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Study of Unrecovered Strain and Critical Stresses in One-Way Shape Memory Nitinol

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Unique thermomechanical properties of Nitinol known as shape memory and superelasticity make it applicable for different fields such as biomedical, structural, and aerospace engineering. These unique properties are due to the comparatively large recoverable strain, which is being produced in a martensitic phase transformation. However, under certain ranges of stresses and temperatures, Nitinol wires exhibit unrecovered strain. For cyclic applications, it is important to understand the strain behavior of Nitinol wires. In this study, the unrecovered strain of different Nitinol wire diameters was investigated using constant stress experiment. Uniaxial tensile test has been also performed to find the range of critical stresses. It was observed that the unrecovered strain produced in the first loading-unloading cycle affects the total strain in the subsequent cycles. Moreover, a critical range of stress was found beyond which the unrecovered strain was negligible while the wires heated up to the range of 70-80°C, depending on the wire diameters. The unrecovered strain of wire diameters of 0.19 mm and less was found to be sensitive to the critical stress. On the other hand, for wire diameters bigger than 0.19 mm this connection between the unrecovered strain and the critical stress was not observed for the same range of heating temperature.

Keywords critical stress, Martensite detwinning, Nitinol, strain response, unrecovered strain, wire diameter

1. Introduction

Nitinol wire, also known as NiTi, is an equiatomic intermetallic compound of binary alloy of Nickel and Titanium with approximate composition of 50 at.% Ni and 50 at.% Ti. Nitinol is one of the most common types of shape memory alloys with two distinct crystal structures. One is Austenite, exists at high temperature with high symmetric crystal structure (two intermediate single cubic), also known as parent phase. The other phase is Martensite with lower symmetric crystal structure (orthorhombic or monoclinic) and exists at low temperature. Nitinol exhibits several unique behaviors such as one-way and two-way shape memory effect and superelasticity. One-way shape memory effect refers to the existence of large elastic strains due to the temperature changes at certain applied stress levels. Two-way shape memory effect concerns with the existence of large recoverable strains due to the temperature change. Superelasticity is when the large elastic strain occurs at high temperatures due to applied stress.

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List of Symbols

A_s	Austenite start temperature (°C)
A_f	Austenite finish temperature (°C)
M_s	Martensite start temperature (°C)
M_f	Martensite finish temperature (°C)
σ_{Ms}	Martensite starting stress in superelastic behavior (MPa)
σ_{Mf}	Martensite finishing stress in superelastic behavior (MPa)
σ_{As}	Austenite starting stress in superelastic behavior (MPa)
σ_{Af}	Austenite finishing stress in superelastic behavior (MPa)
σ_{crsC}	Starting critical stress achieved from constant stress experiment (MPa)
σ_{crfC}	Finishing critical stress achieved from constant stress experiment (MPa)
σ_{crsT}	Starting critical stress achieved from Uniaxial Tensile test (MPa)
σ_{crfT}	Finishing critical stress achieved from Uniaxial Tensile test (MPa)

These unique properties of Nitinol make it a suitable alternative for biomedical device such as orthodontic wires, implants, and also actuators (Ref 1). The research performed by Hutapea and coworkers (Ref 2-9) showed the actuation capability of Nitinol wires to enhance the maneuverability of an active brachytherapy needle. This actuation ability was due to Nitinol high power to volume ratio and their ability to produce a high recoverable strain and actuating force with small wire diameters. It has been shown that the active needle device with Nitinol actuator has a great potential to improve the accuracy of needle placement and also the clinical outcomes (Ref 2, 3). However, to achieve these improvements it is important to investigate the reliability and the consistency in the strain response of Nitinol wires by minimizing the unrecovered strain between actuation cycles (Ref 10).

Martensitic phase transformation in Nitinol wires between Austenite and Martensite is possible by either changing the temperature or by applying stress (Ref 11) that can produce up to 6% recoverable strain (Ref 12, 13). This strain response is influenced by several factors such as transformation temperatures, transformation stresses, the generated strain during phase transformation, the biased stress during thermal cycle, and the maximum temperature of wires during heating cycle. Transformation temperatures are the temperatures at which transformation between two phases (Austenite and Martensite) starts and finishes, namely as A_s and A_f (Austenite start and finish, respectively), M_s and M_f (Martensite start and finish, respectively). Transformation stresses on the other hand are the starting (σ_{Ms}) and finishing stresses (σ_{Mf}) at which the phase transformation occurs between Austenite to Martensite and vise versa (σ_{As}, σ_{Af}) in the superelastic behavior. Biased stress is the constant stress applied to the wire during the thermal cycle. In one-way shape memory behavior, a temperature change induces high recoverable strain at a certain level of the biased stress. However, when the biased stress is lower than a specific stress level (i.e., the critical stress, σ_{crs}, σ_{crf}), a small recovered strain is observed and high unrecovered strain is produced between first and second thermal cycles in one-way Nitinol shape memory wires (Ref 14).

