

# Strathprints Institutional Repository

Henderson, Emma and Nash, David and Dempster, William (2011) *On the experimental testing of fine Nitinol wires for medical devices.* Journal of the Mechanical Behavior of Biomedical Materials, 4 (3). pp. 261-268. ISSN 1751-6161

Strathprints is designed to allow users to access the research output of the University of Strathclyde. Copyright © and Moral Rights for the papers on this site are retained by the individual authors and/or other copyright owners. You may not engage in further distribution of the material for any profitmaking activities or any commercial gain. You may freely distribute both the url (http:// strathprints.strath.ac.uk/) and the content of this paper for research or study, educational, or not-for-profit purposes without prior permission or charge.

Any correspondence concerning this service should be sent to Strathprints administrator: mailto:strathprints@strath.ac.uk



### Research paper

# On the experimental testing of fine Nitinol wires for medical devices

## E. Henderson<sup>*a*,\*</sup>, D.H. Nash<sup>*b*</sup>, W.M. Dempster<sup>*b*</sup>

<sup>a</sup> The National Centre for Prosthetics and Orthotics, University of Strathclyde, 131 St. James Road, Glasgow, G4 OLS, UK <sup>b</sup> Department of Mechanical Engineering, University of Strathclyde, 75 Montrose Street, Glasgow, G1 1XJ, UK

#### ARTICLE INFO

Article history: Received 1 July 2010 Received in revised form 12 October 2010 Accepted 12 October 2010 Published online 17 October 2010

Keywords: Nitinol Shape memory Wire Characterisation Finite element Medical device Superelastic Nickel titanium alloy

#### ABSTRACT

Nitinol, a nickel titanium alloy, is widely used as a biocompatible metal with applications in high strain medical devices. The alloy exhibits both superelasticity and thermal shape memory behaviour. Basic mechanical properties can be established and are provided by suppliers; however the true stress-strain response under repeated load is not fully understood. It is essential to know this behaviour in order to design devices where failure by fatigue may be possible.

The present work develops an approach for characterising the time varying mechanical properties of fine Nitinol wire and investigates processing factors, asymmetric stress–strain behaviour, temperature dependency, strain rate dependency and the material response to thermal and repeated mechanical loading.

Physically realistic and accurately determined mechanical properties are provided in a format suitable for use in finite element analysis for the design of medical devices. Guidance is also given as to the most appropriate experimental set up procedures for gripping and testing thin Nitinol wire.

© 2010 Elsevier Ltd. All rights reserved.

#### 1. Introduction

The move towards increasingly minimally invasive surgical techniques, which can be performed on a higher percentage of the population, has resulted in a need for more technologically advanced interventions. As more complex solutions are developed, the materials considered for use within these devices also become more sophisticated. One material that has found particular favour within the biomedical industry is the shape memory alloy Nitinol. This, near equi-atomic Nickel–Titanium alloy, exhibits both superelasticity and shape or thermal memory capabilities. Coupled with its biocompatibility, it is now being utilised in a variety of applications from orthodontic archwires to surgical guide wires.

Nitinol is used in a variety of different geometrical configurations within the medical devices industry, but is often found as laser cut tubing or fine wire. There are several applications that utilise the material as fine wire, including several stent graft type devices such as the Anaconda endovascular device produced by Terumo Vascutek which is used as the industrial case study in the present work.

<sup>\*</sup> Corresponding author. Tel.: +44 141 548 4034; fax: +44 141 548 3295. E-mail address: emma.m.henderson@strath.ac.uk (E. Henderson).

<sup>1751-6161/\$ -</sup> see front matter © 2010 Elsevier Ltd. All rights reserved. doi:10.1016/j.jmbbm.2010.10.004

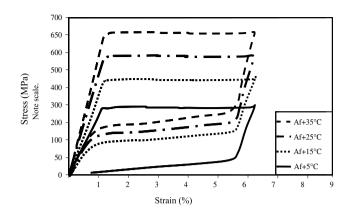


Fig. 1 - The stress-strain response of Nitinol with increasing temperature.

These devices, and others, exploit the superelastic material properties of the wire in its austenitic phase, to allow the large deformation compaction of a device into a small diameter catheter for minimally invasive deployment in vivo. As many of these devices are manufactured from straight drawn wire, coiled into rings and not subsequently heat treated, the primary loading mechanism is seen to be bending (McCummiskey, 2008).

