

The Investigation of the Microstructure and Related Mechanical Properties of Stainless Steel Orthodontic Wires

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Dedication

To my husband John whom I love and admire. He supported me consistently throughout the whole thesis and especially at the more difficult stages.

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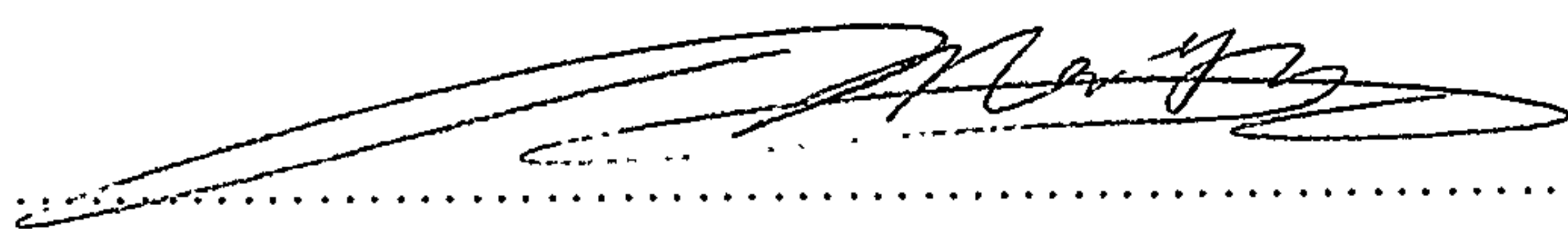
I also would like to give my many thanks to my dear husband John, without whose unending patience, support and devotion, nothing of this venture would have been possible.

Declaration

CANDIDATE'S CERTIFICATE

This is to certify that the work in this thesis was carried out by the candidate while a student at the University of Sydney, and has not been submitted to any other university or institution for a higher degree.

I also certify that the thesis has been written by me. Any help that I have received in my research work and the presentation of the thesis itself has been acknowledged. In addition, I certify that all information sources and literature used are indicated in the thesis.



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Abstract

In this thesis, stainless steel orthodontic wires are examined from the perspectives of both their microstructure and their mechanical properties. TEM and XRD analyses were conducted to determine the microstructure differences between four wires. In the process of this, it was necessary to introduce a new method for XRD analysis when measuring the texture and amount of martensitic phase in very orientated materials such as these wires.

Tensile, 3-point and 4-point bending tests along with SEM analysis provided details of the mechanical properties of the wires. Some anomalies observed in earlier literature about the value of the elastic modulus were resolved in terms of experimental error.

The mechanical properties varied between the wires even though they were all the same composition. These variations could however be explained using the results from the TEM and XRD tests and observing the differences in microstructural features such as the level of twinning, grain size and shape, texture and the amount of martensitic phase.

Chapter 1 Introduction.

This thesis analyses the microstructure of orthodontic wires and their related mechanical properties based upon material science, physics and metallurgy approaches. The rationale for this is that although the application of dental wires is a clinical consideration, their application requires specific wire properties and these properties depend heavily upon the wires' microstructure and mechanical properties.

As discussed by Whittle[1.1], in clinical procedures "apparently standard techniques produce erratic clinical performances". This appears to be caused by the fact that "the critical relationship between the composition, structure, and the attendant properties is currently not well understood." Whittle further remarks that "the relationship is not only complex, but difficult to investigate"[1.1].

As a step in understanding this relationship, this thesis is the first to research the structure of orthodontic wires down to the finest scale so as to provide further insights into their mechanical properties which are sensitive to changes at this level. This thesis is also the first to combine the techniques of transmission electron microscopy analysis (TEM), scanning electron microscopy analysis (SEM), X-ray diffraction analysis (XRD) and mechanical properties tests together to explain the issue of the relationship of microstructure and the wires' mechanical properties based upon material science and metallurgy theory.

In orthodontic application, wires need to have properties such as high strength, a high range of elasticity, high formability, resistance to corrosion in the oral environment, low surface friction and low cost. [1.2; 1.3].

The orthodontic wires used in this study are made from the 18Cr-8Ni 302 austenitic stainless steel. This type of wire satisfies most of the above requirements including high strength, a high modulus of elasticity, good resistance to corrosion and moderate cost. Therefore, this wire has been popular in orthodontic applications since 1929.[1.4; 1.5]

The required properties are the result of the basic wire drawing procedure and the composition of the wires. Because the stainless steel orthodontic wires researched in this thesis have a standard composition (18Cr-8Ni austenitic stainless steel), the different mechanical properties of different sizes of orthodontic wires heavily depend on the effect of the microstructure. One of the main factors controlling this is the forming process.

302 austenitic stainless steel orthodontic wires are produced by several cold drawing processes combined with intermediate annealing heat treatments. This manufacturing process introduces several distinct phenomena of microstructure changes such as elongated grain shape, reduced grain size, texture, martensitic phase transformation, high dislocation density, twinning and small angle boundaries. The combined effects of these changes decides the mechanical properties of the wires.

Therefore, microstructure research for orthodontic wires plays a very important role to examine and explain materials behaviour. Moreover, microstructure research provides the basic information for the manufacturer to control the manufacturing process in order to improve the required properties of the wires.

On the other hand, the mechanical properties tests determine the characteristics of orthodontic wires and hence these tests provide a basis for

the comparison and choice of these wires in the clinical applications. And also, the results of the mechanical properties will create a useful frame work for the microstructure investigation.

In order to bring together these results, the main body thesis is set out in 5 chapters, three of which summarise the different styles of experiments (mechanical property tests, SEM, TEM and XRD). Another chapter summarises the current literature in order to set the thesis into the context of the current research environment. The final chapter summarises the results from the three experimental chapters and states the conclusions of the thesis.

References for chapter 1:

- [1.1]. S. L. Whittle, An Investigation into Physical and Microstructural Properties of Orthodontic Wires and Changes Incident to Bending, Ph.D Thesis, University of Adelaide, Australia, 1995.
- [1.2]. P.C. Kesling, C.K. Kesling and A.J. Wilcock, JR. J Clinical Orthodont/interviews, on orthodontic wires. Journal of Clinical Orthodontics, March 1988, 22 (8): 484-489.
- [1.3]. W.R. Proffit, Contemporary Orthodontics, Second Edition, Mosby, St. Louis, 1993, 289-300 and 470-477.
- [1.4]. J. V. Wilkinson, Some Metallurgical Aspects of Orthodontic Stainless Steel, Angle Orthod. 1962, 48: 192-206.
- [1.5]. R. C. Thurow, Edgewise Orthodontics, Third Edition, Mosby, St. Louis, 1972, 42-71.

Chapter 2 Literature review.

2.1. Introduction.

This chapter reviews the historical literature on stainless steel orthodontic wires and summarises the theory to be used as a basis for the analysis in later chapters.

The main focus in this study is the investigation of the microstructure and the related mechanical properties of different diameter 302-type stainless steel orthodontic wires. One of the main factors affecting the microstructure and their mechanical properties is the forming process since all the orthodontic wires investigated in this thesis have the same composition. So as to better understand the deformed microstructure and the related properties of the orthodontic wires, this chapter describes the wire manufacturing procedure and the required properties for orthodontic wires.

The wire drawing manufacture process involves a plastic deformation process and an intermediate heat treatment, which introduce the change of microstructure and properties. This change can improve the microstructure and the properties such as strength of materials, but can also bring some disadvantages such as brittleness.

In this chapter, elementary plastic deformation theory will be reviewed, in order to help us to understand the phenomena occurring during the wire drawing process. In this section, single crystal behaviour is used as a stage to understand the deformation of polycrystalline aggregates.

Mechanical properties are usually achieved by the combined use of several strengthening mechanisms. Since all the wires researched in this thesis are composed of 302 stainless steel, the discussion will focus on several basic ways in which steel can be strengthened by deformation. This strengthening mechanisms will help us to understand the main reason why wire composed of the similar material but, with different diameters, have different strengths.

Since the wire manufacturing process involves intermediate heat treatment, this chapter will also briefly discuss the basis of heat-treatment. This discussion will focus on how annealing and deformation combine to result in the properties expected during the wire drawing process.

2.2 History of Stainless Steel Orthodontic Wires.

2.2.1. The introduction of stainless steel wires.

Some important stages for the first use of orthodontic wires are: [2.1-2.21]

In 1746, Fauchard fabricated orthodontic appliances consisting of short strips of silver or gold tied to the teeth with lengths of waxed thread [2.1].

In 1903, Edward Hartley Angle introduced the “E” arch constructed of heavy round German silver wire [2.1].

Calvin Suveril Case experimented with the use of light round wire for tooth movement but the properties of the alloys made treatment very difficult [2.4].

Da Costa in France introduced the use of stainless steel which had been developed by Brearly of Sheffield in 1913. Under Sheldon Friel's support, the use of stainless steel in orthodontic wires became fully accepted. Friel, a

graduate of Angle's school, observed that stainless steel was superior to precious alloys in terms of cost, tensile strength and toughness. Moreover, it was less likely to tarnish in the mouth or to retain debris due to 'miniaturisation' Friel recommended austenitic stainless steel since cold work could then be used to harden the material. The disadvantage of stainless steel was, however, that it was difficult to manipulate in comparison to precious alloys [2.4],[2.21].

2.2.2. The Australian Experience: Wilcock's Wires [2.4]

The first Australian stainless steel orthodontic wires were produced during World War II when Japan's entry into the war threatened supply from the US. A committee of dentists and metallurgists was formed to consider Australian manufacture [2.4].

This committee included Mr C.A.J. Wilcock who after the war set up *A.J Wilcock Scientific and Engineering Company*.. which produced (amongst other things) high tensile orthodontic wires.

In the late 1960's, Mr A.J. Wilcock joined his father's company. He changed the source of raw materials to overseas suppliers so as to receive better analysis of the raw materials. With this improved analysis, he developed higher tensile wires ("premium grade"). Since this premium grade wire exhibits the least relaxation, it is still used in orthodontics.

2.3. The alloying element effect in 302 stainless steel and their strengthen mechanism.

2.3.1. Alloying element effect in 302 stainless steel.

The orthodontic wires investigated in this thesis are all made of 302, 18-8 stainless steel. The details of this composition are given in table 2.1. Here, we shall consider the effect of different alloying elements upon the properties of the stainless steel and their strengthening mechanism.

Table 2.1: The compositions of Wilcock wires (type W) (Victoria, Australia) and Dentaurem wires (type G) (Dentaurem, Pforzheim, Germany):

Element	Type 302 composition (weight percent)		
	AISI (ASTM,1990)	Wilcock Wire (Newman, 1963)	Dentaurem Wire
Carbon	0.15 (max)	0.095	0.12
Manganese	2.00 (max)	1.17	2.00
Phosphorous	0.045 (max)	0.022	0.045
Sulphur	0.03 (max)	0.032	0.03
Silicon	1.00 (max)	0.48	1.50
Molybdenum	-	0.21	0.8
Nitrogen	0.01 (max)	-	-
Chromium	17.00-19.00 (max)	18.21	16.00-18.00
Nickel	8.00-10.00 (max)	8.97	6.00-9.00

To produce stainless steel, the chromium content must exceed 11%. If the chromium content is greater than 11% small amounts of carbon are needed in order for it to remain 'stainless'. [2.22]

The presence of chromium in the alloy also contributes largely to the corrosion resistance of stainless steel. No other element added to iron is as effective as chromium in producing resistance to corrosion. [2.22]

Chromium resists corrosion well because of the formation of a strongly adherent oxide coating on the surface which prevents further reaction with the metal below the surface. The formation of such an oxide layer is called passivation. [2.22]

The surface coating is not visible even at high magnification, but the film adds to the metallic lustre of the metal. The degree of passivity is influenced by a number of factors, such as:[2.22]

- alloy composition;
- heat treatment;
- surface condition;
- stress; and
- the environment.

In dental applications the stainless or corrosion resistant characteristics of the alloys can therefore be altered, or lost, by excessive heating during assembly, or by using abrasives or reactive cleaning agents which can alter the surface conditions of the appliance and even by poor oral hygiene practices over prolonged periods [2.23].

The effect of the nickel addition to the austenite class of stainless steel is to stabilise the face-centred structure at room temperature. Other trace elements

are also usually present in stainless steel alloys. These include carbon, nitrogen, silicon, phosphorus, titanium, niobium, manganese, cobalt, nickel, or molybdenum. Each of these elements has the capacity to alter the properties of the steel, such as the strength, the weld-ability, or the effects of heat treatment. Their effect depends on both the concentration and interactions between the elements.[2.25]

2.3.2. Solid solution strengthening mechanism:

The introduction of solute atoms into solid solution in the solvent-atom lattice invariably produces an alloy which is stronger than the pure metal. This type of strengthening is achieved by alloying a metal with appropriate addition of other metallic elements. The host metal is called the solvent, and the alloying element the solute. The solute atoms may form either *substitutional* or *interstitial* solid solution. Alloys are stronger than pure metals because solute atoms ordinarily impose localised lattice strains as their atomic radius would be different than that of the host metal. Consequently dislocation movement is restricted.[2.24]

Carbon is distributed in the inter-atomic spaces, but may also form carbides, depending on the prior history of the alloy.[2.24] The actual hardness attained depends entirely on the amount of carbon in the steel: the more carbon, the harder and stronger, but the product will be less ductile.[2.24]

Carbon and nitrogen atoms enter an iron crystal lattice at a rate that is determined by the available interstitial space, but generally occupy the smaller octahedral rather than the larger tetrahedral interstices, as this requires less strain energy in the movement of nearest neighbour iron atoms.

This can substantially increase the strength of iron and form a steel known as solid solution strengthening by interstitial atoms. [2.26]

Larger alloying elements such as chromium, manganese or nickel have an atomic size approaching that of the iron, and as such become part of the lattice by substitutional incorporation. This has been called solid solution strengthening by substitutional atoms[2.26].

2.4 The stainless steel orthodontic wires used in this investigation.

The investigation in this thesis were carried out on two different brands of orthodontic wires, referred as Wilcock's wires (type W wires) and Dentaurem wires (type G wires) as both of them are made from 18Cr8Ni, 302 type stainless steel. These wires are manufactured in Australia and Germany respectively. The reason for choosing these two brands of wire is that we wished to research why their properties are different despite being composed of the same material.

Type W wires: These have diameters of 0.52 mm (WF) and 1.62mm (WS). The 0.52mm diameter wire is the one used in orthodontic applications. The 1.62mm diameter wires is the starting material before extrusion to form the 0.52mm diameter wire.

Type G wires: These have diameters of 0.88 mm (GF) and 2.18 mm (GS). The 0.88mm diameter wire is the one used in orthodontic practice. The 2.18mm diameter wires is the starting material before extrusion to form the 0.88mm diameter wire.

The composition for type W wires and type G wires are provided by their respective manufacturers and are listed in above table 2.1.

2.5 Required properties of orthodontic wires.

Wilcock[2.3] states that the current approach of many Australian orthodontists is to use extremely light forces to facilitate optimum tooth movement. Accordingly, the orthodontic wires which they use should meet the following requirements:

- they should resist the forces of mastication;
- they should provide a light, continuous force over large displacements without undergoing permanent deformation;
- they should possess good formability and flexibility – they should flex easily without taking a set, should be formable into arch shapes with relative ease, should be resilient and should have good springback; and
- they should possess good biocompatibility and zero stress relaxation properties.

These demands invariably involve balancing metallurgically opposing properties: flexibility, formability and hardness. Wilcock [2.2] observes that flexibility (elastic strain) and resiliency (energy stored per unit volume) are considered the most important properties in the production of orthodontic wires.

Proffit[2.4] states that ideal orthodontic wires should move teeth by light continuous forces. They should have the properties of:

- low stiffness;

- high strength;
- a high range of elasticity;
- high formability;
- being weldable or solderable;
- being resistant to corrosion in the mouth;
- being biocompatible and environmentally stable;
- having low surface friction [2.5], [2.6]; and
- being of low cost.

There is no universal agreement on which properties or tests are most important in assessing orthodontic wires, but tests of elastic behaviour are generally agreed to be the most relevant [2.7].

Elasticity is, however, described in a variety of terms and measured with a wide range of tests [2.7]. Many of these tests enable comparison of wires but may not be clinically relevant [2.5]. Those properties which impact on the clinical application must be isolated.

Accordingly, the main clinical characteristics are described below. These characteristics are dependent on each type of wire. They should therefore be examined separately when selecting wires for a specific application. [2.8], [2.10].

Stiffness is an indication of the rate of force delivery, whereas strength and range are a measure of the maximum capacity of the wire [2.8]:

$$\text{Stiffness} \propto \frac{\text{Load}}{\text{Deflection}}$$

Flexibility is a measure of the extent to which a wire can be strained without undergoing plastic deformation [2.11], [2.12]

$$\text{Flexibility} = \frac{\text{Yield Stress}}{\text{Elastic Modulus}}$$

Resiliency (or more precisely the modulus of resiliency) of a wire is the amount of strain energy which can be stored per unit volume without permanent set. [2.2]:

$$\text{Modulus of Resiliency (per unit volume)} = \frac{(\text{Yield Stress})^2}{\text{Elastic Modulus}}$$

Zero stress relaxation is the ability of a wire to deliver a constant light elastic force when subjected to an external force or to forces of occlusion [2.8].

Zero stress relaxation property requires that the material yield stress be as high as possible. Relaxation in a material implies that a form of slip movement by dislocation is occurring. This relaxation can be related to the internal frictional force so as to overcome the movement of the dislocations.

This property indicates that the wire should have a very high and sharp yield point with low elongation and will depend on the thermomechanical production during manufacture. [2.2]

Formability is the ease with which a material can be plastically deformed. It can be related to the percentage elongation a wire can undergo before fracture. [2.2]

Biocompatibility is the extent that the material co-exists within the body. Stainless steel has good corrosion resistance in the body. [2.2]

2.6 The Wire Drawing Manufacturing Process.

Stainless steel orthodontic wires are generally made from 18-8 (i.e. 18% chromium and 8% nickel) austenitic stainless steel. The strength and hardness of these wires is increased by a reduction in diameter through cold drawing during wire formation.

In wire-drawing operations, metal is pulled through a die by means of a tensile force applied to the exit side of the die. Most of the plastic flow is caused by the compression force due to the interaction of the metal with the die.

The following description of the wire drawing process is based upon Dieter[2.14] and Dixon[2.1]

Wire drawing is usually carried out at room temperature. However, because large deformations are usually involved, the temperature rises considerably during the drawing operation. Some intermediate annealing heat treatments may also be employed between passes of drawing so as to lower the strength of the material in order to enable further deformation.

Wire drawing usually starts with a coil of hot-rolled rod which is first cleaned by pickling so as to remove any scale which would cause surface defects or excessive die wear.

The next step is to prepare the rod. With high-strength materials, a soft surface coating such as copper or tin may be used as a lubricant but, more typically, conversion coatings such as sulphates or oxalates are applied to the rod. These are used in conjunction with a lubricant.

Formation of defects in wires can result from the starting rod material or from the deformation process itself.

For fine wire, such as orthodontic wire, a number of draws are necessary through a series of dies with progressively decreasing diameters. To begin drawing, the end of the wire is pointed, often by electropolishing, passed through the die, and attached to a draw block, or capstan, that is part of a drawing drum. The wire is then pulled by either a chain drive or a hydraulic mechanism.

Drawing speeds may vary from ten to one hundred metres per minute, depending on a number of variables including the characteristics of the die, the type of wire being drawn or the physical property requirements of the finished product.

The drawing force is the sum of three forces:

- the force required to decrease the diameter uniformly (i.e. tensile elongation);

- the force to produce non-uniform shear deformation of the surface layers at the entry to and exit from the die (i.e. redundant work); and
- the force required to overcome friction between the wire and the die wall.

The required force depends on four factors:

- die angle;
- percentage reduction;
- flow stress of the material; and
- die friction.

Die friction is, in turn, dependent upon three factors

- die material;
- lubrication; and
- drawing speed.

Most dies are made from tungsten carbide to enable a long die life. A typical die, as shown in figure 2.1, has a truncated conical shape. In the diagram, three main parts are marked:

- an entrance angle large enough for the undrawn wire and the lubricant that adheres to the die.
- an approach angle (or 'half die angle': α) where the actual reduction in wire diameter occurs; and

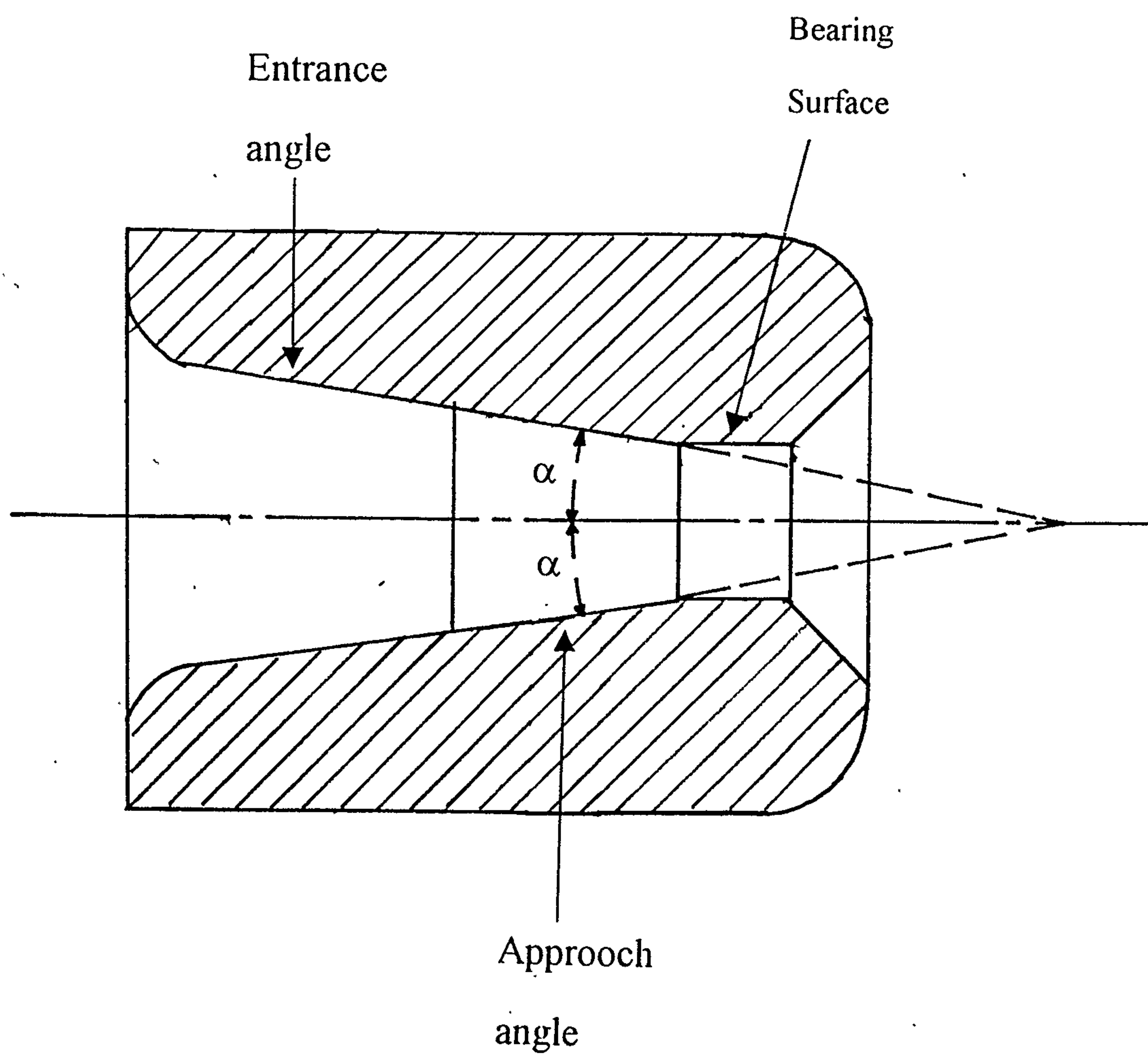


Fig. 2.1 Typical wire drawing die.

- the bearing surface of the die which acts as a guide for the wire exiting the die.

Varying the value of α alters the homogeneity of the surface of the drawn wire. For a given wire diameter, increasing the half die angle increases the amount of shear deformation in the direction opposite to the draw pull. Only the central axis of the wire undergoes pure elongation.

For large half die angles, large shear deformation results in an increased longitudinal tensile stress at the centre of the wire. This tensile stress can exceed the fracture stress, thereby resulting in a cupping type of failure.

For a given half die angle, as percentage reductions increase, the shear deformation becomes less important. Furthermore, because of increased surface shear deformation, the yield and tensile strengths of drawn wires are higher for larger half die angles. This effect on yield and tensile strengths is greater for lower reductions.

Although non-uniform shear deformation decreases with smaller half die angles, wall friction increases. This implies an optimal die angle for minimum drawing force for a given reduction. The existence of this optimal angle is due to the interaction of half die angle, reduction percentage, flow stresses, the type of material, lubrication, and friction.

All else being equal, the optimal half die angle increases with the amount of reduction. Drawing speed has little effect on the drawing force but may profoundly affect the temperature, thereby making lubrication more difficult.

Another factor is 'back-pull' in the opposite direction to the drawing pull. Back-pull can arise directly from frictional forces in a poorly lubricated

system. It might also, however, be deliberately applied in order to increase the life of the die by reducing die wall pressure and therefore reducing frictional forces. Increased back-pull decreases the maximum possible reduction in diameter.

Orthodontic wire attains anisotropy through cold drawing because of the re-orientation of the individual grains. Two distinct types of residual stress patterns may be seen, depending on the amount of reduction, where the reduction per pass is less than one percent:

- longitudinal residual stresses are compressive at the surface and tensile at the axis;
- radial stresses are tensile at the axis and drop off to zero at the free surface; and
- circumferential stresses follow the same trend as the longitudinal residual stresses.

This pattern is consistent with localisation of deformation in the surface layers of the drawn wire.

For larger reductions, the residual stress distribution is the complete opposite of the above pattern:

- longitudinal stresses are tensile at the surface and compressive at the axis;
- radial stresses are compressive at the axis; and

- circumferential stresses follow the same pattern as the longitudinal stresses.

It has been shown that for a given reduction, longitudinal residual stress increases as the half die angle increases. The maximum values of residual stress in cold drawn brass wires is obtained at a reduction of about 15% to 35%.

Much of wire drawing theory assumes a material exhibiting pure plastic deformation without friction. Even with the best lubricated die drawing system, this assumption is unrealistic in that it ignores the presence of friction and shear (redundant) deformation and only considers an idealised situation.

Theoretical analyses using slip field theory have not been applied to wire drawing and, since most theory is calculated with a grid system in two planes, these theories have only limited application to the actual process.

During manufacture, heating and cold drawing is repeated alternately until the wire attains its desired properties. During diameter reduction, the amount of heating at specified intervals depends on various factors, including the diameter of the raw material and the diameter of the finished product.

Heat is used to relieve stresses in cold worked austenite so as to improve tensile elastic properties such as proof stress or yield point. The improvement over untreated wires ranges from ten to twenty percent, allowing ease of forming for orthodontic purposes. [2.15].

If stainless steel wires are subjected to lower temperatures in the range 260 to 460°C for 0.5 to 75 minutes, wires can be tempered so that an increase of up to 25 per cent in (tensile) strength and in relative stiffness occurs. [2.16]

Heating above 400°C reduces the resistance of stainless steel wires to corrosion since the proportion of chromium diminishes in the solid solution adjacent to the grain boundaries due to the formation of chromium carbide at these boundaries. [2.17]

Heat treatment of stainless steel wires is frequently used in the manufacture of orthodontic wires since it enables large reductions in diameter thereby producing wires with improved strength.

An improvement in the properties of stainless steel wires occurs during heat treatment after manufacture, [2.18]. Any improvement in the elastic properties of stiffness and springback resulting from heat treatment, may be accompanied by a corresponding loss of formability or ductility. [2.19]

Severe cold work during drawing leads to the production of martensite from 'deformation induced' metastable austenite. This martensite may be subsequently transformed back to austenite during 'stress relief' heat treatment. [2.20]

This wire manufacturing process involves drawing and tempering which change the properties and microstructure of the wires. These changes will be discussed in later chapters (chapters 3,4,5) in the case of the orthodontic wires and their early stage wires.

2.7. The plastic deformation of metal.

2.7.1. The plastic deformation of single crystal metals.

2.7.1.1. Dislocations.

In a crystal which has undergone slip, lattice defects tend to accumulate along the slip planes. These defects are called dislocations. Dislocations are the conveyors of plastic deformation in metals. Edge and screw are the two fundamental dislocation types (Figure. 2.2) [2.40]

In an edge dislocation, the end of an extra half plane of atoms defines the dislocation line where localised lattice distortion also exists. [2.40]

A screw dislocation may be thought of as resulting from shear distortion; its dislocation line passes through the centre of a spiral, atomic plane ramp. [2.40]

Many dislocations in crystalline materials have both edge and screw components; these are called mixed dislocations. The dislocation line marks the boundary between the sheared and unsheared parts of the slip plane. This is a basic characteristic of all dislocations. [2.40]

Virtually all crystalline materials contain dislocations introduced during solidification, during plastic deformation, or as a consequence of thermal stresses resulting from rapid cooling. In other words, the increase in hardness or strength of a metal with deformation is closely associated with an increase in the concentration of the dislocations. Dislocations end at grain boundaries or at free surfaces. Heat treating a deformed metal specimen can diminish the density of these line defects.[2.41]

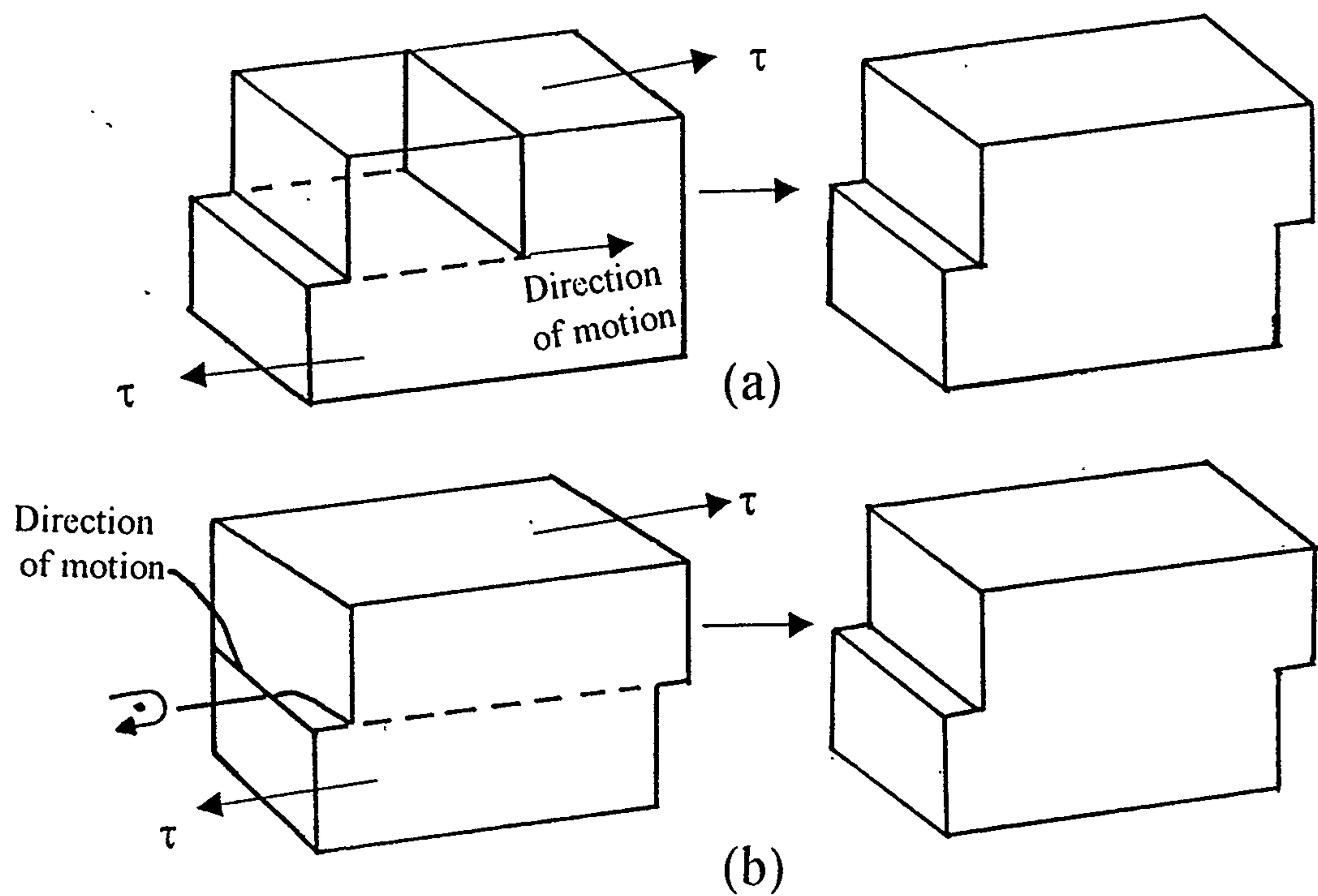


Fig. 2.2 The formation of a step on the surface of a crystal by the motion of (a) an edge dislocation and (b) a screw dislocation. Note that for an edge: the dislocation line moves in the direction of the applied shear stress τ ; for a screw, the dislocation line motion is perpendicular to the stress direction.