The goal of this paper is to determine the range of these critical stresses for different wire diameters through the use of specially designed constant stress experiment and uniaxial tensile test. Several parameters influence the critical stresses such as wire diameter, maximum temperature reached during heating cycle (in constant stress experiment), and the detwinning process during the formation of Martensite in cooling period. Among these parameters, wire diameter is investigated more in details due to its various applications in different fields, for example, the smart needle project of our group.

It has been shown that changes in wire diameters affect the thermomechanical properties of Nitinol wires such as transformation temperatures and stresses (Ref 15). In addition, the maximum temperature that wires experienced through the heating cycle influences the unrecovered strain generated in the first and second thermal cycles (Ref 16). Other studies (Ref 17-24) have shown that the decrease in the wire diameter causes a decrease in the transformation temperatures and an increase in the transformation stresses. Therefore, the decrease in wire diameter could prevent the formation of Martensite from Austenite. Waitz et al. (Ref 17) showed that reducing the wire diameter to less than 50 nm decreases the transformation temperatures in nanocrystalline NiTi. Similarly, Fu et al. (Ref 18) discovered that by decreasing the Nitinol film size, transformation temperatures (especially M_s) decreases, and transformation stresses (σ_{Ms}) increases. They argued that decreasing the size prohibits the formation of Martensite from Austenite. In contrast, Frick et al. (Ref 19) showed that for Nitinol pillars decrease in wire diameter decreased the critical stresses for both forward (Austenite to Martensite) and reversed transformation (Martensite to Austenite) in superelastic behavior. In other studies, Chen and Schuh (Ref 20) discussed the effect of size on transformation temperatures and stresses of Cu-Al-Ni. They discussed that by decreasing the wire diameter less than 100 μm, the transformation temperatures, Austenite transformation stresses, stress hysteresis in a mechanical cycle, and temperature hysteresis in a thermal cycle are increasing. San Juan et al. (Ref 21) claimed that Cu-Al-Ni with diameters of 1.7 and 0.9 μm exhibits better superelastic recovery strain compared to Nitinol pillars of the same diameters. They achieved superelastic recoverable strain up to 5% for more than 100 cycles in micro-compression test. Liu and Mishnaevsky (Ref 22) showed that the formation of Martensite was less probable by decreasing the grain size of Nitinol alloys in nanoscale wire diameters. An et al. (Ref 23) showed the effect of wire diameter on the required power to obtain an actuation response, reported in terms of power consumption and response time for force generation. They showed that by increasing the wire diameter, the required power to reach to the same temperature in Nitinol wires was increasing. Norwich and Fasching (Ref 24) showed the dependency of Nitinol strain response on the wire diameters, wherein decreasing the wire diameter from 0.762 to 0.254 mm significantly improved the consistency (getting the unique total strain in multiple cycles).

It should be noted that most of the studies discussed above, focused on the effect of Nitinol wire diameters on thermomechanical properties, in this case, transformation temperatures and stresses. Moreover, the work done in the literatures used relatively small diameters in the range of micrometer to nanometer diameters. However, there is a great need in different applications to study the thermomechanical behavior of Nitinol wire at a relatively larger scale, 0.1-1.0 mm in diameter. This range of diameters is critical for a number of engineering applications, such as actuators in biomedical devices, surgical implants, stents, rivets, couplings, circuit breakers, electronic chart recorder, and orthodontic wires (Ref 1, 25). In addition, the effect of wire diameter on transformation temperatures and stresses has been discussed, but very limited number of studies investigated the effect of wire diameter on the unrecovered strain and the range of critical stresses. In summary, the goal of this study is to seek fundamental understanding of the effect of Nitinol wire diameter on the unrecovered strain as well as the critical stresses. Understanding of these parameters would help in determining the required biased stress on the wire during thermal cycle and the maximum heating temperature for a consistent strain response.