Sophisticated 'design-by-analysis' techniques, underpinned by numerical methods are often employed to allow the engineering simulation of potential components, without the need to build expensive and time consuming prototypes, and to allow optimisation of product design. These methods require accurate physical representation of the material properties in order to achieve a realistic behaviour of the component under load. This is particularly important with Nitinol, which has highly individual material properties, as a result of 'prior thermo-mechanical processing.

The work herein presents an experimental programme undertaken on fine Nitinol wire for the determination of the mechanical properties for use in finite element simulations. Consideration is given to key factors such as temperature dependence, gripping methods, asymmetry in tension and compression and cyclic response of the wire within the superelastic phase of the material. The paper emphasises the complications of obtaining accurate material characteristics for the Nitinol wire using testing regimes commonly available and therefore the generation of uncertainties experienced in any finite element modelling exercise.

#### 2. Basic Nitinol characteristics

Superelastic Nitinol usually consists of 55.6 wt% Nickel with the balance Titanium and only a trace of other elements. The process dependency of Nitinol can render it with a range of different properties; however it is often the superelastic range which is sought by designers of medical devices, although an increasing number of uses are being found for its thermal shape memory properties. The superelastic region of the material allows for designs to be realised which were previously impossible due to the plastic deformation of most alloys, such as steel, at less than 1% strain. In several different medical devices, where devices are implanted and must be inserted through narrow arteries with complex pathways to the site of repair, Nitinol is being turned to for its largely recoverable strains of up to 8%–10%. Meanwhile, thermal memory is also becoming more sought after in the medical device industry as more in depth understanding allows manipulation of the material with temperature to take on a secondary shape once implanted *in vivo*.

The superelastic phase of the material occurs when it is stable in its austenitic crystallographic structure, and recoverable strains in the region of 8% are achievable (Otsuka and Wayman, 1999). The material, under strain of above  $\sim$ 1%, will undergo stress induced phase change into a martensite crystal structure, and a superelastic plateau will form, as shown in Fig. 1 for several samples of virgin (first cycle) material. Completion of the phase transformation from austenite to fully stress induced martensite occurs at the end of the plateau.

The temperature dependence of the material can also be seen in Fig. 1, which shows first cycle data with increasing temperature beyond the austenite finish temperature  $(A_f)$ . The material is seen to be stable in its superelastic phase at temperatures above the  $A_f$  temperature. However, as the material increases in temperature above this A<sub>f</sub> temperature, the plateau shrinks in width and become less pronounced (ie has a higher gradient). This is not seen with huge effect within the demonstrated temperature regime. The material, at temperatures far beyond the A<sub>f</sub> temperature, will have linear elastic material properties. It is reported in the literature that this breadth of superelasticity exists for approximately 50  $^\circ\text{C}$  above the  $A_f,$  however the breadth is often a result of material composition (Otsuka and Wayman, 1999). The temperature window for superelasticity is a function of alloy composition and of microstructure, which can be modified by prior thermo-mechanical treatment.

#### 3. Experimental characterisation of Nitinol wire

In order to achieve the most realistic results from a finite element analysis, it is necessary to input accurate physically representative material properties. The properties of Nitinol have been shown in the literature (Chen et al., 2001; Favier et al., 2006; Ford and White, 1996; Gong et al., 2002; Liu et al., 1998; Melton, 1990; Nemat-Nasser and Choi, 2004; Otsuka and Wayman, 1999; Pelton et al., 1994; Siddons and Moon, 2001; Wick et al., 1995; Yang et al., 1997) to vary depending on 'prior thermo-mechanical processing, temperature, strain rate, and past loading history, and from experience it is considered advisable that the material is experimentally tested to obtain accurate properties. This is normal practice in industry at present.

