THE MECHANICAL PROPERTIES AND MICROSTRUCTURE OF ORTHODONTIC WIRES

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BY

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SUMMARY

Tensile tests were carried out on alpha-titanium (straightened) and alpha-titanium combination orthodontic wires. 0.2% proof stress, yield strength and the tensile strength were found to be close to the standard values for alpha-titanium. However, the modulus of elasticity of the wire was about 20-30% lower than the standard value for alpha-titanium. Repeated tests showed the same results. It was felt that this decrease in the value of the modulus of elasticity could be due to factors ranging from the manufacturing process (cold working of the wires), the presence of impurities and also due to the fact that most of the standard norms for alpha-titanium were tested on plates, sheets and bigger diameter wires. The range of readings obtained were somewhat similar to that recorded by **Hazel, Sokel and West (1984)**.

Scanning electron microscopy (SEM) was carried out to determine the microstructure of a number of orthodontic wires. A stainless steel wire which was found to be extremely brittle was given for testing by **A.J. Wilcock (Whittlesea, Australia)** to determine the reason for its brittleness. A comparison was made between the microstructure of this stainless steel test wire with other stainless steel orthodontic wires, namely the 0.5085mm (0.20") special plus **(A.J Wilcock, Australia)** and 0.4060mm (0.016") premium plus **(A.J. Wilcock, Australia)**. The test wire was found to have numerous longitudinal striations close to the surface compared to the controls which had fewer and more evenly spaced striations. The transverse section of the brittle test wire showed two distinct types of contrast. The "pitted" parts of the wire were more readily etched compared to the rest of the matrix. The controls had far fewer pits and appeared to be more resistant to the etchant.

The microstructure of alpha-titanium, beta-titanium (TMA), Chinese NiTi, and superelastic NiTi orthodontic wires was also examined using the scanning

electron microscope. The alpha-titanium wire appeared two-phase with the presence of elongated alpha grains in an unresolved matrix. Beta-titanium was difficult to etch and showed raised cone formation in the transverse section. An X-ray microanalysis, using an electron dispersive spectrometer (EDS), of these raised cones showed a higher amount of titanium as compared to the matrix. Titanium has a great affinity for oxygen. It is believed that the cones probably consisted of titanium dioxide. Since oxygen has an atomic number of less than 11 it was not possible to detect its presence with the EDS system used in this study. The Chinese NiTi and superelastic NiTi wires showed the presence of precipitates within pits on the etched surface. These precipitates were analysed and again found to be rich in titanium, thus also suggesting that the precipitates could be titanium dioxide.

Alpha-titanium wires have been reported to become brittle after being placed in the oral cavity for more than six weeks. It has been suggested that this is due to the formation of trihydrides or due to the vanadium content **(Mollenhauer, 1989)**. Transmission electron microscopy (TEM) studies of used (exposed to the oral environment) alpha-titanium wires did not show the presence of any precipitates. It seems unlikely that oxides or hydride formation can explain the proposed changes in the mechanical properties of the alpha-titanium wire.

TEM studies of stainless steel, alpha- and beta-titanium wires exhibited microstructures characteristic of metals that have been heavily cold worked. Heavily dislocated, elongated grains were seen in the longitudinal sections with more equiaxed grains in the transverse sections. Stainless steel and beta-titanium alloys exhibited fine grain structures with cross-sections of approximately 30nm and 80nm respectively. The used and unused alpha-titanium wires showed comparatively much larger structures which are more typical of an annealed condition. The alpha-titanium grains were 0.5-2.0μm in diameter.

SIGNED STATEMENT

This report contains no material which has been accepted for the award of any other degree or diploma in any university. To the best of my knowledge and belief, it contains no material previously published or written by another person, except where due reference is made in the text of the report.

(JAGJIT SINGH)

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CHAPTER 1

INTRODUCTION

Recent advances in orthodontic wire alloys have resulted in a varied array of wires. The development of alpha-titanium, beta-titanium (TMA), nickel-titanium (Nitinol), superelastic nickel-titanium and the different stainless steel wires has made selecting the best archwire for a particular application complex. At present, a plethora of archwires of different sizes and alloys from different manufacturers are available **(Burstone and Goldberg, 1980; Kapila and Sachdeva, 1989)**.

Several studies have been conducted concerning mechanical properties of archwires such as stress relaxation, working range or permanent deformation **(Andreasen and Barret, 1970 ; Andreasen and Morrow, 1978; Harris, Newman and Nicholson, 1988; Jones, Staniford and Chan, 1990; Muira, Mogi and Okamoto, 1990)**. However, data concerning the microstructure and microanalysis of the chemical composition of archwires are lacking.

Many factors affect the structure and properties of archwires. **Bee (1991)** states that "the properties and performance of materials are largely determined by their structures. These structures are in turn determined by chemical composition, heat treatment and processing conditions. In order to understand how materials behave, to develop new or improved materials, we have to study and characterize their structure and composition in as much detail as possible."

Apart from a preliminary study by **Hazel, Sokel and West (1984)**, very little is known of the mechanical properties and microstructure of the relatively new alpha-titanium orthodontic wire. It has been observed by **Mollenhauer (1989)** that six weeks after the insertion of the alpha-titanium wire in the oral cavity, they tend to become brittle to bend. This phenomenon is thought to be due to the formation of trihydrides or due to the vanadium content but there has been no study undertaken to confirm this.

The aims of the present study were to

(1) Determine the mechanical properties of alpha-titanium orthodontic wires.

(2) Make a preliminary comparison of the microstructure of a number of orthodontic wires using scanning electron microscope.

(3) Obtain X-ray analyses, using energy dispersive spectrometer (EDS), of the orthodontic wires.

(4) Determine a suitable technique for the preparation of thin foils (for transmission electron microscopy) from orthodontic wires in both longitudinal and transverse sections.

(5) Carry out preliminary microstructural studies on stainless steel, alpha- and beta-titanium orthodontic wires using transmission electron microscope.

(6) Prepare thin foils of alpha-titanium wire that had been used as a finishing wire in the oral cavity for at least 2 months for transmission electron microscopy (TEM) to determine the cause of brittleness of this wire when exposed to the oral environment.

CHAPTER 2

LITERATURE REVIEW

Historically, relatively few metallic alloys have been used in the fabrication of orthodontic appliances. Although at one time gold was widely used for arch wires, in recent years austenitic stainless steel has been the mainstay of orthodontic wires. It has maintained its popularity because of a good balance of environmental stability, stiffness, resilience, formability and cost. Chromium-cobalt wires have also been used in appliance therapy. Although the mechanical properties of Elgiloy (chromium-cobalt wire) and stainless steel are similar, the former can be given a strengthening heat treatment which allows manipulation of the wire in a softened state. This can be followed by a hardening heat treatment to obtain the desired resilience **(Burstone and Goldberg, 1980).**

Although the properties required in an orthodontic wire will vary, depending upon its application, generally three characteristics are important for a superior wire. First, it should be possible for the wire to be deflected over long distances without permanent deformation; hence, a large springback. This ensures that the clinician can activate his appliances without permanent deformation, which assures better control over tooth movement and minimizes intervals for adjustment. Second, the wire should have a stiffness that is lower than that of stainless steel, which would allow wires to fill the bracket for control and at the same time produce light forces. Third, the wire should be highly formable, that is, capable of being easily shaped, bent, and formed into complicated configurations, such as loops, without fracture **(Barrowes, 1982; Kapila and Sachdeva, 1989; Mohlin, Muller, Odman, and Thilander, 1991).**

Up until the 1930's, the only orthodontic wires available were made of gold. Austenitic stainless steel was introduced as an orthodontic wire in 1929, and shortly afterward gained popularity over gold. Since then several other alloys with desirable properties have been adopted in orthodontics. These include multi-stranded stainless steel, cobalt-chromium, alpha-titanium, beta-titanium, and nickel-titanium wires **(Jones, Staniford, and Chan, 1990; Mohlin, Muller, Odman, and Thilander, 1991)**.

Presently the orthodontist may select, from all the available wire types, one that best meets the demands of a particular clinical situation. The selection of an appropriate wire size and alloy type in turn would provide the benefit of optimum and predictable treatment results or tooth movements.

2.1 METALS AND NONMETALS

Approximately three-quarters of all the elements are considered to be metals. Some of these are metalloids such as silicon or germanium. Some of the properties that an element must have to be considered a metal are:

(1) Ability to donate electrons and form a positive ion.
(2) Crystalline structure - grain structure.
(3) High thermal and electrical conductivity.
(4) Ability to be deformed plastically.
(5) Metallic lustre or reflectivity.

Metalloids, such as silicon, possess one or more of these properties, but they are not true metals unless they have all of the characteristics of metal (**Neely, 1989**).

2.1.1 Crystalline Unit Structures

When metals solidify from the liquid to the solid state, the atoms align themselves in orderly arrays, which is an arrangement that is peculiar to that metal. This arrangement is called a space lattice, which can be represented by a series of points in space. By means of X-ray diffraction, the space lattice of the crystal has been determined for different metals.

Metals solidify into three main lattice structures.

(1) Body-centred cubic (BCC).

(2) Face-centred cubic (FCC).

(3) Hexagonal close-packed (HCP).

As solidification is taking place, the arrangement of the crystalline lattice structure takes on its characteristic pattern. Each unit cell builds on another to form crystalline needle patterns called dendrites.

The crystalline lattice structures begin to grow first by the formation of seed crystals or nuclei as the metal solidifies. The number of nuclei or grain starts formed determines the fineness or coarseness of the metal grain structure. Slow cooling promotes large grains and fast cooling promotes smaller grains. The grain grows outward to form the dendrite crystal until it meets another dendrite crystal that is also growing. The places where these grains meet are called grain boundaries. As the crystal structures grow in different directions, the atoms at the grain boundaries are jammed together in a misfit pattern. Although the nature of the grain boundaries is not entirely understood, it is generally assumed to be an interlocking border in a highly strained condition **(Neeley, 1989)**.

2.1.2 Elasticity and Plasticity

A typical stress-strain graph for a metal such as copper or soft iron is shown in **Fig. 1**. The stress in this case is a simple tensile stress, and the strain is shown as the percent elongation. The first portion of the curve, up to a strain of less than 1%, is a straight line, indicating Hooke's Law behavior with stress directly proportional to strain. This straight-line portion ends at point ***a*** ; the stress at this point is called the ***proportional limit.***

From ***a*** to ***b***, stress and strain are no longer proportional, but if the load is removed at any point between ***O*** and ***b***, the curve is retraced and the material returns to its original length. In the entire region ***Ob***, the material is said to be elastic or to show elastic behaviour. Point ***b***, the end of this region, is called the ***yield point***, and the corresponding stress is called the ***elastic limit***. Up to this point the forces exerted by the material are conservative. When the load is removed, the material returns to its original shape, and the energy put into the material in causing the deformation is recovered. The deformation is said to be reversible (**Giancoli, 1988 ; Sears, Zemansky, and Young, 1987**).

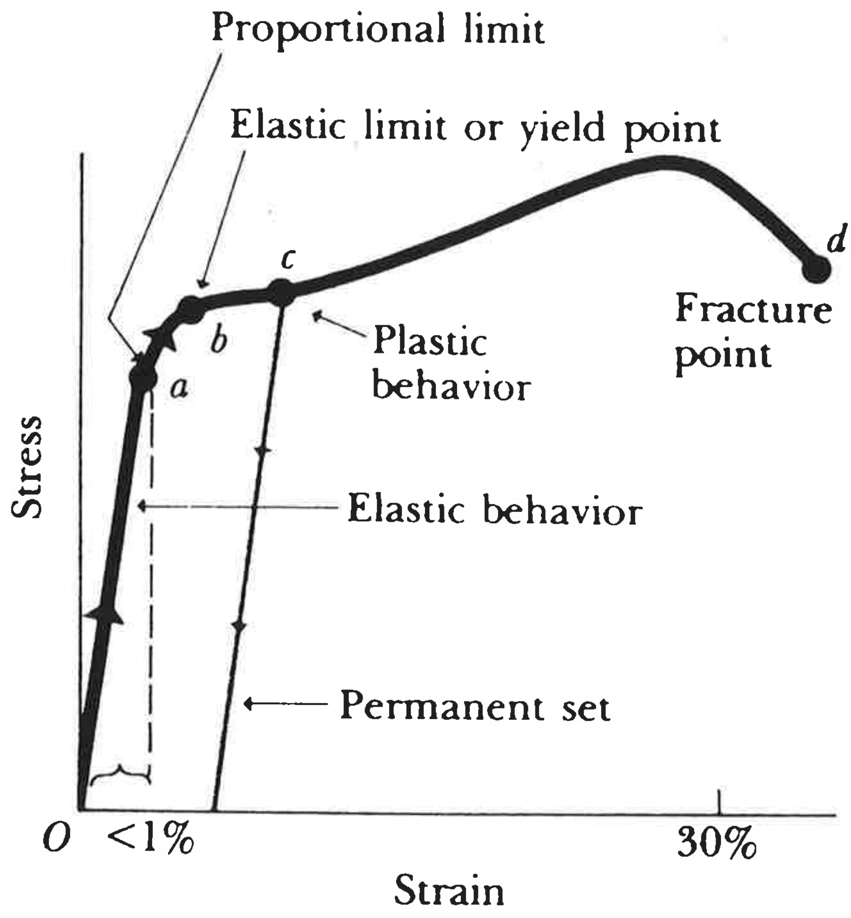


Fig. 1. Typical stress-strain diagram for a ductile metal under tension. (Taken from **Sears, F.W., Zemansky, M.W., and Young, H.D. (1987). University Physics. 7th Edition**).

If a slight increase in loading is applied to the elastic limit, the material will deform for the first time without any increase in loading. ***Yield strength*** is the value of stress on the curve where elongation takes place without the need of applying any additional load. The slope of the curve is horizontal just beyond the yield strength point. If the stress is increased further, the strain increases rapidly, but when the load is removed at some point beyond ***b***, say ***c***, the material does not come back to its original length but traverses the thin line in **Fig. 1**. The length at zero stress is now greater than the original length, and the material is said to have a ***permanent set***. Further increase of load beyond ***c*** produces a large increase in strain (even if the stress decreases) until a point ***d*** is reached at which fracture takes place. From ***b*** to ***d***, the material is said to undergo ***plastic flow***, or ***plastic deformation***. Plastic deformation is irreversible. If a large amount of plastic deformation takes place between the elastic limit and the fracture point, the metal is said to be ductile; but if fracture occurs soon after the elastic limit is passed, the metal is said to be brittle **(Sears, Zemansky, and Young, 1987)**.

As the specimen under tension is strained beyond its yield point, the stress increases to a maximum termed the ultimate tensile strength. The stress required to cause actual fracture of a material is called the ***breaking stress***, or the ***fracture strength***. To provide a definite stress that can be measured on different testing instruments, the property of proof stress is used. The proof stress of a material is the stress required to produce a certain permanent strain. Starting with a certain strain, a line parallel to the straight line portion of the curve ***Oa*** is drawn to its intersection with the curve. The stress corresponding to this point is called the ***proof stress***. The strain at which the proof stress is measured is usually 0.1% or 0.2% strain.

Tensile stress is tensile force per unit area, F/A. Tensile strain is fractional change in length, dl/l_o. Young's modulus Y is the ratio of tensile stress to tensile strain **(Smallman, 1985)**.

$$Y = \frac{F/A}{dl/l_o}$$

The modulus of elasticity value depends on what the material is made of more than how strong or hard it is. For example, the modulus of elasticity of a steel is not changed by annealing or quench hardening.

2.2 WIRE CHARACTERISTICS OF CLINICAL RELEVANCE

Several physical and mechanical properties of orthodontic wires are considered desirable for optimum performance during treatment. These include a large springback, low stiffness, high formability, high stored energy, biocompatibility and environmental stability, low surface friction, and the capability to be welded or soldered to auxiliaries and attachments. Most properties can be assessed simply by the feel the wire has in the hands;-

(a) Flexing the wire in the fingers without taking a set is a quick measure of its flexibility,

(b) Forming an arch with the thumb gives some indication of the force needed to deform the material, that is, its stiffness,

(c) Measuring how much spring is in the arch by deflecting between the thumb and index finger is a measure of resiliency **(Wilcock, 1989).**

2.2.1 Springback

This is also described in the literature as the maximum flexibility, the range of activation, the range of deflection, the working range or the maximum elastic deflection. The springback of a wire is the ratio of the yield strength to the modulus of elasticity. Higher springback values allow increased activation, which is always desirable, unless other properties such as formability of the wire are being sacrificed excessively **(Burstone and Goldberg, 1980)**. This ensures that the clinician can activate his appliance without permanent deformation, which assures better control over tooth movement and minimizes intervals for adjustment.

2.2.2 Stiffness or Load Deflection Rate

The stiffness or load deflection rate of an orthodontic wire is determined by its configuration, the cross-sectional size and shape of the wire, and the modulus of elasticity of the wire **(Goldberg, Morton, and Burstone, 1983)**. This is the force magnitude delivered by an appliance and is proportional to the modulus of elasticity.

Low stiffness or load deflection rates provide (i) the ability to apply lower forces, (ii) a more constant force over time as the appliance experiences deactivation, and (iii) greater ease and accuracy in applying a given force. Orthodontic wires should have a low stiffness which would allow wires to fill the bracket for control and at the same time produce lighter forces **(Burstone and Goldberg, 1980)**.

2.2.3 Formability

Orthodontic wires should be highly formable. This is the capacity of being easily shaped, bent and formed into complicated configurations, such as loops, without fracturing **(Burstone and Goldberg, 1980)**.

2.2.4 Biocompatibility and Environmental Stability

Biocompatibility includes resistance to corrosion and tissue tolerance to elements in the wire. Environmental stability ensures the maintenance of desirable properties of the wire for extended periods of time after manufacture. This, in turn, ensures a predictable behavior of the wire when in use.

2.2.5 Joinability

The ability to attach auxiliaries to orthodontic wires by welding or soldering provides an additional advantage when incorporating modifications to the appliance.

2.2.6 Friction

Space closure and canine retraction in continuous arch wire techniques involve a relative motion of bracket over wire. Excessive amounts of bracket/wire friction may result in loss of anchorage or binding accompanied by little or no tooth movement. The preferred wire material for moving a tooth relative to the wire would be one that produces the least amount of friction at the bracket/wire interface **(Burstone and Goldberg, 1980; Bedner, Gruendeman, and Sandrik, 1991)**.

2.3 MECHANICAL PROPERTIES OF ORTHODONTIC WIRES AND THEIR CLINICAL IMPLICATIONS

2.3.1 Gold Alloy Wires

The composition of the alloys used in gold orthodontic wires is similar to the Type IV gold casting alloys, although there can be wide variation between particular materials. These alloys may contain as little as 15% gold, although 55 to 60% gold is more typical. These wires are alloyed with 11 to 18% copper, 10 to 25% silver, 5 to 10% palladium, 5 to 10% platinum, and 1 to 2% nickel and acquire additional strengthening through the cold-working incorporated during the wire-drawing process. These wires can potentially be strengthened with the proper heat treatment, although they are typically used in the as-drawn condition. The yield strength of wrought gold wires can range from 345 to 1104 MPa depending on the alloy and condition, with corresponding elongations of 3 to 16% The modulus of elasticity of gold-copper alloys is approximately 103 GPa. This combination of properties makes gold very formable and capable of delivering lower forces than stainless steel. These features are very desirable; however, the commercial products typically have yield strengths in the lower end of the range, which limit springback. These wires are easily joined by soldering, and the joints are very corrosion resistant, but the use of gold wires in orthodontics has decreased because of their low yield strength and increasing cost **(Kapila and Sachdeva, 1989)**.

2.3.2 Stainless Steel Wires

(a) Classification

Series 200 steels are austenitic alloys in which a portion of the nickel content of series 300 steels has been replaced by manganese and nitrogen. Except for type 326 (a ferritic-austenitic duplex steel), all series 300 steels are austenitic, the austenite being stabilized by nickel. Series 400 steels that can be hardened by quenching are classified as martensitic steels; those that cannot be hardened by quenching are classed as ferritic. Despite these general classifications, however, martensite, ferrite, and austenitic may be present to some extent in steels of all three series. Of the series 600, only type 630 is martensitic as solution annealed and aged; the others are austenitic as solution annealed and martensitic as aged **(Lyman, 1972)**.

Steel wire products are classified in various ways. The factors of greatest interest are the kind and grades of steel; the size, shape, and mechanical properties of the wire; and the methods of drawing and treatment.

(b) Kind and Composition of Steel Used

Ordinarily, sulphur and phosphorus are kept within specified low limits for each grade of steel, while the silicon, manganese, and carbon contents are varied according to the mechanical properties desired. Occasionally, the sulphur content may be increased to improve the machinability of the steel.

In recent years, there has been a constantly increasing demand for wire having properties that cannot be obtained by cold working and heat treating plain carbon steel. This demand has been met by the addition of alloying elements such as nickel, chromium, manganese, silicon, vanadium, and tungsten, or combinations of these elements **(Smith, 1972)**.

2.3.3 Stainless Steel Orthodontic Wires

Austenitic stainless steel has been used extensively for bands and archwires. It is used primarily because of (i) its high strength, (ii) high modulus of elasticity, and (iii) complete resistance to corrosion in the mouth **(Wilkinson, 1962)**.

In the annealed state, austenitic steel consists of roughly equiaxed grains. High tensile strength, proportional limit, and hardness can be induced in these steels by plastic deformation, or cold-working. The finer the grain size before cold-working, the greater will be the improvement in the mechanical properties. Grain size can be reduced by alternate plastic deformation and recrystallization. In manufacturing practice, a wire is drawn down to size in stages. The drawing process distorts the grains into long fibrous shapes, elongated along the length of the wire. Between each drawing the wire is heated to a temperature which will cause recrystallization of the metal, the grains then being roughly equiaxed again but smaller **(Wilkinson, 1962)**. Carbon interstitial hardening and cold working contribute to the high yield strength and modulus of elasticity of stainless steel. Residual stresses present in a wire subsequent to bending can markedly affect the elastic properties of the wire **(Kapila and Sachdeva,1989)**. Heat treatment, therefore, is used in stress-relieving stainless steel after bending the wire into an arch, loops, or coils. This helps to enhance the elastic properties of the wire. The recommended temperature-time schedule for stress-relieving stainless steel is 399°C (750°F) for 11 minutes.

Commercially available stainless steel wires demonstrate a range of values both for the modulus of elasticity and yield strength. The high modulus of elasticity of stainless steel and its associated high stiffness necessitate the use of smaller diameter wires for alignment of moderately or severely displaced teeth. Unfortunately, decreased wire size results in a poor fit in the bracket and may cause loss of control during tooth movement. However, high stiffness is advantageous in resisting deformation caused by extra- and intra-oral tractional forces. The modulus of elasticity for stainless steel orthodontic alloys can be significantly decreased by the cold-working process associated with wire fabrication **(Khier, Brantley and Fournelle, 1988).**

Drake, Wayne, Powers and Asgar (1982) compared the mechanical properties of three sizes of stainless steel, nickel-titanium, and beta-titanium orthodontic wires in tension, bending, and torsion. Wires made from nickel-titanium alloy had the highest recoverable stored energy and the lowest spring rate, followed by beta-titanium alloy and then stainless steel. This implies that fewer activations or changes in arch wires should be required with nickel-titanium or beta-titanium than with stainless steel.

Joinability with stainless steel is possible by soldering but may be demanding. Stainless steel wires also can be fused together by welding, but this generally requires reinforcement with solder. Stainless steel wires have good corrosion resistance, although solder joints may corrode in the oral cavity **(Burstone, 1981)**.

The austenitic stainless steels commonly used for orthodontic bands and wires contain approximately 18% chromium and 8% nickel. Nickel is the most common cause of metal-induced allergic contact dermatitis in man and produces more allergic reactions than all other metals combined. Second in frequency is chromium **(Park and Shearer, 1983)**. These authors have demonstrated the release of nickel and chromium from stainless steel orthodontic appliances incubated in 0.05% sodium chloride solution at 37^{o}C. The amount released was well below the average dietary intake but this might sensitize patients to nickel and chromium and may cause hypersensitivity reactions in patients with a prior history of hypersensitivity to these metals.

Low levels of bracket/wire friction have been reported with experiments using stainless steel wires. This signifies that stainless steel wires offer lower resistance to tooth movement than other orthodontic alloys **(Kapila and Sachdeva, 1989)**.

2.3.4 Multi-stranded Stainless Steel Wires

Multi-stranded wires are available in varying strand size, shape and number. These wires are composed of specified numbers of thin wire sections coiled around each other to provide a round or rectangular cross-section. **Kusy and Dilley (1984)** investigated the strength, stiffness, and springback properties of a wide selection of single- and multi-stranded wires in a bending mode of stress. Their results showed that the triple-stranded 0.4445mm (0.0175") stainless steel arch wire possesses the stiffness of 0.254mm (0.010") single-stranded stainless steel wire but has at least 20% more strength and springback. Furthermore, the stiffness of the 0.4445mm (0.0175") triple-stranded wire is similar to an 0.406mm (0.016") nickel-titanium wire but only 40% that of an 0.406 mm (0.016") beta-titanium wire. However, nickel-titanium tolerated more than 50% greater activation than the multi-stranded wire. **Kusy and Stevens (1987)** state that although the elastic properties of multi-stranded wires vary widely, several of these wires compare favourably with some of the beta-titanium and nickel-titanium wires.

Ingram, Gipe and Smith (1986) noted that titanium alloy wires and multi-stranded stainless steel wires have low stiffness and high springback as compared to solid stainless steel wires. Unlike stainless steel wires, in which springback decreases with increasing thickness, the titanium and multi-stranded wires have springback properties that are relatively independent of wire size and nearly constant for a particular wire configuration. These findings agree with those made by **Kusy and Dilley (1984)** and **Kusy (1981)**. In contrast, **Schaus and Nikolai (1986)**, using a simulated arch form, noted that multi-stranded wires were less flexible than suggested by theory or previous tests. They indicated that factors such as interbracket distances, wire curvature, direction of activation relative to the curved arch form, bracket width, dimensions of bracket slot relative to wire size, and friction between bracket and wire substantially affect the flexural stiffness of the arch wire.

2.3.5 Cobalt-Chromium Wires

Cobalt-chromium alloys are available commercially as Elgiloy, Azura, and Multiphase. Elgiloy is manufactured in four tempers: soft (blue), ductile (yellow), semi-resilient (green), and red (resilient) in increasing order of resilience. This alloy with a nominal composition of 40% cobalt, 20% chromium, 15% nickel, 7% molybdenum, and 16% iron, has excellent formability in the soft condition and can be heated to 480^{o}C (900^{o}F) to obtain strength properties comparable to stainless steel **(Burstone and Goldberg, 1980)**. Blue Elgiloy is the softest of the four wire tempers. It is recommended for use when considerable bending, soldering, or welding is required. Heat treatment of blue Elgiloy increases its resistance to deformation. Yellow Elgiloy is relatively ductile and more resilient than blue Elgiloy. It can also be bent with relative ease. Further increases in its resilience and spring performance can be achieved by heat treatment. Green Elgiloy is more resilient than yellow. Elgiloy can be shaped with pliers before heat treatment. The most resilient Elgiloy is marked red and provides high spring qualities. Careful manipulation with pliers is recommended when using this wire because it withstands only minimal working. Heat treatment makes red Elgiloy extremely resilient. Since this wire fractures easily after heat treatment, all adjustments should be made before the precipitation-hardening process.

With the exception of red temper Elgiloy, non heat-treated cobalt-chromium wires have a smaller springback than stainless steel wires of comparable sizes, but this property can be greatly increased by adequate heat treatment **(Ingram, Gipe and Smith, 1986)**. The ideal temperature for heat treatment is 482^{o}C (900^{o}F) for 7 to 12 minutes in a dental furnace. This causes precipitation-hardening of the alloy, increasing the resistance of the wire to deformation and results in a wire that demonstrates properties similar to those of stainless steel. Heat treatment at temperatures above 749^{o}C (1200^{o}F) results in a rapid decline in resistance to deformation due to partial annealing **(Filmore and Tomlinson, 1976)**.

The advantages of cobalt-chromium wires over stainless steel wires are superior physical properties, greater resistance to fatigue and distortion, and longer function as a resilient spring. It can be electrolytically polished, easily soldered, and easily heat-treated to remove internal stresses and increase spring performance **(Filmore and Tomlinson, 1976).**

The high moduli of elasticity of cobalt-chromium and stainless steel wires suggest that these wires deliver twice the force of beta-titanium wires and four times the force of nitinol wires for equal amounts of activation. The resultant undesired force vectors are therefore greater with cobalt-chromium and stainless steel wires than with both types of titanium alloys. Clinically, this may translate into faster rates of mesial movement of posterior teeth, thus placing greater demands on intra- and extra-oral anchorage.

Cobalt-chromium and stainless steel wires have good formability and can be bent into many configurations relatively easily. Caution should be exercised when soldering attachments to these wires since high temperatures cause annealing with resultant loss in yield and tensile strengths. Low-fusing solder is recommended for this purpose.

Although larger frictional forces have been noted previously between brackets and cobalt-chromium wires than between brackets and stainless steel wires, a report by **Peterson, Spencer, and Andreasen (1982)** on zero torque/zero angulated brackets indicates similar values for friction between brackets and these two types of alloys. This implies that resistance to tooth movement along stainless steel and cobalt-chromium wires may be comparable.

2.3.6 Titanium Wires

Titanium has a hexagonal close-packed (HCP) crystal structure, called alpha, at room temperature. This structure transforms to a body-centred-cubic (BCC) crystal structure , called beta, at 883^{o}C (1621^{o}F).