If twinned martensite forms during cooling cycle in the absence of a sufficient biased stress, a high value of unrecovered strain forms between initial thermal cycles (Ref 26-32). Based on the biased stress levels, the formation of twinned or detwinned Martensite can be achieved. Formation of active Martensite variants during detwinning process can influence the unrecovered strain during Martensitic transformation (Ref 27, 30). To prevent the unrecovered strain, the biased stress should be beyond certain critical stress ranges. This range of critical stress refers to the range of stress where detwinning process in Martensite phase occurs (Ref 31). There are different Martensite variants (twins) in twinned Martensite crystal structure in different directions. These twins are transformed along the imposed stress direction and form the detwinned martensite crystal structure. This alignment can induce Nitinol wires to undergo a large or a small-shape recovery strain due to occurrence of partial or complete detwinning (Ref 33). If the biased stress is below a certain value, the detwinning process cannot be completed and the unrecovered strain of up to 10% remained in the wires (Ref 34).

In this paper, the effect of wire diameter on unrecovered strains and the critical stresses is presented. Specifically, the effect of maximum temperature on unrecovered strains will be studied. The critical stresses of one-way shape memory Nitinol wires with diameter ranging from 0.10 to 0.29 mm were determined using constant stress experiments. In addition, uniaxial tensile tests were performed to determine the effect of wire diameter on the required stress for the detwinning process. The ranges of critical stresses obtained from the constant stress and the uniaxial tensile experiments will be compared to determine the critical stresses.

2. Experiments and Methods

Nitinol wires from Dynalloy, Inc. (Tustin, CA, USA), commercially known as Flexinol, with A_s (Austenite start) temperature of 70°C at 172 MPa, were tested with various diameters ranging from 0.10 to 0.29 mm. All the wires had a composition of 50.5 at.% Nickel and 49.5 at.% Titanium and length of 100 mm. As-received Flexinol wires have detwinned Martensite crystal structure. They were pre-strained up to 5% and tested and spooled to 104 MPa by manufacturer before shipping. This amount of pre-strain will be released during heating to Austenite in one-way shape memory behavior.

2.1 Differential Scanning Calorimetry (DSC) Test

In order to have the range of transformation temperatures for Nitinol before performing the constant stress experiment and uniaxial tensile test, the DSC tests were performed. Transformation temperatures of Nitinol wires at zero stress were determined with DSC 2920CE machine (TA Instrument, New Castle, DE, USA). Samples were heated to 120°C at a constant rate of 10°C/min and then cooled down to −100°C. Liquid Nitrogen was used as both the cover and purge gas.

2.2 Constant Stress Experiment

The strain-temperature responses of Nitinol wires were obtained from the constant stress experiment. This test was performed to show the one-way shape memory behavior of Nitinol wires. From this experiment, the amount of the unrecovered strain and the total strain were measured for each wire diameter and then the effect of size on the amount of unrecovered strain and critical stresses was investigated.

Figure 1 shows the schematic picture of the test setup where Nitinol wire was loaded at constant stress using a weight hanger. The strain response of Nitinol wire during thermal cycle was measured by connecting the weight hanger to a Linear Variable Differential Transducer (LVDT), a HSD 750-500 manufactured by Macro Sensors (Pennsauken, NJ, USA) with a nominal range of ±12 mm and a scale factor of 0.8 V/mm. The temperature of Nitinol wire was measured with a 0.076 mm Omega K-type thermocouple (Omega Engineering, Stamford, CT, USA), which was attached to the top portion of the Nitinol wire. The contact point between thermocouple and Nitinol wire can influence the reading of the temperature. In order to ensure a proper contact, a conductive paste (Omegatherm 201, Stanford, CT 06907, USA) was applied at the contacts between the Nitinol wire and thermocouple. The output signal of both the thermocouple and the LVDT was collected using SCXI-1321 terminal block (National Instrument, Austin, TX, USA). Note that the test described is similar to the experimental procedure performed by Churchill and Shaw (Ref 13) where they examined the thermo-electro-mechanical response of shape memory alloy (Flexinol wires) during cyclic thermomechanical loading.

