" A Comparison Of The Bending Behaviour Of Nickel-Titanium Orthodontic Archwires "

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DEDICATION

I wish it were possible to adequately express my gratitude to my parents. Their many sacrifices made my education feasible. Their never-ending support and unquestioning love is a debt that I will never be able to repay with the interest it deserves. Their positive influence is a standard to which I can only aspire.

Jasmin, you neglected your own needs whilst attending mine. You are my inspiration, my motivation, my salvation. Your presence is always felt deep within my heart and you are never far from my thoughts. I would like to thank you sincerely for your help and for keeping the dream alive.

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"Finis coronat opus"

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GLOSSARY

Anisotropic:

Having physical properties that differ according to the direction of measurement.

Deformation:

Any change in the geometry, size and or shape, of a body produced by the application of force.

Elastic activation:

A deformation not sufficiently severe to take the most strained element of a body beyond the elastic limit of the material.

Elastic limit:

The limit of load, stress, deformation or strain beyond which the loaded or activated body will exhibit permanent deformation and assume a new passive shape upon complete unloading or deactivation.

Hookean material:

A crystalline material exhibiting a linear relationship between induced normal or shear stress and corresponding strain when subjected to relatively small levels of activation.

Modulus of elasticity:

The slope of the normal stress versus strain graph below the proportional limit, that is, where the material exhibits perfect elasticity.

Moment of inertia:

Property of the cross-sectional area of a beam or wire, referenced to an axis line in the plane of the cross-section, that depends on the shape and size of the cross-section and the specific reference line.

Pseudoplasticity:

The unique material property where stress, or load remains relatively constant, despite a change in strain or deflection.

Strain:

Unit of deformation.

Stress:

Internal force per unit area.

ABBREVIATIONS

A-Ni-Ti	austenitic nickel-titanium alloy.
ANOVA	analysis of variance.
C	Celsius, a unit of thermodynamic temperature.
DSC	differential scanning calorimetry.
E	modulus of elasticity.
g	grams, a measure of mass.
GPa	giga pascal, a measure of pressure which equals 10^9 pascals.
K	Kelvin, a unit of thermodynamic temperature.
kN	kilonewton, a measure of force which equals 10^3 newtons.
min	minute, a measure of time which equals 60 seconds.
mm	millimetre, a measure of length which equals 10^{-3} metres
M-Ni-Ti	martensitic nickel-titanium alloy
MPa	mega pascal, a measure of pressure which equals 10^6 pascals.
N	newton, a measure of force.
Ni-Ti	nickel-titanium alloy.
PP	pseudoplastic plateau.
PYS	pseudo yield stress (or strength).
SME	shape memory effect
TTR	temperature transition range.
YS	yield stress (or strength).
XRD	X-ray diffraction.

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DECLARATION

CANDIDATE'S CERTIFICATE

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

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ABSTRACT

New generation nickel-titanium (Ni-Ti) orthodontic wires display a distinctive pseudoplastic plateau (PP) in tensile testing. A suitable marker for the onset of the plateau is 0.1% pseudo yield stress (0.1% PYS), as established by Stanton, (1995). Pseudoplasticity was the term he used to describe the unique material property depicted in tensile testing where stress, or load, remained relatively constant, despite a change in strain or elongation. An induced austenitic to martensitic crystallographic phase transformation was considered responsible for this physical phenomena. Assessment of pseudoplasticity during bend testing has been a controversial area in the literature with conflicting results reported between cantilever and three point bend testing (Andreasen and Morrow, 1978; Burstone and Goldberg, 1983; Miura *et al.* 1986).

This study had two main objectives. Firstly, to confirm the presence of pseudoplastic behaviour in these wires during bending, whilst establishing the force at which this behaviour was induced. Secondly, to compare the experimental force recorded during bending for the start of pseudoplastic behaviour to that as predicted from 0.1% pseudo yield stress data derived from previous tensile testing.

In the present investigation, four replicate specimens of five commercially available brands of 0.41 mm diameter Ni-Ti wire, from the same batch as those tested previously in tension, were examined by a three point bending test. Bending was performed using a universal testing machine, at ambient conditions (temperature 23 ± 1° C; relative humidity 50 ± 10 %) and undertaken in the 'as supplied' condition from the manufacturer. A crosshead speed of 1.0 mm/min was used and all wires were deflected vertically 4.0 mm at the midspan. Span length was set at 14 mm and each specimen was 50 mm in length. The wires in Experiment 1 were supported by standard edgewise siamese twin brackets (dimensions 0.022" x 0.028") and fastened by the use of stainless steel ligatures. For Experiment 2, a vice grip type jig was used to secure the specimens.

Experiment 1 confirmed the presence of pseudoplastic behaviour in three of the tested wires. Variation in the force generated by the tested wires, (CX, RL and SA), was investigated by a three way analysis of variance (ANOVA). Highly significant differences were found for loading and unloading ($F = 5.239$; $p \leq 0.001$) and also for brand ($F = 7.046$; $p \leq 0.001$). No significant differences were attributed to sample variation ($F = 0.501$; $p \geq 0.05$).

Force levels at which the start of the stress induced austenitic to martensitic phase transformation occurred were substantially higher in Experiment 1 than those predicted by the 0.1% pseudo yield stress data. In addition, no distinctive transition point was identified. These features can be explained by the complex stress distribution pattern produced during bending and the gradual increase in tensile stress from a concentrated area on the surface of the wire. It is proposed that higher forces were required to initiate a crystallographic phase change during three point bending due to the influence of complex stress patterns within the wire. Complex bending stresses may, therefore, account for the disparity between tensile and bending test results.

A reasonable estimate of the force produced by the tested wires as they unloaded across the pseudoplastic plateau was provided by the 0.1% pseudo yield stress data. Experiment 1 demonstrated that the reverse martensitic to austenitic phase transition was not greatly inhibited by complex bending stresses. This feature was considered to be relevant as it was the unloading of the wire that imparted a force to a malaligned tooth. It was concluded that the 0.1% pseudo yield stress data can be used to calculate a reasonable estimate of the force generated by a pseudoplastic Ni-Ti archwire during three point bending, thus providing a meaningful guide to the force produced by these wires in clinical use.

Chapter One
INTRODUCTION

1.1 Introduction

Claims have been made that several of the more recently released nickel-titanium (Ni-Ti) orthodontic archwires possessed "superelastic" behaviour (Watanabe 1982; Burstone, Qin and Morton 1985; Miura *et al.* 1986; Yoneyama *et al.* 1992). This property has been demonstrated in the performance of the wire during bend testing, where a relatively constant force is recorded over an extended portion of the load-deflection curve for both loading and unloading.

1.2 Calculation of Forces Generated

The term pseudoplasticity has been adopted in this study, as opposed to superelasticity, as the most accurate description for that section of the load-deflection curve which upon loading depicts non-linear, that is, plastic deformation. However, recovery from this point still occurs in an elastic manner; hence the deformation must have really been "pseudo-plastic" in nature. Pseudoplasticity can be reliably detected by a tensile test (Watanabe 1982; Miura *et al.* 1986). A recent investigation has confirmed the ability of this test to distinguish between pseudoplastic and non-pseudoplastic Ni-Ti wires (Stanton 1995).

The onset of pseudoplasticity is of clinical importance and needs to be quantified, as after this point, a relatively constant force is produced by an extended deflection of the wire. Stanton (1995) proposed the use of the "pseudo 0.1% yield stress", derived from tensile testing, as a suitable marker for the onset of this phenomena. The "pseudoplastic range" extended from this point until the maximum elastic elongation was reached at the 0.1% yield stress.

Theoretical calculation of the force required to induce pseudoplastic behaviour during three point bend testing can be made by the conversion of the pseudo 0.1% yield stress data. From the values reported by Stanton (1995), which are in the range of 300 to 500 MPa, it would seem that the start of the pseudoplastic range during three point bending is between 0.5 to 0.8N (50-80g) and the end of the range is between 1.7 to 2.1N (170-210g). For ease of conversion 1N approximately equals 100g.

A major question yet to be clarified is whether these theoretical values for the start of the pseudoplastic range in bending, as derived from tensile data, are in agreement with the findings of bend testing. Results published to date are unclear on this issue with approximately eight times as much force required experimentally to reach the pseudo 0.1% yield stress (Watanabe 1982; Miura *et al.* 1986). From the literature, it has been reported that inducement of pseudoplastic behaviour in Ni-Ti wires during bending was more difficult than that predicted by theory alone. Such large discrepancies between theoretical and experimental bending forces can only mean that a fundamental understanding of this material's physical properties during bending is lacking. These inconsistencies highlight a need for further evaluation of the pseudoplastic phenomena of Ni-Ti wires during bending.

This study was undertaken because many authors have also questioned the validity of predicting force systems in bending from tensile properties. It has been recommended instead, that direct measurement during bend testing be employed (Goldberg, Morton and Burstone 1983; Goldberg, Burstone and Koenig 1983; Asgharnia and Brantley 1986; Khier, Brantley and Fournelle 1991).

Chapter Two
AIMS AND OBJECTIVES

The aim of this thesis was to evaluate the behaviour of a selection of proprietary 0.016" (0.41 mm) diameter Ni-Ti pseudoplastic orthodontic archwires via three point bend testing. Specific objectives of this study were to:

- Confirm the presence of pseudoplastic behaviour in Ni-Ti orthodontic wires during three point bending;
- Establish the force levels at which the pseudoplastic behaviour occurred experimentally;
- Compare these experimental findings to the theoretical prediction derived from previous tensile testing (Stanton 1995);
- Evaluate the difference in force produced when two different methods of wire stabilisation were employed;
- Calculate the force levels for various deflections of the Ni-Ti alloy wire;
- Provide data which will contribute to an understanding of the clinical behaviour of Ni-Ti alloy orthodontic archwires;
- Test the null hypothesis that:

  "The force differential between the loading and unloading pseudoplastic plateaus of a Ni-Ti wire during three point bending remains constant as demonstrated in tensile testing."

Chapter Three

REVIEW OF LITERATURE

3.1 Development Of Nickel Titanium Orthodontic Wires

Nickel-titanium (Ni-Ti) wire was originally developed during the 1960's by William Buehler, a metallurgist at the Naval Ordinance Laboratory in Silver Springs, Maryland, USA (Andreasen and Morrow, 1978). Marketed as "Nitinol" (Unitek Corp. Monrovia, Calif.), this 55 cobalt substituted nickel-titanium wire was advocated for use during the levelling and alignment phase of orthodontic therapy (Andreasen and Hillemen, 1971). A desirable property of this Ni-Ti wire was its low modulus of elasticity (Andreasen, Atha and Fahl 1984; Andreasen, Wass and Chan 1985; Khier, Brantley and Fournelle 1991). Clinical use commenced in May 1972. About the same time, a further application was proposed which utilised the Temperature Transition Range (TTR) and the Shape Memory Effect (SME) of the wire to close extraction spaces. Unfortunately, the hypothesis was not supported by clinical trials at this early stage (Andreasen and Brady, 1972; Andreasen and Morrow, 1978).

Early investigators found that "Nitinol" had superior and unique physical properties when compared to conventional stainless steel alloy wires. Ni-Ti alloy wire had a greater elastic limit, a very low modulus of elasticity, and possessed moderate strength. Although Ni-Ti alloy wires delivered lower force levels than stainless steel alloy wires of the same diameter, the stored energy of the "Nitinol" wire was significantly greater than that of an equivalent stainless steel wire (Andreasen and Hillemen, 1971; Andreasen and Morrow, 1978; Lopez, Goldberg and Burstone, 1979). As a result, it was shown that fewer archwire changes would be necessary when using "Nitinol" for the initial phase of orthodontic treatment (Andreasen and Barrett, 1973).

The first commercially available Ni-Ti wire, "Nitinol" claimed to be superelastic (Andreasen, Heilman and Krell 1985). It is now considered, however, to have a principally work-hardened martensitic crystal structure, (Miura *et al.* 1986; Kusy and Wilson 1990; Khier *et al.* 1991; Waters 1992; Yoneyama *et al.* 1992). As such, pseudoplastic properties of this wire are not evident. Being a work-hardened martensitic alloy, (M-Ni-Ti), "Nitinol" is not capable of either a thermal or a stress induced phase transition from the austenitic state to the martensitic state (Waters 1992; Bradley, Brantley and Culbertson 1996).

Austenitic Ni-Ti (A-Ni-Ti) was developed by the Furukawa Electric Co., Ltd, of Japan (Japanese Ni-Ti) and separately by Doctor Tien Hua Cheng and associates at the General Research Institute for Non-Ferrous Metals in Beijing, China (Chinese Ni-Ti). Burstone, Qin and Morton (1985) described the mechanical properties of Chinese Ni-Ti when submitted to flexural testing using a cantilever configuration. Miura *et al.* (1986) examined the mechanical properties of Japanese Ni-Ti and made reference to the superelastic behaviour of this wire both in tensile and three point bending tests.

Although the term "superelastic" was not specifically mentioned in the literature introducing Chinese Ni-Ti (Burstone *et al.* 1985), both Chinese and Japanese Ni-Ti alloy wires displayed unique and similar loading versus deflection curves, indicating that both possessed pseudoplastic behaviour (Khier, Brantley and Fournelle, 1991). A distinction was not made by Khier *et al.* (1991) between the results of cantilever testing (for Chinese Ni-Ti), and three point bending (for Japanese Ni-Ti).

3.2 Austenitic and Martensitic Morphologies

Miura *et al.* (1986) stated that Ni-Ti is a near equi-atomic intermetallic compound, which has a temperature dependant crystal lattice morphology. At high temperatures the crystal structure is in a body centred cubic arrangement called the austenitic phase. At low temperatures, the crystal structure is in a close-packed hexagonal arrangement called the martensitic phase. Wire in the martensitic state is ductile and readily deforms, whilst in the austenitic state it is more difficult to induce deformation.

The transformation from the austenitic phase to the martensitic phase can occur by either lowering the temperature or by applying stress (Waters 1992). The equilibrium existing between the austenitic and martensitic phase is both temperature and stress dependant (Kousbroek 1984). Thus two main groups of martensite can be recognised:

- Stress induced martensite.
- Thermally induced martensite,

3.2.1 Stress Induced Martensite

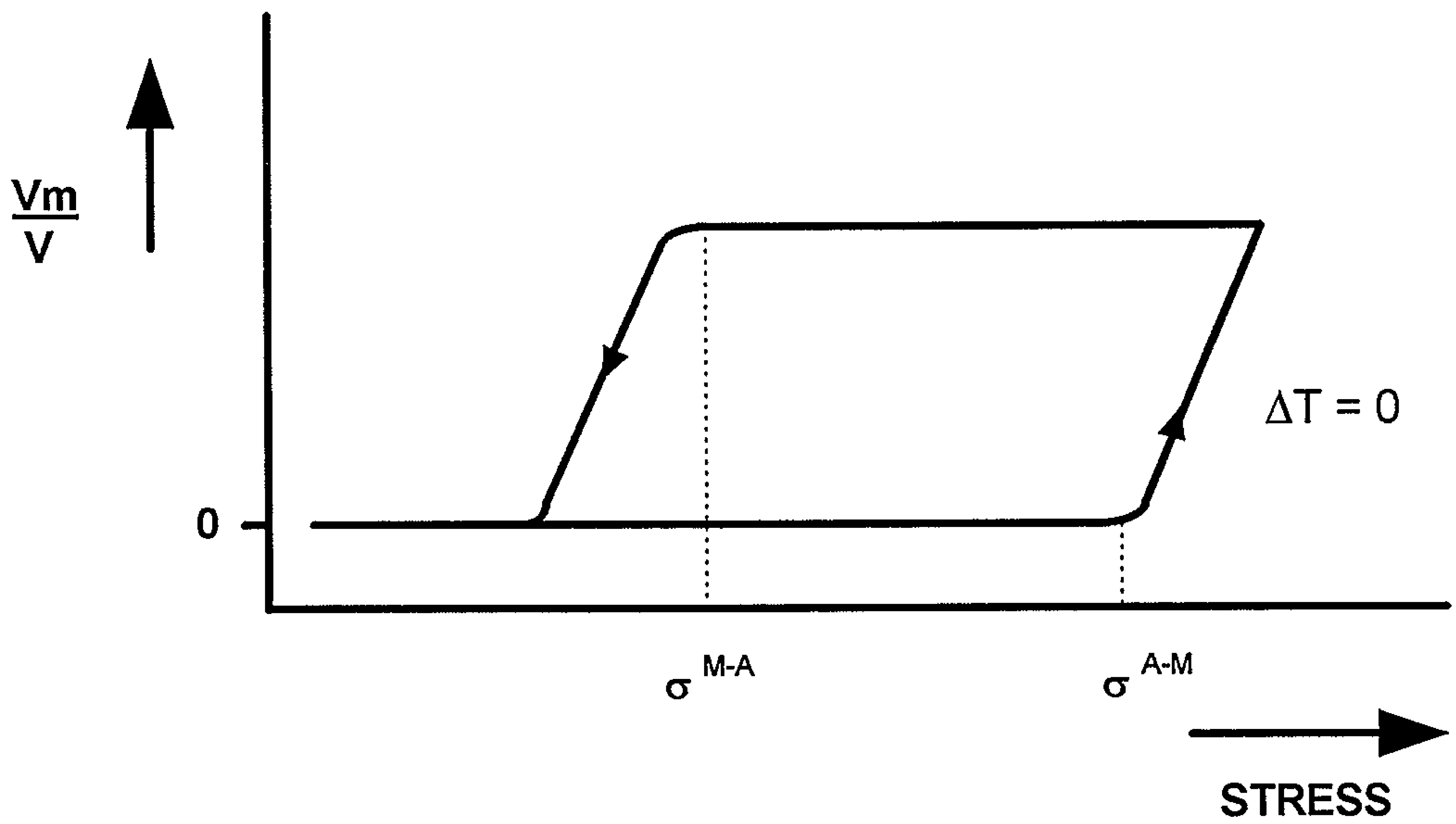


Figure 3.1 Stress induced thermoelastic martensitic transformation in function of the applied stress (adapted from Kousbroek 1984).

σ^{A-M} = stress at which austenite to martensite transformation starts

σ^{M-A} = stress at which the reverse transformation of martensite to austenite starts

$\frac{Vm}{V}$ = fractional volume of the martensite product.

Stress induced martensite is characterised by the formation of the martensitic phase by increasing levels of stress, at a constant temperature. This type of phase transformation can be reversed when the applied stress is decreased. Interestingly, this reverse transformation of martensite back to austenite, takes place at a lower stress than that required for the forward transformation of austenite to martensite (Khier *et al.* 1991). The above-mentioned stages have been identified by Kousbroek (1984). However, the transformation described is a conceptual model based on uniaxial tensile loading and the same result may be more difficult to induce in a three point beam bending configuration.

3.2.2 Thermally Induced Martensite

Thermally induced martensite is characterised by its temperature dependence. Martensite forms and grows constantly as the temperature is lowered, and reverts to austenite as the temperature is increased. Crystal growth rate of the phase is controlled by the rate of change in temperature. Martensitic transformation proceeds as an equilibrium between the chemical driving energy of the reaction and a resistive energy composed mainly of stored elastic energy (Delaey *et al.* 1974). This thermoelastic martensitic transformation occurs over a temperature transition range (TTR) and has the following stages: (Kousbroek 1984).