2.3.7 Titanium Alloy Metallurgy

Titanium alloys are classified as alpha, alpha-beta and beta alloys. Alpha alloys have essentially all-alpha microstructures. In beta alloys, the BCC structure is retained after air cooling from the solution treating temperature. Alpha-beta alloys contain a mixture of alpha and beta phases at room temperature. Within the alpha-beta class, an alloy that contains much more alpha than beta is often called a " near-alpha" alloy. The names "super-alpha" and "lean-beta alpha" are also used for this type of alloy **(Mehl, Bartlo, Betner, Blackburn et. al., 1972).**

Alloying elements favour one or the other of the two structures. The addition of alloying elements also divides the single temperature for equilibrium transformation into two temperatures - the alpha transus, below which the alloy is all-alpha, and the beta transus, above which the alloy is all-beta. Between these two temperatures, both alpha and beta are present **(Polmear, 1981).**

Alloying elements that favour the alpha crystal structure and stabilize it by raising the alpha-beta transformation temperatures include aluminium, gallium, germanium, carbon, oxygen and nitrogen. The principle alloying element in alpha alloys is aluminium, but certain alpha alloys, and most commercial unalloyed titanium, contain small amounts of alpha-stabilizing elements, in addition to the beta-stabilizing elements like vanadium, as strengthening agents.

Two groups of elements stabilize the beta crystal structure by lowering the two transformation temperatures. The isomorphous group consists of elements that are completely miscible in the beta-phase; included in this group are molybdenum, vanadium, tantalum and niobium. The other group forms eutectoid systems with titanium, having eutectoid temperatures as much as $333^{o}C$ ($600^{o}F$) below the transformation temperature of unalloyed titanium. The eutectoid group includes manganese, iron, chromium, cobalt, nickel, copper and silicon.

Two other elements that often are alloyed with titanium are tin and zirconium. These elements have extensive solid solubilities in both alpha and beta phases. They do not strongly promote phase stability, but do retard the rates of transformation, and are useful as strengthening agents.

Alloys of titanium have strength properties equal to that of alloyed steel. Its specific weight is only 60% that of steel, so its strength/weight ratio is outstanding. In addition, the corrosion behavior of titanium and its alloys is excellent. It surpasses stainless steel.

Properties are directly related to microstructure. Single-phase alloys are weldable with good ductility; some two-phase alloys are also weldable, but their welds are less ductile. Two-phase alpha-beta alloys are stronger than the single-phase alpha alloys, primarily because body-centred cubic beta is stronger than close-packed hexagonal alpha. Most important, two-phase alloys can be strengthened by heat treatment because the microstructure can be manipulated by controlling heating, quenching, and aging cycles **(Pollack, 1988)**.

(a) Alpha Alloys

Most of the alpha alloys contain some beta-stabilizing alloying elements. The compositions of these alloys are balanced by a higher aluminium content so that the alloys are essentially one-phase alpha. The alpha alloys have two main attributes :- weldability and retention of strength at high temperatures. The first results from the single-phase microstructure, the second from the presence of aluminium. Alloying elements in solid solution strengthen the alpha-phase alloys. Aluminium is the most effective strengthener of alpha alloys and most importantly, its effect persists to high temperatures. Strengthening effects of carbon, oxygen, and nitrogen disappear above about 260 to 430^{o}C (500 to 800^{o}F). These elements also have embrittling effects above low limits. Hot working of alpha alloys containing more than about 6% aluminium is difficult. Hot workability of high-aluminium alpha is improved by additions of beta-stabilizing alloying elements in amounts small enough so that the beta-phase is present in small quantities in the mill-annealed microstructure.

(b) Alpha-Beta Alloys

Alpha-beta alloys which contain enough beta stabilizing elements to cause some beta-phase to persist down to room temperature, are stronger than alpha alloys. The beta-phase, as strengthened by beta alloying additions in solution, is stronger than the alpha phase. The alpha-beta alloys can be further strengthened by heat treatment. Essentially, this is accomplished by quenching from a temperature in the alpha-beta or beta field, followed by aging at moderately elevated temperature. Quenching suppresses the transformation of the elevated temperature beta-phase that would occur on slow cooling. Aging at elevated temperature causes precipitation of fine particles of alpha in the volumes that were beta grains prior to quenching. This fine structure is stronger than the coarse, annealed alpha-beta structure. Quenching from the beta field and aging usually results in low ductility. Strength of currently available alpha-beta alloys can be increased about 35% by heat treating, compared with the properties of annealed material.

(c) Beta Alloy

One available titanium alloy, Ti-3Al-13V-11Cr, is considered a beta alloy. (Strictly speaking it is a metastable alloy). Its microstructure is predominantly beta-phase after normal titanium alloy annealing procedures. The alloy is weldable in both the annealed (single-phase) and heat treated conditions. Unlike the alpha alloys, this beta alloy can be strengthened by heat treatment. Aging at elevated temperature after solution treatment results in the precipitation of fine particles of alpha and a $TiCr_2$ compound. Ultimate strengths up to about 1500 MPa with 5% elongation are possible after heat treatment. This is an increase over the annealed strength of at least 50%. Aging response of this alloy varies according to hot and cold working history and annealing procedures.

2.3.8 Microstructure

(a) Alpha Structures

Equiaxed alpha grains usually are developed by annealing cold worked alloys above the recrystallization temperature. Elongated alpha grains result from unidirectional working of the metal and are commonly found in longitudinal sections of rolled or extruded alloys.

Although some areas of alpha phase that appear in micrographs of heat treated titanium and titanium alloys may have been present before the heat treatment (frequently called "primary" alpha), other areas of alpha have been produced by transformation from beta **(Lyman, 1972)**. The alpha in these latter areas appears in different forms known as serrated, acicular, platelike, Widmanstatten, and alpha prime **(Mehl, Bartlo, Betner, Blackburn et. al., 1972)**.

(i) Acicular, Serrated and Platelike Alpha

Acicular alpha, which is the most common transformation product from beta with cooling, is produced by nucleation and growth along one set of preferred crystallographic planes of the prior beta matrix or along several sets of planes. In the latter instance, a basket-weave appearance results that is characteristic of a Widmanstatten structure.

Under some conditions, the long grains of alpha that are produced along preferred planes in the beta matrix take on a wide, platelike appearance. Under other conditions, grains of irregular size and with jagged boundaries, called "serrated alpha" are produced.

(ii) Alpha Prime (Martensite)

Alpha prime is a nonequilibrium supersaturated alpha structure produced by diffusionless (martensitic) transformation of beta. The needle-like structure, similar in appearance and in mode of formation to martensite in steel, is often difficult to distinguish from that of acicular alpha, although acicular alpha usually is less well-defined and has curved rather than straight sides **(Lyman, 1972).**

(b) Beta Structures

In alpha-beta and beta alloys, some equilibrium beta is present at room temperature. A nonequilibrium, or metastable, beta phase can be produced in alpha-beta alloys that contain enough beta-stabilizing elements to retain the beta phase at room temperature on rapid cooling from between the alpha transus and beta transus temperatures. The composition of the alloy must be such that the temperature for the start of martensite formation is depressed to below room temperature.

(c) Aged Structures

Aging of alpha prime results in the formation of equilibrium alpha plus beta, but most aged martensite structures cannot be distinguished from unaged martensite by light microscopy. Precipitation of alpha during aging of beta results in some darkening of the aged beta structure. Aging, or stressing, changes metastable beta to alpha or to eutectoid products **(Lyman, 1972)**.

2.3.9 Alpha-Titanium Orthodontic Wires

This new alloy wire is titanium based and contains 5.5-6.75 % aluminium, 3.5-4.5 % vanadium, 0.03 % iron, 0.0125 % max. hydrogen. The alpha-beta titanium alloys have compositions that support a mixture of alpha- and beta-phases and may contain between 10% and 50% beta-phase at room temperature. The alpha-titanium alloy used by **A.J. Wilcock, Whittlesea, Australia** is produced and processed to wire (2.6mm diameter) by I.M.I. Titanium (U.K.) and is known commercially as IMI 318. It is cold reduced - by **A.J. Wilcock** - to 0.5 mm diameter, solution heat treated at 850-880^{o}C, straightened by axially stressing at 450-470^{o}C in argon. It is then cut to 600mm lengths and finally age hardened at 470^{o}C for 8 hours in argon.

The near-alpha titanium wire is very similar in its tensile properties to gold in that it has comparable proof strength and Young's Modulus. This, combined with the corrosion resistance of titanium alloys in the oral environment make it an excellent substitute for gold. It is considerably cheaper than gold. Its importance to the clinician is that it occupies the gap between the lower modulus alloys nickel-titanium and beta-titanium and the high strength, high moduli stainless steel and cobalt-chromium. Thus, low forces combined with a low load deflection rate can be obtained with this alloy in relatively large gauges. This reduces the inherent tendency of archwires manufactured from fine gauge wire to deform in the mouth **(Hazel, Sokel and West, 1984)**. In terms of elastic deflection or springback, near-alpha titanium is similar to high tensile stainless steel. Hence, the clinician can activate the archwire more with this new alloy without permanent deformation with the same force levels as he can with these stainless steel wires.

As an indication of its formability the near-alpha wire has been shaped into representive Edgewise and Begg arch wires to which tipback bends were added. However, it was found that some 0.45mm diameter wires were not easily formed into loops without breaking. This is said to be probably due to the poor surface finish in the wire as the 0.40mm diameter wire was bent into loops without encountering any problems. It is clear, therefore, that the surface finish plays an important role in the formability of near-alpha wire and that attainment of a good surface finish is a necessary step in the production sequence **(Hazel, Sokel and West,1984).**

2.3.10 Beta-Titanium Orthodontic Wires

Orthodontic wires based on beta-titanium alloys became commercially available in the 1980's. An evaluation of these alloys was reported by **Goldberg and Burstone (1979)**. The modulus of elasticity of beta-titanium is approximately twice that of nickel-titanium and less than half that of stainless steel. Its stiffness makes it ideal in applications where less force than steel is required but where lower modulus materials would be inadequate to develop required force magnitudes (**Burstone and Goldberg, 1980**). The beta-titanium alloy offers both resilience and formability. Consequently, it is possible to perform greater tooth movements in the second-order direction while maintaining the form of the arch, and to fabricate all types of loops (**Kusy, 1981**).

The springback for beta-titanium is superior to that of stainless steel. A beta-titanium wire can therefore be deflected almost twice as much as stainless steel wire without permanent deformation. Beta-titanium wires also deliver about half the amount of force as do comparable stainless steel wires. For example, a 0.4572mm x 0.6350mm (0.018" x 0.025") beta-titanium wire delivers approximately the same force as a 0.3556mm x 0.508mm (0.014" x 0.020") stainless steel wire in a second-order activation. The former configuration has the added advantage of full bracket engagement and a resultant greater torque control than the smaller stainless steel wire. The formability of beta-titanium wire is similar to that of stainless steel **(Goldberg and Burstone, 1979)**. It is because of the predominance of BCC beta-phase that beta-titanium has good formability, even after considerable cold-working **(Goldberg and Burstone, 1979)**. The high ductility and formability of the beta-titanium allows it to be formed into arches or segments with complicated loop configurations. However, **Burstone and Goldberg (1980)** recommended that these wires should not be bent over as sharp a radius as stainless steel, so that care in the selection of pliers and bending procedures is required.

One of the features of beta-titanium is its weldability. Wires can be successfully joined by electrical resistance spot welding without the need for reinforcing solder. Finger spring and other auxiliaries of active nature can also be welded directly to an archwire without appreciable losses in the mechanical properties of the wire **(Burstone and Goldberg, 1980)**. **Nelson, Burstone and Goldberg (1987)**, have provided values for these optimal voltage settings. A flat-to-flat electrode configuration is recommended for welding because it produces a strong joint with low levels of distortion. Overheating of the wire causes it to become brittle.

Beta-titanium has a corrosion resistance comparable to stainless steel and cobalt-chromium alloys. Beta-titanium wires demonstrate higher levels of bracket/wire friction than either stainless steel or cobalt-chromium wires. This may imply slower rates of tooth movement during canine retraction and space consolidation with beta-titanium wires than with stainless steel or cobalt-chromium wires. Beta-titanium has good environmental stability and is compatible with the oral tissues **(Goldberg and Burstone, 1980)**.

2.3.11 Nickel-Titanium Orthodontic Wires

Nitinol, a stoichiometric nickel-titanium alloy, was first introduced for use in orthodontics by **Andreasen and Hillman (1971)** and is commercially available as

NiTi, Nitinol, Orthonol, Sentinol and Titanal. Nickel-titanium alloy is approximately 52% nickel, 45% titanium and 3% cobalt. Solid state solution hardening and cold-working are the strengthening mechanisms employed with this alloy **(Burstone and Goldberg, 1980)**. Various properties of the nickel-titanium alloys have been extensively investigated. Of these, however, two unique properties - the superelasticity and the shape memory phenomenon - have attracted considerable attention **(Miura, Mogi et al, 1986; Andreasen and Morrow, 1978)**. Compared with stainless steel, in normal handling, nickel-titanium wire is more difficult to deform permanently. It can almost be bent upon itself without taking a permanent set. It is this characteristic of exceptional elasticity which offers the clinician a real advancement in orthodontic materials application. **Andreasen and Morrow (1978)** state that nickel-titanium requires (i) fewer arch wire changes, (ii) less chair time, (iii) shortens the treatment time required to accomplish rotation and levelling, and (iv) produces less patient discomfort.

The primary advantages of nickel-titanium alloy are the outstanding springiness and flexibility. These properties result in light forces and larger tooth movement **(Kusy, 1981)**.

Heat treatment of nickel-titanium results in substantial alterations in mechanical properties of the alloy. Changes in crystallographic arrangement caused by heating produce the "memory" effect in this alloy. **Andreasen and Morrow (1978)** described the "shape memory" phenomenon as the capability of the wire to return to a previously manufactured shape when it is heated through its transitional temperature range (TTR). This effect is realized by holding the wire in the desired shape while undergoing high-temperature heat treatment. When subsequently cooled, the wire can be deformed within certain strain limits, from which it recovers its original shape if heated through its unique TTR. This change from distorted to original form involves a transformation of nickel-titanium from the martensitic to the austenitic phase.

Hurst (1986), as quoted by **Kapila and Sachdeva (1989)**, evaluated the percentage recovery of five commercially available nickel-titanium wires after subjecting these wires to tensile deformation followed by heating beyond their transitional temperature ranges (TTRs). It was noted that the percentage recovery for NiTi, Nitinol, Orthonol, light and medium Sentinol, and Titanal was about 90% and varied only slightly among these wires. Some clinical uses of the "shape memory" phenomenon have been suggested. These include the possible consolidation of extraction spaces and alignment of crowded teeth. However, further tests and improvements are required for the "shape memory" phenomenon to become widely accepted for clinical purposes.

Garner, Allai and Moore (1986), studied the forces required to overcome a simulated canine retraction assembly using nickel-titanium, beta titanium, and stainless steel wires. When stainless steel archwire was compared with the same size nickel-titanium and beta titanium archwires, the differences in force required to slide the bracket along the wire were increased significantly with stainless steel requiring the least force and beta-titanium requiring the most. These differences were judged to be caused by surface roughness. This roughness is due to the wires being cold-drawn through dies. It was also noted that bracket/wire frictional forces with nickel-titanium wires are higher than those of stainless steel wires and lower than those with beta-titanium wires in zero torque/zero angulated 0.457mm (0.018") brackets. In 0.559mm (0.022") brackets, nickel-titanium and beta-titanium wires demonstrated similar levels of friction that were greater than those with stainless steel or cobalt-chromium wires. Furthermore, **Peterson, Spenser, and Andreasen (1982)** reported that as the angulation between the bracket and archwire increases from 5^{o} to 15^{o}, stainless steel wires require an increasing amount of force to overcome friction when compared to nickel-titanium. At 15^{o} of angulation, nickel-titanium requires 61% less force to overcome friction than stainless steel of the same dimensions.

Despite the advantages already outlined, several other properties of nickel-titanium impose limitations on its use. The poor formability of these wires implies that they are best suited for preadjusted systems. Any first-, second-, and third-order bends have to be overprescribed to obtain the desired permanent bend. Nickel-titanium wire cannot be bent with "sharp-cornered' instruments. Although nickel-titanium feels quite flexible and gives the impression that it is very ductile, it will readily break when bent over a sharp edge. Clinicians generally have no difficulty in putting in first- and second-order bends or the desired torque, although the wire has to be severely overformed in order to get it to take the desired permanent bend **(Andreasen and Morrow, 1978)**. Cinch-backs distal to molar buccal tubes can be obtained by resistance or flame-annealing the end of the wire. This makes the wire dead soft and it can be bent into the preferred configuration. A dark blue color indicates the desired annealing temperature. If the nickel-titanium wire is overheated to a cherry red colour, as with stainless steel wire, there is a risk of making it brittle.

The low stiffness of nickel-titanium provides inadequate stability at the completion of treatment. This stability can be attained by means of stainless steel wire tailored to the desired final occlusion.

Findings on resistance to corrosion of nickel-titanium wires are inconsistent. **Edie, Andreasen and Zaytoun (1981)** report that nickel-titanium is as resistant to corrosion as stainless steel. **Sarkar, Redmond, Schwaninger, and Goldberg (1979)** have found nickel-titanium to be more susceptible to corrosion than other orthodontic alloys. Further, whereas **Schwaninger, Sarker and Foster (1982)** have found no changes in the flexural properties of nickel-titanium following long term immersion in a 1% sodium chloride solution, **Nicholson (1984)** reports an increase in permanent deformation and a decrease in elasticity caused by corrosion or the cumulative effects of cold-working.

Although nickel-titanium arch wires display excellent resilience and low load-deflection rates, their high cost has hampered their universal appeal. As a consequence both the cost factor and the retention of elastic properties have prompted some clinicians to reuse these arch wires **(Buckthal, Mayhew, Kusy, and Crawford, 1986; Kapila, Reichhold, Anderson, and Watanabe, 1991)**.

Recycling involves

(a) repeated exposure of the wire for several weeks or months to mechanical stresses and elements of the oral environment, and

(b) sterilization between uses.

Although **Mayhew and Kusy (1988)** and **Buckthal and Kusy (1988)** have demonstrated no appreciable loss in the elastic moduli, surface topography or tensile properties of nickel-titanium wires after as many as three cycles of various forms of heat sterilization or chemical disinfection, the effects of the oral environment on the wire properties are still inconclusive. The combined effects of repeated clinical use and sterilization on the properties of nickel-titanium wires require further investigation before recycling of these wires is recommended. Some investigations have shown that nickel-titanium is susceptible to corrosion when subjected to either oral fluid or chloride solutions. **(Edie, Andreasen and Zaytoun, 1981; Sarkar, Redmond, Schwaninger, and Goldberg, 1979; Haris, Newman, and Nicholson, 1988**). Those studies propose that some combination of pre-existing surface characteristics and corrosion following *in vivo* use or *in vitro* exposure to chloride solutions causes failure of the wire. **Harris, Newman and Nicholson (1988)** showed that storage of 0.406mm (0.016") nickel-titanium wire in a simulated oral environment (37^{o}C) for at least one month and up to four months when compared to dry, unstrained samples significantly decreases ultimate tensile strength, modulus of elasticity, and 0.2% yield strength.

2.4 CLINICAL APPLICATIONS OF ORTHODONTIC WIRES

The practical applications of orthodontic wires can be optimized by carefully selecting the appropriate alloy type and wire size to meet the demands of a specific clinical situation. **Kusy (1981)** and **Kusy and Greenberg (1982)** have recommended a sequential use of arch wires selected for optimal use of the mechanical properties of their constituent alloys. They suggest that nickel-titanium wires are suitable as a starting and intermediate wire, since it has low stiffness, high strength, and excellent springback properties. On the other hand, its low formability and non-weldability limit its total usefulness. In contrast, beta-titanium wire has a stiffness intermediate between that of nickel-titanium and the stainless steel or cobalt-chromium wires. It has ample strength, good springback, superior formability and weldability. This gives it a balanced set of characteristics for use as an intermediate or finishing wire. **Kusy and Stevens (1987)** noted that triple-stranded wires demonstrate a greater working range than either nickel-titanium or beta-titanium wires of similar or greater dimensions. They also indicate that multi-stranded wires compare favourably with titanium wires and may provide a viable alternative to the more expensive titanium wires for initial levelling. The intermediate stages of treatment require closing loops, gable bends, and attachments. Beta-titanium wires meet these demands while providing a greater range of activation than stainless steel or cobalt-chromium wires. In torsion, the formability and stiffness of stainless steel and cobalt-chromium wires far exceed those of the titanium wires, thereby making these alloys the finishing wires of choice. The lower friction between stainless steel or cobalt-chromium wires and brackets suggest that these wires may be more suitable than other alloys for movement of teeth along a wire.

Whereas the orthodontist has routinely obtained a selection of forces by changing the wire size or shape, an alternative approach might be to vary the wire alloy. **Burstone (1981)** states that the rationale behind primarily varying the material rather than the geometric parameters includes better control over the amount of play between attachment and wire, orientation of wires for directional distribution of forces, preferential orientation of rectangular wires, and over-all reduction in the number of wires used for treatment. He states that advances in orthodontic wire alloys have made it possible to control wire stiffness by varying material properties, namely, the modulus of elasticity. This is known as "variable modulus orthodontics"

Other advantages of "variable modulus orthodontics" are as follows:

(1) The amount of play between bracket and wire is not dictated by the desired wire stiffness, but is under the full control of the clinician.

This implies that the orthodontist determines the amount of bracket wire play desired before selection of the wire. Once the cross-sectional size and shape have been established, the desired stiffness can be implemented by selecting an alloy with an appropiate material stiffness.

(2) The low moduli of elasticity of the newer orthodontic alloys permit the use of light, rectangular wires even during the early stages of treatment. Rectangular wires are preferable over round wires because they can be better oriented in the bracket in such a way that forces work out in the proper directions. They further aid in patient comfort by preventing loops from turning into the cheeks and gingiva. Rectangular wires also maintain better control over root position by delivering both moments and forces.

(3) The use of newer orthodontic alloys with their lower moduli of elasticity offers substantial advantages with a 0.559 mm (0.022") bracket slot.

(4) The selection of an appropriate alloy type and wire size may reduce the number of arch wires needed for alignment by reducing bracket/wire play early in treatment. In addition, since the titanium wires also work more efficiently and over longer periods of time because of their greater springback, the number and frequency of arch wire changes are reduced **(Burstone, 1981; Kapila and Sachdeva, 1989)**.

CHAPTER 3

MATERIALS AND METHODS

3.1 MECHANICAL PROPERTIES

3.1.1 Materials

The following orthodontic wires were used for the tensile tests.

(1) 0.4445mm x 0.6350mm (0.017" x 0.025") rectangular (straightened) alpha-titanium wire **(A.J. Wilcock, Australia),**

(2) 0.4445mm x 0.6350mm (0.017" x 0.025") alpha-titanium combination wire **(A.J. Wilcock, Australia).**

3.1.2 Methods

Tensile Test

Each wire was subjected to two types of tensile tests on an Instron Universal Testing Instrument.

In tensile tests the ends of a wire are fixed into grips, one of which is attached to the load measuring device on the tensile machine, and the other to the straining device. The strain is usually applied by means of a motor-driven cross-head and the elongation of the wire is indicated by its relative movement. The first series of tests used cross-head movement as a measure of extension. The cross-head speed was 1mm/min. A relatively large distance (175mm) between the grips was used to minimise errors in measurement. In the second series of tests an extensometer (gauge length 26.5mm) was used to measure the extension of the wire (**Fig. 2**).

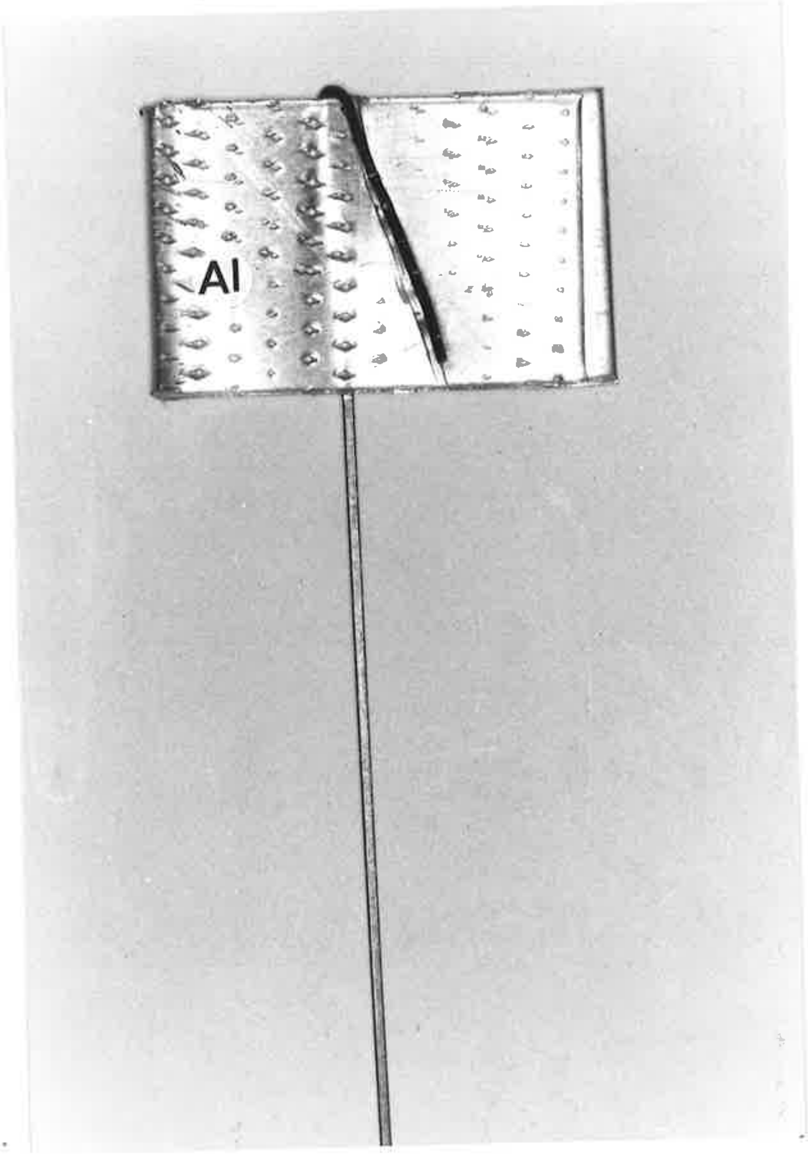
The Instron Universal Testing Instrument plotted the load applied to the wire against the change in length of the wire. Stress is defined as the ratio between a load applied to a sample and the original cross-sectional area. Strain is defined mathematically as the ratio between the change in length of a stressed sample to the original length. The modulus of elasticity is the ratio of stress to strain **(Pollack, 1988)**. From the stress-strain graphs obtained, the modulus of elasticity, yield strength, 0.2% proof stress and tensile strength of the wires were determined.

Some difficulty was experienced in that the wires fractured outside the gauge length. The wires often failed prematurely at the point of the grip. This problem was overcome by feeding the wire into an aluminium casing and winding it around the casing before it entered the groove of the grip (**Fig. 3**).

Fig. 2 Part of the Instron universal testing machine with an extensometer (E) in position.

INSTRON
E
INSTRON

Fig. 3 An alpha-titanium wire wound around an aluminium casing (Al).


Al

3.2 SCANNING ELECTRON MICROSCOPY

3.2.1 Materials

The following orthodontic wires were examined in the scanning electron microscope.

(1) Stainless steel orthodontic wires.
  (a) 0.406mm (0.016") round stainless steel wire (Brittle-Test wire) **(A.J. Wilcock, Australia)**.
  (b) 0.406mm (0.016") round stainless steel premium plus wire **(A.J. Wilcock, Australia)**.
  (c) 0.508mm (0.020") round stainless steel special plus wire. **(A.J. Wilcock, Australia)**.
(2) 0.4445mm x 0.6350mm (0.017" x 0.025") rectangular alpha-titanium orthodontic wire **(A.J.Wilcock, Australia)**.
(3) 0.4445mm. x 0.6350mm (0.017" x 0.025") beta-titanium (TMA-Titanium Molybdenum Alloy) rectangular orthodontic wire **(Ormco, California)**.
(4) Nickel-titanium orthodontic wires.
  (a) 0.406mm (0.016"), round NiTi superelastic wire **(Ormco, California)**.
  (b) 0.406mm (0.016"), round Chinese NiTi wire **(Ormco, California)**.

3.2.2 Methods

Preparation of Wires for SEM

Short lengths (10mm) of each wire were cut to enable a study of the longitudinal and cross-sectional microstructure. Specimens were mounted in bakelite according to standard metallographic practice. The samples were ground on 1200 grade wet silicon paper and polished on a 6μm diamond polishing lap. Final polishing was achieved using a 1μm diamond lap. The polished specimens were ultrasonically cleaned in baths of kerosene and alcohol and dried in a blast of warm air.

To reveal the microstructure it was necessary to etch the surface. Different standard etchants for stainless steel and titanium wires were tried out. These included Keller's reagent, Kroll's reagent, Marble's reagent and many other reagents listed by **Vander Voort (1986)**. These reagents, used at recommended etching and slightly increased times did not produce a suitable etch. After repeated trials, a reagent containing 10% concentrated nitric acid, 2% concentrated hydrofluoric acid and 88% water was found to give a somewhat reasonable etch. The clean and dry polished surface of the wires was etched for the following times.