Six different wire diameters ranging from 0.10 to 0.29 mm were tested using this method at different biased stresses. Nitinol wires were activated by applying an electrical current

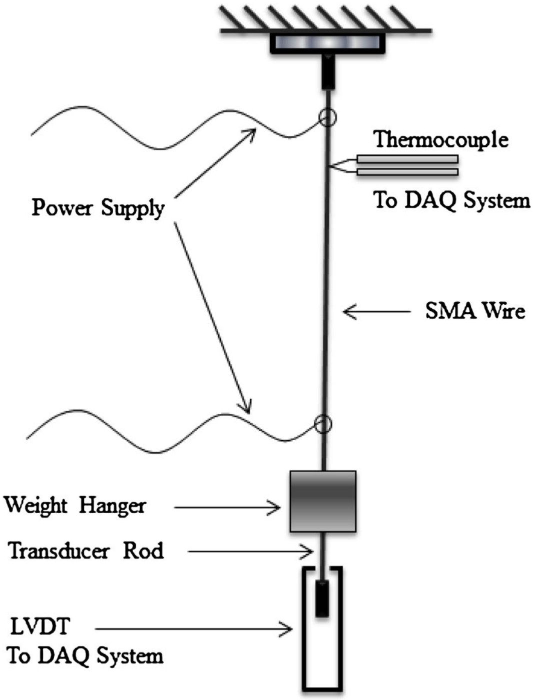

Fig. 1 Schematic of constant stress experiment

Table 1 Applied electrical current values and maximum temperature for each wire diameter

Wire diameter, mm	Electrical current, A	Maximum temperature range, °C
0.10	0.25-0.28	70-80
0.12	0.33-0.37	70-80
0.15	0.41-0.43	73-76
0.19	0.56-0.61	71-80
0.24	0.60-0.67	73-80
0.29	0.70-0.80	75-80

produced by a programmable power supply (BK Precision 1696, Yorba Linda, CA, USA) in a step function mode. The electrical current was applied to the wire in a step function for 15 s. In the first 3 s, the temperature on the wire reached the maximum value of 75°C and for the next 12 s, the wire was kept at this temperature. Due to momentarily disconnection between the tip of thermocouple and the activated Nitinol wire, the temperature had a fluctuation of ±7°C during the 12 s of heating. The maximum temperature was determined by calculating the average temperature value during this heating period. The wire was then air-cooled to room temperature, around 24°C for 45 s. The applied current was employed so that the temperature of the wires reached to 70-80°C, see Table 1. Four cycles of heating and cooling were performed for each wire at each individual biased stress.

2.3 Uniaxial Tensile Test

The stress-strain response of Nitinol wires was obtained by performing tensile test at room temperature in order to determine the range of critical stress of the detwinning process.

An Instron Mini-55 (Artisan Technology Group, Champaign, IL, USA) tensile test machine was used with a 10 N load cell and a strain rate of 4×10^{-5} 1/s (displacement control). The test was performed in an isothermal condition at room temperature. The Nitinol wires with the same length of 100 mm were preconditioned and then tested. The preconditioning temperature performed prior to the tensile test had a significant role on the appearance of stress plateau occurs during the Martensite detwinning process (Ref 35, 36).

The following steps were followed in the preconditioning procedure to ensure the formation of twinned Martensite in Nitinol wires. First, the wires were heated in a range of 70-80°C. This range of temperature is the same range of heating temperature in the constant stress experiment. Note that for 0.24 mm wire, a stress plateau at higher strains during the tensile test was not observed when the preheating range of 70-80°C was applied. For this reason, a preheating range of 120-130°C was used only for the 0.24 mm wire. The constant stress during the preheating procedure was kept at a low level, i.e., the weight of LVDT rod and hanger. The constant stress level is 48.15 MPa for 0.10 mm wires and 9.10 MPa for 0.24 mm wires. The heating time was 15 s. At this point, the detwinned Martensite crystal structure changes to the Austenite crystal structure. The wires were then cooled to the room temperature (24°C) at a negligible biased stress level. After this process, the Austenite structure transforms to a mixture of R-phase and twinned Martensite structure. Based on the DSC results, shown in Table 2, and the phase diagram of Nitinol in Fig. 3, M_f temperatures are below the room temperature for all wire diameters. In order to ensure the formation of fully twinned Martensite crystal structure prior to the tensile test, the wires were placed in an environmental chamber at −40°C for an hour at zero stress.

Finally, in the tensile test, it was possible to change the twinned Martensite (formed after preconditioning procedure) into the detwinned Martensite using an external load. The ranges of stress plateau (σ_{crsT} and σ_{crfT}) were determined by drawing tangential lines at the linear parts of loading and unloading curve, following the procedure described in Ref 36.