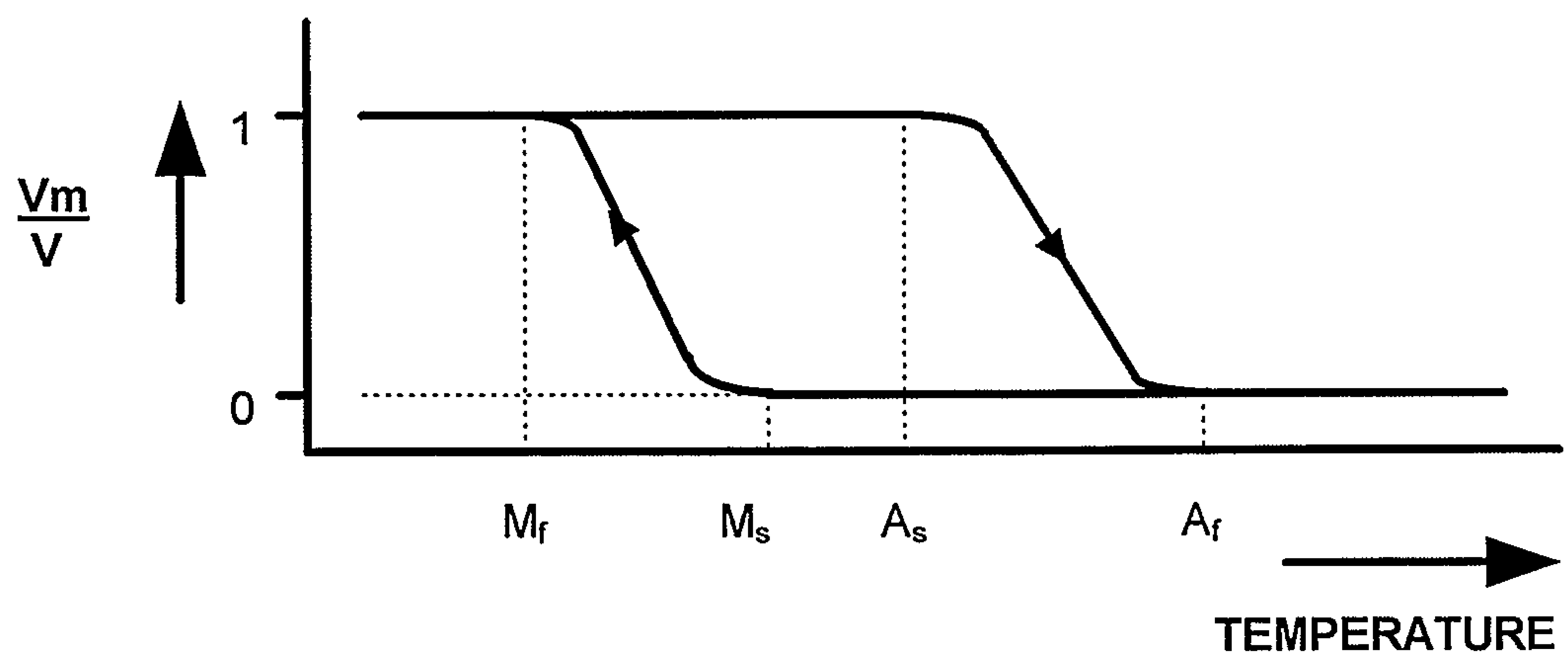


Figure 3.2 Thermoelastic martensitic transformation in a function of temperature (adapted from Kousbroek 1984).

A_s = start of austenitic transformation on heating;

A_f = finish of austenitic transformation on heating;

M_s = start of martensitic transformation on cooling;

M_f = finish of martensitic transformation on cooling.

$\frac{Vm}{V}$ = fractional volume of the martensite product.

Each wire's specific temperature transition range relates to both the composition of the alloy and its manufacturing history (Hurst *et al.* 1990). Recent research into the thermal phase change characteristics of Ni-Ti orthodontic wires has shown that the phase transformations were more complex and involved an intermediate rhombohedral (R) structure (Bradley, Brantley and Culbertson, 1996). When the growth rate of thermally induced martensite is independent of temperature change, it is called "spontaneous" or "burst" martensite and this occurs whenever the chemical driving energy of the reaction greatly exceeds the resistive energy (Delaey *et al.* 1974).

3.3 Pseudoplastic Behaviour

Pseudoplastic behaviour has been attributed to the formation of the martensitic phase from the austenitic state which allows a crystallographically reversible lattice distortion to occur (Miura *et al.* 1986; Waters 1992; Tonner and Waters 1994a). During manufacture, it is necessary for the austenitic phase to predominate in the finished wire to allow superelasticity (Khier *et al.* 1991). Pseudoplasticity can be represented schematically by a stress-strain curve.

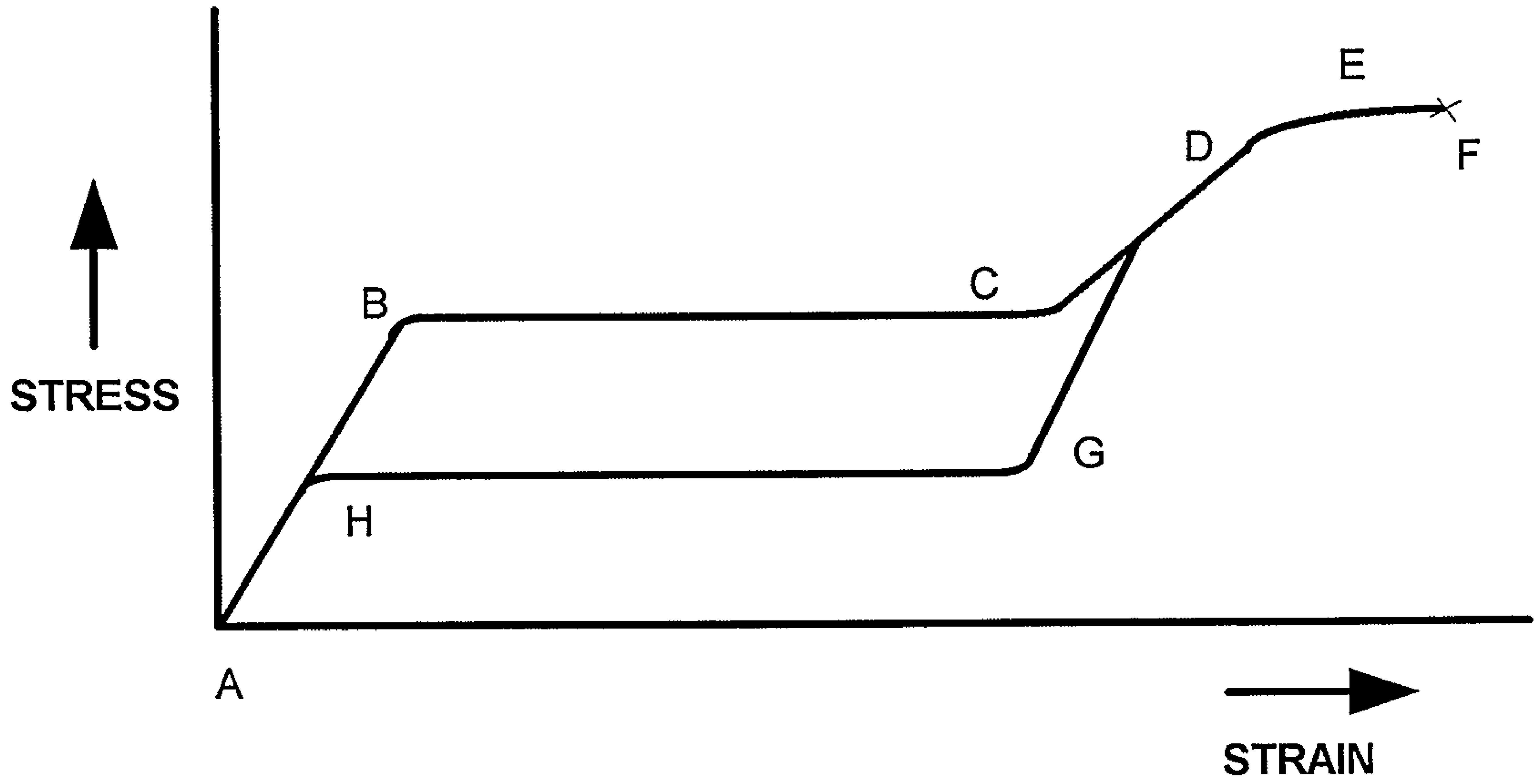


Figure 3.3 Schematic representation of a stress-strain curve showing the pseudoplastic behaviour (adapted from Krishnan *et al.* 1974).

AB represents elastic deformation of the austenitic phase.

At B the first martensite plates begin to form (pseudo 0.1% yield stress).

The transformation is almost complete at point C.

BC represents the martensitic transformation (pseudoplastic range).

CD represents deformation of the matensitic phase to the elastic limit (0.1% yield stress).

AD represents the maximum elastic elongation.

DE represents plastic deformation of the martensite.

At F fracture would occur.

DG represents the unloading of the martensitic phase from the elastic limit.

GH represents the reverse transformation to the austenitic phase.

HA represents unloading of the austenitic phase and its complete recovery at A.

Another method for pseudoplastic behaviour to occur is through a reorientation of the martensitic crystal structure which is closely related to "twinning" (Delaey 1974).

3.4 Shape Memory Effect

The shape memory effect arises if a macroscopic deformation accompanies a martensitic transformation which is not reversed by removing the applied stress. In a subsequent step, the transformation back to the austenitic phase and an accompanying reversal of the macroscopic deformation are induced by heating (Delaey *et al.* 1974). Early investigators noted that the shape memory phenomenon was related to the temperature transition range of the wire. Some Ni-Ti alloys can be plastically deformed at temperatures below their TTR and upon heating back through the TTR, the original shape is regained. For maximum shape recovery, plastic deformation should be restricted to approximately 8 per cent of the original length (Hurst *et al.* 1990; Stanton 1995). Two types of shape memory effects are recognised:

- One-way shape memory effect (as described above),
- Two-way shape memory effect.

3.4.1 Two-Way Shape Memory Effect

Two-way shape memory effect is characterised by the memory of its undeformed shape on heating and also by regaining its deformed shape on cooling (Kousbroek 1984).

3.5 Beam Bending Theory

Behaviour of a beam during bending has been extensively researched in the engineering discipline. Accurate prediction of a beam's performance is made possible by the application of a suitable bending mechanics analysis, which is related to the configuration of the beam and its supports. To convert the real world situation into a manageable mathematical model, a number of assumptions about the beam have been made. A solid beam is considered to be composed of a large number of longitudinal fibres, each having an infinitesimally small cross sectional area in comparison with that of the beam itself. All fibres of the beam are modelled as passively straight. In bending these fibres are deformed and display identical curvatures. Depending on the location of the fibre, the strain of bending may be tensile, compressive or zero. One fibre coincides with the neutral axis.

When a wire is bent, the outside curved surface is placed in tension, whilst the inside curved surface is compressed. Midway between the two extremes is the neutral axis, at which the bending stress is zero. The neutral axis resembles a flat ribbon which runs through the centre of the wire midway between the outer and inner curved sides. It divides the bending stress into tensile and compressive halves. At the neutral axis there is no tension, compression, change in length or force storage (Thurow 1982). The parts of the wire farthest from the neutral axis, that is, the outer and inner curved surfaces are deformed the most during bending. On any beam cross section, the fibre strains are proportional to their distances from the neutral axis. Figure 3.4 below provides a diagrammatic representation of these features in a round wire undergoing three point bending.

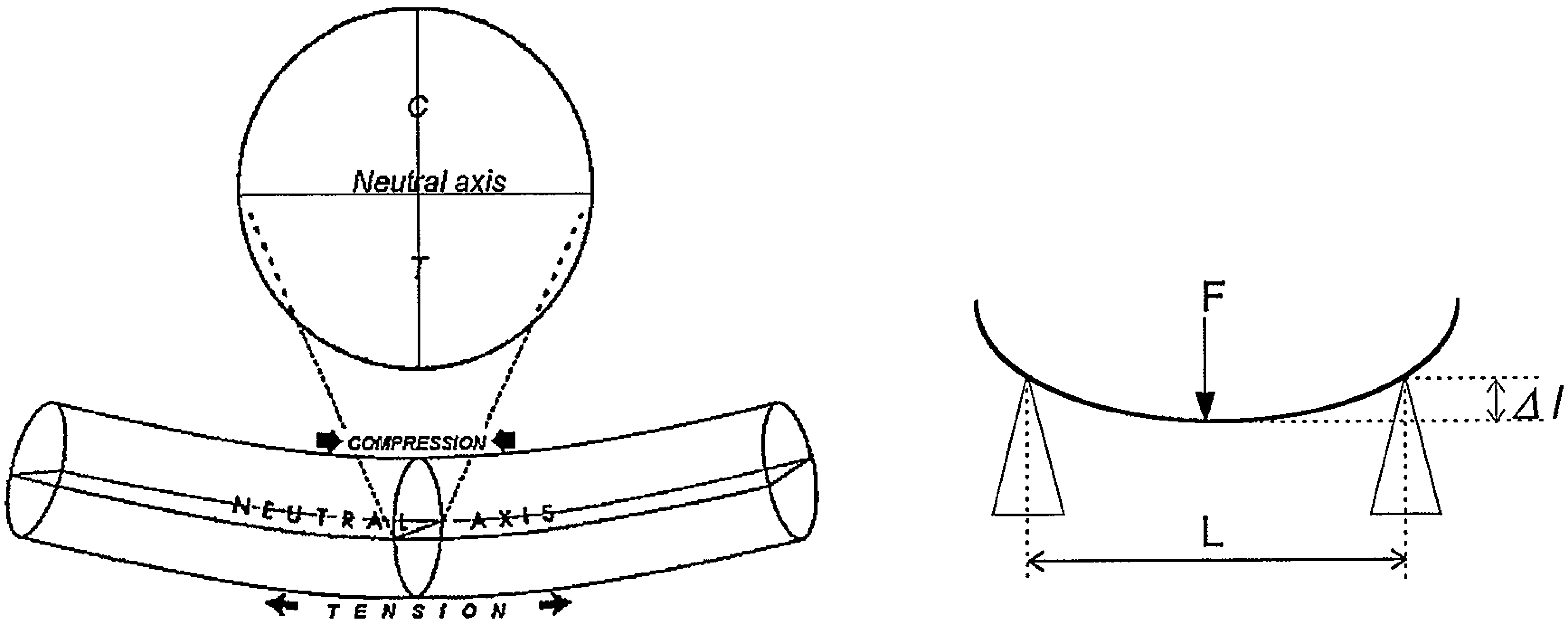


Figure 3.4 Diagrammatic representation of a round wire undergoing three point bending (adapted from Thurow 1982).

For three point bending the maximum stress is developed at the surface of the wire, directly opposite the applied load and it gradually decreases to zero at the supported ends. The stress distribution pattern is represented below in Figure 3.5.

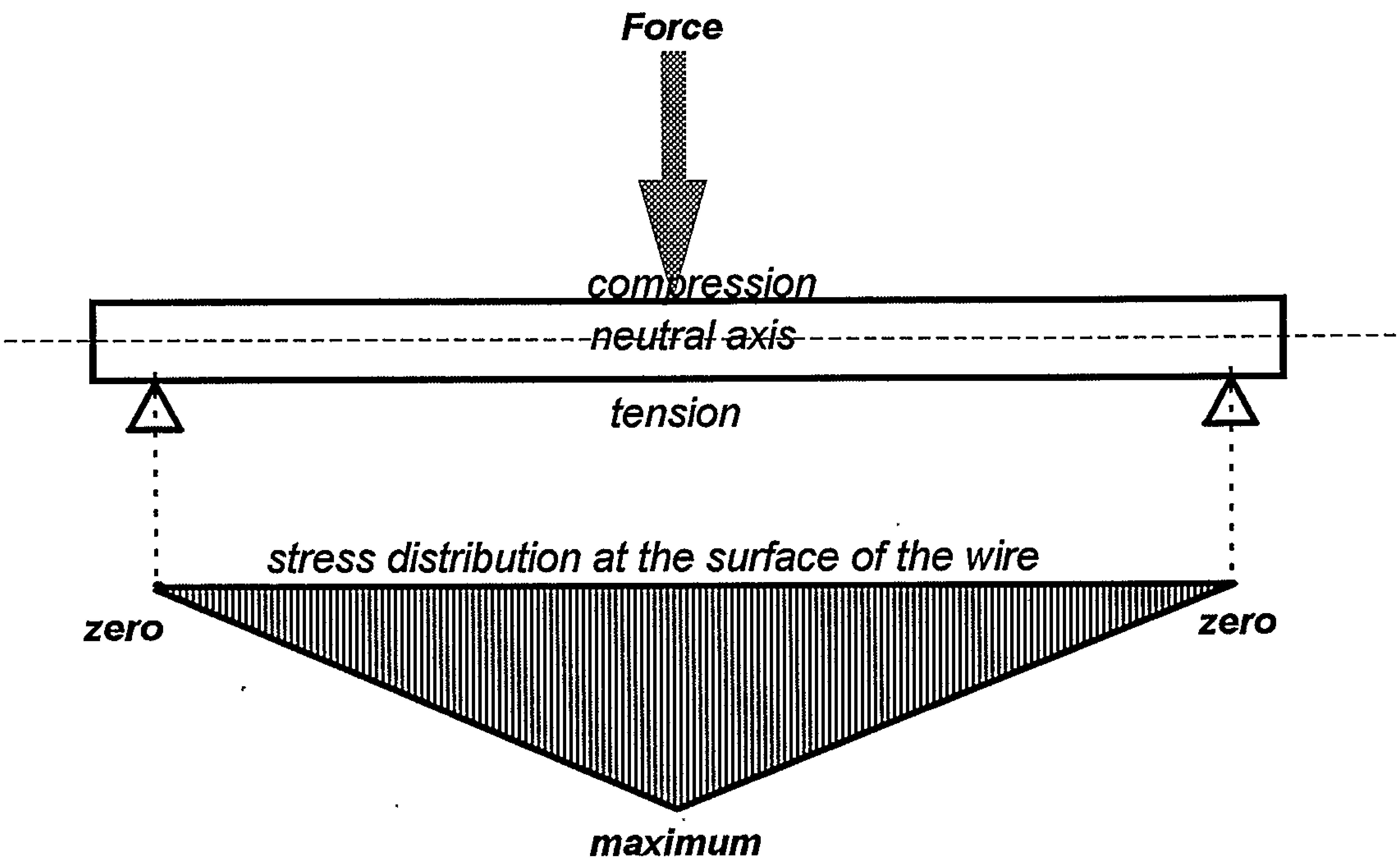


Figure 3.5 Diagrammatic representation of the stress distribution pattern at the surface of a round wire undergoing three point bending (Adapted from Mencik 1992).

Maximum stress is concentrated to a single point, regardless of the applied force. Therefore, the first point on the wire to begin a stress induced austenitic to martensitic phase transformation is that single point on the surface of the wire directly opposite the applied load which has reached the threshold stress. As the applied load is increased, a larger section of the wire can be induced, by elevating a greater area of the wire to the threshold stress.

According to Thurow (1982), the force generated by a deflection of a beam is related to the material's intrinsic properties as well as the geometry of the beam and its supports. Small deformation theory of beam bending considers the linear displacement of the neutral axis and is invoked when the displacement of the neutral axis is less than 45° from its passive position. A number of assumptions in this analysis have been made involving geometry and material behaviour. These are: cross-sectional symmetry of the beam; in-plane bending; elastic activation; Hookean material; and values of the elastic modulus are equal in tension and compression. For three point bending, the following formula describes these relationships when a force is applied at the midspan and the ends are free to slide:

Equation 3.1

$$F = \frac{48\ E\,I\,d}{L^3}$$

where:
(d) is the vertical deflection of the neutral axis from its passive position at the midspan.
(L) is the length of the span between the two supports.
(F) is either the force generated or the load applied.
(E) is the modulus of elasticity for that beam material.
(I) is the moment of inertia of the cross-section.

If the ends of the beam are fixed and are unable to slide, the formula becomes:

Equation 3.2

$$F = \frac{192\ E\,I\,d}{L^3}$$

If the stress has been calculated from previous tensile testing, the following equation can be applied to calculate the force generated:

Equation 3.3

$$F = \frac{\sigma\ \pi\ D^3}{8\ L}$$

where:
(σ) = stress (internal force per unit area).
(π) = 3.14159265
(D) = diameter of the beam.