During the manufacture of the Vascutek Anaconda device, shown in Fig. 2, the Nitinol wire is hand-wound from its straight drawn state into a bundle of up to fourteen turns, secured by a tantalum crimp and suturing, imposing a pre-strain on the wire ring. The ring configuration is then sewn onto the fabric graft into a saddle shape before compaction into the sheath at a high amplitude saddle. Once deployed, the device will exist between two saddle heights that are a function of the artery diameter, device diameter and stiffness coupling between the artery and the device. The process imposes large deformations into each Nitinol wire strand. This is dominated by bending due to the manufacturing process, as observed in finite element cross-sectional plots showing linear variation plots from tension to compression. The level of compaction will induce strains in the magnitude of up to 6%, (McCummiskey, 2008) resulting in the material operating in the superelastic plateau region and cycling between the upper and lower plateau shown loaded by the natural artery pressure pulsations. Furthermore, the conditions are required to be determined at body temperature, due to the human blood temperature, which is maintained constant at 37 °C. Thus the critical issues associated with testing are due to:

- 1. The stress-strain curves in tension and compression;
- 2. The effects of temperature;
- 3. The effects of cyclic loading.

In certain biomedical device applications, including the Terumo Vascutek Anaconda, it is necessary to test fine wires which are of very small diameter (0.1–0.28 mm diameter), and so it is important to ensure the accurate testing of the material in this range. It is also essential to determine the effect of the stress–strain response of the material when subjected to variations in temperature and strain rates, to repeated cyclic loading and to investigate the asymmetrical response of the material in tension and compression.

Considering the unique behaviour of Nitinol, the rationale for testing each separate feature is clear. The measurement of the heat generation effects on a wire of 1 mm diameter (Wagner et al., 2004) has shown that even at very low (<1 Hz) strain rates, there exists an internal heat generation effect when testing the material in air. As the wire considered in the present work will be of 0.26–0.28 mm diameter, except for in the case of tension compression tests and strain rate dependence tests, the dissipation of heat to the surrounding environment is faster due to the increased surface area to volume ratio. As such, it was considered sufficient for the Nitinol in this work to be tested at 5 mm/min to allow minimisation of these effects. This also follows the guidance



Fig. 2 – Terumo Vascutek's Anaconda deployed in a Perspex artery.

given in the current ASTM standards for tensile testing of Nitinol. (ASTM Standards F2516-07e2, 2007).

The literature has shown the temperature dependency of the material (Ford and White, 1996; Otsuka and Wayman, 1999). At the present time, the best industrial practice for material manufacture cannot provide material with a more accurate  $A_f$  temperature specification than  $\pm 3$  °C (McCummiskey, 2008). The wire investigated in the present work was supplied to an  $A_f$  specification of 0–18 °C, a commonly seen specification within the superelastic medical devices industry, therefore there is wide scope for delivered material to have varying  $A_f$  temperatures, particularly when the wire is utilised for in vivo applications, where the environmental temperature is 37 °C.

The superelastic region of the material above A<sub>f</sub> has a limited temperature window in which it acts (Otsuka and Wayman, 1999). A material at high temperatures above A<sub>f</sub> will have a significantly shorter superelastic plateau than that of one at a temperature above but relatively close to A<sub>f</sub>. For a material specification, set at 0–18 °C, suppliers will provide the majority of wires with an A<sub>f</sub> of approximately 5–10 °C and as such, the average in vivo temperature is  $A_f$  + 30 °C. At the limits of the specification, this can change to a range from approximately  $A_f$  + 20 °C– $A_f$  + 40 °C. The testing regime used herein investigates the temperature dependency of the material and aims to quantify the variation in material properties within these limitations. The material that is used to test temperature dependency was supplied with a manufacturer measured Af temperature of 13.7 °C and so testing temperatures are set to range from 33.7 to 53.7 °C.

The asymmetrical response of the material in tension and compression has been well discussed in various works (Liu et al., 1998; McCummiskey, 2008; Melton, 1990; Pelton et al., 1994; Siddons and Moon, 2001; Wick et al., 1995). The high levels of recoverable strain achievable by the material coupled with the complex environment *in vivo* leads to the material often being loaded in bending and or torsion. In the case of the Anaconda, the wire is subject to significant bending whilst compacted in the catheter, and so it is deemed necessary to also quantify the asymmetric response of the material.

The process dependency of the material implies that the actual material employed for the device, should be used to determine the asymmetry behaviour of the wire. The fine diameter of the wire, of diameters 0.1–0.28 mm, and its susceptibility to buckling precludes it from being a viable choice for compression testing with conventional biomedical testing machines. In order to test the material in compression it was necessary to test thicker wires, of nominal diameters 1, 1.8 and 2.4 mm, the use of a range of diameters allowing the effect of processing to be investigated. The material data for these was also compared in tension to the fine wire currently used to determine the accuracy and suitability of such a method.