2.7.1.2. Slip

In cubic metals the two basic methods of deformation are slip and twinning.

Metal can only deform on the slip plane and along the slip direction. The slip plane results from microscopic shear movements, while the direction of shear in the shear plane is called a slip direction. The combination of a particular slip-plane and a slip direction in that plane is referred to as a slip system.[2.37]

In most metals, the plane on which the slip takes place is usually that with the closest atomic packing, while the slip direction is always the closest-packed direction in the slip plane.[2.37]

Slip basically refers to the movement of atomic layers against each other, and this happens by the motion of dislocations. All dislocations, regardless of their type, move in response to shear stresses applied along a slip plane and in a slip direction. Even when an applied stress is tensile or compressive, shear components exist in all but parallel or perpendicular alignments to the stress direction.[2.37]

Dislocations do not move with the same degree of ease on all crystallographic planes of atoms and in all crystallographic directions. Dislocation motion occurs along the slip direction on slip plane. The slip system depends on the crystal structure of the metal and is such that the atomic distortion that accompanies the motion of a dislocation is a minimum. [2.37]

Several slip systems may exist for a particular crystal structure (see table 2.2). The number of independent slip systems represents the different

possible combinations of slip planes and directions. Metals with face centred cubic (fcc) or body centred cubic (bcc) crystal structures have a relatively large number of slip systems. These metals are quite ductile simply because extensive plastic deformation is normally possible along the various slip systems. Conversely, hcp metals, having a few slip systems, are normally quite brittle.[2.38]

In austenitic stainless steel orthodontic wire, only two phases were found: fcc austenitic phase and the bcc martensitic phase. So, in the following, only these two crystallographic systems are discussed.

Face centred cubic metals deform primarily on the close-packed octahedral $\{111\}$ planes in the $\langle 110 \rangle$ close packed directions of which there are three in each $\{111\}$ plane (Fig. 2.3). As there are four different orientations of $\{111\}$ planes, there is therefore a total of twelve possible slip systems which can take part in the deformation. All the familiar face centred cubic metals behave in this way, and it is rare to find a slip plane other than along one of the octahedral planes. Moreover this behaviour persists in solid solutions of face centred cubic structure. [2.38]

Body-centred cubic metals present a somewhat more complicated picture, but in general they all slip in the closest packed direction $\langle 111 \rangle$. The choice of slip plane, however, varies greatly. Work on iron indicates that while the $\{110\}$ plane is a common slip plane, the $\{112\}$ and $\{123\}$ plane are also operative. Planes of these three types share a $\langle 111 \rangle$ direction as their zone axis. Fig. 2.4 A-C illustrates one plane from each of the three systems, with a common $\langle 111 \rangle$ direction. The multiplicity of possible slip systems is

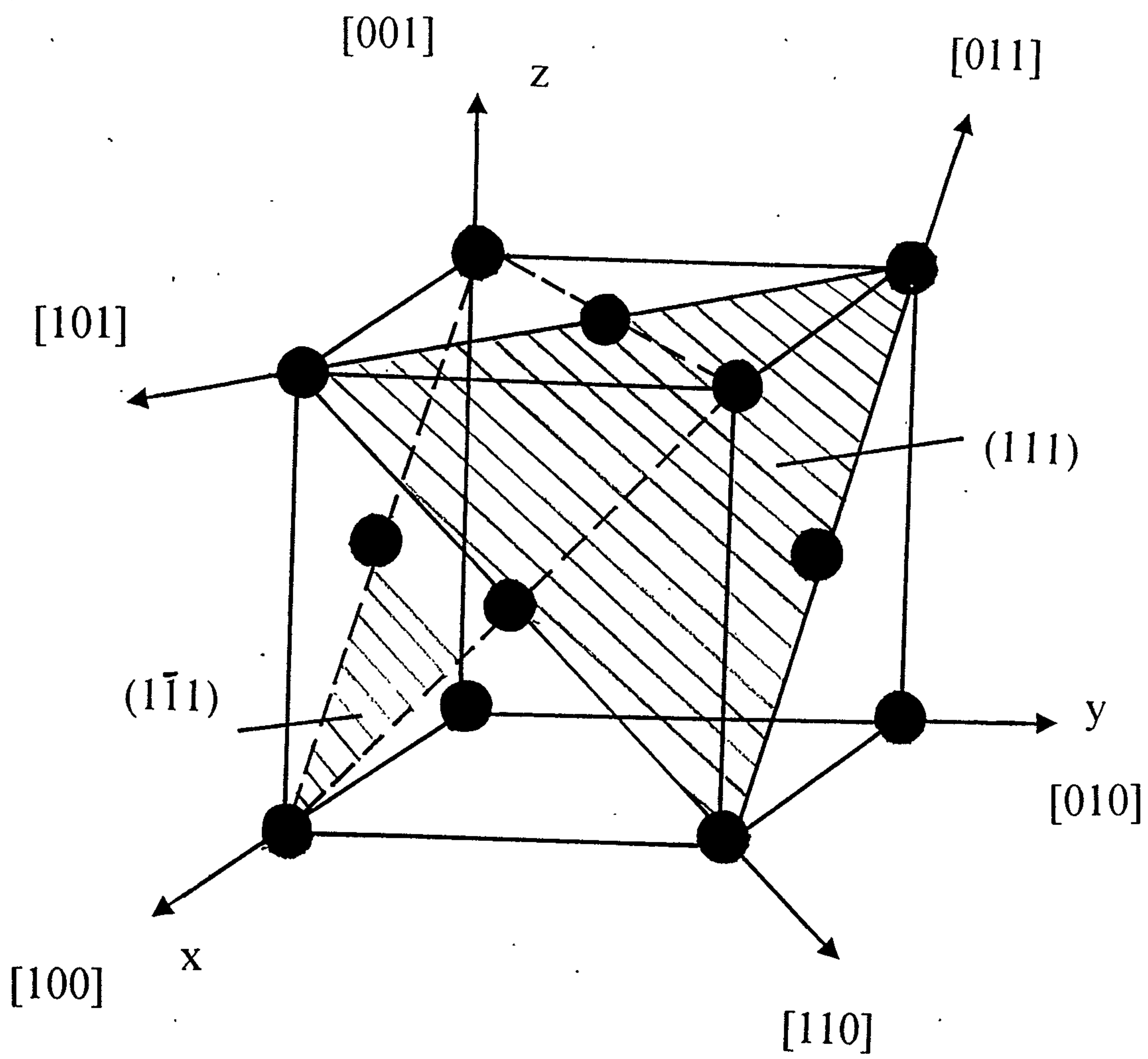


Fig. 2.3 The slip system in face centred cubic structure.

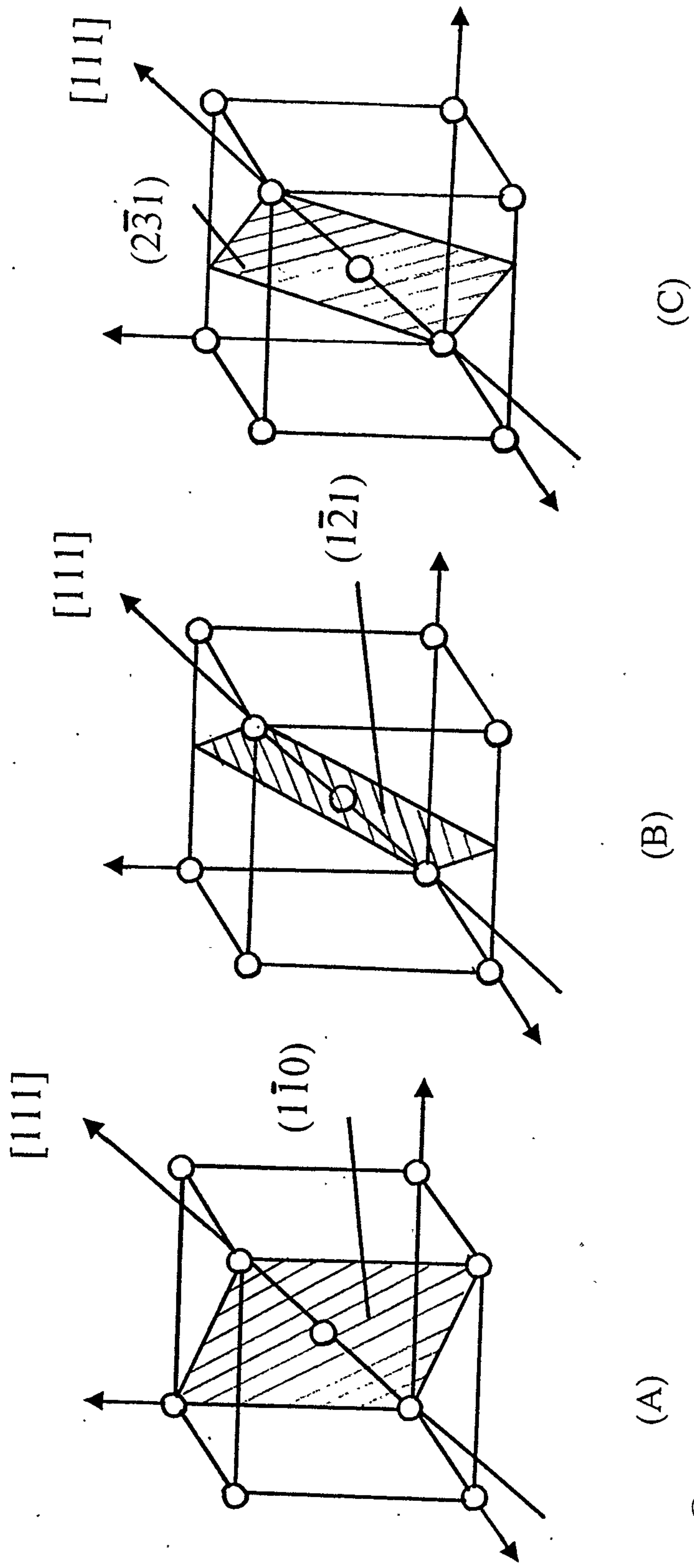


Fig. 2.4 (A-C) The slip system in body centred cubic structure.

sometimes reflected in the very wavy appearance of slip bands in a number of body-centred cubic metals. [2.38]

Table 2.2. Slip systems for face centred cubic, body centred cubic, and hexagonal close-packed metals. [2.38]

Metals	Slip plane	Slip direction	Number of slip systems
	Face-centered cubic		
γ -Fe, Cu, Ni,	{111}	$\langle 1\ \bar{1}0 \rangle$	12
	Body centered cubic		
α -Fe, W, Mo	{110}	$\langle \bar{1}11 \rangle$	12
α -Fe, W	{211}	$\langle \bar{1}11 \rangle$	12
α -Fe, K	{321}	$\langle \bar{1}11 \rangle$	24

2.7.1.3. Twinning

2.7.1.3.1. Deformation by twinning

A number of metals when deformed undergo a sudden localised shear process called twinning, which involves a small but well-defined volume within the crystal. This is in contrast to an individual slip process which, although it occurs through shear, is limited to one crystal plane and is therefore two dimensional in character. Twinning is frequently a catastrophic process which is accompanied by energy release in the form of sound.[2.37]

Microscopic examination of a metal which has twinned reveals that the twinned region is often bounded by parallel or nearly parallel sides which correspond with planes of low indices. These are the twin habit planes or twinning planes. It is also apparent that the twinned region differs appreciably in orientation from the grain in which it formed. The shear stress for forming the twinning is higher than slip and the twinning often forms at the stress concentrated area where the slip cannot continue to develop. Thus, twinning is another form of plastic deformation when it is difficult to deform the crystal by slip.[2.43]

Twinning differs from slip in several respects. For slip, the crystallographic orientation above and below the slip plane is the same before and after the deformation; whereas for twinning, there will be a reorientation across the twin plane (Figure. 2.5). In addition, slip occurs in distinct atomic spacing multiples, whereas the atomic displacement for twinning is less than the interatomic separation.[2.37]

The shear of a very thin twin might possibly be accommodated elastically in the matrix surrounding a twin. However, as a twin grows in size, the accommodation normally requires plastic deformation in the surrounding metal. If the matrix is not able to accommodate the twinning shear of a twin of finite size, then a hole may develop at the point where the accommodation was not obtained.(Figure 2.6) [2.38]. This hole can become the initial source of a crack during fracture. This may be the reason why wires that develop a higher density of twinning (e.g. WF wire), are easier to break in the clinical application.

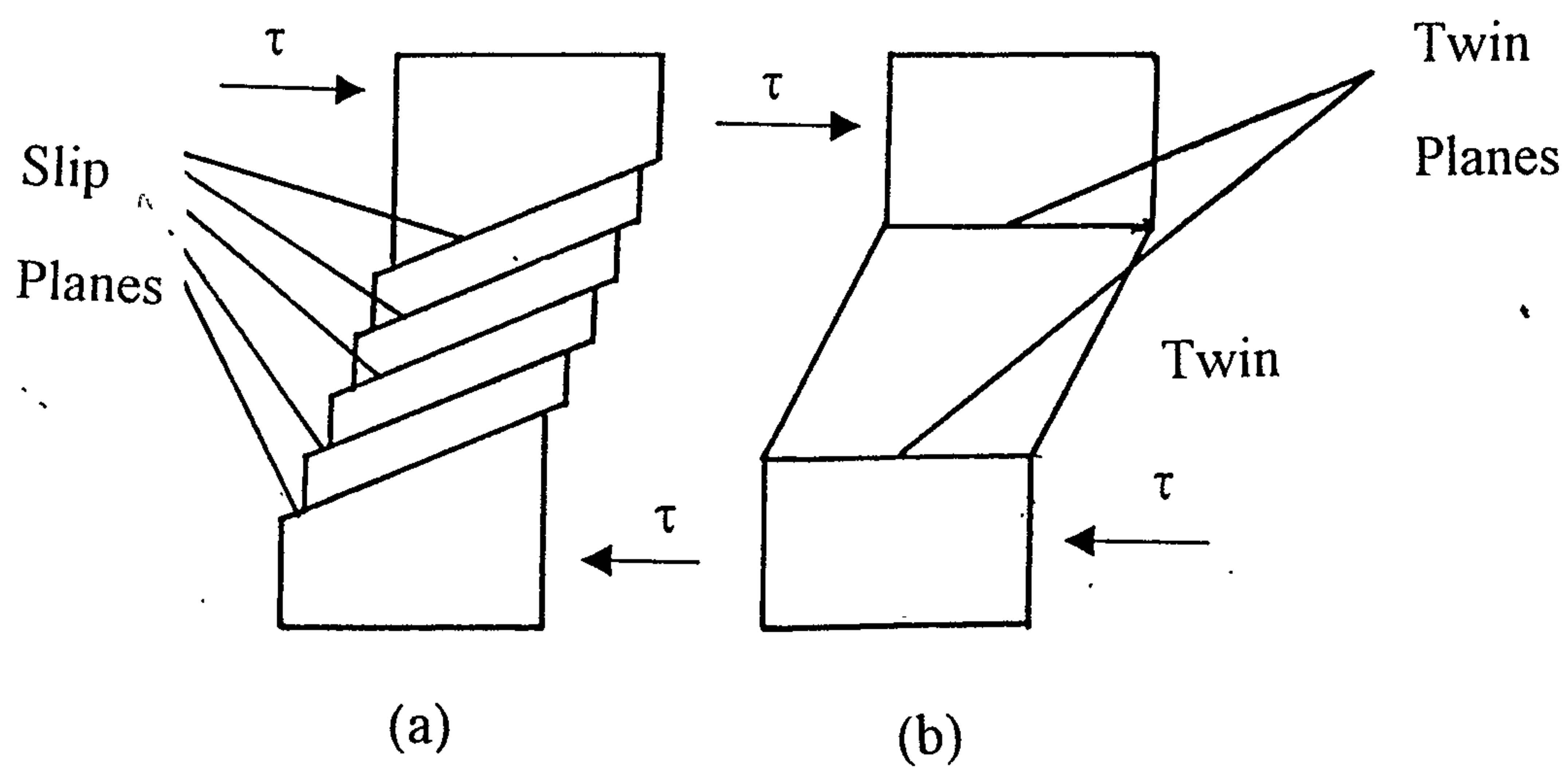


Fig.2.5 For a single crystal subjected to a shear stress τ . (a). deformation by slip; (b). deformation by twinning.

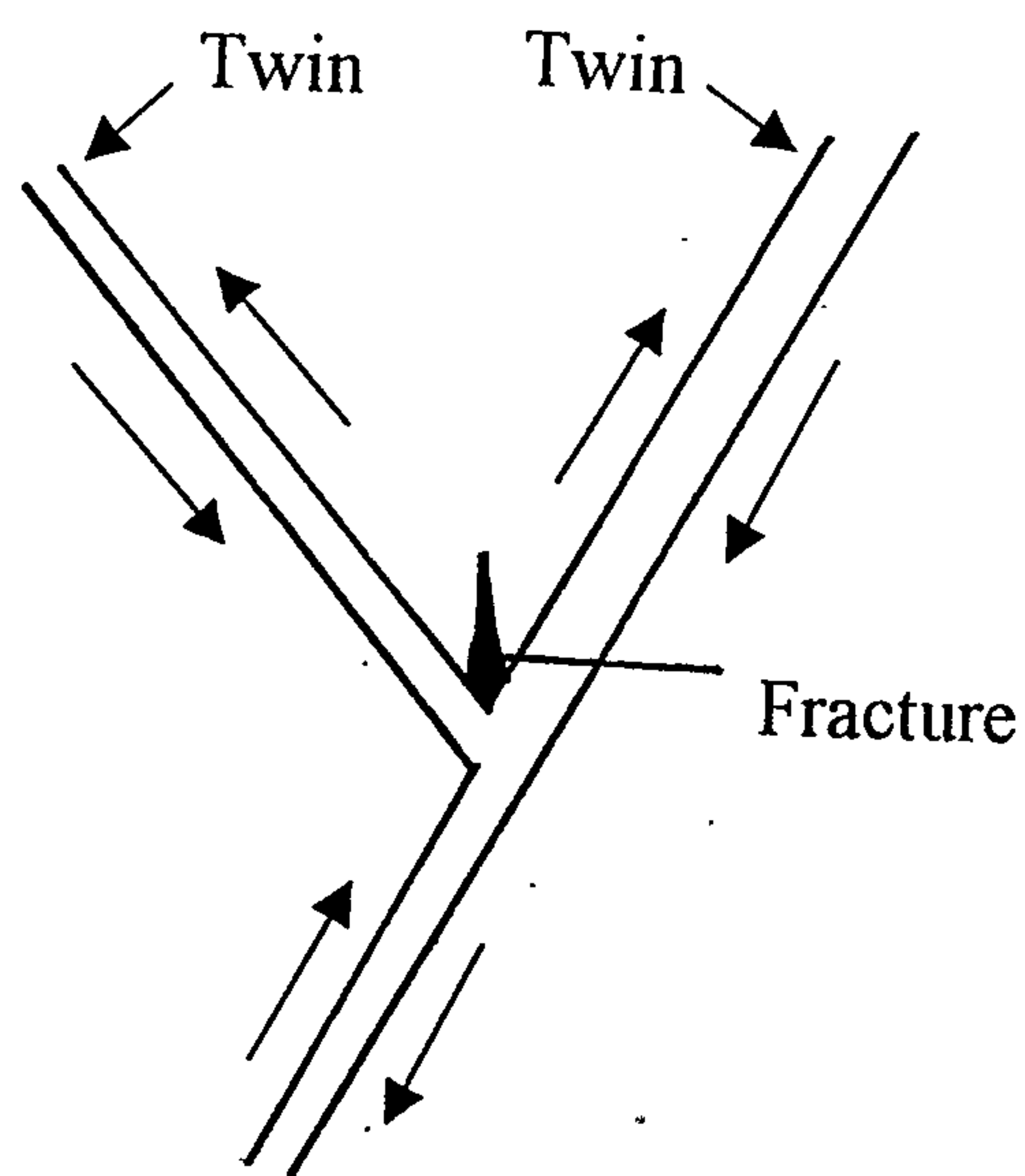


Fig.2.6 A crack induced in an iron crystal at a twin intersection.

2.7.1.3.2. Crystallography of twinning

Twinning is not a dominant mode of deformation in metals which possess many possible slip systems, e.g. face-centred cubic metals. However, it is very significant in metals where the possible slip systems are severely limited, e.g. the close-packed hexagonal metals, where slip is frequently limited to the unique basal system. [2.38]

In polycrystalline aggregates the changes in shape accompanying deformation require the operation of several slip systems; however, this is not possible in the hexagonal metals, so twinning becomes an important deformation mechanism. In recent years it has been found that deformation twinning can also occur in face-centred cubic metals, particularly at low temperatures, and in metals and alloys which possess low stacking fault energies[2.37]. Both 18/8 and 302 stainless steels are low stacking fault energy metals, and as will be seen in the later chapter discussing TEM experiments, fcc twinning structure exist in the wires.

The process of twinning is a co-operative movement of atoms in which individual atoms move only a fraction of the inter-atomic spacing relative to each other. The crystallographic result of such a transition is that the lattice in the twinned region is related to the untwinned crystal by a reflection across the twinning plane which results in the twin lattice being the mirror image of the lattice of the matrix in which it forms. This can result from several different crystallographic transformations. [2.38]

A simple example is given in (Figure 2.7), where the lattice has been twinned along a plane K_1 the twinning plane in the twinning direction η_1 . The open circles are the original atomic positions while the new positions

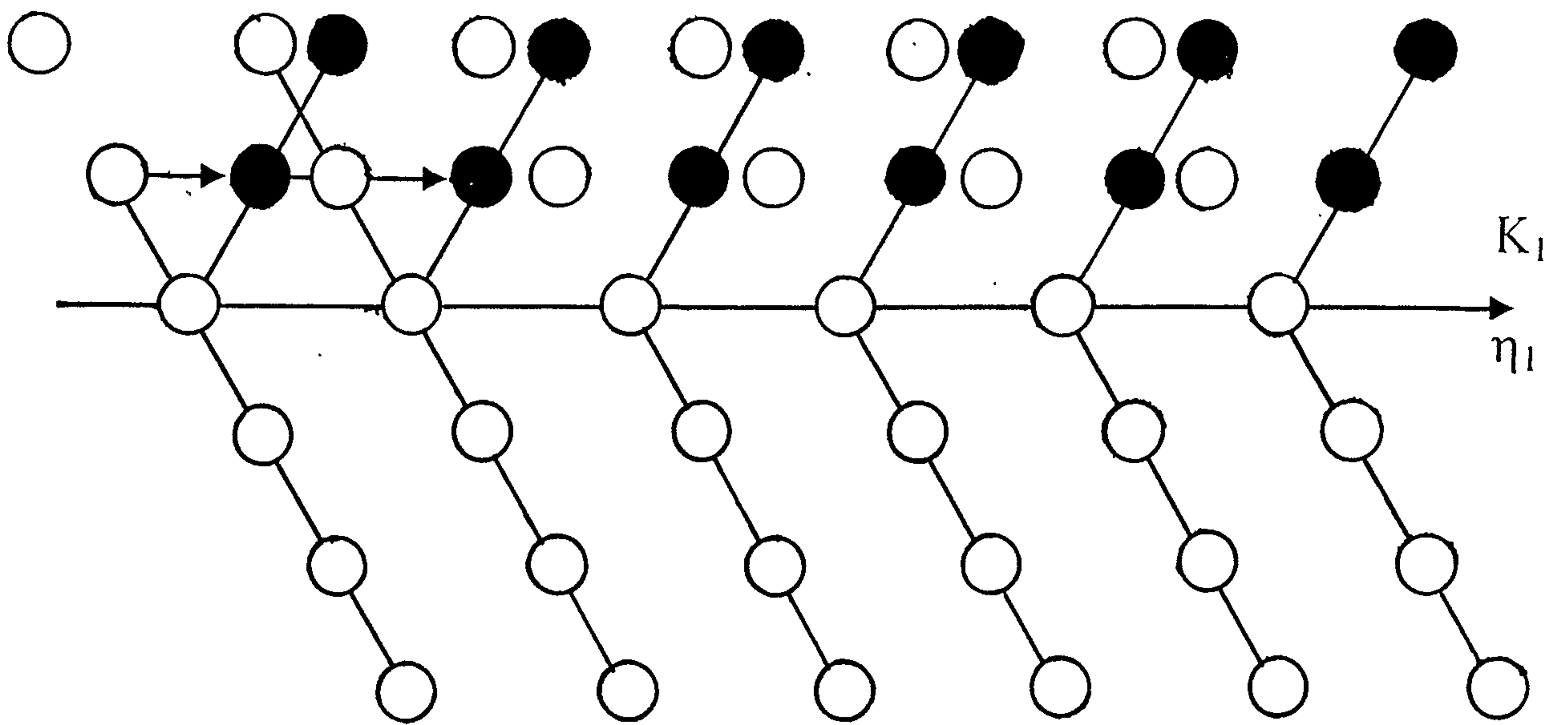


Fig.2.7 A twinning transformation.

following the twin displacements are indicated by the black circles. The twinning lattice is the mirror image of the parent lattice. In contrast to slip, which involves displacements which are equivalent to one or more inter-atomic spacings, twinning results from smaller displacements which successively occur on adjacent planes.[2.37]

The twinning transformation, which leads to a change in orientation of the twinned region, does not alter the symmetry or structure of the crystal.[2.38]

2.7.2. The plastic deformation of polycrystalline metals

In polycrystalline materials the slip direction varies from one grain to another due to the random crystallographic orientations of numerous grains. For each grain, dislocation motion occurs along the slip system that has the most favourable orientation with respect to the applied stress. Each operative slip system would create deformation in a different direction in each grain. In the absence of a crack, mechanical integrity is maintained along the grain boundaries: i.e., the grain boundaries do not usually come apart. Consequently, each individual grain is constrained in the shape it may assume by the neighbouring grains.[2.38]

Polycrystalline metals are stronger than their single-crystal equivalents, which means that greater stresses are required to initiate slip. This is, to a large degree, also a result of geometrical constraints that are imposed on the grains during deformation. Even though a single grain may be favourably oriented with the applied stress with slip, it cannot deform until the adjacent and less favourably oriented grains also become capable of slip; this requires a higher applied stress level.

2.8 The microstructure and properties of metal after deformation and the strengthening mechanisms of deformed structure.

Plastic deformation of single crystals has previously been considered in terms of the movement of dislocations and the basic deformation mechanisms of slip and twinning. Also the plastic deformation of polycrystalline metals has been discussed. The following section examines the microstructure and properties of metal after plastic deformation and also considers the strengthening mechanisms from the deformation.

Strengthening a metal or alloy is achieved essentially by causing the deformation process to become more difficult. This, in essence, means making the movement of dislocations in the material more difficult. Strengthening is almost inevitably accompanied by a loss in ductility. In other words, as we increase the strength of a metallic material it becomes more brittle [2.49]. The strength and mechanical behaviour can be affected by a number of possible factors which will now be considered.

2.8.1. Grain size and boundaries.

2.8.1.1. The change of grains after deformation and resultant properties.

After plastic deformation, the internal grain shape will change along with the change to metal macro shape caused by the applied stress. The internal grain shape change corresponds to the outer shape of the metal, e.g. when drawing the metal, the grain will become elongated along the drawing direction. [2.44]

When the amount of deformation is large, each grain will become elongated and the grain boundary will become hazy. The property of the metal will have directionality as well, e.g. the strength and plasticity is larger in the longitudinal than in the transverse direction of the wires. This structure is called the fibre structure.[2.44]

2.8.1.2. Grain size and its strengthening mechanism.

A fine-grained material (one that has smaller grains) is harder and stronger than one that is coarse grained. The relationship between strength and grain size is expressed by the Hall-Petch equation, which states that the tensile yield stress is related to grain size by [2.49]

$$\sigma_0 = \sigma_i + k_Y D^{-1/2}$$

where:

σ_0 = the yield stress;

σ_i = friction stress opposing motion of dislocation;

k_Y = "unpinning constant," measuring the extent to which dislocations are piled up at barriers; and

D = average grains diameter.

This Hall-Petch equation implies that yield stress is inverse of the grain size; i.e. the smaller the grain size, the higher the yield strength.

Two models have evolved for explaining the dependence of resistance to plastic deformation on grain size.[2.49]

The first model is based upon the concept that dislocations pile up at grain boundaries and these grain boundaries act as barriers to dislocation motion. Thus, a smaller grain size increase the number of grain boundaries and therefore increase the barriers to dislocation movement.