As stress is not evenly distributed through the wire during bending, Equation 3.3 calculates the force which occurs at the surface of the wire only (Figure 3.5). This value diminishes to zero at the neutral axis and, therefore, is not representative of the forces acting throughout the diameter of the wire. Goldberg, Morton and

Burstone (1983) have identified several reasons why flexural moduli calculated from tensile data may not be appropriate. Firstly, large deflections employed in bending tests may invalidate small deformation theory. Secondly, cold working during the manufacturing process of orthodontic wires may render them anisotropic which will diminish the validity of the conversion.

For analytical purposes, orthodontic archwires have been studied as if they were beams, where the span is equivalent to the interbracket distance, the supports are the edges of the orthodontic bracket on each tooth and the load is applied at the midspan. Burstone and Koenig (1974) contend that 'two-tooth' analyses, that is, a single span, are the basic building blocks for understanding the force systems in an orthodontic appliance. Furthermore, by summing a series of two-tooth force systems, the forces acting on each tooth along the arch can be calculated.

3.6 Previous Bending Tests in Orthodontics

Appendix 1 (adapted from Kapila and Sachdeva 1989), provides a summary of reported bending tests. Overwhelmingly, there is a lack of standardisation in the literature, making comparisons between experiments largely impossible. The type of test employed, the span length used and the material properties investigated varied enormously. Unfortunately there has not been a methodology developed specifically for the unusual properties found in nickel-titanium wires. Most researchers have chosen cantilever testing as described by the American Dental Association specification number 32, or its modification with a shorter span length. This standard was originally formulated for stainless steel wires and was developed before Ni-Ti wire was in routine clinical use. Other relevant published standards were also principally formulated for stainless steel wires and include:

- British Standard BS 3507:1976;
- Australian Standard 1964-1977.

In an attempt to quantify the bending characteristics of Ni-Ti alloy wires and extrapolate data which will pertain to their clinical use, some investigators developed their own test methods. Consensus has yet to be reached on whether to adhere to published testing methods, or to modify them as required, or to develop entirely new testing methods. The best approach to this problem of bend testing for Ni-Ti wires is simply not known.

Early research into Ni-Ti archwires was directed towards establishing the superior properties of this material compared to stainless steel for the levelling and alignment phase of orthodontic treatment. During activation, Ni-Ti wires demonstrated greater elasticity, less permanent deformation and were capable of storing a higher spring energy than stainless steel (Andreasen and Hilleman, 1971; Andreasen and Morrow, 1978; Lopez, Burstone and Goldberg, 1979; Drake *et al.* 1982).

Incorporation of nickel-titanium archwires by the orthodontic profession into regular treatment regimes may have been the driving force for the next common area of scrutiny into these wires, namely quantification. Once the exceptional qualities of Ni-Ti archwires were demonstrated, researchers sought to calculate the forces generated by these wires during activation. From examination of Appendix 1, a clear division of thought was revealed as how this task was best accomplished. Some investigators adhered to published standards or their modification and others sought to model the clinical situation.

Of those researchers who used a published standard for their investigation, most chose the American Dental Association Specification Number 32, or its modification with a shorter span length, when they quantified the force produced by an activated Ni-Ti archwire (Andreasen and Morrow 1978; Lopez, Goldberg and Burstone 1979; Drake *et al.* 1982; Goldberg, Morton and Burstone 1983; Burstone and Goldberg 1983; Andreasen, Hilleman and Krell 1985; Burstone, Qin and Morton 1985; Asgharnia and Brantley 1986; Khier, Brantley and Fournelle 1991). As most of these authors deviated from the exact specifications contained in this standard, a critical comparison of their findings was extremely difficult. Often a lack of detail in the literature prevented an analysis of the different experimental findings. Kusy and Stush (1987) did, however, tabulate a comparison of the value for the modulus of elasticity for Ni-Ti alloy archwires in bending from a number of the previously mentioned investigations. Kusy and Stush (1987) suggested a different modulus of elasticity depending on the geometry of the wire, either round or square, which was in agreement with the values reported from the other studies cited. The general findings of all the above mentioned studies confirmed the superior elastic properties and clinically acceptable forces of Ni-Ti archwires when compared with stainless steel wires for similar amounts of activation.

A degree of controversy surrounds the use of the American Dental Association Specification Number 32. Not all investigators believed that it was the best method by which to quantify the forces generated by highly flexible Ni-Ti alloy wires. Criticism has been levelled against the cantilever test itself and also against the small deflection mechanics analysis employed by this standard (Yoshikawa *et al.* 1981; Goldberg, Burstone and Koenig 1983; Schaus and Nikolai 1986; Miura *et al.* 1986; Asgharnia and Brantley 1986; Nikolai, Anderson and Messersmith 1988). Main areas of concern raised by these authors have included:

- Configuration of the cantilever test which allowed slippage of the applied load and an increased span length with highly flexible wires;
- Assumption of a purely elastic model;
- Irrelevance of cantilevers to continuous archwires and the clinical setting;
- Potential erroneous use of the beam deformation formula;
- False positive result of superelasticity in some Ni-Ti wires.

These criticisms surrounding the American Dental Association Specification Number 32 led some investigators to develop their own testing methods. Others, in an attempt to model the clinical situation more precisely, or to examine a particular facet of orthodontic treatment of interest to them, also evolved their own testing protocols. Parameters between the various tests were not standardised nor was consensus achieved as to the preferred approach. As many of the findings were inconsistent, and many different aspects of Ni-Ti alloy archwires had been investigated, the reported results were impossible to compare.

An illustration of this confusion in the literature is highlighted by the following examples. Andreasen and Hilleman (1971) simulated the levelling phase of orthodontic treatment with the use of extracted teeth embedded in plaster and produced load-deflection graphs. Schaus and Nikolai (1986) quantified the transverse flexural stiffness of five preformed archwires in three planes of activation at five different sites on simulated dental arches. Rock and Wilson (1988) compared the results obtained for archwires tested in a simulated dental arch with those from three point bending. Hudgins, Bagby and Erickson (1989) investigated permanent deformation after long term deflection. Mohlin *et al.* (1991) combined a laboratory and a clinical study and recommended that a bending test should be combined with an electron microscopic surface study. Despite exhaustive investigation and reporting, many experiments cannot be satisfactorily compared due to their widely differing aims and methodologies.

Although many of the problems inherent with bend testing could be eliminated by the conversion of data derived from tensile testing, many researchers have found that the forces generated in bending are not accurately predicted from tensile properties (Goldberg, Morton and Burstone 1983; Goldberg, Burstone and Koenig 1983; Asgharnia and Brantley 1986; Khier, Brantley and Fournelle 1991). Furthermore, direct measurement of the forces produced by bending was considered more accurate. Burstone and Goldberg (1983) also contend that use of orthodontic archwires in the clinical setting produces greater forces than theory predicts due to activation beyond their respective elastic limits, that is, partial plastic deformation.

In an attempt to overcome some of the disadvantages of cantilever testing and to provide a more useful clinical model, alternative beam geometries were proposed. Asgharnia and Brantley (1986) found a significant difference between data derived from cantilever bend testing to that from tension and recommended consideration of either 3 or 4 point bend testing as the standard on which to test mechanical properties. Miura *et al.* (1986) claimed that their protocol was the only bending test capable of discriminating between superelastic and non-superelastic behaviour in highly flexible Ni-Ti alloy wires. Kusy and Stush (1987) examined "Nitinol" in both 3 and 4 point bend testing and found the modulus of elasticity to be independent of bending modes (3 or 4 point). Nikoli, Anderson and Messersmith (1988) proposed a five point bending test to simulate archwire activation to a malaligned tooth. The latter authors proposed a three point test with the addition of two extra supports to mimic the configuration of a standard edgewise orthodontic bracket. This was designed as an alternative to the American Dental Association Specification Number 32. Clearly then, no agreement on a bending test method has been established in the literature.

Chronologically, the next common area of interest in the literature relating to nickel-titanium alloy archwires was the property of "superelasticity". Watanabe (1982) claimed that a Ni-Ti alloy with this new property had been developed. The most salient feature of this alloy was depicted in the load-deflection graph where an almost constant force was released as the wire was unloaded over a wide range of deflection. Superelastic behaviour was considered important and significant in the clinical use of these wires.

In a landmark article, Miura *et al.* (1986) claimed that a proprietary brand of nickel-titanium wire had been developed for orthodontic use which possessed superelastic properties. The authors declared that this alloy (Japanese Ni-Ti) was unique and could be distinguished from other brands commercially available when subjected to three point bend testing. The bend testing protocol established by their study used a 14 mm span of wire between two orthodontic brackets welded to supporting steel poles. The test specimen was held in place with ligature wire with a known quantity of force. The midportion of the specimen was deflected 2.0 mm at a crosshead speed of 0.1 mm/min under pressure from a metal pole of 5 mm diameter connected to a load transducer.

Results were presented as a series of load deflection curves. For 0.016" diameter Japanese Ni-Ti, the curve was almost linear to 0.7 mm and then reached 650g at 2.0 mm deflection. During unloading, between 2.0 mm and 1.6 mm deflection the load was seen to drop rapidly. Between 1.6 mm and 0.6 mm deflection the load decreased by a small amount from 350g to 250g. For deflection less than 0.6 mm, the load decreased proportionately and a permanent set of 0.01 mm was recorded. The unloading curve was described as superelastic behaviour by the authors and it was not exhibited by either stainless steel, cobalt-chromium-nickel or "Nitinol" orthodontic wires. Force ranges for superelastic behaviour in 'light', 'medium' and 'heavy' grades of Japanese Ni-Ti (0.016" diameter) were reported as 30 to 70g, 140 to 170g and 250 to 280g, respectively.

Superelasticity was interpreted as a result of the stress induced martensitic transformation of the Japanese Ni-Ti wire as it passed from its austenitic crystal structure to the martensitic arrangement. The American Dental Association Specification Number 32 was not used as the authors believed cantilever testing was unacceptable and would not accurately differentiate between wires which did and did not possess superelastic properties.

Khier, Brantley and Fournelle (1991) compared the bending properties of three superelastic and three non-superelastic Ni-Ti orthodontic wires using a cantilever configuration. Their study used a modification of American Dental Association Specification Number 32. Comparison of the bending graphs produced by this test revealed considerable differences between the plots for superelastic and non-superelastic wires. Similarities were found however, within each group. Specifically the curves during loading and unloading for the non-superelastic wires were steeper, reached a far greater maximum bending moment and had a greater degree of residual permanent deformation after 80° of activation.

In contrast, the curves of the superelastic wires displayed unique characteristics. The gradient of the loading portion of the curves was considerably reduced and non-linear, the maximum bending moment was approximately one-half that of the non-superelastic wires, and the unloading portion of the curves generally contained a near horizontal or plateau region where deactivation took place at almost constant moment values. Also the degree of residual deformation after 80° activation was approximately one-third that of the non-superelastic wires. The differences between the bending properties of the superelastic and non-superelastic wires were attributed to the relative proportions of the austenitic and martensitic microstructures present in each wire. X-ray diffraction (XRD), differential scanning calorimetry (DSC) and optical microscopy were used to determine the metallurgical structures present in the wires. The investigation by Khier *et al.* compared favourably with the findings of Miura *et al.* (1986) and established the superiority of superelastic nickel-titanium alloy wires over non-superelastic wires.

More recent research into Ni-Ti alloy archwires has focussed on the relationship between bending properties and thermal behaviour, using differential scanning calorimetry (DSC). Yoneyama *et al.* (1992) investigated the bending properties of nickel-titanium alloy orthodontic wires by a three point bending test. Wire specimens 30 mm in length were suspended across two 5 mm diameter metal poles with their centres set 14 mm apart. Specimens which were neither bracketed nor ligated were deflected by a third metal pole to a maximum of 2.0 mm with a crosshead speed of 0.2 mm/s at body temperature (310K). Thermal behaviour due to phase transition of the alloy was examined by DSC. Clearly demarcated thermal peaks were detected in the DSC curves of superelastic wires whereas non-superelastic wires had no thermal peaks. Some of the superelastic wires did not have temperature transition ranges at body temperature.

As the pseudoplastic property of Ni-Ti alloys occurs in association with a stress induced martensitic transformation and the unloading orthodontic force relates to the reverse transformation of the martensitic phase to the parent austenitic phase, the finish of the Temperature Transition Range (TTR) for orthodontic purposes must be below body temperature. That is, point A_f, the finish of martensitic to austenitic transformation on heating, must be below body temperature so that the wire is fully in the austenitic phase at mouth temperatures and thus able to exhibit pseudoplastic behaviour in the oral cavity when stress is applied. Waters (1992) classified superelastic wires with a TTR between an unspecified room temperature and body temperature as martensitic active alloys and those which TTR is below room temperature as austenitic active alloys. The austenitic type were claimed to produce higher forces than the martensitic, but both types had a large elastic range and released a nearly constant force over a large portion of the deactivation curve in the load-deflection graphs.

Tonner and Waters (1994a) studied the load-deflection behaviour of a number of commercially available superelastic Ni-Ti alloy orthodontic wires by three point testing over the temperature range 5° - 50° Celsius. Forces at mouth temperature were found to differ by 600% for wires of the same nominal diameter made by different manufacturers. Typically the superelastic alloys had a predominantly austenitic grain structure with some martensite present. Also an intermediate phase in the austenitic to martensitic transformation, called the R structure was present. The relative proportions of these phases at room or mouth temperature is dependant on the exact composition of the alloy and the degree of cold working and heat treatment of the wire during manufacture.

Distinctive changes in the general characteristics of the load-deflection plot of superelastic wires occurred as the ambient temperature was lowered:

- The gradient of the initial slope decreased markedly as temperature was reduced.
- The values of the loading and unloading plateau regions decreased as ambient temperature was reduced.
- Above a critical temperature a wire behaved in a perfectly elastic manner over a wide temperature range.

Below this critical temperature the wire displayed 'permanent' strain and no plateau region. If the temperature was elevated above the critical threshold the wire would recover. The authors also noted that in the majority of cases these superelastic wires had to be deflected at least 2.0 mm over a span of 13 mm to produce a plateau region.

From the literature it is obvious that Ni-Ti alloy wires have undergone intensive investigation. Recent research has shown increased sophistication and complexity as a greater understanding of this material is achieved. Unfortunately, much of this work in the orthodontic sphere has been clouded by confusion in terminology. "Superelasticity" as presented in the literature lacks clarity and is a purely implied material property. It has no obvious beginning nor end and, hence, it cannot be accurately measured. A succinct and clear description of this phenomenon which does reflect material parameters on a load-deflection graph is required to enable suitable quantification and comparison between various commercially available wires.

3.7 Clinical Application of Bend Testing Data

Many researchers have sought to correlate mechanical testing of orthodontic wires to their intended clinical use. From the literature reviewed, it is evident that it is impossible to completely model the clinical situation by laboratory testing alone as there are too many individual variables and interactions to consider.

Burstone and Goldberg (1983) contend that most orthodontic appliances are activated beyond their elastic limit in clinical practice. Accurate prediction of forces generated in the clinical setting will, therefore, require a consideration of both elastic and plastic behaviour as the maximum elastic bending moment is a theoretical value which is usually surpassed in clinical orthodontics. To date, no analysis has included the influence of plastic behaviour.

Prediction of force magnitudes delivered by pseudoplastic Ni-Ti alloy wires is difficult due to their complex unloading curve and the fact that stiffness varies according to the amount of activation (Burstone, Qin and Morton 1985). Hookean behaviour of the wire is assumed in bending mechanics analysis, but the pseudoplastic behaviour Ni-Ti wires deviate from this ideal providing another source of potential error.

Schaus and Nikolai (1986) investigated the relevance of cantilever bending theory when applied to orthodontic archwires. The recommended cantilever test, as detailed by the American Dental Association Specification Number 32, uses a passively straight length of wire and a rotational bending stiffness instead of a force deflection movement similar to the transverse deformation of a levelling archwire. As such it was a poor model of the clinical situation. Asgharnia and Brantley (1986), found that the calculated values for the modulus of elasticity (E) and flexural yield strength (YS) varied with the span length of the test wire when the mathematical formula contained in ADA Specification Number 32 was applied. Since E and YS are intrinsic material properties, and are independent of span length, variation in these results highlights deficiencies present in the bending mechanics analysis used in Specification Number 32.

Cantilever bend testing is largely irrelevant to clinical orthodontics. For example, Ni-Ti continuous archwires are never used as cantilevers, but rather as short spans ("beams") in between adjacent orthodontic brackets. Rock and Wilson (1988) found that an orthodontic archwire in clinical use is far more rigid than beam tests would suggest. They postulated two variables which may explain the difference:

- An increase in rigidity due to the shape of the archwire;
- The restriction caused by ligation of the wire into the brackets.

Many other factors are involved in the clinical use of Ni-Ti archwires in an orthodontic appliance which may also affect force levels (Burstone and Koenig

1974; Waters, Stephens and Houston 1975; Burstone and Goldberg 1983). These factors may be summarised as:

- Curvature of the archwire;
- Orthodontic bracket type, width and angulation;
- Friction within the brackets which limited sliding of the archwire;
- Friction produced by the type of ligation of the orthodontic bracket;
- Effects of occlusion;
- Inter-bracket distance or span length;
- Effects of plastic deformation.

Despite the plethora of tests performed and results reported in the literature no study can truly eliminate all the potential sources of error. Mohlin *et al.* (1991) concluded that laboratory testing alone did not seem capable of identifying major problems with the clinical use of a new material. Kapila and Sachdeva (1989) surmised that the mechanical testing of orthodontic archwires did not necessarily reflect the clinical situations to which these wires are ultimately subjected. They did provide, however, a useful basis for comparison.

Chapter Four
MATERIALS AND METHODS

4.1 MATERIALS

Four proprietary wires marketed as superelastic and one non-superelastic wire (Nitinol) were selected for testing as listed below in Table 4.1. All wires were 0.41 mm (0.016") in diameter and in preformed arch shapes, except for wire RL which was supplied in straight lengths.

Table 4.1 Wires tested.

Name / Description	*Manufacturer*	*Batch Number*	*Code*
Nitinol	3M Unitek	304	MN
Super-elastic	3M Unitek	502	ME
Chinese NiTi	CX Enterprise	Not given	CX
Rematitan Lite	Dentaurum	71744	RL
Sentalloy (medium)	GAC	AB0177	SA

4.2 METHODS

Two test samples 50 mm in length, were prepared from each preformed archwire utalising the straight distal ends. The wires were tested as supplied, under two different testing configurations (Experiments 1 and 2). Four replicate specimens of each brand were tested in each experiment. Calibration of the 1kN load cell was accomplished prior to testing, after the electronics of the testing machine (Shimadzu Autograph AG 50 NE, Tokyo, Japan) had stabilised for approximately thirty minutes. Ambient conditions were 23 ± 1° C and 50 ± 10 % relative humidity.

4.2.1 Experiment 1

In this experiment, the wires were supported in edgewise brackets and secured with stainless steel ligatures. Standard siamese-twin edgewise brackets with dimensions of 0.022" x 0.028" were welded to two supporting abutments and set apart to establish a 14 mm interbracket span. Single point force was applied vertically to the midpoint of the wire via a push rod connected to a load cell, as shown in Figure 4.1. Loading of the test specimens at a cross head speed of 1.0 mm/min was continued until 4.0 mm of deflection was reached and then the direction was reversed with the specimens unloaded at the same rate, back to the origin.