Stainless steel wires	7 minutes.
Alpha-titanium wire	4 minutes.
Beta-titanium (TMA) wire	4 minutes.
Nickel-titanium wires	3 minutes.

The specimens were then washed in water, ultrasonically cleaned in alcohol and then dried in a blast of warm air.

A Philips SEM 505 scanning electron microscope operating at 20 kV was used to examine the specimens and photograph the microstructure. Qualitative microanalysis of the wires was performed using an energy dispersive X-ray spectrometer (EDS) system (Tracor Northern TN 5500 model) on the Philips SEM 505 scanning electron microscope using an accelerating voltage of 20 kV and an electron beam current of 3nA.

3.3 TRANSMISSION ELECTRON MICROSCOPY

3.3.1 Materials

The following materials were examined in the transmission electron microscope.

(1) Discs measuring 3mm in diameter and 2mm thick derived from commercially available sheet titanium I.M.I 318 (Ti-6Al-4V) alloy.

(2) Used and unused 0.4445mm x 0.6350mm (0.017" x 0.025") alpha-titanium wire **(A.J. Wilcock, Australia)**.

(3) 0.4445 x 0.6350mm (0.017" x 0.025") beta-titanium wire **(Ormco, California)**.

(4) 0.406mm (0.016") round stainless steel wire **(A.J. Wilcock, Australia)**.

3.3.2 Methods

Preparation For TEM

(1) An initial study was made of commercially available titanium (Ti-6Al-4V) in order to determine optimum electropolishing conditions and observe typical microstucture in this alloy. Discs measuring 3mm in diameter and approximately 2mm thick were "punched" out from the sheet titanium alloy using a disc punch. 3mm diameter discs were used in order to obtain a proper fit in the specimen holders of the electropolisher and the transmission electron microscope. The discs were ground to approximately 0.1mm in thickness on 1200 grade silicon paper. The thinned sections were electro-polished by a twin-jet technique using an electrolyte containing 10% by volume perchloric acid, 20% glycerol and 70% ethanol. Electrolytic thinning was carried out at about -30^{o}C using liquid nitrogen in alcohol coolant and at a potential of 12.5 volts.

(2) The stainless steel, used and unused alpha-titanium, and beta-titanium specimens were much more difficult to prepare. The width of these wires were too narrow to fit snugly in the specimen holders. 3mm lengths of these wires were used to prepare the specimens. Since the wires did not completely cover the lumen of the specimen holder of the electropolisher, determining the thickness (or thinness in this case) was difficult. The specimens were checked for perforations at short intervals with the help of an optical microscope. Suitable thinned sections of stainless steel wire were obtained by this method. However, preparation of the titanium wires proved to be difficult. The titanium specimens obtained by jet polishing had very small areas of sufficiently thinned areas and often oxides were formed.

In May 1992, the Gatan Dimple Grinder Model 656 and Gatan Ion Beam Mill (Duomill^{TM} model 600) became available for use. The titanium wires were thinned using these machines. The specimens were thinned to approximately 5µm using the Dimple Grinder and were further thinned down to approximately 1µm with the Ion Beam Mill **(Fig. 4)**.

The wire specimens were too narrow to fit in the TEM grid holder. As such folder oyster grids or sandwiching the specimen between two grids were used. It was found to be unsuitable because the thinned areas through which the electron beam could pass through, being very small in area, were frequently covered by the grid mesh.

The specimens were then welded on to grids with large slots. This method worked but it was discontinued because it was felt that the high temperature created during welding could affect the microstructure of the specimen. The wires were then glued with fast setting Araldite^{TM} under an optical microscope. The glueing process had to be carried out slowly and carefully in order to prevent the glue from completely covering these small specimens. The longitudinal and cross-section specimens of alpha- and beta-titanium wires were prepared using this method.

In July 1992, an embedding epoxy resin (Gatan G-1 Epoxy) used for embedding thin wires, fibres, powder and other small objects was introduced in Australia. This resin has made the preparation of TEM specimens, especially cross-section of wires, very much easier. The instructions for embedding the specimens and the curing time should be strictly followed. The 1-2µm thick specimens can dislodge from the embedding material and could create problems if dropped into the pole piece of the electron microscope. The procedure of preparing the specimens for TEM using this material is outlined in **Fig. 5**. The cross-section specimens of stainless steel wire were prepared using this embedding material.

The thinned sections were examined in a JOEL 2000FX transmission electron microscope operating at 200kV.

Fig. 4 Steps in TEM specimen preparation.

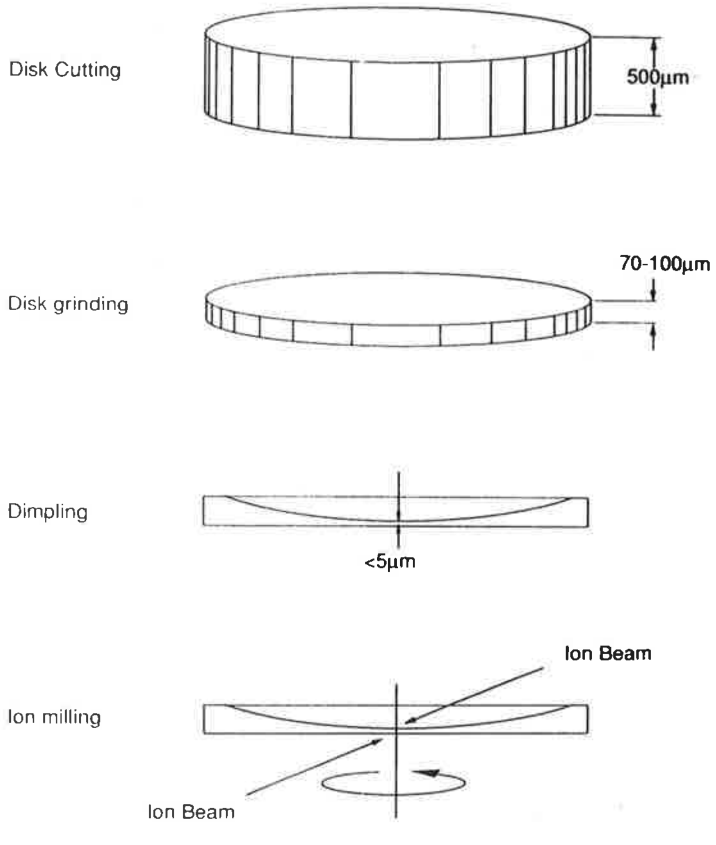
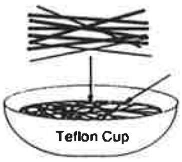

Disk Cutting
500μm
70-100μm
Disk grinding
Dimpling
<5μm
Ion Beam
Ion milling
Ion Beam

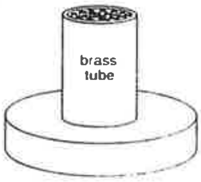
Fig. 5 Steps in the preparation of TEM specimens.

STEP 1



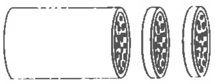
Place strips of 15mm wire in Gatan G-1 expoxy resin and transfer the mixture to a 3mm diameter steel tube

STEP 2



Cure the G-1 epoxy on a hot plate for 10 minutes at 130°C.

STEP 3



Slice the filled tubes using a diamond saw to obtain 3mm discs.

STEP 4

Disc grind, dimple grind and ion mill to perforation.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 MECHANICAL PROPERTIES: TENSILE TESTS

The results for the two different samples of alpha-titanium wires are given below.

4.1.1 Alpha-Titanium (Straightened) Wire

Initial tests were carried out without an extensometer, and the results are presented in **Table 1**.

TABLE 1

	TEST 1	TEST 2	TEST 3
MODULUS OF ELASTICITY (GPa)	88.1	86.3	87.5
YIELD STRENGTH (MPa)	862.0	862.0	862.0
TENSILE STRENGTH(MPa)	1156.0	1198.0	1156.0

Of primary interest in this study was the modulus of elasticity. Data published for all titanium alloys (commercially pure, alpha, near-alpha, alpha and beta, and beta alloys) in their standard supplied conditions (annealed or solution-treated and aged) all give typical values between 100 and 125 GPa **(Metals Handbook, 1990)**. Thus the elastic moduli determined in the current tests are decided, therefore, to carry out further experiments using an extensometer to measure strain more accurately. These results are presented in **Table 2**.

TABLE 2

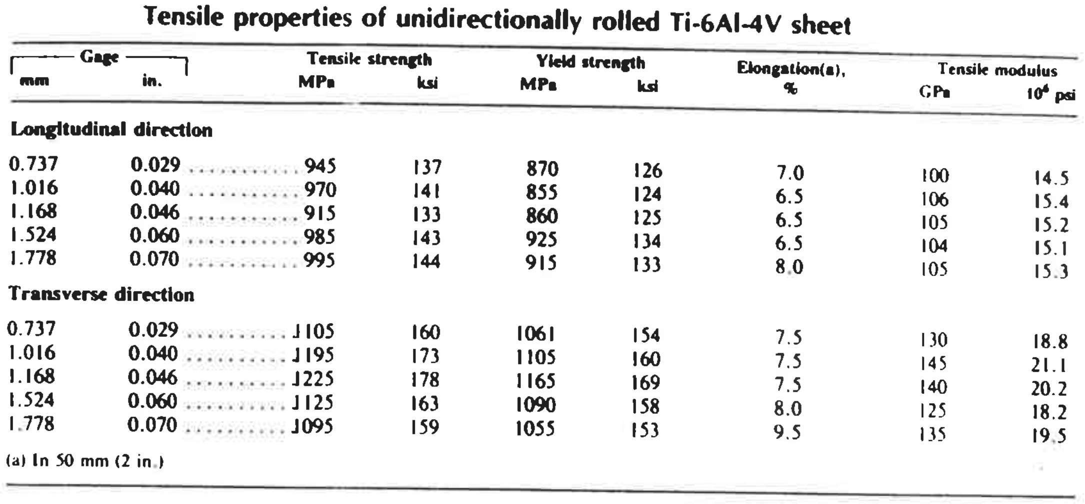
	TEST 1	TEST 2	TEST 3	TEST 4	TEST 5	TEST 6
MODULUS OF ELASTICITY (GPa)	85.15	86.07	85.15	83.38	90.7	89.4
YIELD STRENGTH (MPa)	896.5	*	784.4	*	926.0	*
0.2% PROOF STRESS (MPa)	1048.6	*	936.5	*	1038.0	*
TENSILE STRENGTH (MPa)	1176.6	*	1127.0	*	1166.0	*

* Not determined

Surprisingly, the elastic moduli were very similar to the results obtained in the initial tests **(Table 1)**.

It will be shown later (in the SEM study) that this wire appears to conform to a composition containing aluminium and vanadium. Although no information is available specifically relating to the properties of this material in the heavily drawn condition, some idea of the effect of deformation and section size can be obtained from **Table 3 (Metals Handbook, 1990)** which relates to rolled sheet.

TABLE 3



Tensile properties of unidirectionally rolled Ti-6Al-4V sheet

Gage, mm	Gage, in.	Tensile strength, MPa	Tensile strength, ksi	Yield strength, MPa	Yield strength, ksi	Elongation(a), %	Tensile modulus, GPa	Tensile modulus, 10^6 psi
Longitudinal direction								
0.737	0.029	945	137	870	126	7.0	100	14.5
1.016	0.040	970	141	855	124	6.5	106	15.4
1.168	0.046	915	133	860	125	6.5	105	15.2
1.524	0.060	985	143	925	134	6.5	104	15.1
1.778	0.070	995	144	915	133	8.0	105	15.3
Transverse direction								
0.737	0.029	1105	160	1061	154	7.5	130	18.8
1.016	0.040	1195	173	1105	160	7.5	145	21.1
1.168	0.046	1225	178	1165	169	7.5	140	20.2
1.524	0.060	1125	163	1090	158	8.0	125	18.2
1.778	0.070	1095	159	1055	153	9.5	135	19.5

(a) In 50 mm (2 in.)

[Taken from **Metals Handbook (1990), Volume 2; 10th Edition**: **pg. 610**]

Here it can be seen that there is a marked difference between the tensile modulus in the longitudinal and transverse directions. Nevertheless, the values obtained in the current study are still significantly lower than the minimum in this table.

There is also some discrepancy in the strength values. Those obtained in this study suggest a much higher work hardening capacity than the published data. For example, the yield strengths are more typical of the quoted longitudinal values, while the tensile strengths more closely equate to transverse values. However, this can be explained in terms of the difficulty in the exact determination of the yield strength as the point at which the stress-strain curve deviates from linearity (that is, the limit of proportionality).

4.1.2 Alpha-Titanium Combination Wire

This was also tested using an extensometer and the results are given in **Table 4**.

TABLE 4

	TEST 1	TEST 2	TEST 3	TEST 4
MODULUS OF ELASTICITY (GPa)	90.94	85.8	85.8	91.5
YIELD STRENGTH (MPa)	691.10	*	710.3	*
0.2 % PROOF STRESS (MPa)	979.10	*	806.3	*
TENSILE STRENGTH (MPa)	988.70	*	806.3	*

* Not determined

Once again the modulus of elasticity was found to be low, but repeated tests gave consistent results.

All the values determined are however, similar to those obtained by **Hazel, Sokel, and West (1984).**

4.1.3 Discussion

Despite the reasonably consistent experimental results for the modulus of elasticity in these two alloys, it is unlikely that they represent the true value. The figures listed in **Table 1** are of the order of 15% less than the longitudinal (smaller) values for Ti-6Al-4V sheet in **Table 3**. This could have been anticipated in terms of the influence of movement in the grips, screw drives and frame of the testing machine which can significantly affect the measured elongation of the sample, and hence strain. Although relatively long "grip-to-grip" gauge lengths (175mm) were used in an effort to minimise this effect, the error still remained substantial.

The use of an extensometer should give more accurate values of strain and a true measure of that strain within the limited (26.5mm) gauge length of the wire. In the present case, no significant improvement was observed. The reasons for this are not clear but several factors could affect the result.

A major difficulty in using collated results from previous work is that the precise method by which the modulus of elasticity was measured is frequently omitted and few references to raw data are given. This can produce dramatic differences in value, particularly in materials where the measured modulus is strongly dependent on the strain rate used in the tensile test.

Another consideration is whether given values are obtained directly from mechanical tests, or from very accurate sonic tests (where the elastic modulus is determined by measuring the speed of sound in the material), or calculated from the raw data **(Rosenthal, 1964)**.

In the case of a uniaxial tensile test, the computed modulus of elasticity (derived from Hooke's Law at small deformations) is found to be much larger than the experimental value (usually called Young's modulus). The reason for this discrepancy is the omission of the associated lateral contraction of the specimen in the calculation **(Rosenthal, 1964)**.

However, it is most likely that the reduced values observed in the present study relate to the nature of the very thin wire sample itself. It is unlikely that slipping of the extensometer knife-edges has any effect, since this would be expected to be an intermittent phenomenon with evidence of a jagged line on the stress-strain graph. An attempt was made to minimise any effect of bends in the wire straightening during the test by starting with the wire under slight tension. Nevertheless, this may not have been completely successful and further errors could also have been introduced by the applied stress being dissipated in twisting the rectangular wire section in some areas. These factors could then affect the measured strain.

In addition, it should be noted that similar problems had been experienced in similar tests on other materials **(Brown, 1991)**, and the discrepency might therefore be due entirely to systematic experimental error or faulty equipment.

4.2. SCANNING ELECTRON MICROSCOPY

4.2.1 Stainless Steel Orthodontic Wires

(a) Stainless Steel 0.406mm (0.016") Brittle Wire

Microstructure

Striations parallel to the wire axis were observed in longitudinal sections (**Fig. 6a**). The striations are very closely spaced near the surface as compared to the centre of the specimen. This suggests that the specimen is more heavily deformed near the surface during cold drawing of the wire. A higher magnification image of the centre of the wire (**Fig. 6b**) shows fewer striations and small pits in the wire.

The transverse section of the wire exhibits two distinct types of contrast **(Fig. 6c)**. The dark "pitted" parts of the wire are clearly more readily etched compared with the rest of the matrix. This could be due to the following reasons:-

(i) the presence of a dual phase structure possibly the alpha- and gamma-phases,
(ii) preferential attack by the reagent on localised highly strained areas within the wire, or
(iii) the presence of precipitates or inclusions like oxides in the wire.

Microanalysis

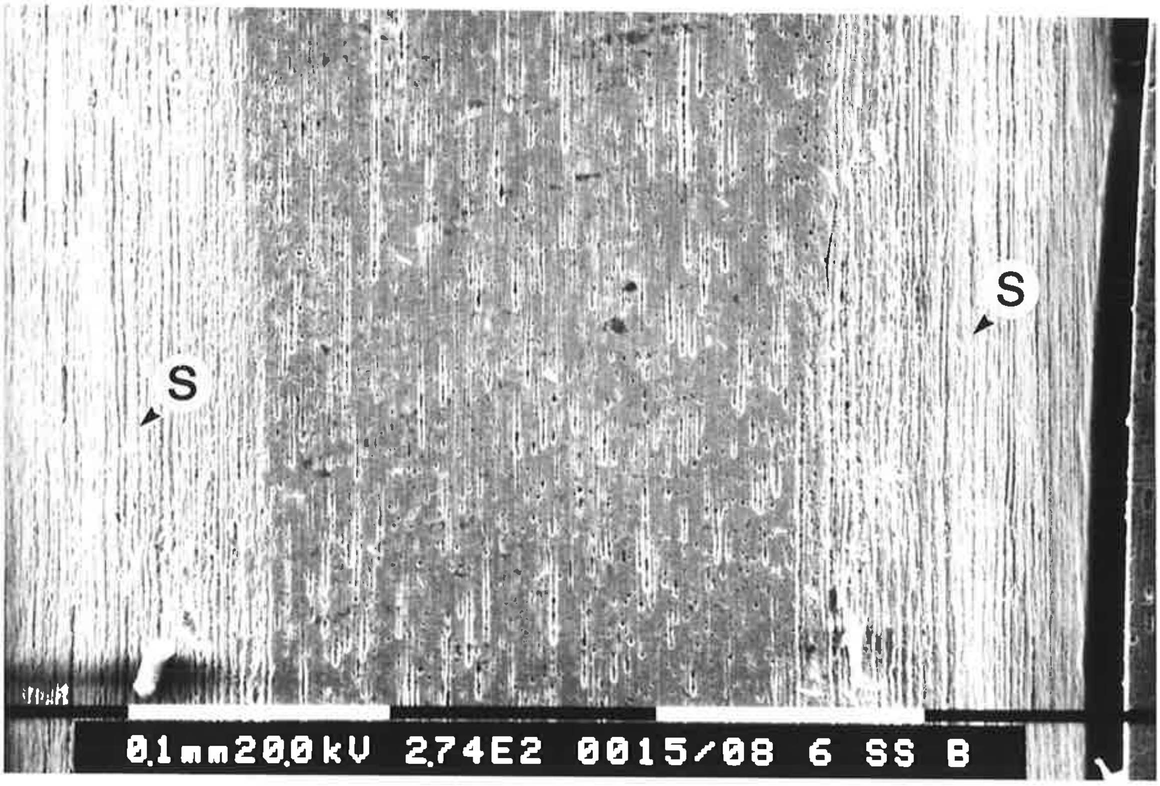
The qualitative microanalysis of this sample using an energy dispersive spectrometer (EDS) shows that it contains iron with only chromium and nickel as major substitutional alloying additions (**Fig. 6d**). Ferritic stainless steels would not be expected in this application since they do not work harden excessively during cold work, and martensitic stainless steels would be too brittle to be drawn into wire.

Martensitic and ferritic stainless steel also contain little or no nickel, and this analysis therefore confirms that this wire is produced from an austenitic grade. The obvious materials are A.I.S.I. types 302, 304, and 316. The A.I.S.I. 316 can be discounted since there is no evidence of its specified 2-3%

molybdenum content. The other two, A.I.S.I types 302 and 304 both have typical chromium and nickel contents of 18.3 and 9.3% respectively, the only difference being the carbon contents (0.12 and 0.05% respectively). Carbon cannot be detected using this EDS system and so these alloys cannot be distinguished here. However, carbide precipitation would not be desirable in these wires (since they could lead to embrittlement) and the lower carbon version 304 is therefore the most likely stainless steel used in the manufacture of this wire.

Fig. 6a Scanning electron micrograph of the longitudinal section of the (brittle) stainless steel wire showing the presence of closely packed striations (S) close to the surface of the wire.

Fig. 6b Scanning electron micrograph of the longitudinal section at the centre of the (brittle) stainless steel wire, at a higher magnification, showing some striations (S) and pits (P) on the surface of the wire.


S
S
0.1mm20.0kV 2.74E2 0015/08 6 SS B

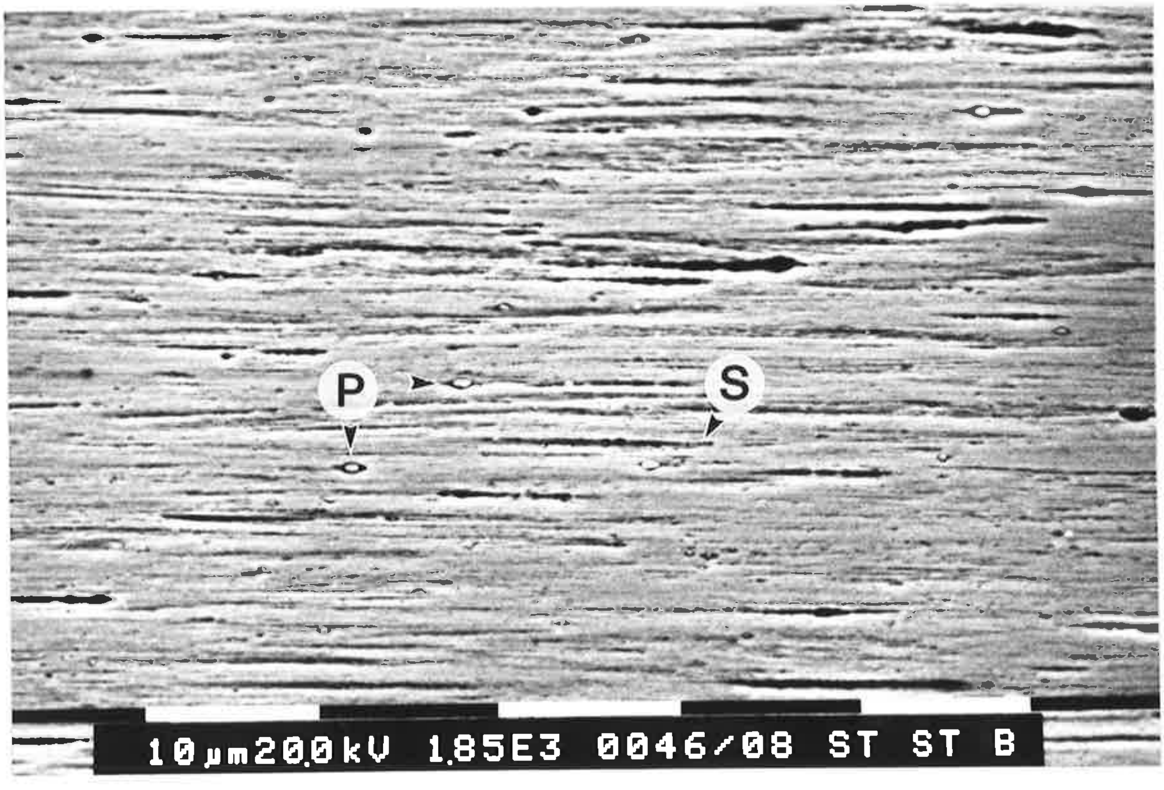
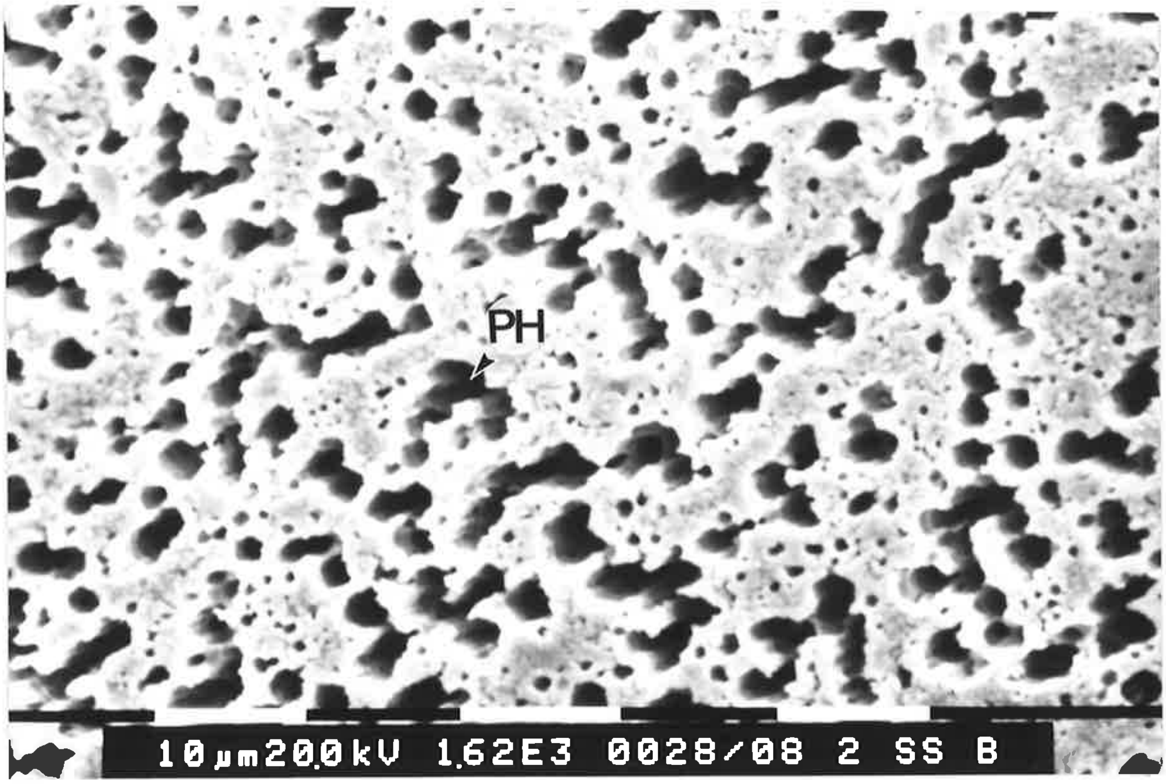
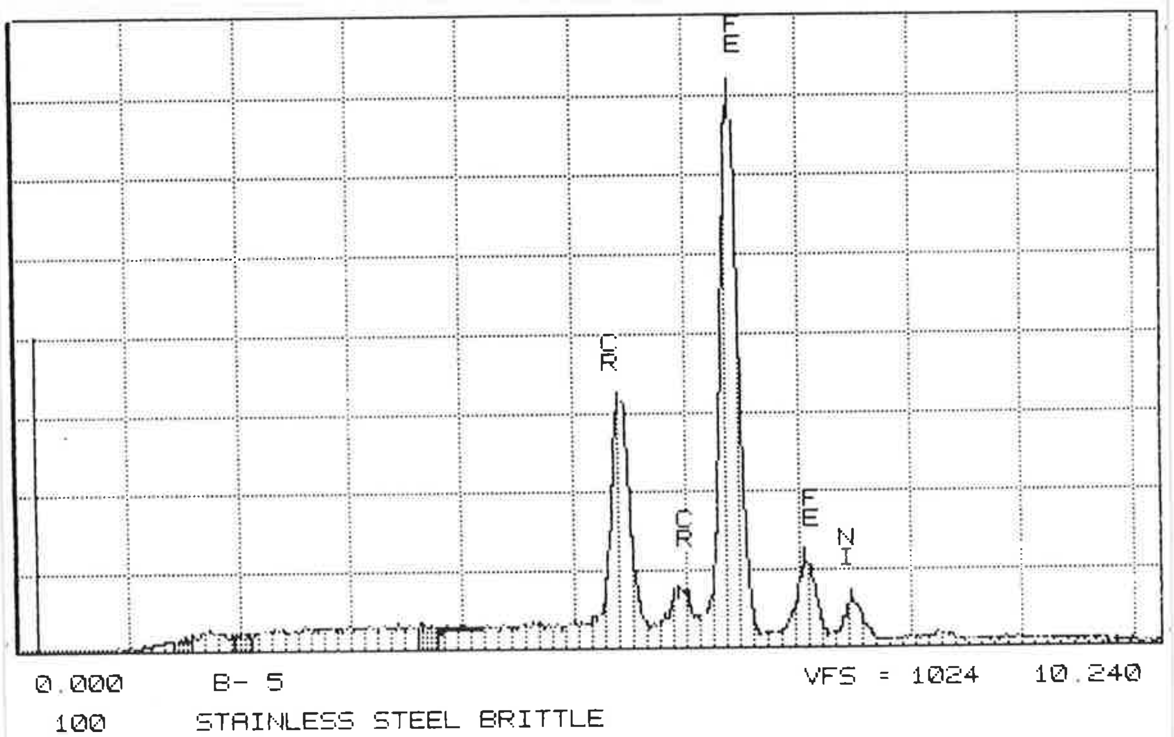

P
S
10µm20.0kV 1.85E3 0046/08 ST ST B

Fig. 6c Scanning electron micrograph of the transverse section of the (brittle) stainless steel wire showing two distinct types of contrast. It appears that there are 2 phases in the wire. One phase (PH) readily dissolves in the etchant.