3. Results and Discussion

3.1 DSC Results

Transformation temperatures of different wires are shown in Table 2. It can be seen that for the wires heated above 75°C, a complete transformation to Austenite occurs; A_f is less than 75°C. For the wires cooled to −40°C transforms into a complete Martensite phase; M_f is higher than −40°C. These observations were used to choose the preconditioning temperature prior to the tensile test.

Table 2 Stress-free transformation temperatures determined from the DSC test for various wire diameters in as-received condition

Wire diameter, mm	M_f, °C	M_s, °C	A_s, °C	A_f, °C
0.10	4	31	58	67
0.12	−11	29	54	65
0.15	−18	26	52	64
0.19	−33	15	42	75
0.24	−30	12	42	64
0.29	−32	15	37	67

3.2 Constant Stress Experiment Results

The purpose of constant stress experiment was to obtain strain-temperature response of Nitinol wires under a constant biased stress. Transformation temperatures were determined by drawing tangential line to the plateau parts of the strain-temperature response at each of the constant stresses of each wire. The results are shown in Fig. 2. By changing the biased stress in each wire diameter, the phase diagram of Nitinol wires was obtained, as shown in Fig. 3. This figure shows the phase diagram of Nitinol wire diameters from 0.10 to 0.19 mm. Figure 4 and 5 shows a typical strain-temperature response of Nitinol wires with 0.19 and 0.29 mm in diameter under various biased stresses, and heated to the temperature range of 70-80°C. For 0.19-mm wire diameter under a biased stress of 48.15 MPa as in Fig. 4(a), a large value of unrecovered strain was observed between the first and second thermal cycles. It was concluded that the biased stress was below the range of critical stress. When the biased stress was increased to 202.60 MPa as in Fig. 4(b), a negligible unrecovered strain was observed indicating that the biased stress was higher than the range of critical stress. Similar trends were observed in smaller diameters of 0.10, 0.12, and 0.15 mm. For larger diameter of 0.29 mm under low-biased stress of 13.6 MPa as in Fig. 5(a), a small unrecovered strain (about 0.5%) was observed. By increasing the biased stress to 240.50 MPa, this small amount of unrecovered strain became negligible as shown in Fig. 5(b).

Based on the constant stress experiment, within the heating range of 70-80°C, two categories of wires were defined and discussed as below. First category was the wire diameters in which the unrecovered strain was dependent on the biased stress (i.e., 0.10, 0.12, 0.15, and 0.19 mm) at the temperature range of 70-80°C. In these wire increasing the applied stress decreased the unrecovered strain after passing the starting critical stress. Figure 6 shows the relationship between the unrecovered strain and the biased stress. Based on the amount of the unrecovered strain shown in Fig. 6(a), the stress value at which unrecovered strain begins to drop can be considered as the starting critical stress (σ_{crsC}) and the value of stress which causes a negligible unrecovered strain can be considered as the finishing critical stress (σ_{crfC}). The critical stress ranges