4.2.2 Experiment 2

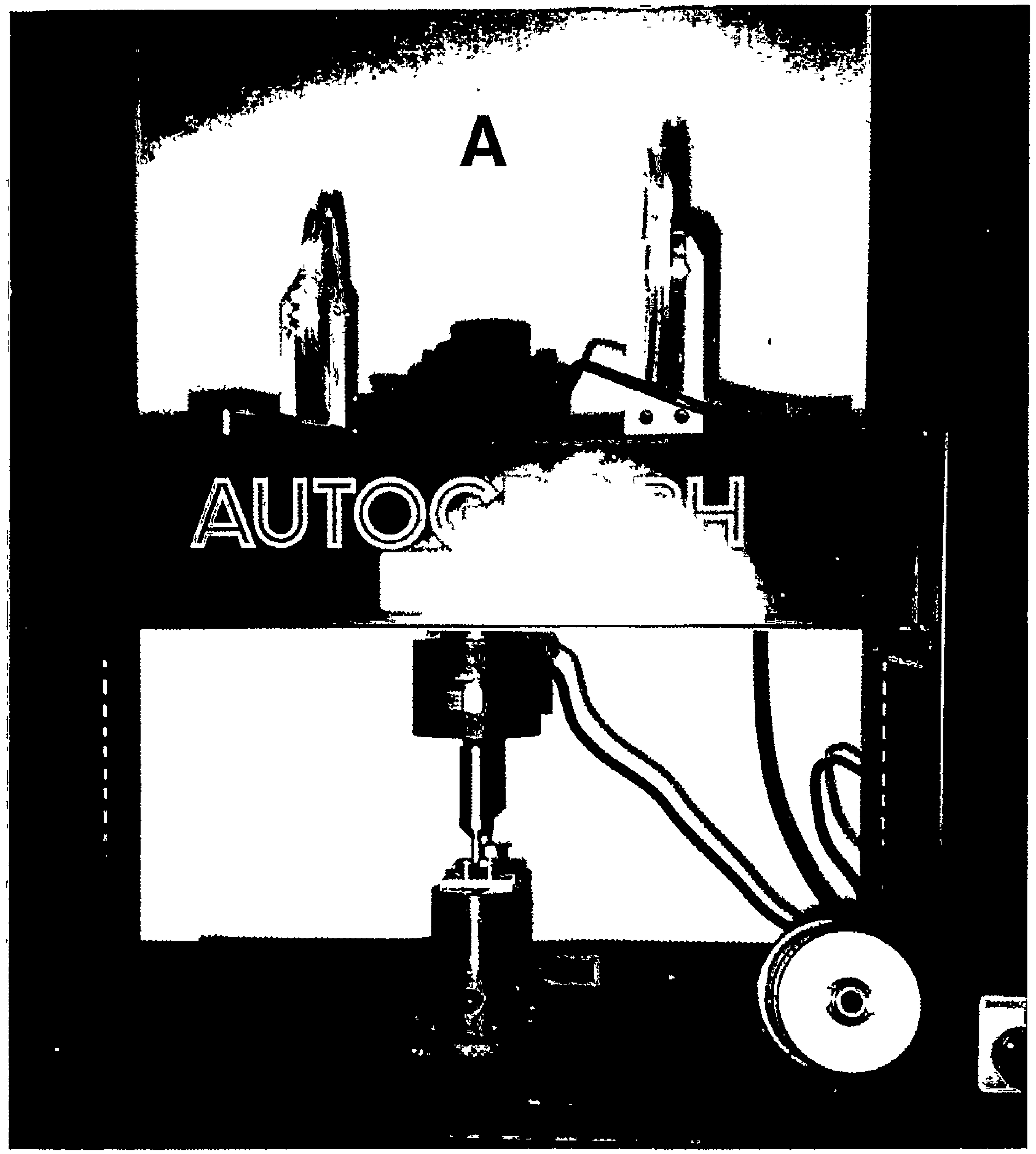
In this experiment, the wires were secured in a vice-grip testing jig which had a span of 14 mm to establish the same span length as that used in Experiment 1. This apparatus was developed to totally eliminate slippage of the wire during testing. Again, single point force was applied vertically to the midpoint of the specimen wire using the same testing conditions as in Experiment 1.

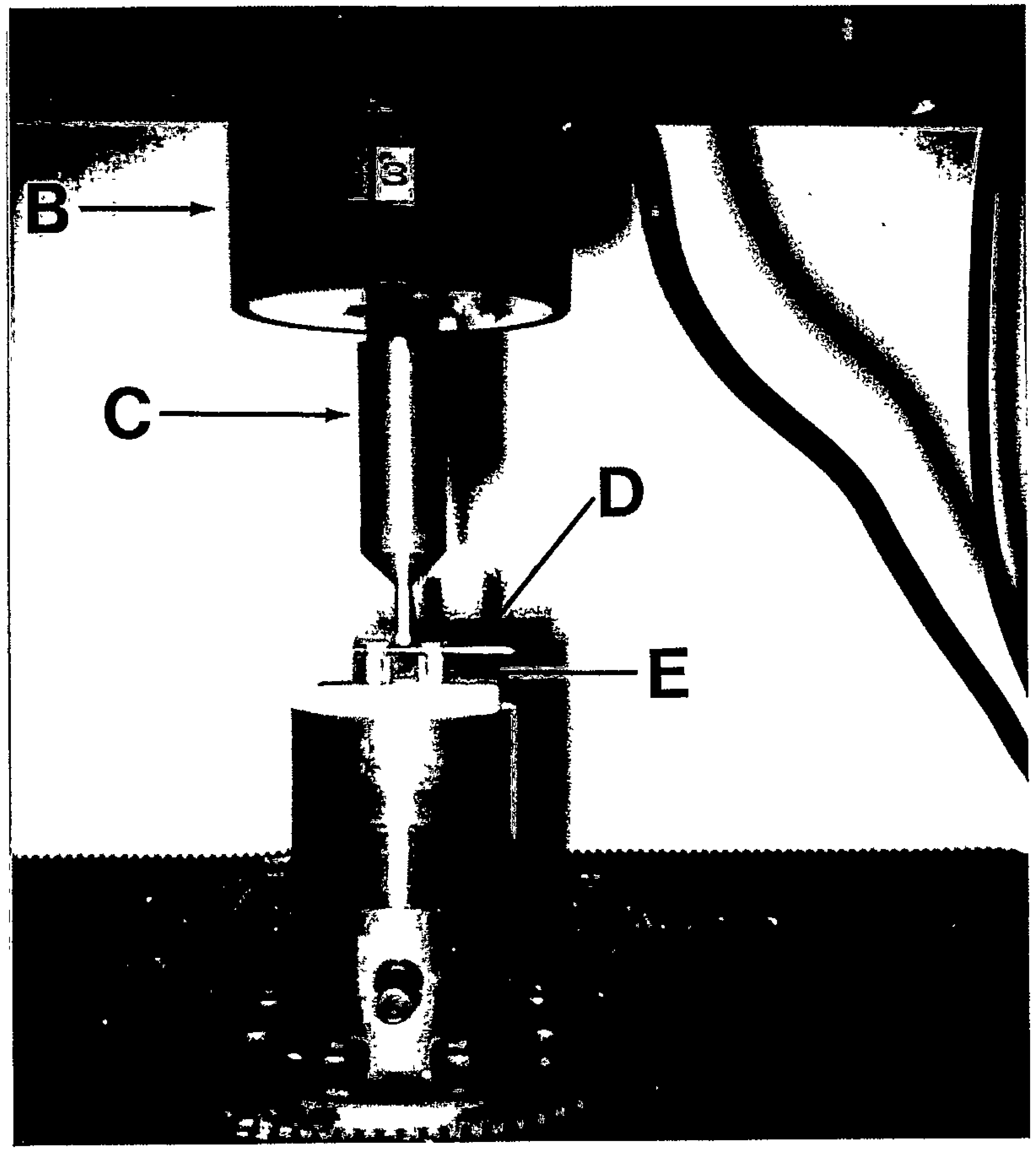
4.3 Data Analysis

All data recorded by the testing machine for force and deflection were stored in spreadsheet format using Microsoft Excel version 5.0. The plot for each wire tested was constructed by the "x-y scatter graph" tool included in that program. The deflection required to induce a phase transformation in the wire was determined by the following method. A straight line was drawn tangentally to the initial linear section of each loading curve for each specimen. The point where the plotted curve began to deviate from this tangent was extrapolated to the X axis and the value was recorded.

The modulus of elasticity was determined by magnifying on-screen images of the plotted curves 200% to inspect the linear portion of the initial loading curve. From the screen, two points were chosen for each specimen to give the longest straight segment. The corresponding values for deflection and force from the spreadsheet data were then used in Equation 6.1 to calculate the modulus of elasticity.

Figure 4.1 Photographs of the experimental apparatus showing the testing machine (A) and detail of the 1kN load cell (B); push rod (C); and the specimen wire (D); held in a siamese twin bracket jig (E).





Chapter Five

RESULTS

5.1 Experiment 1

Individual plots of the load-deflection curves for the four replicate specimens of each brand of wire are displayed in Figures 5.1 to 5.5. For the control wire MN (Figure 5.1) the plotted curves exhibited good reproducibility up to 2.0 mm of deflection. An average force of 5.4N (540g) was recorded at that point, after which slippage of the wire produced progressively erratic plots until 4.0 mm of deflection and an average force of 9.7N was reached. At 4.0 mm of deflection, permanent deformation of the wire specimens was noted so that during unloading the applied force rapidly decreased to the limit of the programmed safety cut-out on the testing machine which was specified at 5% of the maximum load. Hence the plotted curve rapidly approached 0.0N, then stopped without recording an unloading curve back to 0.0 mm deflection.

Wire ME displayed a greater degree of variation in the plots of the individual specimens (Figure 5.2). However, the slope up to 1.0 mm of deflection was reproducible except for one specimen which recorded the highest force at 4.0 mm of deflection. Slippage was noted again after 2.0 mm of deflection (4.2N), and the curves again became progressively erratic with further loading. This feature continued up to 4.0 mm of deflection which gave an average force of 6.9N. No distinct change in the inclination of the loading curves was noted.

Figure 5.1

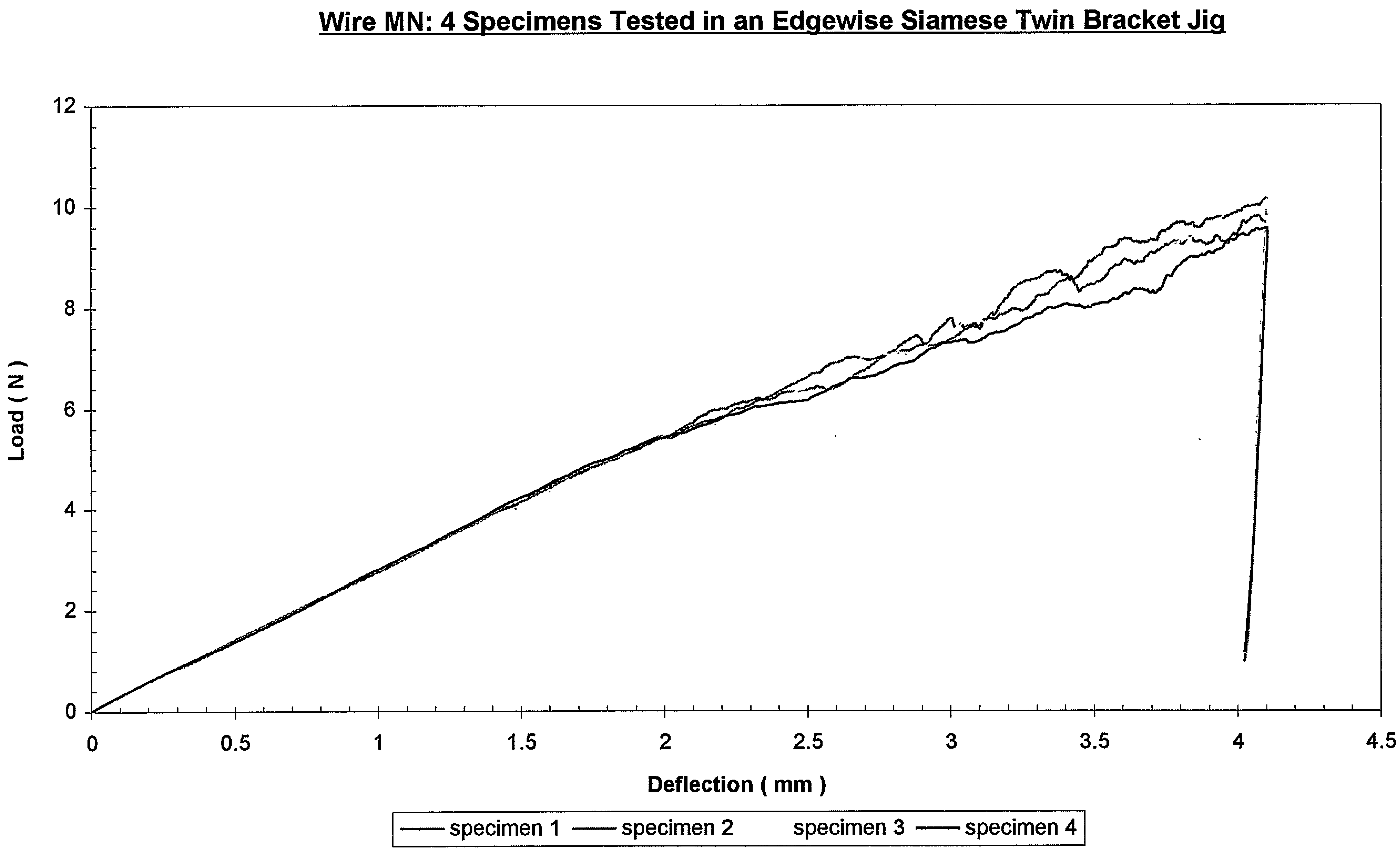
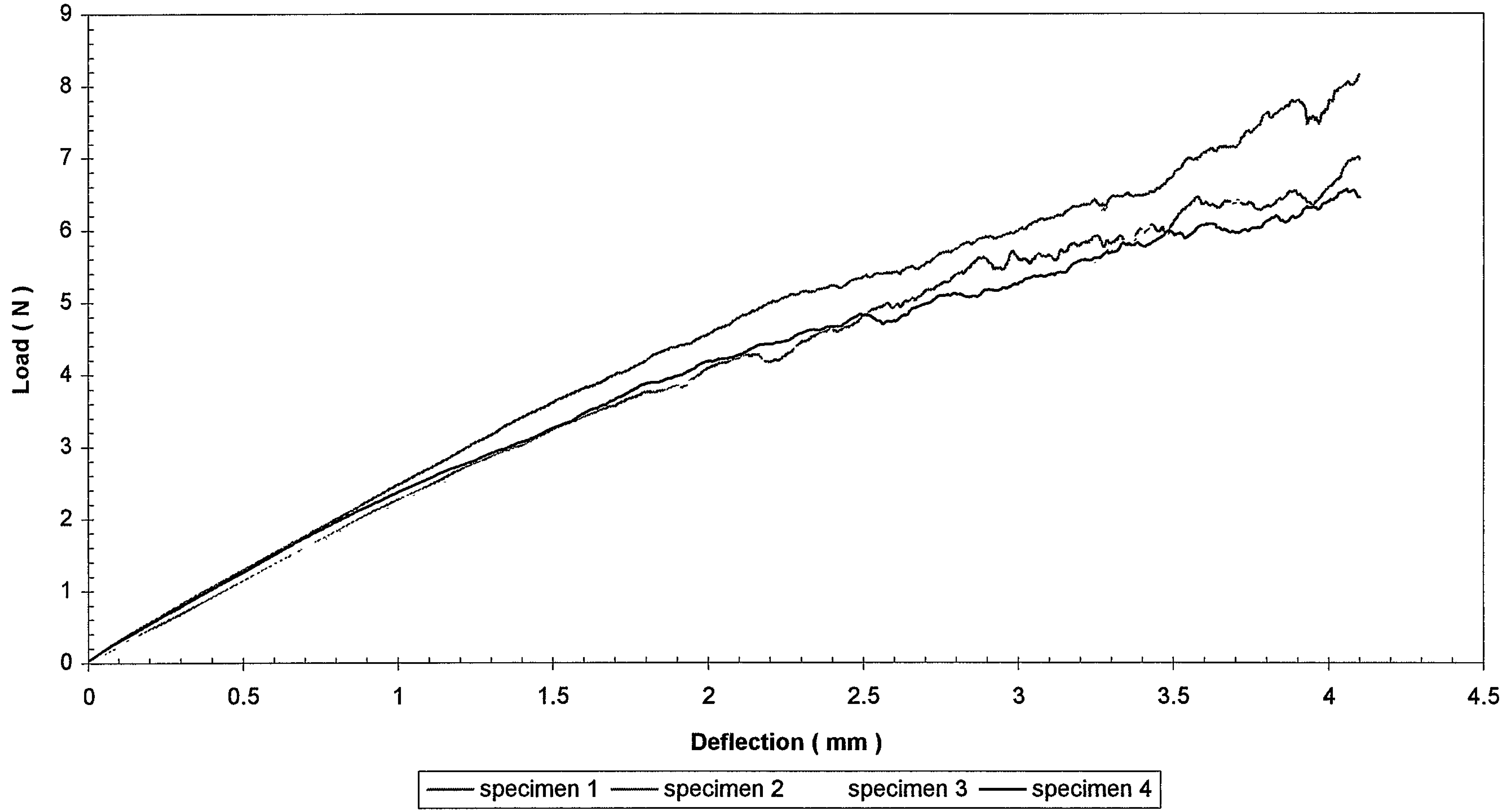

Wire MN: 4 Specimens Tested in an Edgewise Siamese Twin Bracket Jig
Load (N)
0
2
4
6
8
10
12
0
0.5
1
1.5
2
2.5
3
3.5
4
4.5
Deflection (mm)
specimen 1
specimen 2
specimen 3
specimen 4

Figure 5.2

Wire ME: 4 Specimens Tested in an Edgewise Siamese Twin Bracket Jig



All specimens of wire CX, as depicted in Figure 5.3, showed a more consistent plot. Up to 1.0 mm of deflection the curves closely matched and were linear. At this point a change in the inclination of the curves commenced and after 2.0 mm of deflection, greater variation of the curves was displayed due to slippage. This feature continued up to the point of maximum loading at 4.0 mm of deflection, which produced an average force of 6.2N. The unloading portion of these curves produced forces that were greatly reduced in comparison with those obtained during loading. All specimens displayed a plateau region as they were unloaded from 4.0 mm to 1.0 mm of deflection. At approximately 0.8 mm, the inclination of the plot changed decisively and continued back almost to the origin. This section resembled the slope of the initial loading plots.

As demonstrated in Figure 5.4, all curves of wire RL were closely correlated and linear up to 1.0 mm of deflection. Thereafter, a change in the inclination of the curves resulted. After 2.0 mm of deflection, variation became evident due to slippage. Irregular plots were recorded toward the point of maximum loading at 4.0 mm of deflection, with an average force of 5.8N. The unloading portion of the curve produced forces that were greatly reduced in comparison to those obtained during loading. All specimens displayed a plateau region as they were unloaded from 4.0 mm to 1.0 mm of deflection. At approximately 0.8 mm, the inclination of the plot changed convincingly and continued back almost to the origin. This final section resembled that of the initial loading plot up to 1.0 mm.

The most consistent of results were achieved by wire SA (Figure 5.5). Up to 1.0 mm of deflection, the curves almost superimposed. After this point a change in the inclination of the curves resulted. After 2.0 mm of deflection, variation was increased towards the point of maximum loading at 4.0 mm. The average force recorded at this point was the lowest (4.0N), amongst the brands tested. The unloading portion of the curve produced forces that were greatly reduced in comparison to those obtained during loading and displayed the most stable plateaus back towards 1.0 mm of deflection. The final linear section did not resemble the initial loading plot and was very short. It began at only 0.25 mm of the last deflection, at forces less than 0.5N (50g).