Fig. 6d An X-ray analysis (EDS) of the (brittle) stainless steel wire showing the presence of iron, chromium and nickel.


PH
10µm20.0kV 1.62E3 0028/08 2 SS B


FE
CR
CR
FE
NI
0.000
B- 5
VFS = 1024
10.240
100
STAINLESS STEEL BRITTLE

(b) Stainless Steel 0.508mm (0.020") Special Plus Wire

Microstructure

This sample showed many evenly spaced striations throughout the longitudinal section of the wire (**Fig. 7a**). There are some small pits but the number of pits is less than in the previous specimen. Some transverse striations were noticed in this wire and it is believed that these could be deformation flow lines (**Fig. 7b**).

The transverse section shows the reagent etches differentially but the surface is not as deeply etched as the previous specimen (**Fig. 7c**). Here the "pits" are much finer and more evenly distributed throughout the section.

Microanalysis

A qualitative microanalysis using energy dispersive spectrometer (EDS) again showed the presence of only iron, chromium, and nickel with no significant differences from that of the brittle material.

(c) Stainless Steel 0.406mm (0.016") Premium Plus Wire

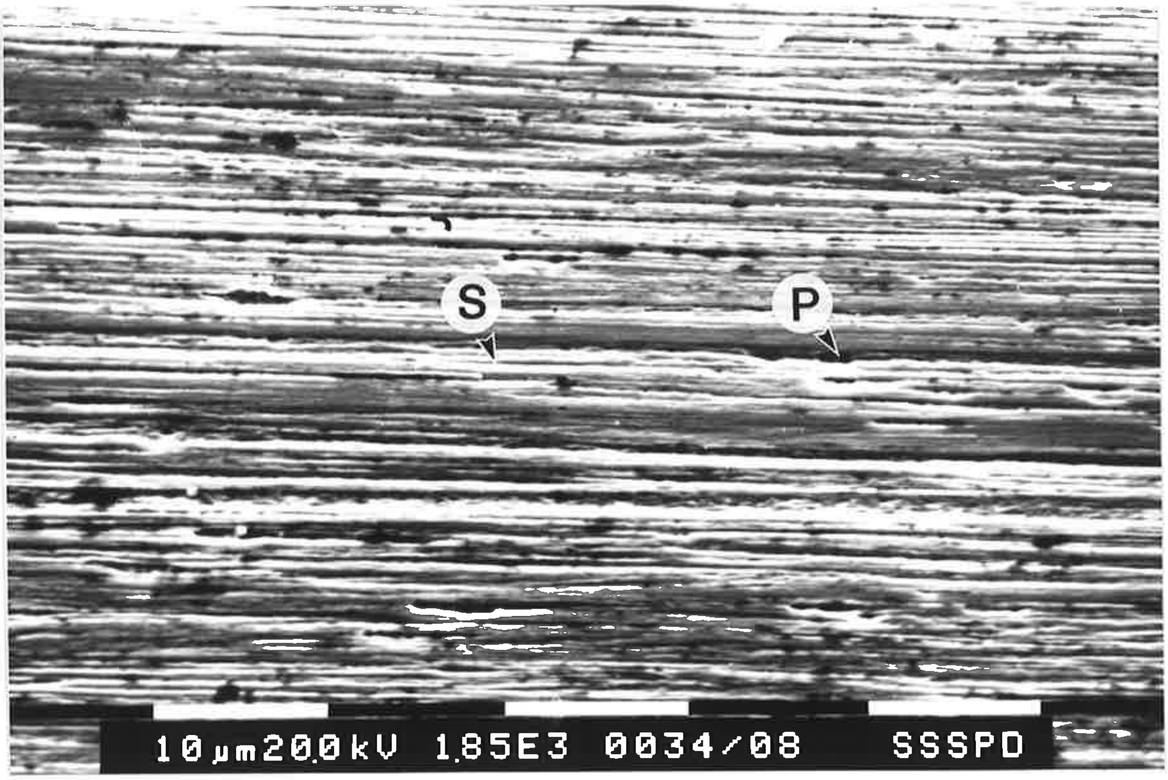
Microstructure

The longitudinal section of this wire shows less striations than either of the previous specimens and there appear to be few pits (**Fig. 8a**).

The transverse section does show some fine "pitting" but the dissolved regions are further refined compared with the previous specimens (**Fig. 8b**).

Fig. 7a A scanning electron micrograph of the stainless steel special plus wire showing striations (S) which are evenly spaced with hardly any pits (P).

Fig. 7b A scanning electron micrograph of the longitudinal section of the wire showing transverse striations (TS) which are believed to be deformation flow lines.


S
P
10 µm 20.0 kV 1.85E3 0034/08 SSSPD

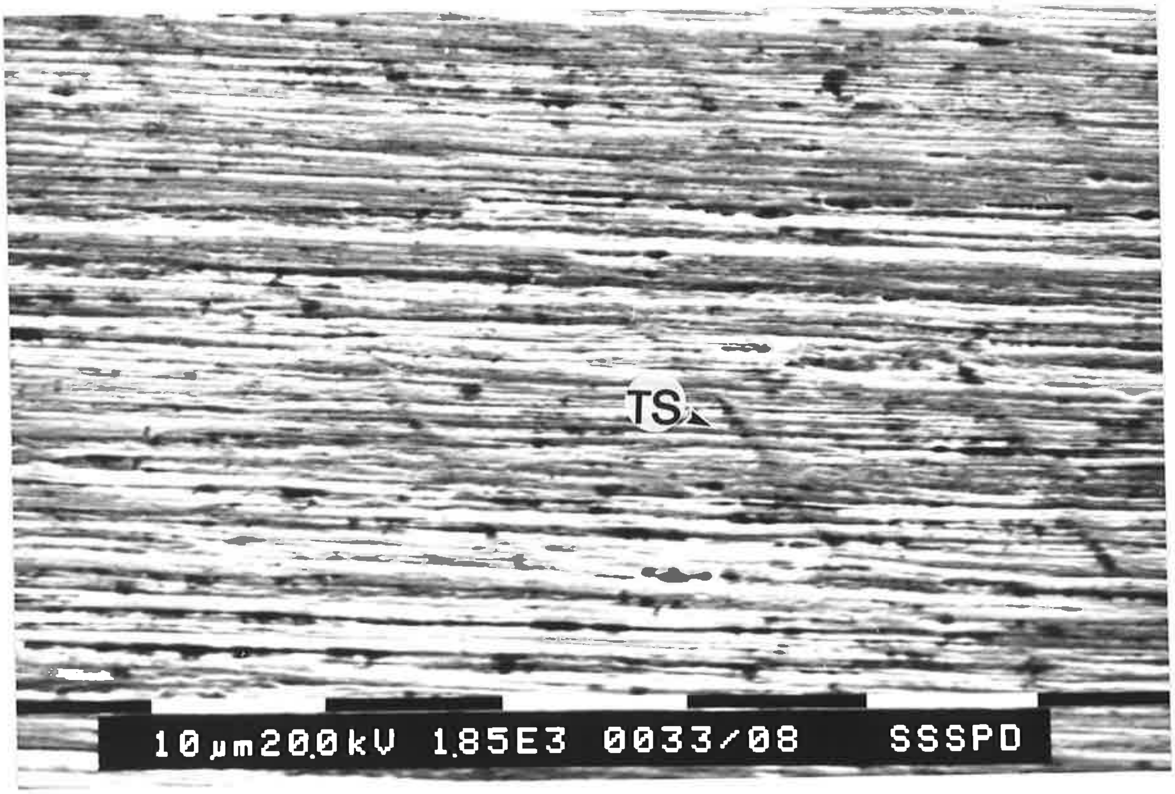

TS
10 µm 20.0 kV 1.85E3 0033/08 SSSPD

Fig. 7c A scanning electron micrograph of the transverse section of the special plus stainless steel wire showing numerous pits in the wire. It again shows a two-phase system with one phase (PH) that readily dissolves in the etchant. However, when compared to the specimen 1, this wire appears to be more resistant to the etchant.

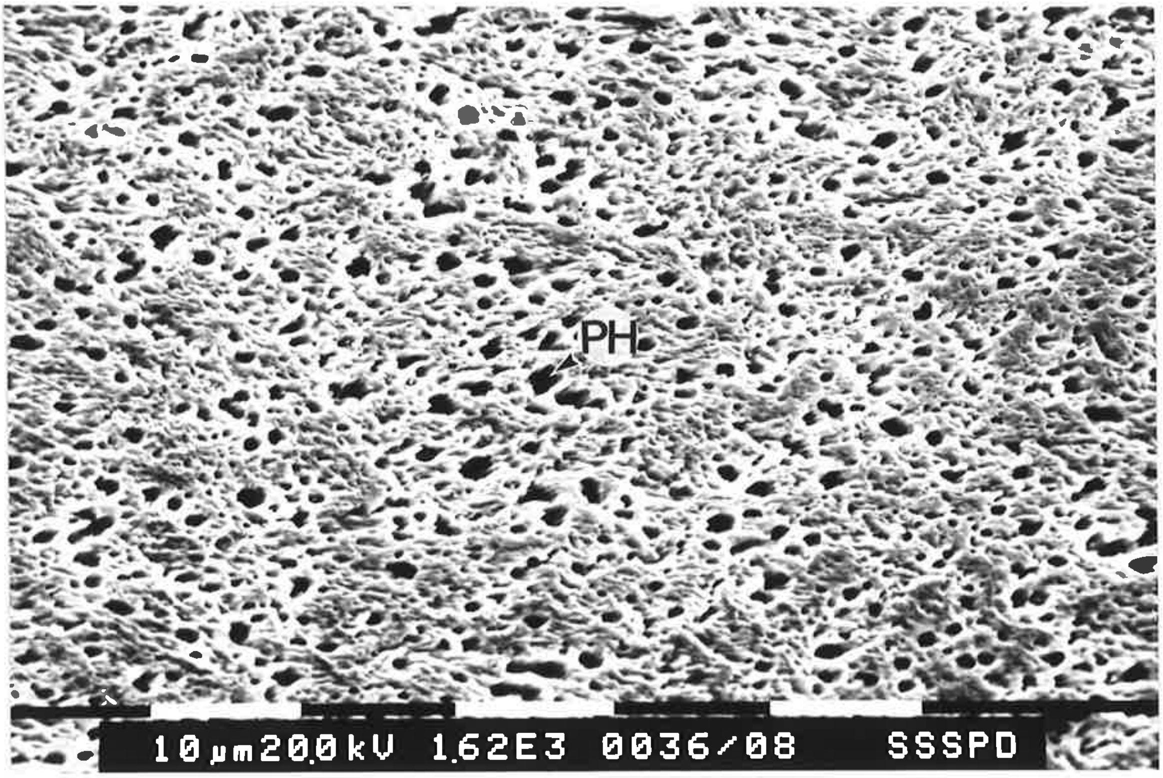
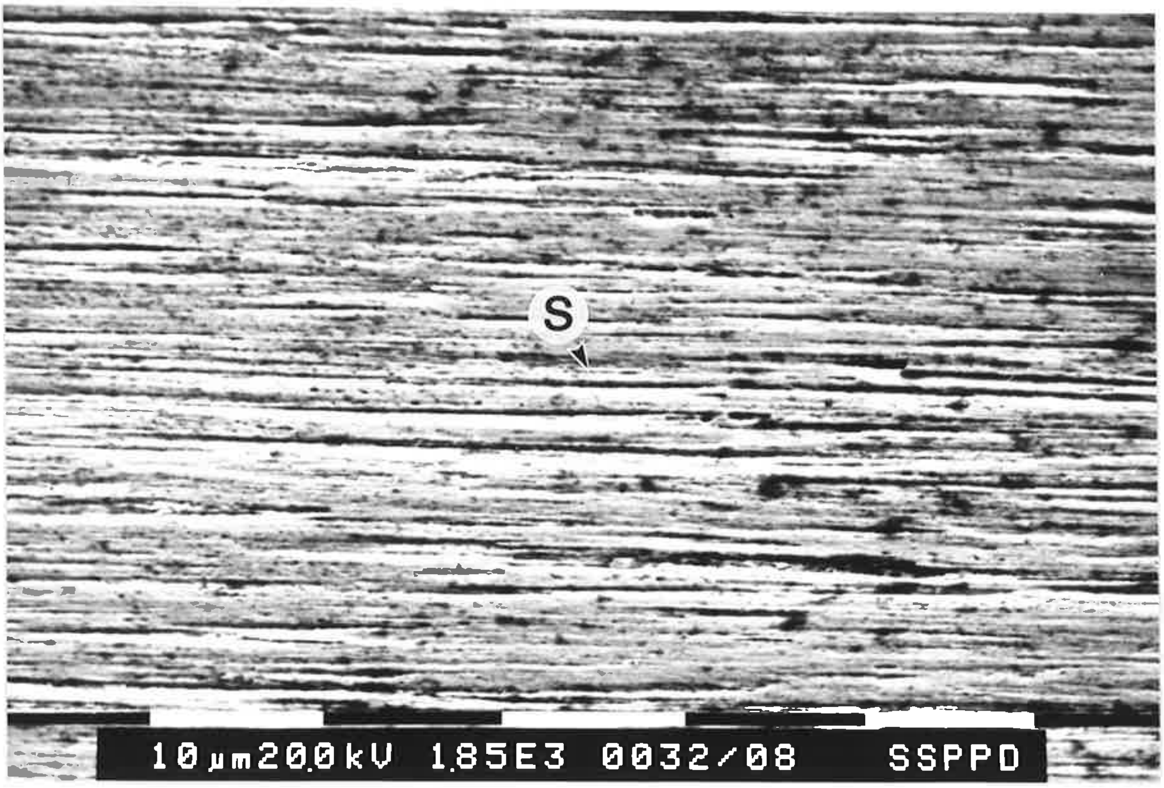
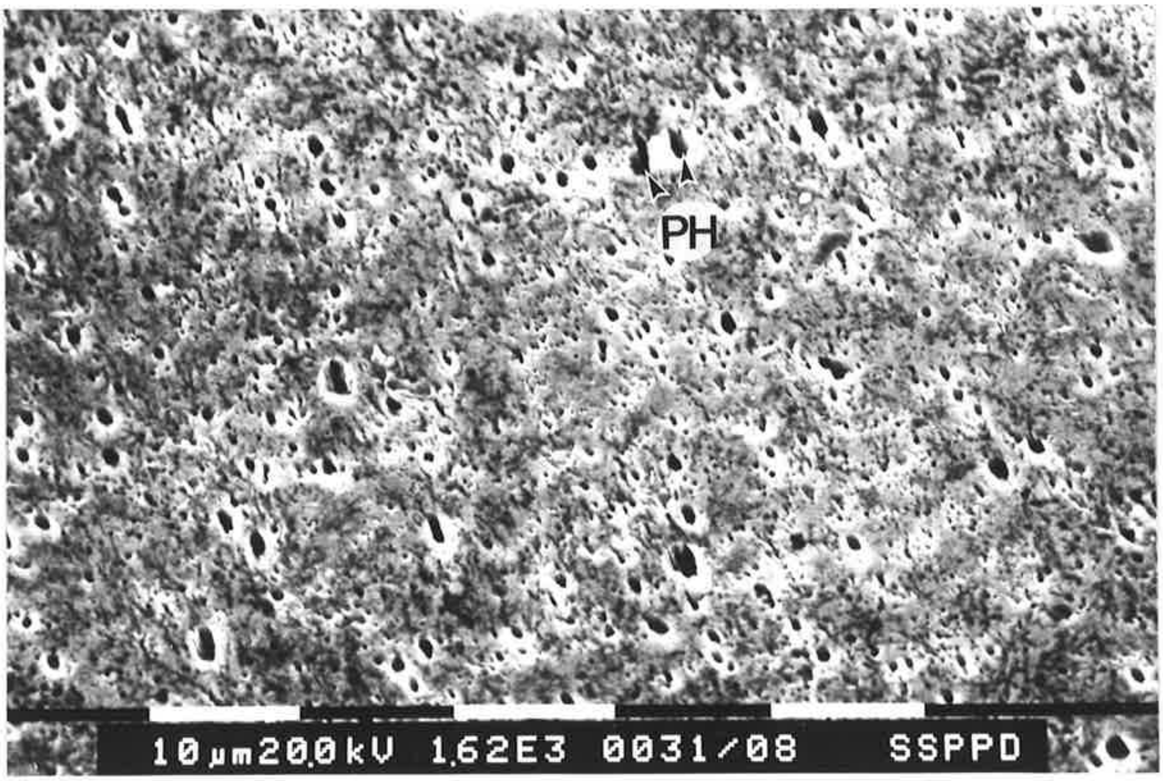

PH
10μm 20.0kV 1.62E3 0036/08 SSSPD

Fig. 8a A scanning electron micrograph of the longitudinal section of the stainless steel premium plus wire showing striations (S) which are evenly spaced throughout the wire with no pits. The striations are less than that in the special plus wire.

Fig. 8b A scanning electron micrograph of the transverse section of the stainless steel premium plus wire showing that the wire is possibly of 2-phases but the dissolved phase (PH) is significantly less than specimens 1 and 2.


S
10µm20.0kV 1.85E3 0032/08 SSPPD


PH
10µm20.0kV 1.62E3 0031/08 SSPPD

4.2.2 Alpha-Titanium Orthodontic Wire

Microstructure

The longitudinal section shows a microstructure consisting of many irregular, elongated grains (**Fig. 9a**). These are aligned parallel to the wire axis as a result of cold working. A higher magnification of the longitudinal section shows some substructure in the matrix (**Fig. 9b**).

The transverse section shows more equiaxed grains with an irregular substructure between them **(Fig. 9c)**. The latter feature is unlikely to arise from factors such as local deformation heterogeneity, and probably represents the existence of a two-phase microstructure.

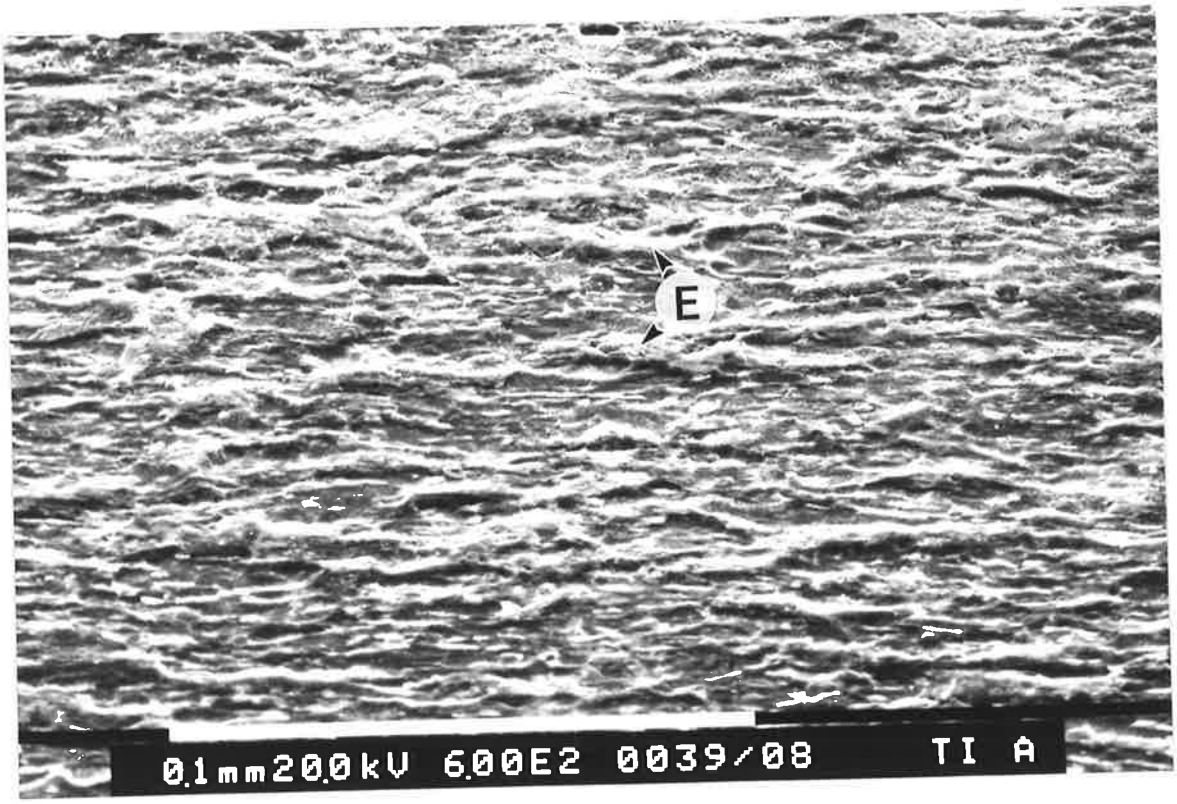
Microanalysis

The wire was analysed using energy dispersive spectrometer (EDS) and found to contain titanium, aluminium and vanadium. The peak of vanadium in the chart appears higher than that of aluminium. This is due to the peak being superimposed on a second peak of titanium and so appears exaggerated (**Fig. 9d**).

The presence of aluminium and vanadium as the only major substitutional alloying additions suggests that this material is of the Ti-6Al-4V type. This is a two-phase (alpha-beta) alloy and does explain the microstructural observations above. The structures can now be interpreted as primary (or recrystallised) alpha grains surrounded by mixed alpha-beta plates.

Fig. 9a A scanning electron micrograph of the longitudinal section of the alpha-titanium wire showing numerous irregular, elongated grains (E) aligned parallel to the wire axis.

Fig. 9b A scanning electron micrograph of the alpha-titanium wire, at a higher magnification, showing elongated grains and areas with a "wavy" substructure (W).


E
0.1mm20.0kV 6.00E2 0039/08 TI A

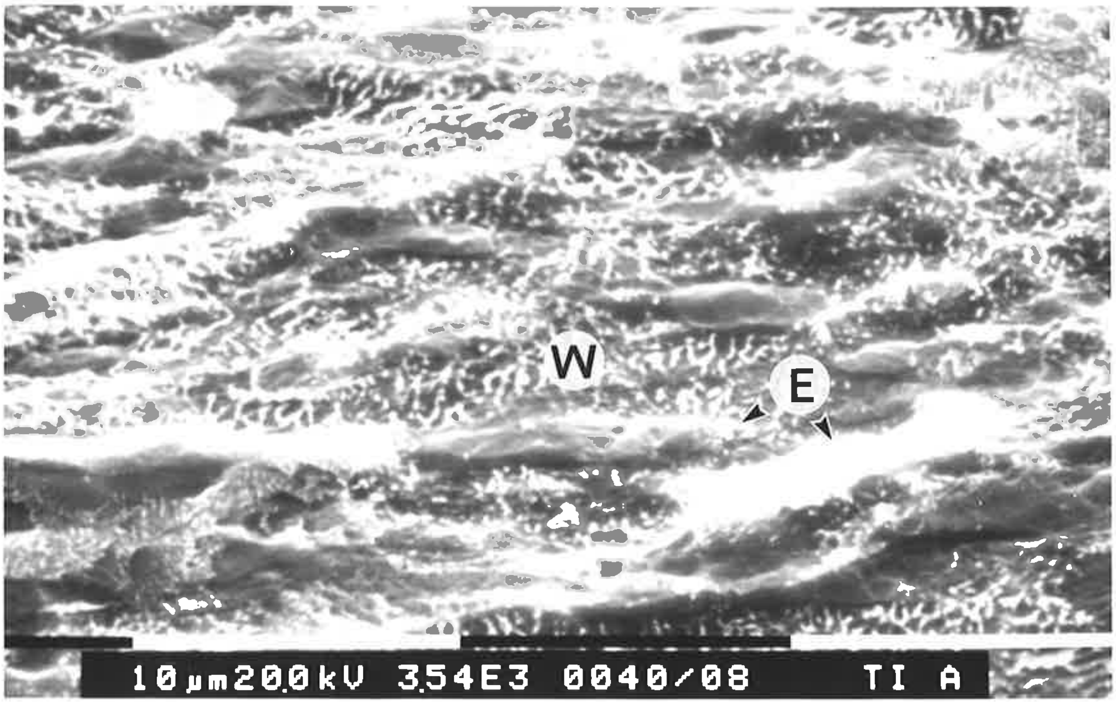
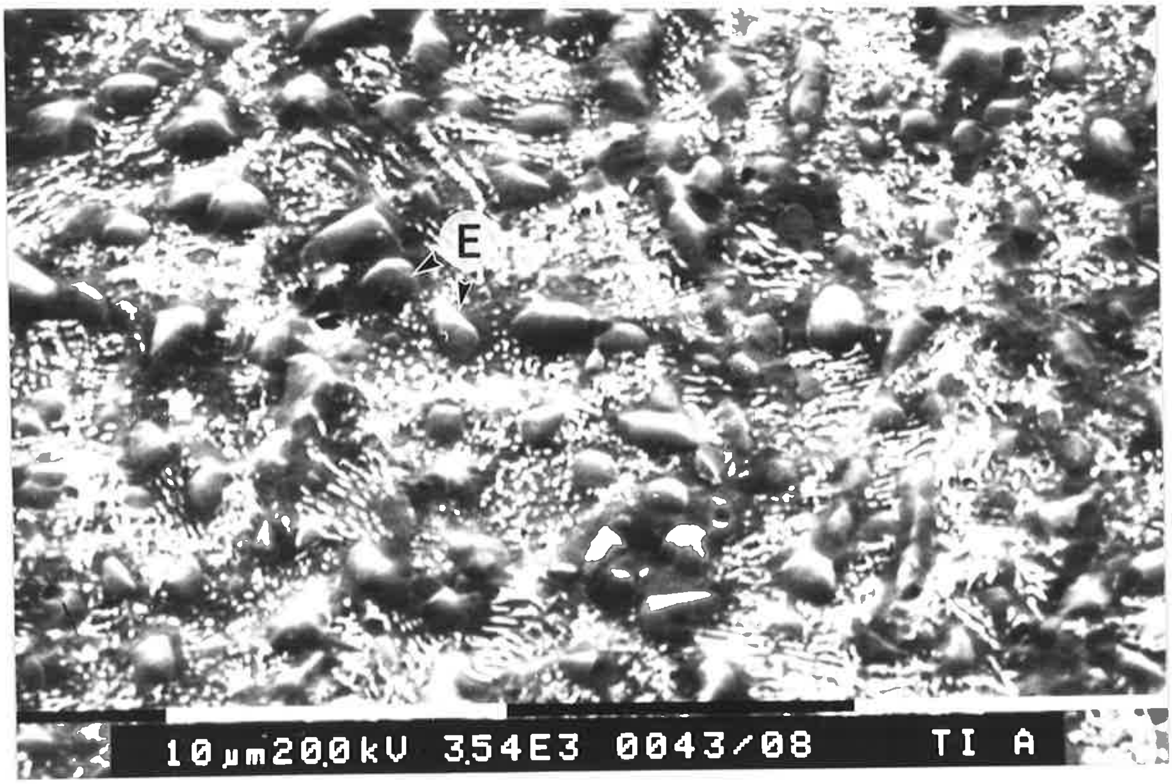
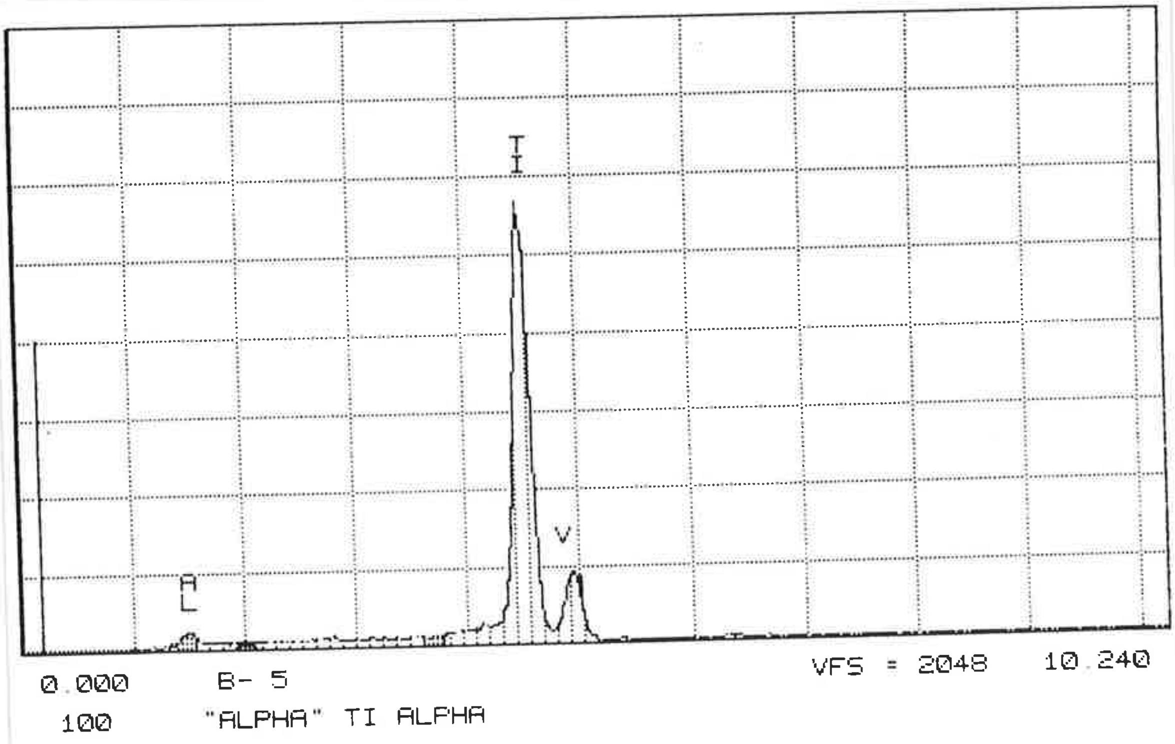

W
E
10µm20.0kV 3.54E3 0040/08 TI A

Fig. 9c A scanning electron micrograph of the transverse section of the alpha-titanium wire showing equiaxed grains (E) and substructural features.