The second model for the grain size dependence of flow stress does not require the presence of dislocation pile-ups at grain boundaries. This model avoids the description of the stresses at grain boundaries and instead concentrates on the influence of grain size on dislocation density. The grain size becoming smaller actually increases the dislocation density. Higher dislocation density results in the structure work hardening. Therefore, higher dislocation density actually increase the yield strength.

Achievement of a smaller grain size is the only method among the strengthening mechanisms that also contributes to the toughness of the material; that is, it also improves the ductility.

In addition to the grain size of the parent material, the sizes and shapes of the constituent phases also significantly affect the mechanical properties of multiphase alloys. Grain size of a metallic material may be regulated by the rate of solidification from the liquid phase and also by plastic deformation followed by an appropriate heat treatment termed recrystallization.[2.37]

2.8.1.3. High-angle grain boundary and its strengthening mechanism.

The ordinary high-angle grain boundary represents a region of random misfit between adjoining crystal lattices. High-angle grain boundaries are boundaries of rather high surface energy. These high energy grain

boundaries usually result in a higher concentration of solute atoms at the boundary than in the interior of the grain. Different slip systems can operate in adjacent regions of the same grain resulting in complex lattice rotations which form deformation bands. Since more slip systems are usually operative near the grain boundary, Hardness will usually be higher near the boundary than in the centre of the grain, As the grain diameter is reduced, more of the effects of grain boundaries will be felt at the grain centre. Thus, the strain hardening of a fine grain size metal will be greater than in a coarse-grain polycrystalline aggregate [2.49],[2.51].

2.8.1.4. Low-angle grain boundary and its strengthening mechanism.

The ordinary high-angle grain boundary represents a region of random misfit between the adjoining crystal lattices. In the low angle boundary, the boundary is composed of a regular array of dislocations.

The subgrains are low-angle boundaries in which the difference in orientation across the boundary may be only a few minutes of arc or, at most, a few degrees. Subgrain boundaries are lower-energy boundaries than grain boundaries. A low-angle boundary contains a relatively simple arrangement of dislocations. The simplest situation is the case of a tilt boundary. In the TEM chapter, as we will see later, the grains in WS, WF and GF wires are all deformed into subgrains with low angle grain boundaries clearly observed.[2.44]

Along the boundary, the atoms adjust their positions by localised deformation to produce a smooth transition from one grain to another. However, elastic deformation cannot accommodate all the misfit, and so,

some of the atom planes must end on the grain boundary. Where the atom planes end, there is an edge dislocation. Therefore, low-angle tilt boundaries can be considered to be an array of edge dislocations. [2.49]

Sub-boundaries or low-angle boundaries can be produced in a number of ways. [2.52] They may be produced during crystal growth, during high-temperature creep deformation or as the result of a phase transformation.

The formation of subgrains in annealed materials can result in a significant increase of strength. For example, Parker and Hazlett found that the yield stress increase in deformed nickel is due to an increase in the density of subgrain boundaries. [2.49].

2.8.2. Phase transformation.

If alloying elements are not completely soluble in the host metal, these foreign atoms can form a second phase so that the whole structure becomes an agglomerate of two different types of grains. This results in an increase in total grain boundary area (smaller grains) and hence higher strength. In addition, alloying may create an opportunity to form non-equilibrium phases, as in the case of steels. Sufficient amounts of carbon would create the opportunity to form a non-equilibrium phase called *martensite*. [2.34].

As will be seen later, both TEM and XRD experimental results have shown that α -martensitic phase forms from the austenite phase in all the wires being researched. Therefore, the discussion here is about the basic theory of the mechanism of martensite phase transformation, martensite strengthening mechanisms and the deformation of two phase aggregates. Also the

similarities and differences between martensite and twinning will be discussed in this section as both have similar appearances in the TEM.

2.8.2.1. The martensitic phase transformation by deformation.

Martensite phase transformation is the phase changes where there is no long range movement of atoms. Indeed, in most cases to be considered the atoms move less than interatomic distance and retain their relationship with their neighbours during the phase change. The prototype of this behaviour is a martensitic transformation where the transformation process is equivalent to the deformation of the parent crystal lattice into the product (martensite) crystal lattice. Because of their nature, martensitic transformations are frequently referred to as "displacive" or "diffusionless".

18-8 austenitic alloys generally have “face-centred-cubic” (fcc) austenite crystal lattices. By cold working (wire drawing process), some austenite can have the sequence of martensite phase transformation to ϵ -martensite (hcp) and to α -martensite (bcc). ϵ -martensite phase is a thermodynamically stable hcp phase whose formation is greatly enhanced as a result of plastic deformation. α -martensite phase is a bcc structure. The martensitic transformation from the fcc to hcp and bcc phase in 18/8 type austenitic stainless steel is complicated by the presence of the hcp (ϵ) phase which is always found to be closely associated with α . Both the transformation products, ϵ and α , are martensitic transformation products. [2.29]

The mechanisms responsible for the formation of ϵ -martensite phase are the subject of arguments. Cina found [2.31, 2.33] that during the continual plastic deformation process, the amount of ϵ became a maximum and later

decreased in quantity while the amount of α progressively increased. This observation led him to believe that α nucleated from ε . But, Otte and Dash, and Breedis & Robertson [2.30, 2.32] believe that ε forms only because of the large shape deformation due to the $\gamma \rightarrow \alpha$ phase transformation.

It has been further argued from symmetry considerations that the transformation from a structure of lower symmetry, (i.e., hcp,) to one of higher symmetry, (i.e., bcc,) is highly unlikely [2.30]. Furthermore, It has been argued [2.30] that a different M_s (temperature of start martensite transformation) temperature should be observed from that due to the $\gamma \rightarrow \alpha$ or $\varepsilon \rightarrow \alpha$ transformation. Since a difference was not found, ε was concluded not to be an intermediate phase. Wayman [2.34] supports the view that ε forms as a consequence of the α formation. With the latter point of view, ε should be expected to increase proportionately with increasing amounts of α . [2.34]

In our deformed 18/8 austenitic orthodontic wire, only α -martensite has been found by using TEM and XRD techniques. This could be explained either by the fact that the procedure of martensite phase transformation by deformation are consistent with Cina 's model or that ε -martensitic has not appeared during the wires drawing process. Since it is not the purpose of this thesis to discuss the model of martensitic phase transformation by deformation, this topic is not examined further here. In the following, the basic and classical martensitic transformation model - Bain distortion is introduced.

2.8.2.1.1. BAIN distortion

A face centred cubic lattice can also be considered as a body centred tetragonal (bct) lattice: see Fig. 2.8 and 2.9 in which (in the upper diagram) a tetragonal unit cell is delineated in the face centred cubic structure. In a tetragonal structure, the crystallographic axes lie at right angles to each other, as in a cubic structure, but one lattice constant c differs in magnitude from the other two. A body centred tetragonal structure has a $c:a$ ratio about 1.4 (see figure 2.8). Similarly, a body centred cubic structure can be considered as a body centred tetragonal structure with $c:a$ ratio of unity. [2.35]

In 1924, Bain suggested that a body centred cubic lattice could be obtained from a face-centred cubic structure by a compression parallel to the C axis and an expansion along the two a axes. Any simple homogeneous pure distortion of this nature which converts one lattice into another by an expansion or contraction along the crystallographic axes belongs to a class known as *Bain distortions*. [2.35]

The Bain distortion indicated above converts a face centred cubic lattice into a body centered lattice with a minimum of atomic movements. However, there is no undistorted plane associated with this Bain distortion, so that the invariant plane strain associated with martensitic transformations cannot be explained by a Bain distortion. The Bain distortion does, however, give an approximate measure of the magnitude of atomic movements involved in a transformation. Thus, in steels, the $\langle 100 \rangle$ axis of austenite is contracted by about 20% to become the martensite c axis and two $\langle 110 \rangle$ axes in austenite are extended by some 12% to become martensite a axes. [2.35].

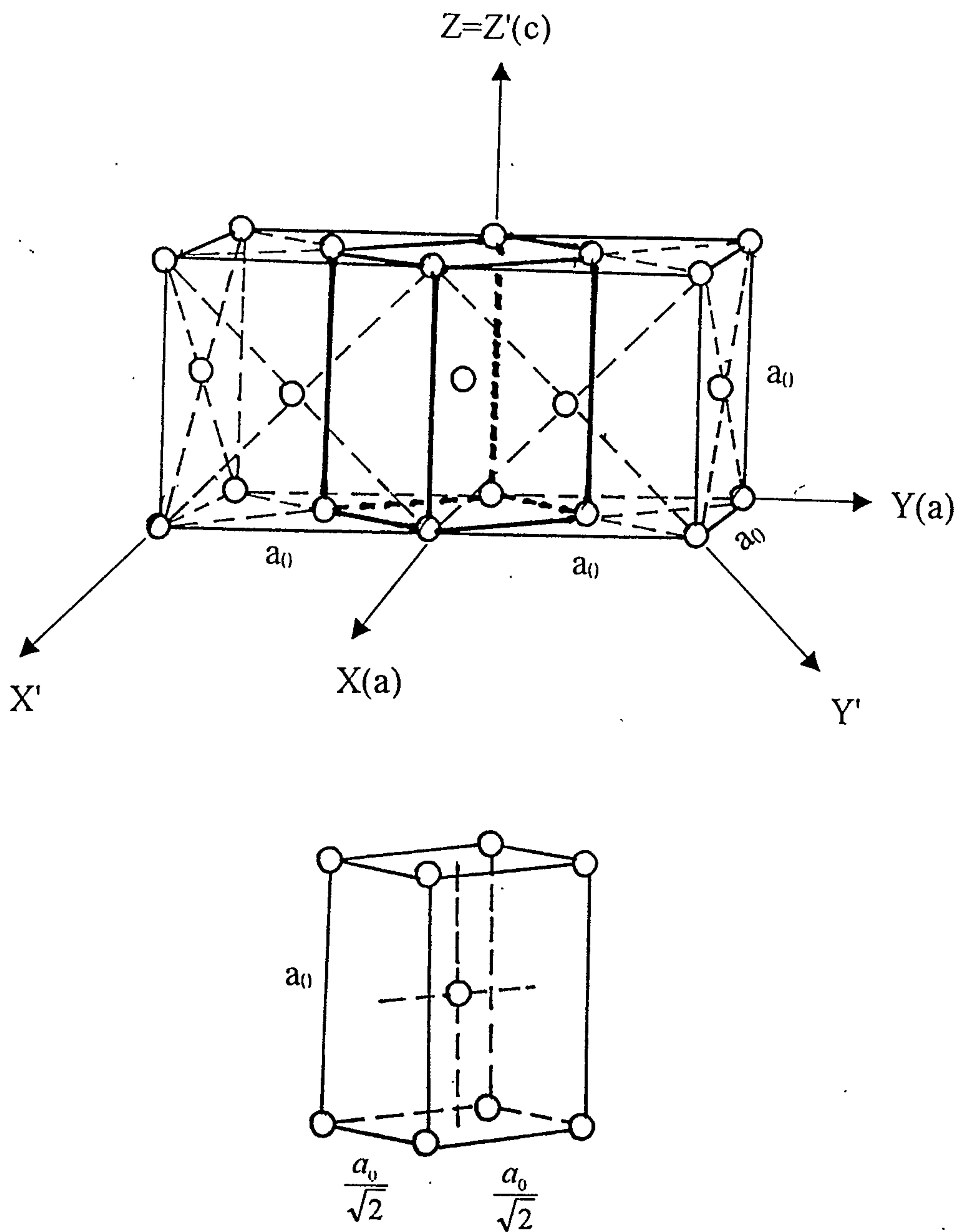


Fig.2.8 Bct lattice delineated in austenite (fcc) structure.

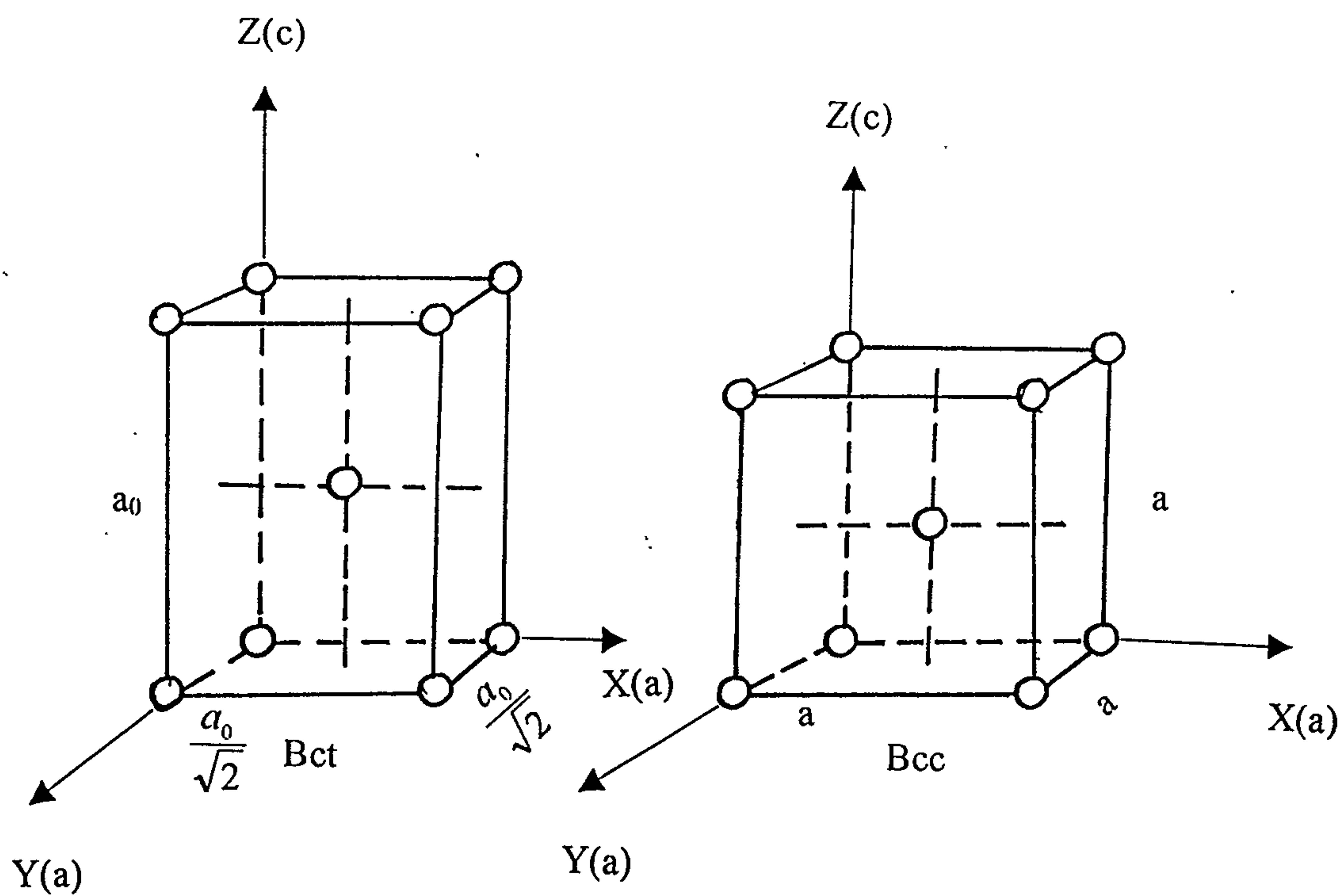


Fig. 2.9 Bct lattice of austenite and bcc lattice of α iron produced by upsetting the former; drawn to scale for comparison.

Because of the manner in which martensite forms, no composition change occurs as the parent lattice is converted into the product phase. Diffusion in either the parent phase or product phase is not required for the reaction to continue. Martensite reactions are, accordingly, commonly referred to as diffusionless phase transformations [2.36].

2.8.2.2. The strengthening mechanisms of martensitic phase transformation.

The transformation of austenite to martensite by a diffusionless shear-type transformation in steel is one of the most common strengthening processes used in engineering materials. Although martensitic transformations occur in a number of metallurgical systems, only alloys based upon iron and carbon show such a pronounced strengthening effect [2.49], [2.53].

The high strength of martensite implies that there are many strong barriers to dislocation motion in this structure. Conventional martensite has a plate structure with a unique habit plane and an internal structure of parallel twins within the plates. The other type of martensite structure is a block martensite containing a high density of dislocations, comparable to those in a highly deformed metal. Thus, one of the mechanisms of strengthening through martensite presence arises from the effective barriers to slip provided by the fine twin structure or the high dislocation density.[2.49]

The second important contribution to the strength of martensite comes from the carbon atoms. Bain and Paxton found that the hardness of martensite is sensitive to carbon content below 0.2%. (the carbon content is lower than 0.2% in 302 stainless steel) [2.49]. The reason for the effect of the carbon content is that strong binding is set up between dislocations and carbon

atoms. The contribution to strength from the barriers in the martensite structure is essentially independent of carbon content, while the strengthening due to carbon atom clustering and dislocation interaction increases approximately linearly with carbon content.[2.49]

2.8.2.3. Deformation of two phase aggregates.

Many alloys have a microstructure consisting of two or more metallurgical phases. For example, the wires investigated here all have austenite and martensite phases. In general they fall into two classes. One class is the aggregated type of two-phase structure in which the size of the second phase particles is of the order of the grain size of the matrix. The other class of structure is the dispersed two-phase structure in which each particle is completely surrounded by a matrix of a single orientation (grain).

Generally the particle size of the second phase is much finer for the dispersed structure and may extend down to submicroscopic dimensions in the early stage precipitation. Fine particles can act as barriers to dislocations by acting as strong, impenetrable noncoherent particles through which the dislocations can move only by sharp changes in curvature of the dislocation line.[2.50]

The strengthening produced by second-phase particles is usually additional to the solid-solution strengthening produced in the matrix. For two-phase alloys produced by equilibrium methods, the existence of a second phase ensures maximum solid-solution hardening because its presence results from supersaturation of the matrix phase. Moreover, the presence of second-phase particles in the continuous matrix phase results in localized internal stresses which modify the plastic properties of the continuous phase. [2.35]

Many factors must be considered for a complete understanding of strengthening from second-phase particles. These factors include the size, shape, number, and distribution of the second-phase particles, the strength, ductility and strain-hardening behaviour of the matrix and second phase, crystallographic fit between the phases, and the interfacial energy and interfacial bonding between the phases. [2.35]

In general terms second phases block slip so that plastic deformation is not uniform in the matrix. Strain in the matrix is localized and increased over the average strain of the specimen. This leads to strengthening from local plastic constraint.[2.50]

In a multiphase alloy, each phase contributes to the overall properties of the aggregate. If the contributions from each phase are independent, then the properties of the multiphase alloy will be a weighted average of the properties of the individual phases. [2.50]

The mechanical properties of an alloy consisting of a ductile phase and a hard brittle phase will depend on how the brittle phase is distributed in the microstructure. If the brittle phase is present as a grain boundary envelop, the alloy is brittle. If the brittle phase is in the form of discontinuous particles at grain boundaries, the brittleness of the alloy is reduced somewhat.

In the wires researched in this thesis, it is very difficult to identify the martensite phase distributed in the microstructure as the austenite phase appears in all the wires as fcc twinning structure which is difficult to distinguish from martensite structure.[2.50]

2.8.2.4. Martensite reactions and deformation twinning. [2.37]

So far, the twinning (see 2.7.1.3) and martensite phase transformation (see 2.8.2) have been introduced separately in the above sections. In fact both are developed by deformation processing (i.e. the wire drawing process). In the later chapter on TEM investigation, it will be shown that both exist in the wires and have the similar appearance. In this section, twinning and martensitic phase transformation are brought together, and the similarities and differences between them are discussed.

These two apparently unrelated types of phenomena actually have much in common. Deformation or mechanical twinning is a mode of plastic deformation, while martensite reactions are a basic type of phase transformation. Twinning occurs as the result of applied stresses. Martensitic reactions occur in metals that undergo phase transformations and the driving force is the chemical free-energy difference between two phases. Stress can sometimes partly trigger a martensitic transformation, but this is an effect of secondary importance.

The similarity between martensite reactions and twinning lies in the analogous way twins and martensite crystals form: in both cases, the atoms inside finite crystalline volumes of the parent phase are realigned as new crystal lattices. In twins, this realignment reproduces the original crystal structure, but with a new orientation. In a martensite plate, not only is a new orientation produced, but also a basically different crystalline structure arises. For example, during martensite transformation, the fcc austenite phase is converted into small crystalline units of a bct phase. On the other

hand, when twinning occurs in the fcc austenite phase, both the parent crystal and the twinned volumes still have the fcc structure.

In martensite reaction, because the new lattice which is formed has a symmetry different from that of the parent phases, the deformation is of necessity more complicated. With mechanical twinning the distortion has already been shown to be a simple shear parallel to the twinning plane or the symmetry plane between a twin and parent crystal. The twinning plane is an undistorted plane; all directions in this plane are unchanged by twinning with respect to both their magnitudes and their angular separations.

The habit plane on which martensite plates form is as in the case of twinning usually assumed to be an undistorted plane. The macroscopic shape deformation in the formation of a martensite plate is believed to be a shear parallel to the habit plane plus a simple (uniaxial) tensile or compressive strain perpendicular to the habit plane. A strain of this nature, known as an invariant plane strain, is the most general that can occur while still maintaining the invariance of the habit plane. Neither a shear parallel to the habit plane nor an extension or contraction perpendicular to it will change the positions or magnitudes of vectors lying in the habit plane.

In both twinning and martensite transformations, each realigned volume of material suffers a change in shape which distorts the surrounding matrix. The change in shape are quite similar, so that martensitic plates and deformation twins look alike, taking the form of small lenses or plates.

2.8.3. Strengthening mechanism due to fine particles.

Forming fine second phase particles in the host metal by the addition of alloying elements can increase the strength of a metal significantly. The differences between this mechanism and that mentioned under 'phase transformation' are: [2.37]

- 1). These fine particles can be formed with extremely small amounts of alloying additions, eg. approximately 0.02% Ti, V, Nb additions to steels.
- 2). These fine particles are not defined as grains due to their sharp and size (approximately 10nm in size)

2.8.4. Work hardening.

2.8.4.1. The work hardening phenomenon.

Work hardening is also called strain hardening. It is a phenomenon whereby a ductile metal becomes harder and stronger as it is plastically deformed. Sometimes the process is also called cold work hardening. The term 'cold' refers to the temperature at which deformation takes place, which is low relative to the absolute melting temperature of the metal. Most metals are hardened at room temperature. [2.38]

The deformation, such as wire drawing, not only changes the external form, but also changes the internal substructure of the grains. This change of the substructure has a major effect on the property of the metal. [2.38]

When the deformation is small, dislocations pile up near the deformed grain boundary. When the deformation is large, the change due to the dislocation and substructure of internal grains will be very complicated. During plastic

deformation, which is also a dislocation motion, will have a complicated reaction with the other dislocations with different Burger's vector. [2.38]

When a mobile dislocation meets other dislocations, initially the large number of dislocations will pile up and become entangled at the dislocation wall and creating a dislocation network. With further deformation, which is the development of this dislocation tangle phenomenon, each grain will develop into fine sub-grains. With higher levels of deformation, the grains will become more fine, the total number of sub-grain boundaries will increase and the dislocation density will also increase. [2.38]

The fine sub-grains will also become elongated as the grains elongate. With the increase of deformation, the grains will be broken. This process creates a significant increase in the dislocation density, and also increases the resistance to further plastic deformation. Therefore, hardness and strength will increase and plastic behaviour and toughness will decrease. This process is called work hardening. [2.44]

Work hardening makes further deformation difficult and so, intermediate heat treatment is necessary in order to reduce the dislocation pile up density in order to restore the ability for further plastic deformation. [2.44]

2.8.4.2. The strengthen mechanism of work hardening.

The dislocation density in a metal increases due to dislocation multiplication during continuing deformation or cold work. Consequently, the average distance of separation between dislocations decreases. The result is that the motion of a dislocation is hindered by the presence of other dislocations. As the dislocation density increases, the resistance to dislocation motion by

other dislocations becomes more pronounced. Thus the required stress for further deformation increases with increasing cold work.[2.44]

2.8.5. Texture.

2.8.5.1. Anisotropy in polycrystalline metals

During the process of plastic deformation, anisotropy of the plastic properties results inevitably from the behaviour of dislocations in the crystals. The directional properties of these dislocations arise from the anisotropy of the elastic properties from the macroscopic restraints imposed on deformation by shear in tensile tests and from other more complex deformation processes.[2.45]

An annealed polycrystalline aggregate is formed by first working an ingot, then recrystallising either at various stages of the deformation, or after deformation is complete. This formation does not usually lead to an array of randomly oriented grains. Most deformation processes tend to induce anisotropy by virtue of the intrinsic crystallographic nature of deformation by slip and twinning, and this is further intensified on recrystallisation. [2.38]

Textures have a number of important technological applications, e.g. texture strengthening, magnetic alloys.

2.8.5.1.1. Development of preferred orientations (textures)

Most methods of deforming a polycrystalline metal result in the predominance of a particular crystallographic direction or plane parallel to

the direction of deformation in a majority of individual crystals comprising the aggregate [2.46].

In a wire drawing process, for a single crystal, the slip direction rotates until it approaches the axis of wires. Rotations occur in the individual grains of aggregates which even in simple processes such as tension suffer more complicated stressing. This leads to the development of preferred orientations or textures. [2.46]

The nature of texture depends on the particular deformation process used and also on the metal's crystal structure which determines the crystallography of the operative slip systems. There are several principal types of deformation texture which are determined primarily by the deformation process employed and the characteristic stress distribution each process imposes on the metal. Some examples of ideal textures are given in Table 2.3. [2.38]

Table 2.3. Some common textures produced by cold work [2.38]

<i>Crystal structure</i>	<i>Mode of working</i>	<i>Texture</i>
fcc	Wire drawing and extrusion	$\langle 111 \rangle, \langle 100 \rangle$, Parallel to wire axis
bcc	Wire drawing and extrusion	$\langle 110 \rangle$ Parallel to wire axis
cph.	Wire drawing and extrusion	$\langle 10 \bar{1}0 \rangle$ Parallel to wire axis

Note: These textures are idealised. In practice, considerable scatter occurs, and very frequently other textures are present.

2.8.5.1.2. Fibre textures.

Fibre texture is characteristic of uniaxial deformation processes such as wire-drawing, extrusion, etc., during the course of which the grains are elongated in the direction of working. Fibre texture is characterised by a crystallographic direction of low indices parallel to the axis (see table 2.3). [2.47]

The perfection of the texture increases with the degree of deformation but it can vary appreciably from the surface to the core of the wire because of macroscopic inhomogeneity in the deformation process.[2.38]

2.8.5.2. Strengthening mechanisms from wires' fibre texture.

As introduced above, a metal which has undergone a severe amount of deformation, such as in wire drawing, will develop a texture. In an ideal wire texture, a definite crystallographic direction lies parallel to the wire axis, and the texture is symmetrical around the wire or fibre axis.

When texture forms in the wires, it means more slip planes form perpendicular to the wire axis (the cross section of the wires). However, fewer slip planes form parallel to the wires axis (the longitudinal section of the wires).

Since the cubic metal can only deform on the slip plane and along the slip direction, this texture makes the wire hard to deform in the longitudinal direction (the wires axis). Therefore, this kind of texture actually causes the deformation process to be more difficult along the wire axis, so, the strength of the wires is increased [2.44]

2.8.6. Residual internal stress.

When a metal is cold worked, most of the energy is expended in changing the shape and in generating heat; however, a small proportion up to about 5% remains stored in the metal. The stored energy is mainly in the form of crystal lattice distortion stress due to dislocations and point defects which are present in concentrations orders of magnitude higher than in annealed metals.

The distribution is, in addition, quite different to that in the annealed state, e.g., there are pile-ups against obstacles, tangles and diffuse sub-boundary arrays. The stored energy in other forms is the internal stress due to the

different amount of deformation between the core and shell of the metal and due to the non-well-distributed deformation between grains or between different areas of the crystal.

The crystal lattice distortion stress is the main reason for the increase in strength of the metal. On other hand, these stresses can reduce the resistance of corrosion. Hence, after deformation, the metal should receive heat treatment to reduce these internal stresses.[2.42]

2.9. Annealing

Many metallic materials are produced initially as large castings which are then further processed in the solid state by forging, rolling, drawing etc to an intermediate or final product. These procedures, which may be carried out hot or cold, and which may involve intermediate anneals, are collectively termed thermomechanical processing. Recovery, recrystallization and grain growth provide the core elements of this processing. To a large extent the mechanical properties and behaviour of a metal depend on the dislocation structure, the size of the grains and the orientation or texture of the grains. The dislocation structure is dependent on the deformation and on the subsequent annealing treatment as well as other material and microstructural parameters. The grain size and texture are determined mainly by the recrystallisation process.[2.42]

2.9.1. Recovery.

The term "recovery" refers to changes in the properties of a deformed material which occur prior to recrystallisation. These changes are such as to partially restore the properties to their values before deformation. It is now known that recovery is primarily due to changes in the dislocation structure

of the material. So, to discuss recovery, it is most convenient to concentrate on the microstructural aspects.[2.42]

Recovery is not confined to plastically deformed materials and may occur in any crystal into which a non-equilibrium high concentration of point or line defects has been introduced. Even in the case of deformed metals, dislocations as well as point defects will be formed during deformation. However, as most of the excess point defects will anneal out at low temperatures, this does not usually constitute a separately identifiable stage of the recovery of a cold worked metal. [2.42]

Dislocation recovery is not a single microstructural process but a series of micromechanisms which include: dislocation tangles; cell formation; annihilation of dislocations within cells; subgrain formation and subgrain growth. Whether any or all of these occur during the annealing of a particular specimen will depend on a number of parameters, including the material, purity, strain, deformation temperature and annealing temperature. In many cases, some of these stages will have occurred during the deformation as dynamic recovery. Although the recovery stages tend to occur in the order shown, there may be significant overlap between them.[2.42]

The recovery and recrystallization are competing processes as both are driven by the stored energy of the deformed state. Once recrystallization has occurred and the deformation substructure has been consumed, then clearly no further recovery can occur. The extent of recovery will therefore depend on the ease with which recrystallization occurs. Conversely, because recovery lowers the driving force for recrystallisation, a significant amount

or prior recovery may in turn influence the nature and the kinetics of recrystallization. The division between recovery and recrystallization is sometimes difficult to define, because recovery plays an important role in nucleating recrystallization. Additionally, there are circumstances in which there is no clear distinction between the two phenomena.[2.42]

During recovery, the microstructural changes in a material are subtle and occur on a small scale. The microstructures as observed by optical microscopy do not usually reveal much change and for this reason, recovery is often measured indirectly by some bulk technique such as some physical or mechanical property.