Figure 5.3

Wire CX: 4 Specimens Tested in an Edgewise Siamese Twin Bracket Jig

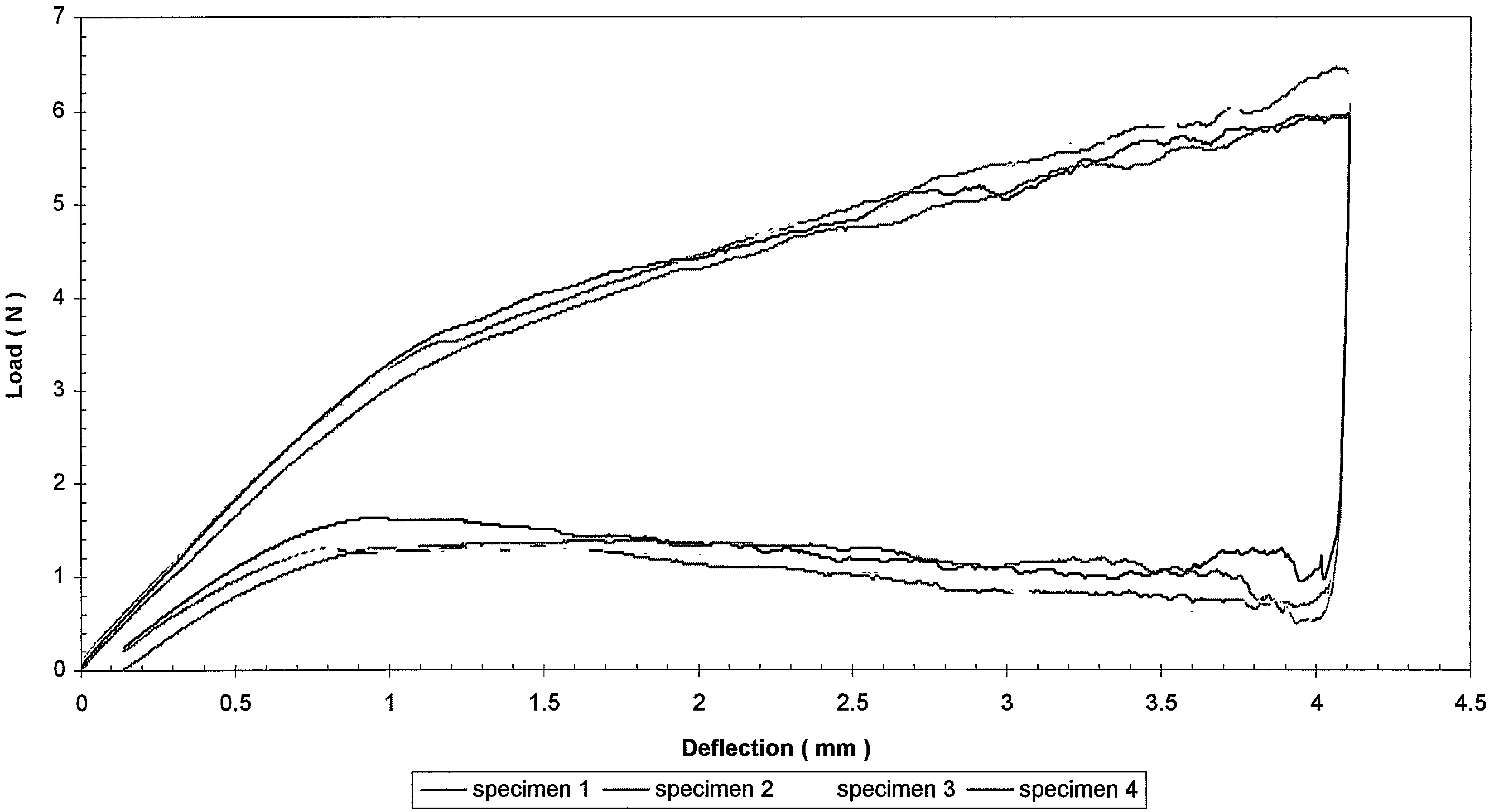
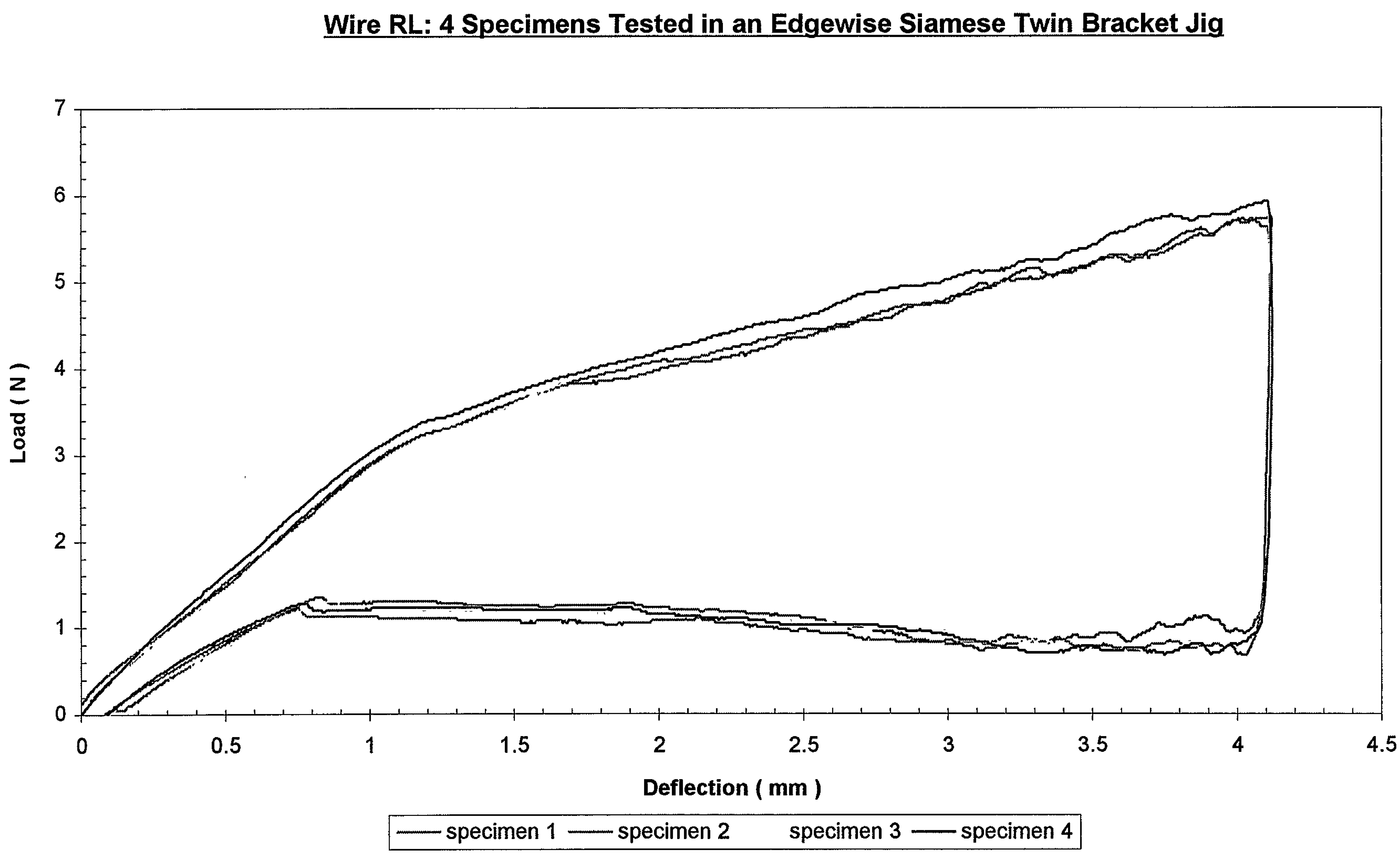


Figure 5.4



Wire RL: 4 Specimens Tested in an Edgewise Siamese Twin Bracket Jig
Load (N)
7
6
5
4
3
2
1
0
0
0.5
1
1.5
2
2.5
3
3.5
4
4.5
Deflection (mm)
specimen 1
specimen 2
specimen 3
specimen 4

Figure 5.5

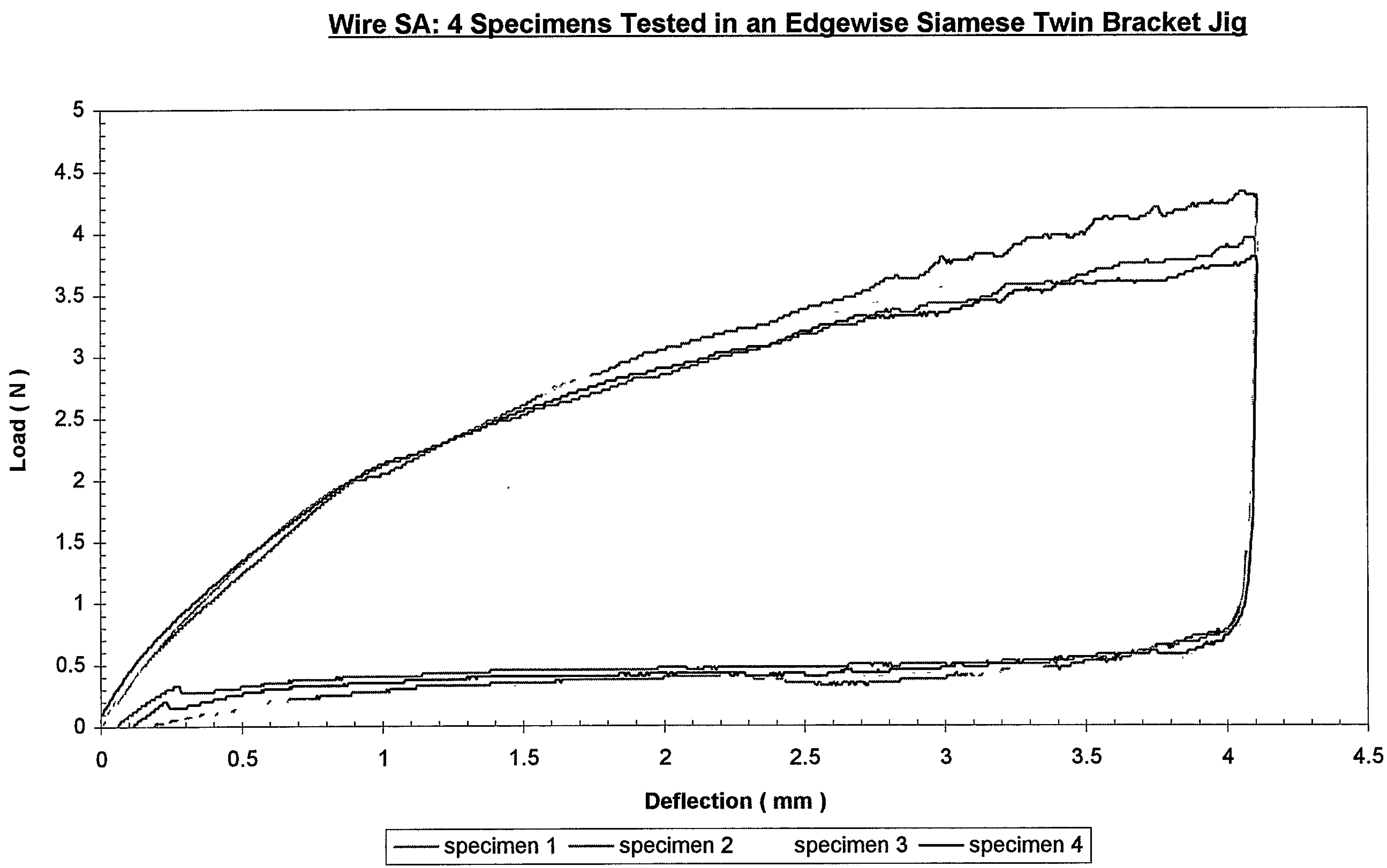

Wire SA: 4 Specimens Tested in an Edgewise Siamese Twin Bracket Jig
Load (N)
5
4.5
4
3.5
3
2.5
2
1.5
1
0.5
0
0
0.5
1
1.5
2
2.5
3
3.5
4
4.5
Deflection (mm)
specimen 1
specimen 2
specimen 3
specimen 4

Figure 5.6

Average plots of tested Wires in an Edgwise Siamese Twin Bracket Jig

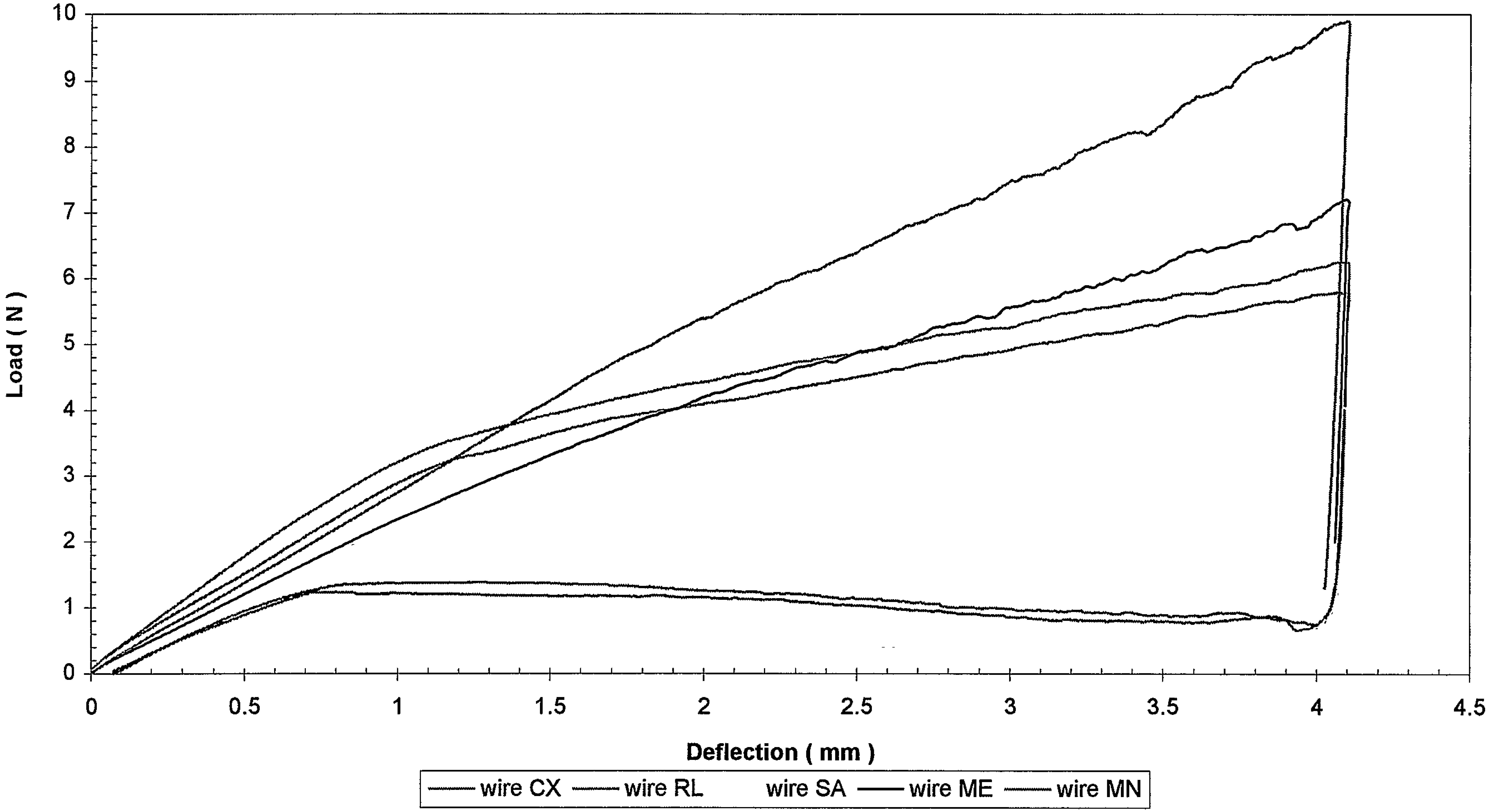


Figure 5.6 shows an 'averaged' load-deflection curve for each of the five brands of wire tested. It provides an insight into the difference in behaviour of the various proprietary brands and the forces generated by these wires. All wires demonstrated a relatively linear relationship up to 1.0 mm deflection. Near this point wires CX, RL and SA showed a distinct change in the inclination of the plot which was maintained until 4.0 mm of deflection was reached. After approximately 3N, however, the effects of slippage caused erratic plots for each wire on the load-deflection curve and continued up to the point of maximum loading. Erratic plots were also recognised on the unloading curve, but to a lesser extent.

Average forces produced at 1.0, 2.0, 3.0 and 4.0 mm of deflection, during loading of the wires are displayed in Table 5.1. The highest load achieved at 1.0 mm of deflection was by wire CX (3.2N; 320g), followed by RL (2.9N), then MN (2.7N), ME (2.3N), and finally SA (2.1N). At a maximum deflection of 4.0 mm, the highest load was achieved by wire MN (9.7N), followed by ME (6.9N), then CX (6.2N), RL (5.8N) and finally SA (4.0N).

Table 5.1 Average force (N) produced at various deflections on the loading curve for the tested wires in Experiment 1.

	Force (N) at various deflections for a 14.0 mm span			
Wire	*1.0 mm*	*2.0 mm*	*3.0 mm*	*4.0 mm*
MN	2.7 ± 0.08	5.4 ± 0.06	7.5 ± 0.21	9.7 ± 0.32
ME	2.3 ± 0.11	4.2 ± 0.25	5.6 ± 0.32	6.9 ± 0.61
CX	3.2 ± 0.12	4.4 ± 0.09	5.3 ± 0.19	6.2 ± 0.28
RL	2.9 ± 0.11	4.1 ± 0.09	4.9 ± 0.15	5.8 ± 0.07
SA	2.1 ± 0.06	2.9 ± 0.09	3.5 ± 0.02	4.0 ± 0.21

No unloading curves were recorded for wires MN and ME as the applied load rapidly decreased to the limit of the programmed safety cut-out on the testing machine, due to permanent deformation of the specimens. Unloading curves were obtained for wires CX, RL and SA. Average forces produced at 4.0, 3.0, 2.0 and 1.0 mm of deflection, during unloading of the wires are summarised in Table 5.2. The highest load at 4.0 mm of deflection was wire CX (0.8N; 80g), followed by wire

RL (0.7N) and wire SA (0.7N). The same order was maintained for 1.0 mm of deflection, with the highest load was recorded by wire CX (1.4N), followed by wire RL (1.2N) and finally wire SA (0.3N).

Table 5.2 Average force (N) produced at various deflections on the unloading curve for the tested wires in Experiment 1.

Wire	*Force (N) at various deflections for a 14.0 mm span*			
	4.0 mm	*3.0 mm*	*2.0 mm*	*1.0 mm*
CX	0.8 ± 0.24	1.0 ± 0.15	1.3 ± 0.10	1.4 ± 0.16
RL	0.7 ± 0.17	0.9 ± 0.06	1.2 ± 0.06	1.2 ± 0.07
SA	0.7 ± 0.09	0.5 ± 0.05	0.4 ± 0.04	0.3 ± 0.08

5.2 Modulus of Elasticity

The modulus of elasticity is a measure of stiffness and was calculated from the data recorded for the linear section of the initial loading curve for each specimen tested in Experiment 1. Results are shown in Table 5.3 below.

Table 5.3 Modulus of Elasticity (GPa) for all samples tested.

Wire	*Modulus of Elasticity (GPa)*				
	sample 1	*sample 2*	*sample 3*	*sample 4*	*average*
MN	112.05	112.56	107.41	106.77	109.70
ME	98.91	92.86	91.98	97.32	95.27
CX	132.61	134.28	142.82	142.82	138.13
RL	113.05	107.11	105.61	123.64	112.35
SA	82.43	89.30	92.73	94.9	89.84

Wire CX displayed the highest modulus of elasticity with an average of 138.13GPa. In descending order, the next wire was RL, followed by MN, then ME and finally wire SA with an average of 89.84GPa.