Fig. 9d Energy dispersive X-ray spectrum of the alpha-titanium wire showing the presence of titanium, aluminium and vanadium.


E
10µm20.0kV 3.54E3 0043/08 TI A


TI
V
AL
0.000
B- 5
VFS = 2048
10.240
100
"ALPHA" TI ALPHA

4.2.3 Beta-Titanium Orthodontic Wire

Microstructure

This wire was much more difficult to etch as compared to the other wires. The longitudinal section showed a few broad striations and large shallow pits on the surface (**Fig. 10a**).

Transverse sections of this wire showed what appeared to be coarse pits in the surface but these were proved to be raised cones (**Fig. 10b**), using a Y-modulation image (**Fig. 10c**).

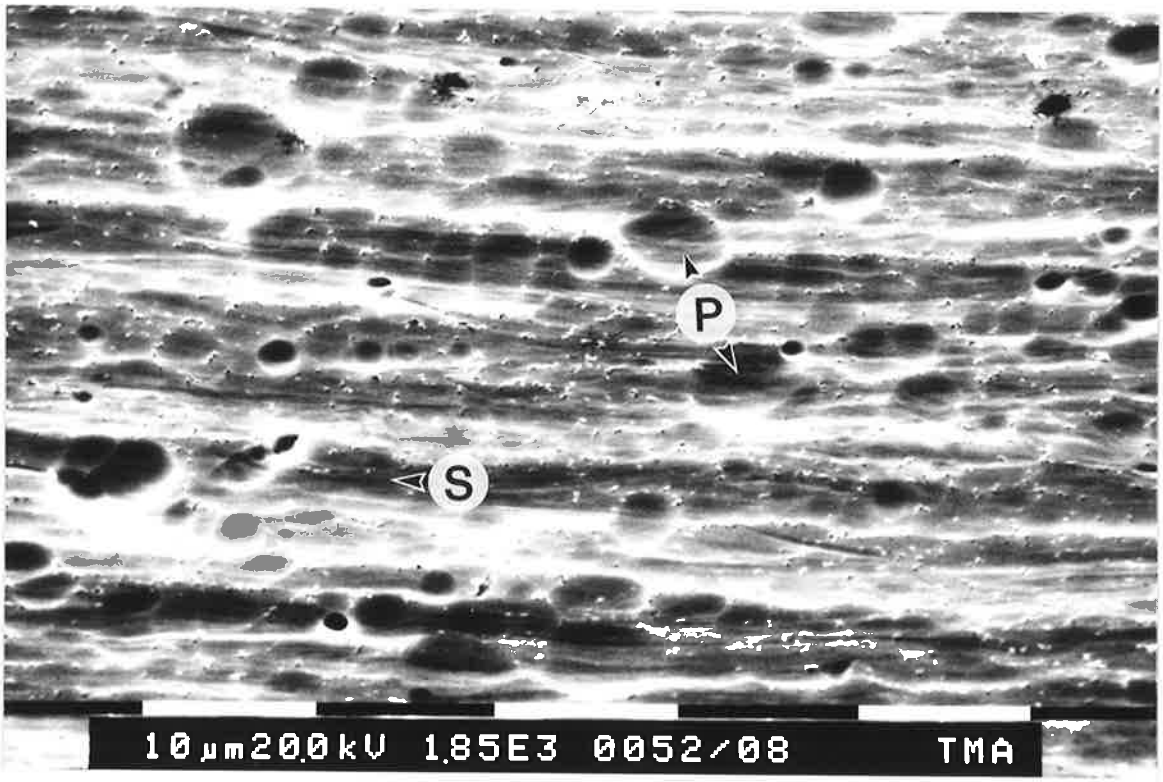
Microanalysis

The wire consists mainly of titanium with small proportions of molybdenum, tin and zirconium (**Fig. 10d**). This analysis is consistent with an obsolete commercial aircraft alloy "beta 111" which has a nominal composition Ti-11.5Mo-6Zr-4.5Sn **(Metals Handbook, 1990)**

The microanalysis of the cones showed a higher proportion of titanium compared to the matrix. Titanium has a great affinity for oxygen and it is possible that the raised cones contain titanium dioxide which could have formed during the manufacturing process. [Since oxygen has an atomic number of less than 11 it is not possible to detect its presence using this EDS system.

Fig. 10a A scanning electron micrograph of the longitudinal section of the beta-titanium wire showing the presence of very few spaced out striations (S) and shallow wide pits (P) on the surface.

Fig. 10b A scanning electron micrograph of the transverse section of the beta-titanium wire showing numerous raised cones (C).


P
S
10µm20.0kV 1.85E3 0052/08 TMA

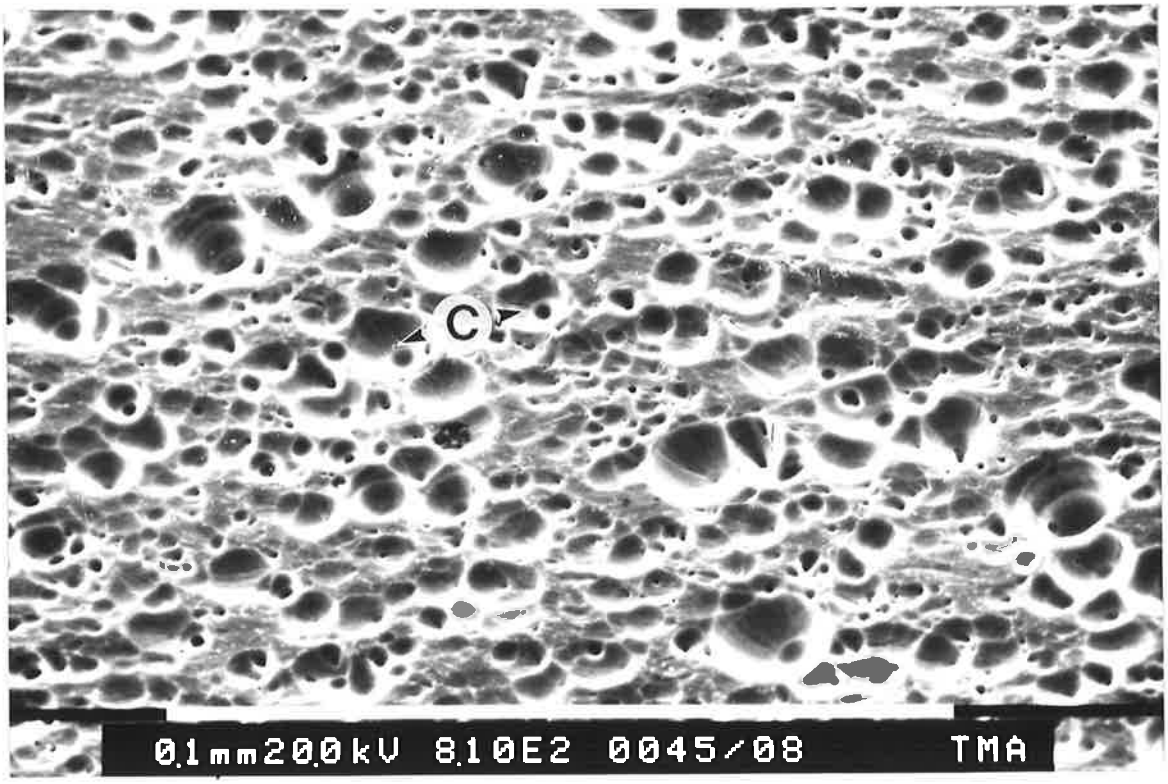
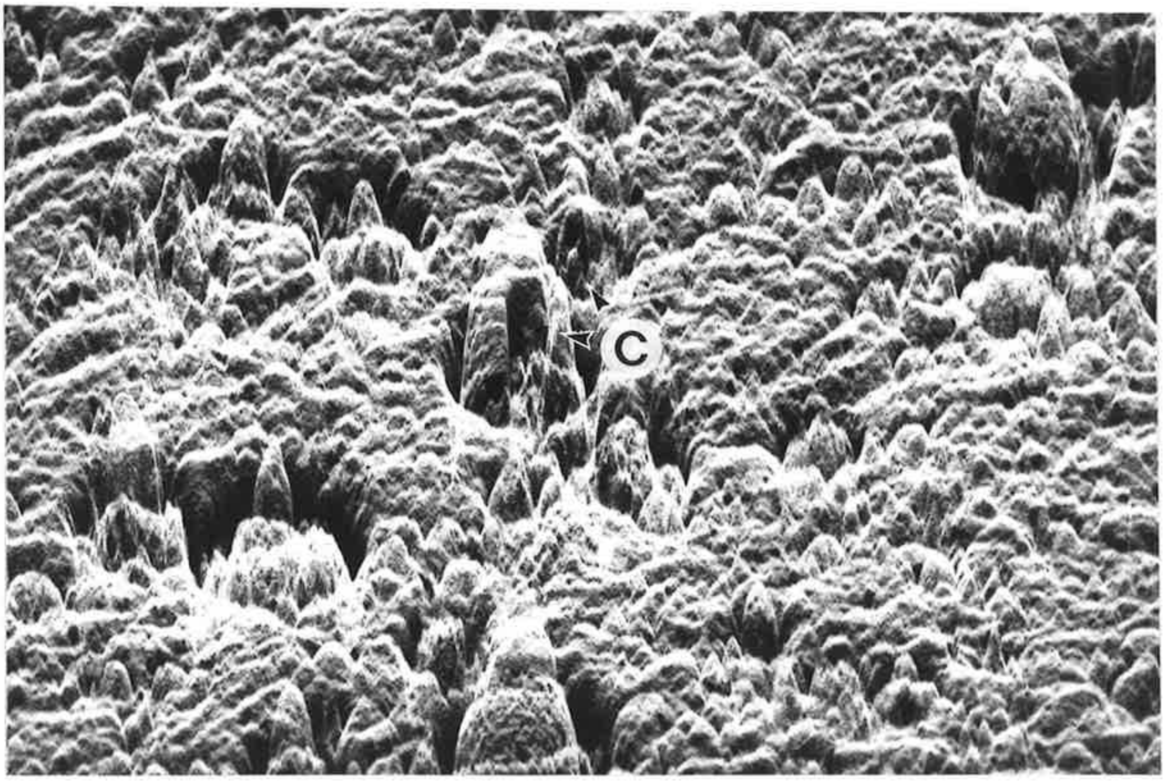
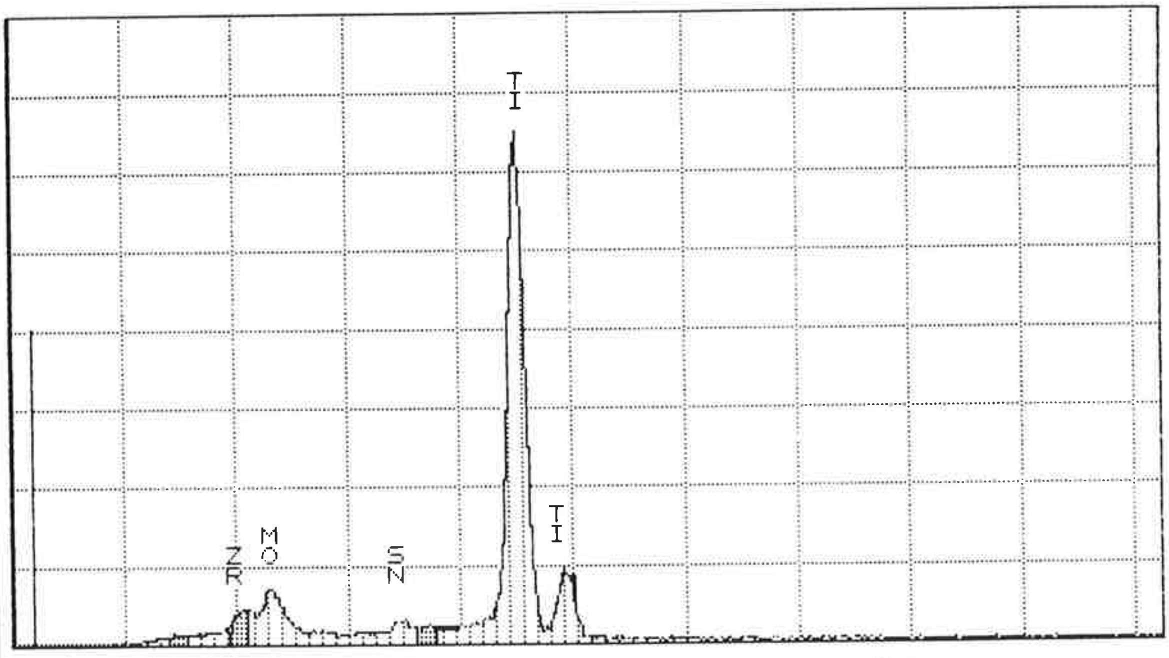

C
0.1mm20.0kV 8.10E2 0045/08 TMA

Fig. 10c A scanning electron Y-modulation image of the beta-titanium wire showing the presence of raised cones (C) of posssibly titanium dioxide.

Fig. 10d Energy dispersive X-ray spectrum of the beta-titanium wire showing the presence of titanium with small amounts of molybdenum, tin and zirconium. Oxygen is not recorded since it has an atomic number of less than 11 which cannot be analysed using this technique.


C


TI
TI
ZR
MO
SN
0.000
B- 5
VFS = 2048
10.240
100
TMA BETA TI

4.2.4 NiTi Orthodontic Wires

(a) Chinese NiTi Wire

Microstructure

Longitudinal sections show that the wire was not attacked aggresively by the etchant. There is evidence of fine, parallel striations and a few separate, and some joined pits (**Fig 11a**) with frequent evidence of particles in the pits.

Transverse sections, however, show deep pits also containing particles (**Fig. 11b**).

Microanalysis

The analysis of the matrix shows the presence of only nickel and titanium (**Fig. 11c**). However, spot analyses of the particles revealed that they were virtually pure titanium which suggests that these particles could be titanium dioxide (**Fig. 11d**).

(b) NiTi Superelastic Wire

Microstructure

The longitudinal sections revealed a few elongated pits. The matrix structure is not clearly revealed, but the grains do not seem greatly elongated and there appears to be plate-like substructure. Again precipitates were observed within the pits **(Fig. 12a)**.

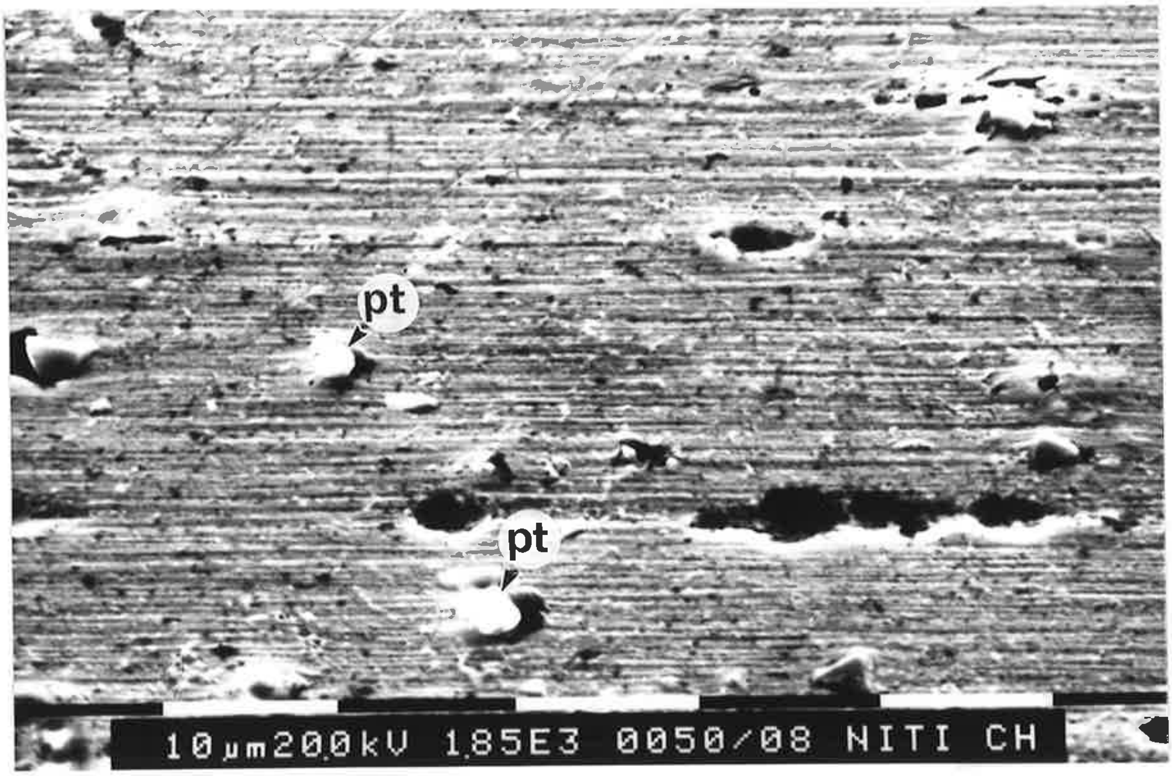
Transverse sections showed more numerous pits also with precipitate particles in them. **(Fig. 12b)**.

Microanalysis

The spectrum obtained is virtually identical to that produced by the Chinese NiTi wire showing the presence of only titanium and nickel (**Fig. 12c**). As before the analysis of the precipitates within the pits shows a higher proportion of titanium in relation to the amount of nickel which again suggests the presence of titanium oxides (**Fig. 12d**).

Fig. 11a A scanning electron micrograph of the longitudinal section of the Chinese NiTi wire showing that the wire was hardly attacked by the etchant. It shows a few separate and a few joined pits (P). It also shows the presence of precipitate (pt) particles in the pits. These particles are believed to be titanium dioxide.

Fig. 11b A scanning electron micrograph of the transverse section of the Chinese NiTi wire showing the presence of pits with precipitates (pt).


pt
pt
10µm20.0kV 1.85E3 0050/08 NITI CH

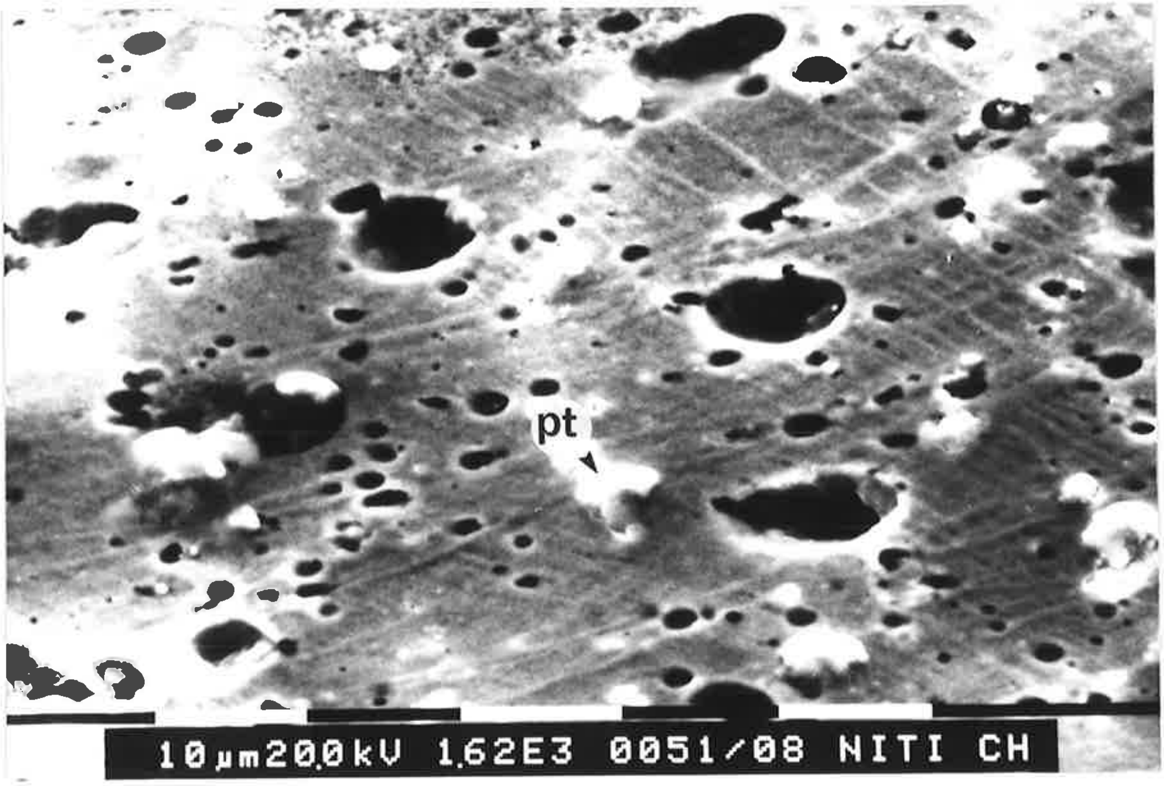
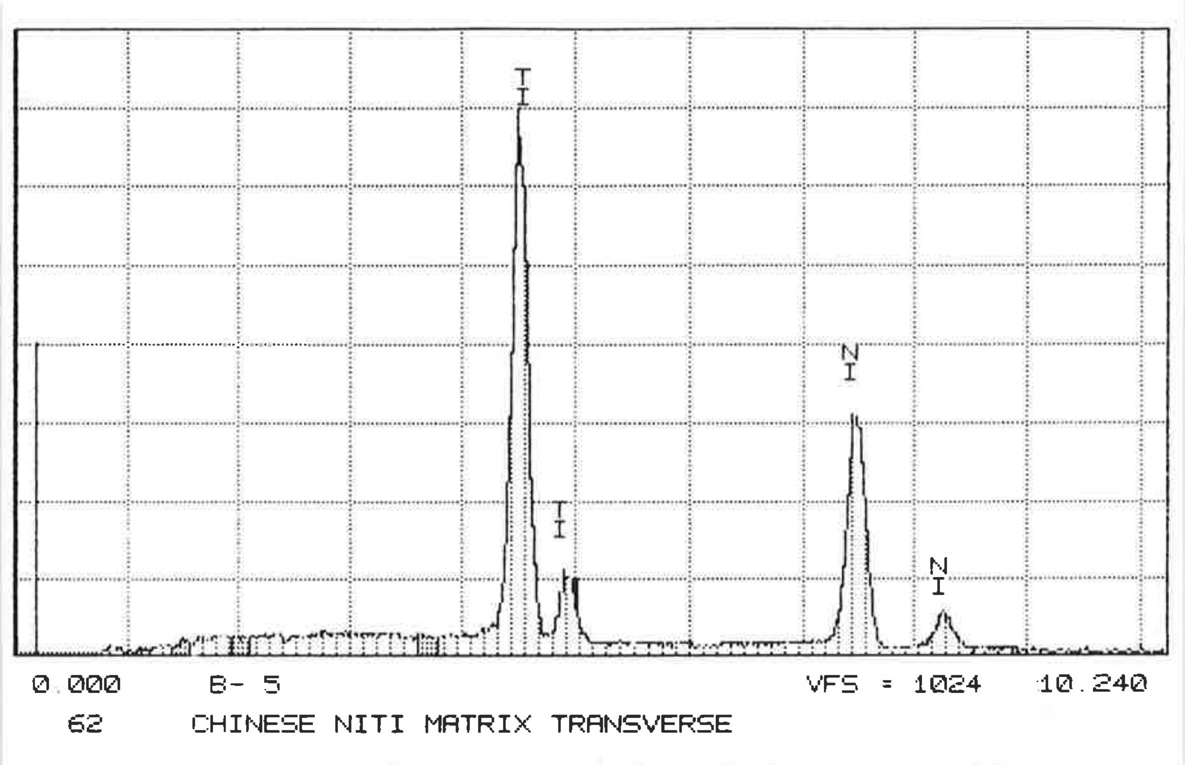

pt
10µm20.0kV 1.62E3 0051/08 NITI CH

Fig. 11c Energy dispersive X-ray spectrum of the matrix of the Chinese NiTi wire showing the presence of nickel and titanium.

Fig. 11d Energy dispersive X-ray spectrum of the precipitates of the Chinese NiTi wire showing higher proportion of titanium than nickel as compared to that of the matrix suggesting the presence of titanium dioxide.


TI
TI
NI
NI
0.000
B- 5
VFS = 1024
10.240
62
CHINESE NITI MATRIX TRANSVERSE

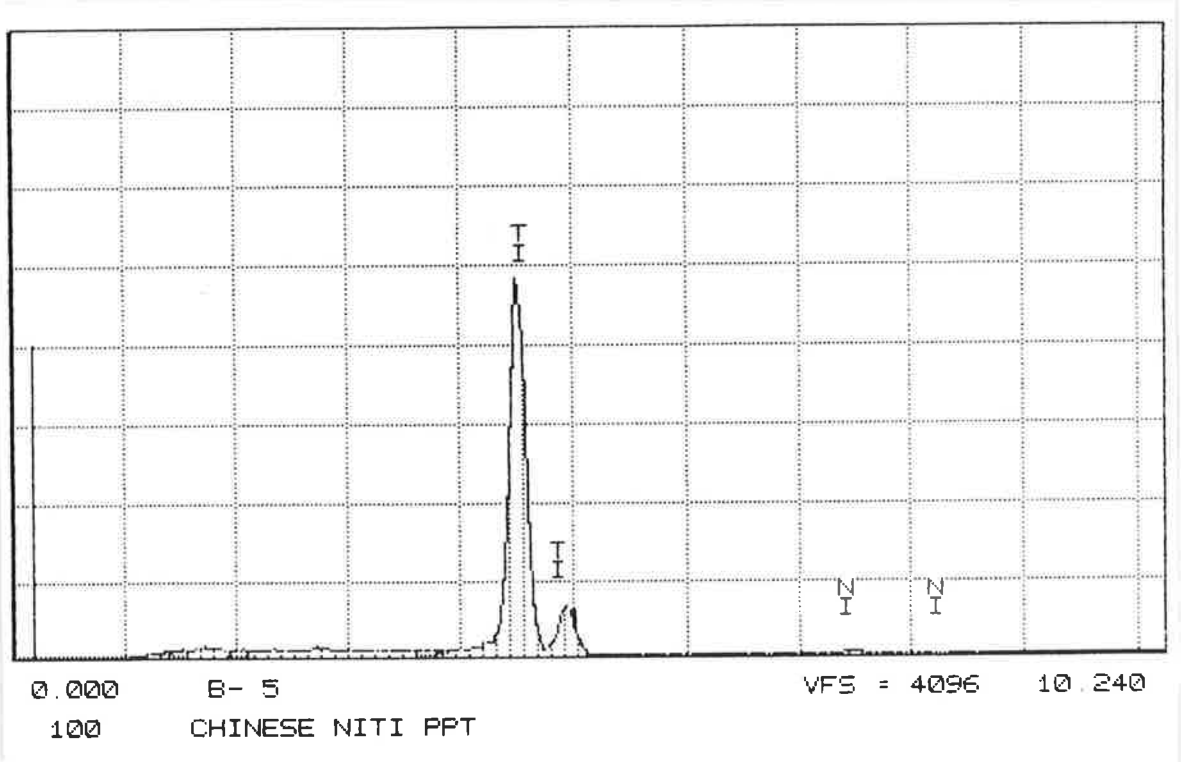
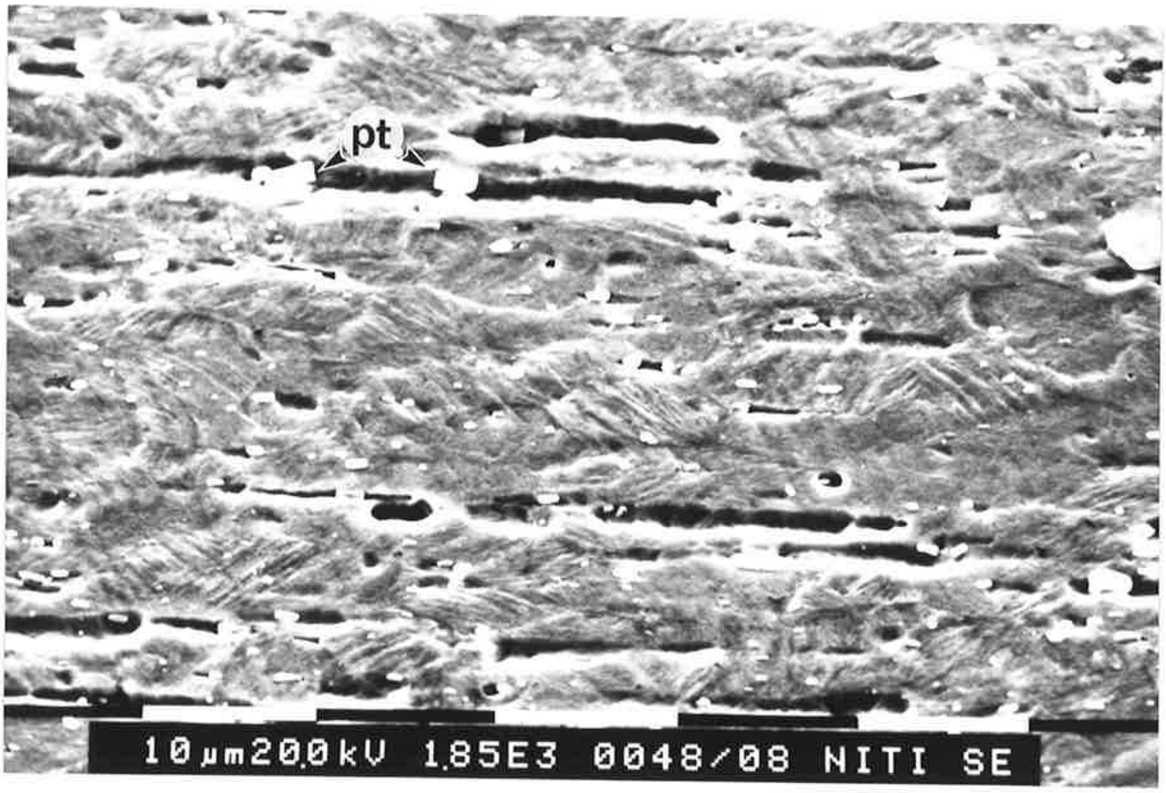

TI
TI
NI
NI
0.000
B- 5
VFS = 4096
10.240
100
CHINESE NITI PPT

Fig. 12a A scanning electron micrograph of the longitudinal section of the NiTi superelastic wire showing the presence of joined pits with white precipitates (pt) in them.