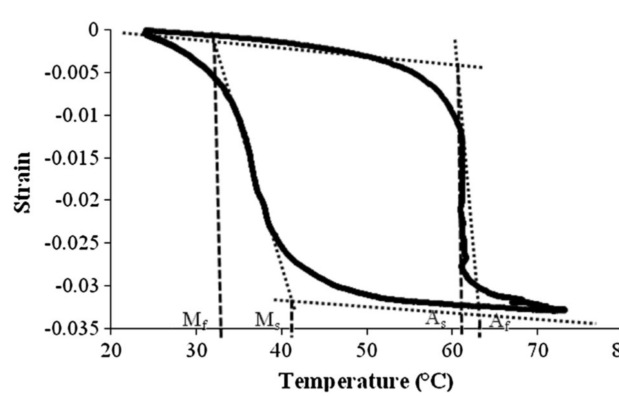

Fig. 2 Obtaining transition temperatures from strain vs. temperature plot

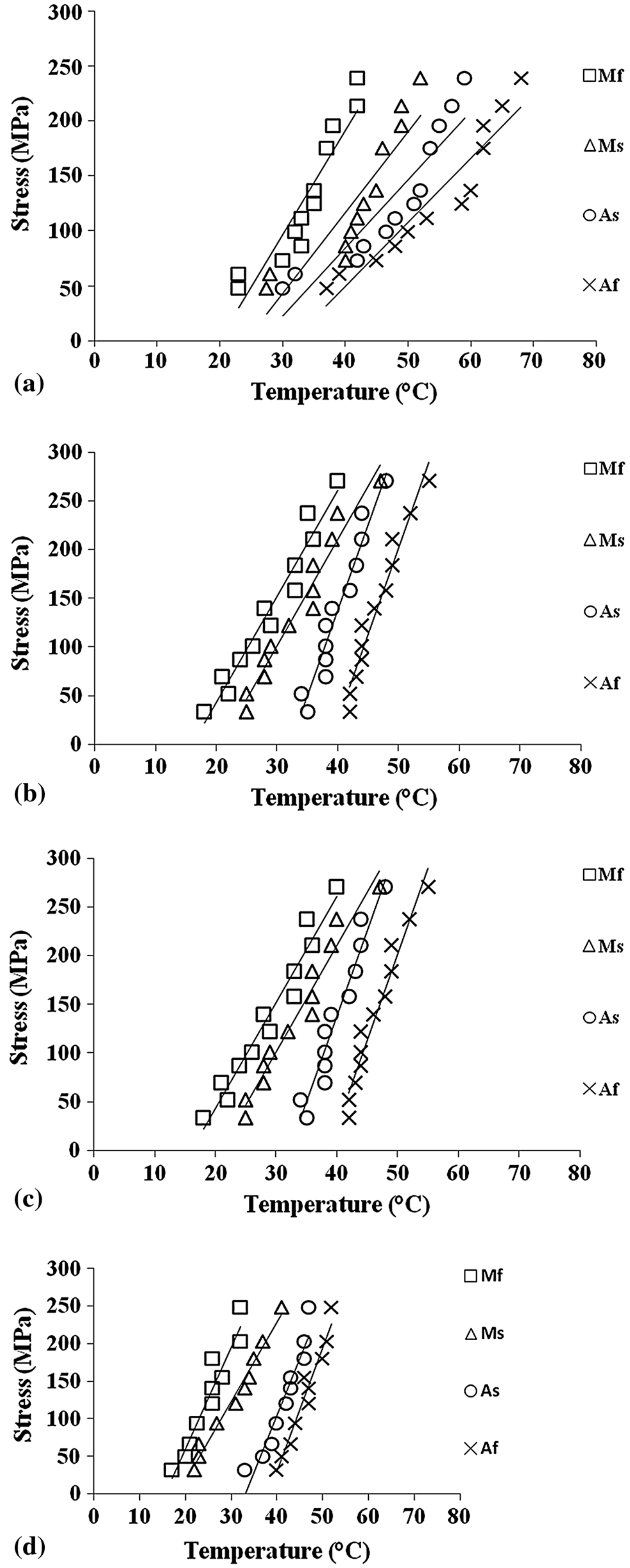

Fig. 3 Variation in transformation temperatures with applied constant stress for Nitinol wires with diameters of (a) 0.10 mm, (b) 0.12 mm, (c) 0.15 mm, and (d) 0.19 mm

achieved from constant stress experiment are summarized in Table 3. It can be observed in Table 3 and Fig. 6(a) that by decreasing the diameter of Nitinol wires the starting and finishing critical stress were decreased. In the second category

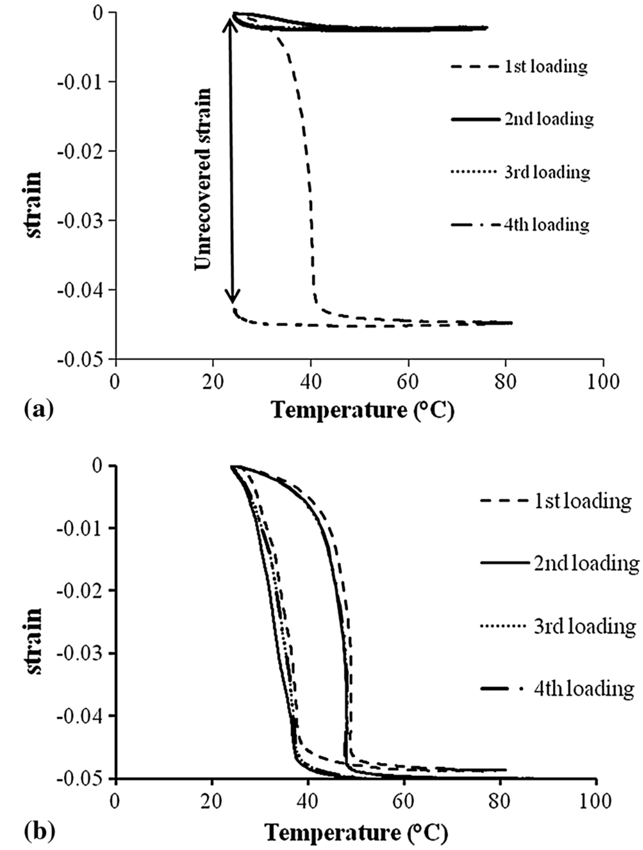

Fig. 4 Strain response of 0.19-mm wire diameter under constant stress of (a) 48.15 MPa and (b) 202.60 MPa