5.3 Experiment 2 (Vice Grip Jig)

Appendix 2 shows the average load deflection curves of the five wires tested in the vice grip jig. This apparatus was designed to eliminate slippage of the specimens which was noted in Experiment 1. The modification did not prove to be successful, as slippage still occurred in all the tested wires. As the specimens were tightly constrained, high loads were recorded for 1.0, 2.0, 3.0, and 4.0 mm of deflection and no unloading data were obtained. The results were confused and varied. As such, further analyses were not warranted and the graphs are contained in appendices 2 to 7 to demonstrate that slippage could not be eliminated from three point bending testing, even when a vice was used. Erratic peaks occurring in the graph in Appendix 3, were due to slippage of the specimen wire within the testing jig. It is also worth referring to the large forces produced in Experiment 2, which are summarised in Table 5.4.

Table 5.4 Average force (N) produced at various deflections on the loading curve for the tested wires in Experiment 2.

	Force (N) at various deflections for a 14.0 mm span			
Wire	*1.0 mm*	*2.0 mm*	*3.0 mm*	*4.0 mm*
MN	12.6 ± 0.92	32.6 ± 7.59	52.0 ± 12.07	73.1 ± 20.51
ME	12.2 ± 1.17	32.8 ± 3.04	51.6 ± 15.49	67.8 ± 17.81
CX	17.0 ± 2.16	31.3 ± 4.44	47.4 ± 6.78	62.9 ± 9.60
RL	11.2 ± 0.69	23.8 ± 2.61	35.3 ± 5.01	47.6 ± 7.31
SA	10.7 ± 1.11	21.2 ± 2.43	30.3 ± 4.68	39.1 ± 7.19

5.4 Statistical Analysis

For statistical analysis, loading in the vice grip jig was designated as Test 1 (T1), loading in the edgewise siamese twin bracket jig was Test 2 (T2), and unloading in the edgewise siamese twin bracket jig was Test 3 (T3). Four samples for each of the wires CX, RL and SA were evaluated for the force they produced at 1.0, 2.0, 3.0 and 4.0 mm of deflection. Variation in the force generated by these wires was investigated by a three way analysis of variance (ANOVA). The ANOVA examined whether the source of variation resulted from either T1, T2 or T3, the brand (CX, RL, SA), or the specimens used (S1, S2, S3 or S4). The results are provided in Table 5.5

Table 5.5 Three way analysis of variance for force by test; (loading in a vice grip jig; loading in an edgewise siamese twin bracket jig; unloading in an edgewise siamese twin bracket jig), brand and sample.

Source of variation	*Sum of squares*	*Degrees of freedom*	*Mean square*	*F ratio*	*p-value*
Main effects	27891.410	7	3984.487	41.631	0.000
Test	26398.877	2	13199.438	137.913	0.000
Brand	1348.703	2	674.351	7.046	0.001
Sample	143.830	3	47.943	0.501	0.682
2-way interactions	2744.658	16	171.541	1.792	0.041
Test Brand	2005.856	4	501.464	5.239	0.001
Test Sample	272.772	6	45.462	0.475	0.826
Brand Sample	466.030	6	77.672	0.812	0.563
3-way interactions	944.083	12	78.674	0.822	0.627
Test Brand Sample	944.083	12	78.674	0.822	0.627

Not significant: $p \geq 0.05$; Significant: $p \leq 0.01$; Highly significant: $p \leq 0.001$

The main effects were highly significant differences between tests T1, T2, and T3, ($F = 137.913$; $p \leq 0.001$) and between brands ($F = 7.046$; $p \leq 0.001$). No significant differences were detected within the samples ($F = 0.501$; $p \geq 0.05$).

Although apparent from a comparison between Tables 5.1 and 5.4, the significance of the difference between variation in force for loading in a vice grip jig (T1) and in an edgewise siamese twin bracket (T2) was investigated by a three way ANOVA. The source of variation which resulted from either the type of test (T1 or T2), the brand (CX, RL, SA), or the samples (S1, S2, S3 or S4), were considered. The results are contained in Table 5.6.

Table 5.6 Three way analysis of variance for force by test; (loading in a vice grip jig; loading in an edgewise siamese twin bracket jig), brand and sample.

Source of variation			*Sum of squares*	*Degrees of freedom*	*Mean square*	*F ratio*	*p-value*
Main effects			29886.595	7	4269.514	21.649	0.000
	Test		26963.354	1	26963.354	136.719	0.000
	Brand		2869.702	3	956.567	4.850	0.003
	Sample		53.539	3	17.846	0.090	0.965
2-way interactions			3752.022	15	250.135	1.268	0.237
Test	*Brand*		1859.054	3	619.685	3.142	0.029
Test	*Sample*		47.881	3	15.960	0.081	0.970
Brand	*Sample*		1845.088	9	205.010	1.040	0.415
3-way interactions			1766.141	9	196.238	0.995	0.449
Test	*Brand*	*Sample*	1766.141	9	196.238	0.995	0.449

Not significant: $p \geq 0.05$; Significant: $p \leq 0.01$; Highly significant: $p \leq 0.001$

The main effects on force were a highly significant difference between loading in a vice grip jig (T1) and an edgewise siamese twin bracket jig (T2); ($F = 136.719$; $p \leq 0.001$) and significant difference between brands ($F = 4.850$; $p \leq 0.001$). No significant differences were detected within the samples ($F = 0.090$; $p \geq 0.05$).

The significance of the difference between variation in force for loading in an edgewise siamese twin bracket (T2) and unloading in the same jig (T3) was investigated by three-way ANOVA. The source of variation which resulted from either the type of treatment (T2 or T3), the brand (CX, RL, SA), or the samples (S1, S2, S3 or S4), were considered. The results are contained in Table 5.7

Table 5.7 Three way analysis of variance for force by test; (loading in an edgewise siamese twin bracket jig; unloading in an edgewise siamese twin bracket jig), brand and sample.

Source of variation	*Sum of squares*	*Degrees of freedom*	*Mean square*	*F ratio*	*p-value*
Main effects	281.796	6	46.966	69.555	0.000
Test	260.371	1	260.371	385.601	0.000
Brand	21.339	2	10.670	15.801	0.000
Sample	0.086	3	0.029	0.042	0.988
2-way interactions	3.690	11	0.335	0.497	0.899
Test Brand	3.285	2	1.164	2.432	0.095
Test Sample	0.339	3	0.113	0.167	0.918
Brand Sample	0.066	6	0.011	0.016	1.000
3-way interactions	0.452	6	0.075	0.111	0.995
Test Brand Sample	0.452	6	0.075	0.111	0.995

Not significant: $p \geq 0.05$; Significant: $p \leq 0.01$; Highly significant: $p \leq 0.001$

The main effects on force were a highly significant difference between loading (T2) and unloading (T3) in an edgewise siamese twin bracket jig ($F = 385.601$; $p \leq 0.001$). A highly significant difference in force was also found to be due to brand ($F = 15.801$; $p \leq 0.001$). No significant differences were detected within the samples ($F = 0.042$; $p \geq 0.05$).

The significance of the difference between variation in force for unloading in an edgewise siamese twin bracket (T3) was investigated by a two-way ANOVA. The source of variation which resulted from either the brand (CX, RL, SA), or the samples (S1, S2, S3 or S4), were considered. The results are contained in Table 5.8

Table 5.8 Two way analysis of variance for force by brand and sample for unloading in an edgewise siamese twin bracket jig.

Source of variation		*Sum of squares*	*Degrees of freedom*	*Mean square*	*F ratio*	*p-value*
Main effects		4.093	5	0.819	14.634	0.000
	Brand	3.978	2	1.989	35.563	0.000
	Sample	0.114	3	0.038	0.682	0.569
2-way interactions		0.284	6	0.047	0.845	0.544
Brand	*Sample*	0.284	6	0.047	0.845	0.544

Not significant: $p \geq 0.05$; Significant: $p \leq 0.01$; Highly significant: $p \leq 0.001$

The main effect on force was a highly significant difference between brands ($F = 35.563$; $p \leq 0.001$). No significant difference was detected within the samples ($F = 0.682$; $p \geq 0.05$).

The difference between brands was investigated using a multiple comparison (Duncan) test technique. A significant difference ($p \leq 0.05$) existed between brands CX and SA. No significant differences were found between brands SA and RL or between brands CX and RL. The results are contained in Table 5.9.

Table 5.9 Multiple comparison (Duncan) test of brands.

Mean	*Brand*	*Brand SA*	*Brand RL*	*Brand CX*
9.810	SA	NS	NS	*
13.649	RL	NS	NS	NS
18.114	CX	*	NS	NS

*NS = Not Significant; * = Significant: $p \leq 0.05$*

Chapter Six
DISCUSSION

6.1 Compared Results

There is enormous variation in test design and reported results. These factors prevent an accurate comparison between the present study and those previously reported. The majority of the discussion will, therefore, focus on the difference between the results obtained by tensile testing (Stanton 1995) and the present investigation, after two relevant published papers have been referred to.

Watanabe (1982) presented load / deflection curves of a first generation Ni-Ti wire, assumed to be Nitinol and a newly developed Ni-Ti wire, assumed to be Japanese Ni-Ti. At 1.0 and 2.0 mm of deflection, by three point bending and without ligation of the wire, the load for both of these wires was similar to the findings in the present study. Watanabe (1982) recorded approximately 2.0N, (200g), for both types of Ni-Ti wire at 1.0 mm of deflection and at 2.0 mm, the load was approximately 3.5N for Nitinol and 2.5N for Sentalloy. The present study recorded approximately 2.7N, (270g), for wire MN, and 2.1N for wire SA, at 1.0 mm of deflection and approximately 5.4N and 2.9N, respectively, at 2.0 mm of deflection. Compared to this present investigation, higher loads were recorded by Watanabe (1982) during unloading and also when ligation was used to secure the test specimen into the bracket jig.

Miura *et al.* (1986) observed 7.9N for Nitinol and 6.5N for Japanese Ni-Ti wire at 2.0 mm deflection on the loading curve, whereas the present study produced 5.4N for Nitinol, and 3.0N for Japanese Ni-Ti. During unloading, Miura *et al.* (1986) reported approximately 2.5N for Japanese Ni-Ti whereas the present study achieved 0.4N. Variation in these results may be due to differences in the amount of ligation applied, batch variation, or on going development of Japanese Ni-Ti by its manufacturers.

6.2 Theoretical and Experimental Force Values

The intent of the present study has been a comparison between the theoretical force for the start of the austenitic to martensitic phase transformation derived from tensile testing data and that derived from Experiment 1 for this mark. Table 6.1 compares the predicted and experimental values, where the 0.1% pseudo yield stress provided by Stanton (1995) was adopted.

Table 6.1 Comparison between 0.1% pseudo yield stress, predicted load (N) produced by three point bending at this stress, and the load obtained in Experiment 1 for loading at 1.0 mm deflection and unloading at 3.0 mm of deflection.

Wire	*0.1% pseudo yield stress (MPa)*	*Predicted load (N) for transformation*	*Load (N) at 1.0 mm deflection for loading*	*Load (N) at 3.0 mm deflection for unloading*
MN	-	-	2.69 ± 0.09	-
ME	433	0.84	2.33 ± 0.11	-
CX	329	0.64	3.20 ± 0.14	0.98 ± 0.15
RL	433	0.84	2.89 ± 0.11	0.86 ± 0.06
SA	296	0.57	2.11 ± 0.06	0.41 ± 0.07

Use of Equation 3.3 enabled the calculation of the theoretical force for the start of the austenitic to martensitc phase transformation during three point bending, for a 14.0 mm span. For all wires, the experimental data was significantly higher than theory predicted. These findings are in general agreement with those authors who have claimed that the forces generated in bending are not accurately predicted from tensile properties and recommended instead, bend testing (Goldberg, Morton and Burstone 1983; Goldberg, Burstone and Koenig 1983; Asgharnia and Brantley 1986; Khier, Brantley and Fournelle 1991).

The discrepancy that existed between predicted and experimental forces has been raised by a number of authors (Burstone and Goldberg 1983; Goldberg, Morton and Burstone 1983; Goldberg, Burstone and Koenig 1983). These authors, referring to their own test designs, have proposed a variety of mechanisms to account for the inconsistency between experimental and predicted forces, which can be summarised as:

- Erroneous engineering theory which has assumed complete elastic behaviour through all cross sections of the wire;
- Small deflection theory which is invalidated by deflections of the test wire greater than 10% of the span length;
- Anisotropic properties within the wire due to the effects of heat treatment, cold working and drawing in their production; and
- Non uniform stress distribution across the wire during bending.

Of these factors, the first proposal is not relevant to this investigation as the test wires did behave in a predominantly elastic manner. The second proposal was directed towards those studies which used cantilever testing and the bending theory contained within the American Dental Association Specification Number 32. As that protocol was not adopted in the present study, it is not relevant to this investigation. Although impossible to control, the influence of batch variation should have been significantly reduced by using the same batch of wire for both tensile and three point bending tests. Therefore, in this investigation, a significant effect was contributed by the last proposal, whereby a non-uniform stress distribution was largely responsible for the differences between experimental and predicted forces.

As demonstrated in Figure 3.4, a bent wire has its outside surface tensed, whilst its inside curved surface is compressed, and midway between the two extremes is the neutral axis which divides the bending stress into tensile and compressive halves. For three point bending, maximum stress is developed at the surface of the wire, directly opposite the applied load and it gradually decreases to zero at the supported ends as represented in Figure 3.5. Therefore, the first point on the wire to begin a stress induced austenitic to martensitic phase transformation is that single point on the surface of the wire, directly opposite the applied load, which has reached the threshold stress. As the applied load is increased, a larger section of the wire can be induced by elevating a greater area of the wire to the threshold stress. Such complex stress distribution patterns produced during bending may be the key determinant of the force required for the progress of the austenitic to martensitic phase transformation. When a pseudoplastic Ni-Ti wire was loaded in tension, a uniform tensile stress was distributed throughout the entire wire and an almost instantaneous phase transition resulted at the 0.1% pseudo yield stress. After this point a pseudoplastic plateau was obtained on the graph (Stanton 1995). When the same wire was subjected to three point bending no immediate phase transition was observed (Figure 5.6). Instead, a gradual change was noted, which indicated that the complex stress distribution pattern produced during bending actively inhibited the instantaneous phase transformation that was observed in tension. As a result, much higher forces were recorded experimentally during three point bend testing for the start of the austenitic to martensitic phase transformation than theory would have predicted from tensile data. Similar discrepancies between tensile and bend testing have been reported by Goldberg, Burstone and Koenig (1983).

After reviewing Figures 5.3 to 5.5, 1.0 mm of deflection was selected as a representative reference point for the start of this phase transformation during bending, according to the protocol detailed in paragraph 4.3. Part of the discrepancy that existed between predicted and experimental findings may be explained by a lack of sensitivity of this chosen point to the actual start of the transformation. Even if, however, 0.5 mm deflection was considered, which is well below any change in the slope of the loading curve, a large discrepancy between predicted and experimental results would still remain.

Table 6.1 also compared the predicted force for the start of the austenitic to martensitic phase transformation to that force achieved experimentally for 3.0 mm of deflection on the unloading curve. This point on the unloading curve was selected as representative of the force produced by each specimen along the pseudoplastic plateau. For wires CX, RL and SA, forces produced at 3.0 mm of deflection were very similar to the prediction. From this relationship, it would seem that once the martensitic phase had been established, bending stresses did not greatly inhibit the reverse transition of martensite back to austenite during unloading. A satisfactory estimate of the forces generated by Ni-Ti wires as they unload across the pseudoplastic plateau was unexpectedly provided by the 0.1% pseudo yield stress data. This feature was considered to be clinically relevant, as it is the unloading of the wire which imparts a force to the tooth during orthodontic treatment, not the loading force. The 0.1% pseudo yield stress data can therefore be used to provide a reasonable estimate of the force produced by Ni-Ti wires in clinical use.

The 0.1% pseudo yield stress data and Equation 3.3 when combined should provide a satisfactory estimate of the force generated by pseudoplastic wires as they unload across the pseudoplastic range for any span length that is factored into the formula. As the load produced by the tested wires does not vary considerably in the pseudoplastic plateau region, the predicted force should be a reasonable estimate for the entire extent of wire deflection which may occur in the pseudoplastic range. That is, the actual amount of wire deflection is largely irrelevant whilst in this range. A benefit of this approach is the elimination of the need for repeated bending experiments at different span lengths to cover the wide range of interbracket distances which occur clinically. Table 6.2 provides an example of the force estimated for varying span lengths for wires CX, RL and SA. The force generated across the pseudoplastic plateau in Experiment 1 by these wires was included for comparative purposes.

Table 6.2 Comparison between the experimental unloading force range for a 14 mm span and the predicted force for a variety of interbracket spans which may be experienced clinically.

	Tested force (g)	*Predicted force (g)*						
Wire	*14 mm*	*12 mm*	*14 mm*	*15 mm*	*16 mm*	*23 mm*	*24 mm*	*25 mm*
CX	139 to 76	76	65	61	56	39	38	36
RL	124 to 76	100	85	80	75	52	50	47
SA	30 to 65	68	58	55	51	36	34	33

A 12.0 mm interbracket span may be encountered when aligning imbricated mandibular incisor teeth. Crowding in the premolar region in non-extraction treatment is represented by the 14.0, 15.0 and 16.0 mm spans. The increased span length for extraction therapy is represented by the 23.0, 24.0 and 25.0 mm interbracket spans. For the tested wires, at all spans and at all deflections within the pseudoplastic range, physiologically acceptable forces were generated for orthodontic movement of teeth (Quinn and Yoshikawa, 1985; Proffit, 1986; Reitan and Rygh, 1994).

6.3 Clinical Significance

Rock and Wilson (1988) found that an orthodontic archwire in clinical use is far more rigid than beam tests would suggest. They postulated two variables which may explain the difference:

- An increase in rigidity due to the shape of the archwire;
- The restriction caused by ligation of the wire into the brackets.

Many factors are involved in the clinical use of Ni-Ti alloy archwires which may affect force levels (Burstone and Koenig 1974; Waters, Stephens and Houston 1975; Burstone and Goldberg 1983). These factors may be summarised as:

- Curvature of the archwire;
- Orthodontic bracket type, width and angulation;
- Friction within the brackets which limits sliding of the archwire;
- Friction produced by the type of ligation;
- Effects of occlusion;
- Inter-bracket distance or span length;
- Effects of plastic deformation.

Although the exact magnitude of the force delivered by an unloading pseudoplastic Ni-Ti wire to a mal-positioned tooth in the mouth cannot be accurately calculated from laboratory testing, guidelines for clinical use can be established. For all the 0.4 mm diameter pseudoplastic wires tested (CX, RL and SA), clinically acceptable forces were produced as they unloaded from either 1.0, 2.0, 3.0 or 4.0 mm of deflection, when the wire was able to slide through the bracket (Experiment 1). If the wire was held rigidly and slippage was limited, inordinately high forces were produced from all the wires. Although this arrangement is not entirely related to the clinical situation it is, nevertheless, recommended that an archwire should not be ligated tightly to the aligned teeth before engagement of a mal-aligned tooth.

6.4 Published Bending Test Standards

All of the published standards were produced before Ni-Ti wires were commonly used in clinical practice. As such, the protocols for these tests were not considered suitable for the evaluation of highly elastic wires. Where loading of the wire was specified, it was in a cantilever configuration, which did not detect the presence of superelastic behaviour or the pseudoplastic range. Because of these considerations, Australian Standard (AS 1964 - 1977), British Standards (BS 4545 - 1970 and BS 3507 : 1976), and the American Dental Association Specification Number 32 for Orthodontic Wires Not Containing Precious Metals (1977) were not considered appropriate for use in this study.