Fig. 12b A scanning electron micrograph of the transverse section of the NiTi wire showing the presence of pits with white precipitates (pt) in them. In comparison with the Chinese NiTi the pits in this wire are much smaller.


pt
10µm20.0kV 1.85E3 0048/08 NITI SE

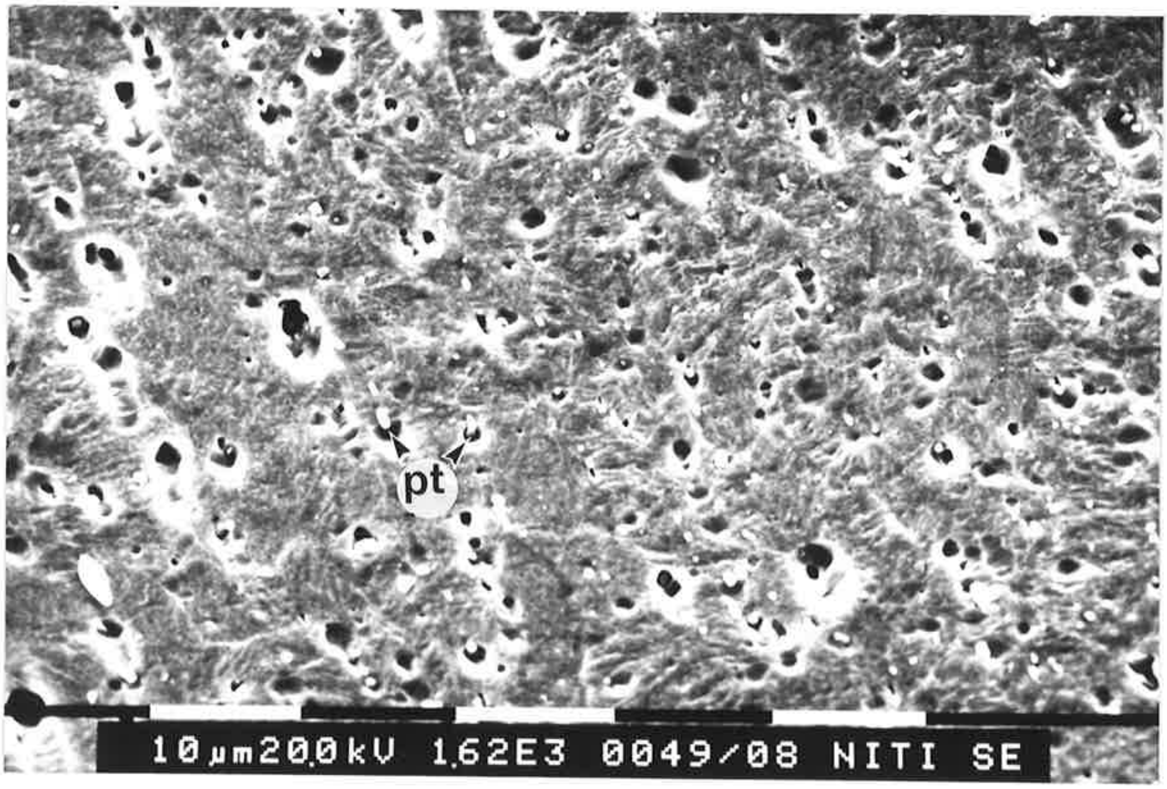
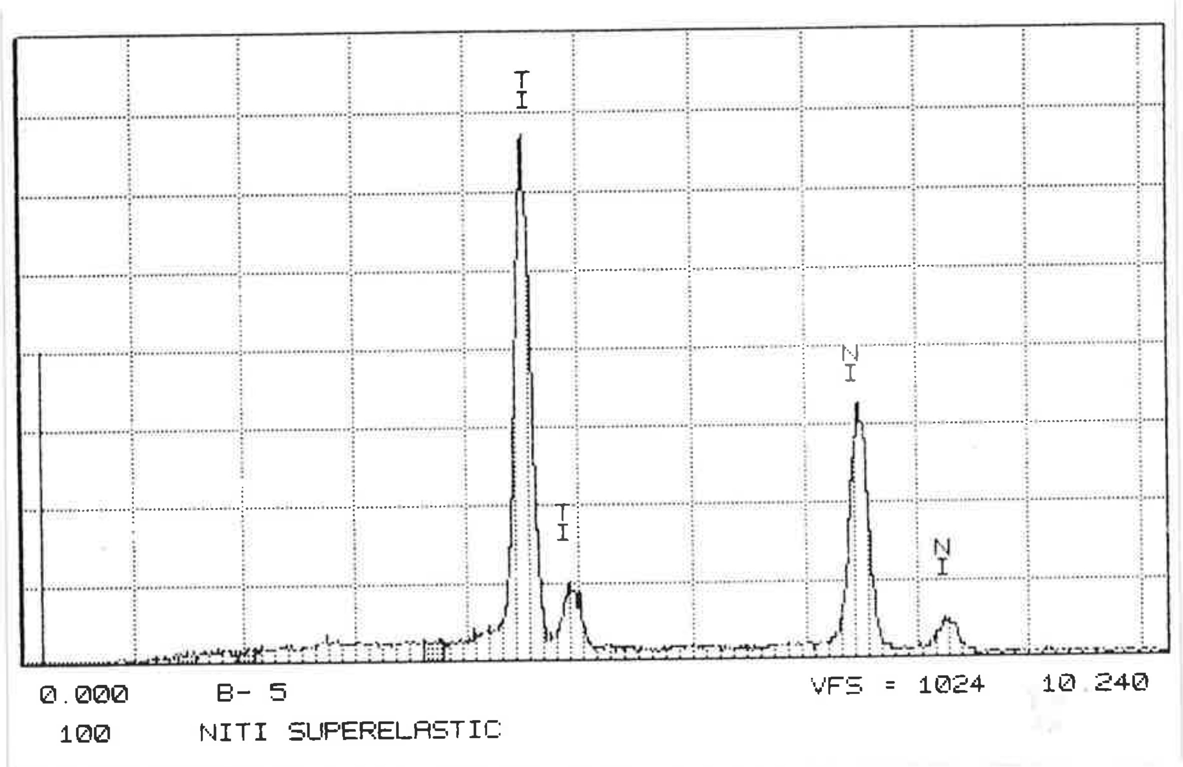
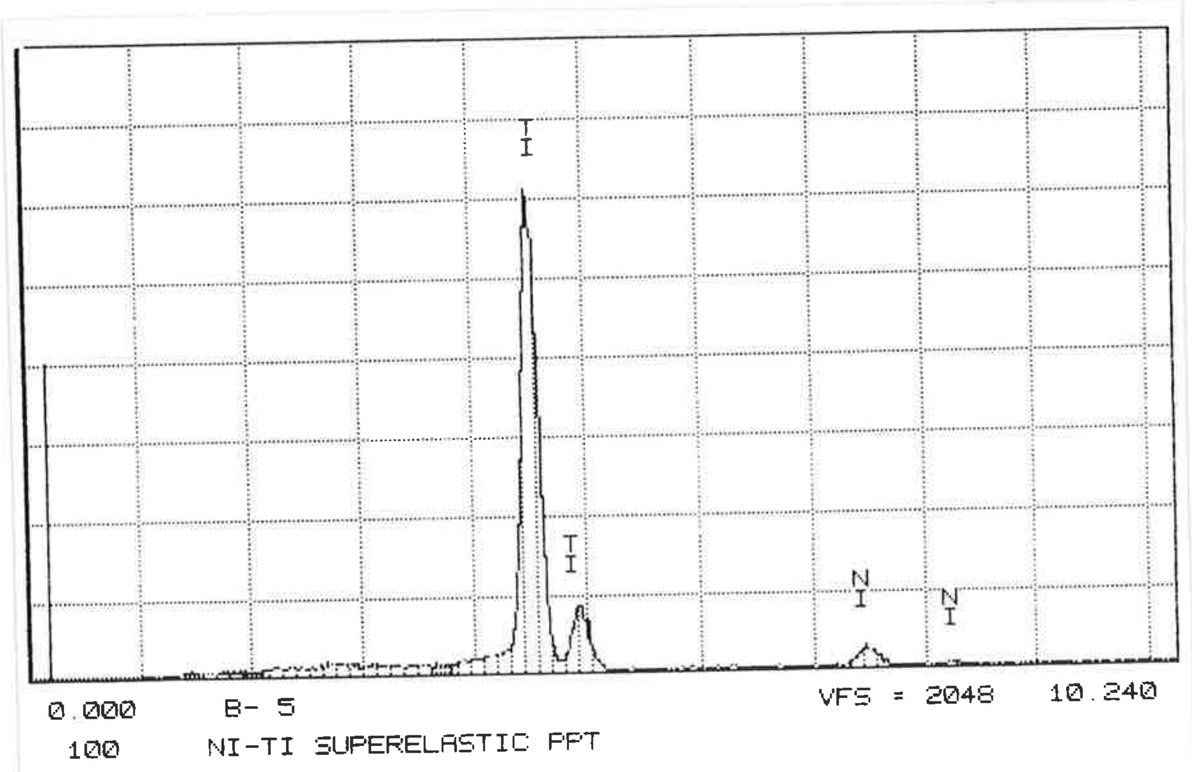

pt
10µm20.0kV 1.62E3 0049/08 NITI SE

Fig. 12c Energy dispersive X-ray spectrum of the matrix of the superelastic NiTi wire showing the presence of nickel and titanium.

Fig. 12d Energy dispersive X-ray spectrum of the precipitates of the superelastic NiTi wire showing a higher proportion of titanium than nickel as compared to that of the matrix.


TI
TI
NI
NI
0.000
B- 5
VFS = 1024
10.240
100
NITI SUPERELASTIC


TI
TI
NI
NI
0.000
B- 5
VFS = 2048
10.240
100
NI-TI SUPERELASTIC PPT

4.2.5 Discussion

A major difficulty in this part of the study was to find etching techniques which would clearly reveal the microstructures of the wires. Most of the standard chemical and electrolytic etches recommended for stainless steels and titanium alloys **(Vander Voort, 1984)** were not only tested as specified, but also manipulated in terms of etching time, temperature and applied voltage (where applicable).

Even the best reagent which was finally used for the wires had relatively limited success. There were frequent indications of striated structures, and differential etching effects were observed which could be associated with two-phase structures. However, virtually no detailed microstructural information was obtained which could be used to characterise each material (and eventually explain their mechanical properties).

In addition, the wires used in this study showed very different microstructural features compared to the published features of corresponding metals in other forms **(Metals Handbook, 1990)**. Moreover, there is very little published datum on the SEM microstructure of wires at all.

The stainless steel wires appeared to consist of two phases, one of which was more readily attacked by the etchant.

The observed striated structures in the longitudinal sections of wire are consistent with the elongation of a two-phase mixture in the direction of drawing during cold working. The greater concentration of these striations near the surface could also be predicted from the mechanics of deformation through a wire-drawing die. The greater number of closely spaced striations near the surface of the brittle sample may explain the frequent observation by orthodontists that when these wires break, the end of the broken wire almost always has a sharp edge which peels off from the other broken end. The difference in the number of striations, porosity and the presence of pits could also produce marked differences in mechanical properties between different wires.

In terms of failure of the wires, small well dispersed voids or pits or other phases are less deleterious than larger more concentrated defects. In the case of these stainless steels, the distribution of "pits" improves with increasing wire quality and this does correlate well with clinical experience.

Although the microstructure of the alpha-titanium wires could not be observed in detail, it also appeared to contain two phases. In addition, the X-ray microanalysis using energy dispersive spectrometer (EDS) showed the presence of titanium, aluminium, and vanadium, which is consistent with an alpha-beta alloy composition.

The beta-titanium revealed little of the actual microstructure. Cones and surface pits were observed and precipitates in the cones showed a much higher proportion of titanium when compared with the matrix. It is believed that these precipitates are titanium dioxide. This refractory oxide would be resistant to most etchants and therefore remain unattacked while the surrounding matrix dissolved.

The microanalysis of this alloy showed the presence of molybdenum, tin, and zirconium as substitutional alloying additions, which is consistent with a beta-forming composition.

In the NiTi wires, some fine striations were observed but again there were few obvious microstructural features.

The microanalysis of Chinese NiTi and the ordinary NiTi wires showed the presence of only titanium and nickel.

These materials also exhibited pits containing precipitates which had a higher proportion of titanium as compared with the matrix. This again suggests that titanium dioxide is present in the wire.

It is clear from the above that no definitive conclusions can be reached regarding the microstructures of these wires. It will become clear in the next section how fine the actual distribution of phases is in all these materials. It is relevant here then to consider the effects of etching on a fine-grained, especially duplex (or dual-phase) structure, when the grains are elongated in the longitudinal direction parallel to the wire axis.

Etchants in general may produce observable contrast in the light microscope or SEM by attacking different phases, or even different crystallographic orientations of the same phase, at different rates. However, chemical (or electrochemical) dissolution is usually more rapid at regions of higher energy. These include grain boundaries, phase boundaries, and areas of high dislocation density (which are produced during cold working). In a cold-worked, fine grained structure, the close proximity of the stress fields associated with grain boundaries and regions of high dislocation density may overlap and produce a general high energy region.

Under these conditions, uniform etching might be expected. At best, relatively indistinct striations representing small differences between high and higher energy areas might be produced, while even small numbers of heterogeneities such as non-metallic inclusions (which are unaffected by etching) will appear (perhaps erroneously) as major microstructural features.

It is suggested, therefore, that even scanning electron microscopy (SEM) is not a suitable technique for the study of the microstructure of orthodontic wires.

4.3 TRANSMISSION ELECTRON MICROSCOPY

4.3.1 Stainless Steel

Microstructure

Longitudinal sections of the stainless steel wires revealed a microstructure consisting mainly of long, narrow grains **(Fig. 13a)**. The average width of the grains was approximately 30nm. There are also quite complex variations in contrast across this specimen. Although this was not analysed in great detail it is clear that most are derived from the presence of a very high dislocation density within the grains. There was little existence of significant oxide formation.

In the transverse section, the grains appeared more circular or elliptical **(Fig. 13b)**. Again there was a range of apparent size (most probably due to sectioning effects), but the mean minimum dimension was again of the order of 30nm. Similar contrast variations were also observed, and also areas suggestive of a "swirled" pattern in the grains.

These observations are typical of the microstructure expected in heavily cold-drawn wire. Progress through each successive die reduces the wire diameter by deforming the original equiaxed grains into very elongated grains aligned in the drawing direction and a finer more circular morphology in the cross-section. The final very fine grain size found in this material suggests a fine initial grain size in the original material and/or a large reduction during cold-drawing (with few or no intermediate annealing treatments).

A typical electron diffraction pattern **(Fig. 13c)** shows rings containing many discrete diffraction spots. This is not representative of a heavily oxidised specimen, but occurs due to the pronounced texture produced by wire drawing, or by the superimposition of the regular spot patterns from a large number of fine grains in different orientations.

Indexing this pattern identifies the 3 major rings as being derived from the {011}, {002}. and {112} crystal planes of a BCC alpha-iron structure **(Fig. 13d)**.

This is a surprising result since the SEM study indicated that the material was most likely an austenitic A.I.S.I. TYPE 304 grade (with a FCC structure).

However, the presence of a considerable amount of a BCC phase was confirmed simply using a magnet, which was found to strongly attract all the stainless steel archwires tested.

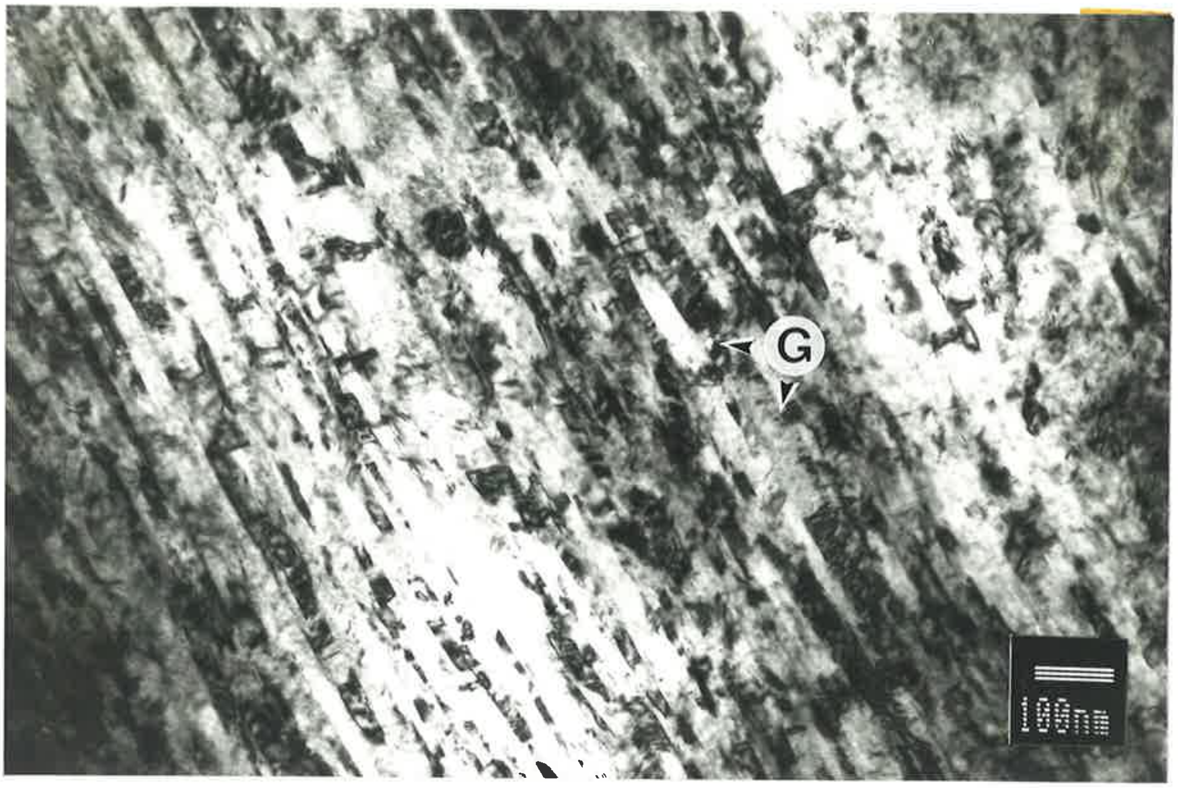
Another example of a diffraction pattern from a similar material is shown as **Fig. 13e**. Here again the major rings r_1, r_3, and r_5 **(Fig. 13f)** correspond with the previous {011}, {002}, and {112} planes of a BCC phase but the weak r_2, r_4 lines are consistent with {002}, and {022} planes of the FCC gamma phase. So in some areas small amounts of austenitic still exist and the structure is actually duplex (alpha+gamma) stainless steel.

These observations are consistent with a deformation-induced martensitic transformation, although austenitic stainless steels retain their stable FCC structure to extremely low temperatures with little or no tendency to form martensite, this behaviour can be changed when the material is strained. It has been shown that the gamma to alpha (martensite) transformation in 304 stainless steel can be induced by plastic deformation at room temperature **(Hecker, Stout, Staudhammer, and Smith, 1982)**. It is proposed here that wire-drawing process used in the manufacture of these wires does produce a significant amount of strain-induced transformation of gamma to martensite. This martensite, forming in a low carbon content alloy, will exhibit little tetragonality in its crystal structure, and appears in the diffraction patterns as a BCC structure.

It can be concluded then that archwires in the form they are used, actually consist of a heavily deformed duplex (or dual-phase) alpha+gamma structure, and their properties could best be considered in terms of a type of aligned composite structure.

Fig. 13a shows the presence of heavily dislocated and elongated grain structures of stainless steel. The cross-section of the grains (G) measure about 30 nm.

Fig. 13b shows the transverse section (TEM) of stainless steel wire. The grains (G) measuring 30nm in diameter appear circular or elliptical in shape.


G
100nm

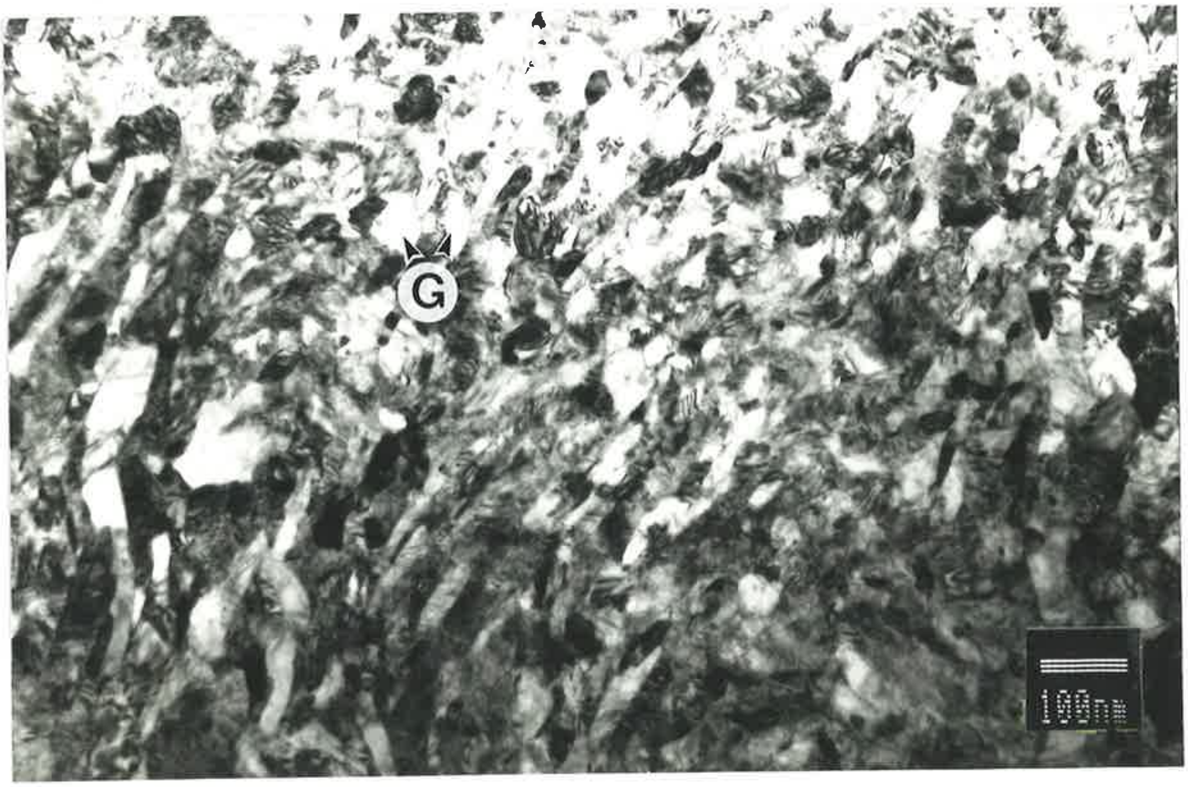
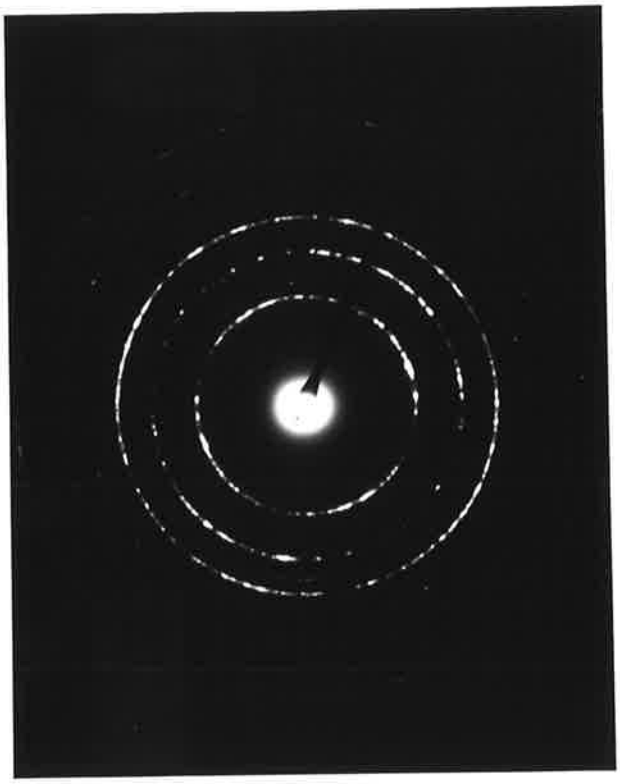
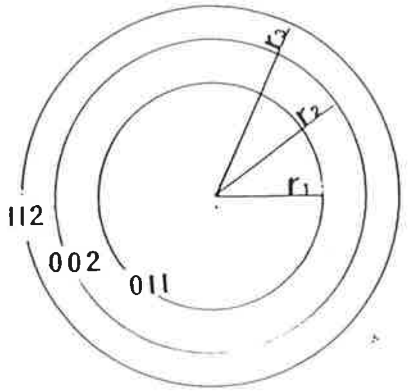

G
100nm

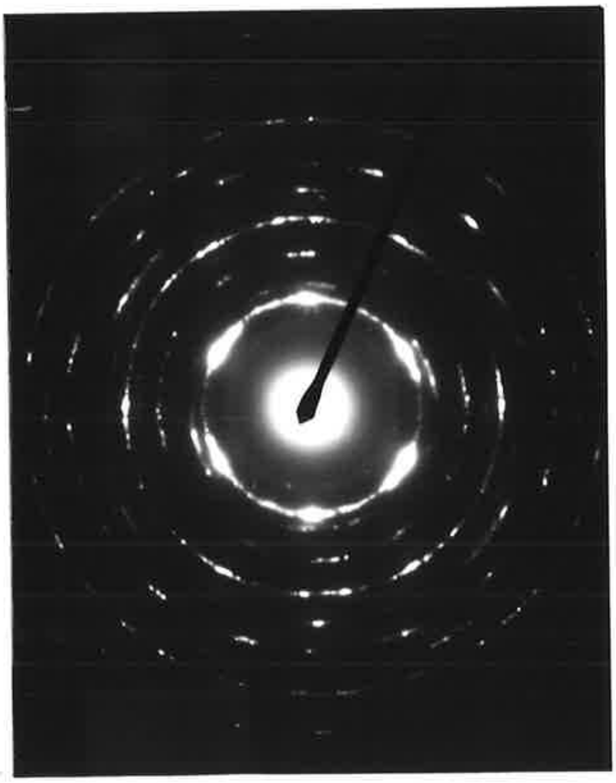
Fig. 13c The electron diffraction pattern shows rings containing many discrete diffraction spots. It indicates the presence of a metal of strong texture.

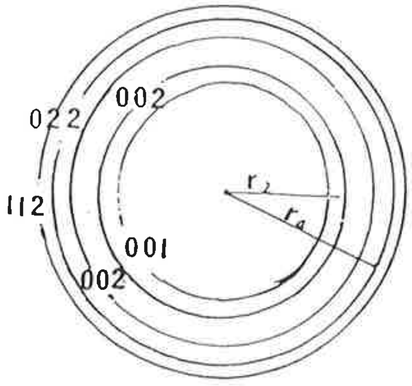
Fig. 13d Indexing of the diffraction pattern identifies three major rings derived from the BCC alpha-iron structure.

Figs. 13e and 13f Apart from the three major rings (r_1,r_3,r_5) of the BCC phases, the r_2 and r_4 rings indicate a gamma phase.




r3
r2
r1
112
002
011




022
002
r2
r4
112
001
002

4.3.2 Commercially Available Annealed Alpha-Titanium

An initial study was made of a commercial (Ti-6Al-4V) alloy in order to determine optimum electropolishing conditions for this material and to observe typical microstructures in this alloy.