In order to design, model and optimise complex medical devices such as the Anaconda (Fig. 2) effectively using a finite element approach, it is necessary to ensure good representation of the material properties. For example, the 'shape memory alloy' (SMA) material model available within the ANSYS finite element system (Ansys Inc., PA.) is typical of current practice, and is based on the Auricchio constitutive model (Auricchio et al., 1997). The effectiveness of the model has been discussed elsewhere by the author (McCummiskey, 2008). The experimental programme developed herein provides a systematic approach to ensure a physically accurate depiction of the mechanical properties of Nitinol. Thereafter these can be fully incorporated in a suitable finite element model.

#### 3.1. Tensile and compressive stress–strain testing

Both tensile and compressive stress-strain testing for all wires considered was carried out on a Tinius Olsen H5K-S tensile tester, of maximum load capacity 5 kN. The testing machine was set up with load cells of varying capacity in conjunction with the diameter of the wire and maximum load applied (Tinius Olsen Ltd. Salfords, UK). The load cells were accurate to  $\pm 0.5\%$  of the applied load from 2% to 100% of the load cell capacity. The temperature of the test environment was controlled using an environmental chamber designed and built by Terumo Vascutek (Terumo Vascutek Ltd. Inchinnan, UK). The chamber consists of an enclosed Perspex box, incorporating a fan heater and thermostat. The temperature can be held accurate to within  $\pm 2$  °C as per control testing. QMAT software, written and provided by Tinius Olsen (Tinius Olsen Ltd. Salfords, UK) with the tensile tester was set to produce 1000 data points per loading and per unloading cycle of force and displacement.

Due to the fine diameters of the wire, crosshead displacement was used to determine the extension levels

in the wire. Video extensometer was tried, however the highly polished surface of the wire combined with the large extensions imposed on the small cross section, caused slippage of the reference flags used for monitoring the extension.

The strain rates for all experimental testing subsequent to the strain rate dependency testing was determined based on the results of the initial strain rate testing, and is defined at that point.

#### 3.2. Wire samples

The wires tested were manufactured for use in the superelastic regime with 55.6 wt% Ni, and the balance Titanium, and with  $A_f$  temperature specified to be less than 18 °C. This material corresponds to NDC (Nitinol Devices and Components Inc. Freemont, CA.) product code SE-508 (50.8 at.% Ni). The selected test samples were of nominal diameter 0.26, 0.28, 1.0, 1.8, and 2.4 mm with reported active A<sub>f</sub> temperatures of 13.7 °C, 11.7 °C, 5.3 °C, -5 °C, and 11.4 °C respectively. Further control over these Af temperatures was not available, due to manufacturing limitations. While within the testing regime the variation in Af temperatures can be dealt with, by ensuring tests are carried out at temperatures relative to A<sub>f</sub>, it is worth noting that within a design context, this implies that for a device produced to operate under constant temperature, such as in vivo, there may be a variety of different material properties, dependent on the proximity of the A<sub>f</sub> temperature to the operating temperature. Thus for any design, a range of potential material properties will be considered.

#### 3.3. Gripping approach

Slippage in the grips of a tensile specimen can cause a reduction in the required load, and as such lead to erroneous data. Various methods for gripping the fine wires were considered. For the present work, several types of grips were tested, and light duty pneumatic vice style grips were incorporated into the test routine, manufactured by Tinius Olsen (Tinius Olsen Ltd. Salfords, UK), code HT42. These grips provide active control of the test specimen by applying constant load rather than constant geometry passive grips. The thicker wires, of diameter greater than 1 mm were gripped using chuck style grips, as loading levels were above the range of the pneumatic grips. Visual examination was conducted under optical microscope after completion of these tests to ensure no slippage had occurred.

#### 3.4. Experimental strain rate

It is well reported that Nitinol exhibits strain rate dependency. In order to ensure consistency in the present work both the tension and compression material response data were examined at an extension rate of 50 mm/min for multiple cycling tests and 5 mm/min for single cycle tests. The increased strain rate for the multiple cycling tests could be verified for accuracy by comparison with the single cycle lower strain rate tests. This extension rate is in accordance with a maximum strain rate equivalent to 6 mm/min as stated in the ASTM standard tension test method (ASTM Standards F2516-07e2, 2007).