Although the majority of researchers have chosen ADA specification number 32, or a modified version using a shorter span length, many concerns have been raised regarding the validity of data derived from such cantilever testing. Miura *et al.* (1986) contend that due to the good springback properties of all Ni-Ti alloy wires, including those that do not undergo pseudoplastic behaviour, a false-positive result for the presence of pseudoplasticity can occur during cantilever testing from inherent errors. Those authors also claim that during testing according to ADA Specification Number 32, the Ni-Ti alloy wire bends under loading and the point of application begins to slide giving an effective increase in the length of the test span and, moreover, a decrease in the load. These intrinsic problems with the test design produce data that is unreliable for determining pseudoplastic behaviour.

Other criticisms of Specification 32 relate to errors in the calculation of the modulus of elasticity (Nikoli Anderson and Messersmith 1988). Sizeable curvatures and accompanying deflections of Ni-Ti alloy wires produced by the test itself are the cause of such errors. Also, it has been argued that data derived from deflection of the specimen beyond 2.5 mm should be excluded from the calculation of the modulus of elasticity value, as correct application of the beam deformation formula assumes maximum deflection of the beam does not exceed one-tenth of its length (Nikoli, *et al.* 1988). Calculated values for the modulus of elasticity (E) and flexural yield strength (YS) have been shown to vary with the span length of the test wire when applying the mathematical formula in ADA Specification Number 32 (Asgharnia and Brantley 1986). Since E and YS are intrinsic material properties, and are independent of span length, variation in these results highlights deficiencies present in the bending mechanics analysis employed in Specification 32.

Cantilever bend testing is largely irrelevant to continuous archwire mechanics used in clinical orthodontics. Continuous archwires are never used as cantilevers, but rather as short spans or beams in between adjacent orthodontic brackets. Therefore, a three point beam arrangement was considered to be a more relevant testing model.

6.5 Intratest Variation Experiment 1

Intrabatch variation in the load-deflection characteristics of nickel-titanium wires was reported by Tonner and Waters (1994b). The effect of temperature on the loading and unloading characteristics of superelastic Ni-Ti wires was investigated by these authors during three point bend testing. Wide variation in the plot for replicate specimens at a given temperature was noted. The degree of variation for the initial slope of the loading curve, and for the unloading pseudoplastic plateau, was considered greater than that which normally existed between replicate specimens when compared to 18/8 stainless steel. Variation for the initial slope of the loading curve was 7.5% and the unloading plateau was 10%. According to these authors the stress at which the reverse martensitic transformation occurred appeared to be more dependent on the processing heat treatment and work hardening history of the wire, rather than on the diameter.

Analysis of variance for the present study found, however, that the variation in the force produced by the tested Ni-Ti wires as they were loaded and unloaded was significantly related to brand, but not sample (Table 5.1). Plots of the individual specimens for each brand can be seen in Figures 5.1 to 5.5. Descriptive statistics of the four specimens tested for each of the wires (CX, RL, SA) are presented in Table 6.3. The force produced at 1.0 mm deflection for loading is representative of the forward austenitic to martensitic transformation and the force produced at 1.0 mm of unloading, (that is at 3.0mm of deflection on the unloading curve), is representative of the reverse martensitic to austenitic transition. The force at both of these marks for all specimens of a particular brand is remarkably similar as noted by the low standard deviations and sample variances listed in Table 6.3. Variation is considerably less than that reported by Tonner and Waters (1994b).

Table 6.3 Descriptive statistics of load (N) for wires (CX, RL, SA), samples (S1, S2, S3, S4) at 1.0 mm deflection on the loading curve and 3.0 mm on the unloading curve.

	Wire	*Mean*	*Standard Deviation*	*Sample Variance*	*Range*	*Min*	*Max*
Loading at 1.0 mm	*CX*	3.20	0.14	0.02	0.30	3.00	3.30
	RL	2.89	0.11	0.01	0.28	2.75	3.03
	SA	2.11	0.06	0.00	0.15	2.05	2.20
Unloading at 3.0 mm	*CX*	0.98	0.15	0.02	0.30	0.83	1.13
	RL	0.86	0.06	0.00	0.13	0.80	0.93
	SA	0.42	0.07	0.00	0.15	0.35	0.50

6.6 Friction

Friction is unpredictable and multifactorial in nature representing a complex interrelationship of various factors (Ireland, Sherriff and McDonald, 1991). Ho and West (1991) showed that all tested factors and interactions had a statistically significant effect on friction. Therefore, conclusions proposed by various authors may only relate to the testing conditions of their own particular study. Tidy (1989) claimed that friction remains a constant proportion of the total force applied That is, friction rises in proportion to the applied force. This feature may have been operating after approximately 2.0 mm of deflection was reached for each of the tested wires in Experiment 1, as slippage became apparent after this point. As reflected in the plotted graphs, recordings became more erratic after 2.0 mm of deflection, but continued on an essentially linear inclination up to the unloading point. Possibly this linear extension of the graph was due to the combined effects of slippage and friction. Similarly, these two factors may have contributed to the long pseudoplastic plateau region as the wire unloaded.

6.7 Modulus Of Elasticity

The modulus of elasticity was calculated according to the protocol detailed in paragraph 4.3, using the equation listed below:

Equation 6.1

$$E = \frac{4\,L^3}{3\,\pi\,D^4} \times \frac{F}{\Delta l}$$

where:

(E) is the modulus of elasticity.

(L) is the interbracket span between the two supports.

(F) is either the force generated or the load applied.

(D) is the diameter of the wire.

(Δl) is the vertical deflection of the wire.

In this study wire MN recorded an average value of 109.70 GPa, which is considerably greater than 44.4 GPa, as reported by Kusy and Stush (1987). The highest modulus of elasticity was wire CX (138.13 GPa) and the lowest was wire SA (89.84 GPa). Such differences are not surprising given that small variations in the manufacturing procedure or composition of Ni-Ti wires can have enormous effects on phase transformations (Bradley, Brantley and Culbertson, 1996).

6.8 Statistical Significance

Highly significant differences in force were found between loading and unloading of the wire in an edgewise siamese twin bracket jig ($F = 385.601$; $p \leq 0.001$). A highly significant difference in force was also found to be due to brand ($F = 15.801$; $p \leq 0.001$). However, no significant differences in force were attributed to variation in the samples (Table 5.6). Statistical analysis of Experiment 1 indicated that there was a difference between brands which are currently commercially available, and that quality control within the batches was acceptable. Although statistically significant differences were detected between the brands tested, these differences may not be clinically relevant. Similar forces were achieved for similar amounts of activation for all of the samples evaluated. A small but statistically insignificant amount of variation between samples can be inferred from the low standard deviations and sample variances listed in Table 6.3.

6.9 Technical Difficulties

No unloading curve was recorded for wires MN and ME due to technical difficulties with the testing machine's software. Communication with the manufacturer in Japan failed to resolve the impasse of a pre-programmed automatic safety cut-out which could not be altered by the user. An improved software package which permits cyclical testing of the specimen was not available at the time when this present study was conducted.

Chapter Seven

SUMMARY

This study investigated the bending behaviour of five Ni-Ti orthodontic wires. They were selected on the basis of previous tensile testing and represented certain aspects of the pseudoplastic plateaus produced by Stanton (1995). Wire MN was a non pseudoplastic Ni-Ti wire included as a control. Wire ME was included because it had produced a plateau with the shortest elongation and also had recorded the highest stress. Wire SA was included because it had recorded a plateau with the lowest stress. The longest pseudoplastic elongation was represented by Wire RL. Wire CX was included because it recorded an average result.

Specifically, this study confirmed the presence of pseudoplastic behaviour during three point bending and calculated the force level at which this phenomenon occurred in wires CX, RL and SA for loading and unloading in an edgewise siamese twin bracket jig. In addition, the force produced by 1.0, 2.0, 3.0 and 4.0 mm of deflection for both loading and unloading was established. During loading no immediate austenitic to martensitic phase transformation occurred. Instead, a gradual transition was noted. However, a long pseudoplastic plateau region was recorded on the unloading curve for wires CX, RL and SA.

Pseudoplastic behaviour in Ni-Ti orthodontic wires was reliant upon the austenitic to martensitic phase transformation. Stanton (1995) proposed that this phase transformation commenced at the 0.1% pseudo yield stress in tensile testing. Forces achieved experimentally for the start of the austenitic to martensitic phase transformation during three point bending, (2.1-3.2N), were significantly higher than those predicted from a mathematical calculation based on the 0.1% pseudo yield stress data (0.5-0.8N). Complex stress patterns produced during loading of the wire during three point bending may be the cause of such a discrepancy.

Forces produced during unloading of the tested wires were greatly reduced in comparison with the same activation of the wire when loaded. Of note was the unusual relationship between the 0.1% pseudo yield stress and the unloading pseudoplastic plateau. Although the predicted force for the start of the austenitic phase transition taken from tensile testing did not correlate with the loading curve, this prediction (0.5-0.8N) did show a reasonable approximation to the force at which the reverse martensitic phase transformation occurred on the unloading curve (0.5-1.0N). That is, the 0.1% pseudo yield stress provides a reasonable estimate of the force imparted from a Ni-Ti wire as it unloads across the pseudoplastic plateau.

Interestingly, once the martensitic phase had been established, bending stresses did not greatly inhibit the reverse transition of martensite back to austenite. This feature was considered to be clinically relevant, as it is the unloading of the wire which imparts a force to the tooth during orthodontic treatment, and not the loading force.

Highly significant differences ($p \leq 0.001$) were found in the force produced by the tested wires for loading, unloading and brand. No significant differences were attributed to sample variation. This finding indicated that identifiable variation existed between commercially available proprietary wires, all of which possessed excellent intrabatch quality control. The differences between brands, however, may not be clinically relevant when the force values are compared in table 5.2.

Wires CX, RL and SA produced light forces over a long unloading range which were considered physiologically suitable for tooth movement. Forces ranged from 0.8-0.7N at 4.0 mm of deflection to 1.4-0.3N at 1.0 mm of deflection. Although it is not possible to extrapolate experimental results directly to clinical use, it may be cautiously assumed that these wires are capable of producing a near optimal force for a long range of activation. The tested wires demonstrated, at all deflections within the pseudoplastic range, suitable forces for orthodontic tooth movement.

Three point bending in a vice grip jig is not recommended since Experiment 2 produced widely varied responses. Variation within the samples and between the brands was significant. Differences could be explained by the unreliable nature of this type of bend testing. High values were recorded when no slippage was apparent and lower values were recorded when slippage occurred. Although not addressed by previous investigations, slippage could not be entirely eliminated from three point bend testing, even when the wire specimen was locked in a vice. This finding would suggest that the influence of slippage during three point bend testing has been largely neglected in the literature. Forces increased approximately ten-fold when the wire was rigidly held by the vice grip compared to the edgewise siamese twin bracket grip, in which the wire was allowed to slip.

Chapter Eight
CONCLUSION

Ni-Ti archwires (CX, RL and SA) demonstrated pseudoplastic behaviour during three point bend testing. During loading, the forward austenitic to martensitic crystallographic phase transformation was a gradual transition which did not change immediately at the 0.1% pseudo yield stress, as occurred in tensile testing (Stanton 1995). During unloading of these wires, forces were markedly reduced and a long pseudoplastic plateau region was evident on the curve of the load / deflection graph. From the present study into the bending behaviour of Ni-Ti orthodontic archwires, the following conclusions can be drawn:

- Pseudoplastic behaviour was induced in wires CX, RL and SA during three point bending.

- During loading, the forward austenitic to martensitic transformation occurred gradually and at forces significantly higher than those predicted from the 0.1% pseudo yield stress data.

- During unloading, the reverse martensitic to austenitic transformation occurred rapidly and at forces significantly lower than for the forward transformation.

- Complex patterns of stress distribution are present in a wire during bending, with one-half in compression and the other one-half in tension. This complex pattern of stress may have inhibited the forward austenitic to martensitic transformation, but had negligible effect on the reverse martensitic to austenitic transformation.

- The forces exerted by wires CX, RL and SA across the pseudoplastic plateau during unloading approximated the forces predicted from 0.1% pseudo yield stress tensile data.

- The tested wires demonstrated, at all deflections within the pseudoplastic range, suitable forces for orthodontic tooth movement.

- The 0.1% pseudo yield stress data can provide a reasonable estimate of the force generated by a Ni-Ti wire in clinical use. Although it is not possible to extrapolate experimental findings directly to the clinical situation, the 0.1% pseudo yield stress does provide a useful guide. Use of tensile data removes the complications of slippage, friction and complex stress distributions within the wire, all of which confound a clear interpretation of bending tests.

- Highly significant differences ($F = 385.601$; $p \leq 0.001$) were found between the forces produced by loading and unloading of Ni-Ti wires during three point bending in an edgewise siamese twin bracket jig. An increasing force differential was demonstrated in Figures 5.3 to 5.5 between the loading and unloading pseudoplastic plateaus. Therefore, the null hypothesis that:

  > "The force differential between the loading and unloading pseudoplastic plateaus of a Ni-Ti wire during three point bending remains constant as demonstrated in tensile testing."

  can be confidently rejected.

- Three point bending was a reliable method when the wire was permitted to slide, as in the edgewise siamese twin bracket jig. However, after approximately 2.0 mm of deflection, the load/deflection curve became increasingly erratic. Conclusions regarding a wire's performance should, therefore, be based on data recorded up to 2.0 mm of deflection. Otherwise, the increased variation of the curve may distort interpretation of the graph.

- Three point bend testing is an unreliable method to evaluate pseudoplastic Ni-Ti archwires when the wire is held in a vice grip. Variation that occurred within the specimens of the same brand and variation between the brands tested produced inconclusive results. Slippage of the specimen during testing with this apparatus was reduced but not totally eliminated. Force values recorded in the vice grip jig were approximately ten times more than those recorded in an edgewise siamese twin bracket jig.

Chapter Nine

FUTURE RESEARCH

As the evaluation of the load-deflection graphs in this study was descriptive research, the true nature of the crystallographic response to three point bending is unclear. To study the crystallographic response of Ni-Ti wire during three point bending, examination of the crystal ultrastructure is required.

The detection of a crystallographic phase transition in pseudoplastic Ni-Ti wires would be possible using electron microscopic techniques. Demonstration of an austenitic to martensitic phase transformation and its behaviour during three point bending would enhance the current understanding of the pseudoplastic phenomena. It is for this reason that an ultrastructural investigation is recommended as the next phase of this investigation. In light of the growing popularity of thermally active pseudoplastic orthodontic wires in clinical use, any future study should also include these wires especially the recently released copper-nickel-titanium alloys.

Measurement of the oral temperature range would be an important consideration for any investigation into thermally sensitive alloys. The oral temperature range would be necessary to confirm that these wires were operating within their temperature transition range, as the anterior part of the mouth may experience temperatures which are below body temperature (37° C). If the temperature transition range of a Ni-Ti wire is markedly above 37° C, a thermally induced phase transformation will not result

A more comprehensive understanding of the crystallographic response in Ni-Ti wires during bending could result in improved physical properties of these products. This knowledge could lead to a considerable reduction in the length of time currently taken for orthodontic treatment.

Chapter Ten

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Chapter Eleven
APPENDICES

Appendix 1 Summary of the various investigators, testing conditions, wires tested and the properties evaluated (adapted from Kapila and Sachdeva, 1989). *For key to abbreviations, see page 66.*

Investigator	*Year*	*Test Type*	*Wires Tested*	*Length or Span*	*Properties Evaluated*	*Mounting*	*Comment*
Waters *et al.*	1975	5 point beam	SS, Msd	13 mm	stiffness*	point contact & 3mm single width brackets	
Andreasen and Morrow.	1978	cantilever	SS, Nitinol		bending moment, spring rate, M_r	Tinius-Olsen tester as per ADA #32	
Lopez *et al.*	1979	cantilever	SS, Nitinol	0.25 inch	permanent deflection versus reflection	Tinius-Olsen tester as per ADA #32	3 separate tests: deflection, deflection & hold, permanent deformat-ion then deflection
Goldberg and Burstone.	1979	cantilever	SS, β-ti		YS, E, YS/E, spring rate, formability	Tinius-Olsen tester as per ADA #32	
Waters *et al.*	1981	wrap test*	Co-Cr, SS, Nitinol, Msd,		yield diameter		
Drake *et al.*	1982	cantilever	SS, Nitinol, β-ti	25.4 mm	YS, E, YS/E, spring rate, M_r	Tinius-Olsen tester as per ADA #32	
Schwaninger *et al.*	1982	cantilever	Nitinol		flexural YS		
Burstone and Goldberg.	1983	cantilever	SS, β-ti, NiTi	10 mm for SS, & β-ti 5 mm for NiTi	maximum bending moment & springback	torque gauge	
Burstone *et al.*	1985	cantilever	SS, Nitinol, Chinese - NiTi	5 mm	maximum bending moment & springback		
Ingram *et al.*	1986	wrap test*	SS, Msd, Nitinol, Co-Cr	90 mm	yield diameter		

Investigator	*Year*	*Test Type*	*Wires Tested*	*Length or Span*	*Properties Evaluated*	*Mounting*	*Comment*
Miura *et al.*	1986	3 point beam test	SS, Nitinol, β-ti, Jpn NiTi	14 mm	deflection versus load graphs	single width bracket and ligation	
Asgharnia and Brantley.	1986	cantilever	SS, β-ti Co-Cr Nitinol	1 inch	E, YS	Tinius-Olsen tester	ADA specificat-ion #32
Schaus and Nikoli.	1986	beam test via simulated arch wire †	SS, β-ti, Nitinol, Msd	5 sites & 5 spans: 5.8, 6.5, 7.3, 8.2, 14.0 mm	flexural stiffness	archwire attached via self ligating bracket (.022 x .028)	attempts to simulate clinical use
Kusy and Stush.	1987	3 point beam	Nitinol, β-ti		E		
Goldberg *et al.*	1983	automated spring tester	SS, β-ti, Co-Cr, Nitinol, Msd		Flexural E		
Rock and Wilson.	1988	3 point beam & simulated archwire ‡	SS, β-ti, Nitinol, Msd	15 mm beam & 15 mm inter-bracket width	deflection versus force generated	beam free to slide archwires to .018" slot edgewise brackets	attempts to compare beam testing to simulated clinical use
Nikoli *et al.*	1988	5 point beam	NiTi, β-ti, Msd	20 mm & 30 mm for single wires 5 mm & 10 mm for Msd	deflection versus load graphs produced. wire stiffness & elastic range calculated	beam free to slide. Supports were single knife edges not actual edgewise brackets	a proposed alternative elastic bending test to ADA specificat-ion # 32
Khier *et al.*	1991	cantilever	Nitinol, β-ti, Jpn NiTi	0.25 inch (6 mm)	bending moment versus angular deflection	torque meter	ADA specificat-ion #28
Kapila *et al.*	1992	3 point beam	Nitinol, austen-itic NiTi	14 mm	deflection versus load graphs	.022" slot edgewise brackets, beam held with elastic modules	compares load-deflection after clinical recycling and sterilising
Tonner and Waters.	1994	3 point beam	SS, Msd Jpn NiTi NiTi		load - deflection behaviour over 5 to 50°C		

KEY:

Wires: SS = stainless steel; β-ti = beta-titanium; Co-Cr = cobalt chromium; Msd = multistranded; Jpn NiTi = Japanese nickel-titanium

Properties: YS = yield strength; YS/E = relates to springback; E = modulus of elasticity; M_r = modulus of resilience or stored energy.