Microstructure

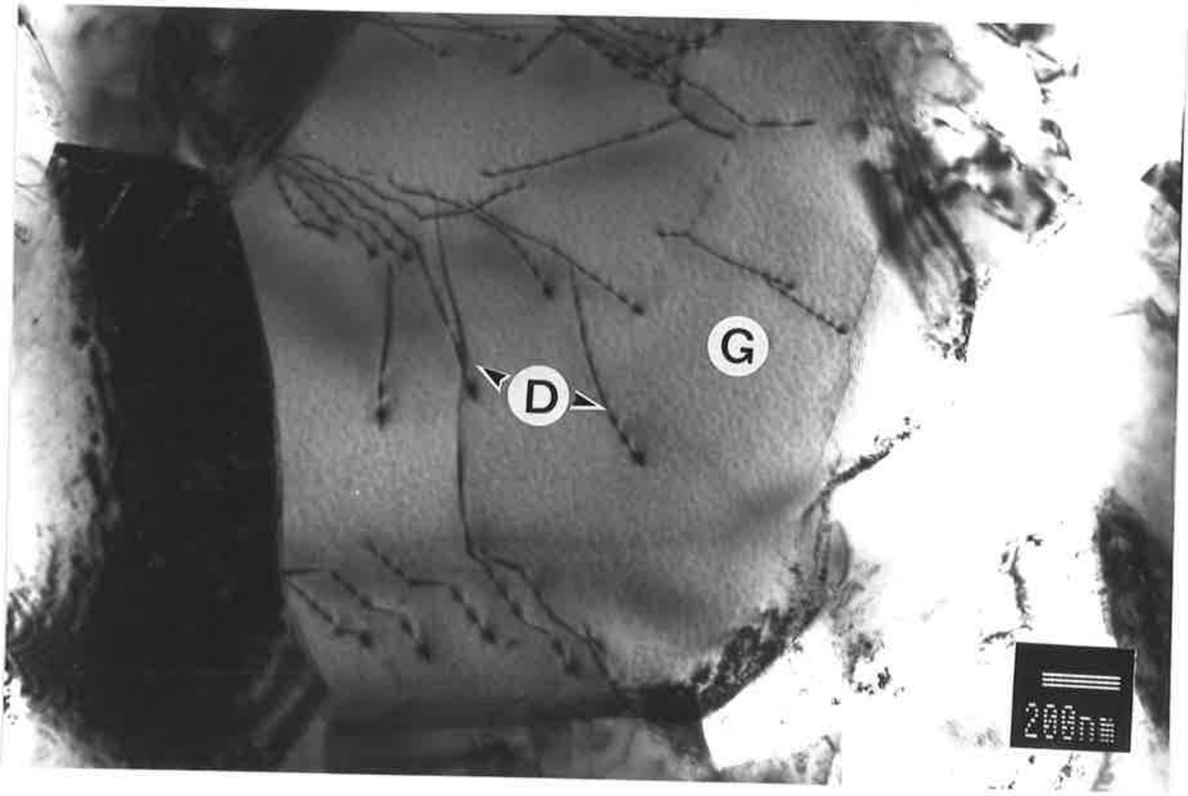
The microstructure of the annealed Ti-6Al-4V alloy is shown in **Fig. 14a**. It shows clean grains, containing a few individual dislocations, a fine grain size (approximately 2μm), and no evidence of any precipitates.

More relatively clean but finer grains (approximately 1μm) are shown in **Fig 14b**. Here most of the dislocations are arranged in regular networks, with coarse hexagonal and very fine rectangular dislocation arrays forming sub-grain boundaries.

A somewhat higher dislocation density can be seen in **Fig. 14c**, although most are still arranged in sub-grain boundaries. There is also some evidence of the elongated grain structure associated with the manufacture of rolled alloy plate from which this sample was obtained.

Fig. 14a A transmission electron micrograph of Ti-6Al-4V wire showing a fine clean grain (G) containing a few individual dislocations (D).

Fig. 14b A transmission electron micrograph of Ti-6Al-4V wire showing relatively clean grains (G) with no precipitates. The dislocations are arranged in regular hexagonal shaped networks (H).


D
G
200nm

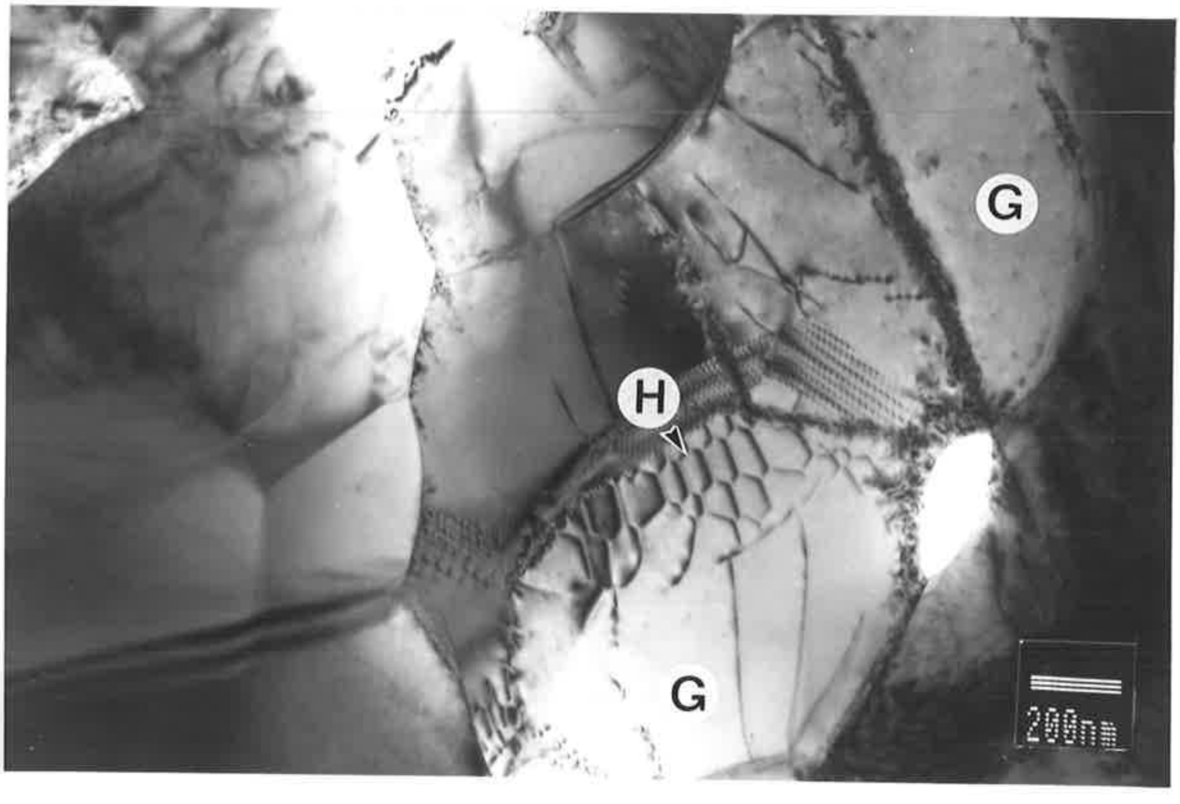
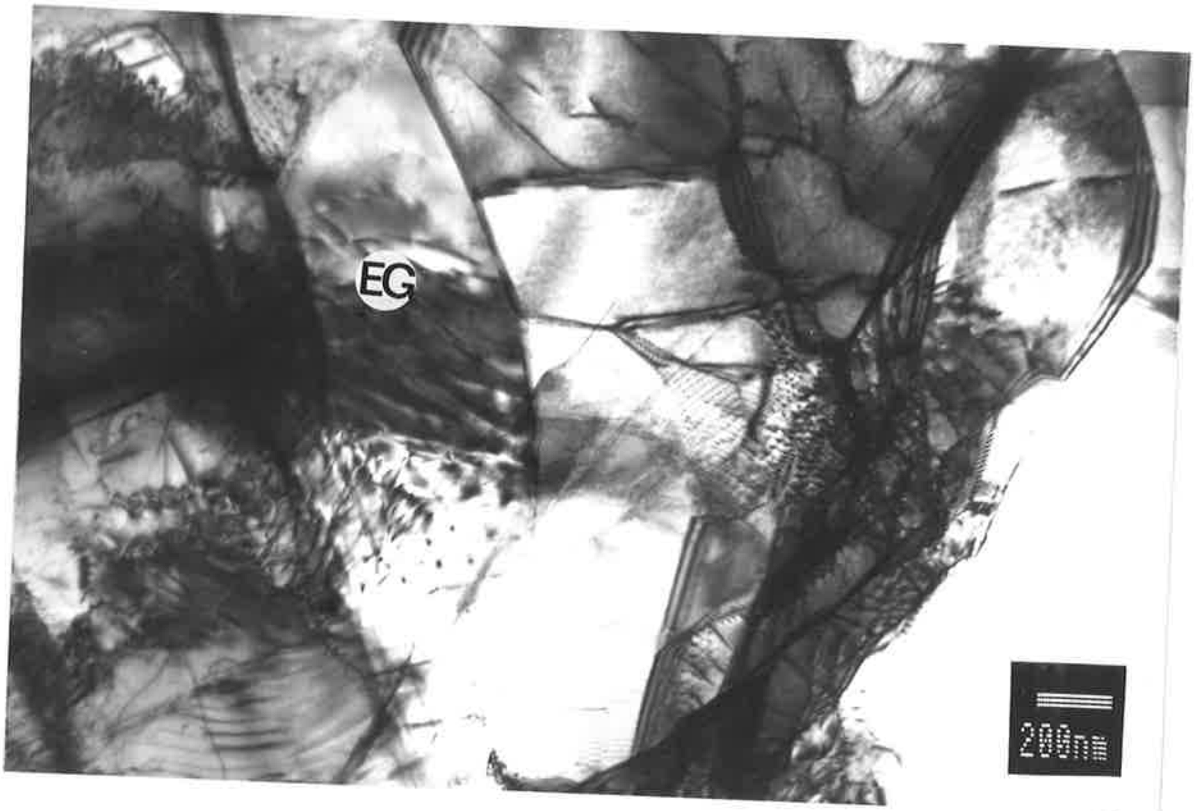

G
H
G
200nm

Fig. 14c A transmission electron micrograph of Ti-6Al-4V wire showing the presence of elongated grain structure (EG).


EG
200nm

4.3.3 Alpha-Titanium Wire

Microstructure

A micrograph taken from a longitudinal section is shown in **Fig. 15a**. In this area, the structure is predominantly plate-like, with areas of contrast typical of a high dislocation density. There appears to be a wide range of widths of approximately 250nm to almost 2μm for the large central plate. Again, this may just be a sectioning effect through "pancake"-shaped grains produced by deformation during manufacture. Within the larger plates there are also banded features (T). While there could be areas containing both alpha- and beta-phases, it is more likely that they are internal twins of the alpha-phase. These frequently arise in deformed HCP structures since because of the limited number of slip systems on which dislocations can be generated and then move, strain must be accomodated by alternative deformation mechanisms, such as twinning **(Love, 1985)**.

Another typical area, containing plates of different sizes is shown in **Fig. 15b**.

In the transverse sections, elliptical grains (with internal structures, also possibly twinning) up to 1-1.5μm in size were observed in a heavily dislocated matrix which showed some evidence again of internal twinning **(Fig. 15c)**.

A selected area diffraction pattern obtained from an area of the matrix containing only finer plates **(Figs. 15d and 15e)** revealed the presence of only the HCP alpha-titanium phase.

Another pattern however, obtained from the clearly defined elliptical grain in **Fig. 15c**, can be indexed in terms of the BCC beta phase **(Figs. 15f and 15g)**.

It can be concluded then that this material has a deformed duplex structure, consistent with an (alpha+beta) phase titanium alloy composition.

The observed microstructure is considerably coarser than that of the stainless steel (by a factor of at least fifty times). This could be explained by a number of factors:-

(a) the initial stock material from which the wire was drawn had a coarser structure,

(b) the starting material was smaller than the stainless steel stock and therefore required less drawing,

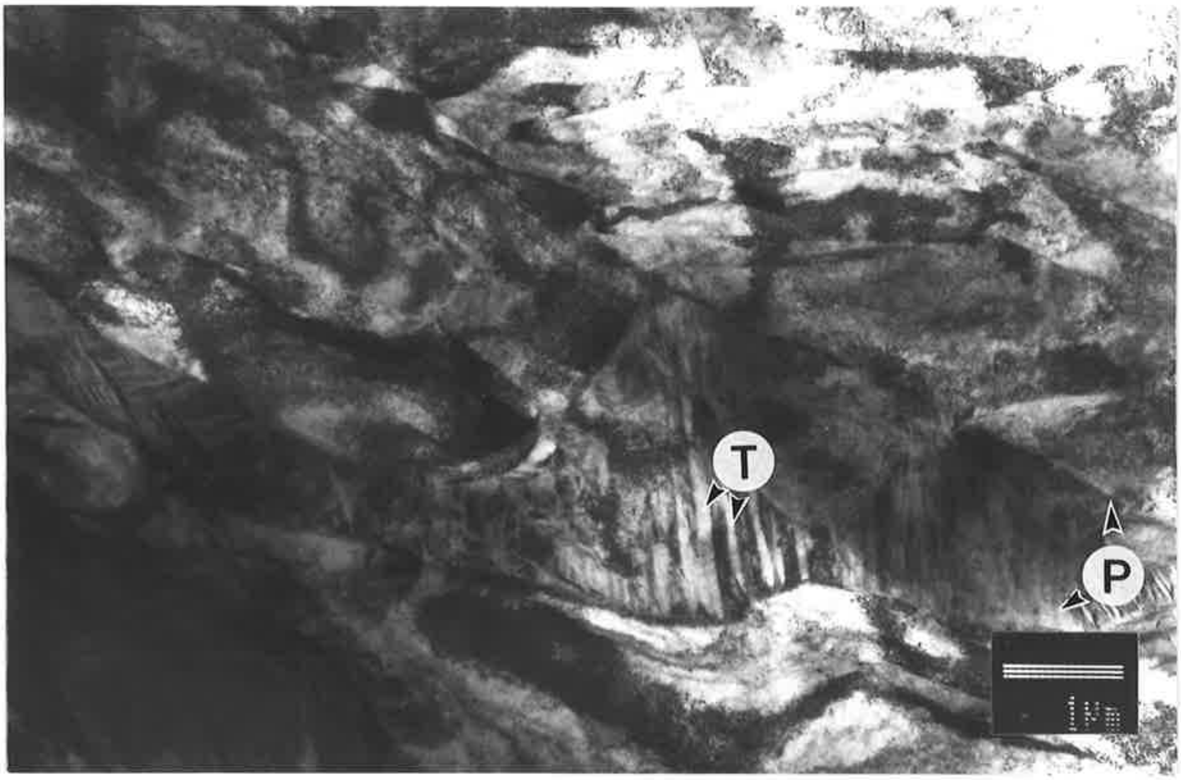
(c) intermediate anneals were applied to the material during the drawing sequence, or

(d) the material was partially annealed after drawing.

Although details of commercial practice are not generally available, (d) can be eliminated on the basis that there is no sign of recovery or recrystallisation processes which must precede grain growth and coarsening. While (a) and (b) are possible, it is thought that the large effect arises from some form of annealing treatment between drawing passes or sequences of passes, which will lead to a much smaller deformation in the final passes.

Fig. 15a The TEM micrograph of the longitudinal section of alpha-titanium wire shows heavily dislocated plates (P). The diameter of these plates range from 250nm to 2µm. Internal twinning (T) is seen in the large alpha plate.

Fig. 15b This TEM micrograph again shows plates of varying sizes. Twinning (T) is also present within the plates.


T
P
1µm

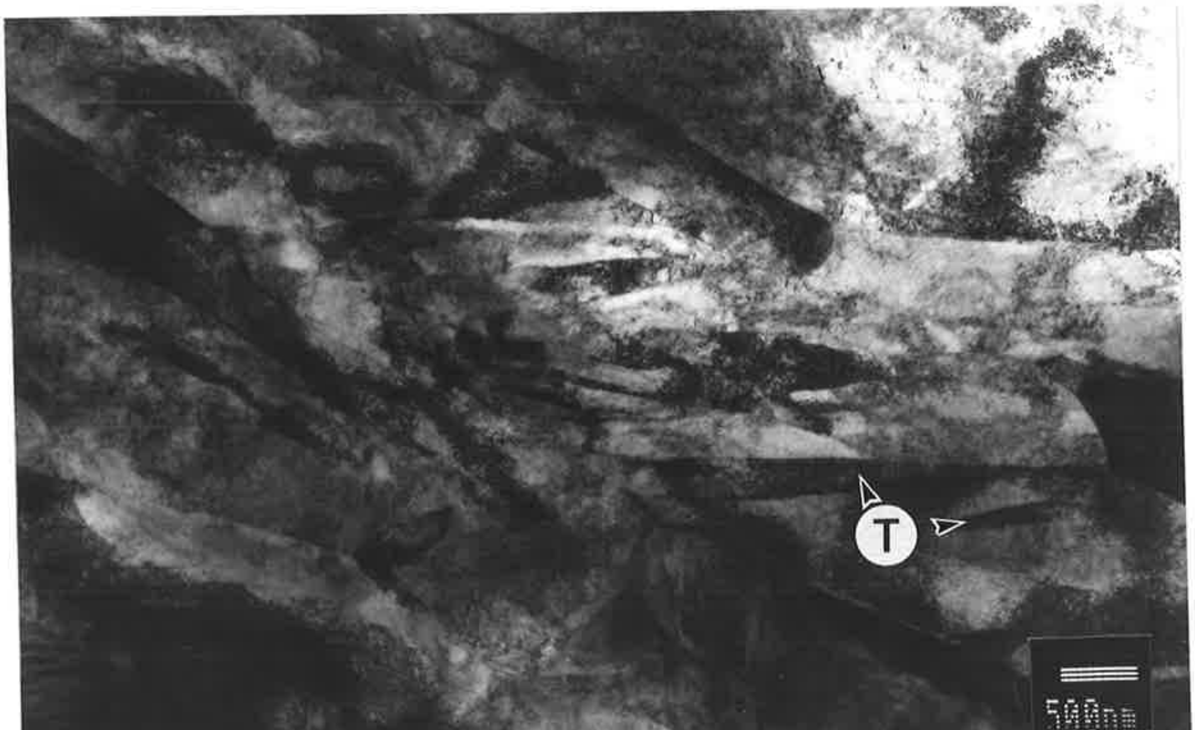
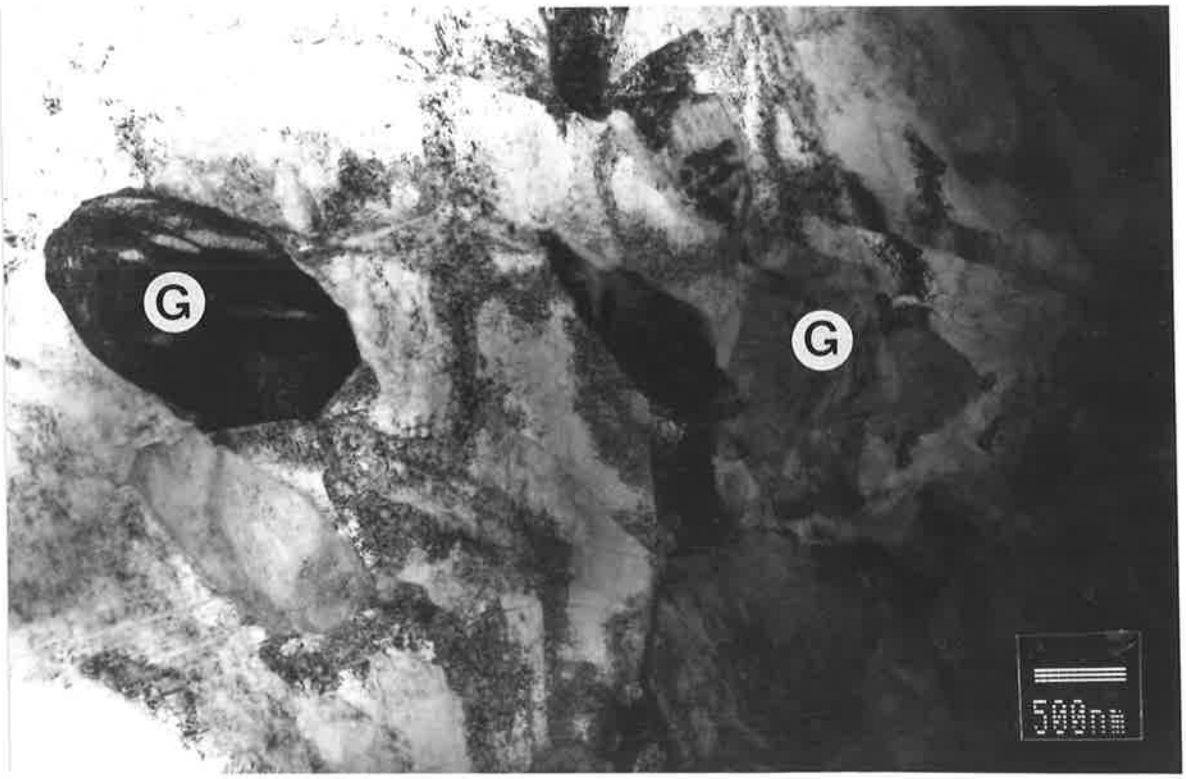
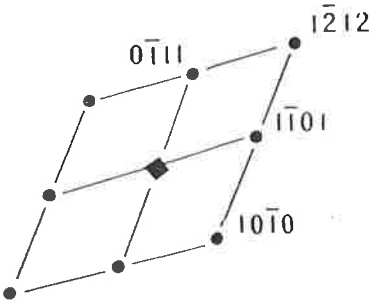

T
500nm

Fig. 15c The TEM micrograph shows a transverse section of the alpha-titanium wire. Elliptical grains (G) of varying sizes are seen.

Figs. 15d and 15e The electron diffraction pattern of a fine plate is seen in **Fig. 15e.** Indexing the pattern reveals the presence of only HCP alpha-titanium phase.



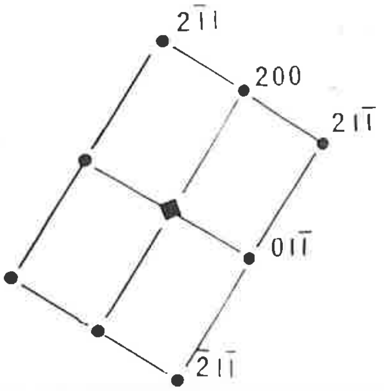

d



e

Figs. 15f and 15g The indexing of this selected area diffraction pattern shows the presence of a BCC beta-phase. The zone axis of the pattern is $[0\bar{1}1]$.

f



g

4.3.4 Alpha-Titanium Used in the Mouth

Microstructure

A preliminary study was made on the effect of the oral environment on the structure of alpha-titanium archwires. This was undertaken using transverse sections prepared from an archwire which had been in use in the mouth for a period of 2 months. It has been claimed that the properties of this material change **(Mollenhaur, 1989)** under these conditions, the wires becoming brittle with time.

The aim here was to determine if any obvious reason could be found for this behaviour, such as the development of oxide or hydride precipitates which would have the effect of strengthening the wire (through precipitation or dispersion hardening) but reducing ductility.

The structure of the used alpha-titanium is very similar to the unused, consisting of heavily dislocated/twinned alpha plates and smaller elliptical beta grains. There is no obvious evidence of any precipitate particles **(Fig. 16a).**

The diffraction pattern **(Fig. 16b)** from one plate reveals only a HCP alpha crystal structure.

It is not immediately clear why the {0001} and {0002} reflections are not visible. Further work is required to determine the structural factors or the effects of microstructural features (such as twins) which might cause these omissions. There is no clear evidence of any precipitates; additional unindexed spots all correspond to strong matric reflections.

It seems unlikely therefore, that oxide or hydride formation can explain the proposed changes in the mechanical properties of alpha-titanium archwires in use. This might be expected, given the following considerations;-

(a) Pure titanium is nontoxic; commercially pure titanium and some titanium alloys generally are biologically compatible with human tissues and bones.

(b) At human body temperature titanium is inert and has a extremely good corrosion behaviour. Due to this, titanium is used extensively

> in prosthetic devices such as heart-valve parts and load-bearing leg-bone replacements or splints or as implants to replace missing teeth. In general, body fluids have pH values from 7.4 into the acidic range that also contain a variety of organic acids and other components - media to which titanium is totally immune **(DONACHIE, 1988)**.

Titanium is really chemically active only at elevated temperatures when it will oxidise in air, and react with oxygen and nitrogen at the surface to produce an enriched layer called the "alpha case". Oxygen is the more potent since it is absorbed at a much greater rate than nitrogen. Alpha case is detrimental because of the brittle nature of the oxygen-enriched alpha structure.

Oxygen and nitrogen also have a potent effect on strength. As the amount of oxygen and nitrogen increases, the toughness decreases until the material eventually becomes quite brittle **(DONACHIE, 1988)**.

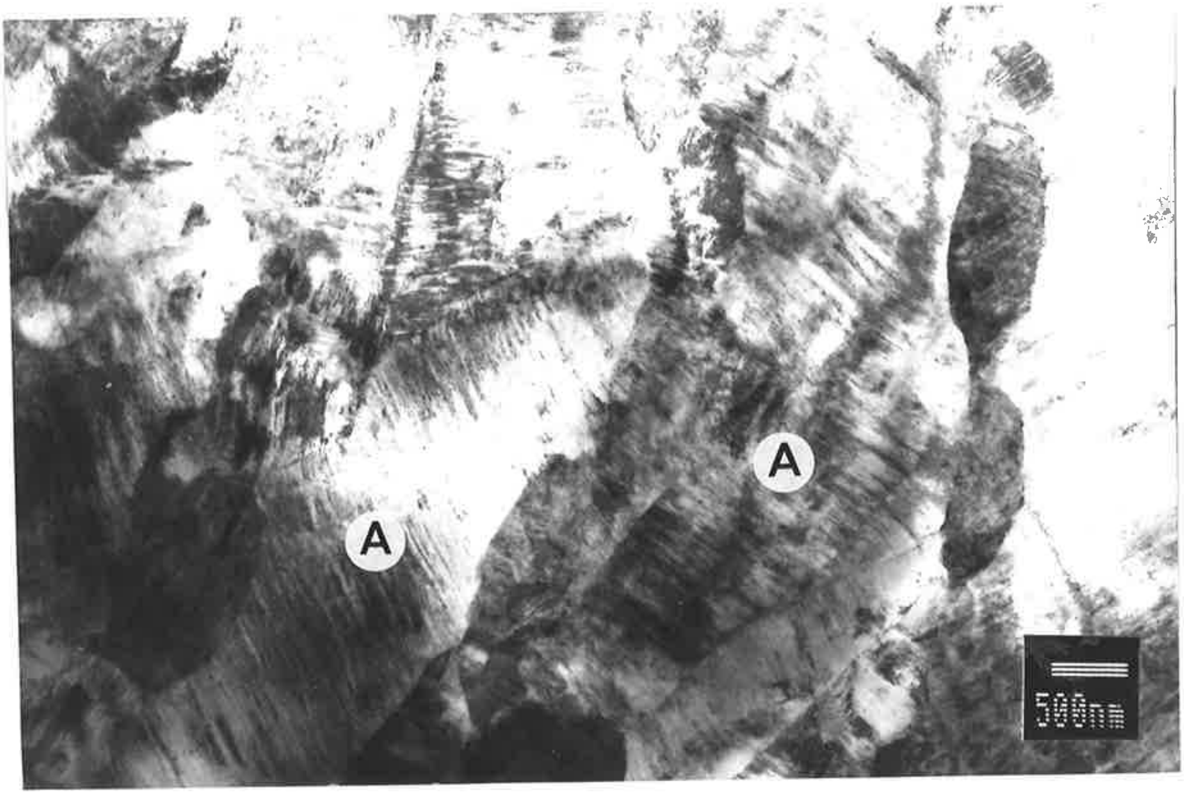
Titanium also reacts with hydrogen formed by the decomposition of water vapour. Unless the heat treatment is performed in a vacuum furnace or in an inert atmosphere, and unless surface cleanliness is maintained, there is a direct effect on the titanium's properties. Small amounts of hydrogen (100 to 200 ppm) can be tolerated in titanium alloys with the specific limiting amount determined by the type of alloy.

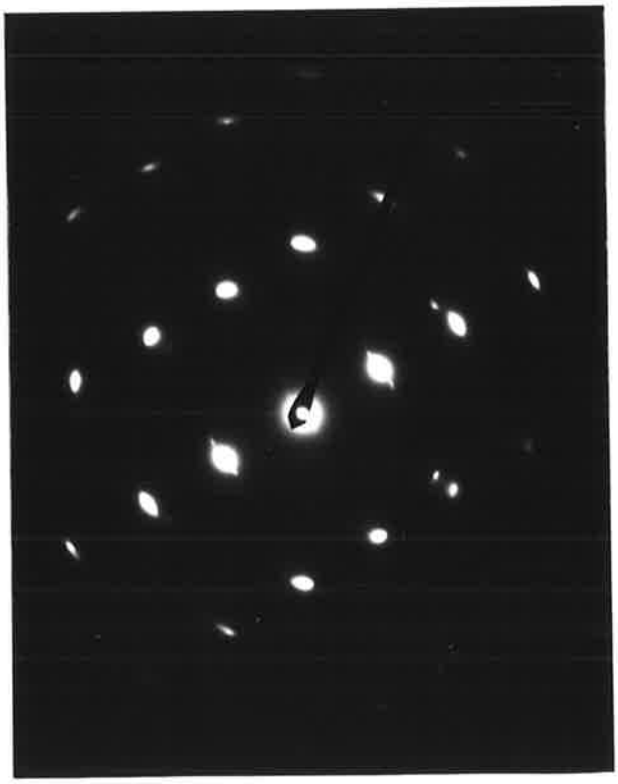
From this information it is not surprising that no precipitates were observed in the current study of wires used at ambient temperatures.