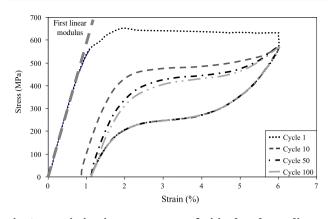


Fig. 3 – Variation in  $\sigma$ - $\epsilon$  response of Nitinol under cyclic loading for up to 100 cycles (NB. All curves return along the same unload line.) ( $d_w = 0.28$  mm, loading rate 5 mm/min).

#### 3.5. Variation in stress-strain response with low magnitude cycling

Nitinol's cyclic variation in stress-strain response has been reported in the literature (Gong et al., 2002; Nemat-Nasser and Choi, 2004; Yang et al., 1997). As the Anaconda device must be subject to a minimum of 400 million cycles in vivo to meet with ISO 25539-1 (2003), it is necessary to consider not just the initial response of the material, but also the longer term response to determine if the stress-strain response stabilised over time. This will enable the most suitable stress-strain response to characterise the material for use in finite element simulations to be determined.

Nitinol wire of diameter 0.28 mm was tested through 100 cycles at 50 mm/min and a temperature of  $A_f$  + 30 °C to test its variation in response with low frequency strain cycling.

The variance in response is shown in Fig. 3 where the variation in the first linear modulus, shortening and depreciation of the upper plateau magnitude and the subsequent increase in residual strain are seen through mechanical cycling.

From Fig. 3 it is seen that through cycling, the start of the phase transformation from the material's parent phase into martensite occurs at a progressively lower stress level. Plastic strain is seen to be induced in the material during the initial cycling, and that the magnitude of additional plastic strain decreases with increasing cycling. The transformation plateau of the material is seen to degrade during the load cycling of the material. The degradation of the plateau, in terms of its starting stress and length, are due to the residual martensite which remains in the stable austenite structure after the first cycle. These cause the energy required for the nucleation of the stable austenite into martensite to diminish, and as such the starting stress levels to decrease (Sakuma et al., 2003).

The first linear modulus region (up to  $\sim 1\% \varepsilon$ ) of the tested Nitinol (as shown by the dashed line on Fig. 3) decreases with cycling, with the proportion of reduction reducing each time, until the material remains largely stable after 20–30 loading cycles. The variation in residual strain, shown in Fig. 4 largely stabilises by 30–40 loading cycles at a value of 0.85%  $\varepsilon$  after 100 cycles. This residual strain is known to continue

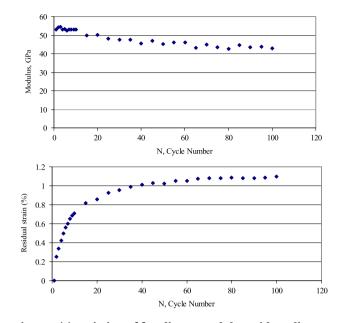


Fig. 4 – (a) Variation of first linear modulus with cycling, and (b) variation in residual strain with cycling.

increasing, albeit with lesser gradient. Upon comparing the austenitic start stress at the start of the loading plateau ( $\sigma_s^{AS}$ ) and austenitic finish stress at the end of the loading plateau ( $\sigma_f^{AS}$ ) it is seen that during the first cycle the  $\sigma_s^{AS}$  has a value of 566 MPa at a strain of 1.89% and by the 100th cycle a value of 353 MPa occurring at a strain of 2.65%, a reduction of over 37%. The strain value increases by almost 30% due to the residual strains occurring in the material through cycling.

#### 3.6. Variation in stress-strain response with temperature

As noted earlier, superelasticity is reported to have a limited region of effectiveness for a temperature window above the A<sub>f</sub> temperature, and the plateau length decreases and becomes less pronounced with separation from this A<sub>f</sub>. Variances in the production and processing techniques of the material and also during production of the Anaconda device, could potentially change the operating temperature relative to A<sub>f</sub> and thus the response of the material. Wire of 0.26 mm diameter was tested at temperatures of  $A_f$  + 20 °C,  $A_f$  + 30 °C and  $A_f$  + 40 °C. Five repeat tests were run at an extension rate of 5 mm/min. The stress-strain curves obtained for each temperature is shown in Fig. 5 which clearly shows the increase in first linear modulus, plateau stress, and in residual strain with increasing temperature. These effects appear to be more prevalent between  $A_f$  +20 °C and  $A_f$  +30 °C, with the highest temperature  $A_f + 40$  °C comparing more closely to  $A_f + 30 \circ C$ .