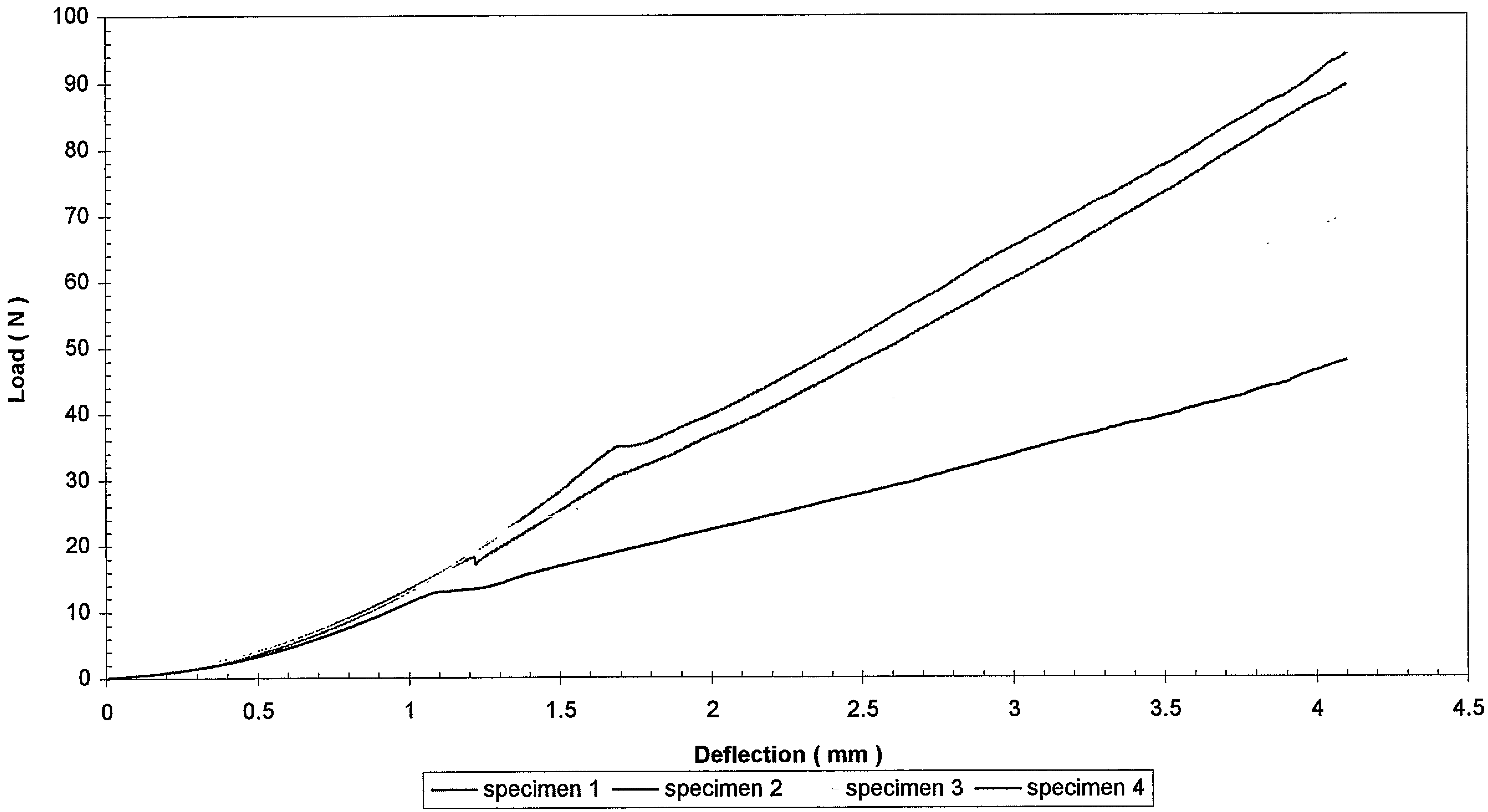
ADA #32 = American Dental Association Specification Number 32.

* wire sections were wrapped around mandrels of varying diameters and deformation was measured on a graph after unwrapping. Yield diameter was arbitrarily set as a 2 mm arc height for a 5 cm cord of wire.

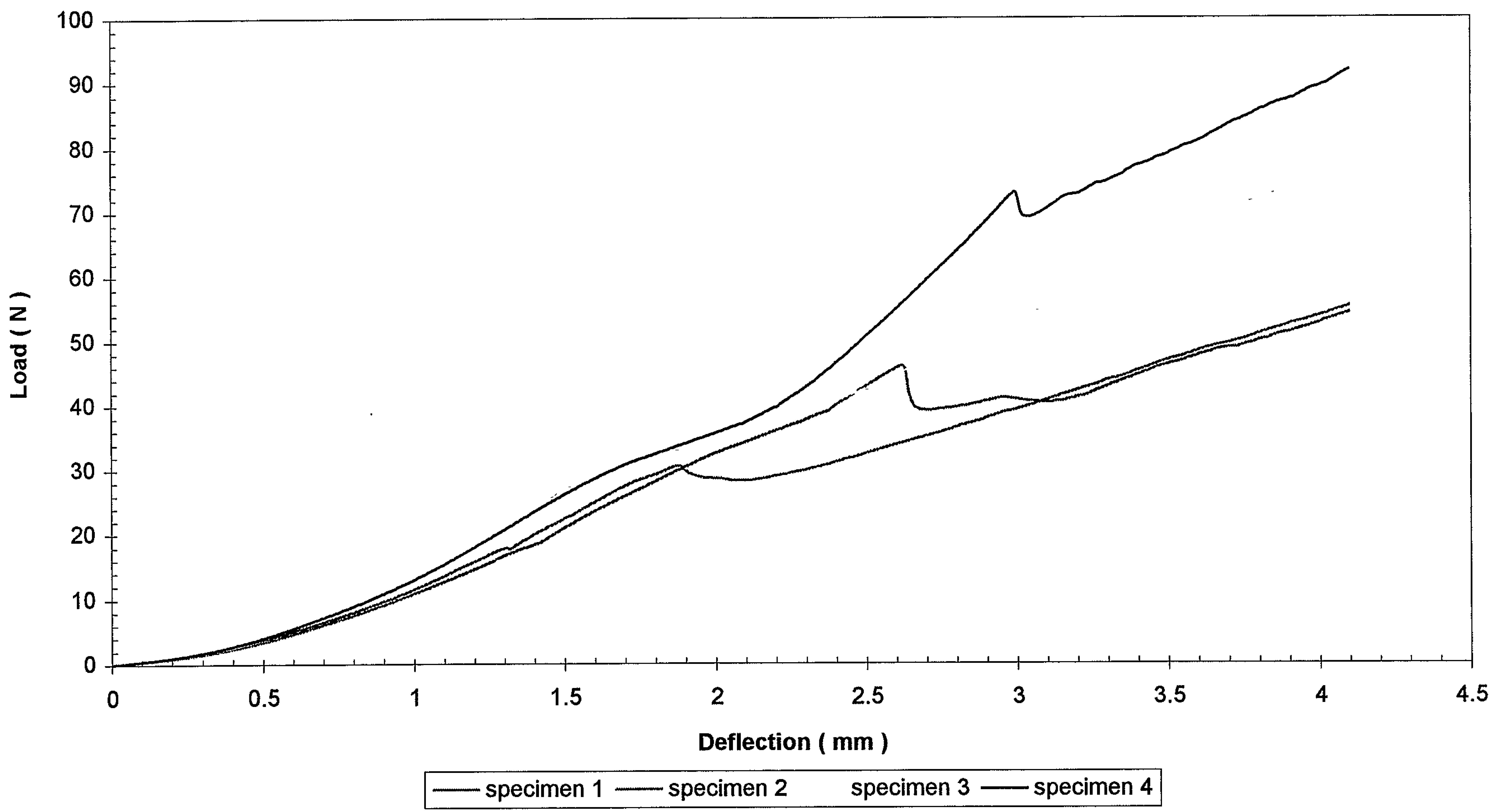
† beam testing via simulated dental arches with dowel segments machined to represent average tooth size. Archwire attached to dowels via self ligating standard twin edgewise bracket (.022 x .028 slot). A load line with a test weight was attached to the archwire at various locations and deflection was measured.

‡ 3 point beam test consisted of a 30 mm length of wire free to slide over a 15 mm span. A phantom head jaw mounted on a bracket for testing simulated an archwire in the mouth. A central incisor was removed to create a 15 mm interbracket span.

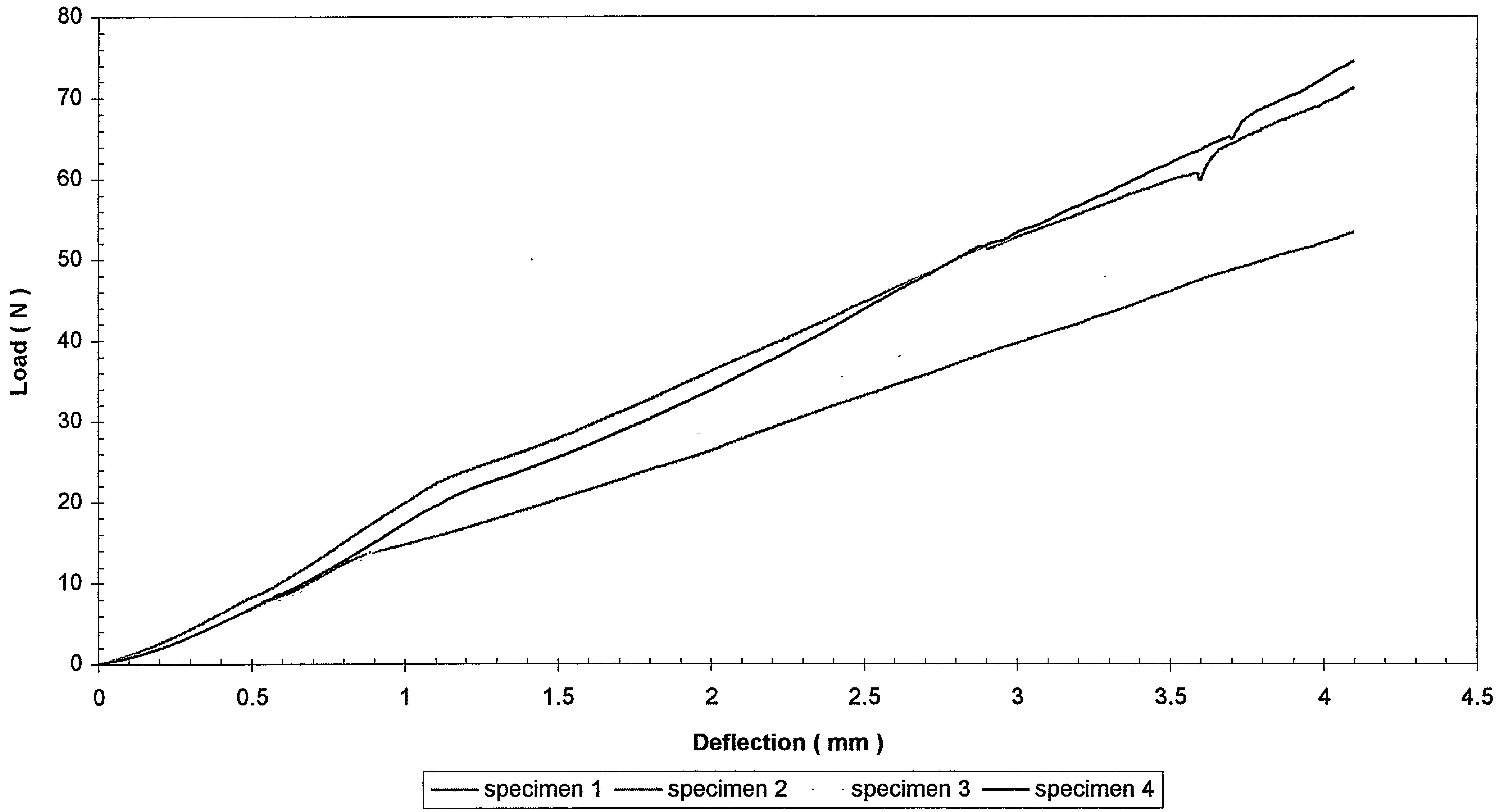
Wire MN: 4 Specimens Tested in a Vice Grip Jig



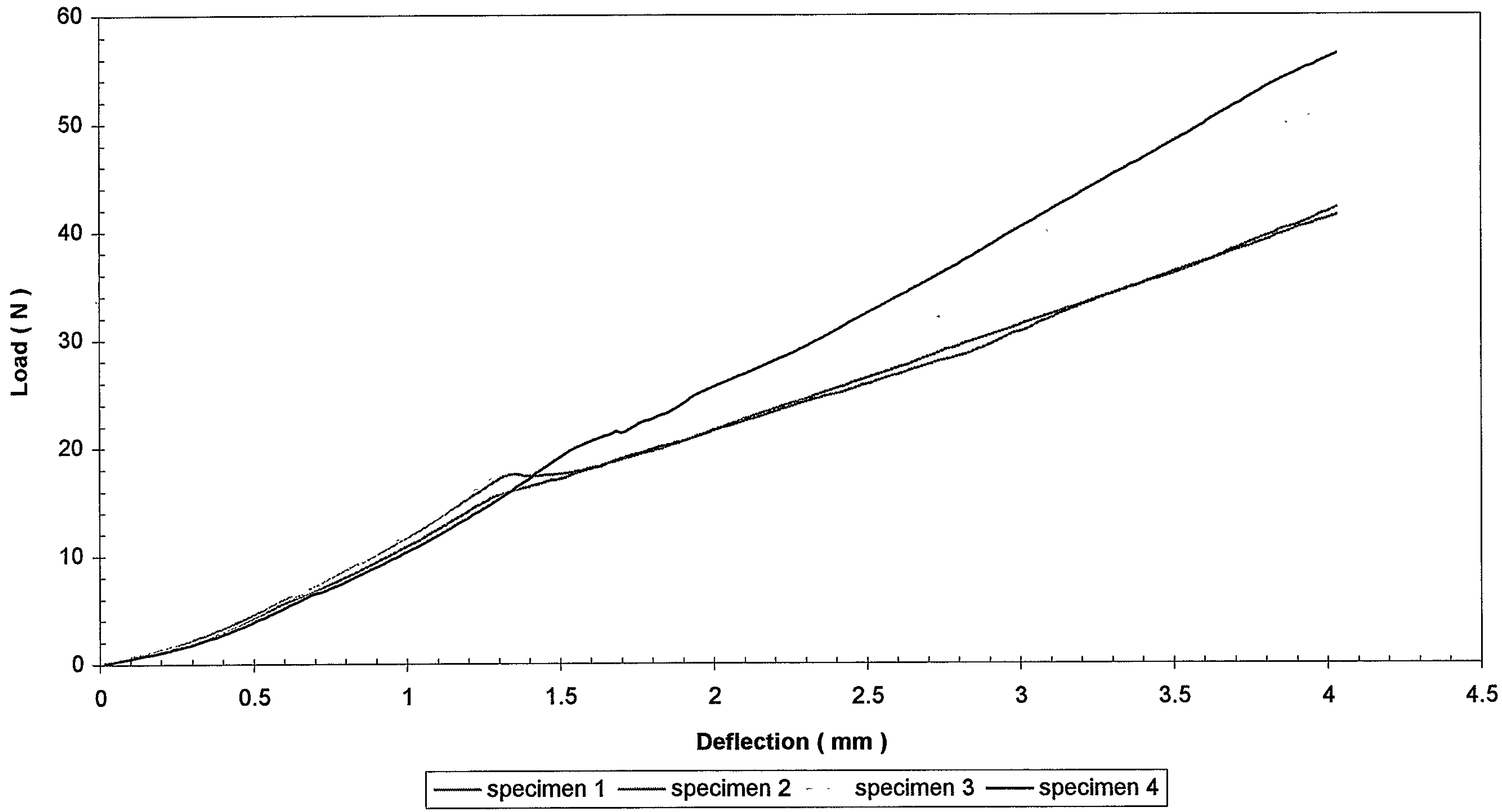
Wire ME: 4 Specimens Tested in a Vice Grip Jig



Wire CX: 4 Specimens Tested in a Vice Grip Jig

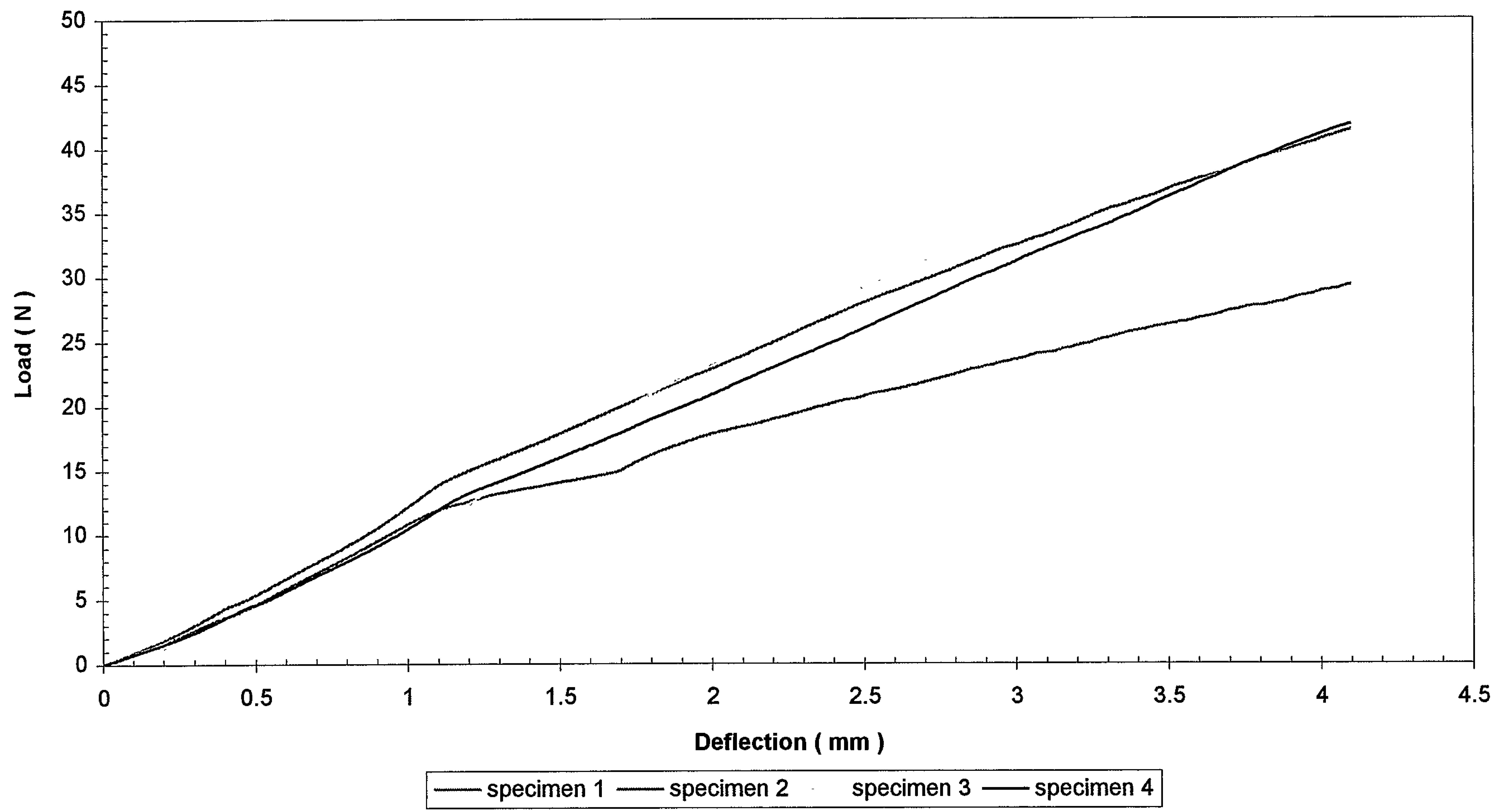


Wire RL: 4 Specimens Tested in a Vice Grip Jig



Appendix 6

Wire SA: 4 specimens Tested in a vice Grip Jig



Average Plots of Wires Tested in a Vice Grip Jig