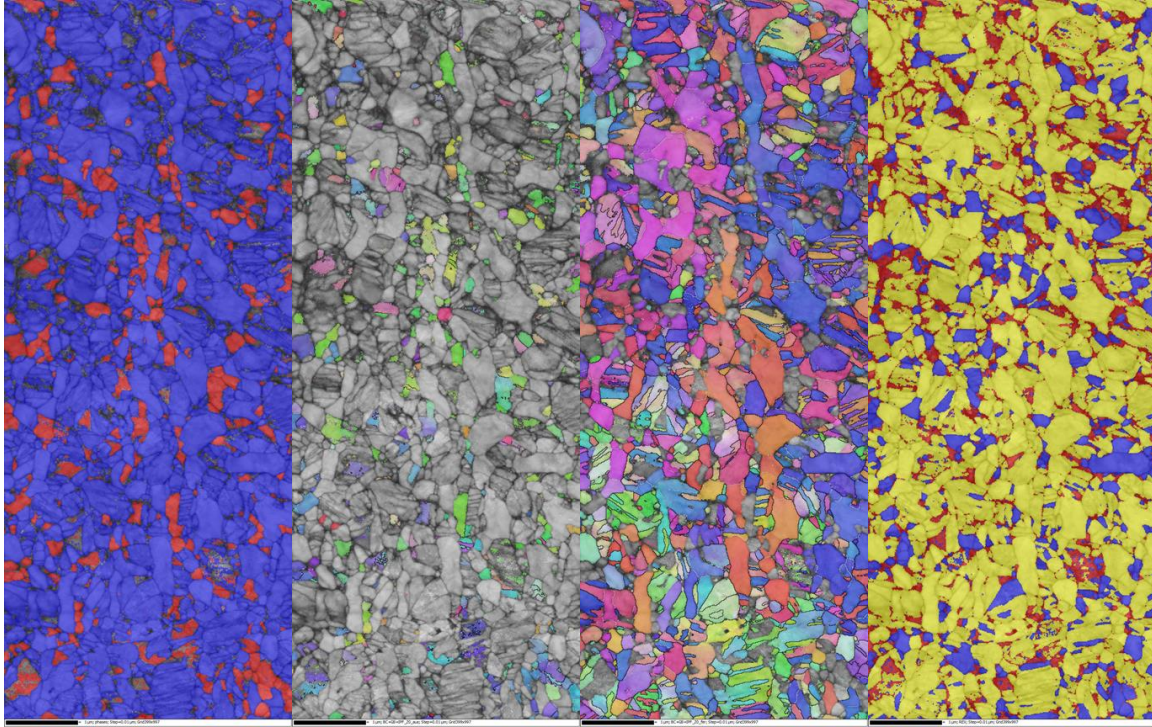


FeCMn steel – 0.7 mm Rolled RD - 620 °C, 10 min

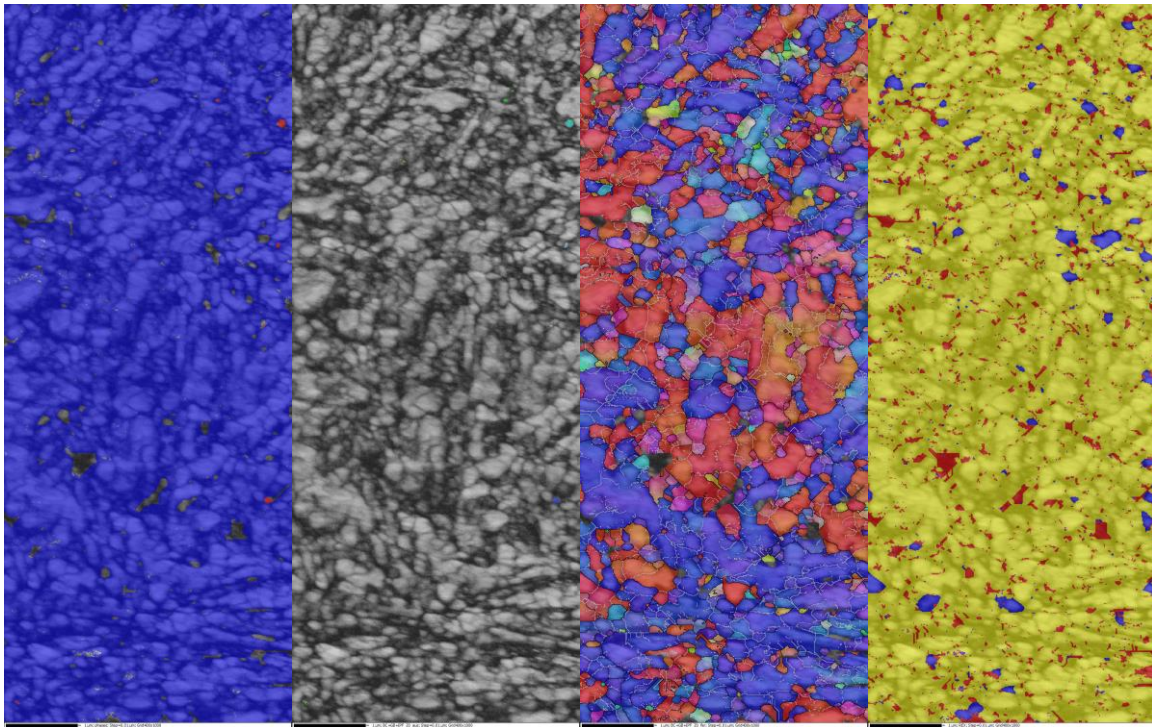


Appendix 1: Scanning Electron Microscopy Maps of Specimens

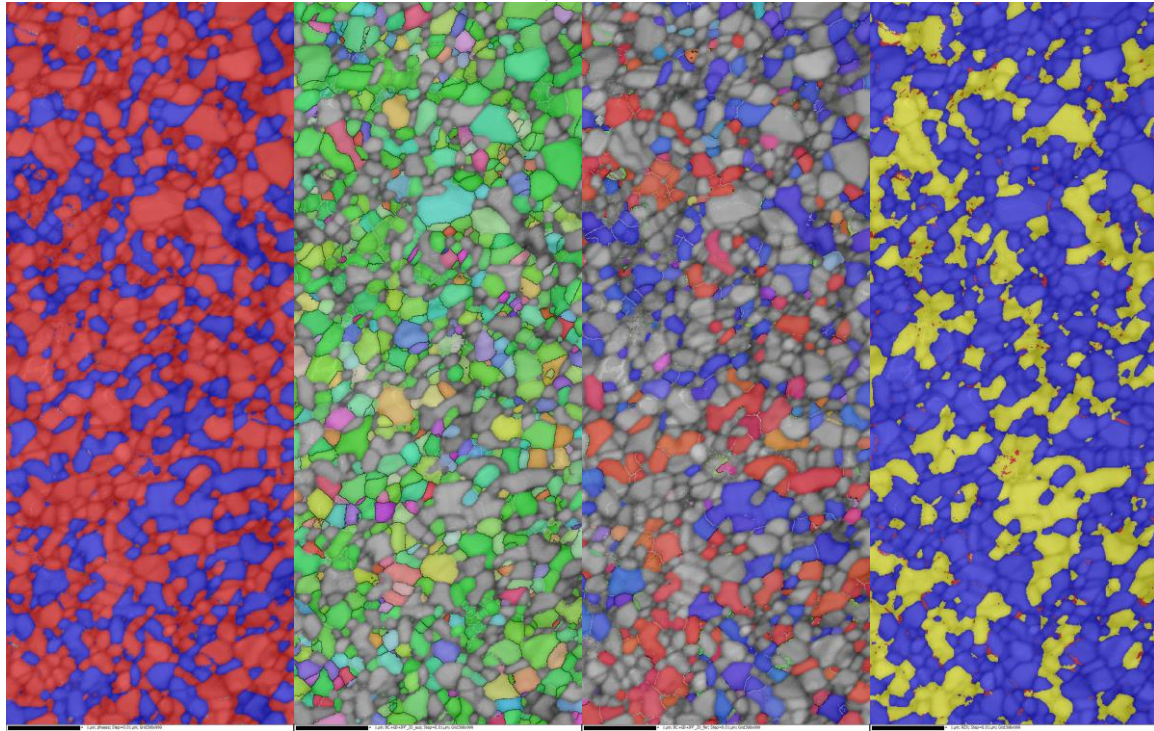
FeCMn steel – 0.7 mm Rolled RD - 650 °C, 10 min



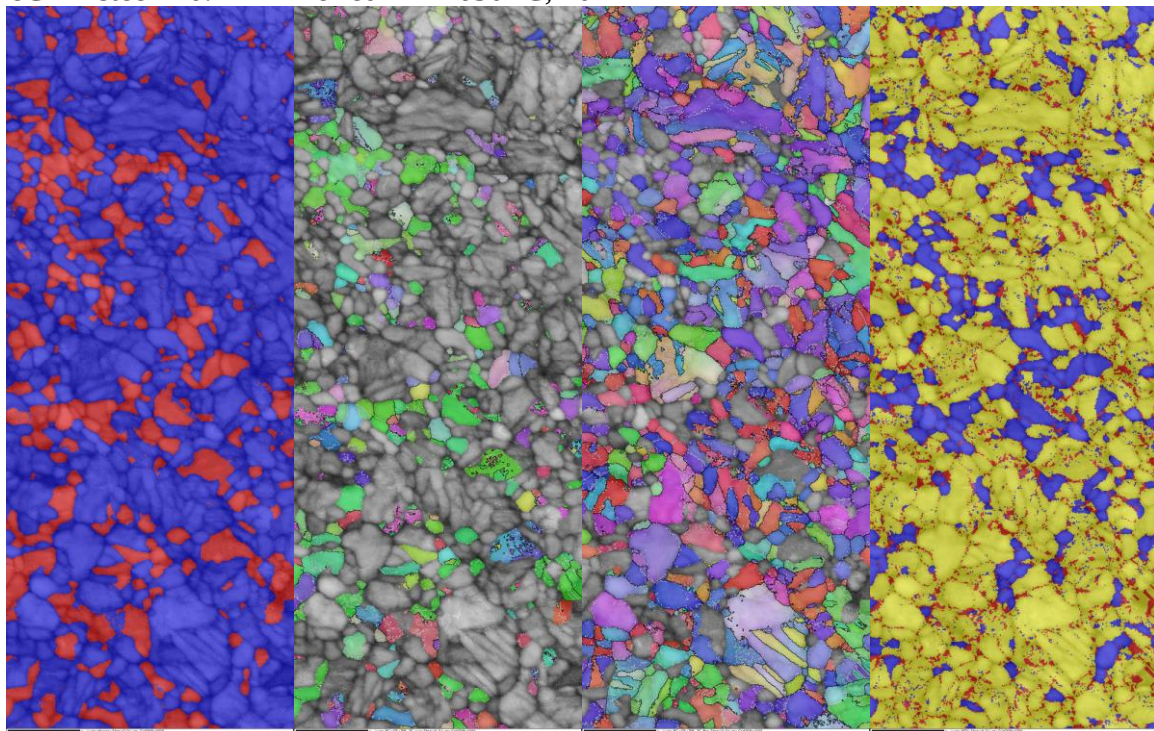
FeCMn steel – 0.7 mm Rolled TD – Cold Rolled