The increase in temperature in addition to the variation in stress-strain effect shows an increasing saw tooth effect with temperature. This is seen to be more pronounced at small diameters of wire, and is believed to be due to nucleation bursts of stress induced martensite across the cross section of the specimen (McCummiskey, 2008). At higher temperatures, the energy levels within the material are increased and so the effect becomes more prominent.

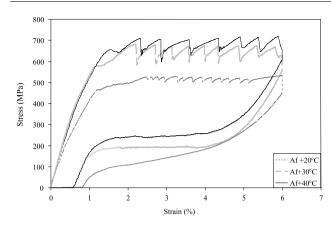


Fig. 5 – Variation of stress–strain curve with temperature during first load cycle. (wire diameter = 0.26 mm, loading rate = 5 mm/min).

#### 3.7. Testing of asymmetry in tension and compression

It is widely reported that Nitinol exhibits an asymmetrical response in tension and compression. Pelton et al. (1994) and Wick et al. (1995) investigated the bending behaviour of Nitinol to determine its stress-strain response and found the stress-strain correlation of the material in bending to be somewhat different to that found in tension. Further investigations have shown that this occurs due to the asymmetry between the compressive and tensile stress-strain curves in the material (Siddons and Moon, 2001). Siddons and Moon have investigated, not only the asymmetry exhibited by the material in tension and compression, but also the degradation of the transformation plateaus and the increment in residual strain that occurs on load cycling of Nitinol tubing. The tests took place over a period of 100 cycles however, it was reported that the material reached a largely steady state condition during the first 40 cycles. Liu et al., also investigated the asymmetric stress-strain response of Nitinol, reporting more specifically on the variation in response of a range of samples subjected to different annealing processes. (Liu et al., 1998).

It is very difficult to test fine diameter wire (0.1–0.3 mm diameter) in compression as it may buckle due to the slenderness ratio of the experimental test set up. Typically sample sizes must have a minimum length of 100 mm and therefore a length to diameter ratio of 333 for the thickest wire, which is prone to buckling. For this reason, it was necessary to employ an alternative technique using a more appropriate slenderness ratio.

Three thicker wires of diameter 1.0, 1.8, and 2.4 mm, were sourced for testing in compression. The diameters chosen were thick enough for compression testing while still undergoing the same processing as fine wires and thus still being defined as in the wire range rather than the processed characteristics of rod. In addition, tensile testing of the thicker wires was undertaken and compared to the tensile curves of the fine wire, and upon deeming the response of the two materials comparable, it was assumed that the degree of asymmetry was also comparable, and thus the compression curves for both wires would be similar.

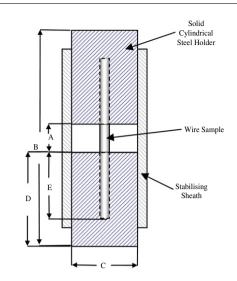


Fig. 6 – Holder for compression tests on Nitinol wire and associated dimensions.

Table 1 – Dimensions of test rig.			
Test rig size (mm)	1	1.8	2.4
A (mm)	1.5	2	2.5
B (mm)	44	60	60
C (mm)	20	20	20
D (mm)	22	33	33
E (mm)	10	18	23

While the sourced, thicker wires were less susceptible to buckling than the fine wires, due to their lower slenderness ratio,  $\sim$ 20, it was still necessary to produce a specially designed specimen holder for each wire diameters to try and minimise the effects of buckling on the wire. The aim of the specimen holder was to steady the wire in the grips and prevent buckling of the wire by limiting the movement of the wire transversely without causing a tight constraint. For this reason, the hole fit required had to be very precise to allow Poisson's effects whilst still supporting the wire in order to prevent buckling. A schematic of the specimen holder is shown in Fig. 6, and dimensions in Table 1. Dimensions were defined as per the Poisson's effects of expansion under compression. Using this holder, the sample was placed in the tensile tester using flat jaw grips.