Recovery may result in a small change in grain size and shape. These microstructural changes which occur during recovery affect the mechanical properties, and therefore recovery is often measured by changes in the yield stress or hardness of the material, although these changes are often small. The mechanical properties of a recovered materials are of some practical importance and their relationship to the microstructural changes. So, recovery is usually used for reducing the stress and retaining the work hardening behaviour.[2.42]

2.9.2. Recrystallisation.

Unlike recovery, recrystallisation causes the mechanical and physical properties of deformed metal to return completely to those of the annealed state. The mechanical properties such as: hardness, yield strength and ultimate tensile strength change slowly during the recovery period, but during recrystallisation these properties alter drastically even over a very small temperature range. (Figure 2.10)[2.44]

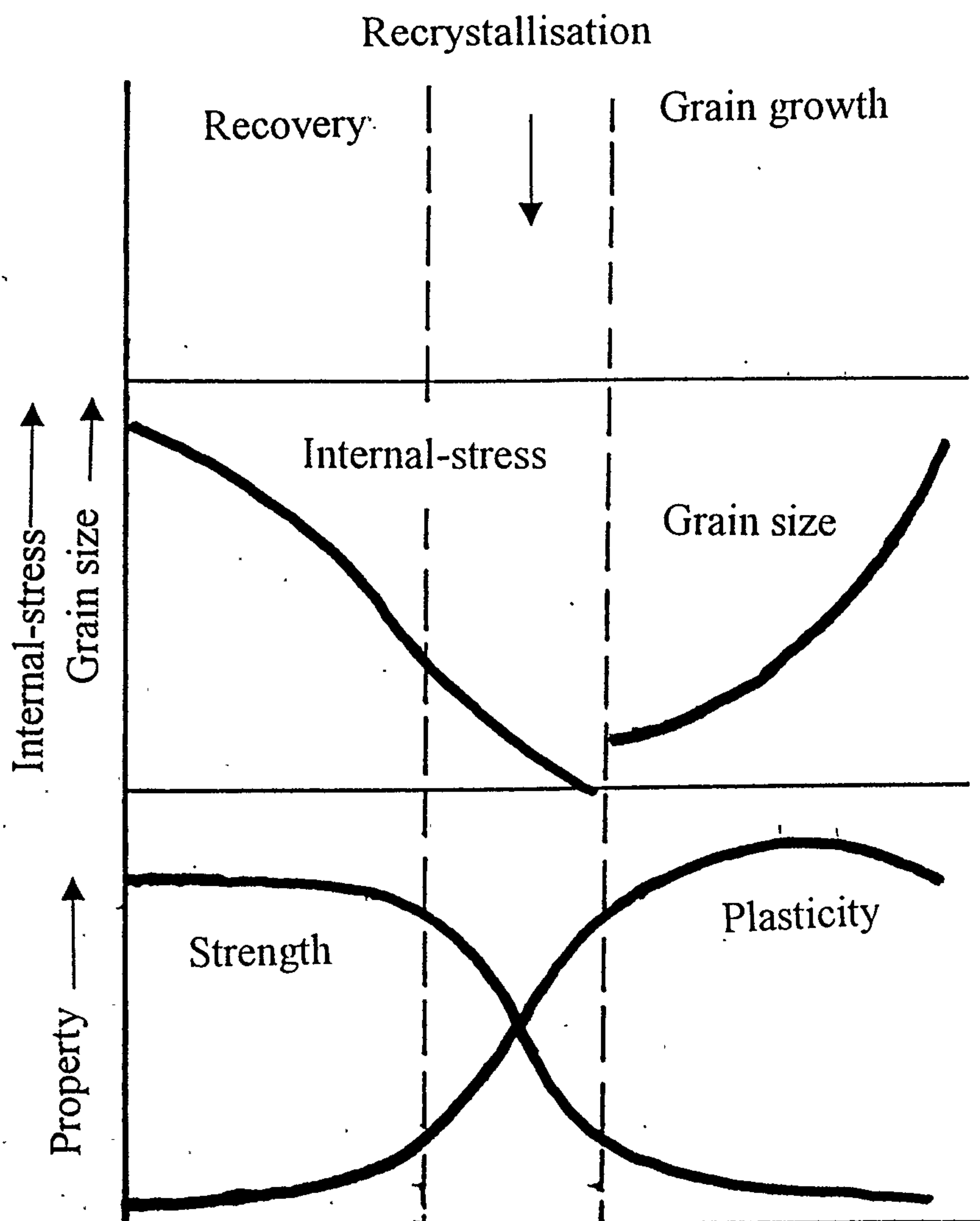


Fig.2.10 The change of grain size and property of deformation metal during annealing temperature.

After recovery, the point defect and crystal lattice distortion are reduced, but the grain is still deformed and elongated. When the metal has been heated to a higher temperature, the deformed and elongated grain will become new equiaxed crystal grains. This process is a nucleation and growth process. After recrystallisation, new grains will just change shape, the type of the crystal lattice will not change.[2.44]

2.9.3. Grain growth.

After recrystallisation, if heating is continue or an increase of the heat treatment temperature, the crystal grain will continue growing. This process is not avoidable as this is a energy reduction procedure.[2.44]

The above discussion summarises the theory from material science and metallurgy theory which will be used as a basis for the next three chapters' research into the microstructure and their mechanical properties of wires. These chapters are chapter 3 which investigates the mechanical properties, chapter 4 which quantifies the phases amount and texture by using X ray diffraction technique and chapter 5 which investigates the microstructure details using TEM techniques.

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Chapter 3. Mechanical properties test for WS, WF, GS and GF stainless steel orthodontic wires.

3.1. Introduction:

In this chapter, the mechanical properties of the four orthodontic wires studied will be considered. Initially the reasons for conducting these tests are discussed and then other mechanical property research is briefly reviewed so as to assist in interpreting or comparing the results obtained in this chapter.

3.1.1. Mechanical Property Tests.

Austenitic stainless steel has been the primary alloy used in orthodontic wires since 1929. The greater strength, higher modulus of elasticity, good resistance to corrosion, and moderate cost have been responsible for its popularity.[3.1, 3.2]

In the selection of an appropriate stainless steel wire, the main criteria are optimum predictable treatment results in clinical application. The clinician must be conversant with the mechanical properties and able to relate these properties to the relevant clinical applications. Mechanical property tests determine the characteristics of orthodontic wires and hence these tests provide a basis for the comparison and choice of these wires.

In our particular study, the results of the mechanical properties will not only serve the above purposes, but they will also create a useful framework for microstructure investigation into why, as discussed in other chapters, wires of the same composition but of different sizes have different mechanical properties

The two main measures to be considered are yield strength and the modulus of elasticity (fig.3.1). Yield strength (YS) is the point beyond which permanent deformation of the wires will occur. The modulus of elasticity (E) describes the resistance to elastic deformation.

For wires of the same composition, YS is heavily dependent on the deformation level and on microstructural detail, such as; grain size and shape, the amount of martensite phase and texture, etc. The standard value of E for stainless steel wires is about 200GPa [3.3]. This parameter depends upon the nature of the interatomic forces of the metal. The strengthening method, alloying, heat-treatment and cold working only affect this parameter slightly.[3.4]

These two mechanical property parameters (E and YS) are the most closely related to the clinical effectiveness of the wires. Therefore, as will be discussed shortly, a reasonable amount of the testing has been conducted to determine these values for stainless steel orthodontic wires.

These parameters are commonly estimated using tensile or bending tests. Tensile tests are often preferable to bending tests for the determination of properties since they introduce fewer variables. On the other hand, bending tests better simulate the manner in which orthodontic wires are used clinically. Thus, bending tests may be considered to be more representative of clinical use than the conventional tension tests. In this respect, 4 point bending tests more closely match actual clinical applications than 3 point bending tests.

As will be seen in the following literature survey, different types of mechanical tests may result in different values for YS and E. The same test

on the same wires at different stages (e.g. in their as received and heat-treated states) can also result in different values of YS and E. Since accurate estimates of these values are needed in the development of a framework for examining microstructure, reasons for the differences in values should be determined.

For this chapter, we used three techniques for measuring the mechanical properties:

- tensile tests;
- 3 point bending tests; and
- 4 point bending tests.

Once these tests had been conducted, further analysis was carried out using a scanning electron microscope to evaluate the fracture surfaces developed. The resulting SEM micrographs show the influence of microstructure on the fracture path and help determine the mechanisms of the fracture. The fracture mechanism can also be related to ductility of the wires.

3.1.2 Literature Survey

This section reviews this literature, thereby providing a context for the results from this study and accordingly creating a basis for ensuring that accurate results have been determined and that the conflicts in other research can be avoided in our specific case.

The following tables summarise earlier research results concerning yield strength and the elastic modulus of stainless steel wires:

Table 3.1: Summary of comparison E and YS in tensile and bending test for as received austenite stainless steel orthodontic wires by previous investigators.

Reference	Wire type	Diameter (mm)	Tension test		Bending test		Comments
			E (GPa)	0.1%YS (MPa)	E (GPa)	0.1%YS (MPa)	
Twelftree and associates 1977 [3.5]	Premium Plus	0.406	174	2380			E value to be 150Gpa-174Gpa, which is about 20% lower than the standard value.
	Premium	0.406	170	2520			
	Special Plus	0.406	164	2160			
	Special	0.406	150	2180			
	Unisil	0.406	153	2270			
Brantley and associates 1978 1[3.6]	Dentarum	0.406	160	2120			E in bending test should be identical within the experimental error to E for uniaxial tensile test for the same wires.
	Tru-Chrome	0.762	179		186*		
	Tru-Chrome	1.016	185		169*		
	Tru-Chrome	1.270	173		164*		
	Permachrome	0.762	188		194*		
	Permachrome	1.016	177		165*		
	Permachrome	1.295	177		153*		
	Tru-chrome	0.254	161	1516	232*	1517*	
	Tru-chrome	0.356	159	1379	218*	2000*	
	Tru-chrome	0.406	177	1241	217*	2000*	
Asgharnia and Brantley 1986 [3.7]	Tru-chrome	0.457	172	1448	216*	2413*	Values of E and YS in bending were almost invariably higher than the corresponding values obtained in tension tests for most stainless steel wires. The only general agreement between the E in bending and tension was for the 1.016 mm stainless steel wires.
	Tru-chrome	0.508	181	1172	215*	2344*	
	Tru-chrome	0.432x0.635	163	1103	192*	1655*	
	Tru-chrome	0.457x0.635	165	1241	194*	1931*	
	Permachrome	1.016	161	965	159*	2000*	

Continued

Drake and associates 1982 [3.8]	#		0.406	142	1147	94*	1294*	The YS in tension tests to be lower than in bending tests. E in tension tests is higher than in bending tests.
	#		0.432x0.635	134	1196	123*	1588*	
	#		0.483x0.635	128	1196	123*	1646*	
Kusy and Dilley 1984 [3.9]	Wildcat	3Sx0.203				188**		The E value remains at a level of about 200Gpa in three and four point bending tests, which shows independent of the amount of work-hardening. This result contrasts with other dental literature, in which a broader range of values has been reported, i.e. 126Gpa- 262Gpa.
	Permachrome	0.254				198**		
	Wildcat	0.457				192**		
	Wildcat	3Sx0.203				191***		
	Permachrome	0.254				204***		
Yoshikawa and associate 1981 [3.10]	Wildcat	0.457				192***		E value in bending tests are about 10% lower than the standard E value.
	Permachrome	0.406				181*		
	Permachrome	0.457				179*		
	Permachrome	0.508				178*		
Goldberg and associate 1983 [3.11]	Permachrome	0.406		148		177*		E value in bending is higher than the tension tests, both are lower than the standard E value.

Austenite stainless steel wires which name not given.

S number of strands

* Value are obtained by cantilever bending

** Value are obtained by 3 point bending test.

*** Value are obtained by 4 point bending tests.

**** All above tension value are obtained by uniaxial tensile test.

• .All above value are the mean value for the total numbers of same wires tested. Refer to the original articles for other specifications of the wires and company details.

•

Table 3.2: Summary of comparison E for as received and heated treatmented austenite stainless steel orthodontic wires by previous investigators.

Investigator Year	Wire type	Size (mm)	Tension test			Bending test		
			YS(MPa)		E(GPa)	YS (MPa)		E(GPa)
			As received	Heat treated	As received	Heat treated	As received	Heat treated
Goldberg and associate 1977 [3.12]	Permachrome	0.254x0.508	1780†	354†	149	183		
	Permachrome	0.406	1880†	302†	156	203		
	Permachrome	0.762	1010†	411†	158	200		
	Permachrome	0.914	1550†	277†	157	177		

The heat treatment condition was heat treatment of five minutes at 1010°C, followed by air cooling

Comment: Goldberg and associate found E to be 20% lower in as-received wires. They also found the same wires after annealing, (heat treatment of five minutes at 1010°C, followed by air cooling) to have a value of E closer to the standard value. Thus, they suggested that E is 20% decreased from the standard value by the extensive cold working associated with the wire manufacturing process. This results agrees very strongly with the earlier results of Zapffe[3.13] and Howe and associate.[3.14].

Continued						
Asgharnia and Willian 1986 [3.15]	Tru-chrome	0.254	161	175	232	234
	Tru-chrome	0.356	159	168	218	231
	Tru-chrome	0.406	177	176	217	237
	Tru-chrome	0.457	172	179	216	234
	Tru-chrome	0.508	181	182	215	231
	Tru-chrome	0.432x0.635	163	172	192	211
	Tru-chrome	0.457x0.635	165	159	194	206
	Permachrome	1.016	161	159	159	169

The heat treatment condition were heat treatment of ten minutes at 482°C, followed by air cooling.

Comment: Asghamnia and Brantley found higher value E in bending for most of the heat-treated (heat treatment of ten minutes at 482°C) stainless steel wires. And they found both the values of YS and E are similar between the as received and heat-treated wires. This probably due to the low temperature (482°C) annealing.can not totally recovery the cold work structure.

Continued

Khier and associate 1988 [3.16]	Tru-Chrome	0.406	2000‡	2482‡	181	176	2137‡	2275‡	215	238
	Tru-Chrome	0.432x0.635	1792‡	2413‡	165	172	1654‡	2137‡	190	208
	Tru-Chrome	0.762	1654‡	2413‡	177	181	1930‡	2137‡	186	202
	Tru-Chrome	1.270	1310‡	2068‡	173	172	2413‡	2689‡	163	178
	Permachrome	0.406	2206‡	2413‡	201	197	2551‡	3240‡	221	239
	Permachrome	0.432	1930‡	2000‡	196	192	2620‡	2826‡	198	215
	Permachrome	0.762	1517‡	1861‡	189	185	2068‡	2275‡	194	208
	Permachrome	1.295	1310‡	1517‡	184	181	1723‡	1861‡	180	192

The heat treatment condition were heated at 482°C for ten minutes followed by air cooling.

Comment: Khier and associate found higher value E in bending tests for both as received and heat treated wires (heated at 482°C for ten minutes). They also found E value are similar between the as received and heat treated wires; YS value are higher in heat treated wires than the as received wires in both tension and bending tests.

† Value for 0.1% permanent offset.

‡ Value for 0.2% permanent offset.

*All above tension value are obtained by uniaxial tensile test., and all above bending values are obtained by cantilever bending tests

*All above value are the mean value for the total numbers of same wires tested.

The above results show conflicting values for the mechanical property measures. Later, the results for this study will be seen to differ somewhat from the above. The reasons for this difference will be given in the discussion at the end of the chapter.

3.2. Testing theory.

This section sets out the theory which will be used in later sections to interpret and apply the experimental results.

3.2.1. The tensile test.

The modulus of elasticity (E), or Young's modulus is the slope of the initial linear portion of the stress-strain curve (see fig.3.1). It is a measure of the stiffness of the material. The greater the modulus, the smaller the elastic strain resulting from the application of a given stress.

The modulus of elasticity is determined by the binding forces between atoms. Since these forces cannot be changed without changing the basic nature of the material, it follows that the modulus of elasticity is one of the most structure insensitive of the mechanical properties. Alloying, heat treatment and cold work affect it only slightly.

The tensile test results can be presented three different diagrammatic forms:

- as load-extension diagrams;
- as engineering stress-strain diagrams, or
- as true stress-strain diagrams.

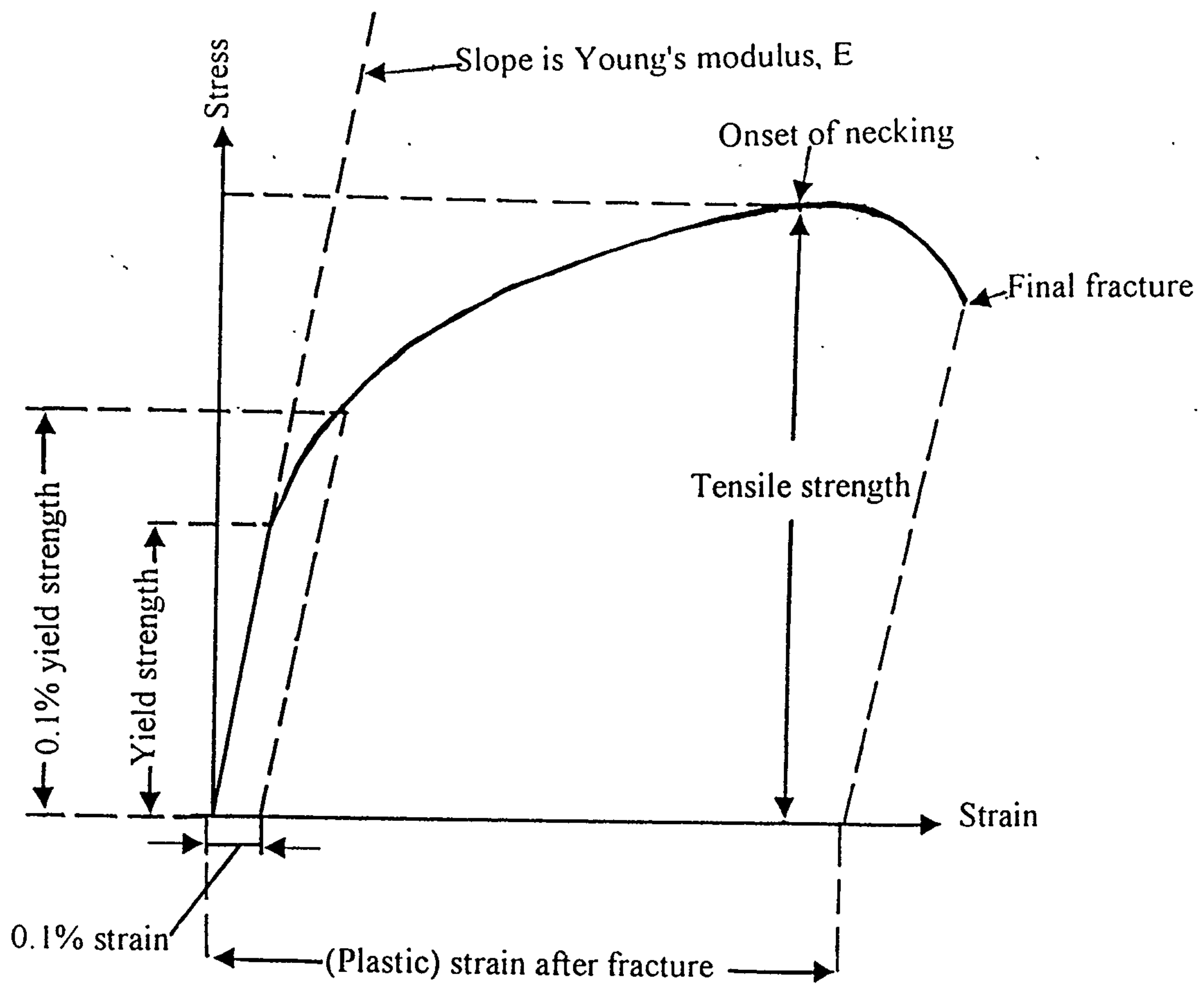


Fig. 3.1 The engineering stress-strain curve.

A load-extension diagram is rather crude since it depends on the specimen's dimensions. The other two forms of diagram are, however, independent of this size effect.

An engineering stress-strain curve is constructed from the load elongation measurements. (fig. 3.1) The stress considered in this stress-strain curve is the average longitudinal stress in the tensile specimen. It is obtained by dividing the load (F) by the original area (A_0) of the cross section of the specimen, i.e.

$$\sigma = \frac{F}{A_0}$$

The strain used for the engineering stress-strain curve is the average linear strain; obtained by dividing the elongation of the gage length of the specimen, δ , by its original length, L_0 . So,

$$\varepsilon = \frac{\delta}{L_0} = \frac{\Delta L}{L} = \frac{L - L_0}{L_0}$$

Since both stress and strain values are obtained by dividing the load and elongation by constant factors, an engineering stress-strain curve will have the same shape as the corresponding load-elongation curve.[3.19],[3.4]

An engineering stress-strain diagram is constructed by considering the initial sample size when calculating the stress and strain. Whereas a true stress-strain diagram is based on the instantaneous dimensions of the sample during the test (Fig.3.2).

True and engineering stress-strain results are related through the following formulae [3.4]

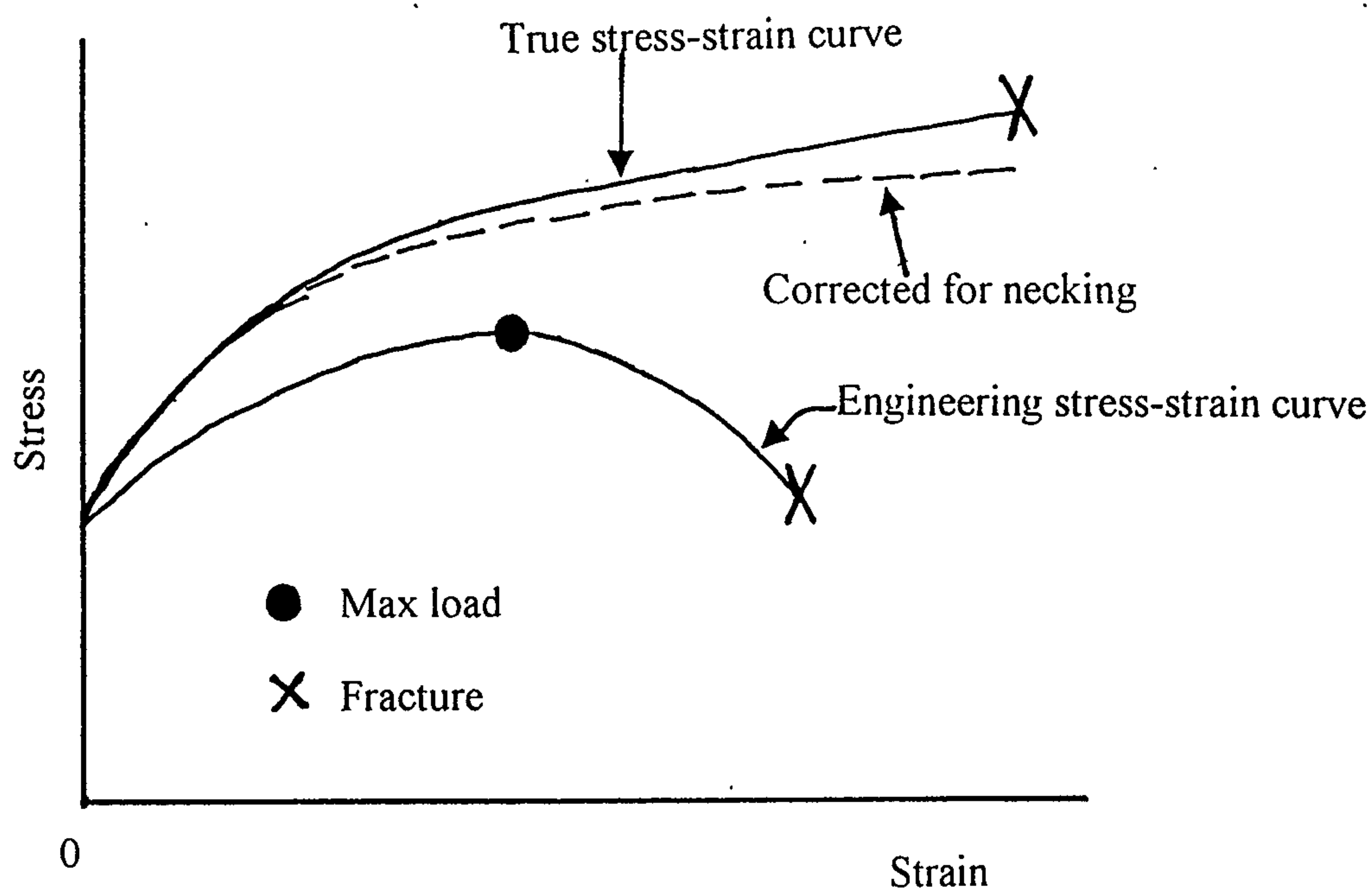


Fig. 3.2 Comparison of engineering and true stress-strain curves.

$$\sigma_t = \sigma(1+\varepsilon);$$

$$\varepsilon_t = \ln(1+\varepsilon)$$

where

σ_t is the true stress;

σ is the engineering stress;

ε_t is the true strain; and

ε is the engineering strain.

The analysis in this chapter follows the standard approach of presenting results through engineering stress-strain diagrams. Engineering tension tests are widely used to provide basic information on the strength of materials. The difference between engineering and true stress-strain curves is minimal for plastic strain less than 10%.

In an engineering stress-strain curve (see fig.3.1), the elastic region stress is linearly proportional to strain. The slope of this initial linear portion of the stress-strain curve is the value of elastic modulus. When the load exceeds a value corresponding to the yield strength, the specimen undergoes gross plastic deformation. It is permanently deformed even if the load is released to zero. Yield strength (σ_Y) is F/A_0 at the onset of plastic flow. The yield strength is the stress required to produce a small amount of plastic deformation. The yield point is a gradual transition from elastic to plastic behaviour for most materials. This property is determined by the stress corresponding to the intersection of the stress-strain curve and a line running parallel to the elastic part of the curve at an offset of a specified strain. The offset is usually specified as a strain of 0.1 or 0.2 percent. [3.4]

In the case of stainless steel materials, the stress to produce continued plastic deformation increases with increasing plastic strain, this is termed "work hardening". The volume of the specimen remains constant during plastic deformation and, as the specimen elongates, the cross-sectional area decreases uniformly along the gage length. [2.34]

Eventually a point is reached where the decrease in specimen cross section area is greater than the increase in deformation load arising from strain hardening. This condition will first be reached at some region of the specimen that is slightly weaker than the rest. All further plastic deformation will concentrate in this region, and the specimen will begin to neck or thin down locally. [3.20]

Because the cross-section area is now decreasing far more rapidly than the deformation by strain hardening, the actual load required to deform the specimen falls off and the engineering stress continues to decrease until fracture occurs.[3.20, 3.4, 3.19]

3.2.2. Fracture surface analysis of the tensile test specimens. [3.4, 3.17]

Tensile fracture examination can give a possible clue to low values of strength or ductility of the specimen. Tensile test fracture is the separation of the wire into two pieces in response to an imposed stress. There are two modes of fracture: ductile and brittle.

Any fracture process involves two steps; crack formation and propagation. The mode of fracture is highly dependent on the mechanism of crack propagation.

Ductile fracture is characterised by the following features:

- There is extensive plastic deformation in the vicinity of an advancing crack.
- The process proceeds relatively slowly.
- There is generally evidence of appreciable gross deformation at the fracture surfaces (twisting, tearing, and a fibrous appearance). Ductile fractures start with the initiation of voids, most commonly at second-phase particles.
- The ductile fracture crystallographic mode is shear. A shear fracture occurs as the result of extensive slip along the active slip plane.

On the other hand, for a brittle fracture, the following features are found:

- There is very little accompanying plastic deformation.
- Cracks spread extremely rapidly.
- The surface appears bright or granular. For a brittle fracture, transgranular-cleavage surfaces usually contain a large number of cleavage steps and a “river pattern” of branching cracks. The surfaces of brittle fractures are smooth.
- The crystallographic mode for brittle fracture is cleavage. This type of fracture is controlled by tensile stresses acting normal to the crystallographic cleavage plane.

In reality, tensile fracture surfaces frequently consist of a mixture of fibrous and granular fractures, i.e. a mixture of ductile and brittle fractures.

In the tensile fracture of moderately ductile metals, plastic deformation eventually produces a necked region. Fracture begins at the centre of the specimen and then extends by shear. This results in the familiar “cup-and – cone” fracture. Very ductile metals may actually draw down to a line or a point before separation. This kind of failure is usually called rupture.

Fig.3.3 shows the stages of ductile “cup-and-cone” fracture produced through uniaxial tension. Necking begins at (A) the point of plastic instability. At (A) the increase in strength due to strain hardening fails to compensate for the decrease in cross-sectional area. This failure occurs at maximum load.

The formation of a neck introduces a triaxial state of stress in the region. A hydrostatic component of tension acts along the axis of the specimen at the centre of the necked region. Many fine cavities form in this region(B). Under continued straining, these cavities grow and coalesce into a central crack(C). This crack grows in a direction perpendicular to the axis of the specimen until it approaches the surface. It then propagates along localised shear planes at roughly 45° to the axis to form the “cone” part of the fracture(D). The central crack forms early and tends to concentrate the deformation at its tip in narrow bands of high shear strain. These shear bands are at angles of 50° - 60° to the transverse direction. Sheets of voids are nucleated in these bands and the voids grow and coalesce into local fractures of the void sheet.