FeCMn steel – 0.7 mm Rolled TD - 620 °C, 10 min

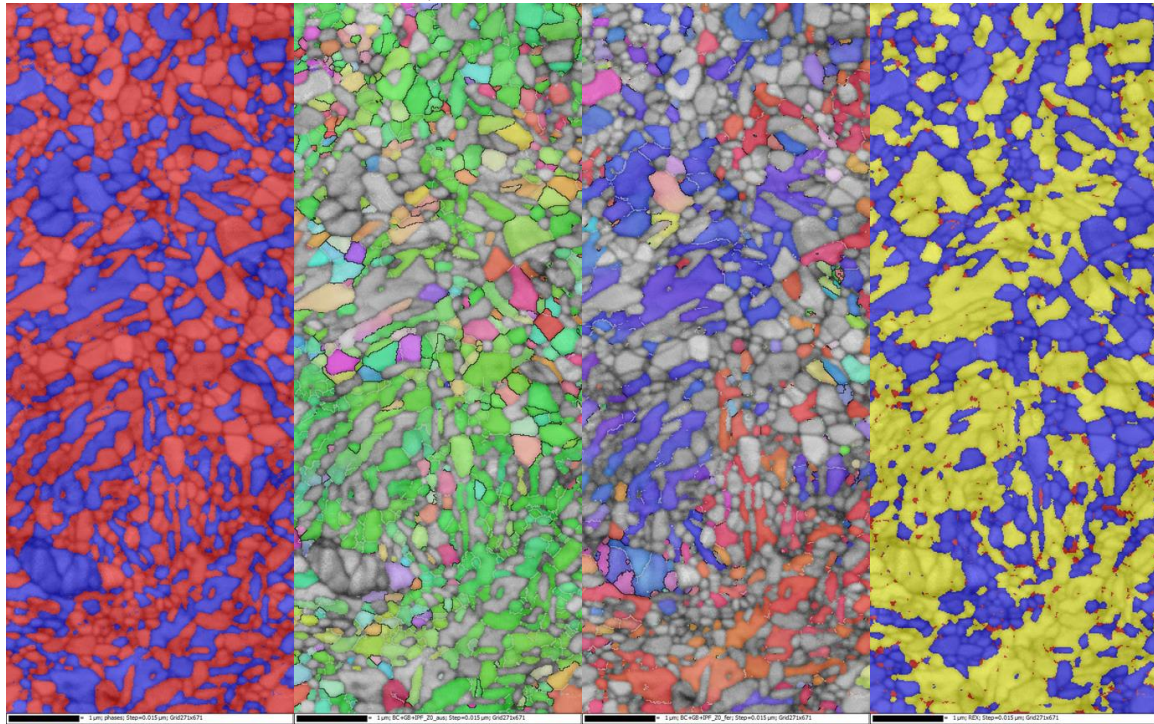


FeCMn steel – 0.7 mm Rolled TD - 650 °C, 10 min

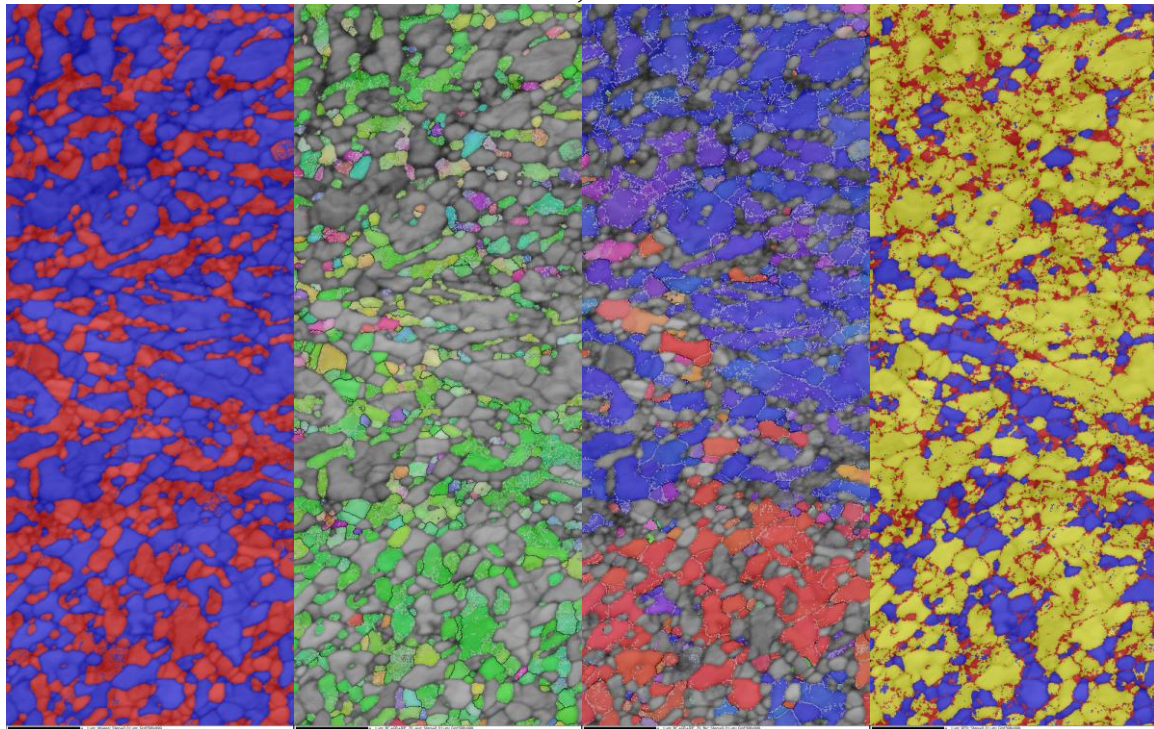


A1.2 FeCMnAl Steel Specimens

FeCMnAl steel - 1.8 mm - 650 °C, 10 min

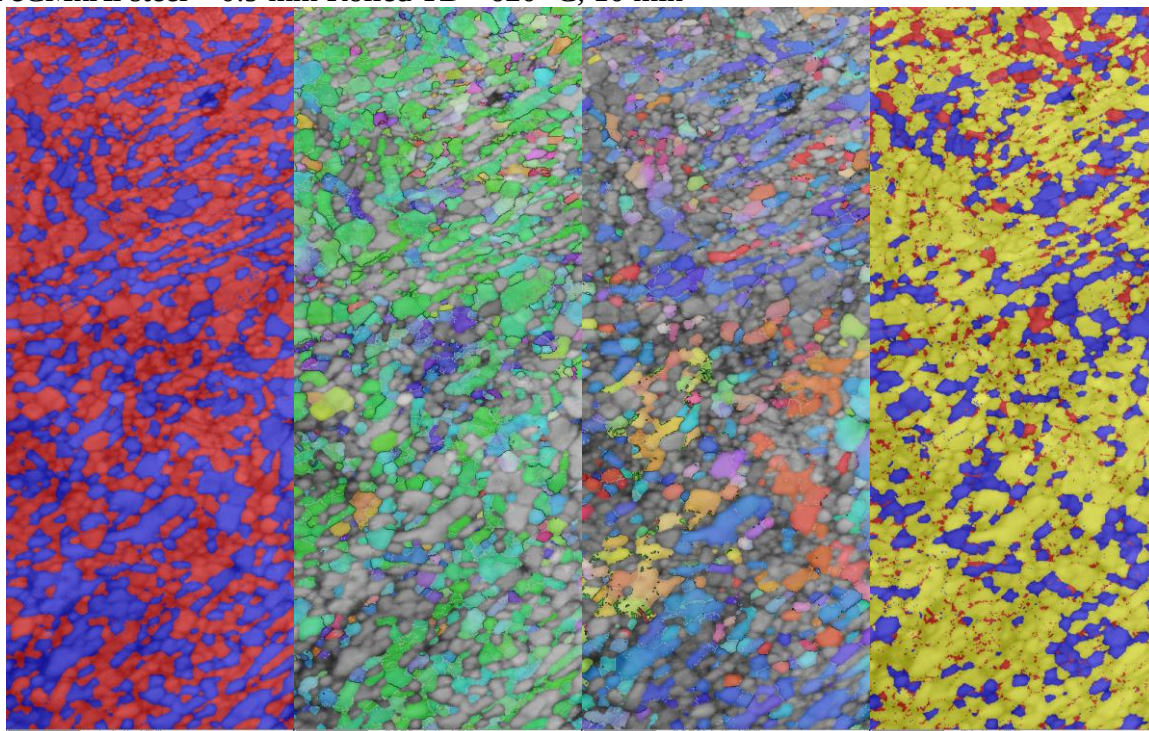


FeCMnAl steel – 0.9 mm Rolled TD - 620 °C, 5 min

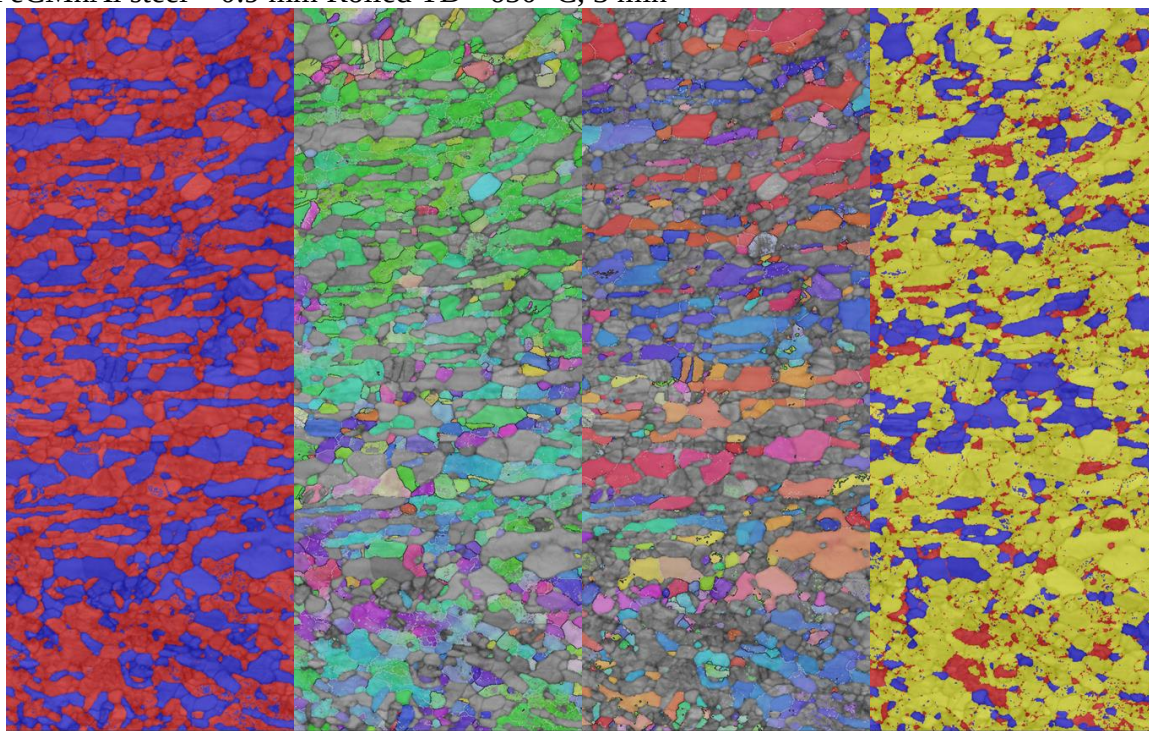


Appendix 1: Scanning Electron Microscopy Maps of Specimens

FeCMnAl steel – 0.9 mm Rolled TD - 620 °C, 10 min

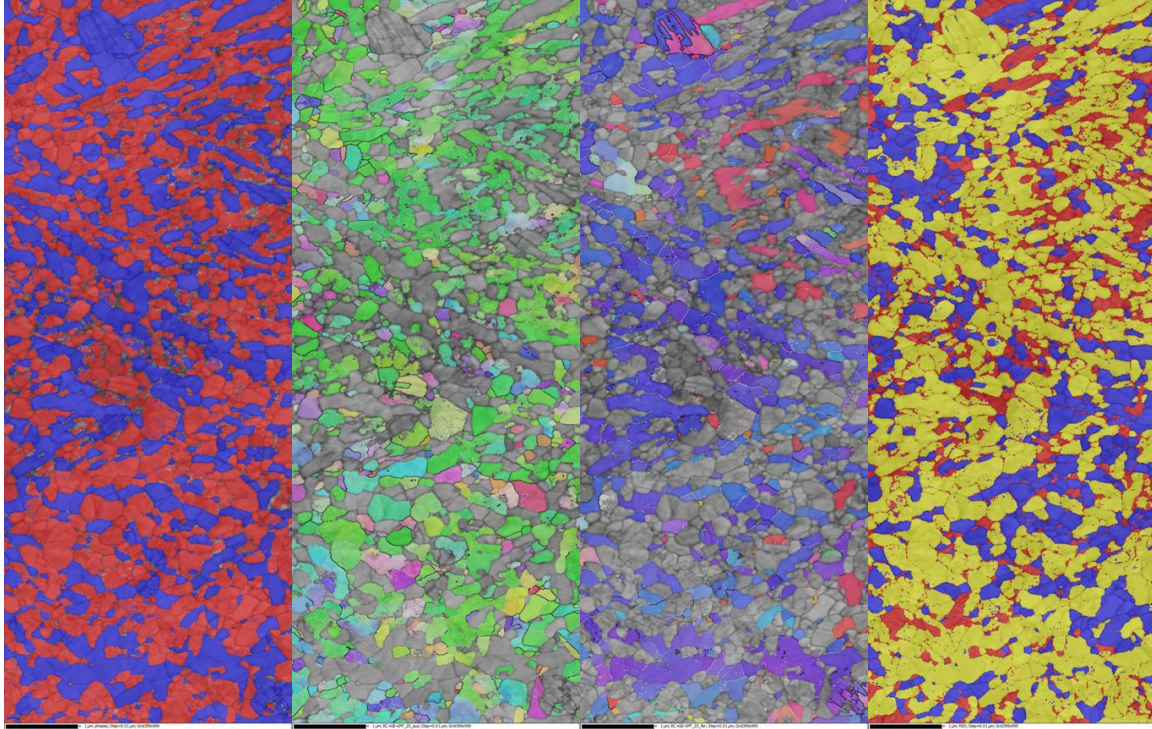


FeCMnAl steel – 0.9 mm Rolled TD - 650 °C, 3 min

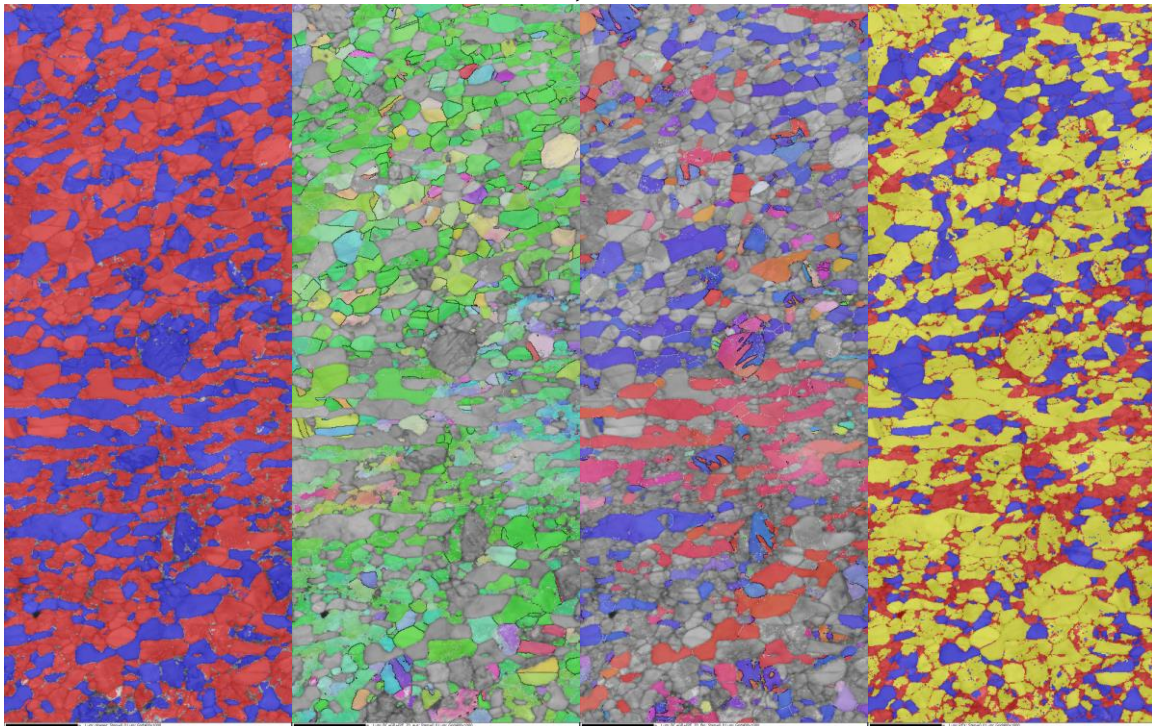


Appendix 1: Scanning Electron Microscopy Maps of Specimens

FeCMnAl steel – 0.9 mm Rolled TD - 650 °C, 5 min

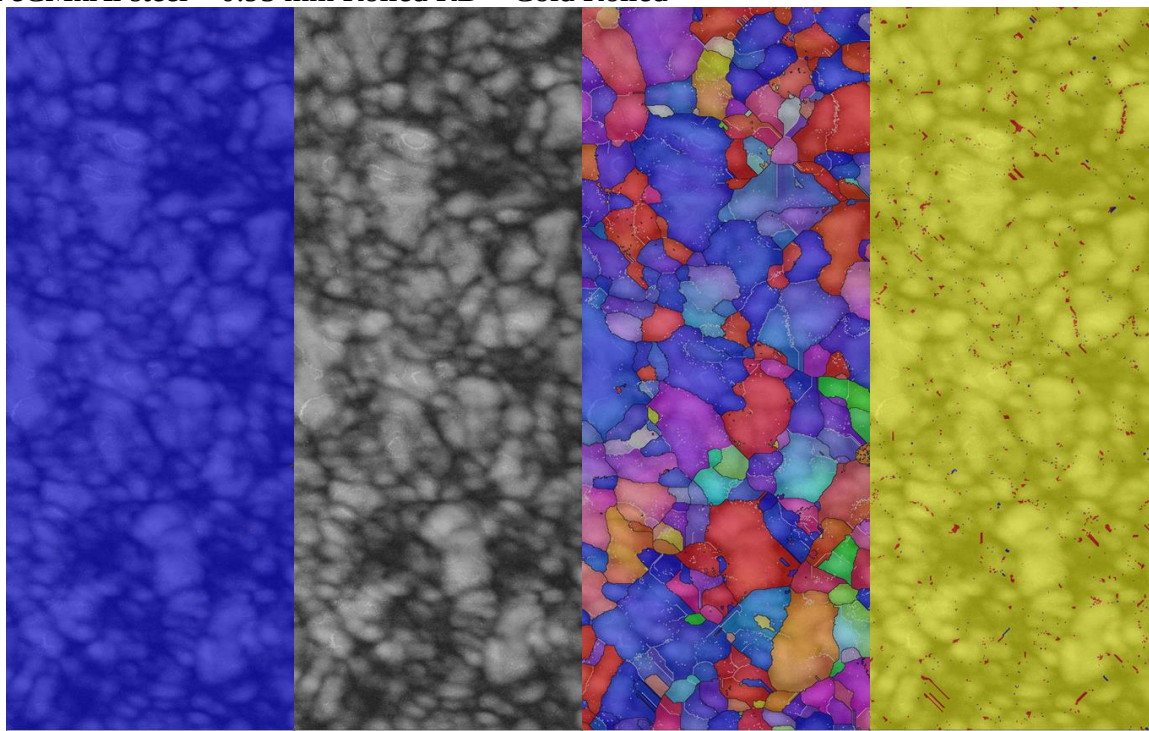


FeCMnAl steel – 0.9 mm Rolled TD - 650 °C, 10 min

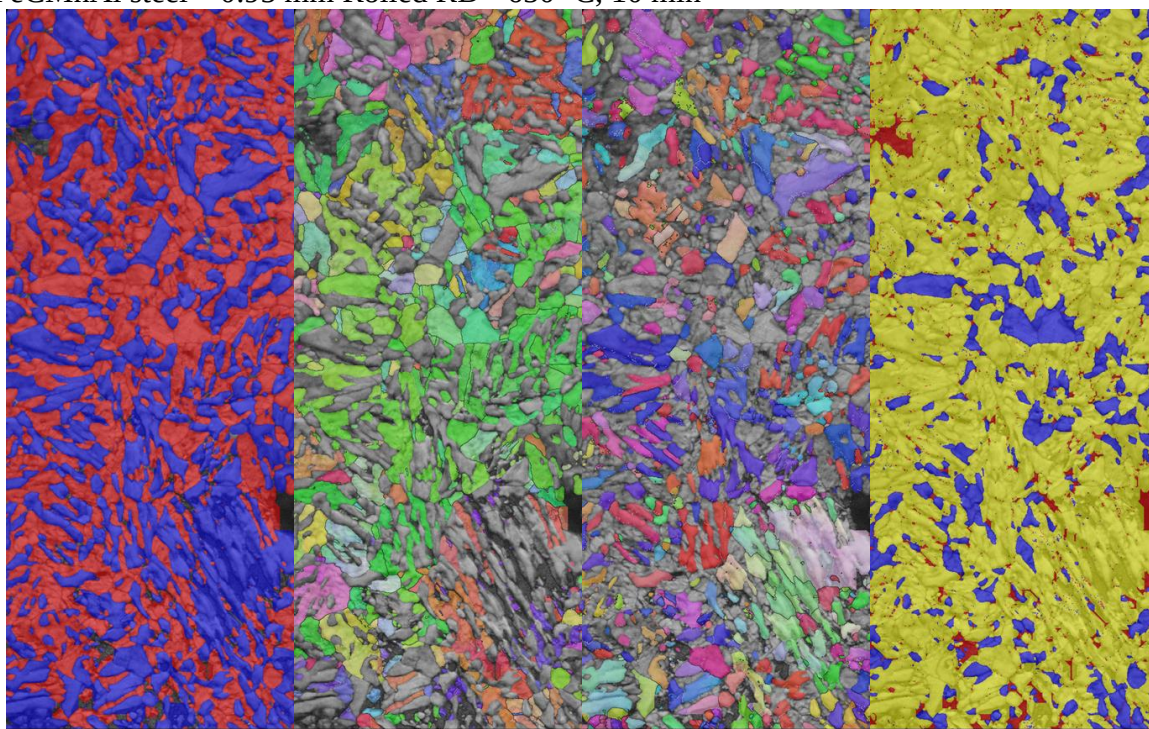


Appendix 1: Scanning Electron Microscopy Maps of Specimens

FeCMnAl steel – 0.95 mm Rolled RD – Cold Rolled



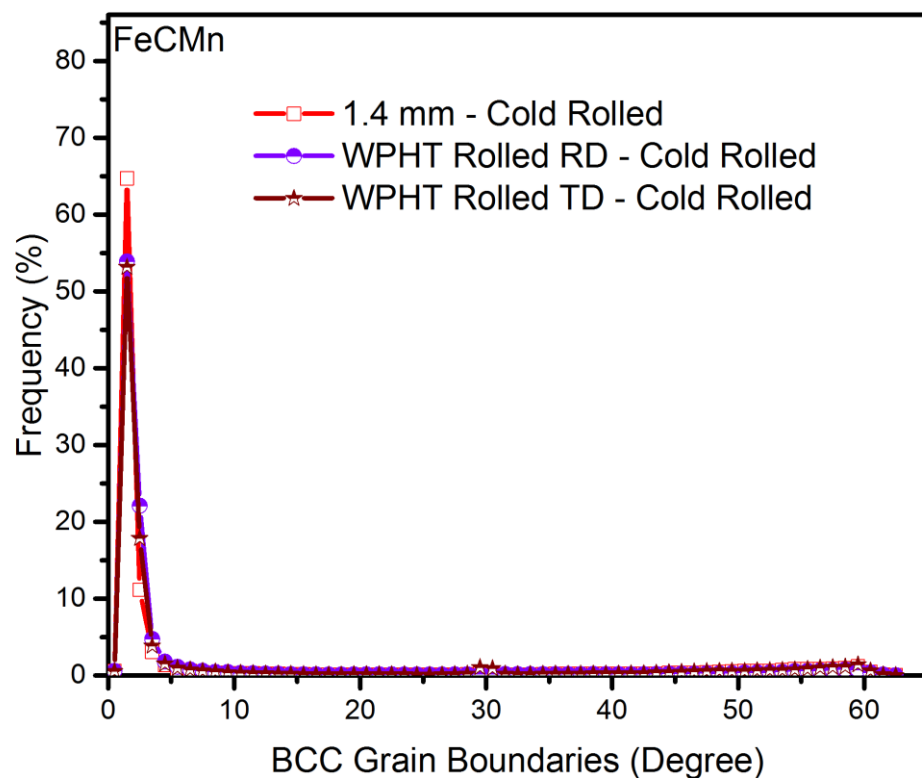
FeCMnAl steel – 0.95 mm Rolled RD - 650 °C, 10 min



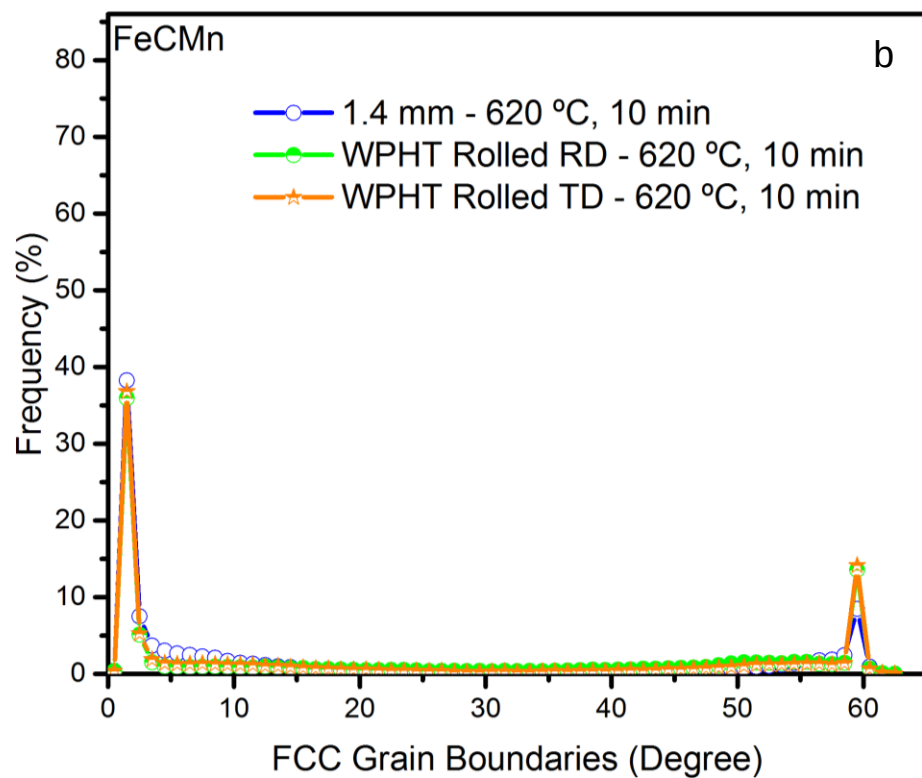
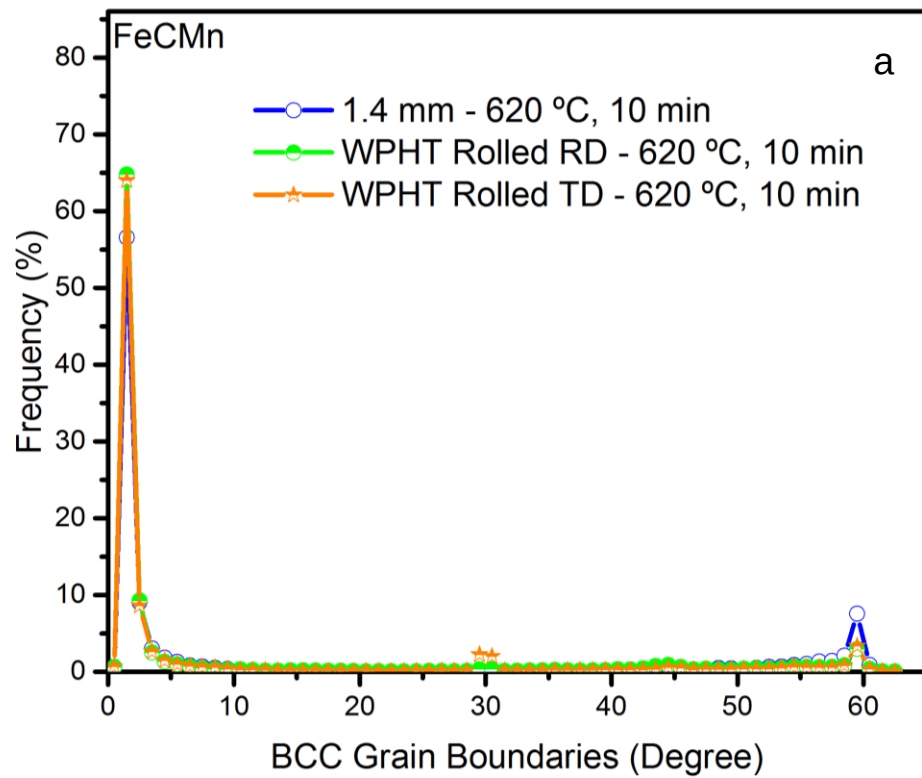
Appendix 2 : Grain Boundaries Distribution, Local Misorientation Distribution and Kernel Average Misorientation Maps

A2.1 : FeCMn Steel

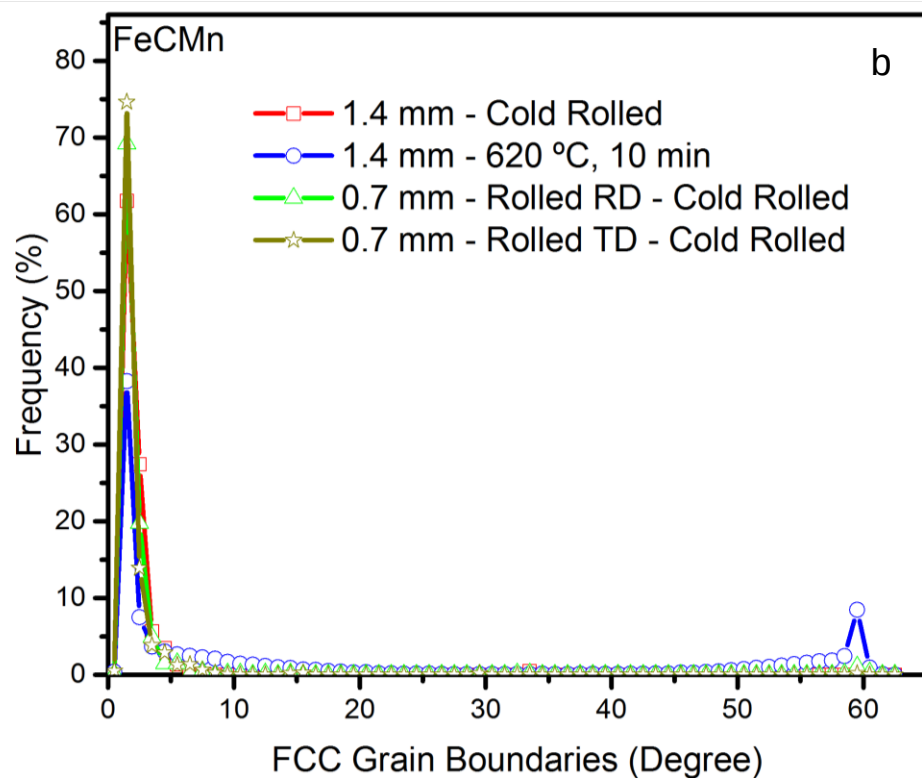
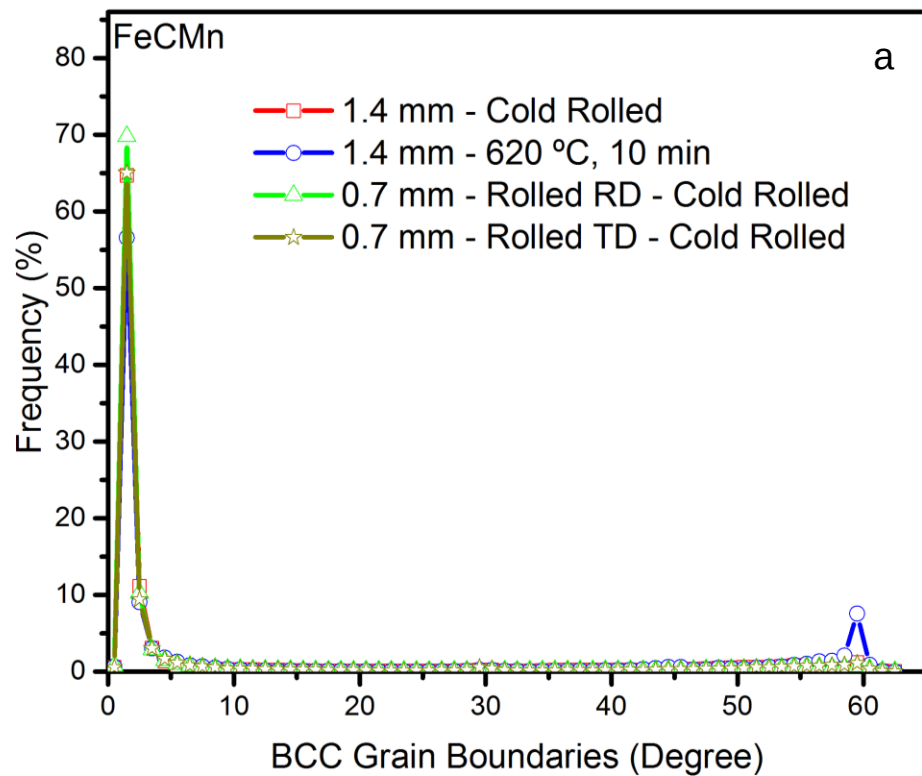
A2.1.1 : Grain Boundaries Distribution of the FeCMn steel Specimens



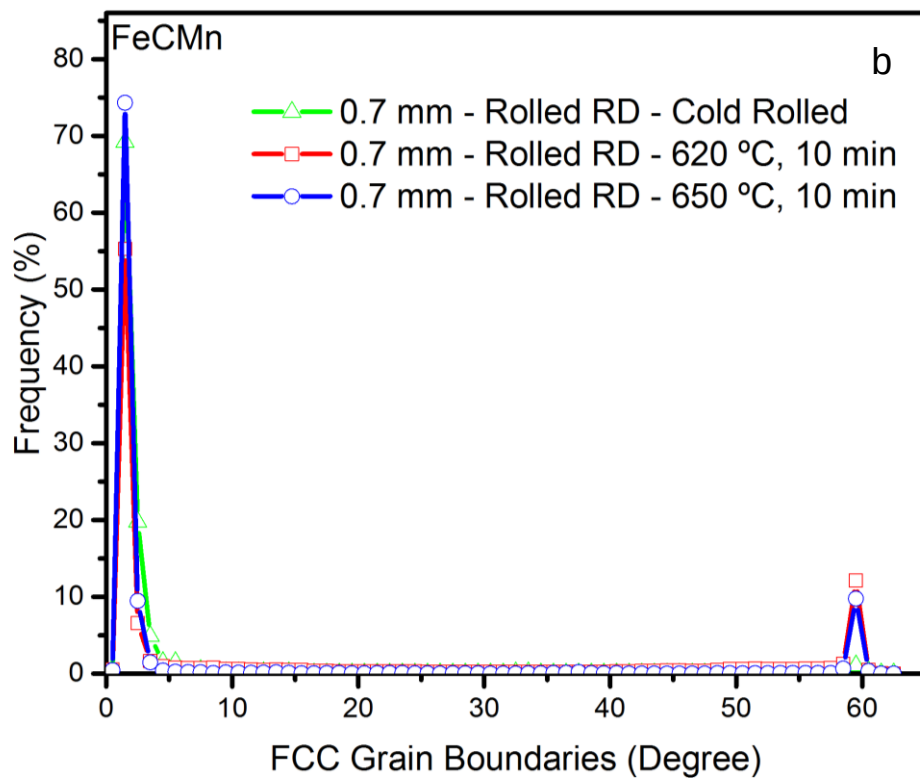
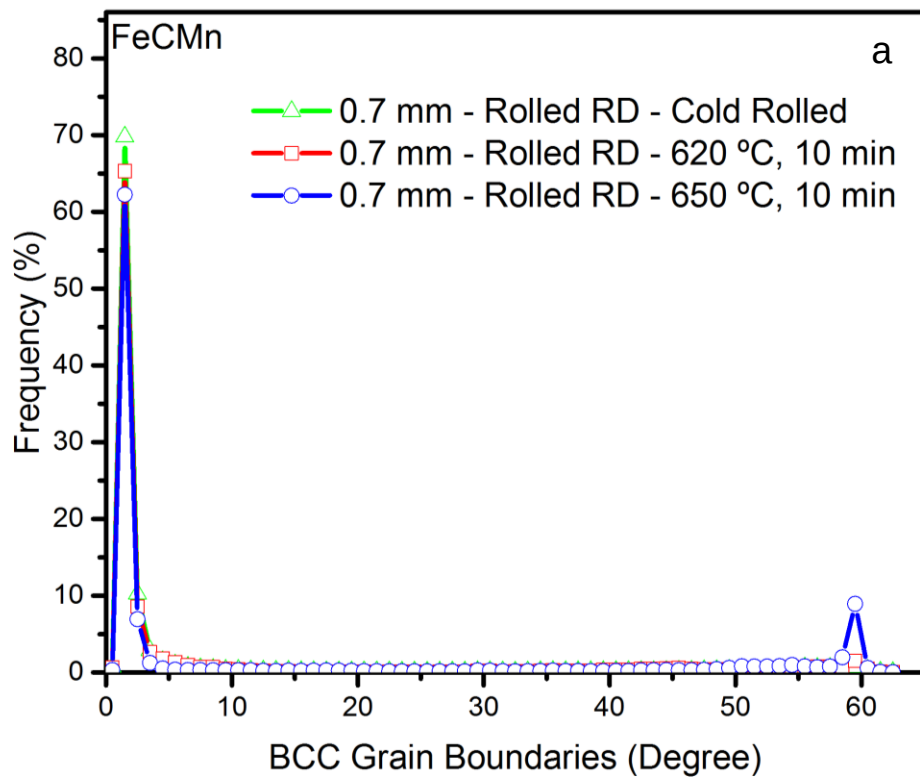
Histogram distribution of the α/α' phase grain boundaries in the FeCMn steel, effect of the cold rolling WPHT



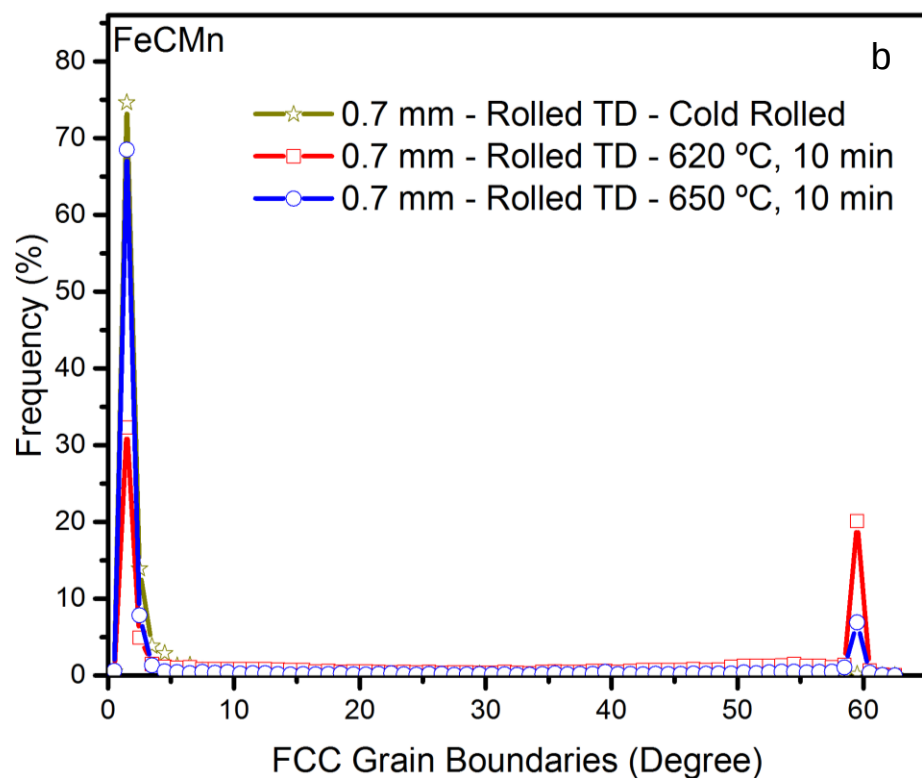
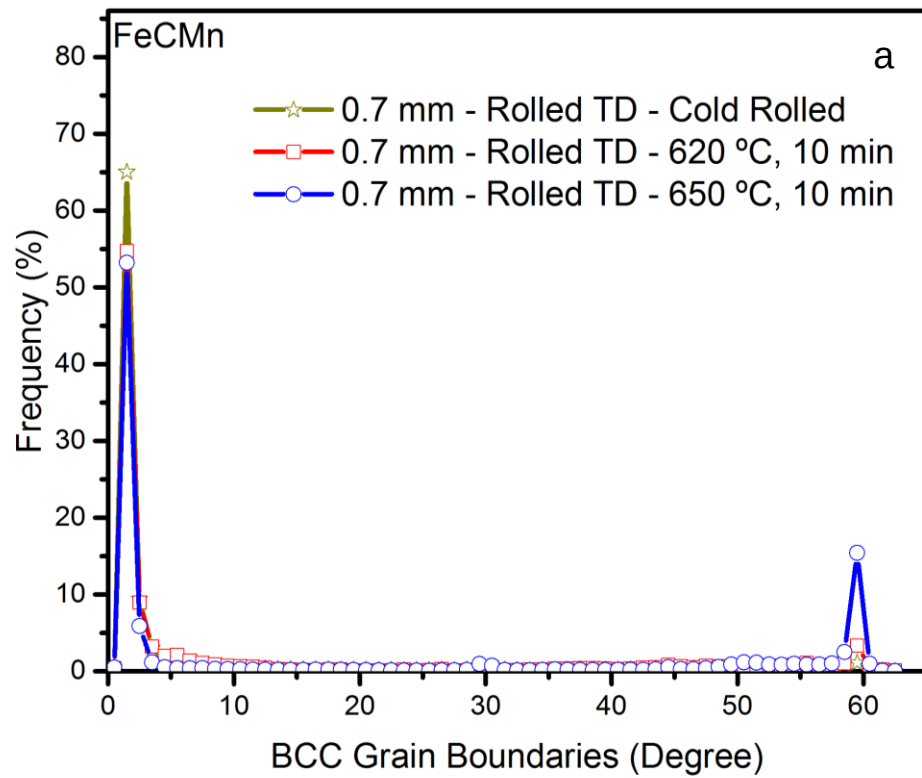
Histogram distribution of the (a) α/α' and (b) γ grain boundaries in the FeCMn steel, effect of the IA at 620 °C for 10 min on the WPHT cold rolled samples



Histogram distribution of the (a) α/α' and (b) γ grain boundaries in the FeCMn steel, effect of the first annealing at 620 °C for 10 min and the following cold rolling

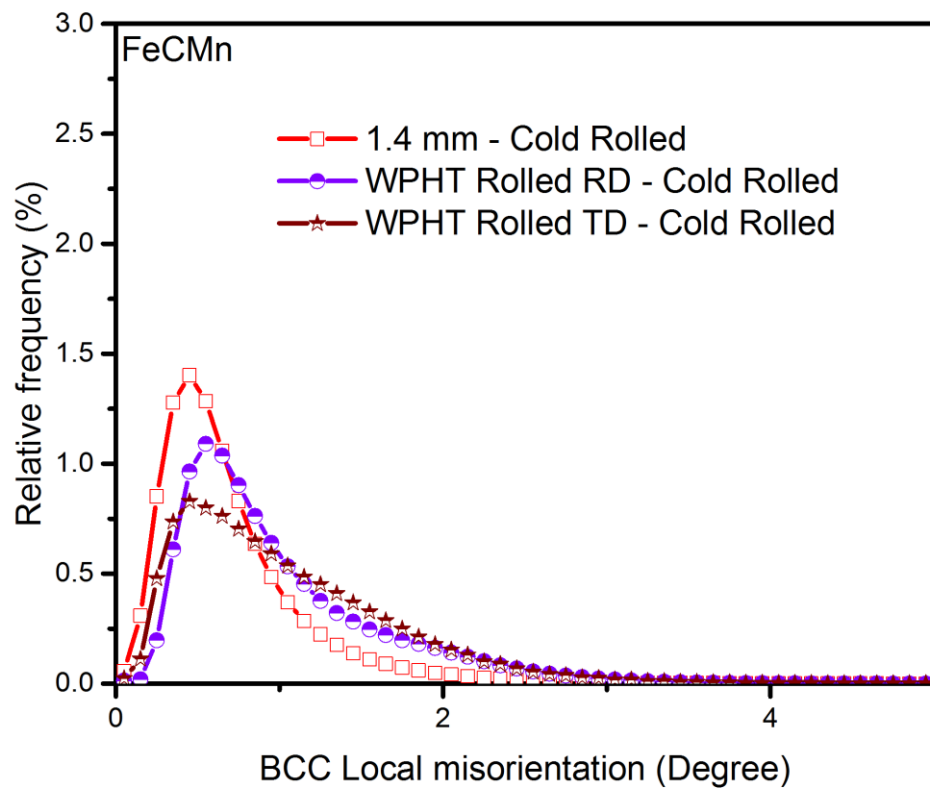


Histogram distribution of the (a) α/α' and (b) γ grain boundaries in the FeCMn steel, effect of the second annealing on the RD cold rolled samples

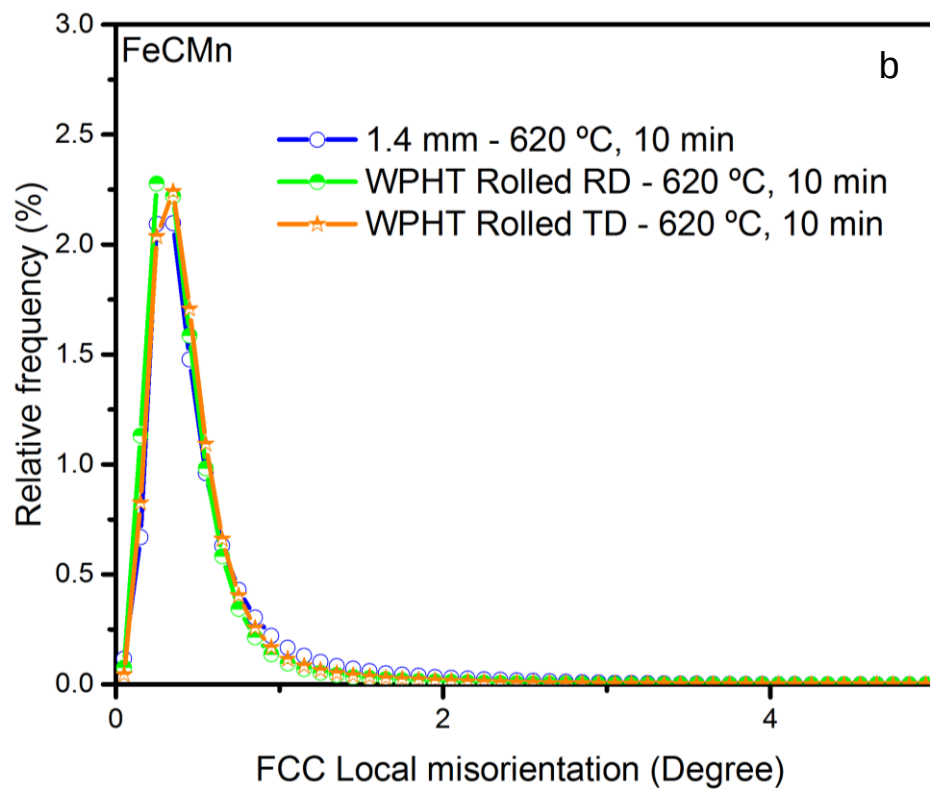
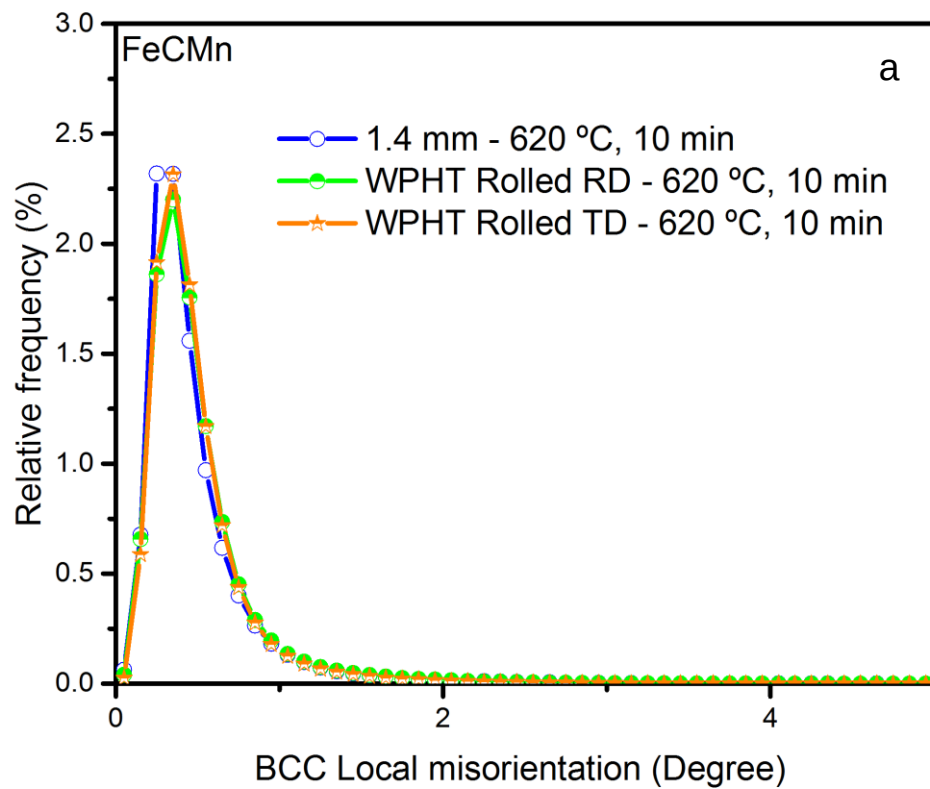