The material was tested in compression between 4% and 0.01% strain for 50 cycles, at a temperature of  $A_f$  + 30 °C to investigate the asymmetry in tension and compression. A typical curve showing the asymmetry of the material is seen in Fig. 7 for a 1.8 mm wire.

The asymmetry of the Nitinol wire is evident in Fig. 7 above. The austenite starting stress ( $\sigma_s^{AS}$ ) is seen to be 645 MPa in tension at a strain of 1.67%, whilst in compression the value rises in magnitude to 853 MPa but occurs at a decreased strain of -1.43% an increase in magnitude of 24.3%. The linear region modulus has a higher magnitude in compression and the plateau in compression is less pronounced even during the first cycle of the material as shown. The gap length of the specimen holder prohibited the present work from testing in compression above 4% strain however it is reported in the

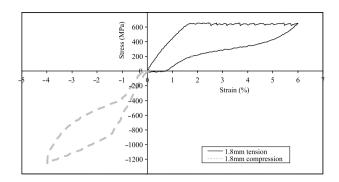


Fig. 7 – Asymmetry of 1.8 mm Nitinol wire in tension and compression ( $A_f$  + 30 °C).

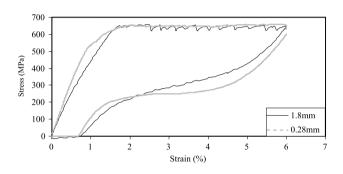


Fig. 8 – Comparison of 1.8 and 0.28 mm wire diameters response in tension.

literature (Siddons and Moon, 2001) that a shorter plateau is also observed in compression.

Whilst the wires tested for compression were sourced to the same specification as those initially investigated, the high sensitivity of the properties of Nitinol to production methods and processing implies that there could be a significant difference in the responses of the two wires. Fig. 8 shows the comparison of tensile stress–strain response curves for the 1.8 mm wire with that of the 0.28 mm wire.

Both wires show similar stress-strain responses, with almost identical plateau levels, however the first linear modulus is higher for the finer wire as is the load-unload modulus seen at the end of the hysteresis loop. The overall values of residual strain are within 0.07%. A contributing factor in the discrepancies of the response could be due to the change in gripping method required as gripping of the fine wire was held using pneumatic grips, whilst the thicker wire used manual chuck grips and as discussed, these grips may not be as effective as the pneumatic grips. Further developments may be required in order to gain more accurate information on the asymmetrical response of the wire in compression, however the results thus far have given a good indication of the asymmetrical response of the wire in tension and compression.

#### 4. Conclusions

The work presented herein has defined the need for a specific methodology for the testing of fine Nitinol wire. Testing of

such wires has been systematically undertaken to examine the changes in stress strain response so that material properties can be utilised within finite element simulations with confidence. The variation in properties seen in the material with varying strain rate, gripping, temperature, and with low magnitude cycling has been clearly presented. The asymmetrical response of the material has also been investigated and any finite element simulation with bending must adopt this approach in its material model.

It was found that with the available resources, testing independent of strain rate could not be fully achieved for the tested wire in air. However, a test strain rate of 5 mm/min was deemed adequately slow when considering factors such as the increased surface area of the wire under test. Various gripping methods for the thin and highly polished wire were examined and the benefits of pneumatic grips have been clearly highlighted.

Low magnitude cycling has also been shown to affect the stress-strain response of the material, with the largest change in response occurring during the first 40 cycles. Subsequent to this, the wire appears to shake down to a stable behaviour. Consideration must be given to the behaviour of the material within its chosen application in order to determine the most appropriate material cycle to use in representing its response. A variation of the material response through increasing temperature is also seen, which must be considered to ensure that the material remains superelastic throughout its manufacture and life in service.

The asymmetrical response of the material in tension and compression has been investigated and a testing method proposed to obtain reasonably accurate stress-strain characteristics for fine diameter wire without the need for expensive equipment. Reasonable validation of this response has been shown in the form of comparison of the tension stress-strain response of the material for the thicker, tested wires and fine wires, considered for characterisation, however it is noted that further work into this area would be beneficial to gain a more detailed understanding of the asymmetry of the material.

Overall, in considering the numerous dependencies of the material and the complex operating environment experienced for a many internal medical devices, the work has presented a testing methodology that can be utilised to obtain a higher level of accuracy within the finite element medium, than single tensile tests. The work has highlighted the several areas in which care must be taken to ensure valid and viable results, and has also exposed several areas in which discrepancies exist in conventional testing methods.