While the average direction of crack growth is radially outward in the direction transverse to the tensile axis, on a finer scale the crack zig-zags back and forth across the transverse plane by void sheet formation. Thus,

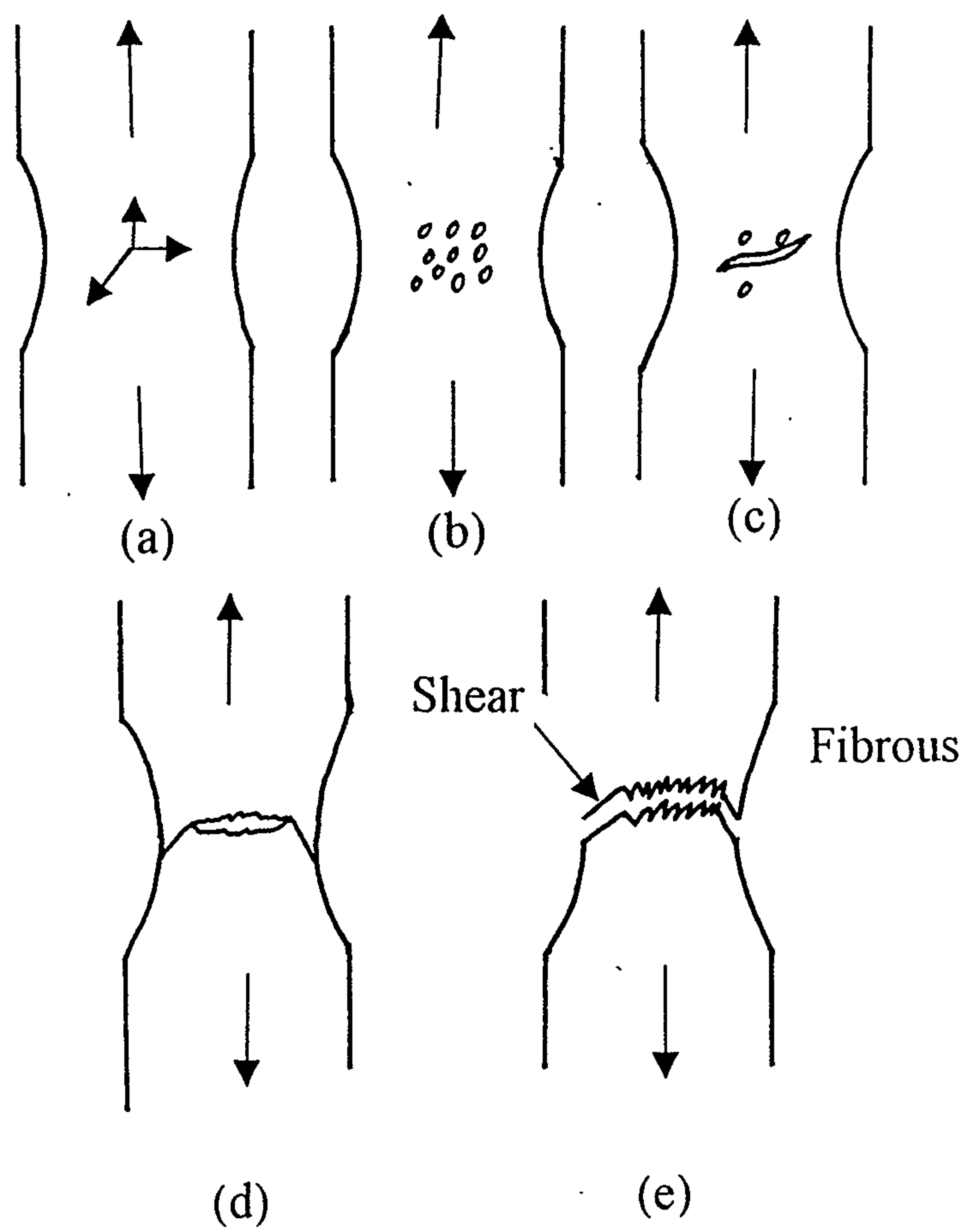


Fig. 3.3 Stages in the formation of a cup and cone fracture.

crack growth in a ductile fracture is essentially through a process of void coalescence (E).

Nonaxial loading will cause asymmetrical type - "partial cup and cone fracture". Lack of symmetry is caused by nonhomogeneity of the material or by a defect or flaw of some sort such as segregation, a blowhole, or a inclusion of foreign matter such as slag. The "partial cup-cone" type tensile fracture as described here will be observed in the results presented shortly.

3.2.3. Flexure testing. [3.18, 3.19]

Flexure testing results from the application of a symmetric bending moment to a sample. Such a moment, when applied to a component so as to result in bending, is referred to as a bending moment and is denoted by M . The standard convention: is that to the left of any section, a anti-clockwise moment is considered negative.

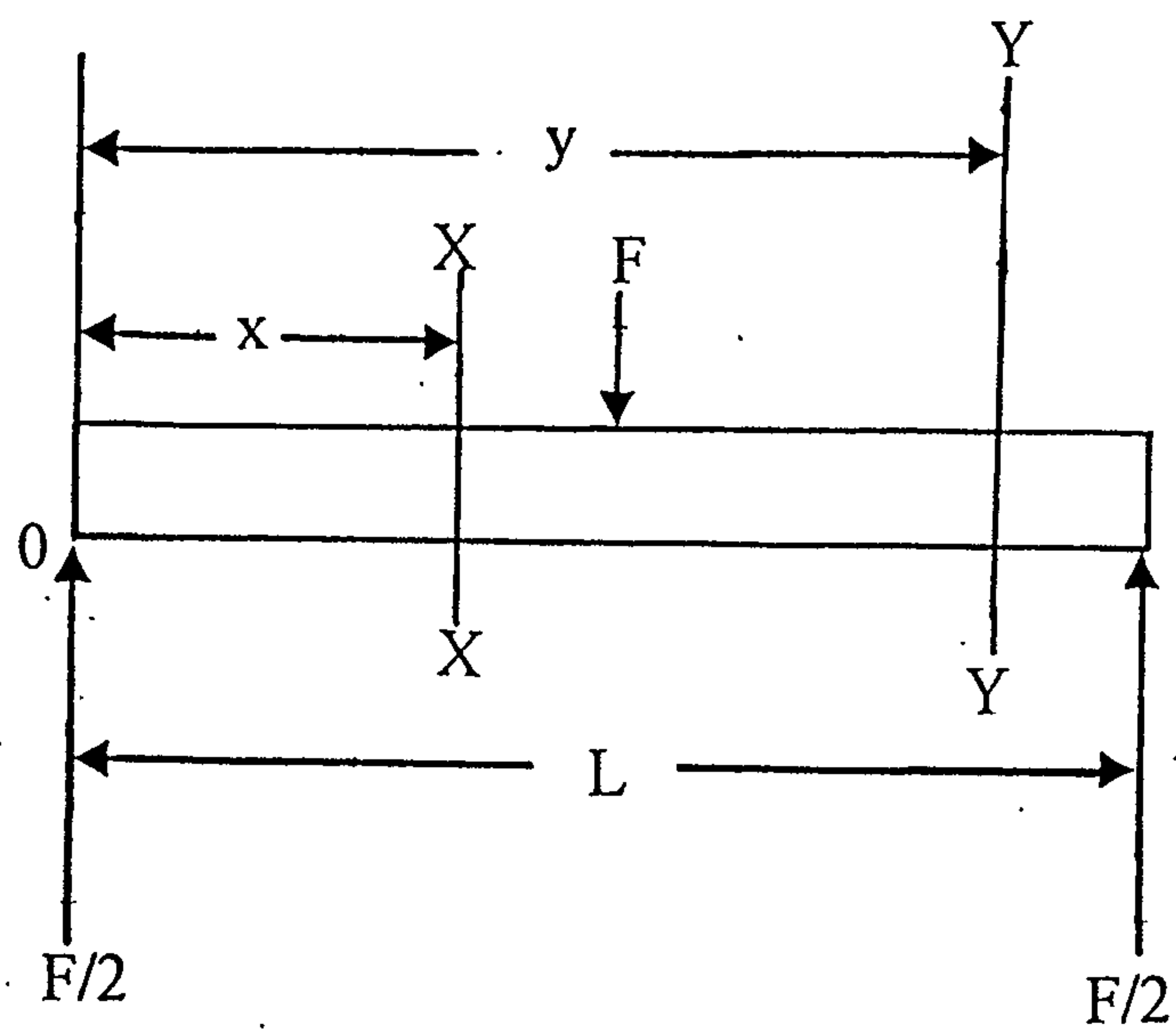
3.2.3.1. Three point bending moment.

Clearly, from figure 3.4, the load per support is $F/2$. The bending moment at any section is the net effect of all forces acting on that part of the wire to the left of the section, clockwise direction being taken as positive.

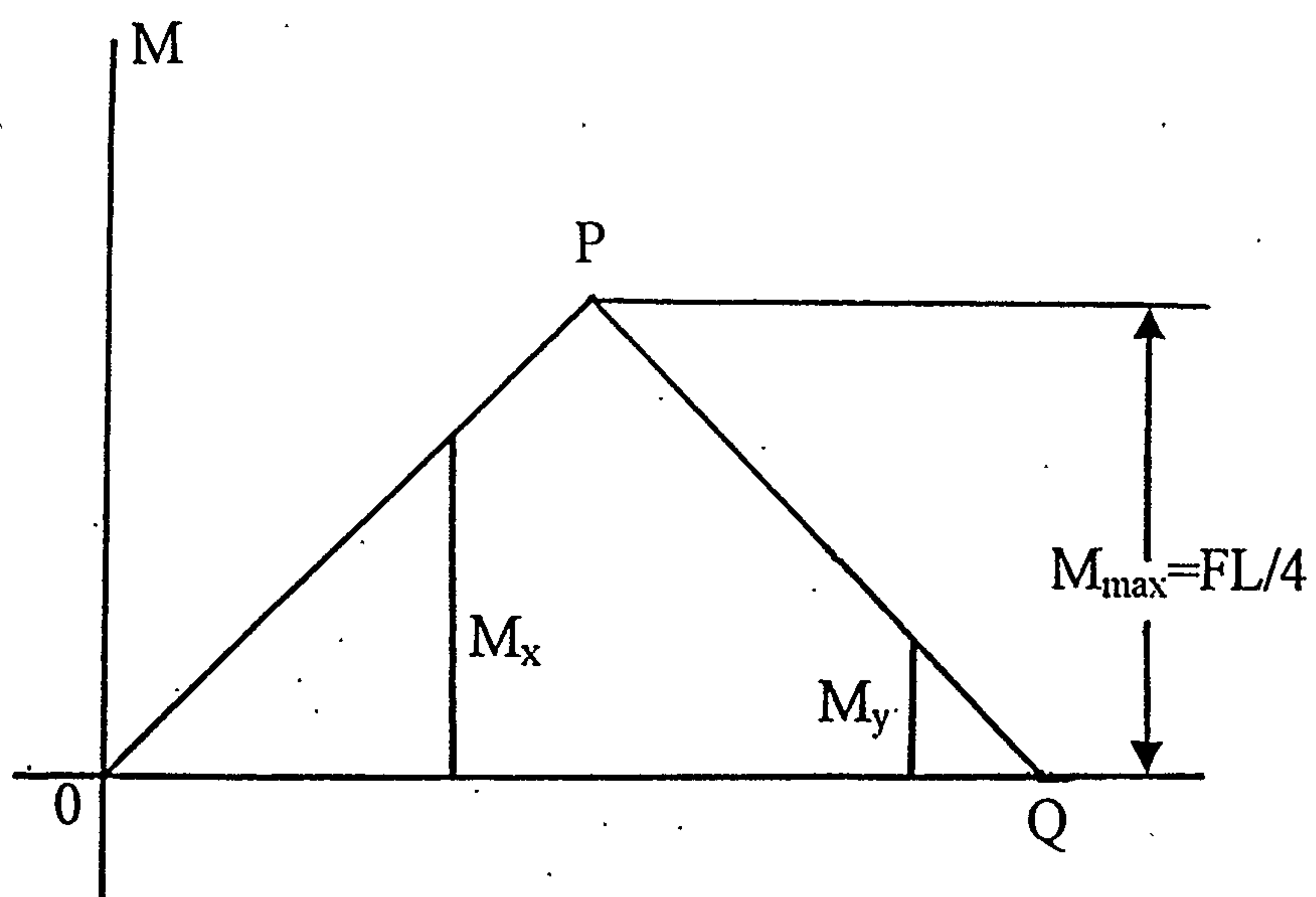
At any section XX to the left of the load,

$$M_x = \frac{F}{2}x$$

where $x < \frac{L}{2}$.



(a)



(b)

Fig. 3.4 Diagrammatic representation of the three point bending moment.

Thus for the left half of the wire, M is proportional to the distance from the left support and its graph is the straight line OP . When $X=0$, P at the left support, $M=0$.

At the centre

$$M = \frac{F}{2} \times \frac{L}{2} = \frac{FL}{4} \text{ where } x = \frac{L}{2}$$

This value of M is clearly the maximum value.

At any section YY to the right of the load:

$$\begin{aligned} My &= \frac{F}{2}y - F\left[y - \frac{L}{2}\right], \\ &= \frac{F}{2}(L - y) \end{aligned}$$

where $y > \frac{L}{2}$.

At the right support when $y=L$, $M=0$

Thus for the right half of the wire, M is proportional to the distance from the right support and its graph is the straight line PQ .

3.2.3.2. Four point bending moment

Figure 3.5 represents a 4 point bending in which each support load is $F/2$. At any section XX ,

$$M_x = - (F/2)X \text{ where } x < a.$$

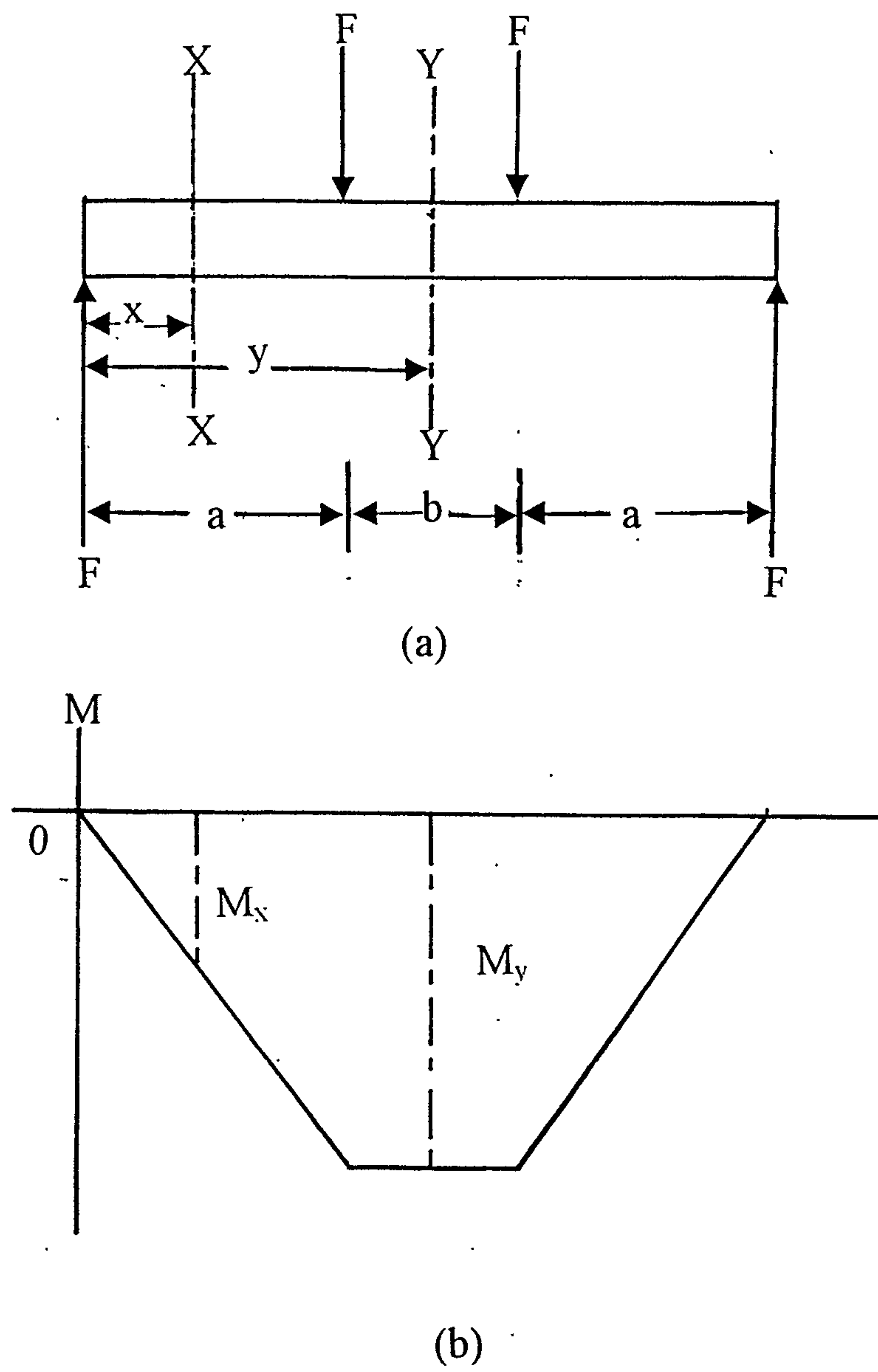


Fig. 3.5 Diagrammatic representation of the four point bending moment.

Hence M is proportional to X , i.e. it is zero at the ends and has a value at the supports of Fa .

At any section YY ,

$$\begin{aligned} M_y &= -(F/2)y + (F/2)(y - a) \\ &= -(F/2)a \end{aligned}$$

Since a is a constant, the bending moment between the supports is also constant.

3.2.4. Three point and four point bending [3.18, 3.19, 3.21, 3.22, 3.23]

A bending moment applied to a wire produces tensile and compressive strains separated by an unstrained or “neutral” plane as in Fig. 3.6.(a).

The region where the wire section and this neutral plane intersect is known as the neutral axis of the section. Since orthodontic wires are symmetrical, this axis is mid way between the loaded edges. The strain at any point due to bending (and hence the stress σ) is proportional to the distance y from the neutral plane or axis. (Fig. 3.6 (b)).

The force generated by the deflection of the wire is related to the material's intrinsic properties as well as to the geometry of the wire and its supports. A number of assumptions in this analysis have been made involving geometry and materials behaviour. These assumptions are:

- cross-sectional symmetry of the wire;
- in-plane bending;
- elastic activation in the wires; and

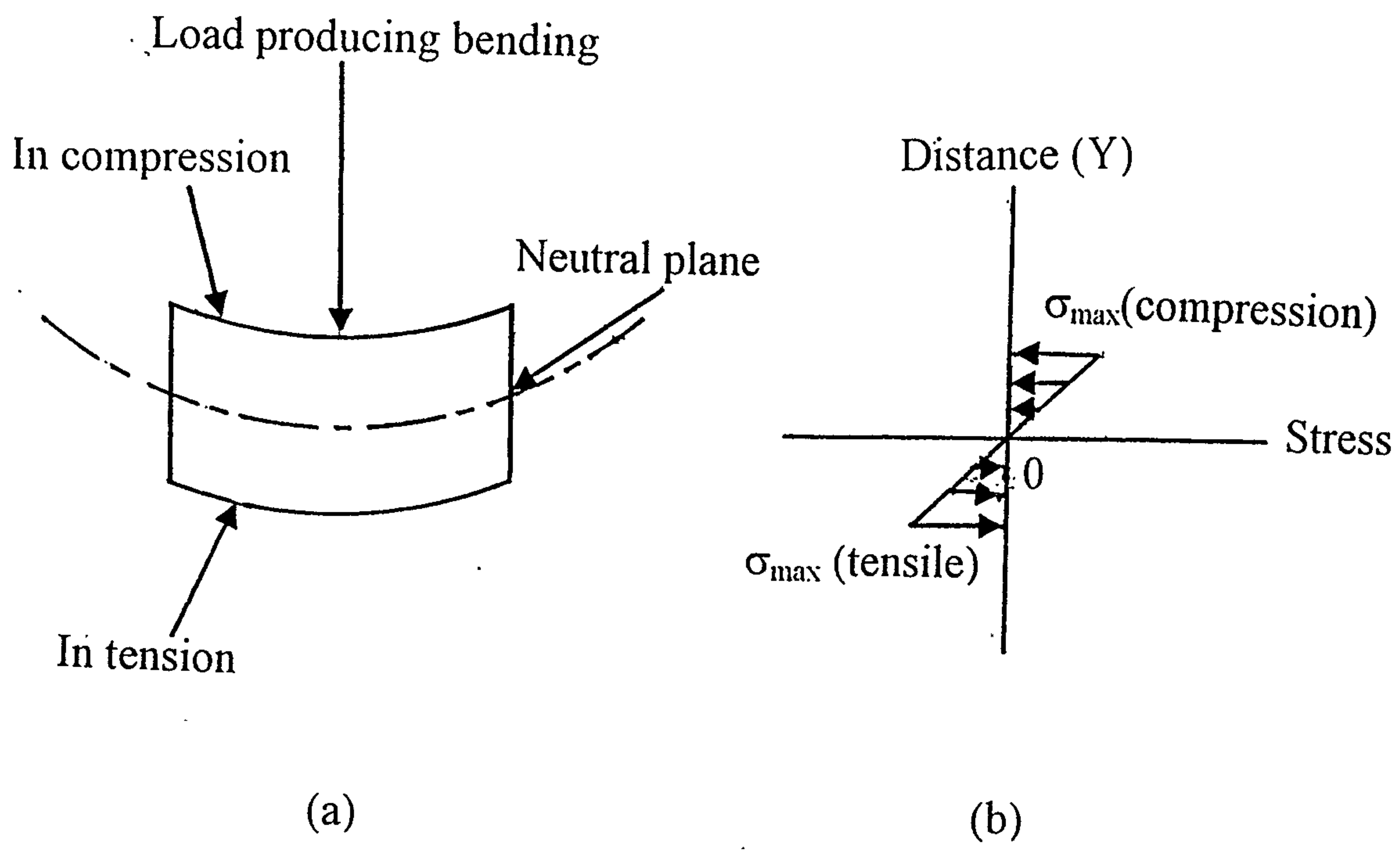


Fig. 3.6 Diagrammatic representation of the stress distribution of the round wire undergoing the bending.

- values of the elastic modulus being equal in tension and compression.

3.2.4.1. Three point bending.

In three point bending, the following formula describes the relationship between stress and strain when a force is applied at the midspan and the ends are free to slide:

$$\frac{M}{I} = \frac{E}{R} = \frac{\sigma}{y}$$

where,

M=bending moment;

I=second moment of area of the section about the neutral plane, for a circular section, $I = \frac{\pi d^4}{64}$, where d=diameter of the section;

E=Young's modulus of elasticity for the material;

R=radius of curvature; and

σ =the tensile or compressive stress in the material at any distance y from the neutral plane.

Thus,

$$\sigma = \frac{M}{I} y,$$

Then, the tensile or compressive strain (ε) in the material at any distance y from the neutral plane the strain will be:

$$\varepsilon = \frac{\sigma}{E} = \frac{EY/R}{E} = \frac{Y}{R}$$

For the stress and the strain on the surface of the circular wires: [3.23]

(fig.3.7)

$$\sigma = \frac{MY}{I} = \frac{\left(\frac{F}{2}\right)\left(\frac{L}{2}\right)\frac{D}{2}}{\frac{\pi D^4}{64}} = \frac{8FL}{\pi D^3} \quad \varepsilon = \frac{6D\Delta L}{L^2}$$

where,

D is diameter of the wires; and

ΔL is the deflection in bending.

3.2.4.2. Four point bending.

In four point bending, the following formula describes the relationships between stress and strain on the surface of the circular wires, when the force is applied at the wire and the ends are free to rotate [3.23] (fig.3.8).

$$\sigma = \frac{MY}{I} = \frac{a\left(\frac{F}{2}\right)Y}{\frac{\pi D^4}{64}} = \frac{\left(\frac{L_1 - L_2}{2}\right)\left(\frac{F}{2}\right)\left(\frac{D}{2}\right)}{\frac{\pi D^4}{64}} = \frac{8(L_1 - L_2)F}{\pi D^3}$$

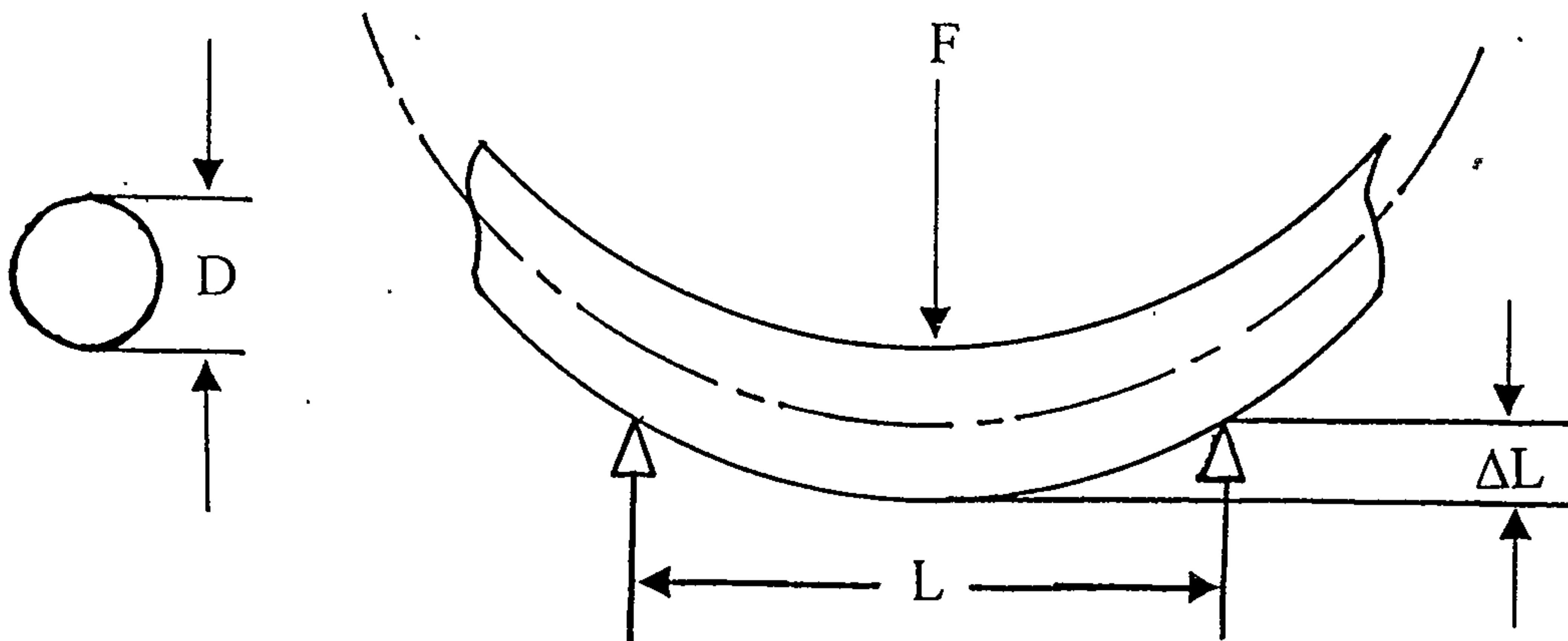


Fig. 3.7 Diagrammatic representation of the three point bending.

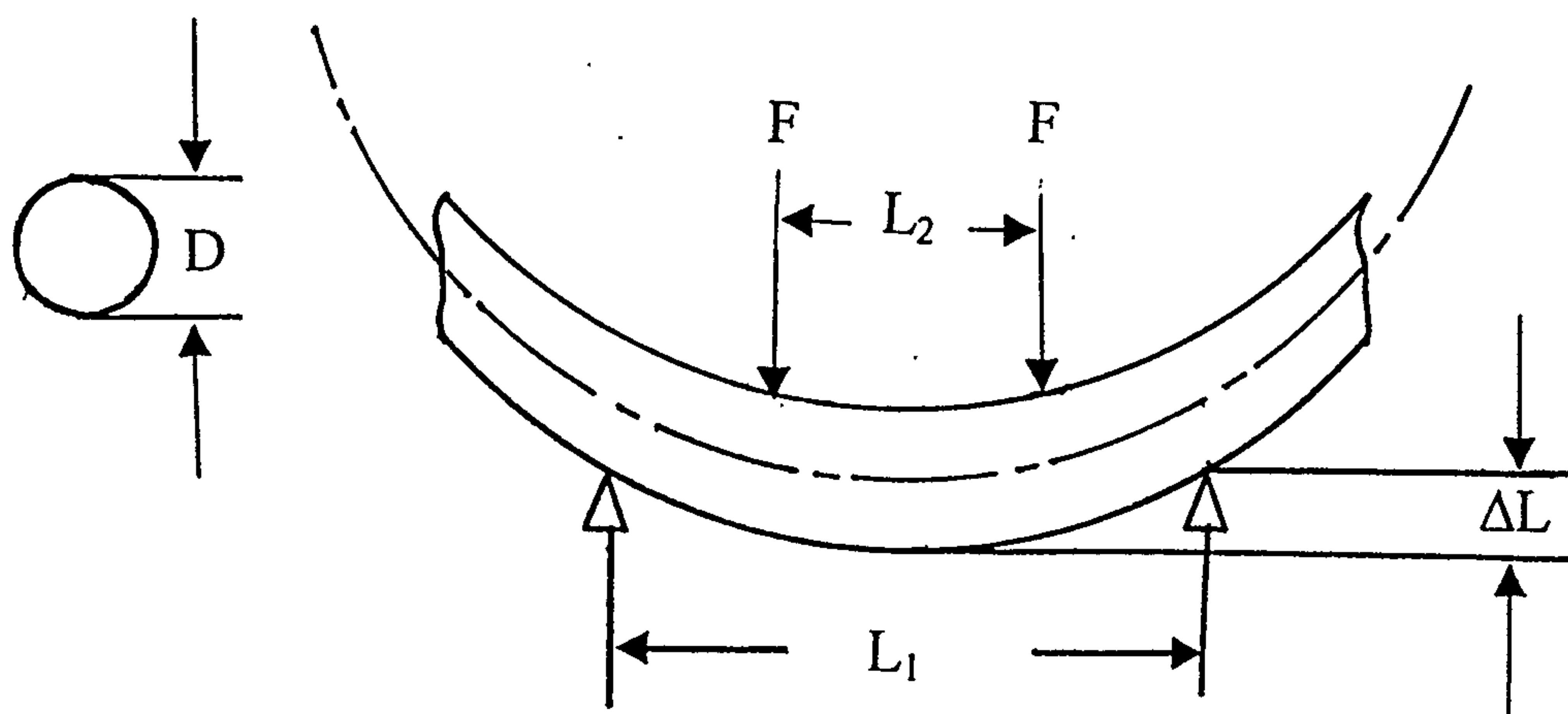


Fig. 3.8 Diagrammatic representation of the four point bending.

$$\varepsilon = \frac{6((L_1 - L_2)D\Delta L)}{L_1^3 - 3L_1L_2^2 + 2L_2^3}$$

3.3. Materials and Methods

3.3.1. Materials

The previous section discussed the theoretical basis for the approach of this chapter. This section discusses the materials upon which these tests were conducted.

The four austenitic stainless steel 302 orthodontic wires tested in the present study, together with their diameters, manufacturers' names, symbols and batch numbers have been listed in table 2.1 of chapter 2.

As described in chapter 2 (section 2.4), 0.52mm Premium Plus and 0.88mm Dentaurem stainless steel orthodontic wires are ready for orthodontic applications. The 1.60mm & 2.18mm wires are early drawing stage wires for the 0.52mm and 0.88mm wires respectively.

In order to compare the elastic moduli in the annealed and as-received conditions, a set of three 0.52mm WF wires (Premium Plus) were heated in a vacuum furnace at 1000°C for half an hour and then cooled in the vacuum furnace to room temperature.

Before presenting the results of the wires mechanical properties tests, the microstructure characteristics of these four wires are summarised here based upon the TEM and XRD experimental results which are detailed later in this thesis:

- the four wires are composed of austenitic and α -martensite phases and the ordering of martensite phase amounts is $GS > WF > GS > GF$;
- the four wires have (111)austenite and (110)martensite texture;
- the subgrains in the WF, WS and GF wires are elongated whereas the grains in the GS wires are hexagonal.;
- the ordering of subgrains or grain sizes is $WF < GF < WS < GS$;
- austenitic and martensitic twinning structures exist in all of these wires;
- small angle grain boundaries exist in the WF, WS and GF wires.

3.3.2. Experimental method

3.3.2.1. Tensile tests.

All four wires were supplied from the manufacturer in coiled form. The specimens were made by cutting the wires from the coil to a length of approximately 300mm. The wires were then straightened by hand prior to testing, in accordance with the specifications of the British Standards Institute (1976)[3.24]. Before the tests, each specimen's diameter was measured by a micrometer.