Histogram distribution of the (a) α/α' and (b) γ grain boundaries in the FeCMn steel, effect of the second annealing on the TD cold rolled samples

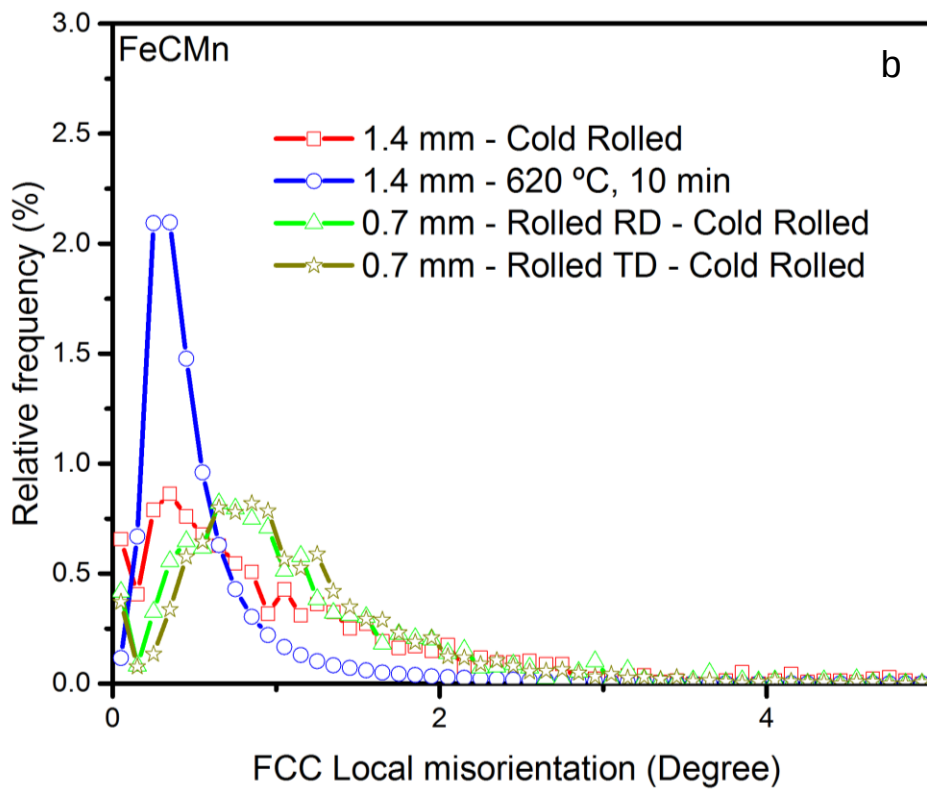
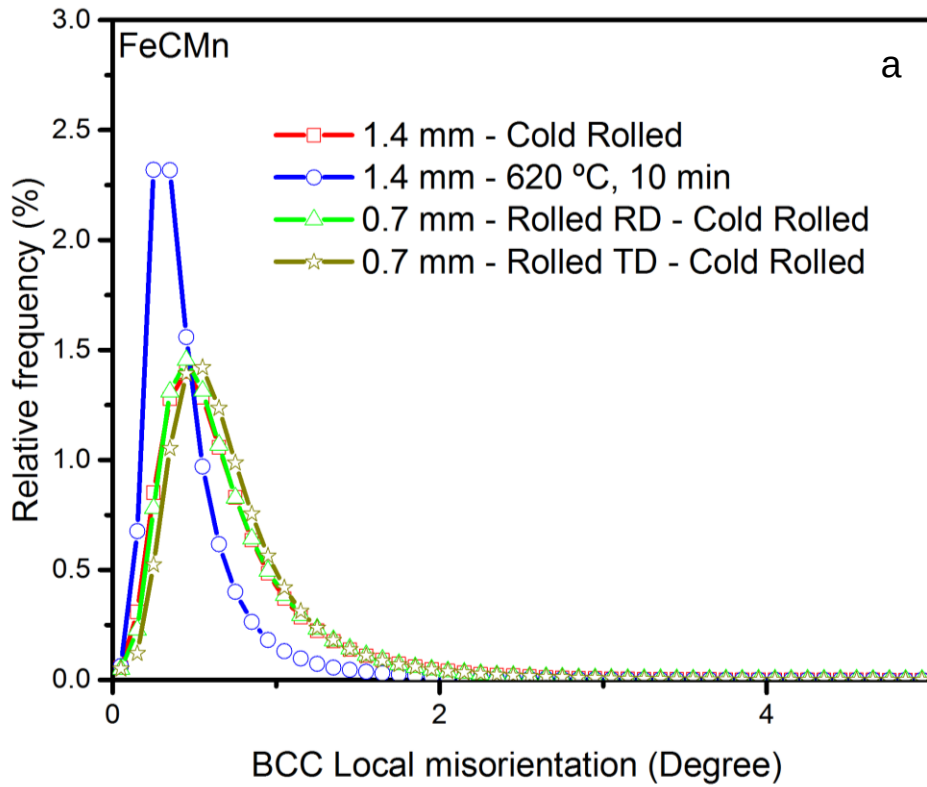
A2.1.2 : Kernel Average Misorientation Histogram Distributions and the Maps of the FeCMn Steel



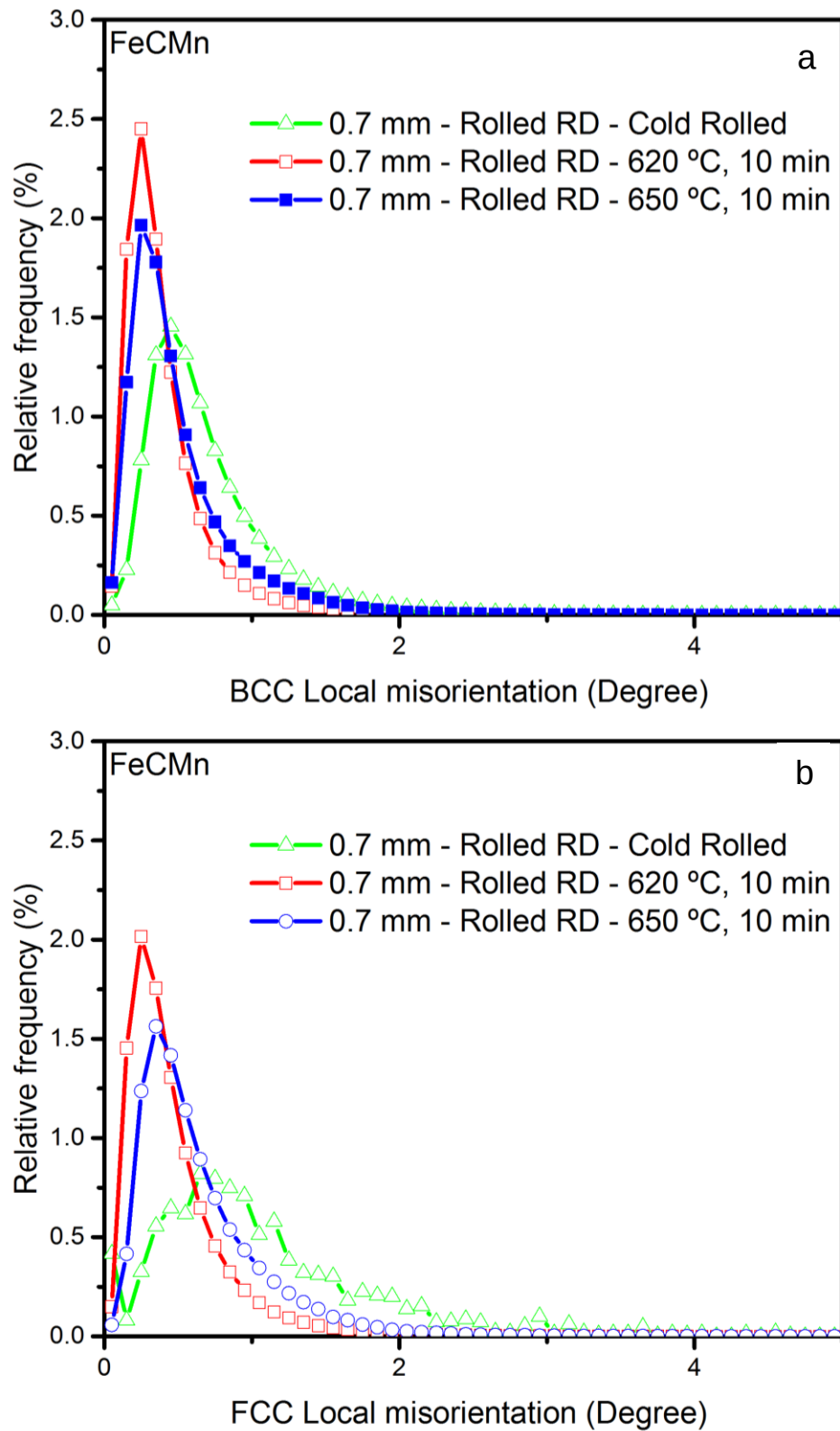
Histogram distribution of the α/α' local misorientation in the FeCMn steel, effect of the cold rolling WPHT



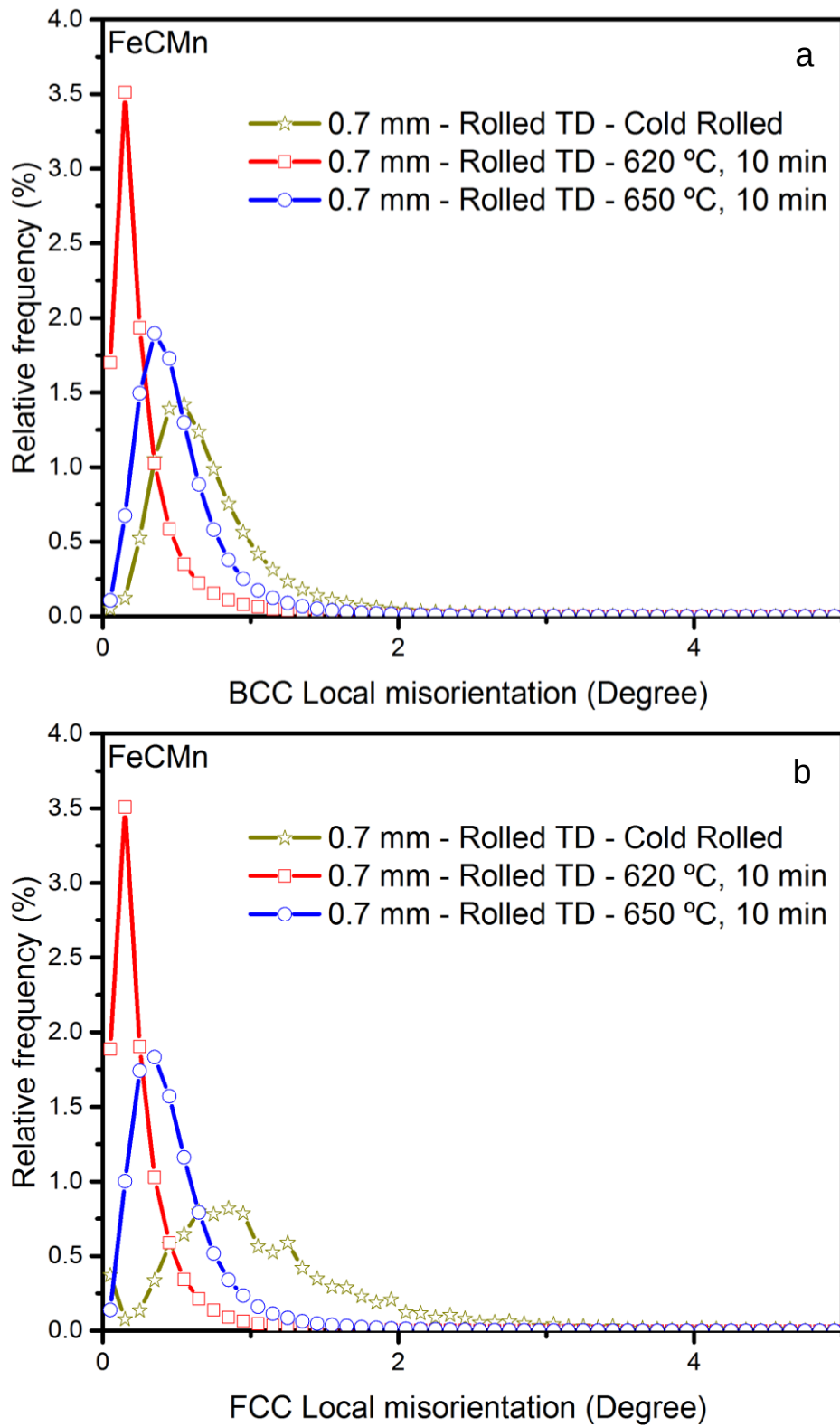
Histogram distribution of the (a) α/α' and (b) γ local misorientation in the FeCMn steel, effect of the IA on the WPHT cold rolled samples



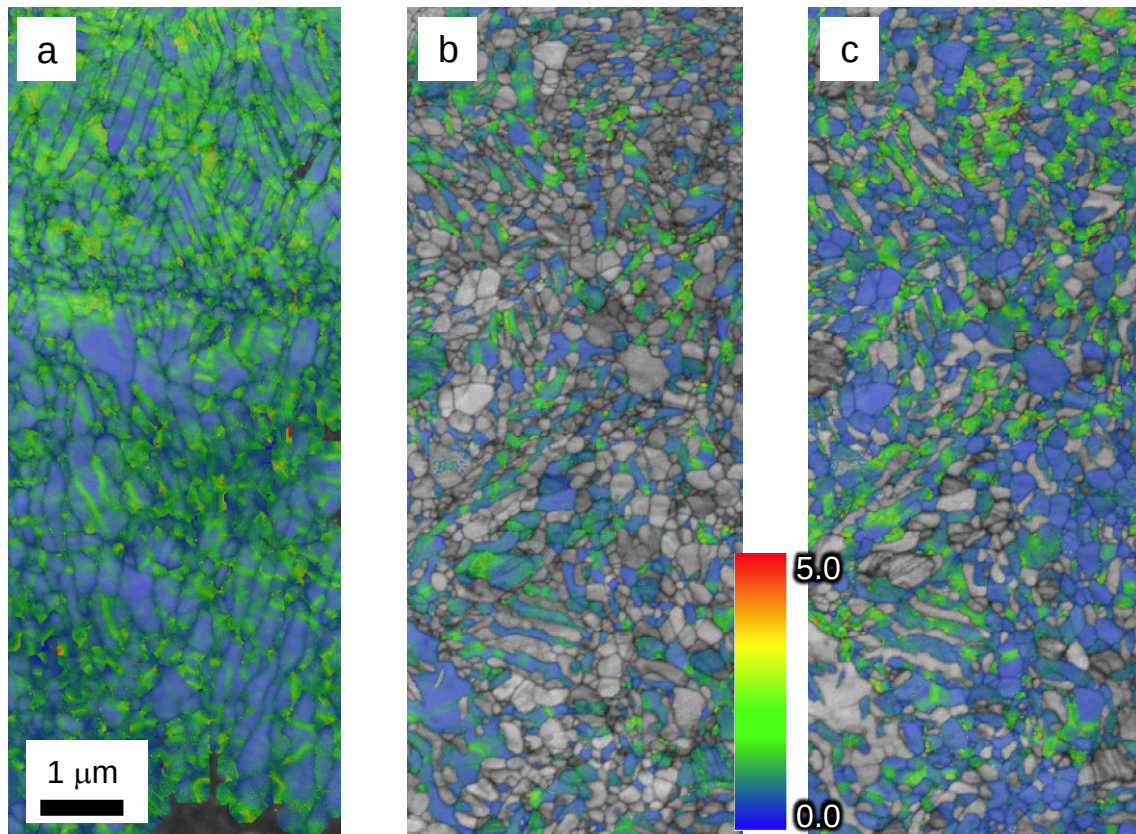
Histogram distribution of the (a) α/α' and (b) γ local misorientation in the FeCMn steel, effect of the first annealing at 620 °C and the following cold rolling



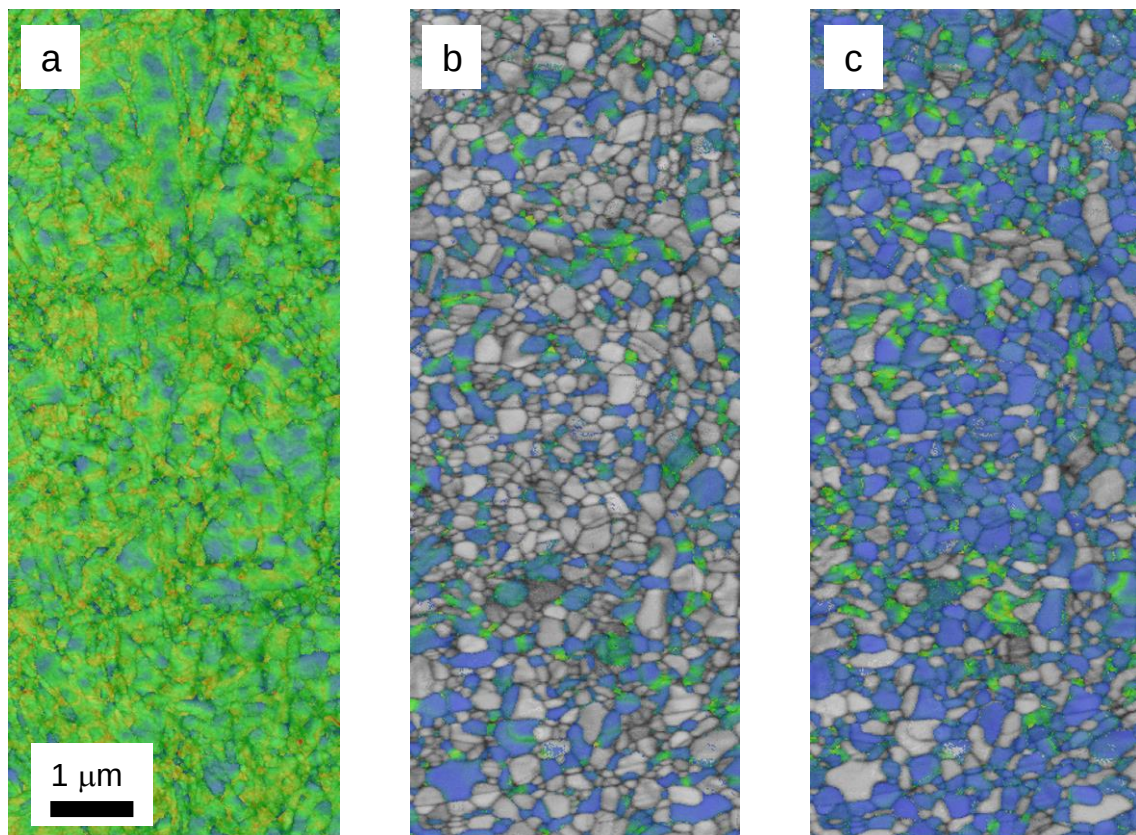
Histogram distribution of the (a) α/α' and (b) γ local misorientation in the FeCMn steel, effect of the second annealing on the RD cold rolled samples



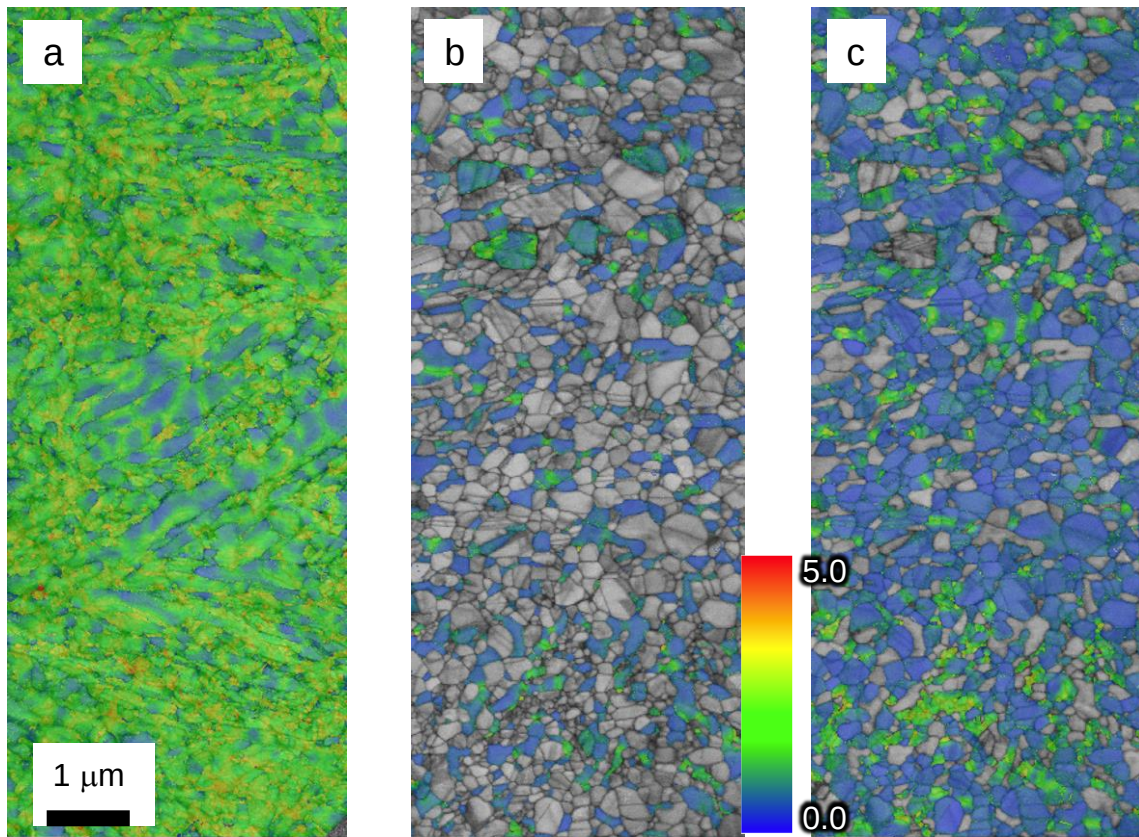
Histogram distribution of the (a) α/α' and (b) γ local misorientation in the FeCMn steel, effect of the second annealing on the TD cold rolled samples



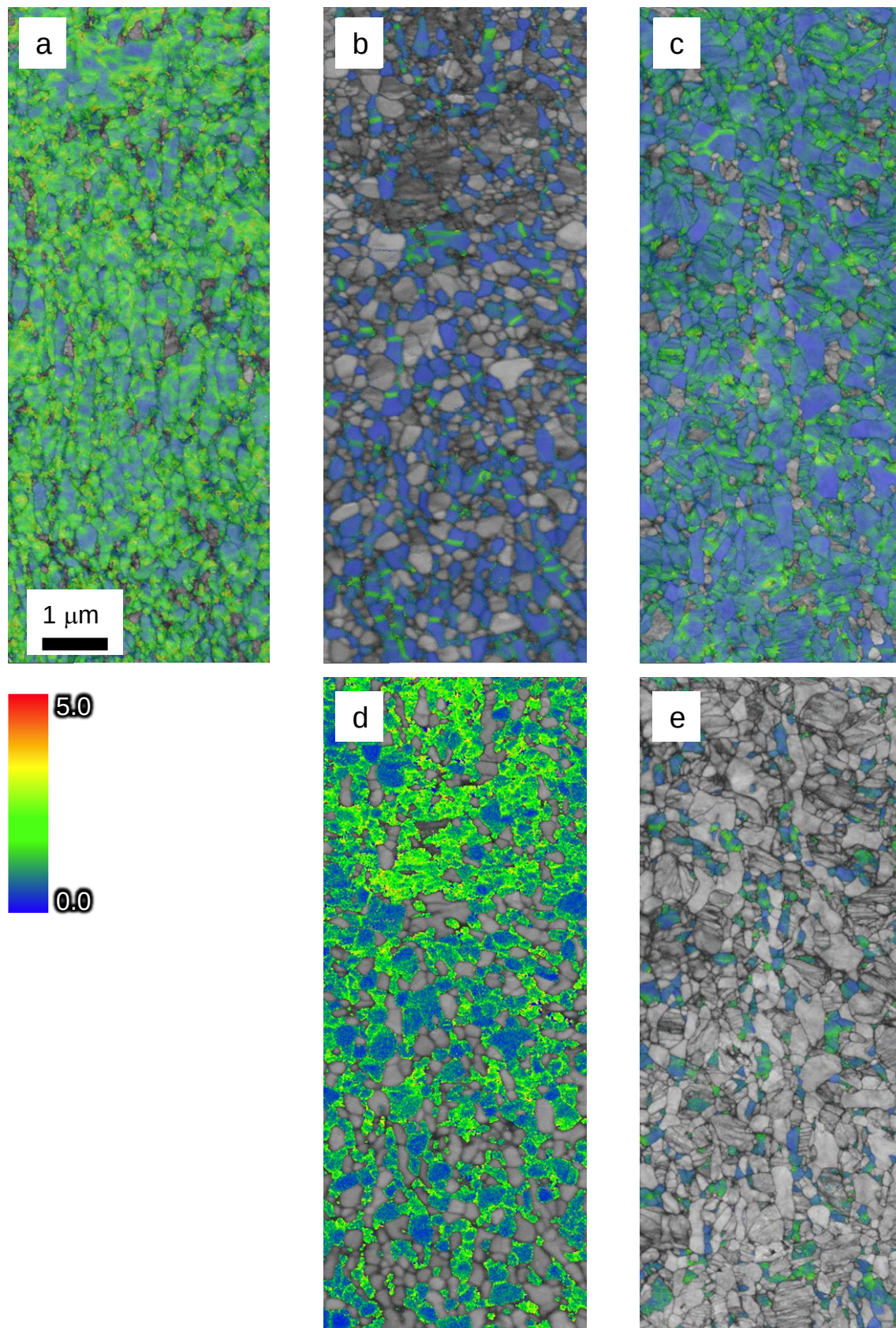
SEM-TKD KAM IQ-maps of the 1.4 mm FeCMn steel (a) as-received α/α' phase. The (b) α/α' and (c) γ phases of the 1.4 mm FeCMn steel after IA at 620 °C for 10 min.



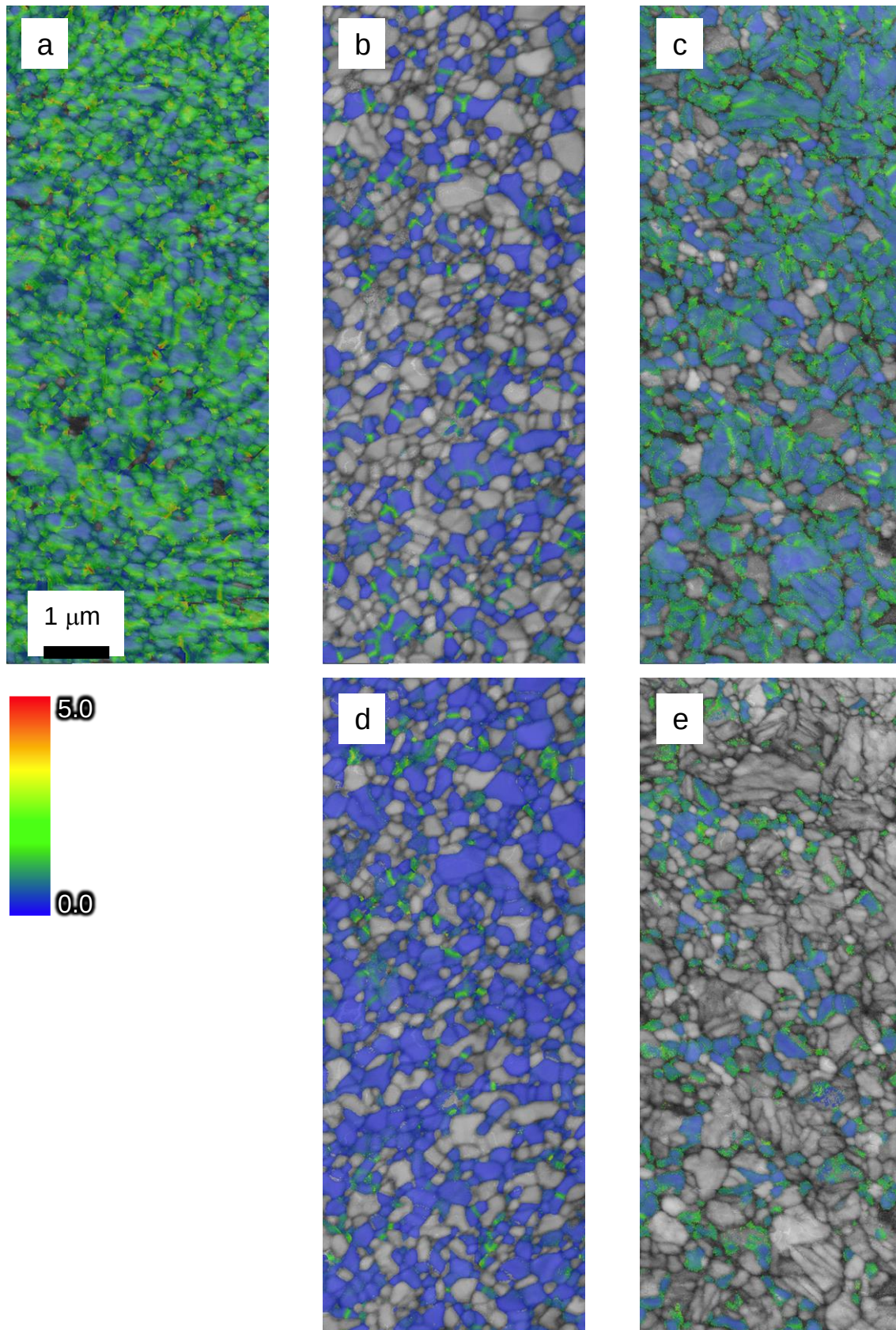
SEM-TKD KAM IQ-maps of the FeCMn steel which cold rolled in the RD direction WPHT (a) the α/α' phase of the cold rolled specimen, (b) the α/α' and (c) the γ phases of the specimen after the IA at 620 °C for 10 min.