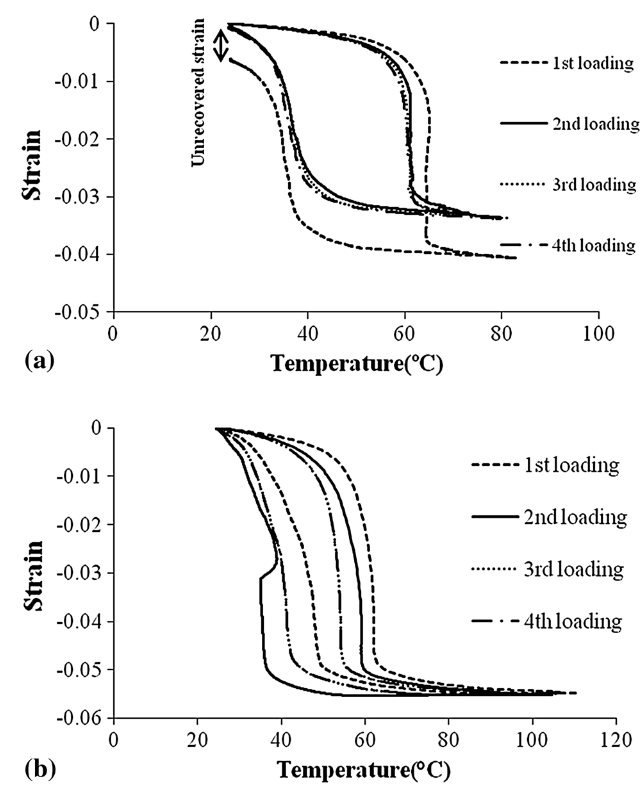

Fig. 5 Strain response of 0.29-mm wire diameter under constant stress of (a) 13.60 MPa and (b) 240.50 MPa

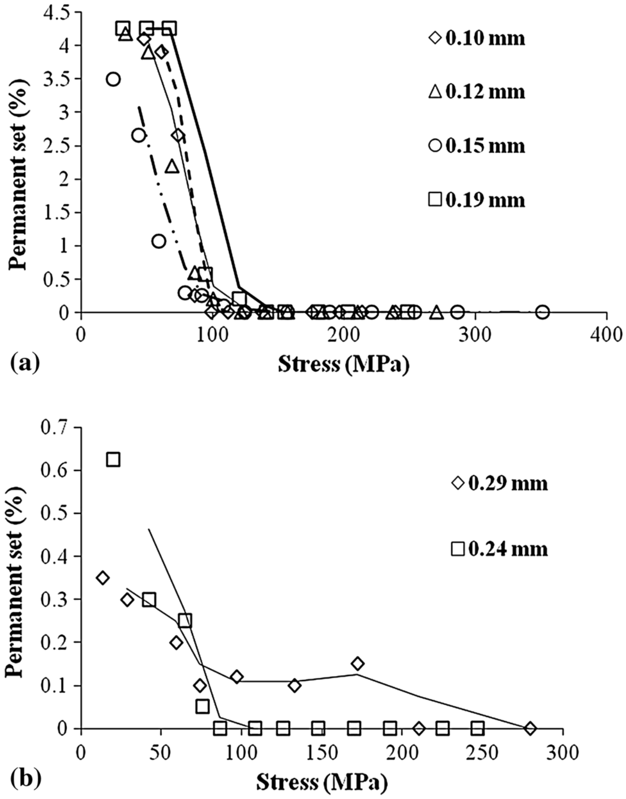

Fig. 6 Variation in unrecovered strain with applied constant stress for Nitinol wires with diameters of (a) 0.10, 0.12, 0.15, 0.19 mm and (b) 0.24 and 0.29 mm

Table 3 The critical stresses for detwinning process in Martensite phase determined from constant stress experiment (σ_{crsC}, σ_{crfC}) at the heating range of 70-80°C