The tensile tests were carried out on a universal testing machine – Autograph AG-50NE, Shimadzu, Kyoto, Japan.

The grips used were the wrap-around type suggested by Ware and Masson (1975)[3.25] and a test length of 80mm was set between the grips. An extensometer with gauge length of 10mm was used to eliminate erroneous

readings for the elastic modulus caused by slippage in the grips and machine compliance. To further avoid the slippage of the extensometer on the wires as seen by Drake et al (1982)[3.8], I also used a small drop of glue to fix the extensometer gage on to the wires. Since the surfaces of the gages were ground down in the extensometer, the gage length accuracy was affected. This will be considered further in the later discussion section.

Three specimens of each wire were tested. An initial load of 0.5N was used to eliminate slack in the wires in the manner suggested by the British Standards Institute (1976),[3.24]. Stress-strain curves were obtained from the tests and these curves were used to calculate 0.1% yield strength (YS), U.T.S., elastic modulus (E) and the elongation at fracture.

After the tensile test, a 505 Phillips scanning electron microscope (SEM) in the back scatter mode was used to examine the cross-sectional and longitudinal sections of the fracture parts of the specimens. This technique was used because, as mentioned above, SEM micrographs can show the microstructure of the fractured part, thereby assisting with the identification of the mechanisms of the fracture. This identification can be used to confirm the results concerning the ductility of the wire as determined in the tensile tests.

3.3.2.2. Three point bending tests

In the 3 point bending tests, prior to testing, the four wires were cut to a length of 45mm and then straightened by hand. Then, the wires were placed on two supports, 14mm apart. A single point force was applied vertically to the midpoint of the wires via a push rod connected to a load cell. Loading of the test specimens at a cross head speed of 1.0mm/min was continued until

4% strain was reached. At this point, the direction of the load was reversed so that the specimens were unloaded at the same rate, back to the original state.

Three samples of each wire were tested. The stress-strain curves were obtained from the 3 point bending test, and were used to calculate the 0.1% yield strength and elastic modulus.

After the tests, a scanning electron microscope (SEM) was employed to check the tensile surfaces at the bending part.

3.3.2.3. Four point bending tests

In the 4 point bending test, prior to testing, the 0.52mm and 0.88mm wires were cut to a length of 45mm and straightened by hand. The wires were then placed on two supports, 14mm apart. The 1.62mm and 2.18mm wires were cut to lengths of 60mm and also straightened by hand. They were then placed on supports, 32.5mm apart. Two equal forces were applied vertically to each of the wires. The two forces were applied to points 2.5mm from the centre in each direction.

These forces were applied vertically to the wires via two push rods connected to a load cell under the same testing conditions as in the 3 point bending tests.

Three samples of each wire were tested. The stress-strain curves were obtained from the 4 point bending test, and were used to calculate the 0.1% yield strength and elastic modulus.

A scanning electron microscope (SEM) was used to examine the tensile surfaces of the bending part.

3.4. Results

3.4.1. Tensile test

Fig.3.12A and Fig 3.12B show representative stress strain curves from the tensile tests on WF(as received), WF(heat treated), WS (as received), GF (as received) and GS wires (as received). In the case of Fig 3.12A, five curves are shown in which the extensometers was used. In contrast, Fig 3,12B shows four curves when an extensometer was not used,

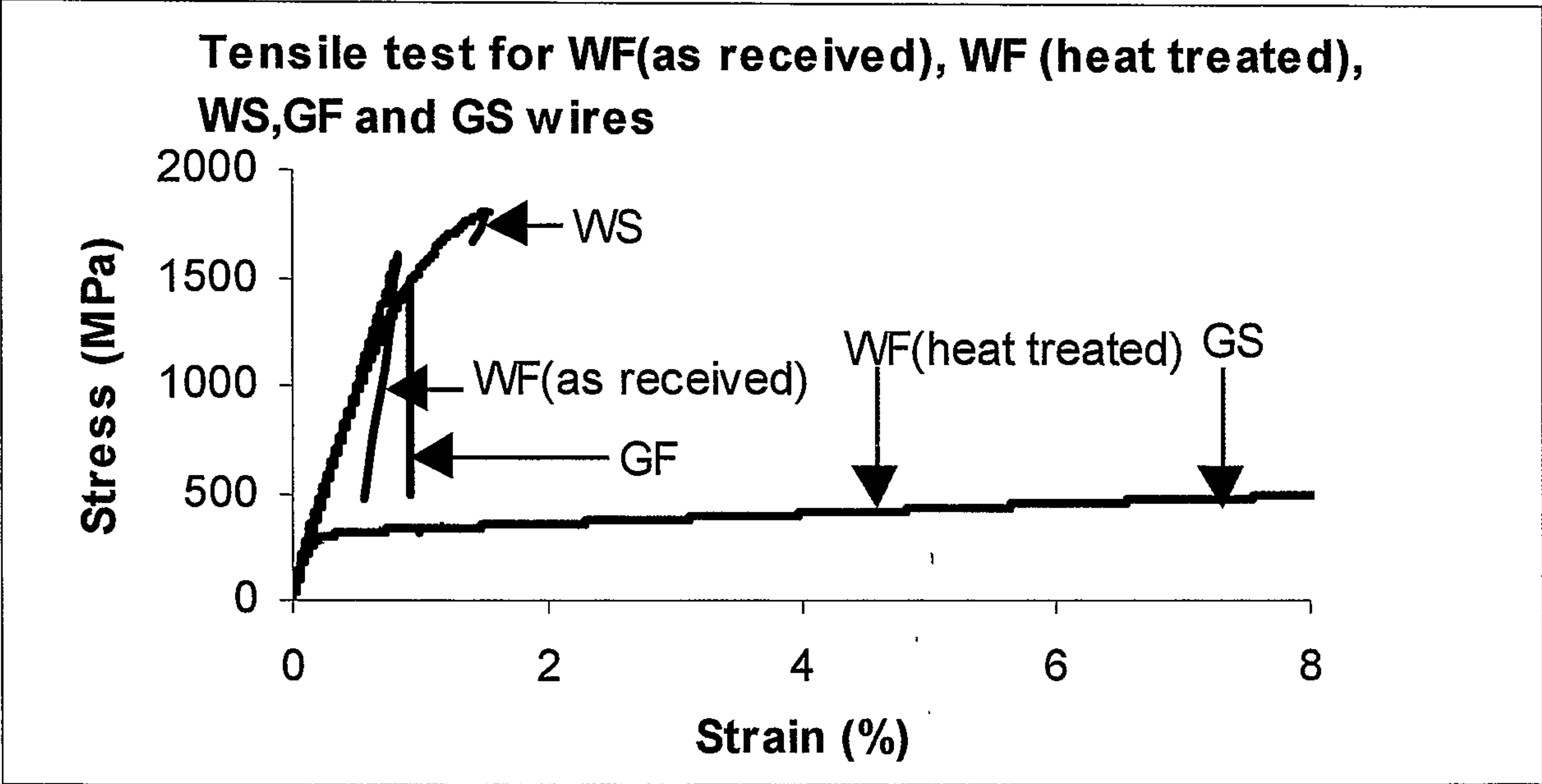


Fig. 3.12A Representative stress/strain curves of tensile test by using the extensometer for five wires.

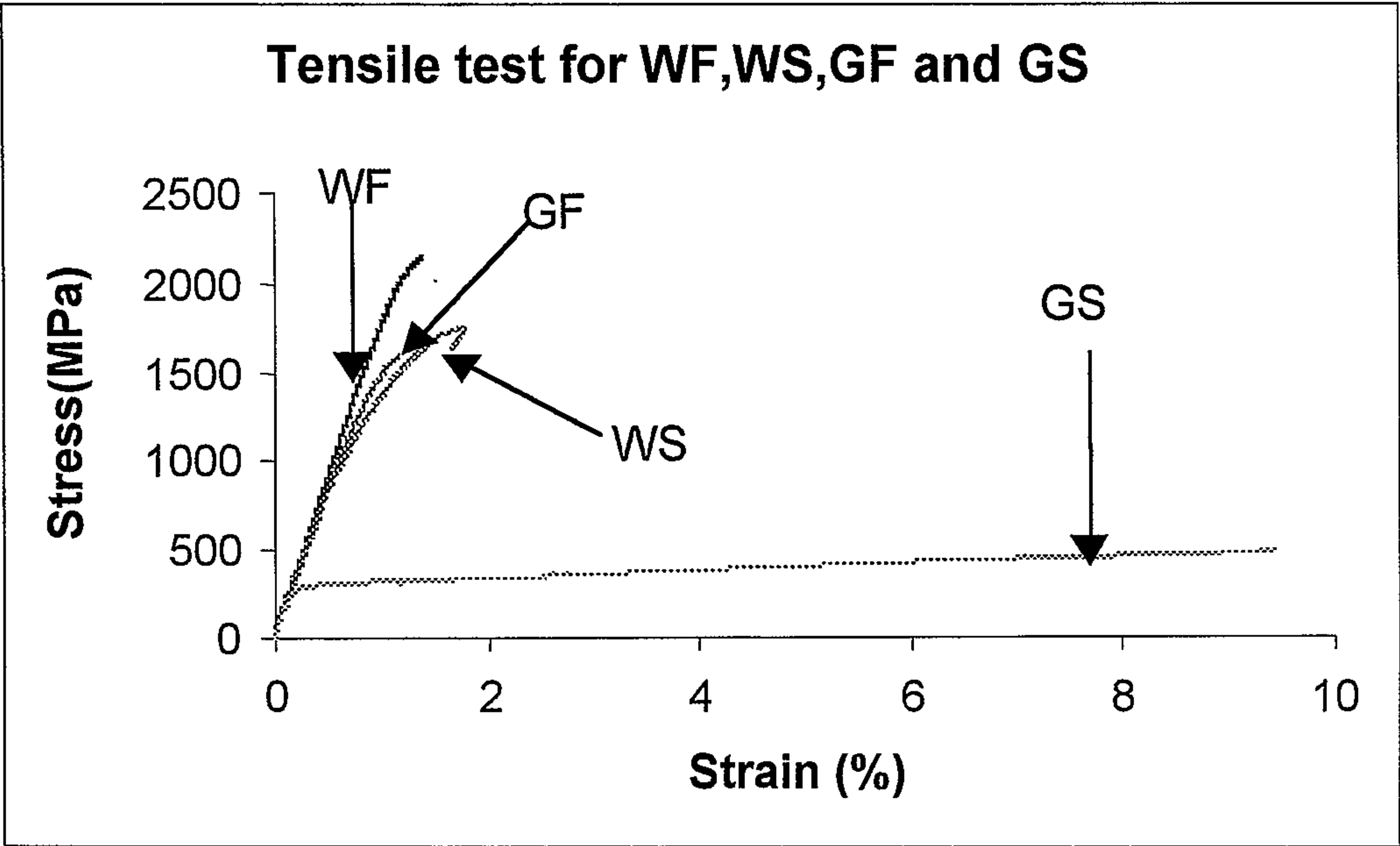


Fig. 3.12B Representative stress/strain curves of tensile test without using the extensometer for four wires.

From these figures and detailed results in table 3.3, the following results can be concluded:

- In the tensile tests using the extensometer, all five wires have nearly identical values for the elastic modulus within experimental error. The results are tabulated below in table 3.3. This value for E is approximately 200GPa, which is consistent with the standard stainless steel elastic modulus which is 200 GPa. As will be seen later, the value for E as determined through the tensile tests with an extensometer is different from the values from the tensile tests not using an extensometer and from the flexure. Possible reasons for this difference will be considered in the discussion section of this chapter.
- In the tensile tests not using an extensometer, the mean value of E for all the as-received wires was approximately 170GPa, which is 15% lower than the standard E values. The possible reasons for this will be discussed later in this chapter.
- Yield stress results are also listed in table 3.3. The ordering of 0.1% YS value for the five wires were:

$$WF(\text{as received}) > GF > WS > GS = WF(\text{heat treated})$$

The reason for the above ordering of yield strength will be discussed later.

- The shape of stress-strain curves of these five wires shows that WF wires after heat treatment and GS wires are ductile materials and that GF and WS wires and as received WF wires are brittle materials.

The tensile parameters, such as 0.1% YS, UTS (ultimate tensile strength), elongation at fracture, the value of E and the measuring range for E for each wire are given in tables 3.3.

Table 3.3A. The mechanical properties as found from the tensile test with using the extensometer.

Specimen	Wires condition	Test No.	Ave. wire's diameter	E (GPa)	Measuring range of E		0.1%YS (MPa)	UTS (MPa)	Elongation at fracture(%)
					Stress range (MPa)	Strain range (%)			
WF	As received	1	0.520	190	100-1000	0.04-0.5	>1600	1600	0.81
	As received	2	0.520	192	250-990	0.15-0.55	>1520	1520	0.82
	As received	3	0.515	199	0-1011	0-0.5	>1850	1850	0.93
		Mean	0.518	194±5			>1656	1656±250	0.85±0.12
WF	Heated at 1000°C for half hour	1	0.515	199	50-200	0-0.085	280	#	#
	Heated at 1000°C for half hour	2	0.515	194	20-150	0-0.08	280	#	#
	Heated at 1000°C for half hour	3	0.517	195	20-125	0-0.057	260	#	#
		Mean	0.516	196±5			273±20	#	#

WS	As received	1	1.60	199	50-500	0-0.25	1250	1750	1.52
	As received	2	1.61	197	20-250	0-0.125	1100	1750	1.75
	As received	3	1.60	204	30-400	0-0.17	1200	1750	1.50
		Mean	1.60	200+8			1183+150	1750+2	1.59+0.25
GF	As received	1	0.88	197	20-200	0-0.1	1230	1400	0.93
	As received	2	0.87	198	20-500	0-0.22	1550	1600	1.02
	As received	3	0.87	197	30-500	0-0.24	1250	1380	0.85
		Mean	0.87	197+3			1343+220	1460+250	0.93+0.15
GS	As received	1	2.180	201	30-200	0-0.15	250	430	8.1
	As received	2	2.200	203	5-200	0-0.13	300	480	8.0
	As received	3	2.180	199	10-200	0-0.12	280	450	6.0
		Mean	2.187	201+5			277+30	453+50	7.4+2

Table 3.3B. The mechanical properties as found from the tensile test without using the extensometer.

Specimen	Wires condition	Test No.	Ave. wire's diameter	E (GPa)	Measuring range of E		0.1%YS (MPa)
					Stress range (MPa)	Strain range (%)	
WF	As received	1	0.520	180	100-1050	0.04-0.5	2100
	As received	2	0.520	155	150-950	0.05-0.55	2350
	As received	3	0.515	170	100-1000	0-0.5	2200
		Mean	0.518	168±25			2217±250
WS	As received	1	1.60	185	10-500	0-0.25	1050
	As received	2	1.61	160	20-300	0-0.25	1200
	As received	3	1.60	175	30-450	0-0.15	1150
		Mean	1.60	173±25			1113±100

GF	As received	1	0.87	170	20-400	0-0.15	1350
	As received	2	0.88	180	20-500	0-0.20	1250
	As received	3	0.87	160	30-500	0-0.25	1550
		Mean	0.87	177±10			1383±250
GS	As received	1	2.185	150	30-200	0-0.20	350
	As received	2	2.190	175	35-200	0-0.15	315
	As received	3	2.180	170	30-200	0-0.15	320
		Mean	2.185	165±20			328±30

3.4.2. Flexure tests

3.4.2.1. Three point bending tests

Fig. 3.13 shows four representative stress-strain curves from the three point bending tests on as-received WF, WS, GF and GS wires.

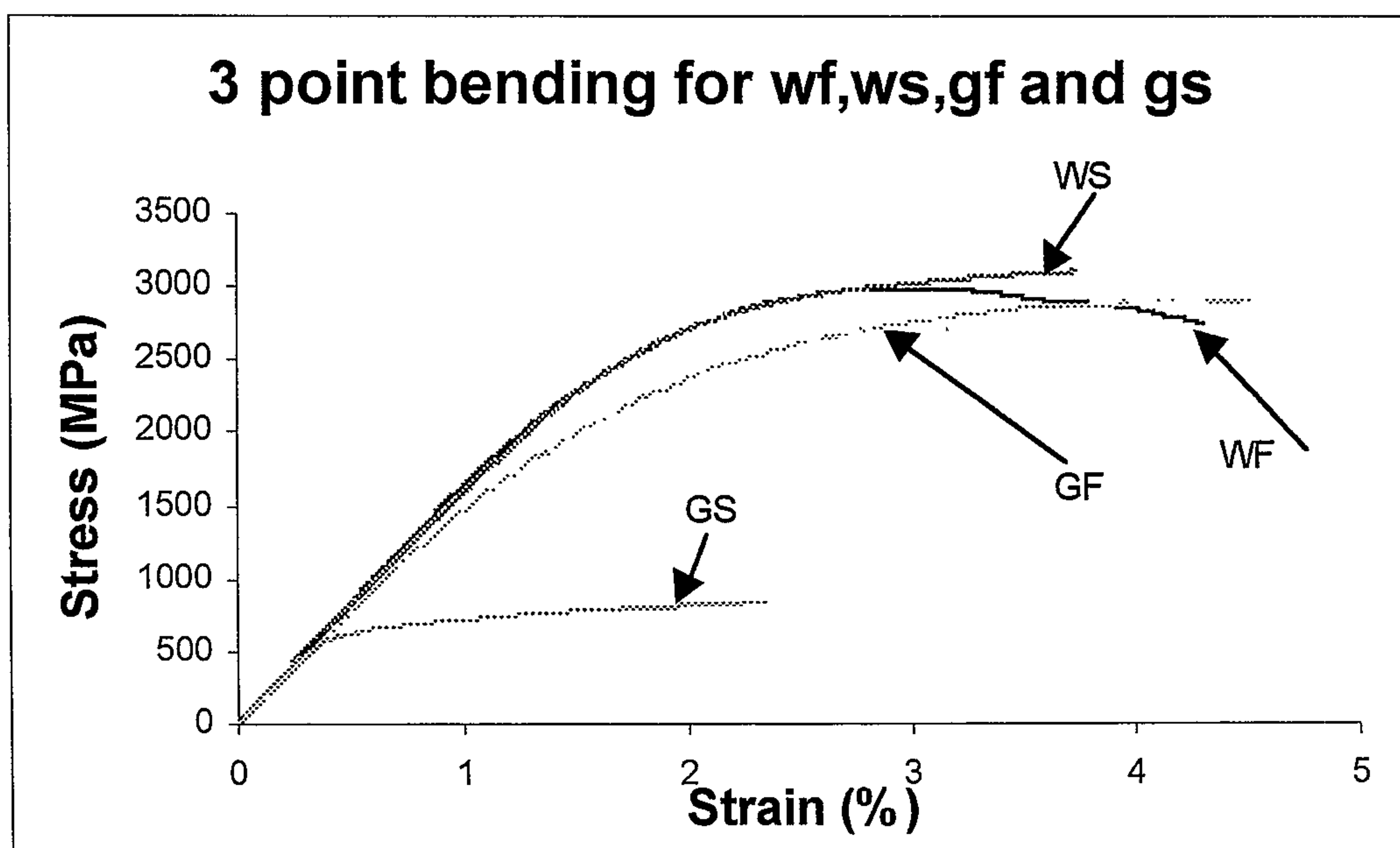


Fig. 3.13 Representative stress/strain curves of three point bending tests for the four wires.

From this figure and the summary of the 3 point flexure data in table 3.4 below, the following can be concluded:

- The mean value of E for all as-received wires is approximately 170GPa, which is 15% lower than the standard E values and the E value from the stainless steel standard value. The possible reasons for this will be discussed later in the discussion section of this chapter.

- The ordering of 0.1% YS for the four wires in three point bending tests is:

$$WF > WS > GF > GS$$

- The 0.1% YS in the three point bending is invariably higher than the corresponding values obtained in tension tests.

Table 3.4 The mechanical properties which resulted from the 3-point bending test.

Specimen	Test No.	Wires' Diameter. (mm)	Elastic modulus (Gpa)	Measuring range of E		0.1% YS (Mpa)
				Stress range (MPa)	Strain range (%)	
WF	1	0.52	164	10-2000	0-1.2	2800
	2	0.52	165	10-2000	0-1.2	2900
	3	0.52	171	0-2000	0-1.1	2700
	Mean	0.52	167±8			2800±200
WS	1	1.62	178	0-1500	0-0.86	2300
	2	1.61	162	0-1500	0-0.90	2250
	3	1.61	165	0-1500	0-0.90	2200
	Mean	1.61	168±15			2250±100
GF	1	0.87	161	25-1000	0-0.6	1850
	2	0.88	158	10-1000	0-0.6	1800
	3	0.87	152	5-1000	0-0.7	1750
	Mean	0.87	157±10			1800±100
GS	1	2.20	165	0-400	0-0.25	630
	2	2.18	162	0-400	0-0.24	650
	3	2.20	150	0-400	0-0.28	600
	Mean	2.19	159±15			626±50

3.4.2.2. Four point bending test .

Fig. 3.14 shows four representative stress-strain curves from the four point bending tests on as-received WF, WS, GF and GS wires.

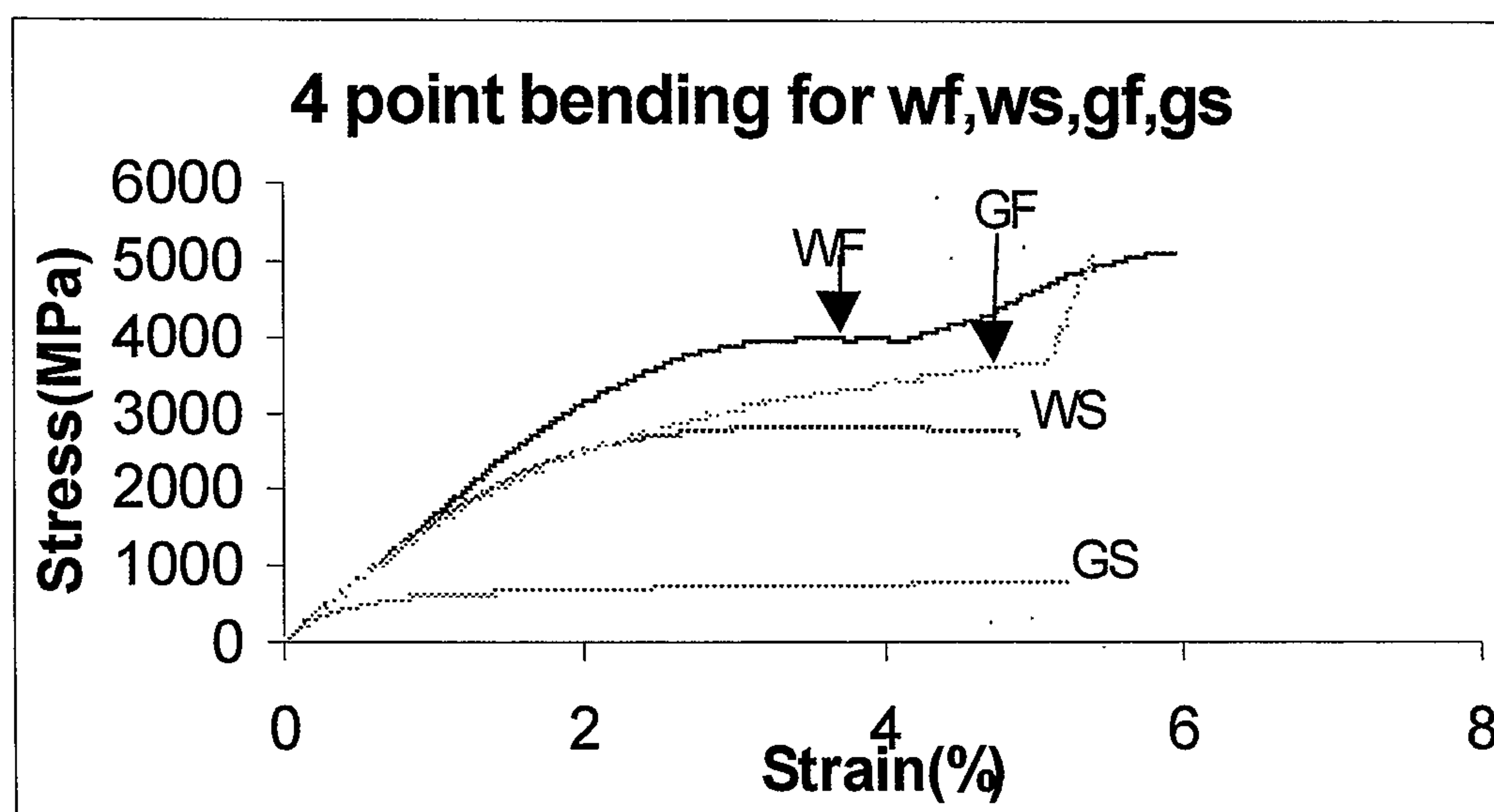


Fig. 3.14 Representative stress/strain curves of four point bending tests for the four wires.

From this figure and the summary of the 4 point flexure data in table 3.5, the following can be concluded:

- The mean values for E of all as-received wires are approximately 170GPa, which is similar to the values obtained from the three point bending tests but 15% lower than the standard E value and the E value from the tensile tests.
- The ordering of 0.1% YS for four wires in four point bending tests is:

$$WF>WS>GF>GS$$

- The 0.1% YS in four point bending tests is lower than the corresponding values obtained in three point bending tests but higher than the corresponding values obtained in tension tests.

The parameters of four point bending: 0.1% YS and E values for each wire in each test are listed in the tables 3.5

Table 3.5 The mechanical properties which resulted from the 4-point bending test.

Specimen	Test No.	Diameter of the wires (mm)	Elastic modulus (Gpa)	Measuring range of E		0.1% YS (Mpa)
				Stress range (MPa)	Strain range (%)	
WF	1	0.52	170	10-1000	0-0.57	2300
	2	0.51	163	20-1000	0-0.57	2350
	3	0.52	165	10-1000	0-0.60	2250
	Mean	0.52	166±10			2300±100
WS	1	1.62	173	10-1100	0-0.66	1950
	2	1.60	167	5-1000	0-0.60	1800
	3	1.61	173	5-1000	0-0.60	1900
	Mean	1.61	171±8			1883±150
GF	1	0.88	158	0-1000	0-0.60	1600
	2	0.87	157	0-1000	0-0.59	1700
	3	0.88	156	10-1000	0-0.61	1700
	Mean	0.88	157±3			1683±100
GS	1	2.18	163	10-200	0-0.12	420
	2	2.19	161	20-200	0-0.10	390
	3	2.20	163	50-200	0-0.18	430
	Mean	2.19	162±5			413±40

3.4.3. Scanning electron microscope examination.

3.4.3.1. Scanning electron microscope examination of tensile fracture part.

SEM micrographs provide information about the macro-mechanism of the wire fracture. The micrographs of the fracture surface of WF, WS and GF wires are shown in fig. 3.15(a) & 3.15(b), fig. 3.16(a) & 3.16(b) and fig. 3.17(a) & 3.17(b), respectively.

These pictures clearly demonstrate that a “cup & cone” fracture model is relevant in these wires. The micrographs of the fracture part of the GS wire are shown in fig. 3.18(a) & 3.18(b), in which a semi cup and cone fracture mode can be observed.

Higher magnification images of the fracture surfaces of WF, WS, GF and GS wires are shown in figures 3.19; 3.20; 3.21, and 3.22, respectively. These figures show three regions:

- (a) the centre fibrous region; clearly showing tearing dimples and equiaxed dimples;
- (b) the second region which is the shear fracture region; and
- (c) the outer surface region of the wires.

These three regions are shown in separate higher magnification photos beside each of the main pictures.

The micromechanisms of the wires fractures can be confirmed from the above figures as will be discussed later in this chapter.

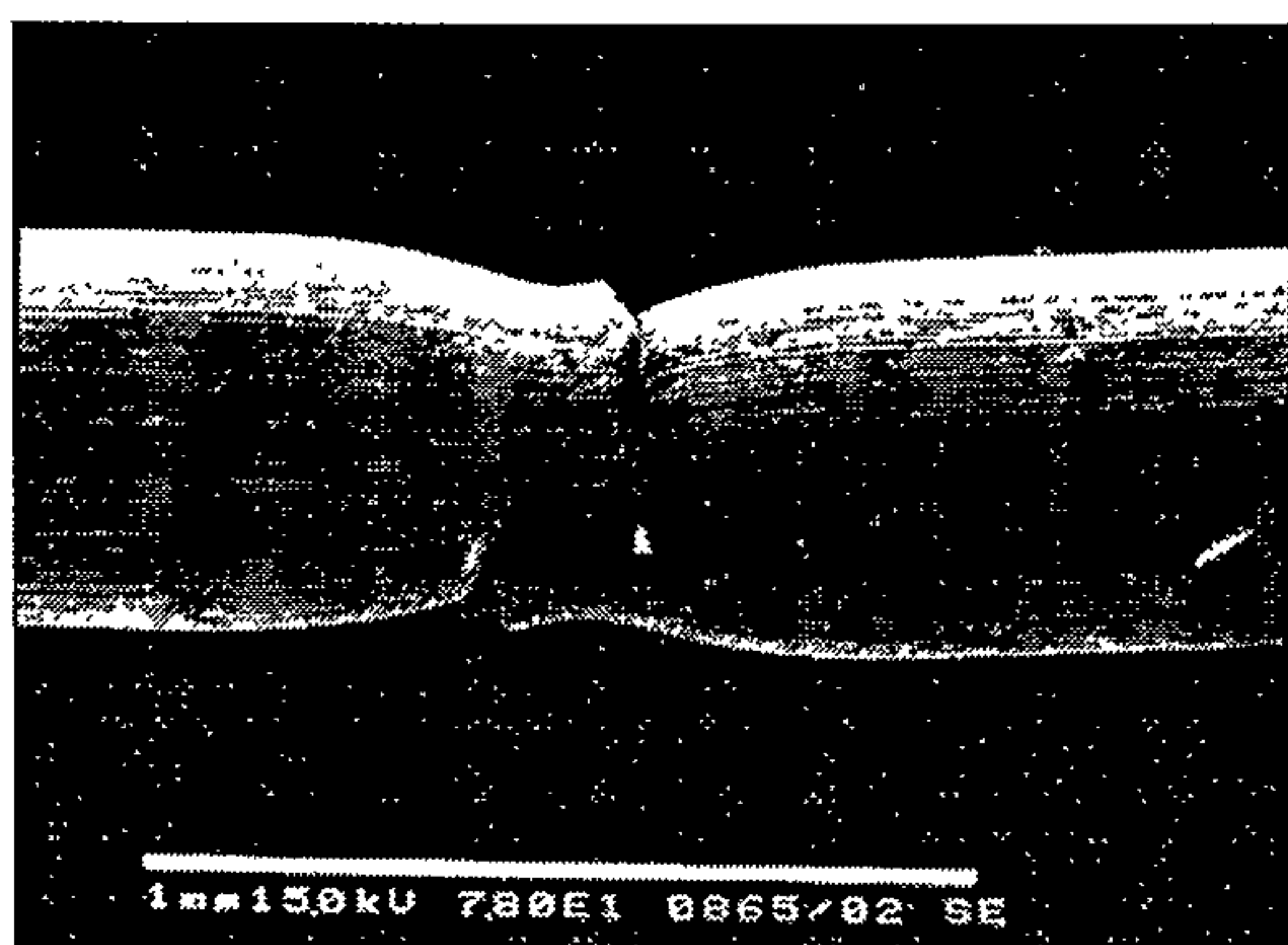


Fig. 3.15(a). Tensile fracture part of WF wire.