SEM-TKD KAM IQ-maps of the FeCMn steel which cold rolled in the TD direction WPHT (a) the α/α' phase of the cold rolled specimen, (b) the α/α' and (c) the γ phases of the specimen after the IA at 620 °C for 10 min.



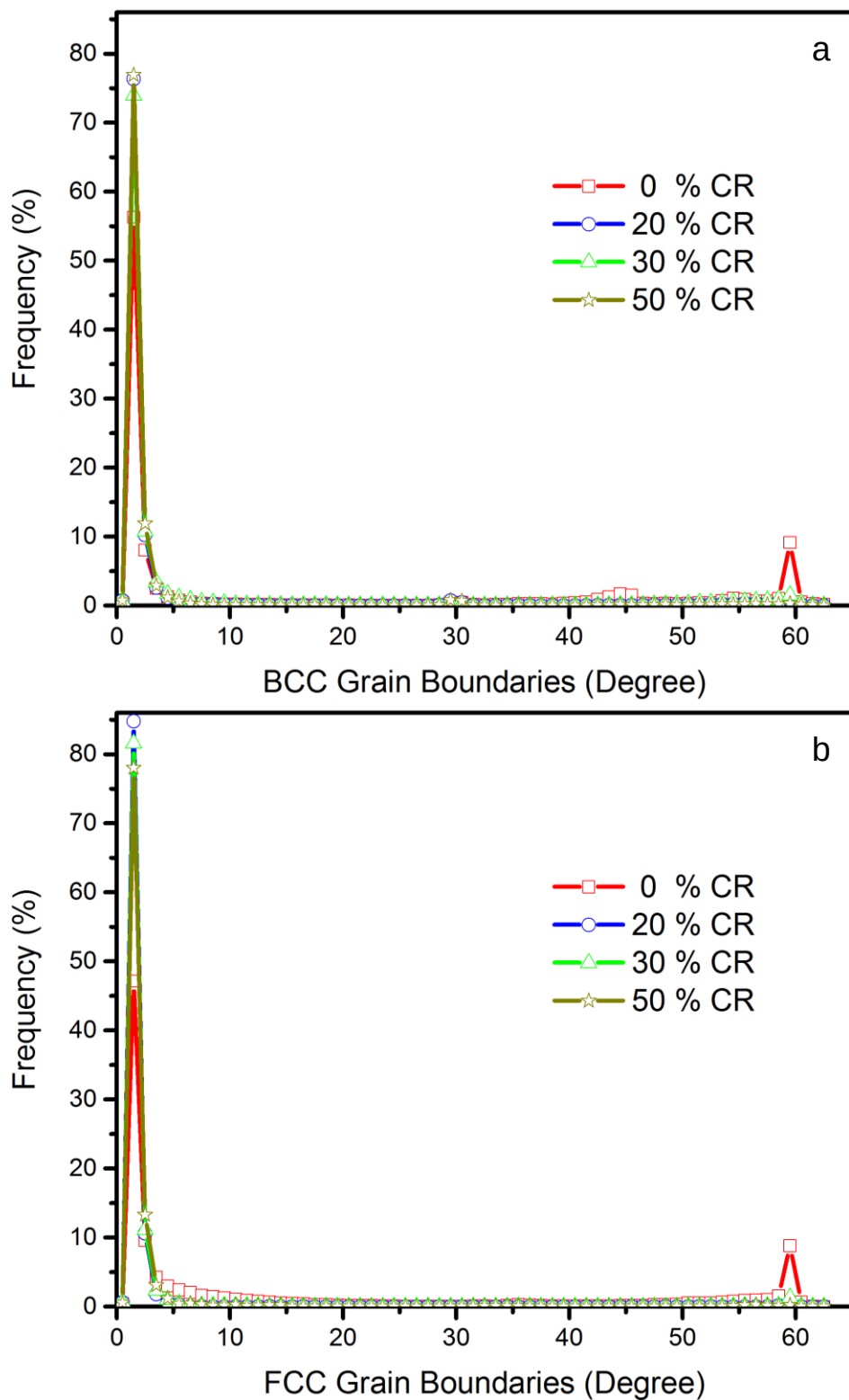
SEM-TKD KAM IQ-maps of the 0.7 mm rolled RD FeCMn steel (a) the α/α' phase of the cold rolled specimen, (b) the α/α' and (d) the γ phases of the specimen after the IA at 620 °C for 10 min and (c) the BCC and (e) the γ phases of the specimen annealed at 650 °C for 10 min.



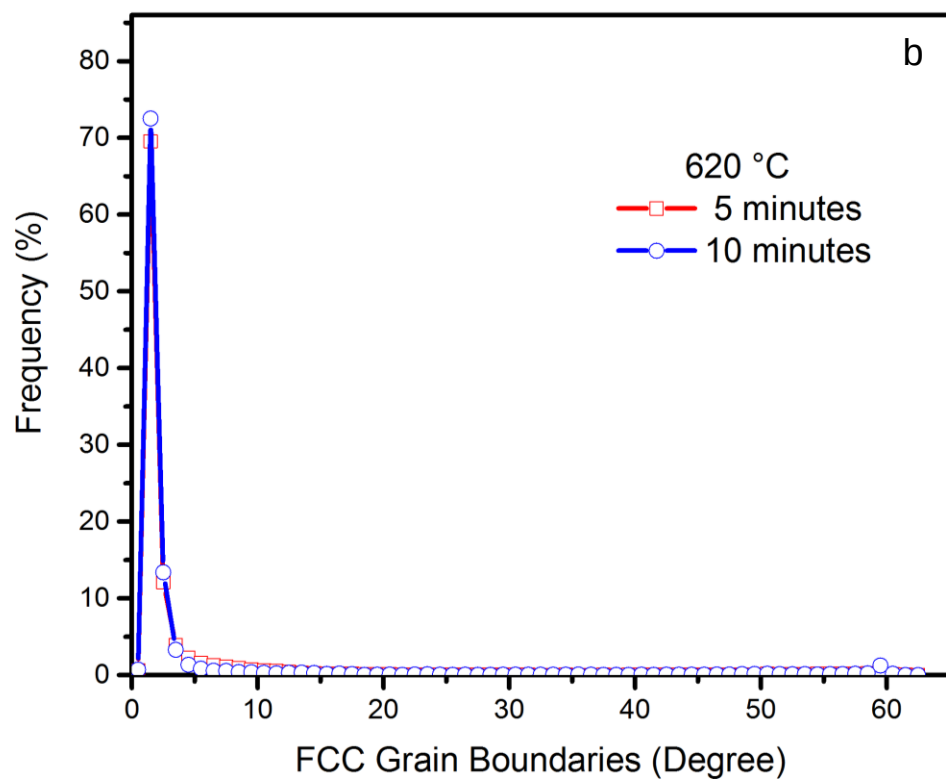
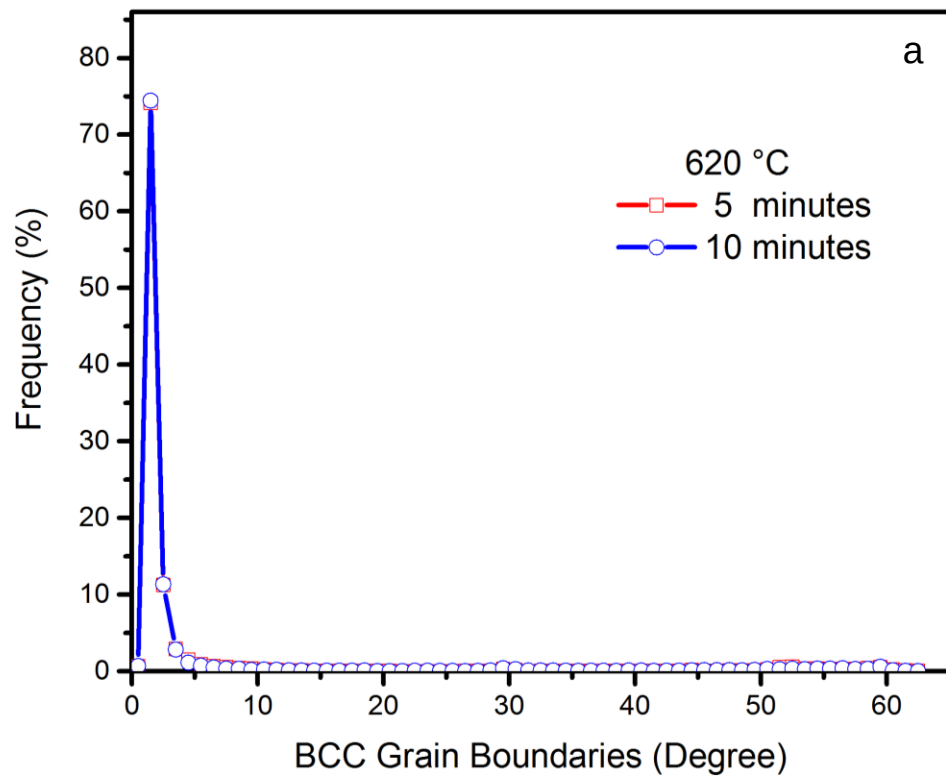
SEM-TKD KAM IQ-maps of the 0.7 mm rolled TD FeCMn steel (a) the α/α' phase of the cold rolled specimen, (b) the α/α' and (d) the γ phases of the specimen after the IA at 620 °C for 10 min and (c) the α/α' and (e) the γ phases of the specimen annealed at 650 °C for 10 min.

A2.2 : FeCMnAl Steel

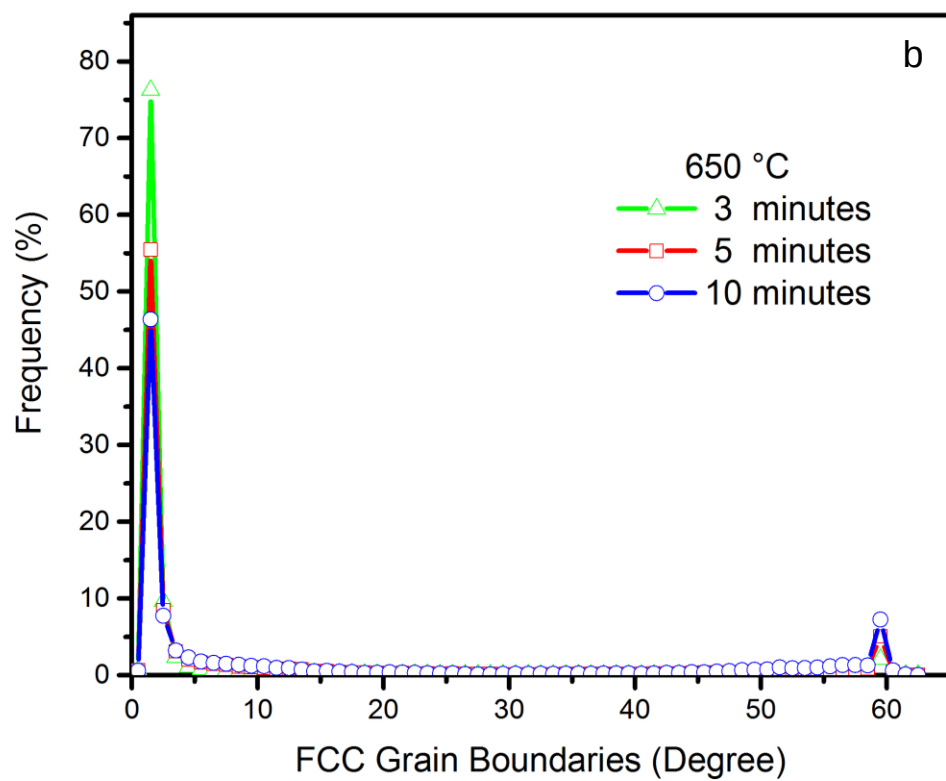
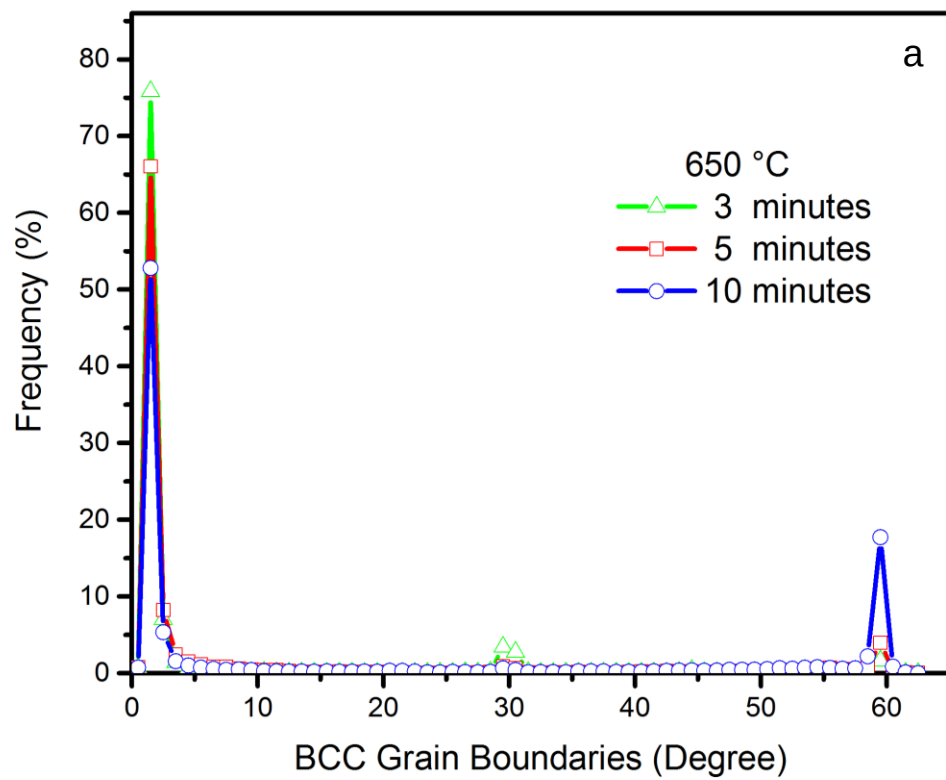
A2.2.1 : Grain Boundaries Distribution of the FeCMnAl Steel Specimens



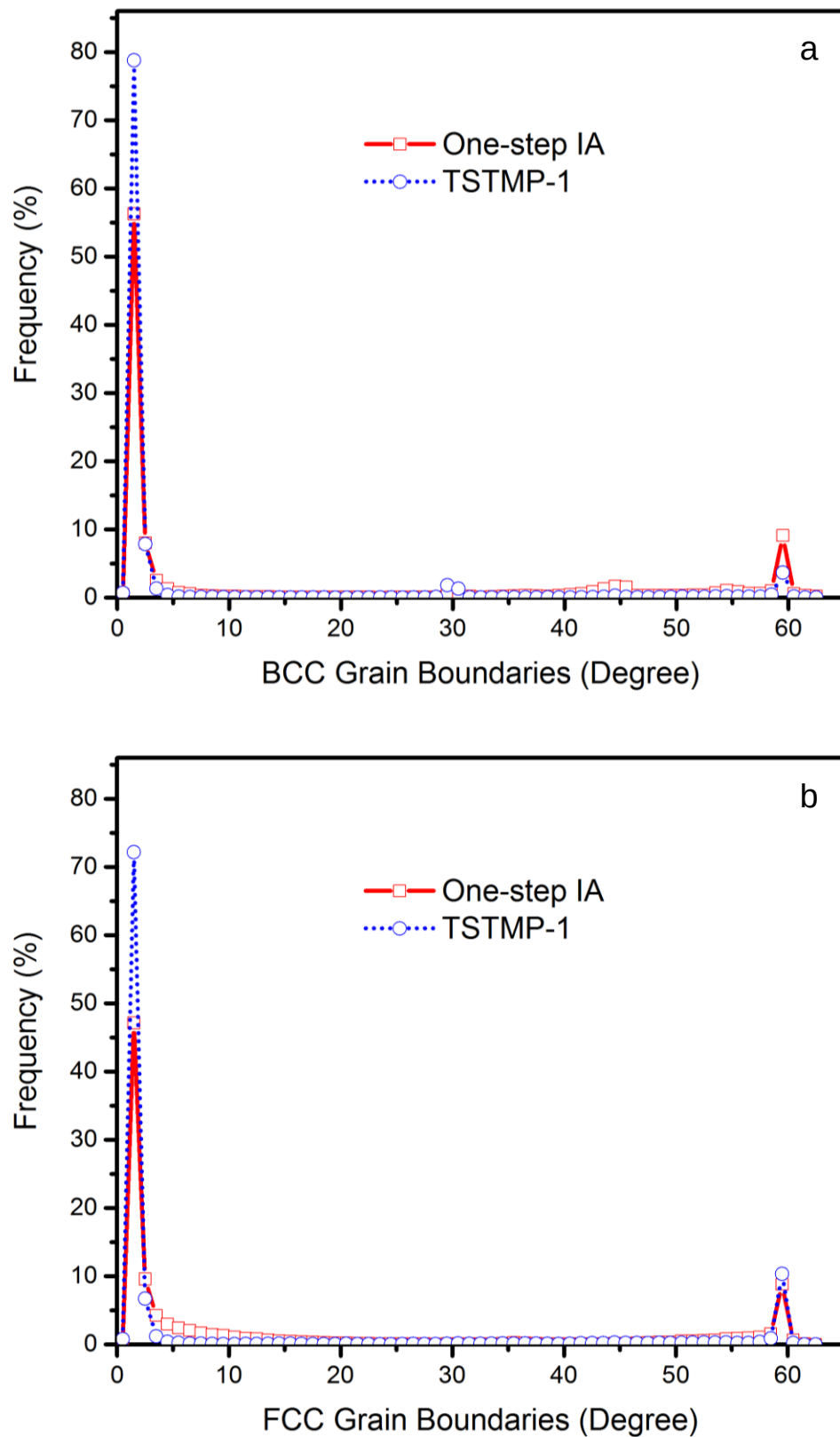
Histogram distribution of the (a) α/α' and (b) γ grain boundaries after cold rolling. The 0% CR sample is the 1.8 mm sample with 10 min IA at 650 °C without cold rolling.



Histogram distribution of the (a) α/α' and (b) γ grain boundaries in the 0.9 mm FeCMnAl steel samples after the IA at 620 °C

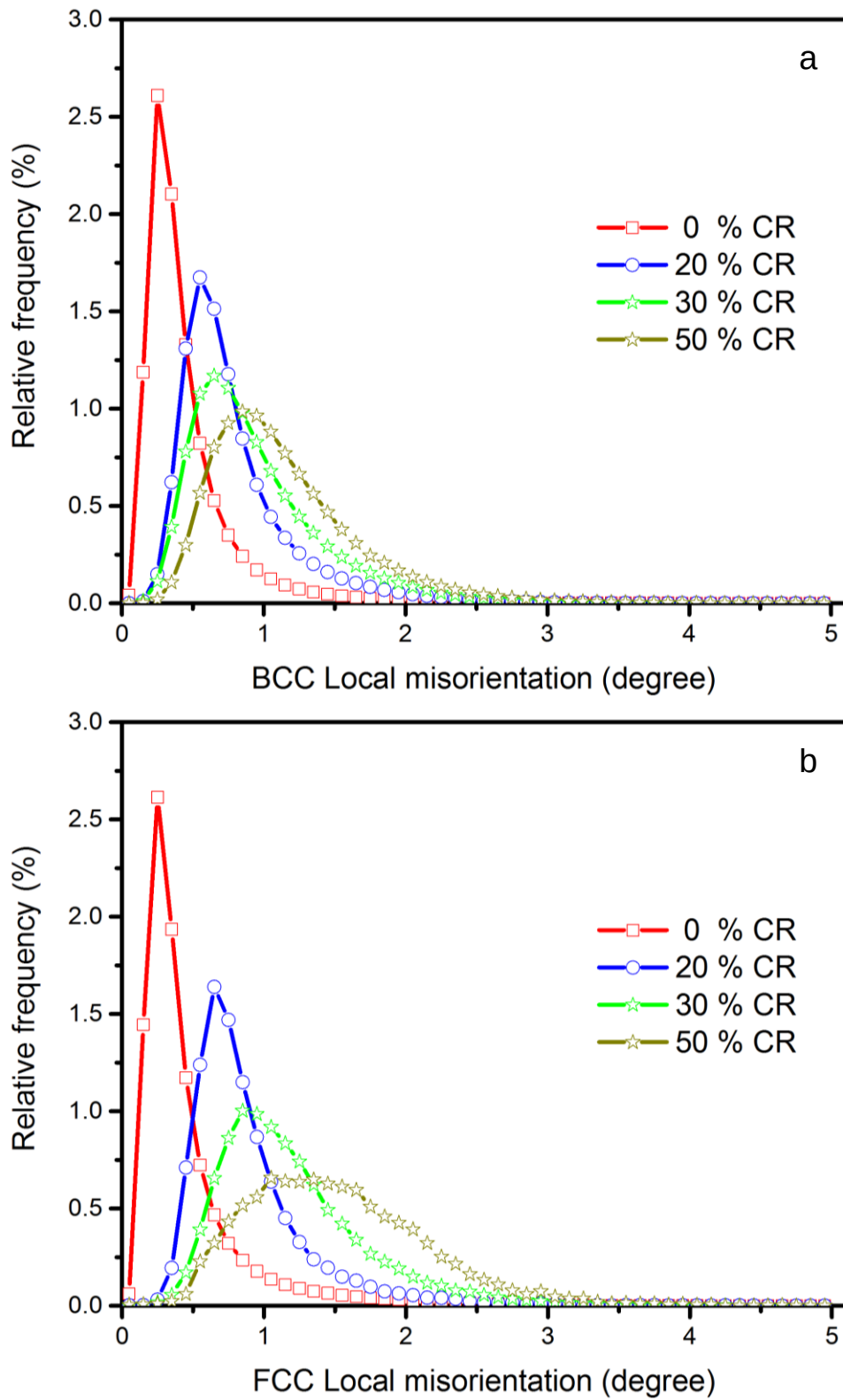


Histogram distribution of the (a) α/α' and (b) γ grain boundaries in the 0.9 mm FeCMnAl steel samples after the IA at 650 °C

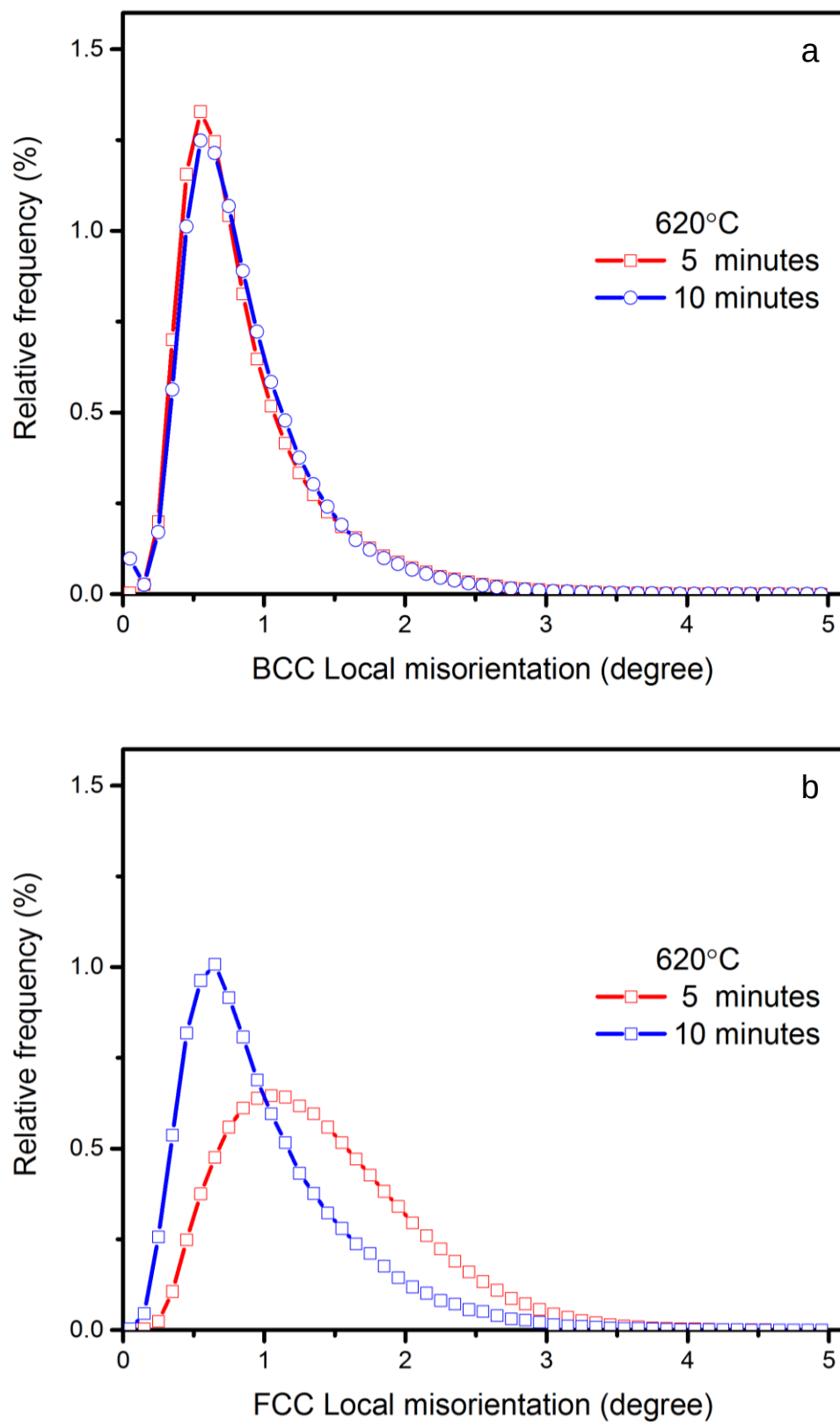


Histogram distribution of the (a) α/α' and (b) γ local misorientation in the FeCMnAl steel samples after one-step IA and the TSTMP conditions (chapter 7)

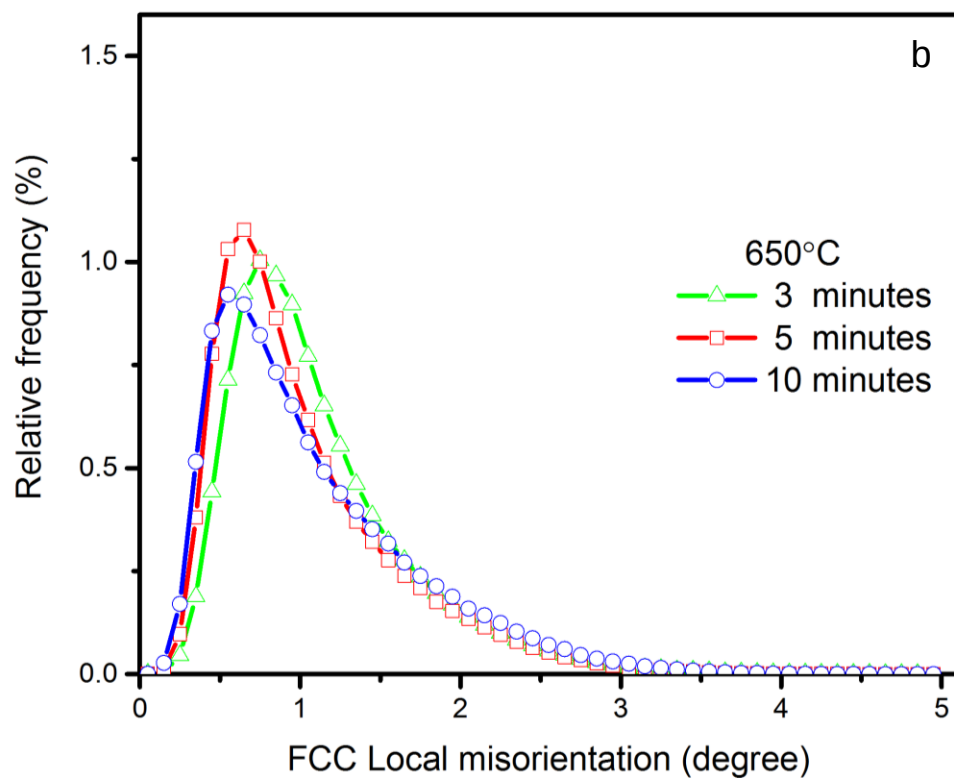
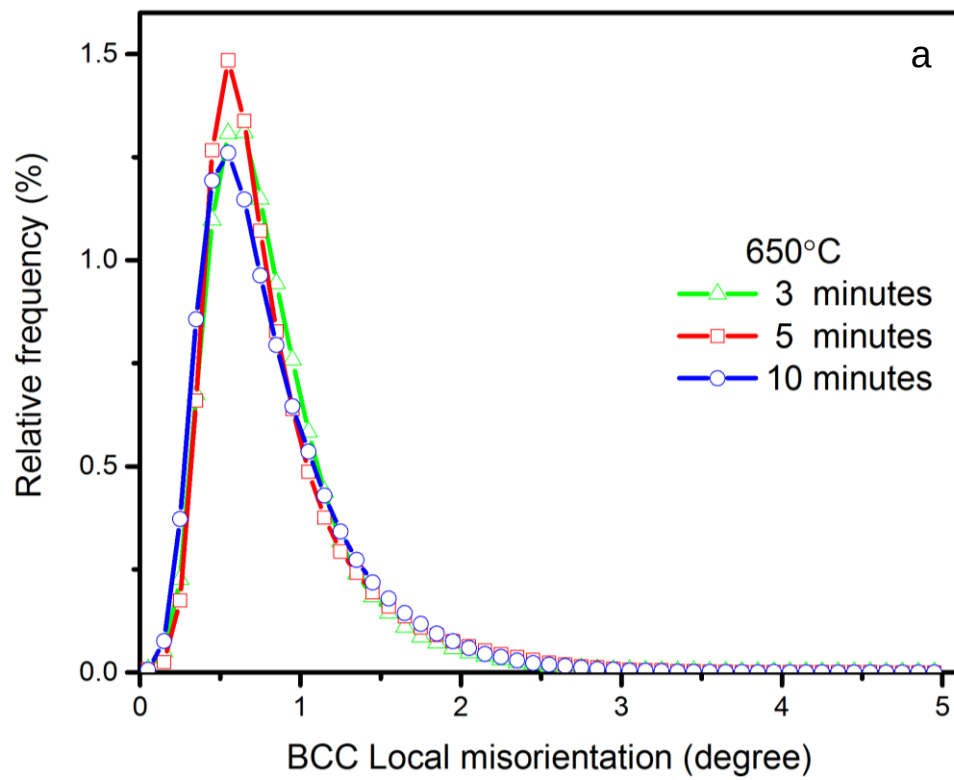
A2.2.2 : Kernel Average Misorientation Histogram Distributions and the Maps of the FeCMnAl Steel