#### Acknowledgements

The authors would like to acknowledge support from Terumo Vascutek Ltd, and from EPSRC through the University of Strathclyde Collaborative Training Account—Research Associate Industrial Secondment scheme.

#### REFERENCES

- ASTM F2516-07e2. 2007. Standard test method for tension testing of Nickel–Titanium superelastic materials. The American Society for Testing and Materials.
- Auricchio, F., Taylor, R.L., Lubliner, J., 1997. Shape-memory alloys: macromodelling and numerical simulations of the superelastic behaviour. Comput. Methods Appl. Mech. Engrg. 146, 281–312.
- Chen, W.W., Wu, Q., Kang, J.H., Winfree, N.A., 2001. Compressive superelastic behaviour of a NiTi shape memory alloy at strain rates of  $0.001-750 \text{ s}^{-1}$ . Internat. J. Solids Structures 38, 8989–8998.
- Favier, D., Liu, Y., Orgeas, L., Sandel, A., Debove, L., Comte-Gaz, P., 2006. Influence of thermomechanical processing on the superelastic properties of a Ni-rich Nitinol shape memory alloy. Mater. Sci. Eng. A Struct. Mater. 429, 130–136.
- Ford, D.S., White, S.R., 1996. Thermomechanical behavior of 55Ni45Ti Nitinol. Acta Mater. 44 (6), 2295–2307.
- Gong, J.M., Tobushi, H., Takata, K., Okumura, K., 2002. Superelastic deformation of a TiNi shape memory alloy subjected to various cyclic loadings. Proc. Inst. Mech. Eng. L 216 (1), 17–23.
- ISO 25539-1:2003. Cardiovascular implants—endovascular devices—part 1: endovascular prostheses. International Organization for Standardization.
- Liu, Y., Xie, Z., Van Humbeeck, J., Delaey, L., 1998. Asymmetry of stress–strain curves under tension and compression for NiTi shape memory alloys. Acta Mater. 46 (12), 4325–4338.
- McCummiskey, E., 2008. The development of an analysis tool for the design of endovascular devices. Ph.D. Thesis. University of Strathclyde, Glasgow, UK.

- Melton, K.N., 1990. Ni–Ti based shape memory alloys. In: Duerig, T.W., Melton, K.N., Stöckel, D., Wayman, C.M. (Eds.), Engineering Aspects of Shape Memory Alloys. Butterworth-Heinemann, pp. 21–35.
- Nemat-Nasser, S., Choi, J.Y., 2004. Strain rate dependence of deformation mechanisms in a Ni–Ti–Cr shape memory alloy. Acta Mater. 53 (2), 449–454.
- Otsuka, K., Wayman, C.M., 1999. Shape Memory Materials. Cambridge University Press.
- Pelton, A.R., Rebelo, N., Duerig, D.W., Wick, A., 1994. Experimental and FEM analysis of the bending behaviour of superelastic tubing. In: First International Conference on Shape Memory and Superelastic Technologies. pp. 353–358.
- Sakuma, T., Yadama, M., Iwata, U., Ochi, Y., Hosogi, M., Okabe, N., 2003. Evaluation of degradation of critical stress for inducing martensite by residual martensite volume fraction in Ti–Ni alloys. Trans. Mater. Res. Soc. Jpn. 28 (3), 639–642.
- Siddons, D.J., Moon, J.R., 2001. Tensile and compression performance of superelastic NiTi tubing. Mater. Sci. Technol. 17 (9), 1073–1078.
- Wagner, M., Sawaguchi, T., Kaustraeter, G., Hoeffken, D., Eggelar, G., 2004. Structural fatigue of pseudoelastic NiTi shape memory wires. Mater. Sci. Eng. A Struct. Mater. 378, 105–109.
- Wick, A., Vohringer, O., Pelton, A.R., 1995. The bending behaviour of NiTi. J. Phys. IV Colloq. 5 (8), 789–794.
- Yang, F., Wu, H.K., Pu, Z.J., 1997. The effect of strain rate and sample size effect on the superelastic behaviour of superelastic alloys. In: Proceedings of the Second International Conference on Shape Memory and Superelastic Technologies. CA.