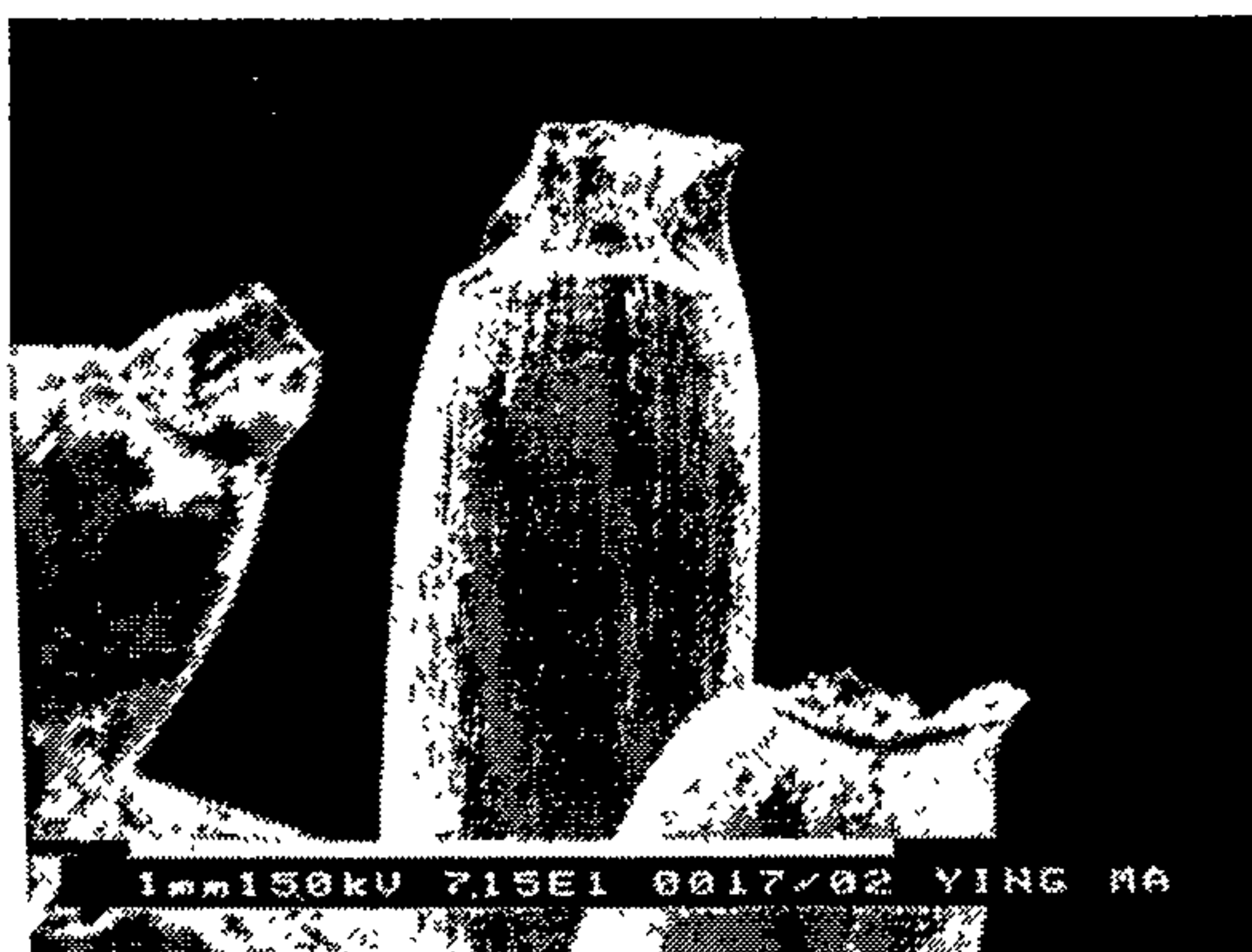


Fig. 3.15(b). Tensile fracture part of WF wire.

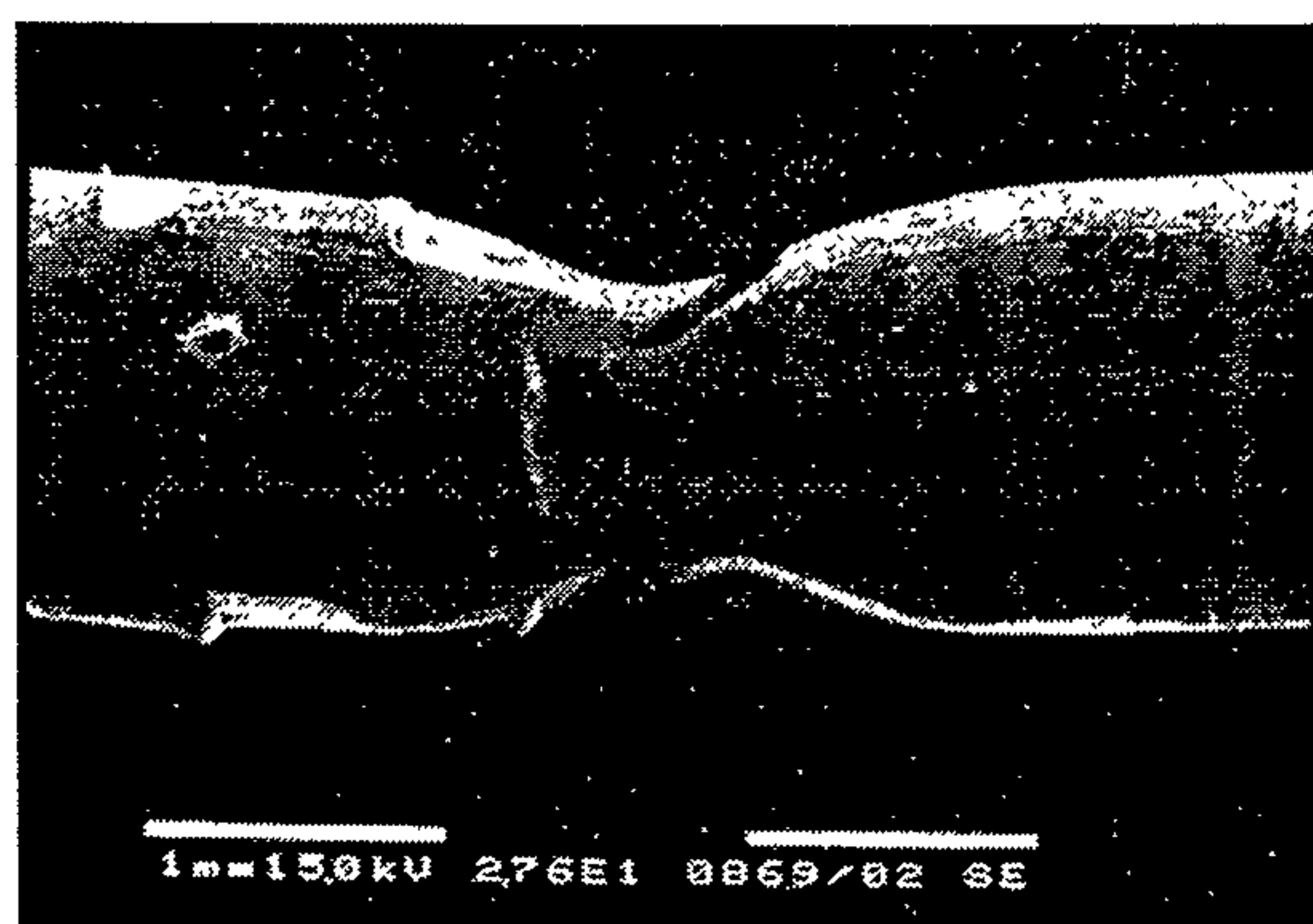


Fig. 3.16(a). Tensile fracture part of WS wire.



Fig. 3.16(b). Tensile Fracture part of WS wire.



Fig.3.17(a). Tensile fracture part of GF wire.



Fig.3.17(b). Tensile fracture part of GF wire.

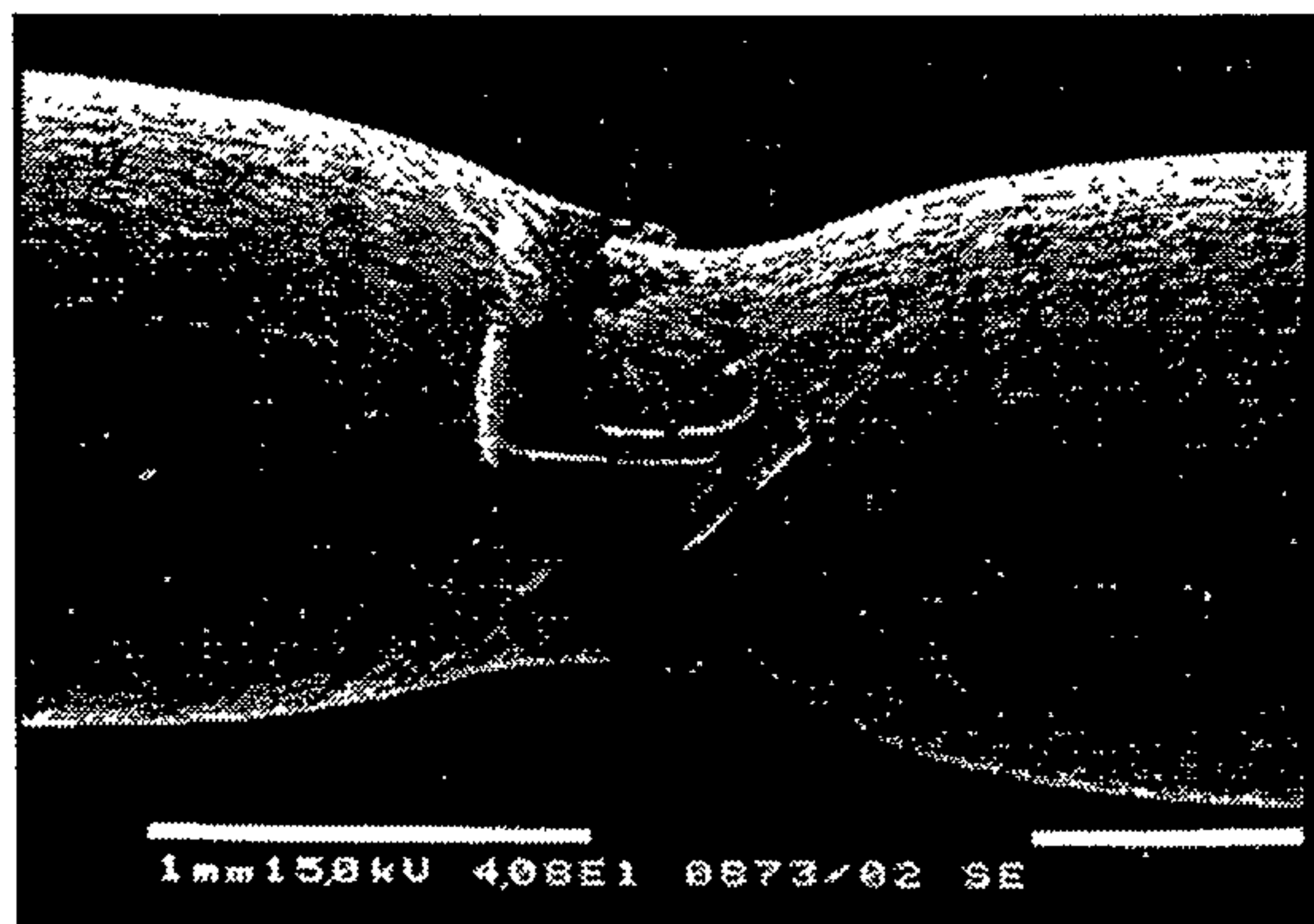


Fig. 3.18(a). Tensile fracture part of GS wire.

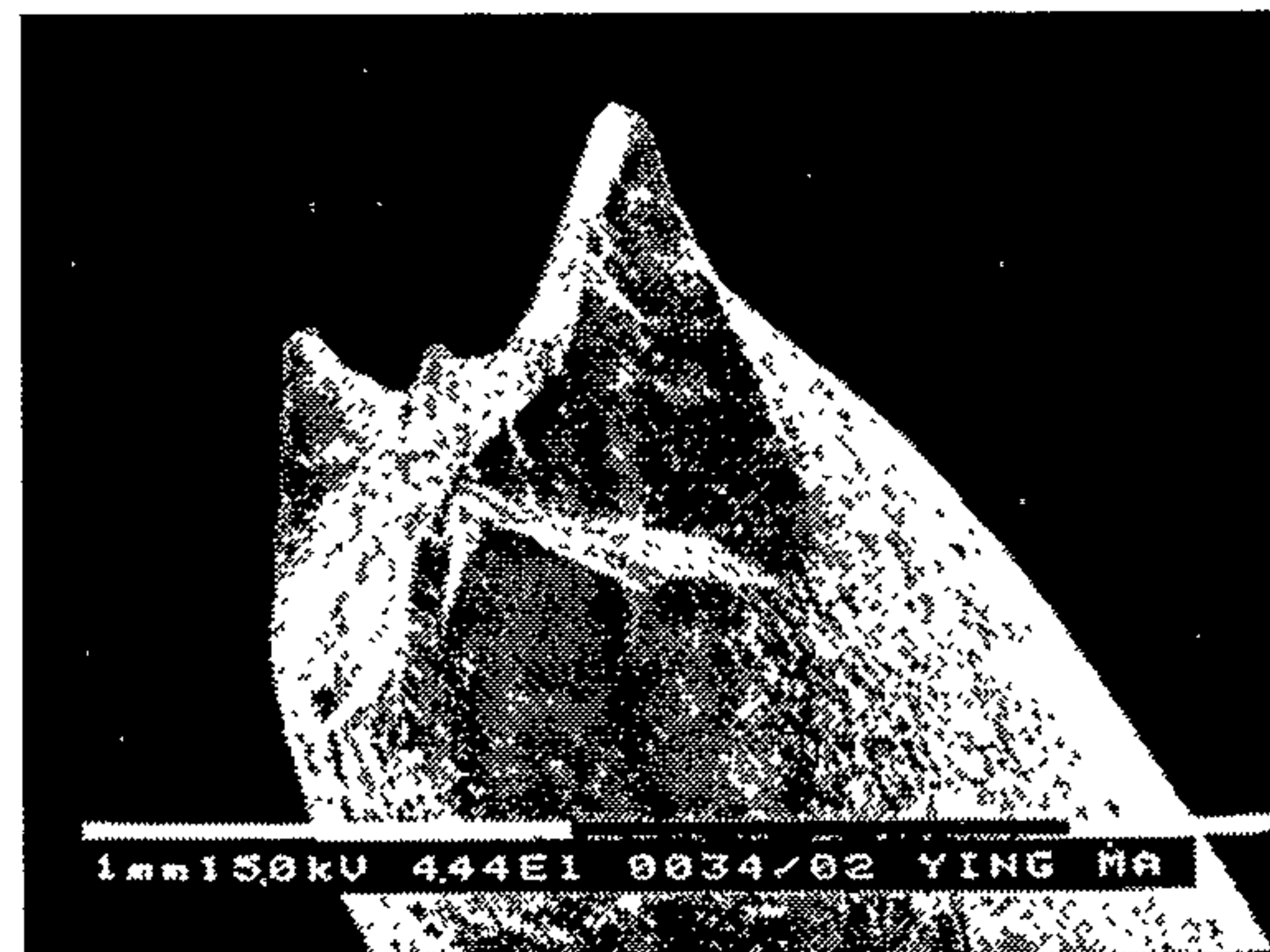


Fig. 3.18(b). Tensile fracture part of GS wire.

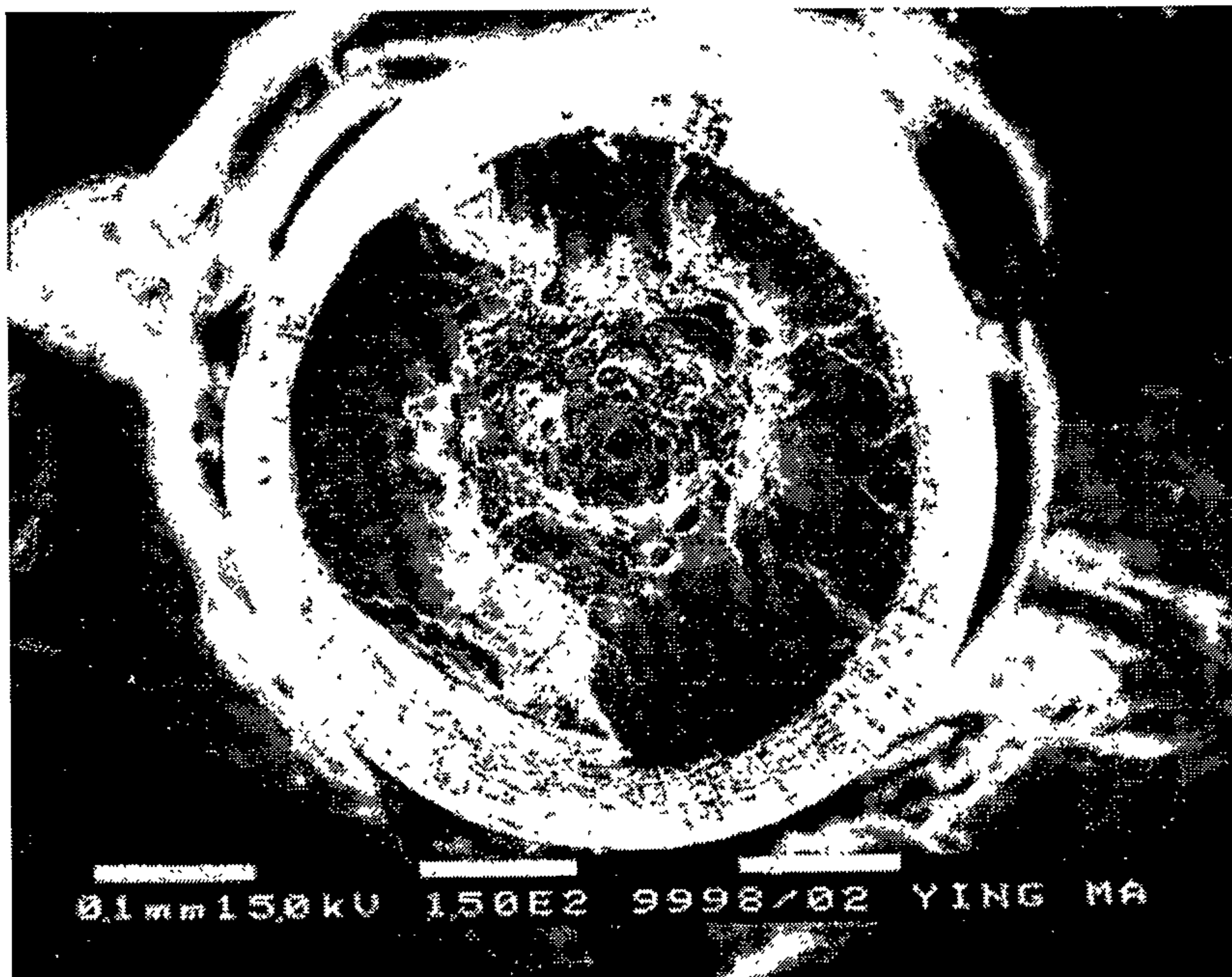


Fig. 3.19 Cross-section of tensile fracture part of WF wire.

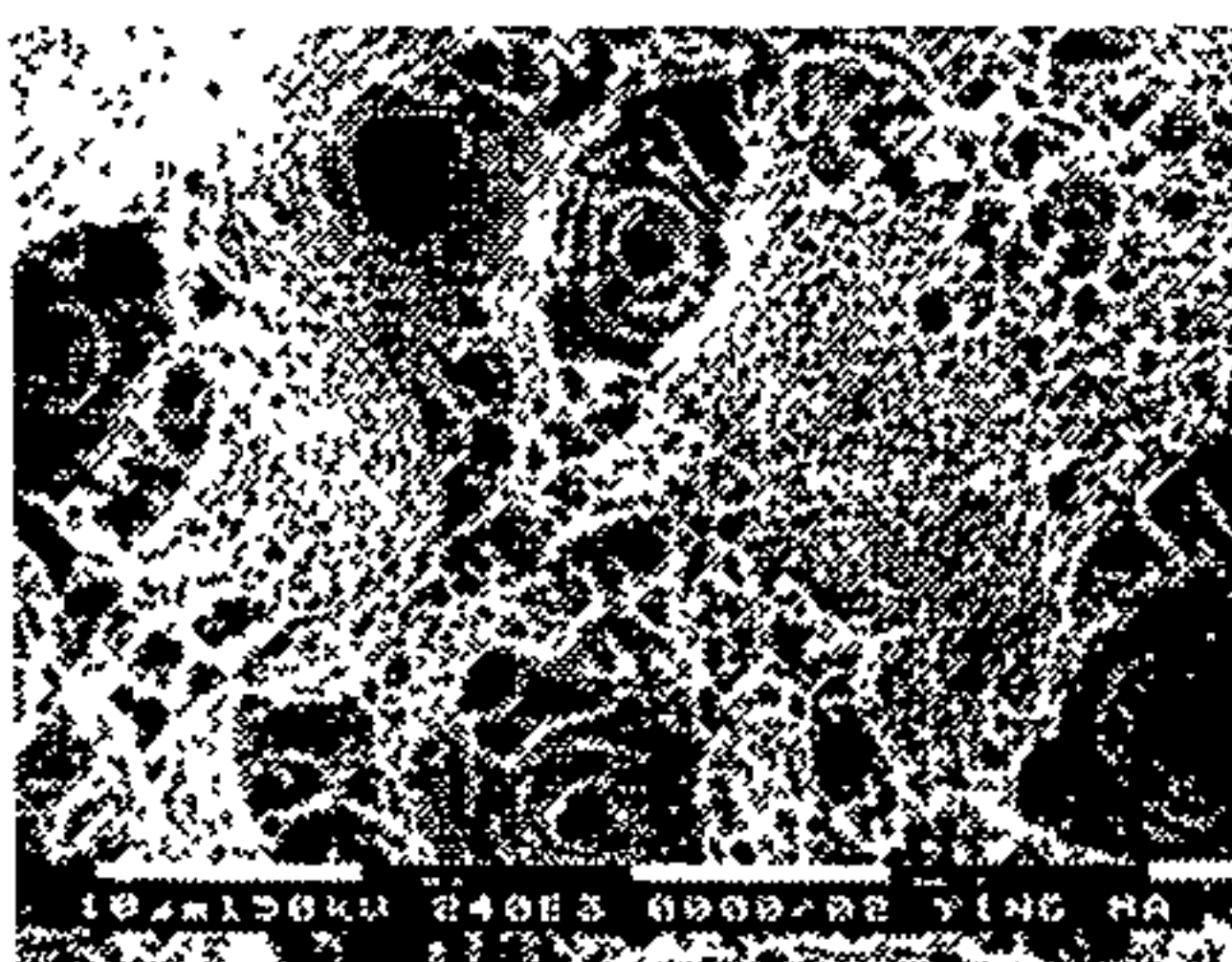


Fig 3.19 (a)
Center region.

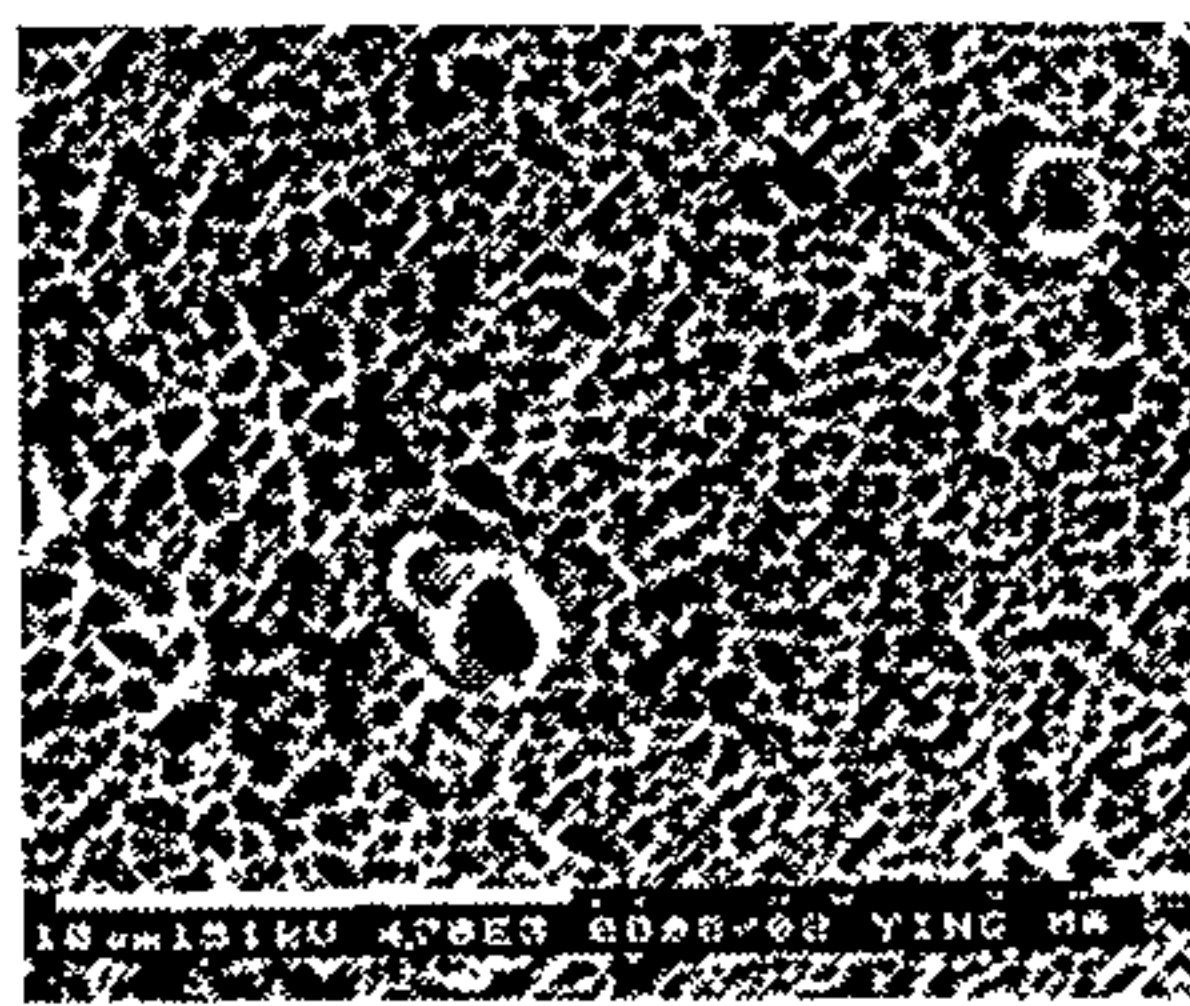


Fig. 3.19 (b)
Second region.

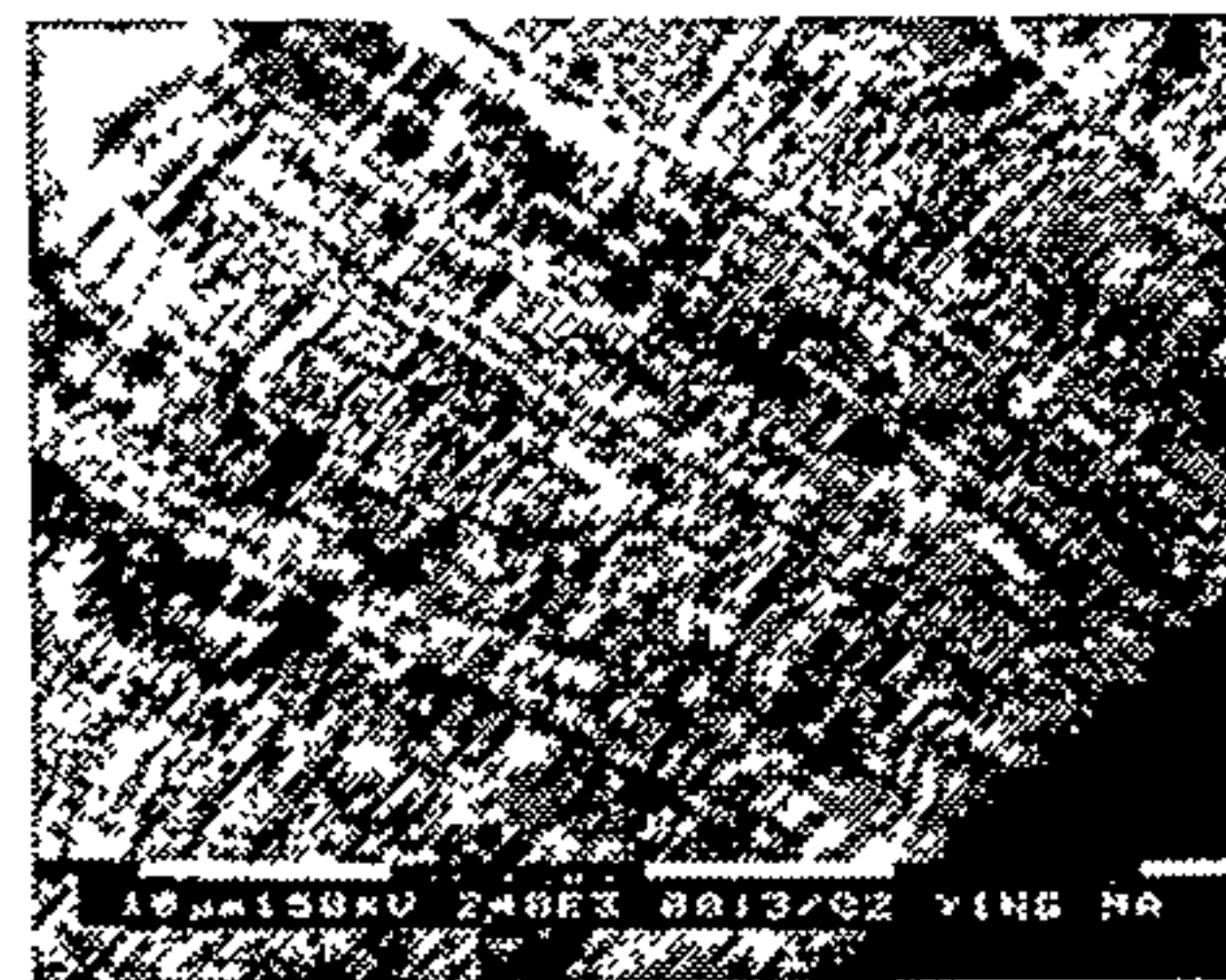


Fig 3.19 (c)
Outer region.