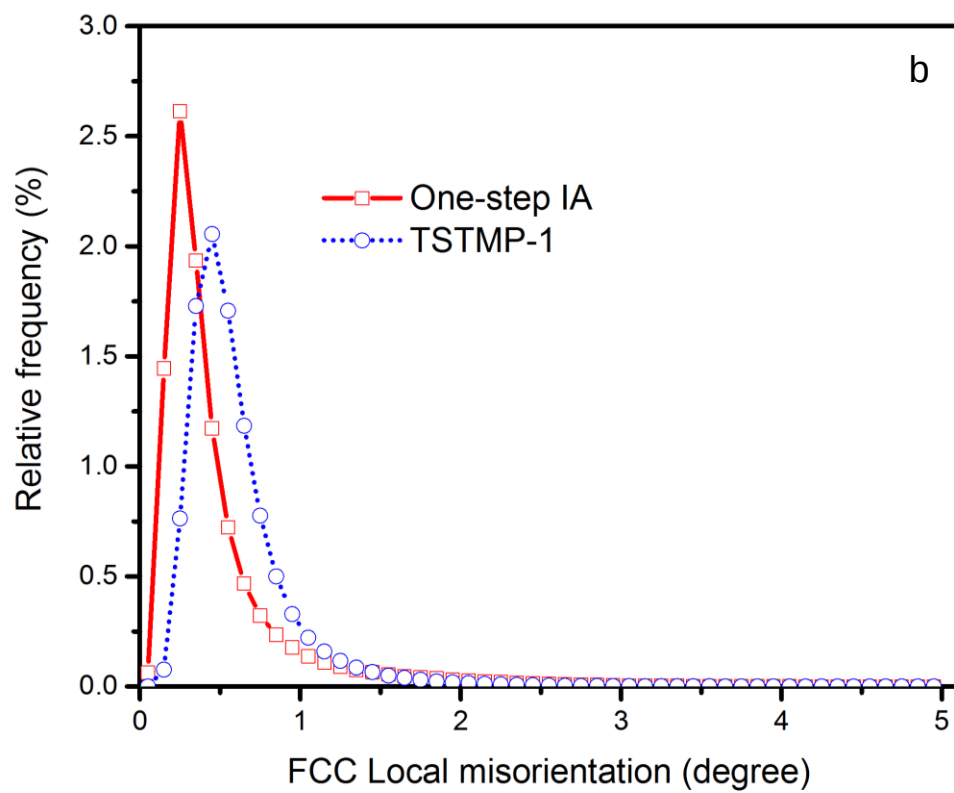
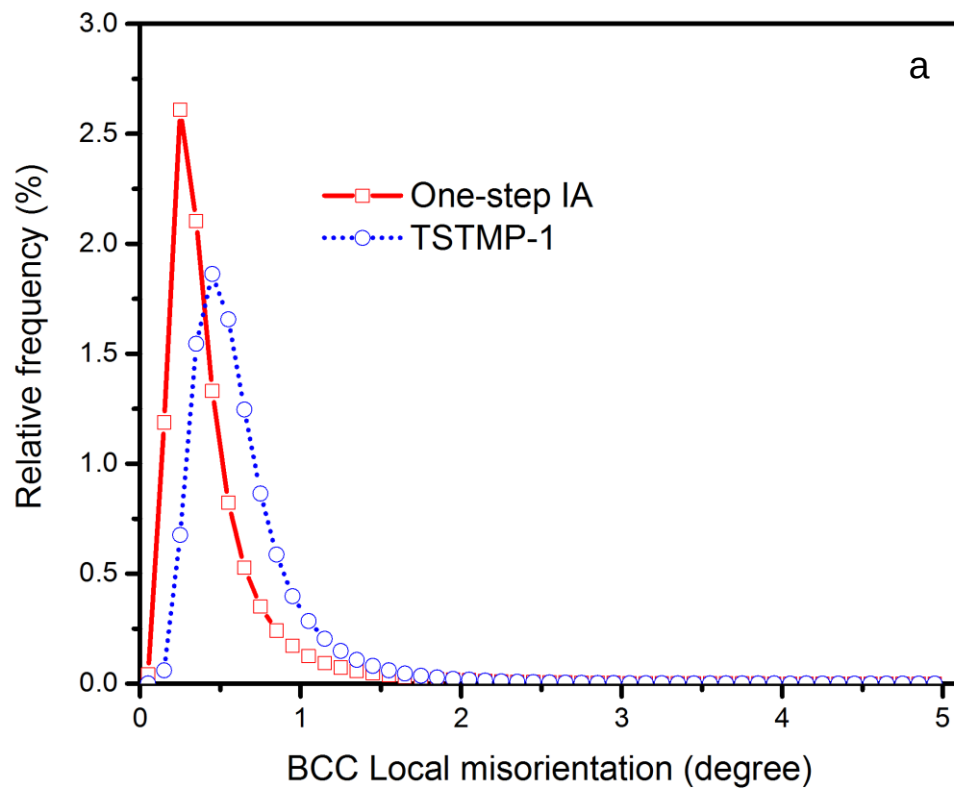
Histogram distribution of the (a) α/α' and (b) γ local misorientation after cold rolling. The 0% CR sample is the 1.8 mm sample with 10 min IA at 650 °C without cold rolling.



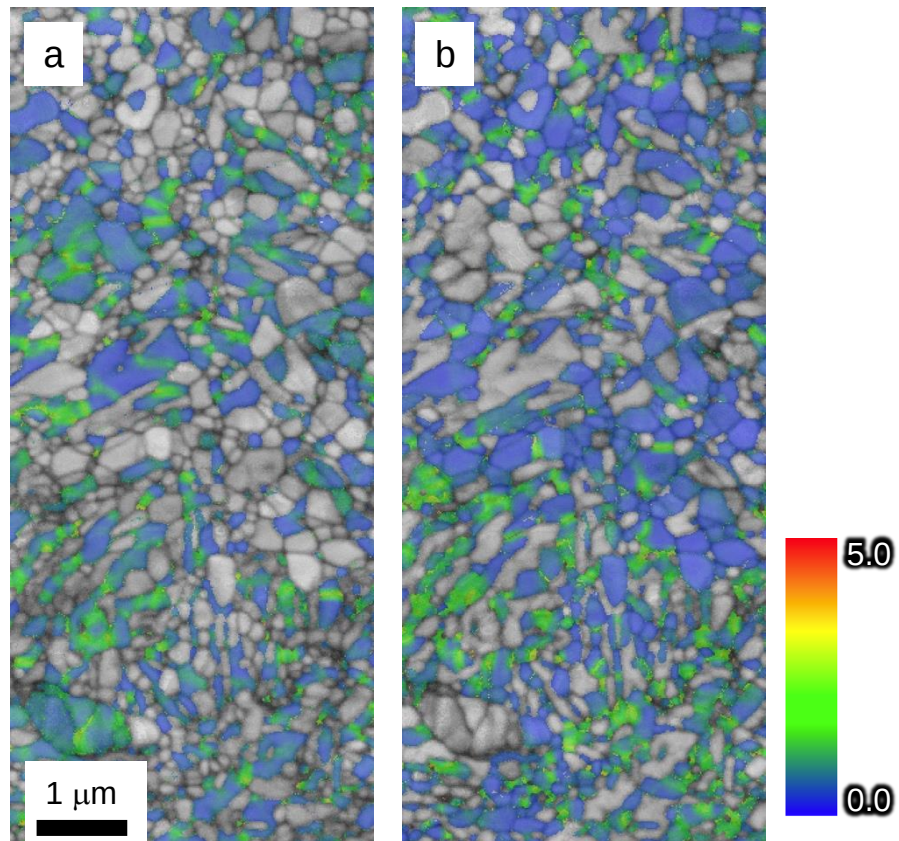
Histogram distribution of the (a) α/α' and (b) γ local misorientation in the 0.9 mm FeCMnAl steel samples after the IA at 620 °C



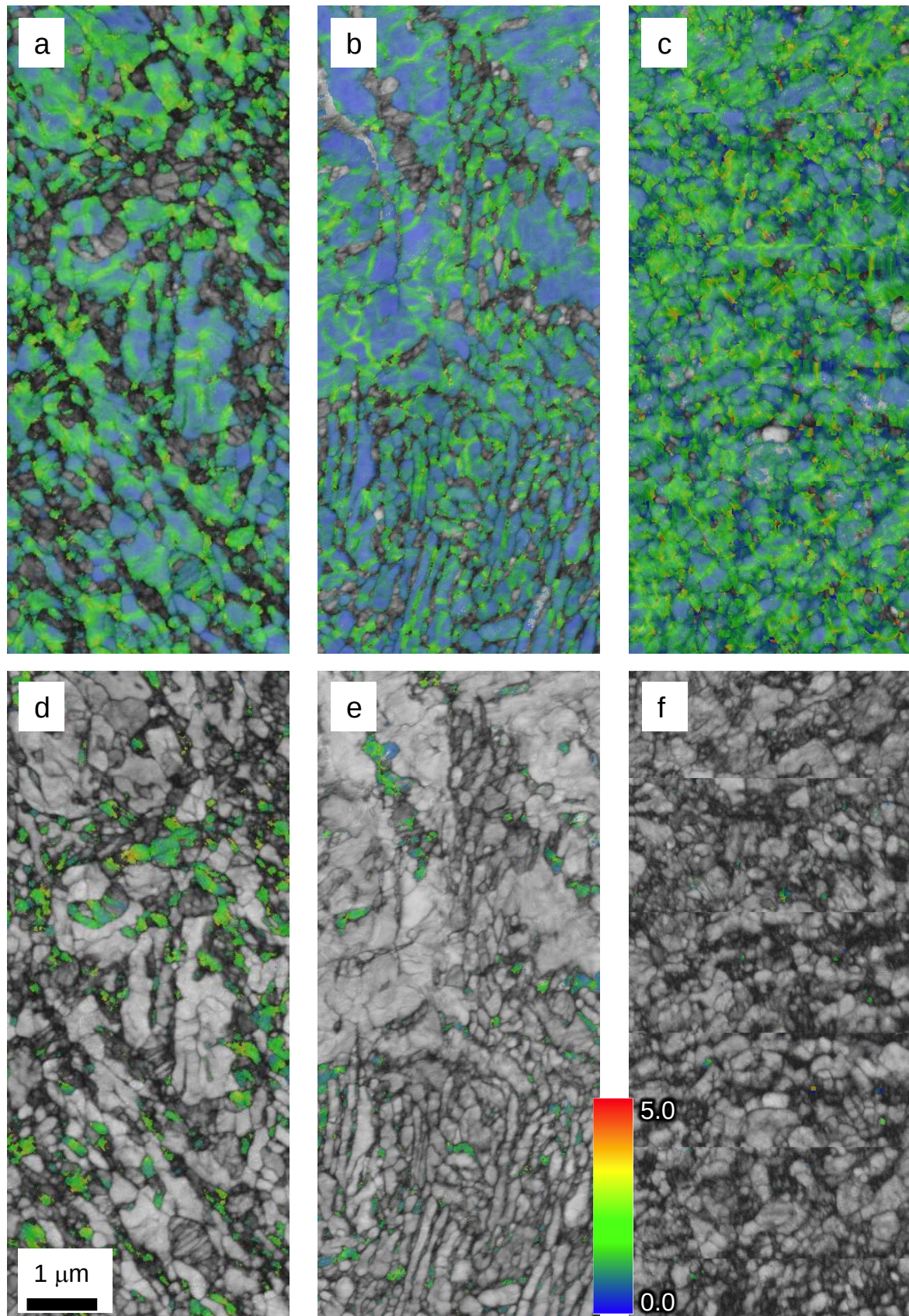
Histogram distribution of the (a) α/α' and (b) γ local misorientation in the 0.9 mm FeCMnAl steel samples after the IA at 650 °C



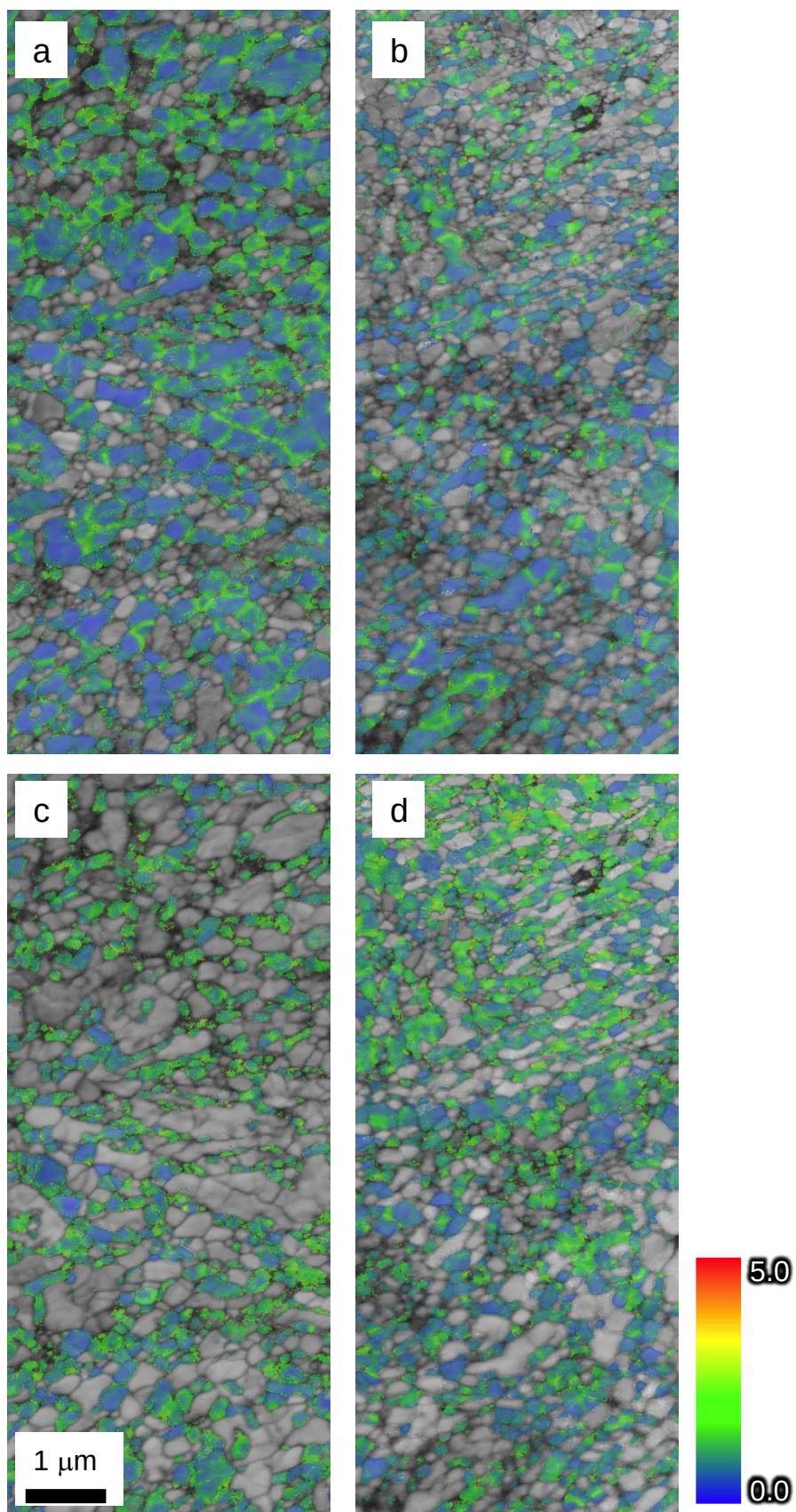
Histogram distribution of the (a) α/α' and (b) γ local misorientation in the FeCMnAl steel samples after the one-step IA and the TSTMP conditions (chapter 7)



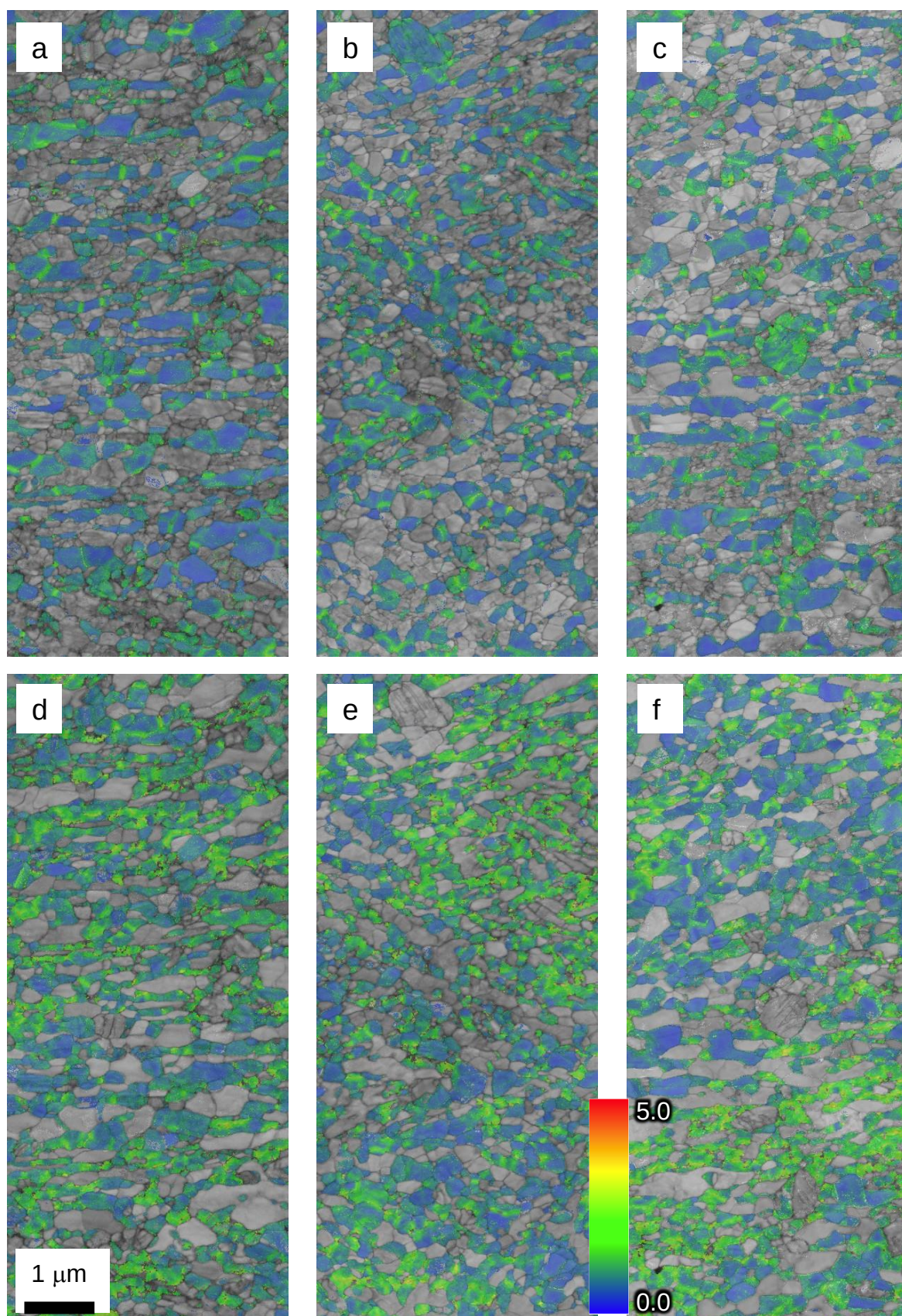
SEM-TKD KAM IQ-maps of the (a) α/α' and (b) γ phases in the 1.8 mm FeCMnAl steel annealed at 650 °C for 10 min.



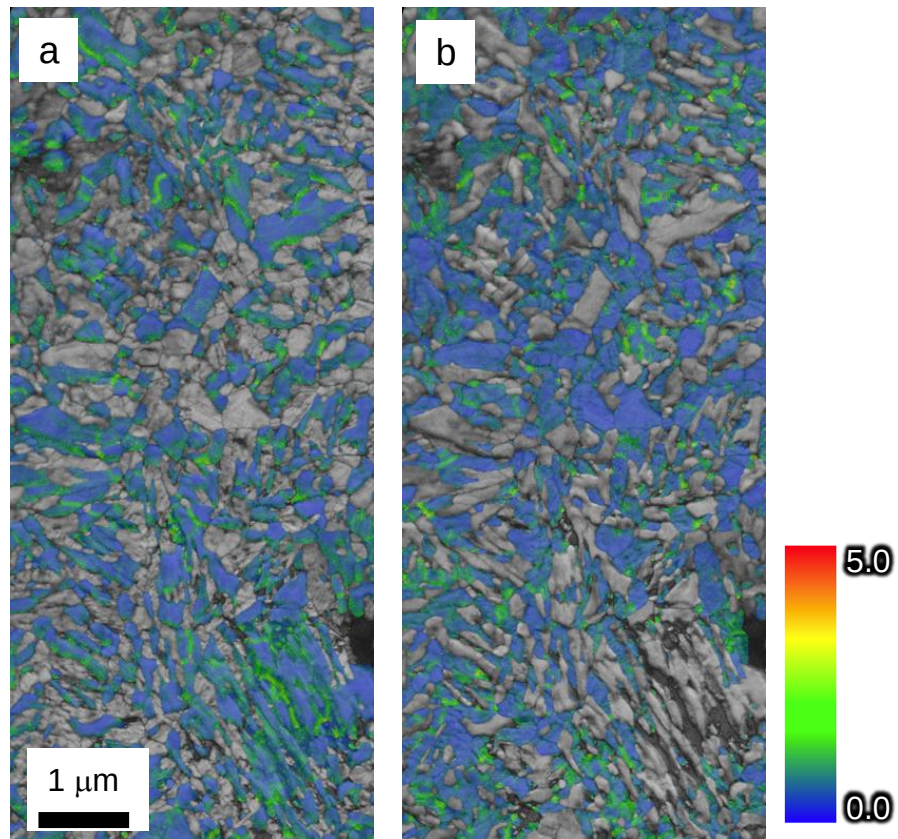
SEM-TKD KAM IQ-maps of the IA 1.8 mm FeCMnAl steel at 650 °C for 10 min, the α/α' phase after (a) 20%, (b) 30%, and (c) 50% TD cold rolling and the γ phase after (d) 20%, (e) 30%, and (f) 50% TD cold rolling.



SEM-TKD KAM IQ-maps of the 0.9 mm FeCMnAl steel after the IA at 620 °C, the α/α' phase after (a) 5 and (b) 10 min and the γ phase after (c) 5 and (d) 10 min



SEM-TKD KAM IQ-maps of the 0.9 mm FeCMnAl steel after the IA at 650 °C, the α/α' phase after (a) 3, (b) 5 and (c) 10 min and the γ phase after (d) 3, (e) 5 and (f) 10 min.



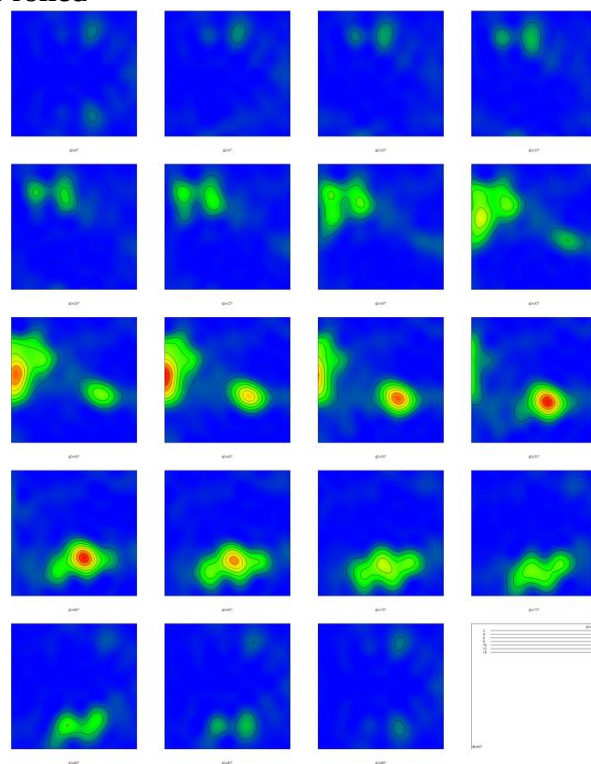
SEM-TKD KAM IQ-maps of the (a) α/α' and (b) γ phases in the FeCMnAl steel annealed at 700 °C for 3 min, quenched at 200 °C and then in the water, 5% cold rolled in the RD and then the IA at 650 °C for 10 min.

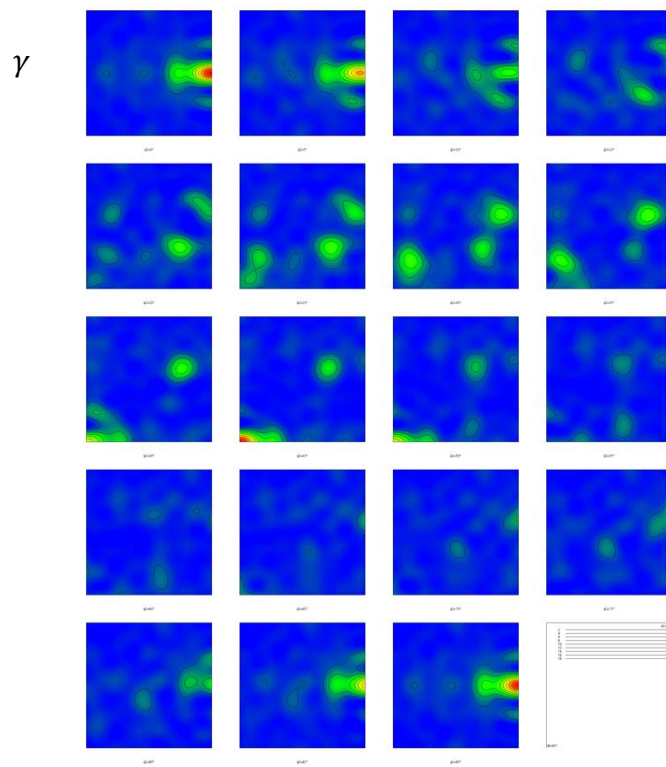
Appendix 3 : Orientation Distribution Function Maps of the FeCMn Steel and the FeCMnAl Steel

A3.1 : FeCMn Steel

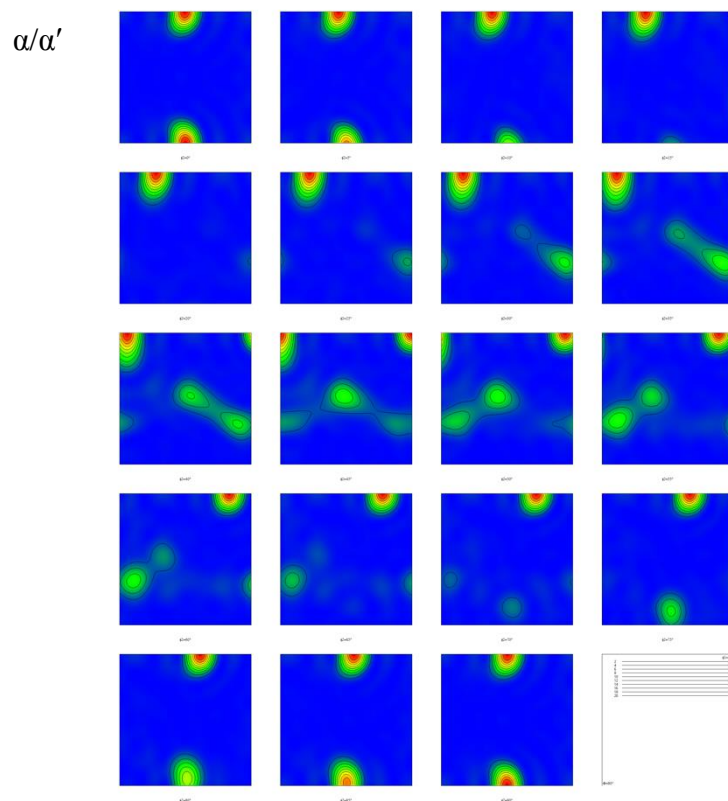
The 1.4 mm FeCMn steel - cold-rolled

α/α'

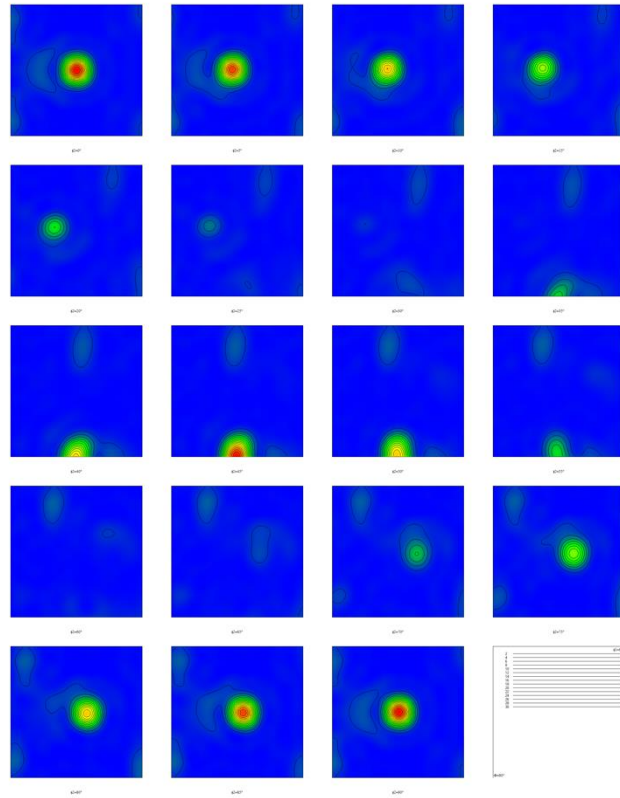




The FeCMn steel - IA at 620°C for 10 min

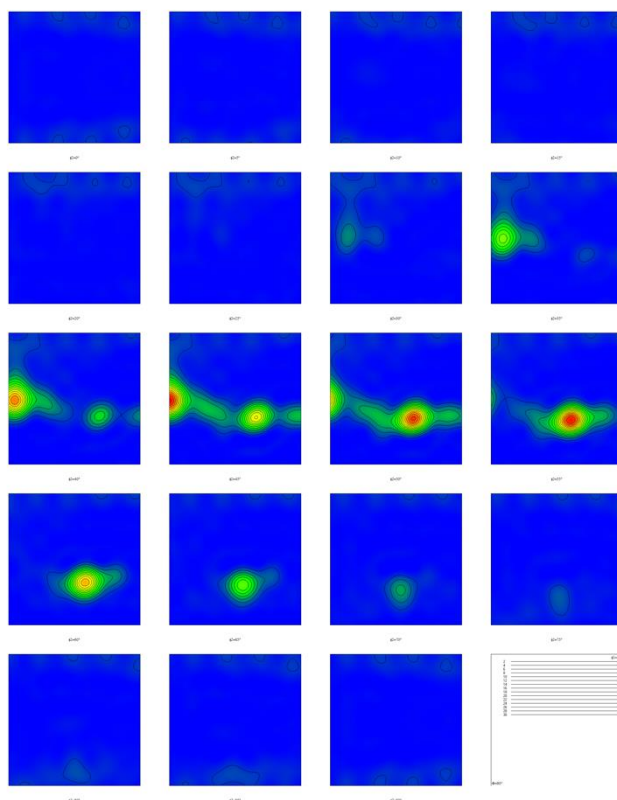


γ

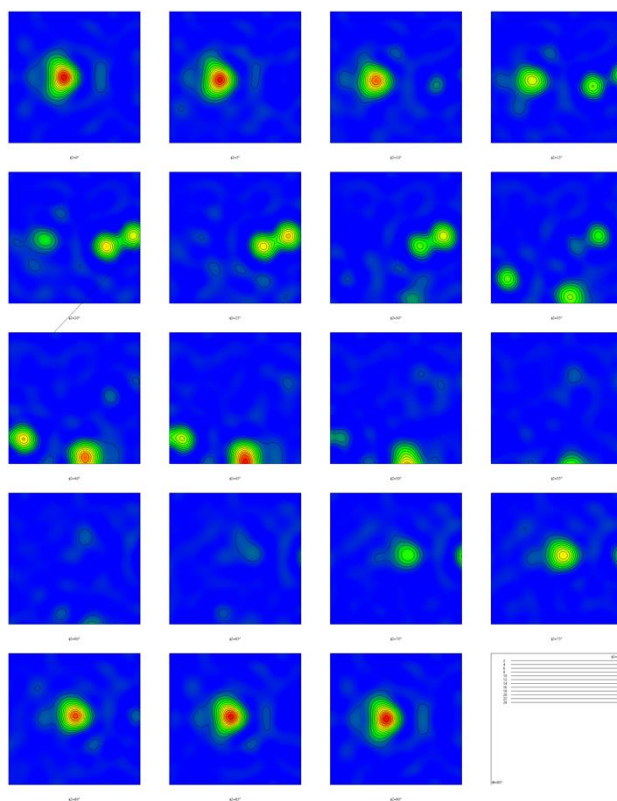


The FeCMn steel - IA at 620°C, 10 min –50% RD cold-rolled

α/α'