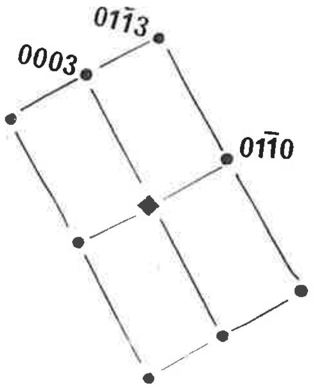
However, although hydrides were not present, it is possible that hydrogen diffuses from the oral cavity into the wire as free hydrogen atoms. This would not be detected with a TEM. An atom probe would be required for this. However, hydrogen in its free form has little effect on the mechanical properties. It is upon precipitation that ductility suffers **(POLMEAR, 1981)**.

Fig. 16a The TEM micrograph of used alpha-titanium wire shows heavily dislocated alpha plates (A). There is no evidence of any precipitates to indicate the presence of oxides or hydrides.

Fig. 16b The electron diffraction pattern from an alpha plate reveals the presence of HCP alpha crystal structure. The zone axis is $[2\bar{1}\bar{1}0]$.




b



c

4.3.5 Beta-Titanium

Microstructure

In longitudinal sections, the structure again consists of long, narrow, heavily dislocated grains **(Fig. 17a)**. These are intermediate in size between those of stainless steel and alpha-titanium, with widths of 50-100nm. The finer plate-like substructure is once more probably due to internal twinning, which is also a common form of deformation in BCC crystal structures.

The diffraction pattern **(Fig. 17b)** can be indexed in terms of two overlapping beta-titanium zones **(Fig. 17c)** and was therefore, obtained from an area including a high angle grain boundary.

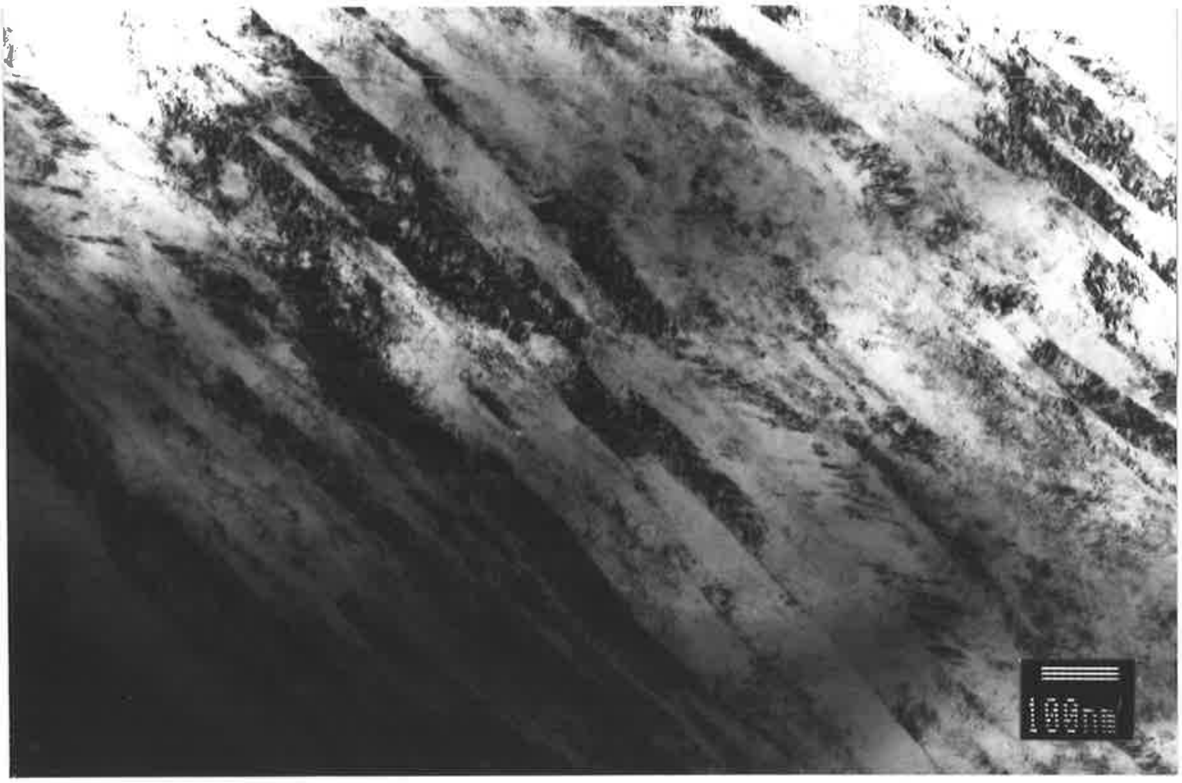
There is no evidence of a clear ring pattern nor extra precipitate spots which might be associated with oxides. The diffuse contrast in **Fig. 17a** therefore, does arise from the high dislocation density rather than a surface oxide film.

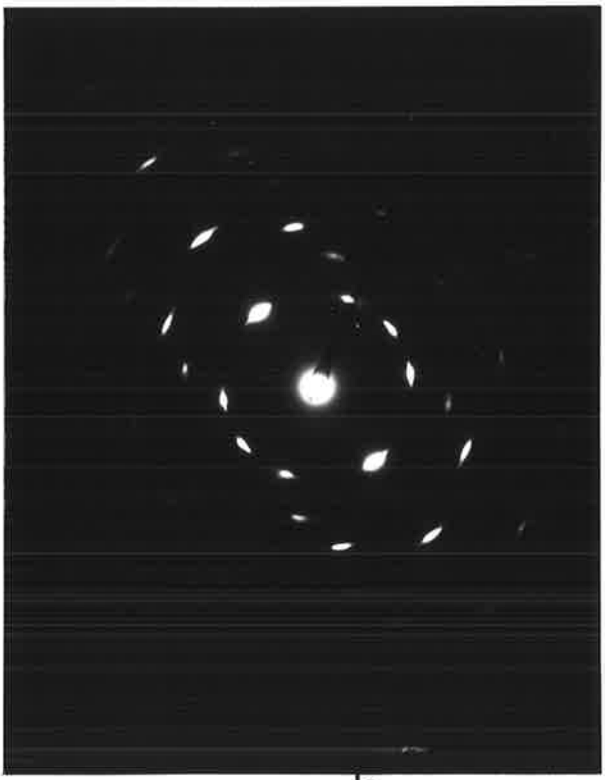
The transverse sections of this alloy reveal unexpected microstructure. In **Fig. 17d**, a high dislocation density and significant twinning can be readily seen, but the matrix grain structure is not clear.

Without a detailed history of the manufacturing process, it is not possible to draw definite conclusions regarding the development of this microstructure. However, it is possible that such a structure could be produced by a relatively small final reduction to size after an intermediate annealing process.

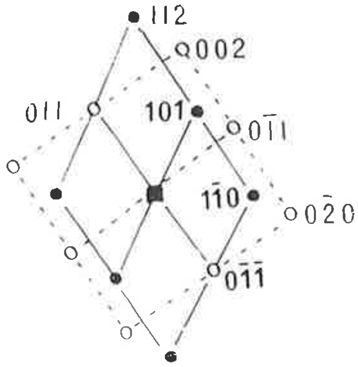
Fig. 17a The longitudinal sections of beta-titanium show long narrow, elongated grains. The grain diameter is between 50-100nm.

Figs. 17b and 17c The indexed diffraction pattern reveals two overlapping beta-titanium zones at a high grain boundary. The zone axis of is $[11\bar{1}]$ and zone axis of is [100].



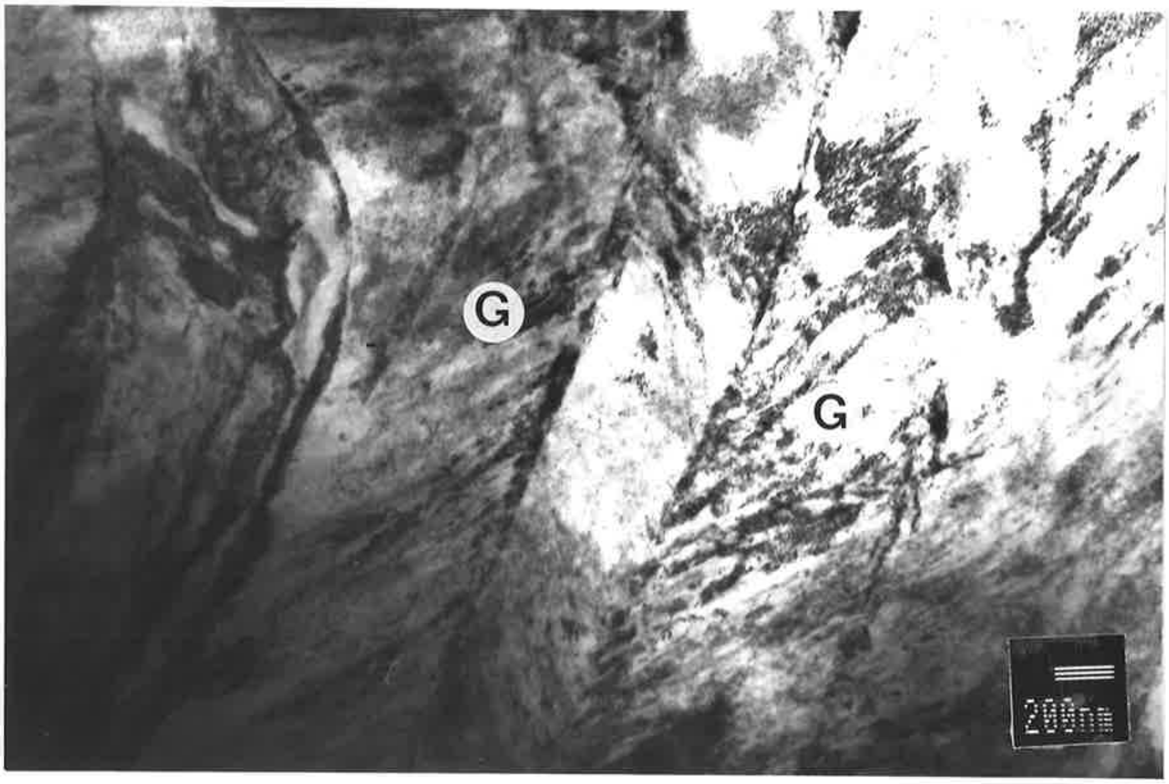


b



c

Fig. 17d The transverse section of beta-titanium shows highly dislocated grain structure with significant amount of twinning present.


G
G
200nm

4.3.6 Discussion

All of the alloys studied using transmission electron microscopy exhibited microstructures characteristic of metals which had been heavily cold worked to produce wire. This led generally to elongated, thin grains in the longitudinal direction (the wire axis) with more equiaxed grains in the transverse direction.

The noted differences in scale between features common to all the materials (e.g. grain size) can be explained to some extent in terms of possible variations in starting material or detailed manufacturing procedures. No detailed conclusions can be drawn however, without access to (confidential) commercial information.

Nevertheless, it is most important to note here the properties of metals are determined largely by their microstructures and that a fine grain size in particular is unique in imparting both high strength and improved toughness (resistance to fracture).

It would be interesting to determine the changes in the properties of the titanium wires when produced with finer microstructures similar to the stainless steel wire. With grain refinement and, possibly improvement of grain shape, the properties of the titanium wires could be improved. With its other advantages over stainless steel (60% of the density and 55% of the modulus of stainless steel, and greater corrosion resistance), the use of the titanium archwires could be extended in orthodontic treatment.

However, the stainless steel wires themselves could also probably be improved and their applications extended. Manipulation of processing parameters can change not only grain size and dislocation density but also the relative proportions of martensite- and gamma-phases, with the potential to produce major changes in mechanical properties.

It is quite clear that very little is known concerning the detailed microstructures of orthodontic wires.

Conventional techniques (including SEM) do not have the resolution to provide useful microstructural data. It is vital therefore, to undertake more TEM studies. The current work has made an initial contribution in obtaining some basic information. It is hoped that this can be built on with more extensive studies. These will not only allow the understanding of the mechanical behaviour of current materials, but also permit the development of new and better wires, based on sound scientific principles.

CHAPTER 5

CONCLUSIONS

The main purpose of this study was to determine the mechanical properties of alpha-titanium orthodontic wires and to study in detail the microstructure of orthodontic wires.

The findings are as follows:-

(1) The mechanical properties determined for the alpha-titanium wires are considered unreliable, due to the inadequacy of the available testing machine. While values for the yield strength, 0.2% proof stress and tensile strength were comparable with some values in the literature, they were not consistent in, for example, the yield strength/tensile strength ratio. However, it should be noted that there are no data available on heavily drawn fine wires.

The modulus of elasticity was consistently low compared with published values, but similar to that reported by **Hazel, Sokel, and West (1984)**.

(2) Scanning Electron Microscopy revealed little useful information concerning the microstructure of orthodontic wires. Despite using a vast range of chemical and electrochemical etchants, under many different conditions, very little contrast could be produced in the wire samples.

Some indication was obtained of an unexpected dual-phase structure in the supposedly austenitic stainless steel through differential etching. The distribution of these phases became finer and more uniform with increasing wire quality. Striations in the direction of the wire axis were also observed, and these could be associated with the brittleness sometimes found in the alloys.

The alpha-titanium alloy also appeared two-phase through differential etching. Microanalysis showed the presence of aluminium and vanadium as substitutional solute additions, consistent with an alpha+beta Ti-6Al-4V type alloy.

Beta-titanium and the NiTi alloys proved very difficult to etch at all. They provided little microstructural information. However, all of them were found to contain titanium-rich particles which were probably oxides produced during manufacture. These are probably deleterious to wire properties.

It is concluded that SEM is not a suitable technique for the study of fine microstructures in orthodontic wires.

(4) Transmission electron microscopy gave valuable microstructural information. While all the alloys studied had elongated, narrow grains in the drawing direction, and a more equiaxed transverse microstructure, there were significant differences in the scale of each structure. The mean grain diameter (transverse) or grain width (longitudinal) was approximately 30nm in the stainless steel, 1500nm in the alpha-titanium, and 80nm in the beta-titanium. These are thought to be due to differences in the manufacturing practice.

Diffraction experiments confirmed the presence of

(a) alpha- and gamma-phases in the stainless steel (due to a deformation-induced martensite transformation),

(b) alpha- and beta-phases in the alpha titanium confirming an alpha+beta alloy, and

(c) only beta-phase in the beta-titanium.

(5) No evidence of oxide or hydride formation was found in the alpha-titanium which had been used in the mouth.

(6) The detailed microstructural characterisation of these alloys will lead to a deeper understanding of the mechanical properties of each of these materials. Hopefully, they will also allow modifications to be made to the manufacturing system to provide improved properties, or tailor properties for a specific purpose. In this way the range of applications for all the wires could be extended.

CHAPTER 6

FUTURE WORK AND CONCLUDING REMARKS

FUTURE WORK

(1) Mechanical testing should be performed using a calibrated, high accuracy extensometer and load cell on more sophisticated equipment, such as a servohydraulic testing machine

This programme should include not only alpha-titanium, but also other stainless steel, beta-titanium and NiTi wires in order to provide a wider database of the mechanical properties of a range of alloys in fine wire form.

Mechanical testing of the wires should also be extended to determine other properties, such as resilience and springback characteristics.

(2) Even more detailed TEM investigations should be carried out on the materials used in this study, and the work extended to other orthodontic wires. This will build up a database of microstructural information which will be invaluable in understanding mechanical properties, and changing them for particular orthodontic applications.

(3) Microanalytical data should also be obtained from the individual phases in each alloy, to provide greater detailed information and understanding of these materials.

(4) Efforts should be made to ascertain if mechanical properties of alpha-titanium do in fact change through exposure to an oral environment (over different periods of time), and if so further attempts should be made to determine the mechanisms (precipitate formation or otherwise) by which this occurs.

(5) The potential to change mechanical properties in orthodontic wires through changes in manufacturing methods should be assessed. This can only be

done in collabration with a wire producer. However, initial data could be obtained by simply deforming and/or heat treating existing wires in the laboratory and re-examining their mechanical properties.

CONCLUDING REMARKS

Titanium metallurgy grew out of the application of titanium to gas turbines. From there the metal was applied to airframes. Within a relatively short history of development, the record of accomplishment has been remarkable.

Newer titanium alloys like the beta alloys such as Ti-10V-2Fe-3Al and Ti-15V-3Cr-3Al-3Sn are now available. They offer an attractive alternative to the alpha-beta alloys because of their increased heat treatability and their formability characteristics **(DONACHIE, 1988)**.

Titanium has also found applications in dentistry especially in the manufacture of dental implants and orthodontic wires. Osseointegrated titanium implants are now widely used to provide anchorage for the full range of tissue integrated prosthetic rehabilitation, ranging in complexity from the missing single tooth through to the extensive maxillofacial defect causing loss of the entire dentition and adjacent facial parts.

Higher-modulus alloys may become significant in the future. Titanium has a low modulus relative to its density. It is about 50% of the modulus of steel, but about 60% of the density of steel. The modulus is, to a degree, also a function of crystal orientation. Thus there is a hope that a practical process could permit the attainment of higher moduli without a corresponding loss of the strength properties. With these advances in titanium, newer and better titanium alloys will be available for orthodontic use.

In the future, additional progress in orthodontic arch materials, including composite plastic "wires" as well as new metal alloys, can also be anticipated.

Developmental work has proceeded with tooth-coloured ceramic brackets that are dimensionally accurate and stable as steel. It is possible, and indeed probable, that research in this direction will eventually provide successful tooth-coloured attachments. Tooth-coloured wires will also be needed at that point, and it may eventually be possible to achieve this goal by using coatings on steel or titanium wires, or by using archwires made from high-strength plastics **(PROFFIT, 1986)**.

One of the shape memory plastics named polynorbornen was developed in Japan, in 1983. This polymer has a molecular mass of over 3 million. It has much the same characteristics seen with shape memory alloys. The transition point ($35^{O}C$) is between that of conventional plastic and rubber. As it is nearly tooth coloured and can be moulded to an arch-shaped wire, the shape memory plastic archwire, combined with ceramic brackets, will be less noticeable than conventional orthodontic appliances. However, the plastic archwire, at this stage, is ineffective in controlling dental arch width, expanding the dental arch, opening the bite, and torquing teeth **(NAKASIMA, REN HU, ICHINOSE, AND SHIMADA, 1991)**.

Nevertheless, from the current study it is clear that there are further possibilities for the existing alloys with the potential for extension of applications for both stainless steel and titanium wires. This could be achieved by manipulation of the manufacturing process to produce tailored microstructures with specific or superior mechanical properties.

CHAPTER 7

BIBLIOGRAPHY

ANDREASEN, G.F. AND BARRET, R.D.(1970)
An evaluation of cobalt-substituted nitinol wire in orthodontics.
Am. J. Orthod. 63 : 462-470.

ANDREASEN, G.F. AND HILLMAN, T.B.(1971)
An evaluation of 55 cobalt substituted nitinol wire for use in orthodontics.
Am. Dent. Assoc. 82 : 1373-1375.

ANDREASEN, G.F. AND MORROW, R.E. (1978)
Laboratory and clinical analysis of nitinol wire.
Am. J. Orthod. 73 : 142-152.

BARROWES, K. J. (1982)
Archwire flexibility and deformation.
J. Clin. Orthod. 16 : 803-811.

BEDNAR, J.R., GRUENDEMAN, G.W., AND SANDRIK, J.L. (1991)
A comparative study of frictional forces between orthodontic brackets and arch wires.
Am. J. Orthod. Dentofac. Orthop. 100 : 513-522.

BEE, J.V. (1991)
New instruments complete world-class facility at The University of Adelaide.
Lumen. The University of Adelaide News Magazine.
20 (16) : 1

BROWN, I.H. (1991)
Private Communication.
Dept. of Mechanical Engineering. University of Adelaide.

BUCKTHAL, J.E. AND KUSY, R.P. (1988)
Effects of cold disinfectants on the mechanical properties and the surface topography of nickel-titanium arch wires.
Am. J. Orthod. Dentofac. Orthop. 94 : 117-122.

BUCKTHAL, J.E., MAYHEW, M.J., KUSY, R.P. AND CRAWFORD, J.J. (1986)
Survey of sterilization and disinfection procedures.
J. Clin. Orthod. 20 : 759-765.

BURSTONE, C.J. AND GOLDBERG, A.J. (1980)
Beta titanium : A new orthodontic alloy.
Am. J. Orthod. 77(2) : 121-132.

BURSTONE, C.J. (1981)
Variable-modulus orthodontics.
Am. J. Orthod. 80(1) : 1-16.

DONACHIE, M.J. (1988)
Titanium. A Technical Guide.
Ohio, ASM International.

DRAKE, S.R., WAYNE, D.M., POWERS, J.M., AND ASGAR, K. (1979)
Mechanical properties of orthodontic wires in tension, bending, and torsion.
Am. J. Orthod. Dentofac. Orthop. 82 : 206-210.

EDIE, J.W., ANDREASEN, G.F. AND ZAYTOUN, M.P. (1981)
Surface corrosion of nitinol and stainless steel under clinical conditions.
Angle Orthod. 51 : 319-324.

FILMORE, G.F., AND TOMLINSON, J.L. (1976)
Heat treatment of cobalt-chromium alloy wire.
Angle Orthod. 46 : 187-195.

GARNER, L.D., ALLAI, W.W. AND MOORE, B.K. (1986)
A comparison of frictitional forces during simulated canine retraction of a continuous edgewise arch wire.
Am. J. Orthod. Dentofac. Orthop. 90 : 199-203.

GIANCOLI, D.C. (1988)
Physics For Scientists And Engineers.
New Jersey, Prentice Hall : 277-279.

GOLDBERG, A.J., AND BURSTONE, C.J. (1979)
An evaluation of beta-titanium alloys for use in orthodontic appliances.
J. Dent. Res. 58 : 593-600.

GOLDBERG, A.J., MORTON, J., AND BURSTONE, C.J. (1983)
The flexure modulus of elasticity of orthodontic wires.
J. Dent. Res. 62 : 856-858.

HARRIS, E.F., NEWMAN, S.M. AND NICHOLSON, J.A. (1988)
Nitinol arch wire in a simulated oral environment : changes in mechanical propoerties.
Am. J. Orthod. Dentofac. Orthop. 93 (6) : 508-513.

HAZEL, R.J., SOKEL, B.N. AND WEST, V.C. (1984)
Near-alpha titanium - a potentially new orthodontic archwire material.
Aust. Orthod. J., 8 : 90-91.

HECKER, S.S., STOUT, M.G., STAUDHAMMER, K.P. AND SMITH, J.L. (1982)
Effects of strain state and strain rate on deformation-induced transformation in 304 stainless steel: Part 1. Magnetic measurements and mechanical behavior.
Metall. Trans. 13A : 619-626.

INGRAM, S.B., GIPE, D.P. AND SMITH, R.J. (1986)
Comparative range of orthodontic wires.
Am. J. Orthod. Dentofac. Orthop. 90 : 296-307.

JONES, M.L., STANIFORD, H. AND CHAN, C. (1990)
Comparison of superelastic NiTi and multistranded stainless steel wires in initial alignment.
J. Clin. Orthod. 24 (10) : 611-613.

KAPILA, S., REICHHOLD, G.W., ANDERSON, R.S., AND WATANABE, L.G. (1991)
Effects of clinical recycling on mechanical properties of nickel-titanium alloy wires.
Am. J. Orthod. Dentofac. Orthop. 100: 428-435.

KAPILA, S. AND SACHDEVA, R. (1989)
Mechanical properties and clinical applications of orthodontic wires.
Am. J. Orthod. Dentofac. Orthop. 96(2) : 100-109.

KHIER, S.E., BRANTLEY, W.A. AND FOURNELLE, R.A. (1988).
Structure and mechanical properties of as-received and heat-treated stainless steel orthodontic wires.
Am. J. Orthod. Dentofac. Orthop. 93 (3) : 206-212.

KUSY, R.P. (1981)
Comparison of nickel-titanium and beta-titanium wire sizes to conventional orthodontic arch wire materials.
Am. J. Orthod. 79 : 625-629.

KUSY, R.P. AND DILLEY, G.J. (1984)
Elastic modulus of triple-stranded stainless steel arch wire via three- and four-point bending.
J.Dent. Res. 63 : 1232-1240.

KUSY R.P. AND GREENBERG, A.R. (1982)
Comparison of the elastic properties of nickel titanium and beta-titanium arch wires.
Am. J. Orthod. 82 : 199-205.

KUSY, R.P. AND STEVENS, L.E. (1987)
Triple-stranded stainless steel wires - evaluation of mechanical properties and comparison with titanium alloy alternatives.
Angle Orthod. 57 : 18-32.

KUSY, R.P.AND WILSON, T.W. (1990)
Dynamic mechanical properties of straight titanium alloy arch wires.
Dental Mater.: 228-236.

LOVE, L.C. (1985)
Principles of Metallurgy.
Virginia, Reston Publishing Company Inc.

LYMAN, T. (1972)
Metals Handbook. 8th Edition. Atlas of Microstructures of Industrial Alloys.
Ohio, American Society For Metals

MAYHEW, M.J. AND KUSY, R.P. (1988)
Effects of sterilization on the mechanical properties and surface topography of nickel-titanium arch wires.
Am. J. Orthod. Dentofac. Orthop. 93 : 232-236.

MEHL, R.F., BARTLO, L.O., BETNER, D.R., BLACKBURN, M. J., ET.AL. (1972)
Microstructure of titanium and titanium alloys. In : Lyman, T. (ed) Metals Handbook, 8th Edition. Atlas of Microstructure of Industrial Alloys.
Ohio, American Society For Metals : 321-334.

METALS HANDBOOK. VOL 2. 10TH EDITION (1990)
Properties and selection: Nonferrous alloys and special purpose materials.
Ohio, ASM International.

MOHLIN, B., MULLER, H., ODMAN, J., AND THILANDER, B. (1991)
Examination of Chinese NiTi wire by a combined clinical and laboratory approach.
Eur. Orthod. J. 13 : 386-391.

MOLLENHAUER, B. (1989)
Updates of original articles. New approaches to the Begg technique.
Aust.Orthod.J. 11(1) : 54-55.

MUIRA, F.,MOGI, M.,OHURA, Y AND HAMANAKA, H. (1986)
The super-elastic property of the Japanese NiTi alloy wire for use in orthodontics.
Am. J. Orthod. Dentofac. Orthop. 90(1): 1-10.

MUIRA, F., MOGI, M. AND OKAMOTO, Y. (1990)
New application of superelastic NiTi rectangular wire.
J. Clin. Orthod. 24(9) : 544

NAKASIMA, A., REN HU, J., ICHINOSE, M., AND SHIMADA, H. (1991)
Potential application of shape memory plastic as elastic material in clinical orthodontics.
Eur. J. Orthod. 13 : 179-186.

NEELY, J.E. (1989)
Practical Metallurgy And Materials Of Industry.
New Jersey, Prentice Hall. Third Edition.

NELSON, K.R., BURSTONE, C.J. AND GOLDBERG, A.J. (1987)
Optimal welding of beta-titanium orthodontic wires.
Am. J. Orthod. Dentofac. Orthop. 92 : 213-219.

NICHOLSON, J.A. (1984)
An analysis of nitinol in a simulated oral environment.
Am. J. Orthod. 85 : 453.

PARK, H.Y. AND SHEARER, T.R. (1983)
In-vitro release of nickel and chromium from simulated orthodontic appliances.
Am. J. Orthod. 84 : 156-159.

PETERSON, L., SPENCER, R., AND ANDREASEN, G. (1982)
Comparison of frictional resistance for nitinol and stainless steel wire in edgewise brackets.
Quintessence Int. 13. : 563-571.

POLMEAR, I.J. (1981)
Light Alloys. Metallurgy of the Light Metals.
London, Edward Arnold (Publishers) Ltd

POLLACK, H.W. (1988)
Materials Science and Metallurgy.
New Jersey, Prentice Hall. Fourth Edition.

PROFFIT, W.R. (1986)
Contemporary Orthodontics.
St. Louis, The C.V. Mosby Company.

ROSENTHAL, D. (1964)
Introduction to Properties of Materials.
United States, Van Nostrand : 89

SARKAR, N.K., REDMOND, N., SCHWANINGER, B. AND GOLDBERG, J. (1979).
The chloride corrosion behavior of four orthodontic wires (Abstract).
J.Dent. Res. 58A ; 98.

SCHAUS, J.G. AND NIKOLAI, R.J. (1986)
Localized tranverse flexural stiffnesses of continuous arch wires.
Am. J. Orthod. 89 : 407-414.

SCHWANINGER, B., SARKAR, N.K. AND FOSTER, B.E. (1982).
Effect of long-term immersion corrosion on the flexural properties of nitinol.
Am. J. Orthod. 82 : 45-49.

SEARS, F.W., ZEMANSKY, M.W., AND YOUNG, H.D. (1987)
University Physics. 7th Edition.
Massachusetts, Addison-Wesley Publishing Company : 299-301.

SMALLMAN, R.E. (1985)
Modern Physical Metallurgy.
London, Butterworths: 77-78.

SMITH, H.H. (1972)
Steel wire and wire products. In: Lyman, T. (ed) Metals Handbook.
Ohio, American Society for Metals.

VANDER VOORT, G.F. (1984)
Metallography. Principles and Practice
New York, McGraw-Hill Book Company.

WILCOCK, A.J. (1989)
Applied materials engineering for orthodontic wires.
Aust. Orthod. J. 11(1) : 22-29.

WILKINSON, J.V. (1962)
Some metallurgical aspects of orthodontic stainless steel.
Angle Orthod. 48 : 192-206.