Fig. 3.20 Cross-section of tensile fracture part of WS wire.

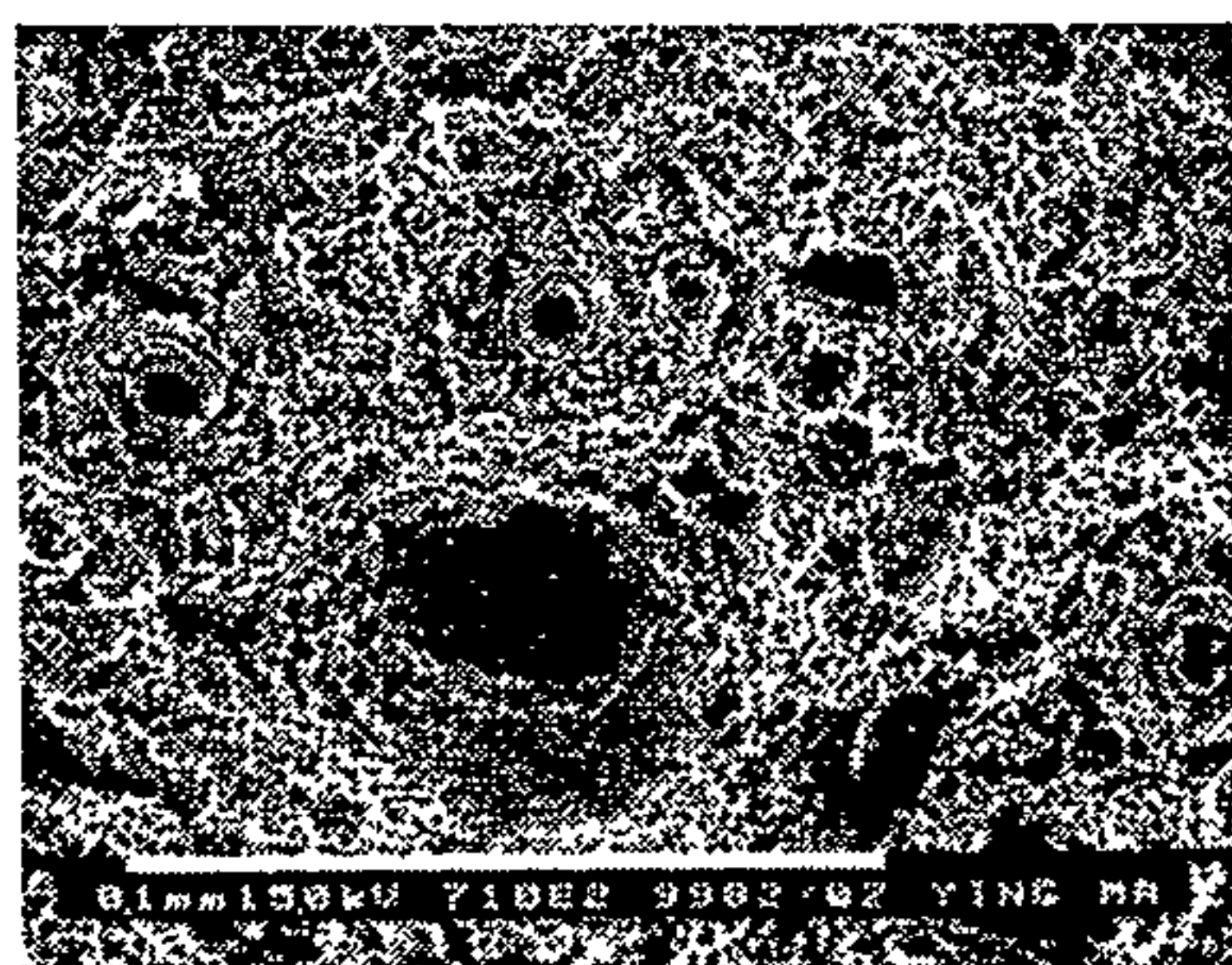


Fig 3.20(a)
Center region.



Fig. 3.20(b)
Second region.



Fig. 3.20(c)
Outer region.

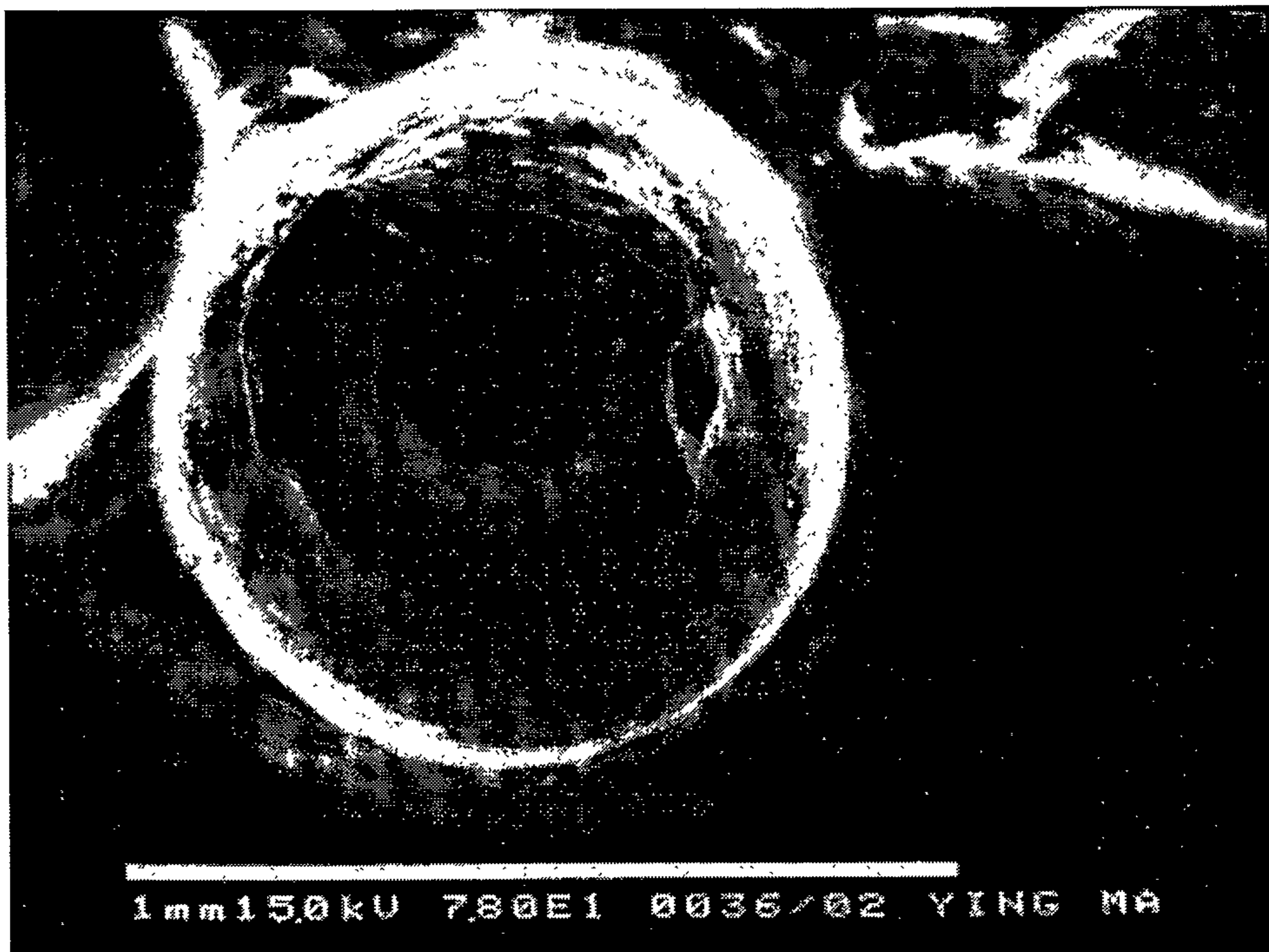


Fig. 3.21. Cross-section of tensile fracture part of GF wire.

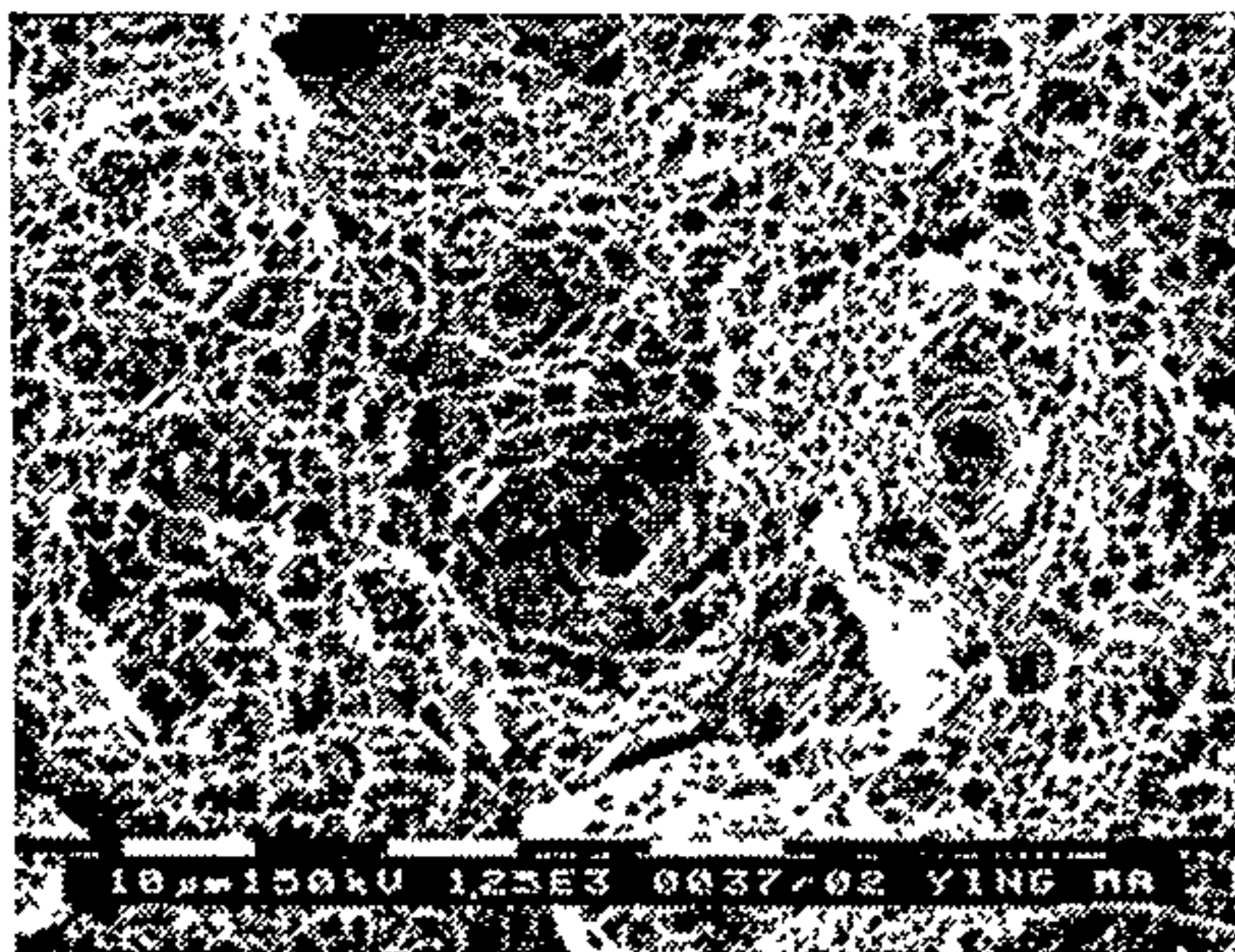


Fig. 3.21(a)
Center region.

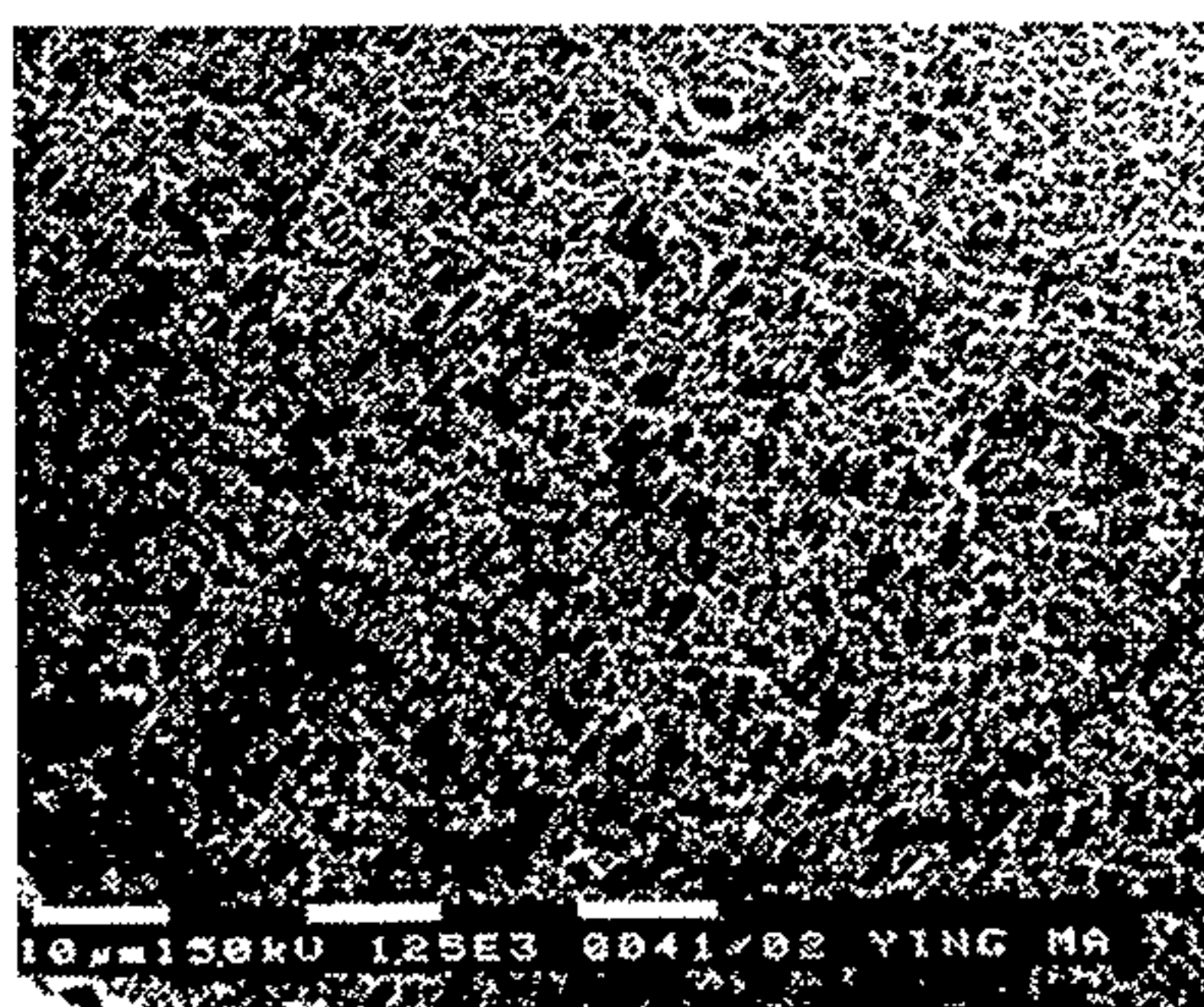


Fig. 3.21(b)
Second region.



Fig. 3.21(c)
Outer region.

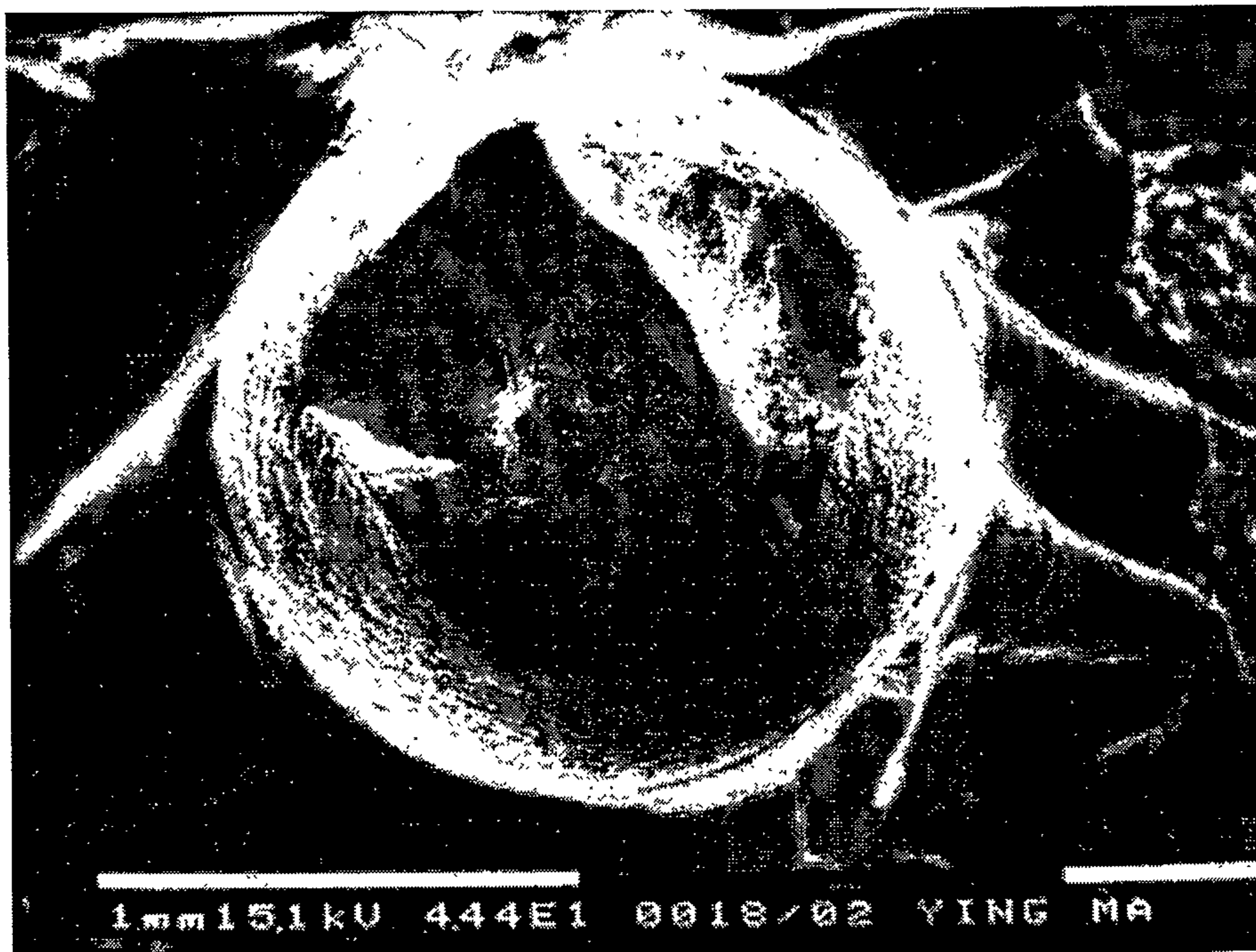


Fig.3.22. Cross-section of tensile fracture part of GS wire.

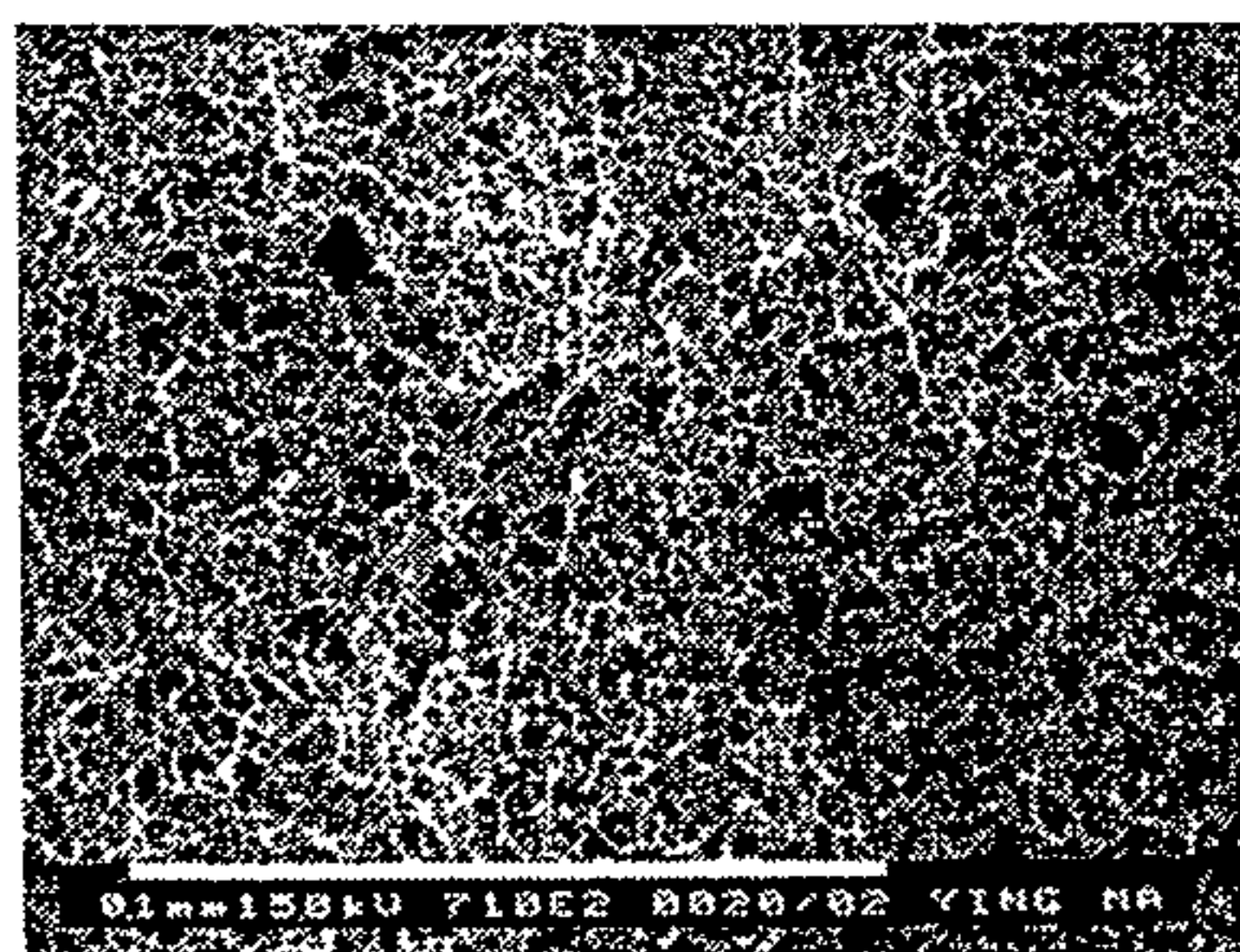


Fig.3.22 (a)
Center region.

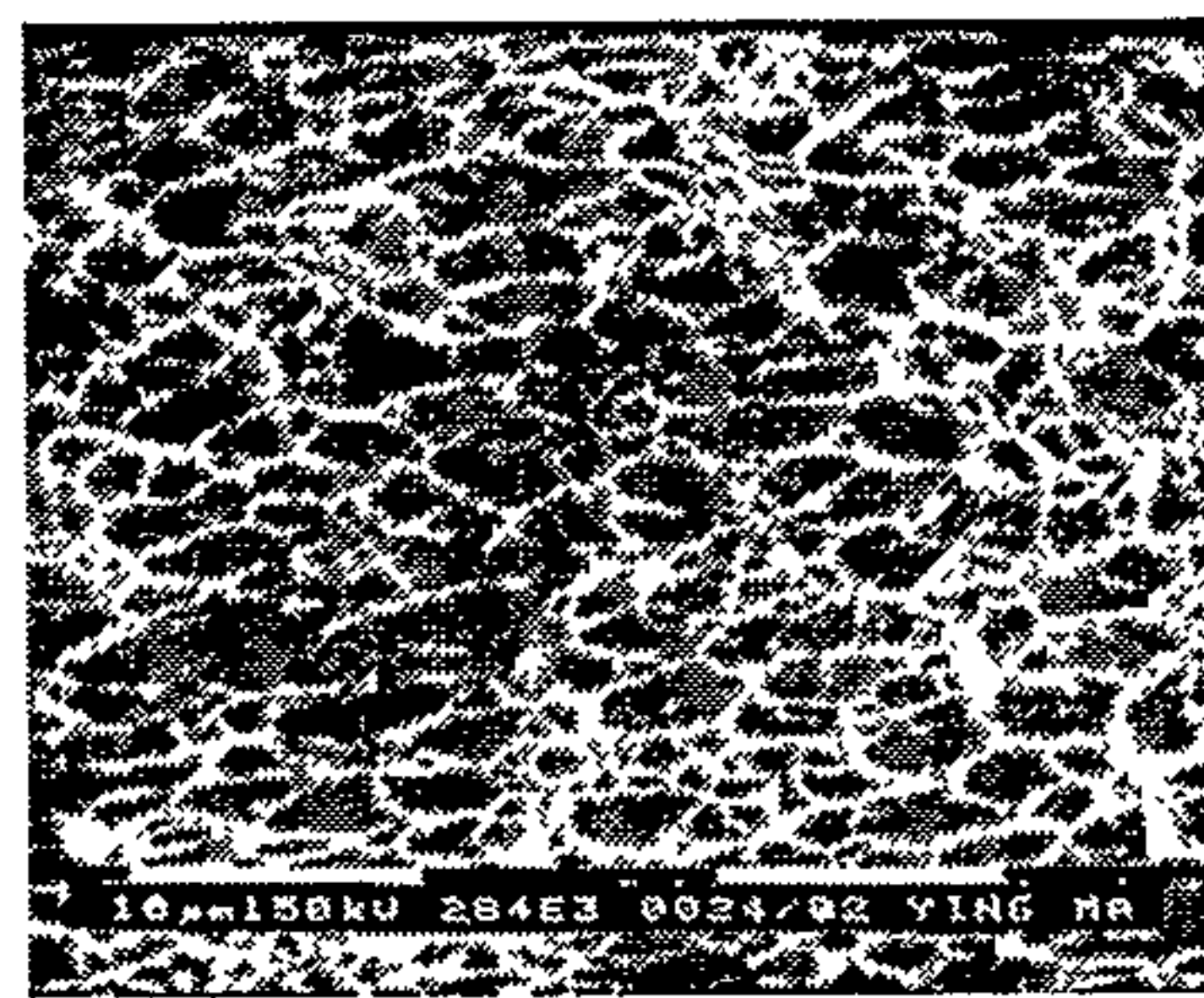


Fig.3.22(b)
Second region.

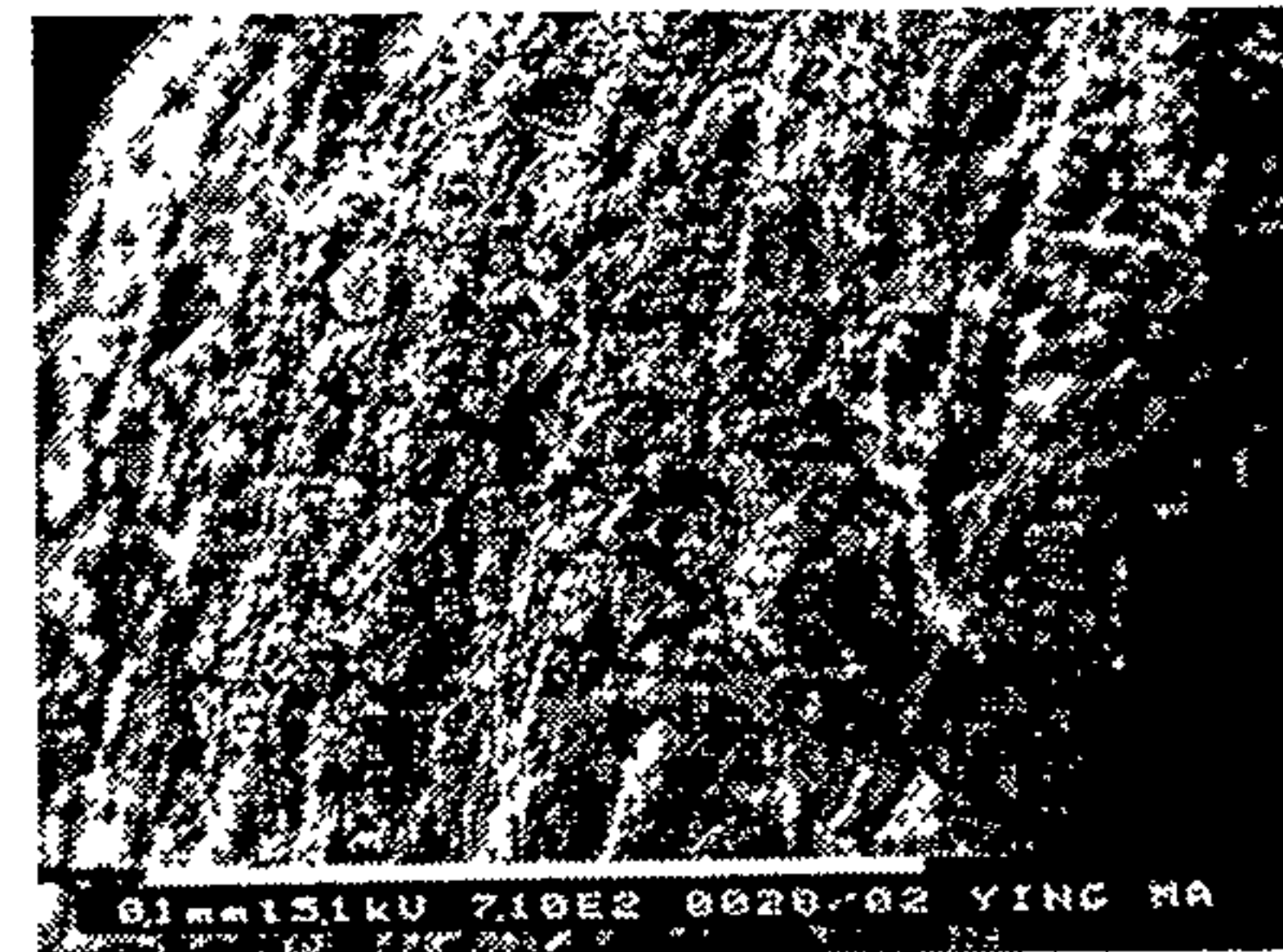


Fig.3.22(c)
Outer region.