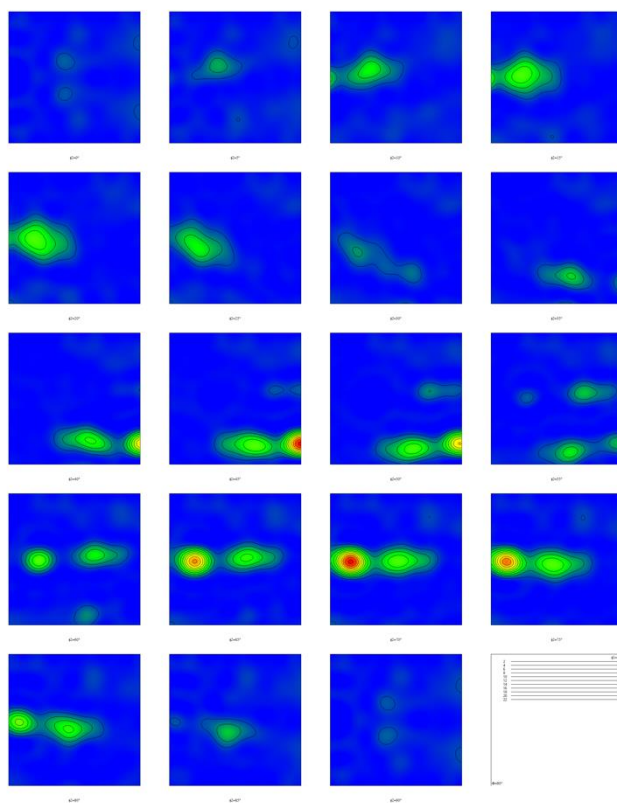


γ

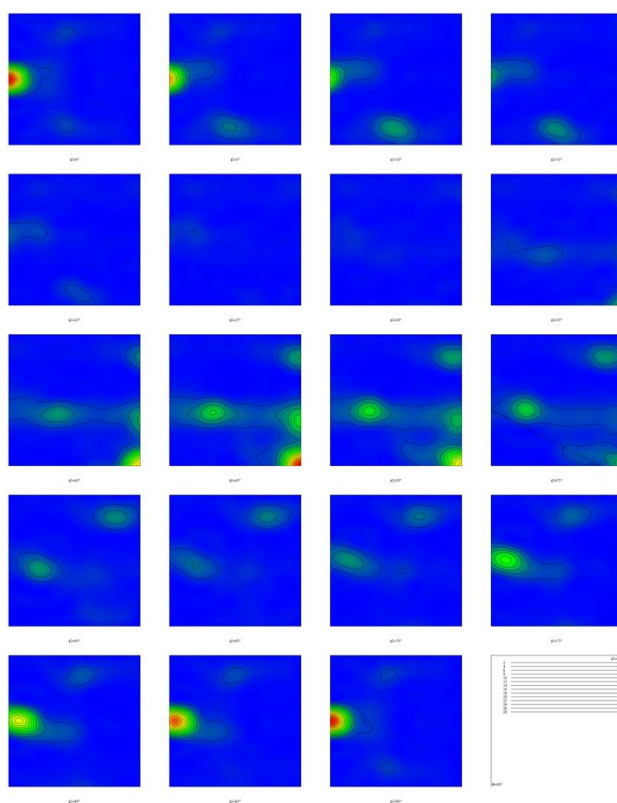


The FeCMn steel - IA at 620°C, 10 min – 50% RD cold-rolled - IA at 620°C, 10 min

α/α'

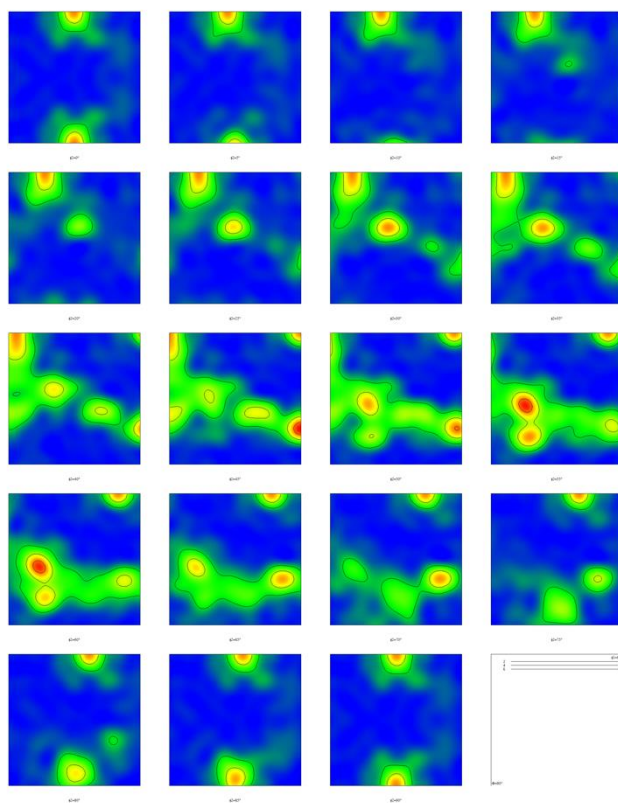


γ

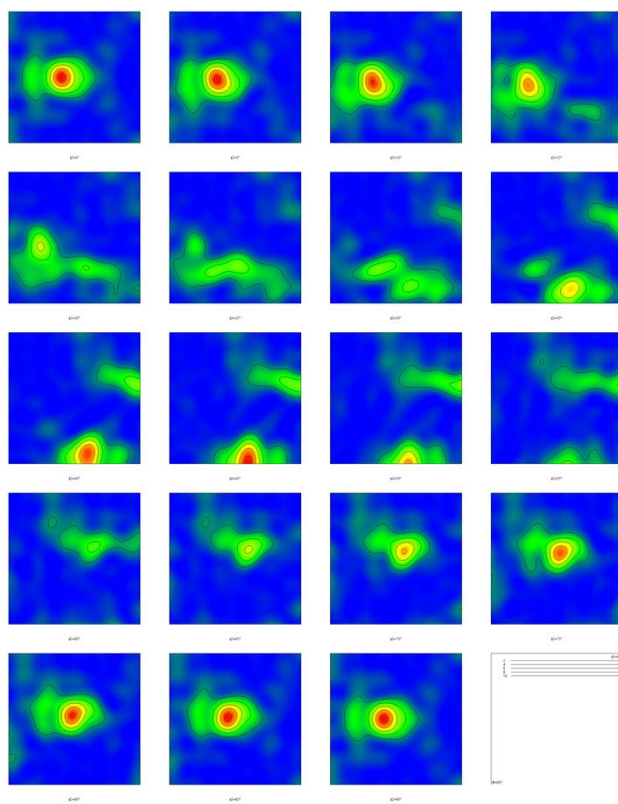


The FeCMn steel - IA at 620°C, 10 min –50% RD cold-rolled- annealed at 650°C, 10 min

α/α'

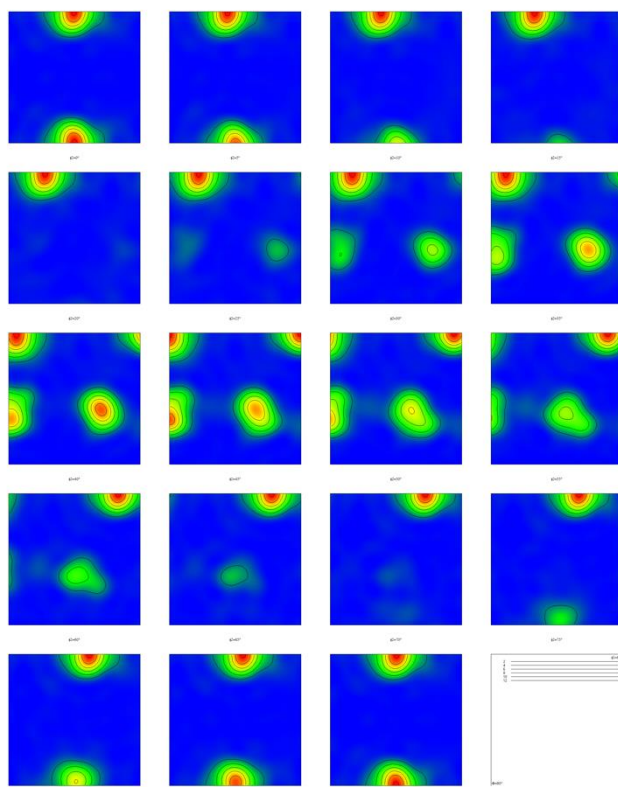


γ

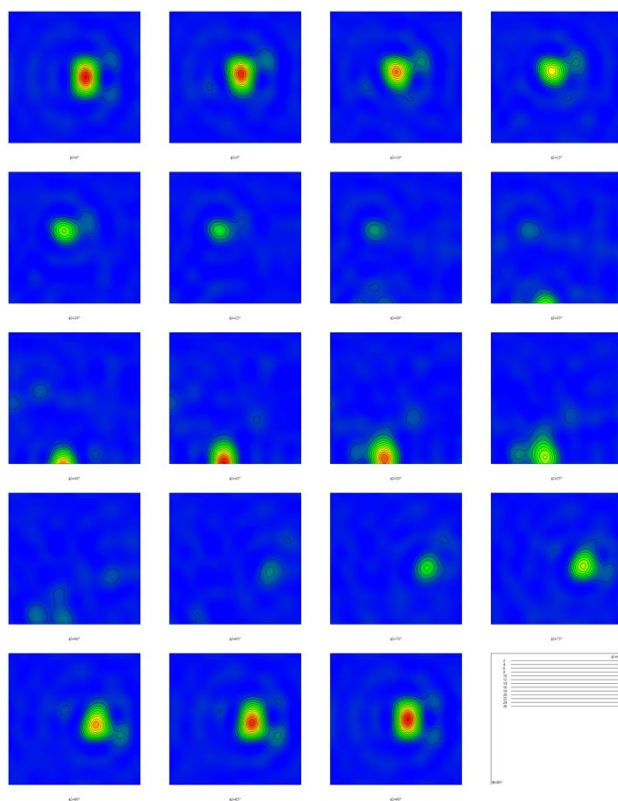


The FeCMn steel - IA at 620°C, 10 min –50% TD cold-rolled

α/α'

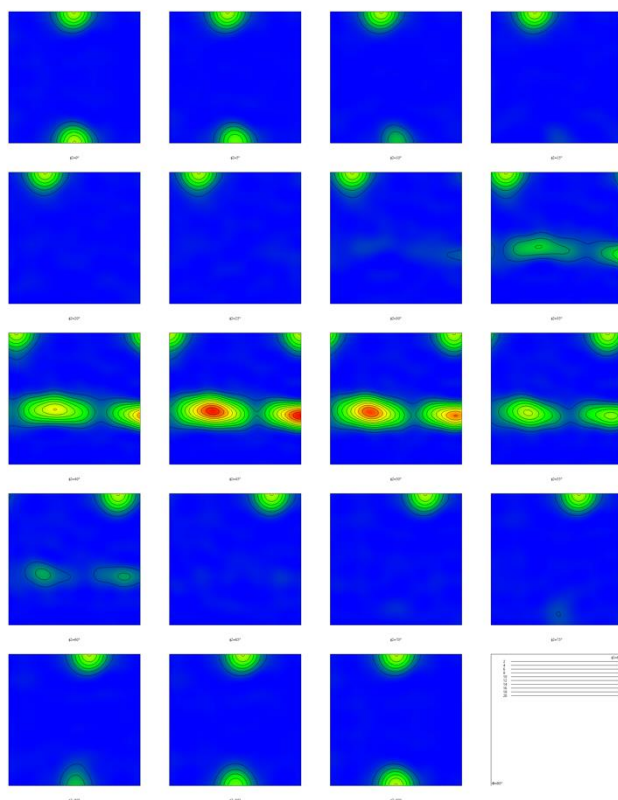


γ

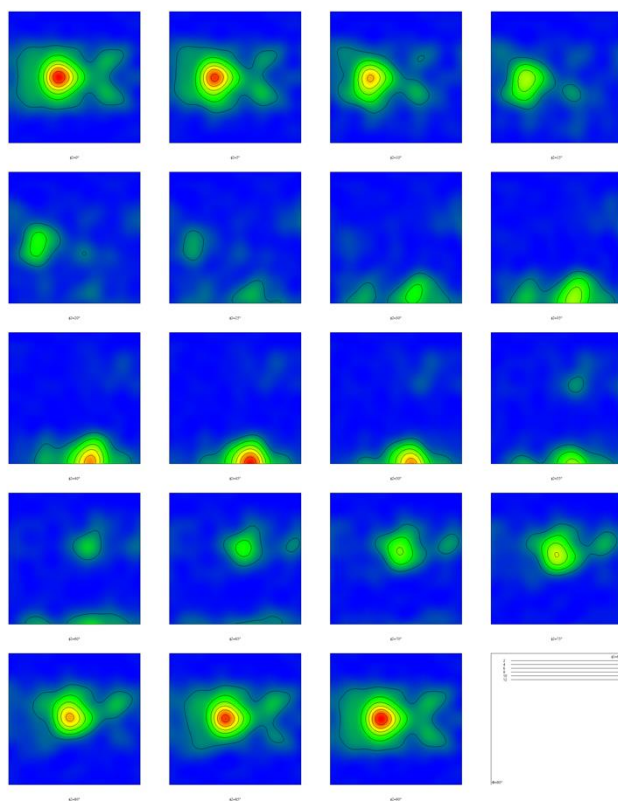


The FeCMn steel - IA at 620°C, 10 min – 50% TD cold-rolled- IA at 620°C, 10 min

α/α'

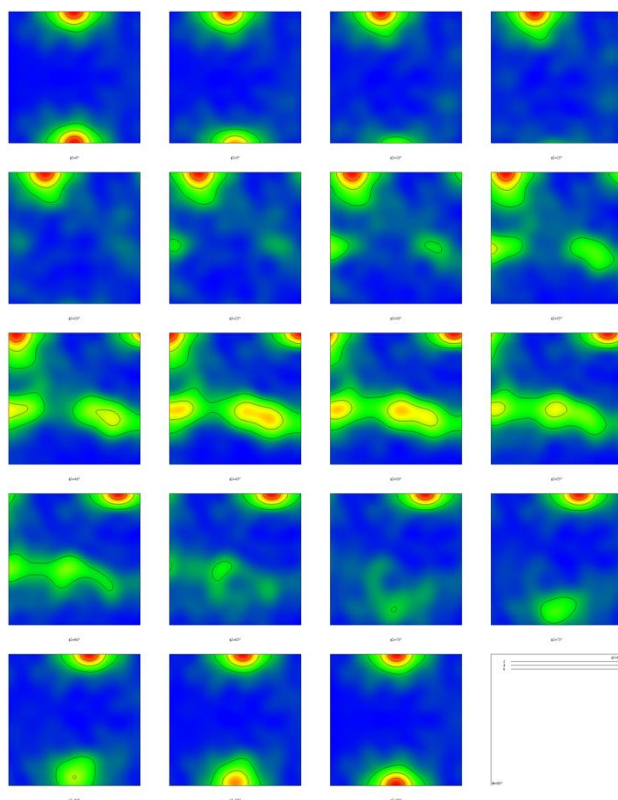


γ

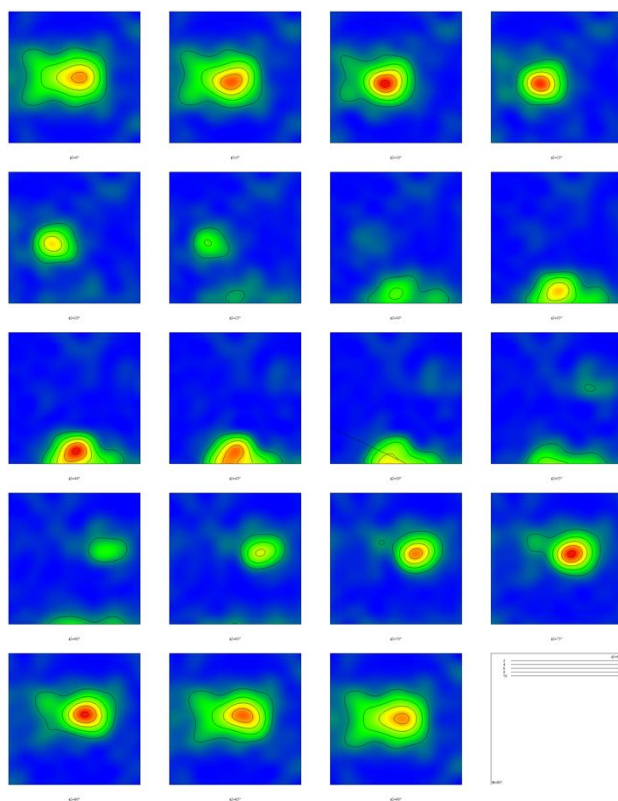


The FeCMn steel - IA at 620°C, 10 min –50% TD cold-rolled- annealed at 650°C, 10 min

α/α'

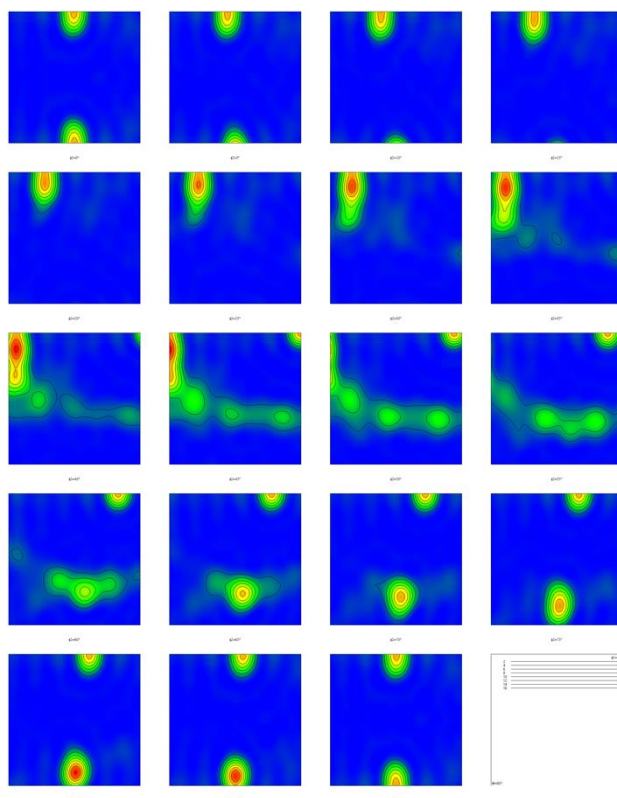


γ

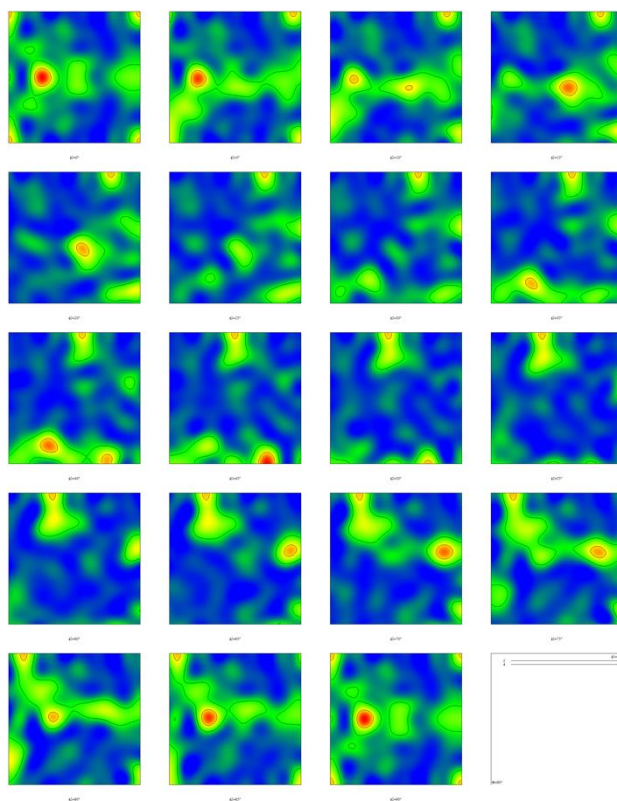


The FeCMn steel –WPHT 50% RD cold-rolled

α/α'

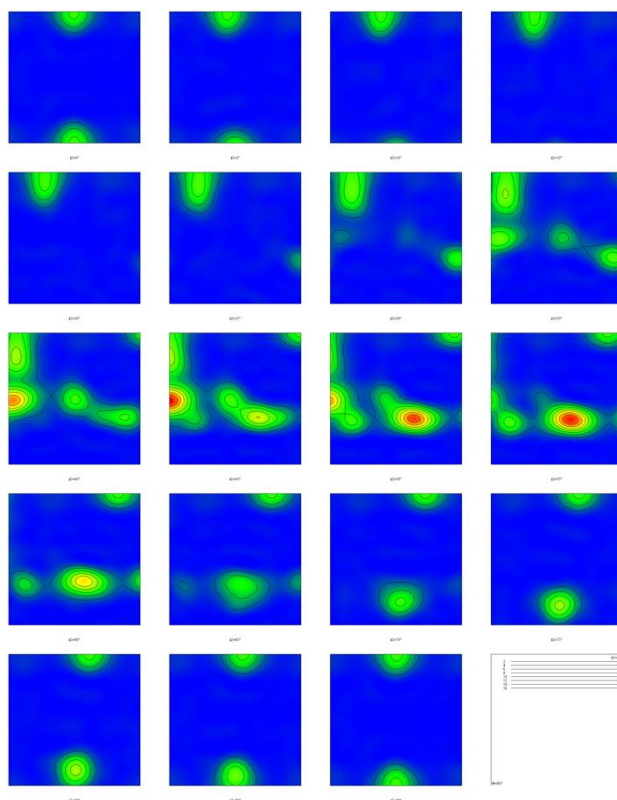


γ

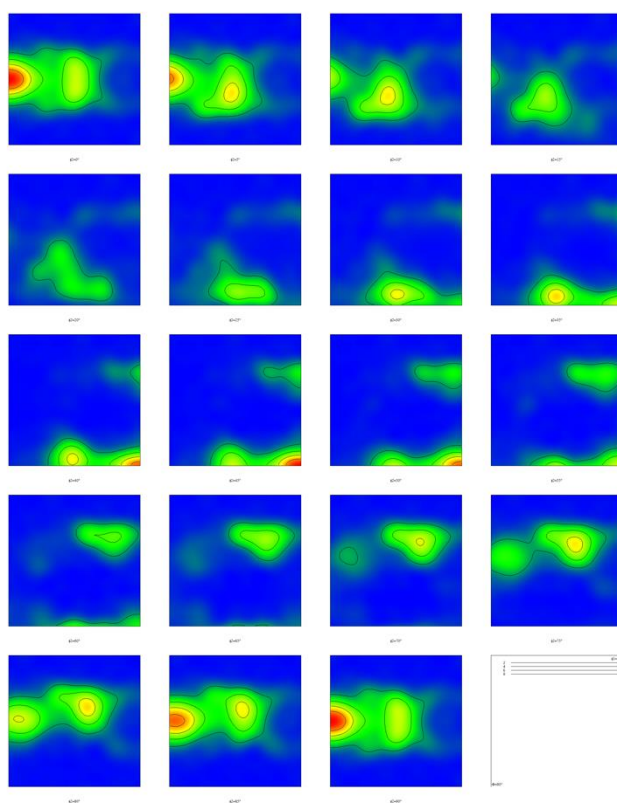


The FeCMn steel – WPHT, 50% RD cold-rolled - IA at 620°C, 10 min

α/α'

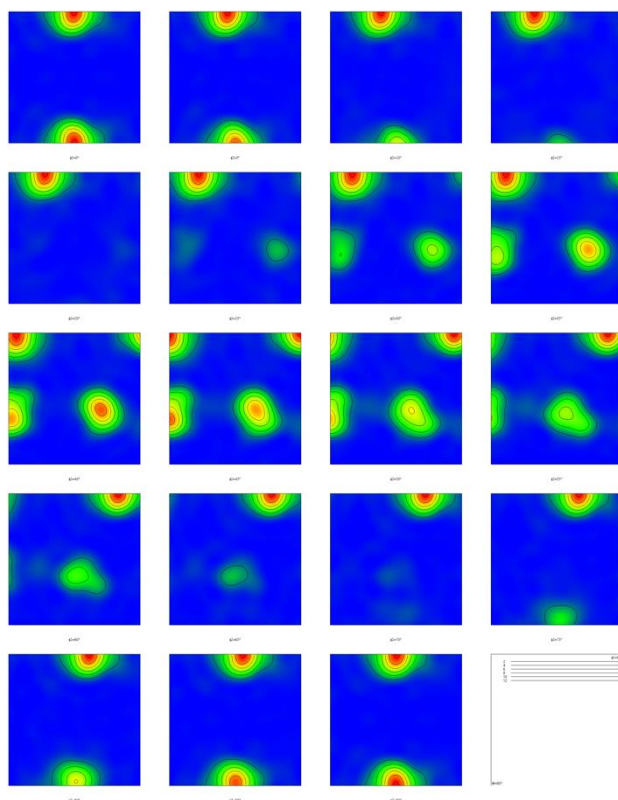


γ

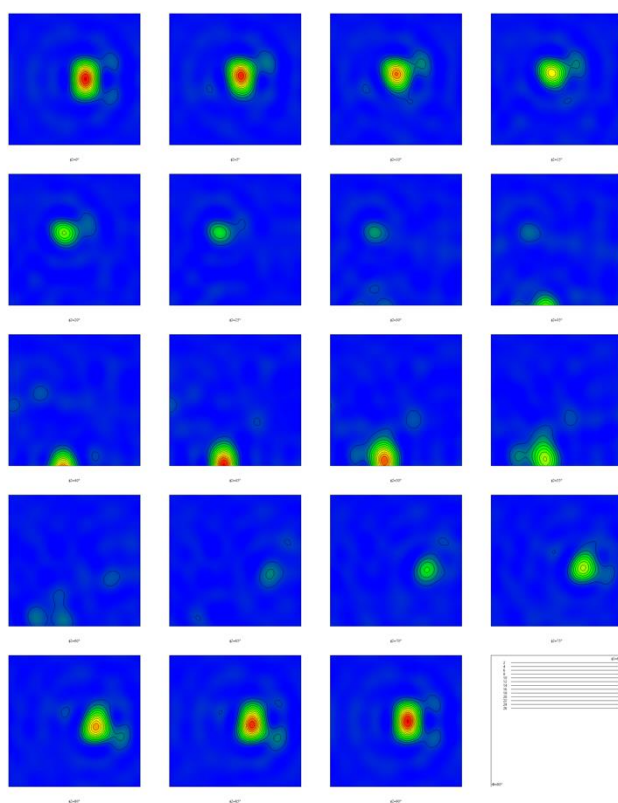


The FeCMn steel –WPHT 20% TD cold-rolled

α/α'

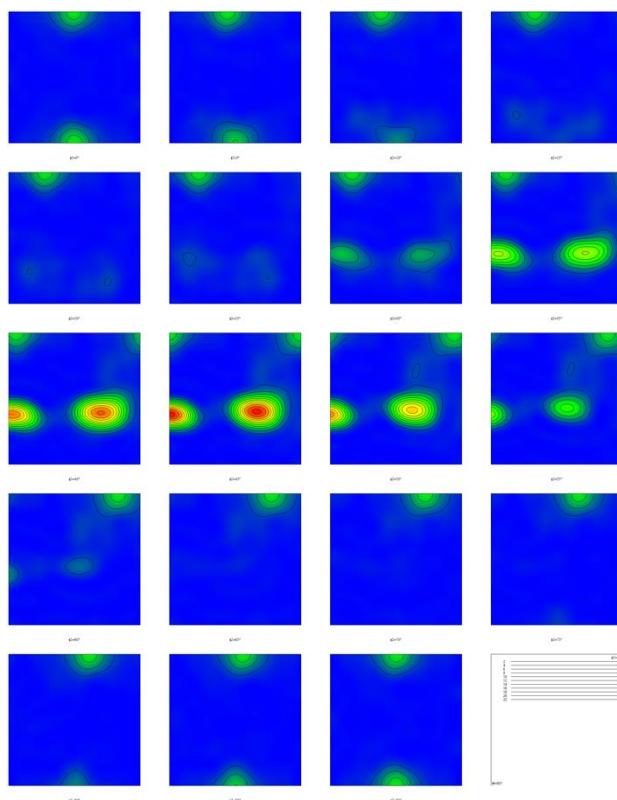


γ

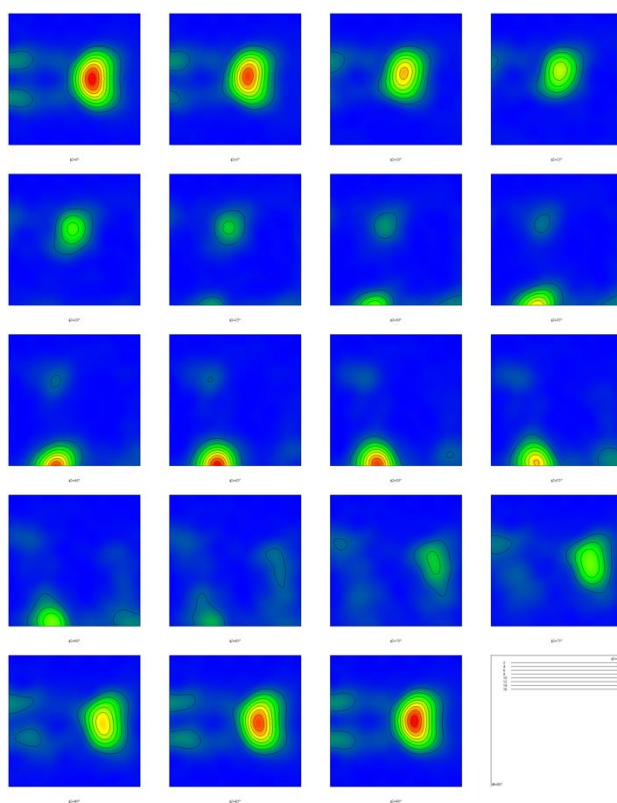


The FeCMn steel – WPHT 20% TD cold-rolled - IA at 620°C, 10 min

α/α'



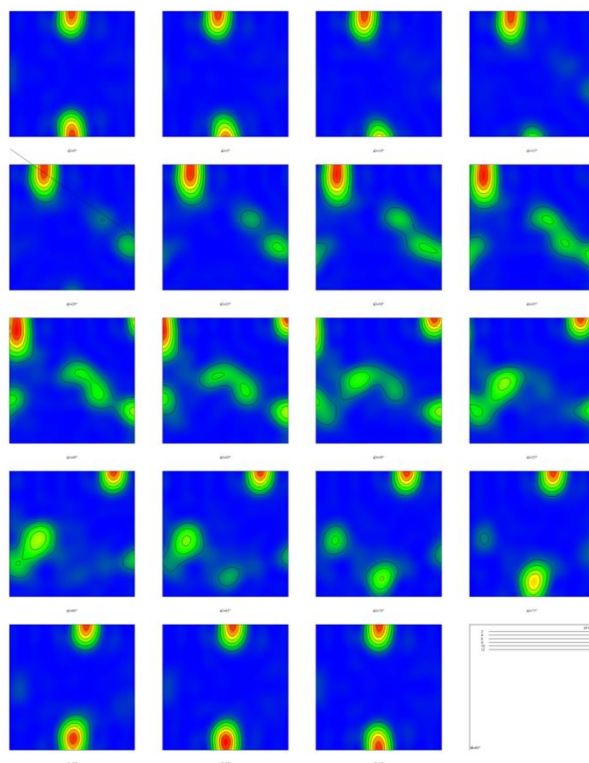
γ



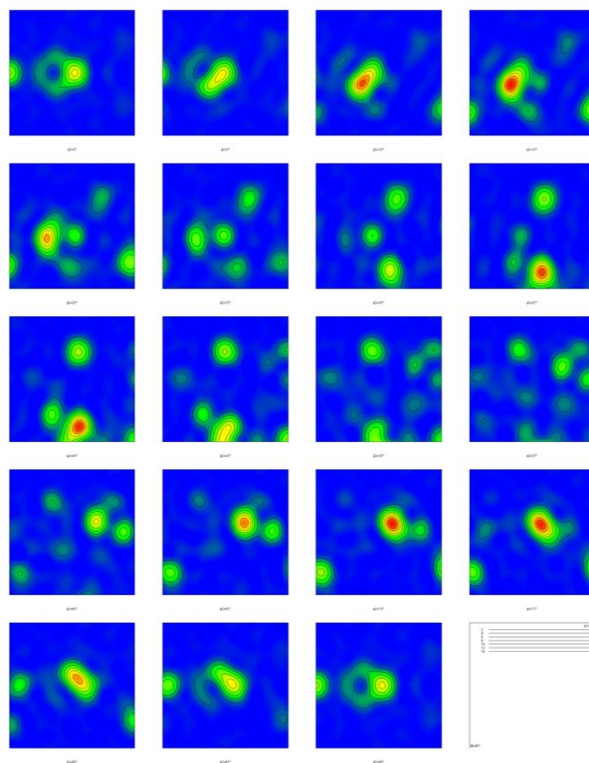
A3.2 : FeCMnAl Steel

The 1.8 mm FeCMnAl steel – cold-rolled

α/α'

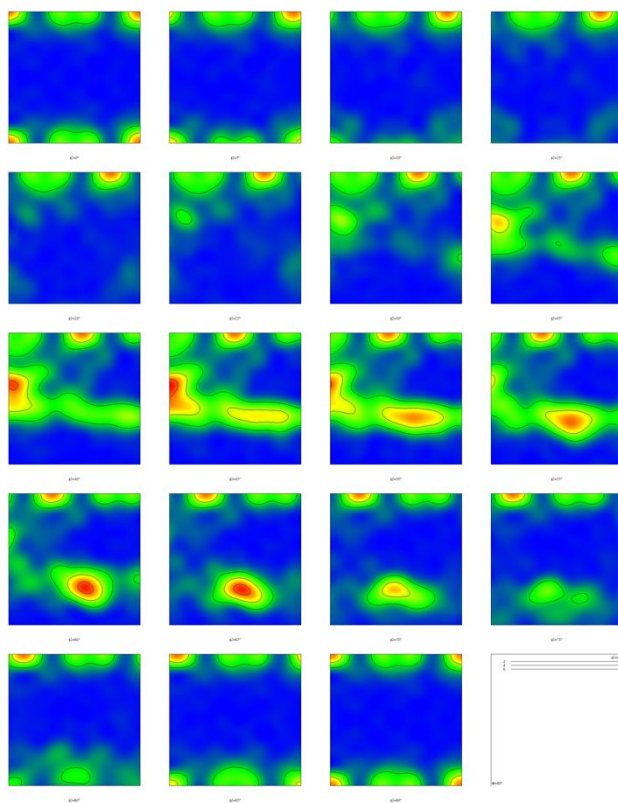


γ

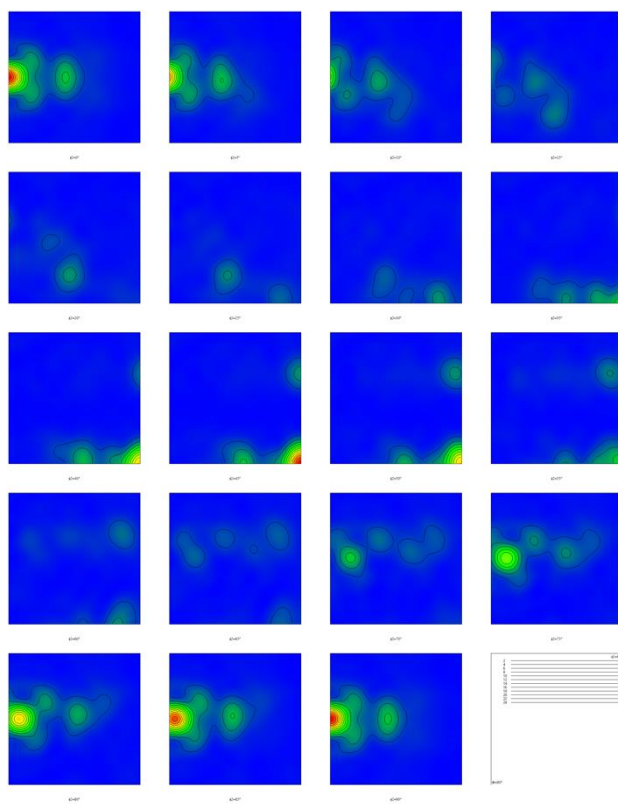


The 1.8mm FeCMnAl steel - IA at 650°C for 10 min

α/α'

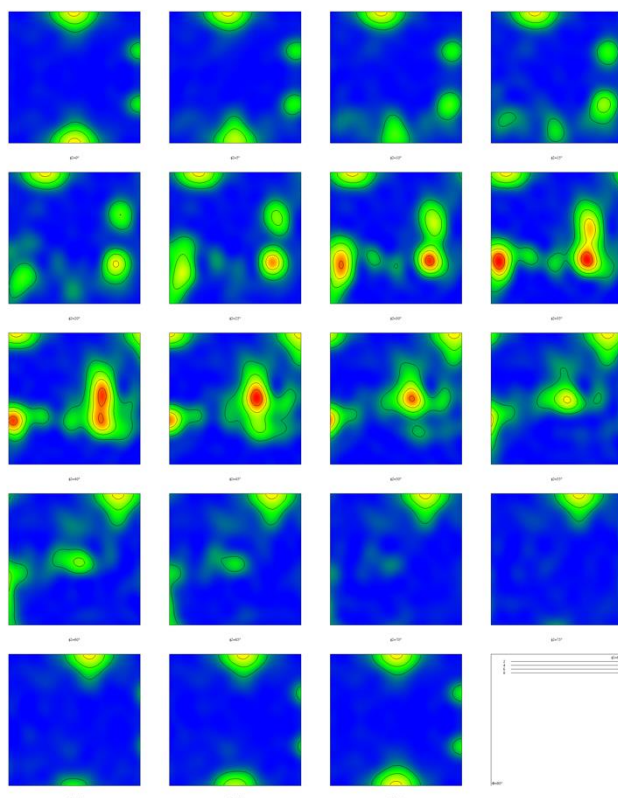


γ

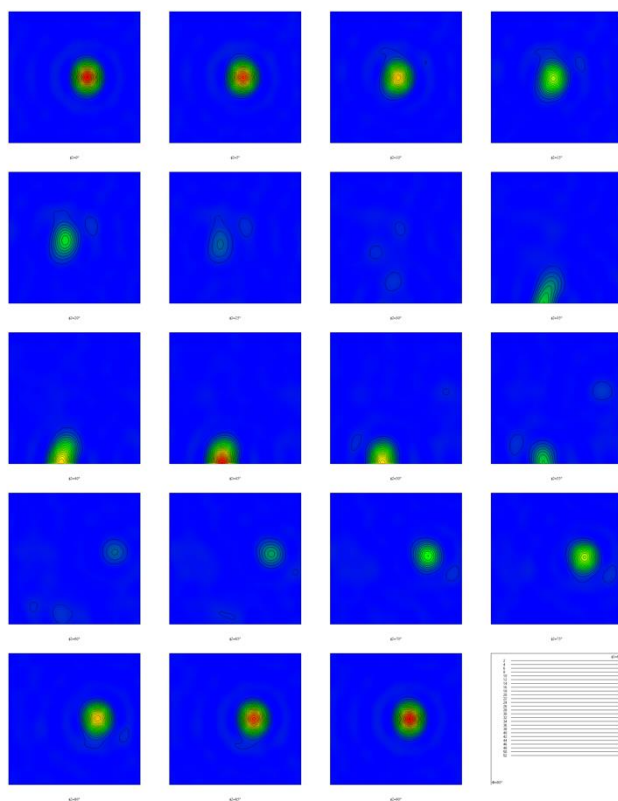


The FeCMnAl steel - IA at 650°C, 10 min - 20% TD cold-rolled

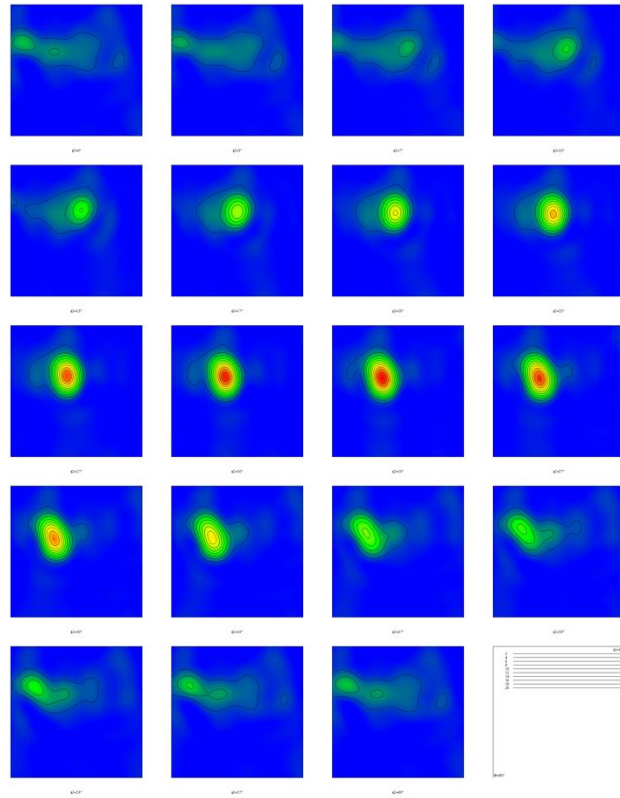
α/α'



γ

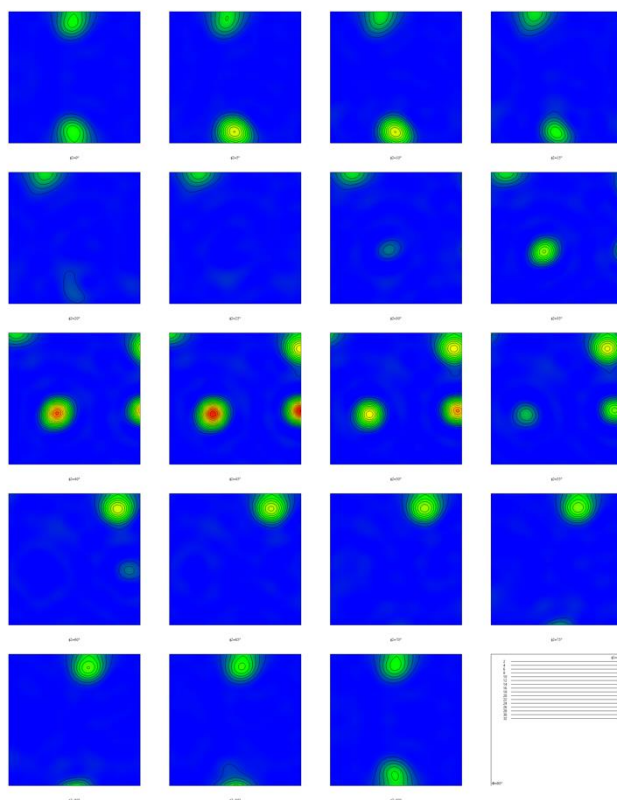


ε -martensite

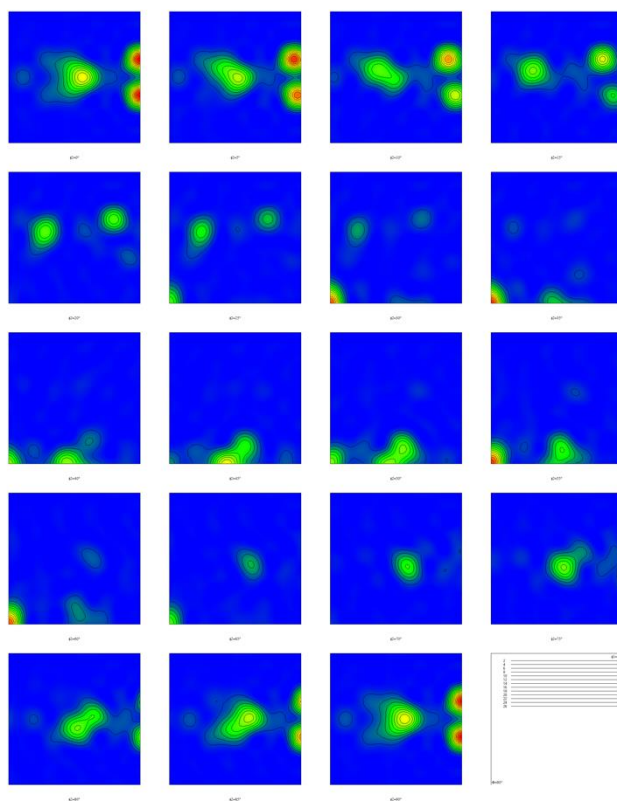


The FeCMnAl steel - IA at 650°C, 10 min - 30% TD cold-rolled

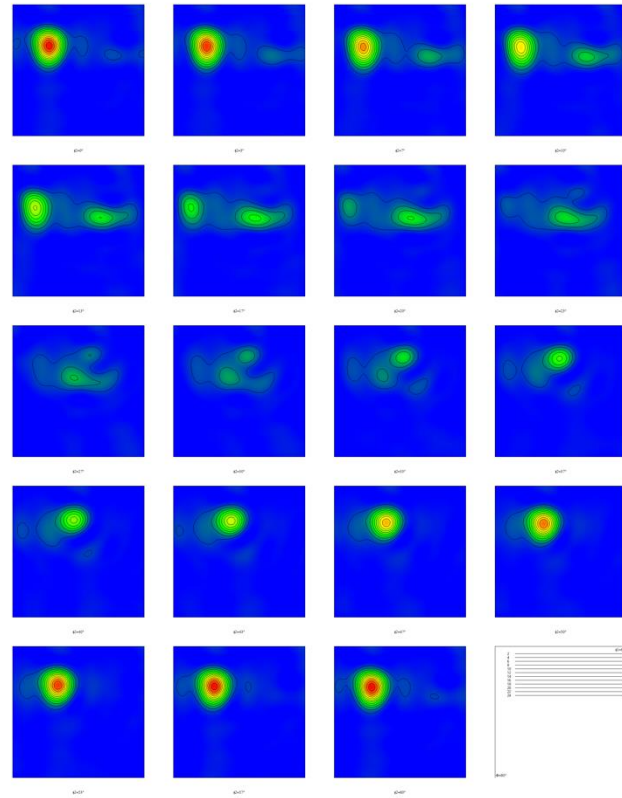
α/α'



γ

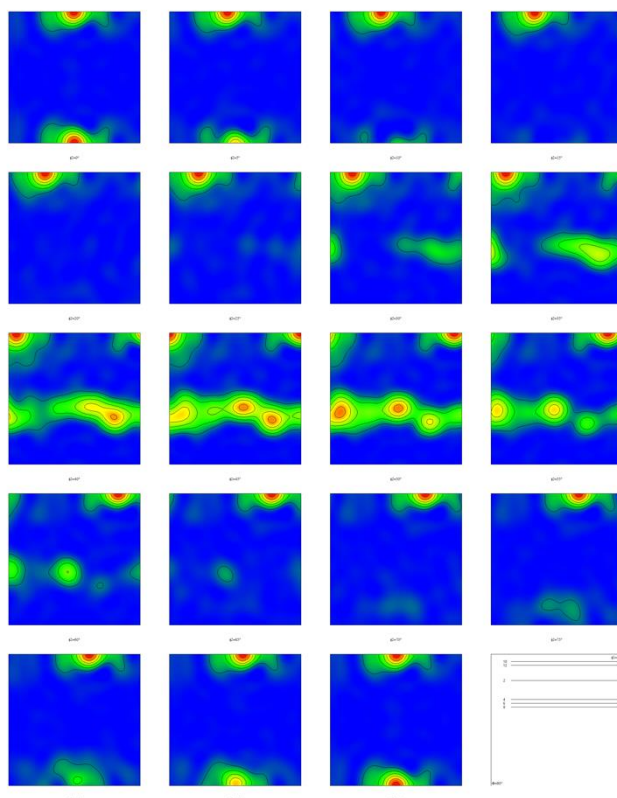


ε -martensite

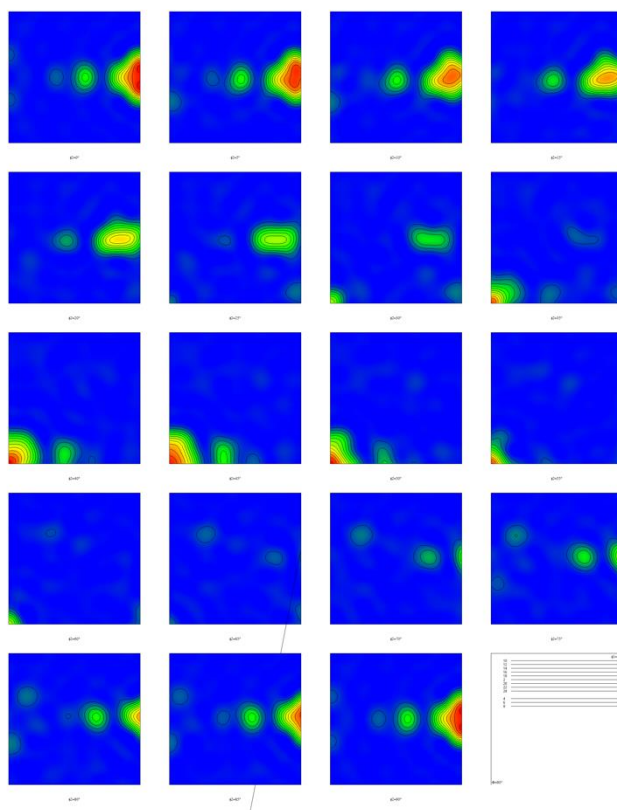


The FeCMnAl steel - IA at 650°C, 10 min - 50% TD cold-rolled

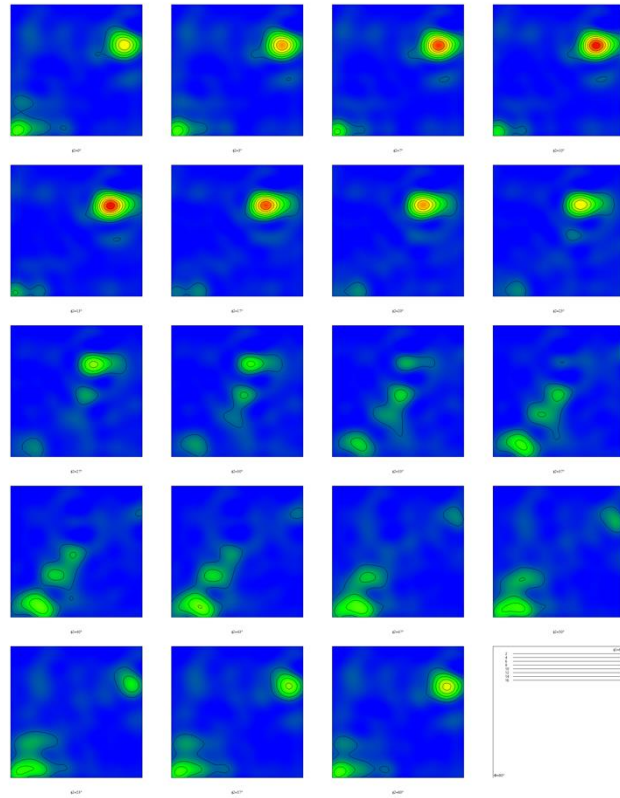
α/α'



γ

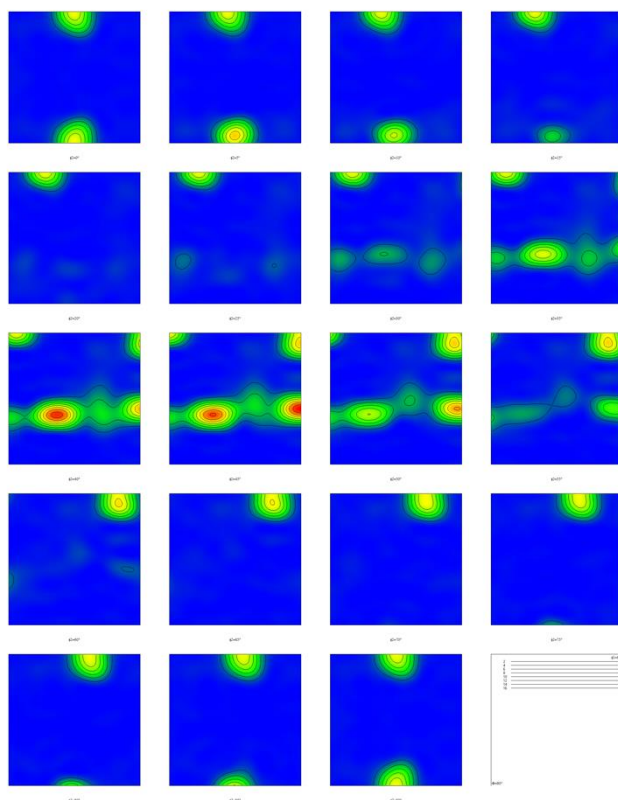


ε -martensite

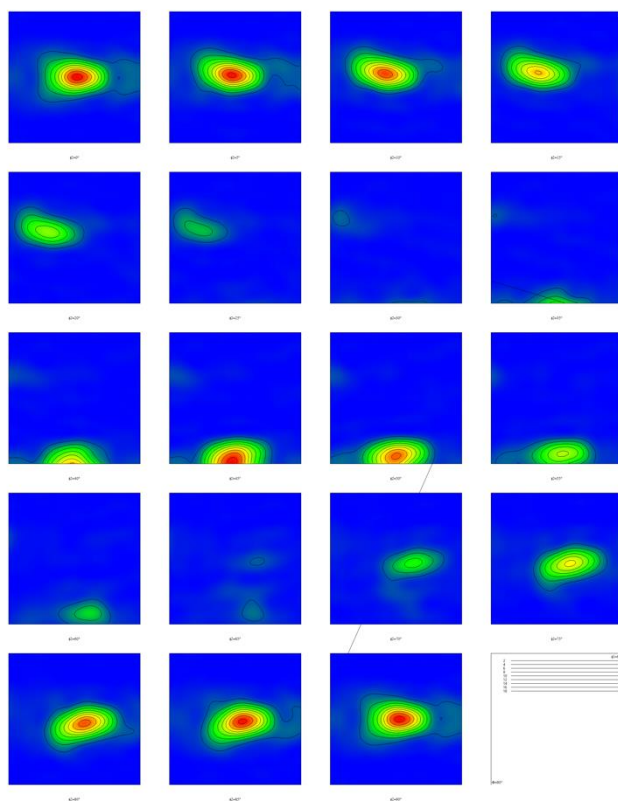


The 0.9 mm FeCMnAl steel - IA at 620°C, 5 min

α/α'

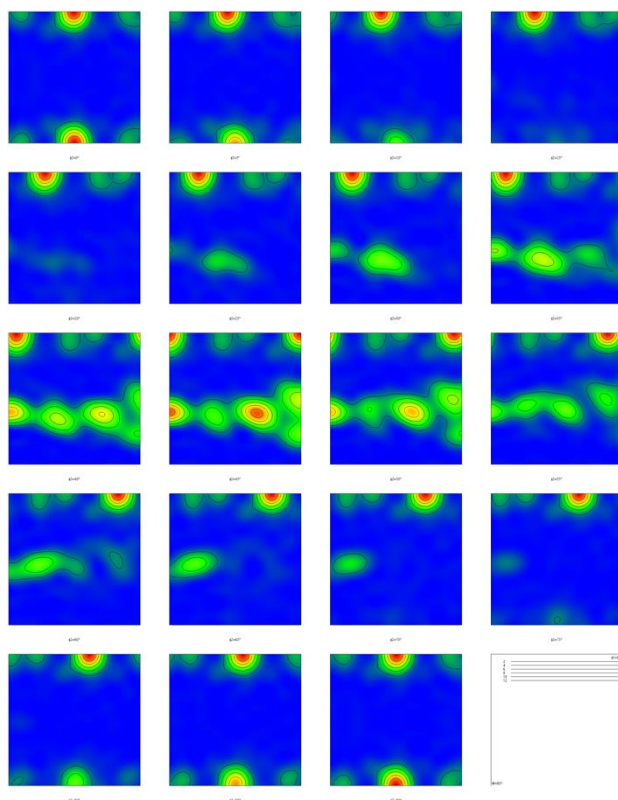


γ

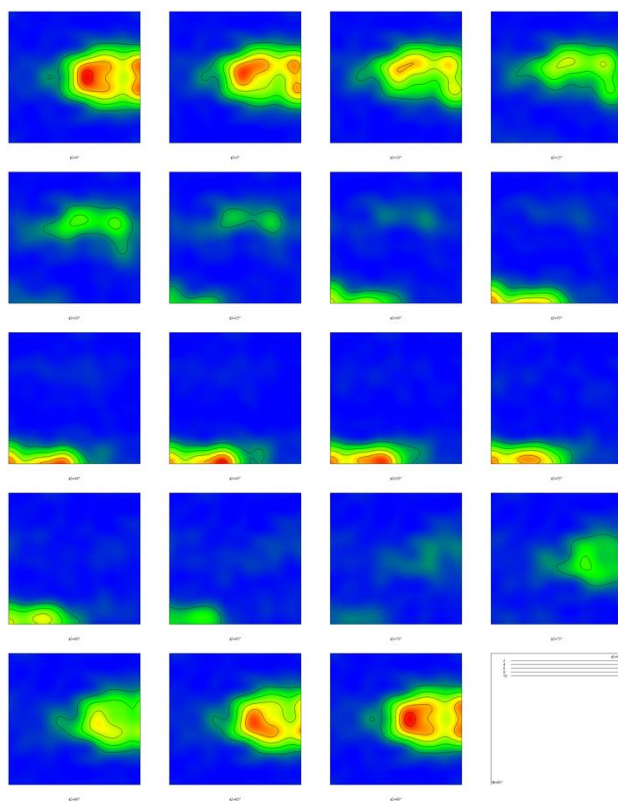


The 0.9 mm FeCMnAl steel - IA at 620°C, 10 min

α/α'

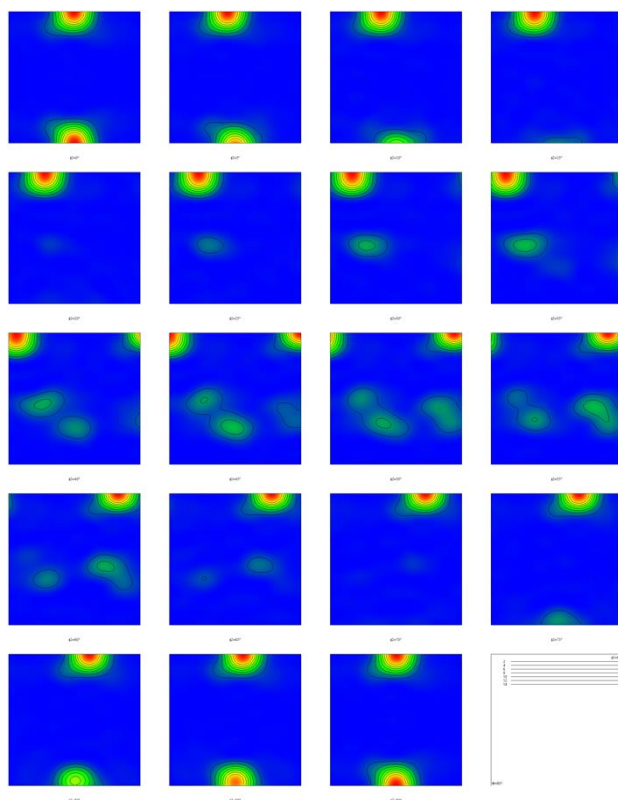


γ

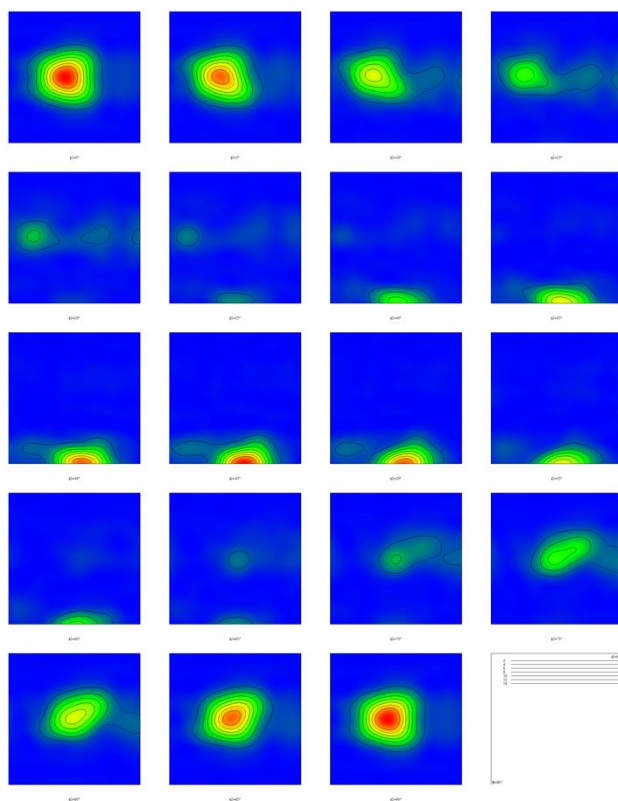


The 0.9 mm FeCMnAl steel - IA at 650°C, 3 min

α/α'

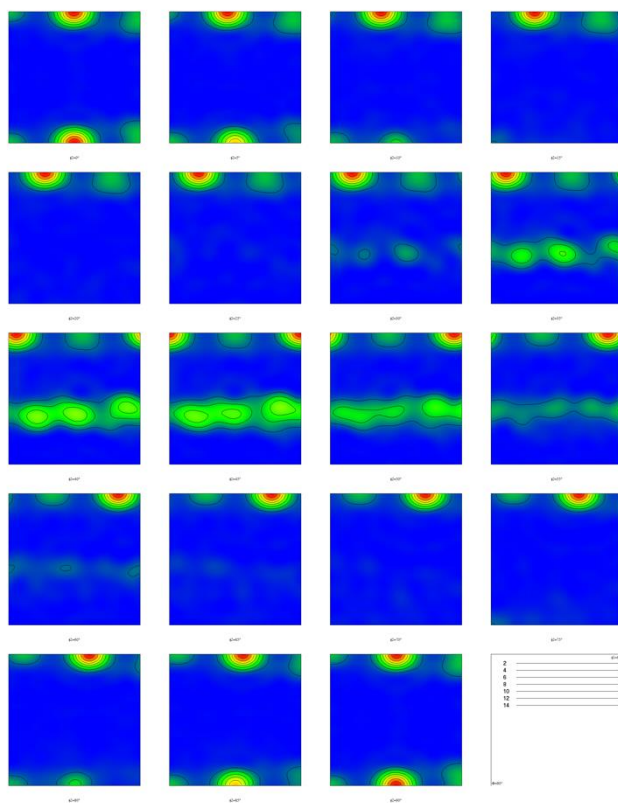


γ

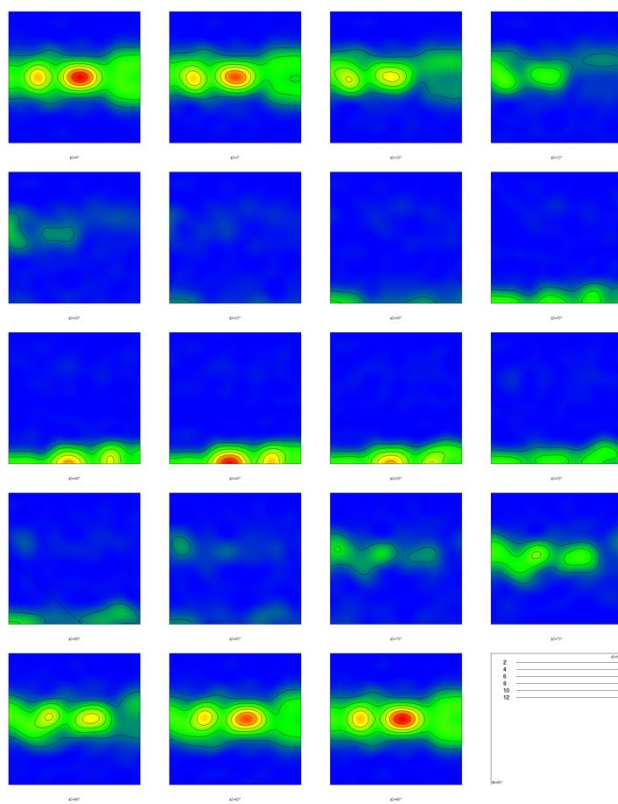


The 0.9 mm FeCMnAl steel - IA at 650°C, 5 min

α/α'

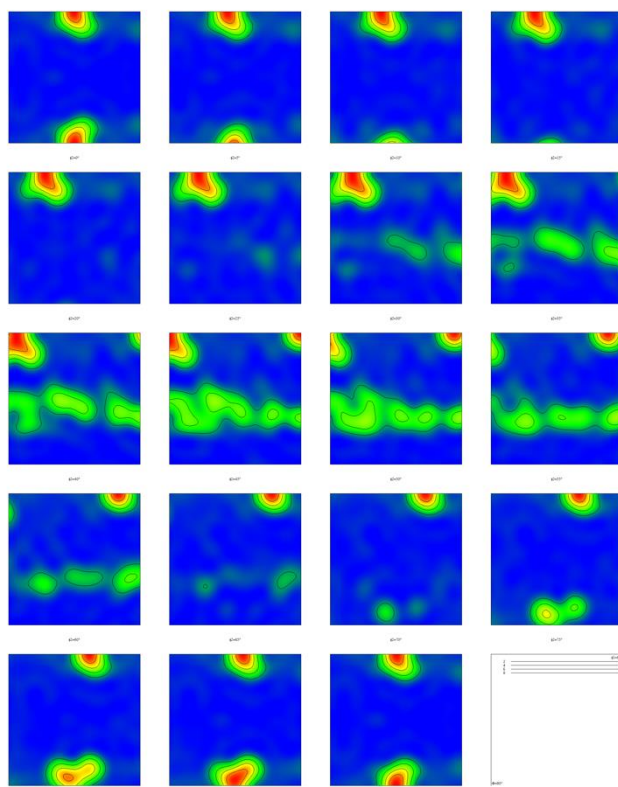


γ

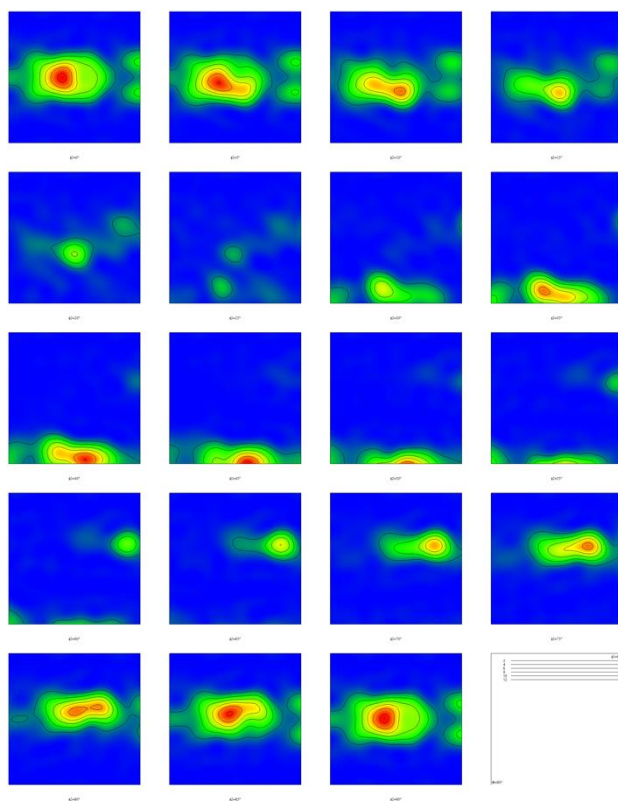


The 0.9 mm FeCMnAl steel - IA at 650°C, 10 min

α/α'

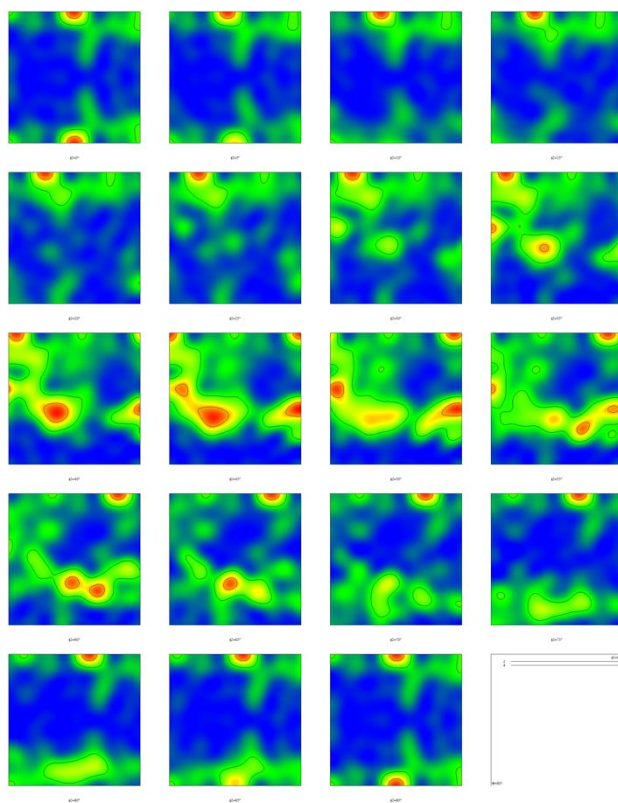


γ

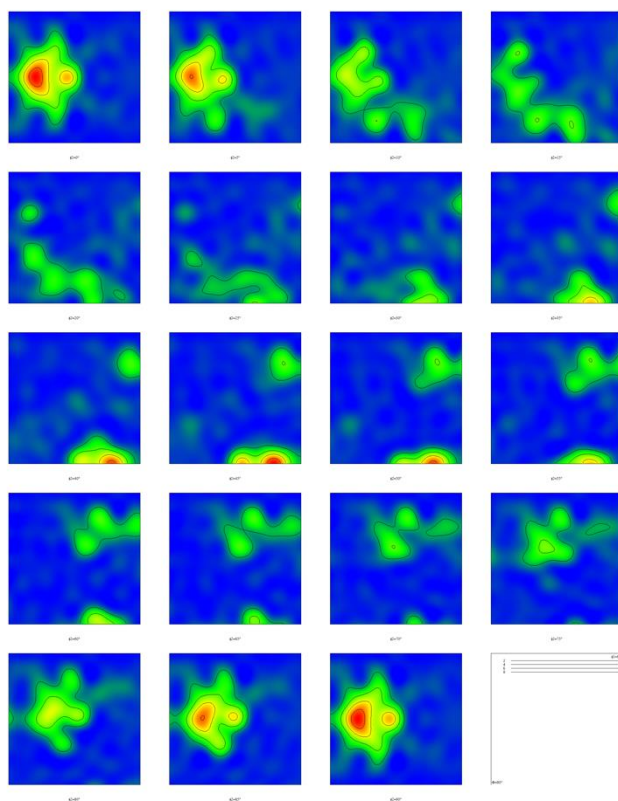


The FeCMnAl steel - annealed at 700°C, 3 min, quenched at 200°C and then in water

α/α'

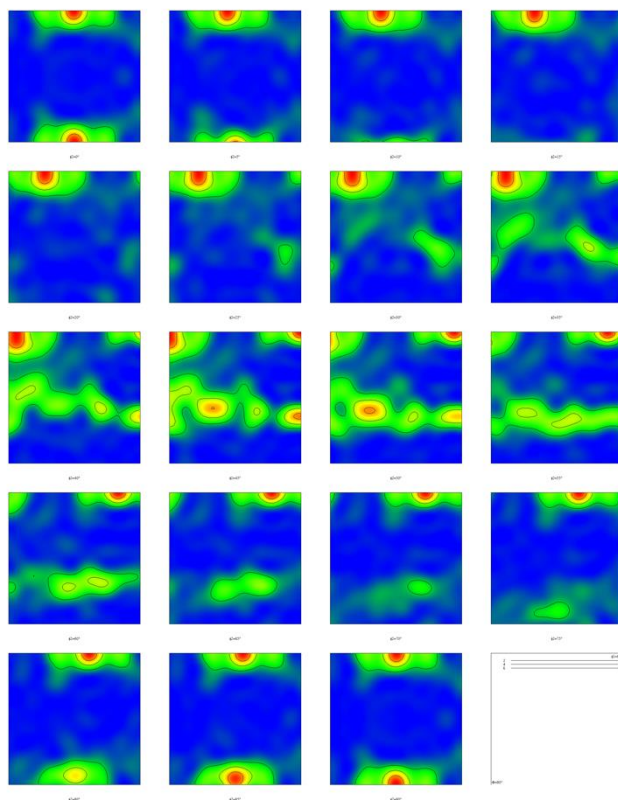


γ

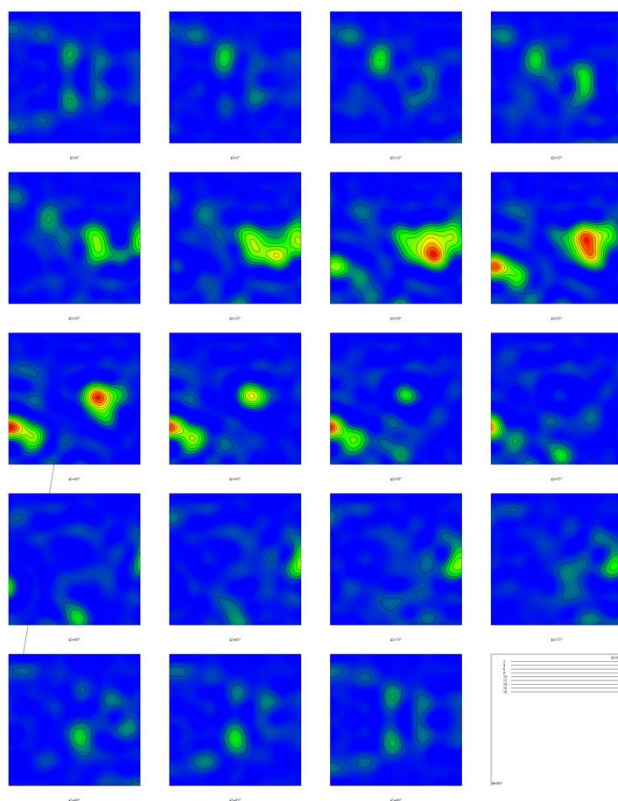


The FeCMnAl steel - annealed at 700°C, 3 min, quenched at 200°C and then in water – 5% cold-rolled RD

α/α'

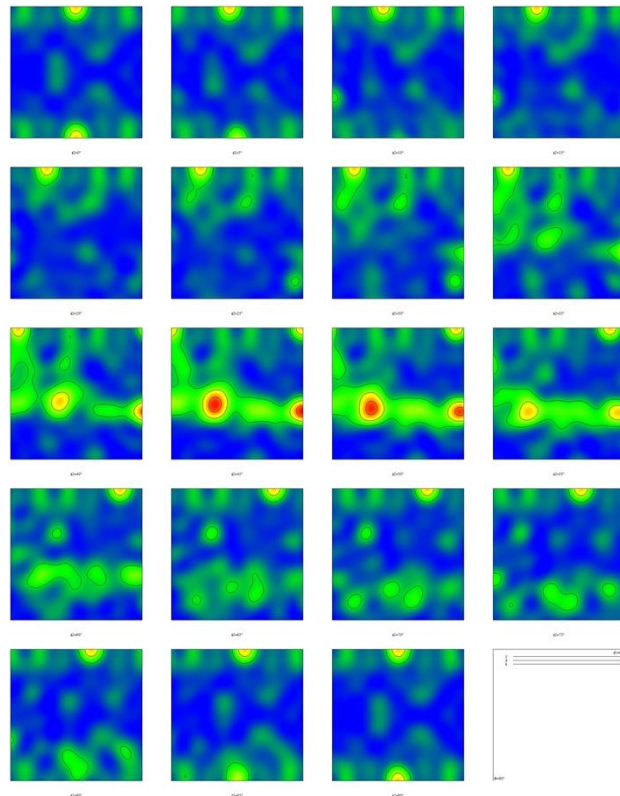


γ

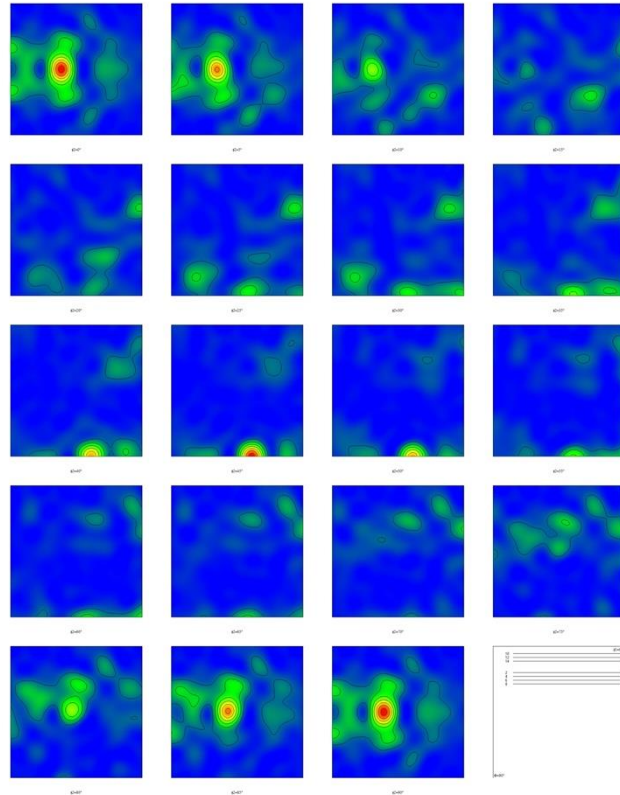


The FeCMnAl steel - annealed at 700°C, 3 min, quenched at 200°C and then in water – 5% cold-rolled RD - IA at 650°C, 10 min

α/α'



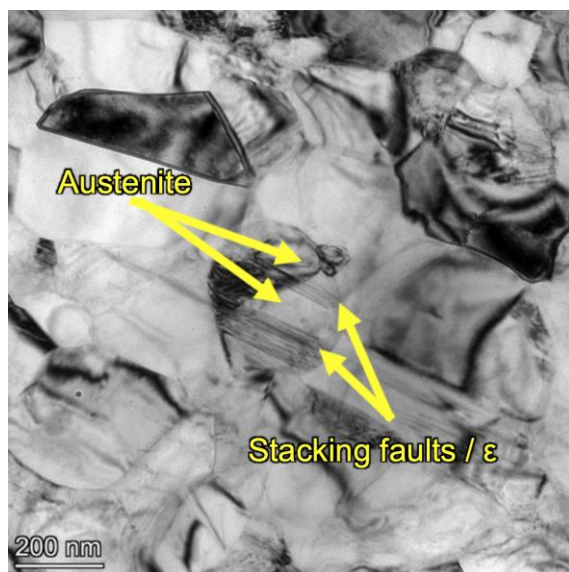
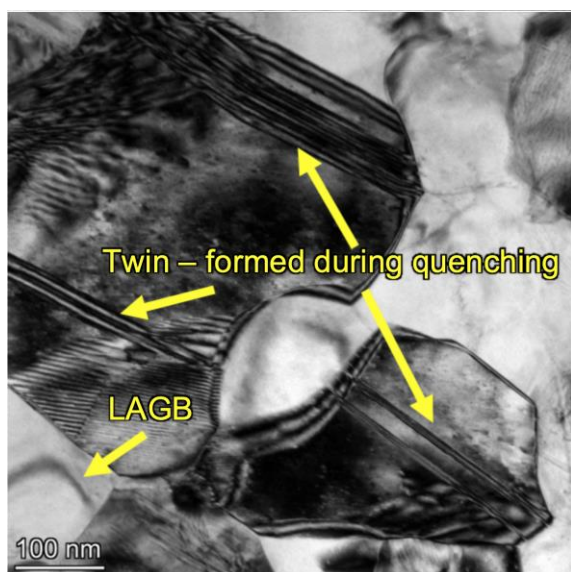
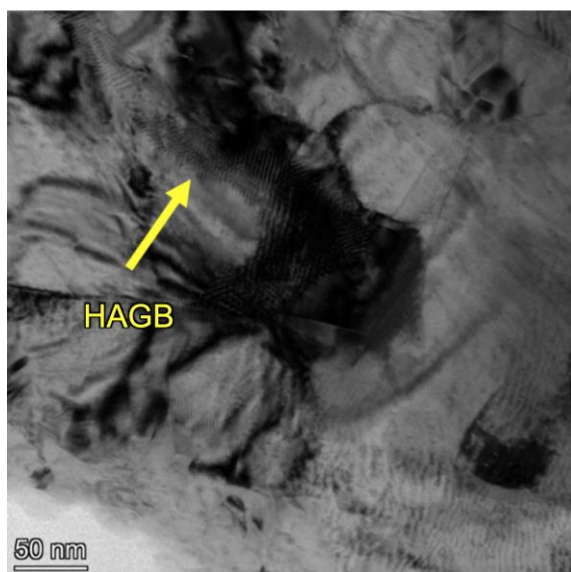
γ

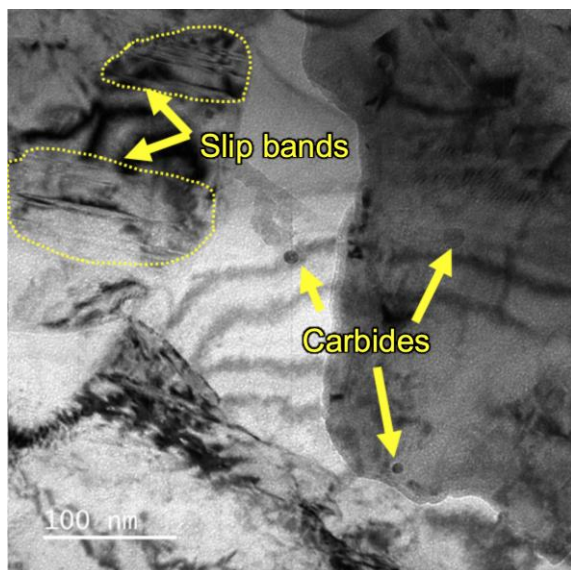


Appendix 4 : Transmission electron microscopy images of the FeCMnAl steel specimen during the interrupted tensile test

This appendix provides the TEM image of the FeCMnAl steel specimen during the interrupted tensile test. The specimen was subjected to the TSTMP which includes: (1) the IA at 650 °C for 10 min, (2) 50% TD cold rolling and (3) the IA at 650 °C for 10 min.

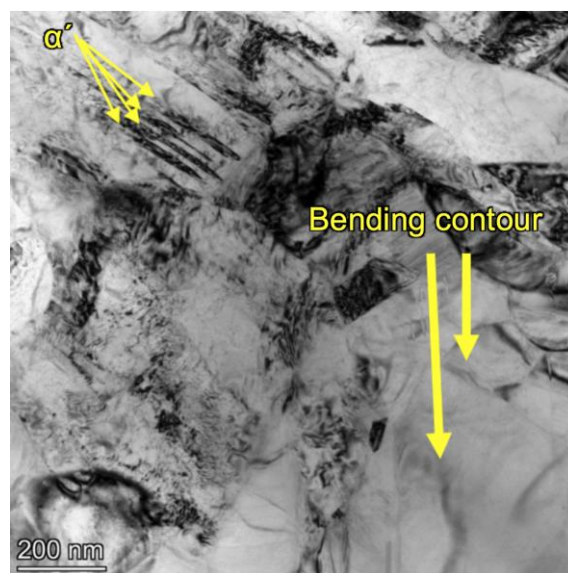
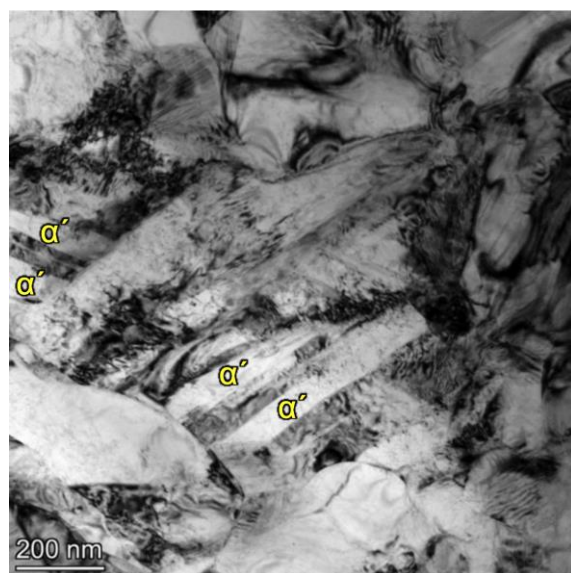
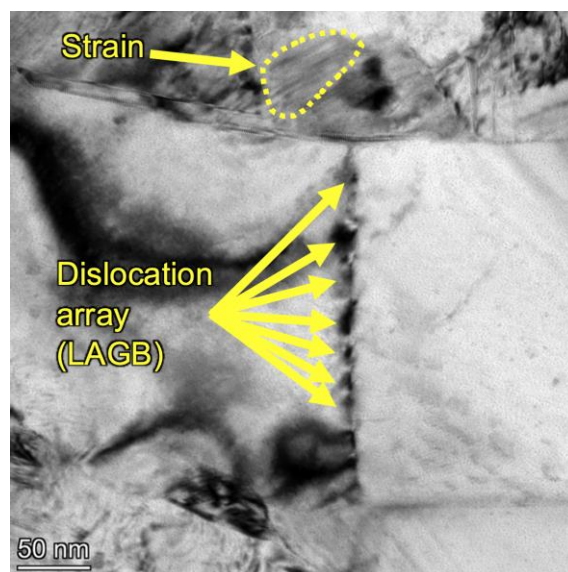
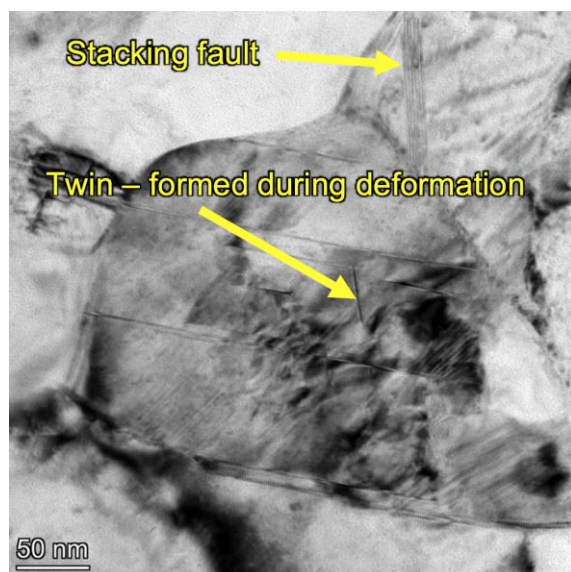
- Strain: 0%



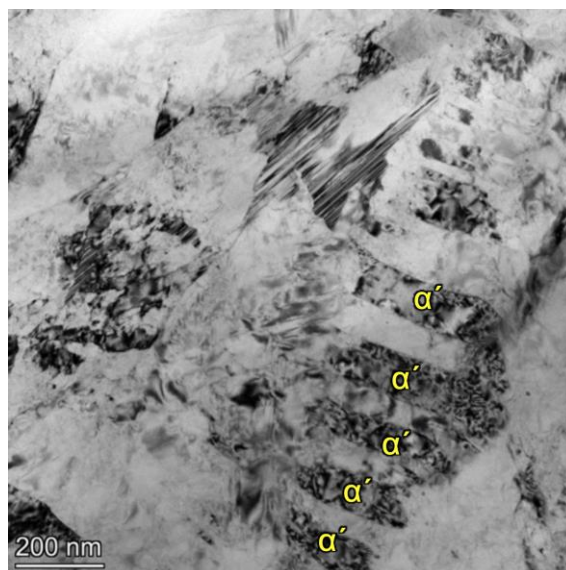
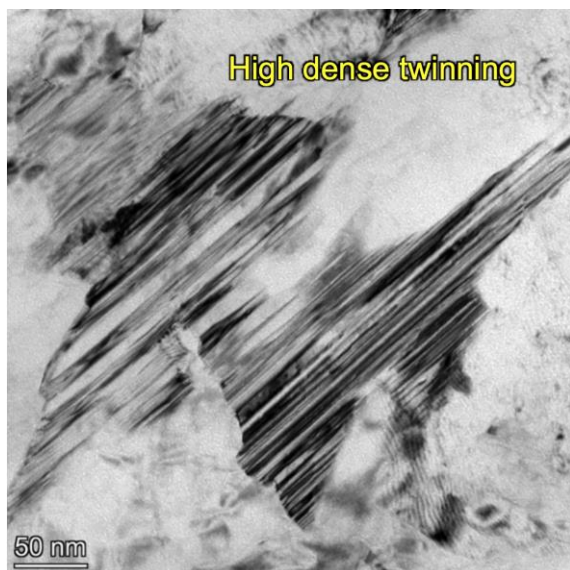


- Strain: 0.7%





- Strain: 6.5%



- Strain: 24.4%