Diameter, mm	σ_{crs}, MPa	σ_{crf}, MPa
0.10	86.40	99.10
0.12	100.62	121.84
0.15	108.60	124.20
0.19	120.00	140.80
0.24	NA	NA
0.29	NA	NA

of wires, the unrecovered strain was independent from biased stress (0.24 and 0.29 mm) at temperature range of 70-80°C. In these wires, the unrecovered strain with respect to the total strain was much smaller than the values observed in the other diameters discussed in the previous sentences. As can be seen in Fig. 6(b), the amount of unrecovered strain was relatively small. By increasing the amount of biased stress there was no sharp drop in the amount of unrecovered strain. This was an indirect method of measuring the critical stresses of different diameters.

It should be mentioned that there were differences between transition temperatures obtained from the DSC and the constant

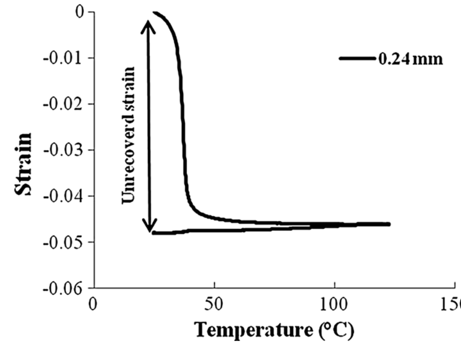

Fig. 7 Strain vs. temperature response for 0.24-mm diameter Nitinol wire at 9.1 MPa constant stress heated to high temperature range between 120 and 130°C

stress experiments. In the DSC test, the transformation temperatures were obtained from the change in the rate of heat flow of the sample during heating and cooling at zero stress, while the stress free transition temperatures from the constant stress experiment were achieved by a linear extrapolation to zero stress which can contain errors and inaccuracy due to the reading of temperature in constant stress experiment and experimental errors.

Standard deviation of ±15 MPa was included in the critical stress results (Table 3) due to the variation of weight of hanger and LVDT rod as a constant minimum load. By increasing the amount of constant stress in each wire diameter with application of the same amount of current (similar heating temperature, same diameter at different stress level), different strain-temperature responses were achieved. According to the phase diagrams of Nitinol (Fig. 3), achieved from constant stress experiment and DSC test for each wire diameter (Table 2), the whole cycle of transformation from Martensite to Austenite in heating has been performed since the wires were heated higher than 70°C which was beyond the Austenite finish temperature achieved from both methods. During cooling from high temperature to room temperature (22°C), based on the constant stress (biased stress), the transformation from Austenite to Martensite at very low stress was completed (see Fig. 3). During cooling, Austenite reverts back to Martensite and based on the amount of biased stress twinned or detwinned Martensite could be formed (Ref 37, 38).

By increasing the range of the maximum temperature in the heating cycle from 70 to 80°C and 120 to 130°C range, a high unrecovered strain was observed in wire diameter of 0.24 mm. Figure 7 demonstrates the high unrecovered strain for 0.24 mm wire at a low-biased stress (9.1 MPa) while heated to a high temperature (about 120 ± 5°C).

3.3 Uniaxial Tensile Test Results

When Nitinol wire was loaded with the initial crystal structure of twinned Martensite, beyond elastic deformation, a plateau was observed in stress-strain plot due to the reorientation of twinned to detwinned Martensite. A complete Martensite reorientation can produce up to 6% shape memory strain in Nitinol wires. The starting and finishing stresses (σ_{crsT}, σ_{crfT}) of small slope plateau are the range of critical stresses. Length of plateau was also affected by the amount of reorientation process, which was related to the maximum preconditioning temperature. Comparison within each range of temperature, between different diameters, and between different preconditioning temperatures in the same diameter, was performed. It has been observed that by increasing the wire diameter the critical stresses due to Martensite detwinning were increased. By increasing the preheating temperature to 120-130°C, the critical stress and the length of strain plateau have been increased in each wire diameter. Table 4 shows the σ_{crsT} and σ_{crfT} achieved from uniaxial tensile test (the ranges of stress plateau) for two different preconditioning temperatures.

Results of the tensile tests are presented in Fig. 8 and 9. As shown in Fig. 8, a stress plateau was observed for 0.10-0.19 mm wires. Note the wires were preconditioned at 70-80°C. It could be seen in this figure that the ranges of critical stress increased as the diameter increased. However, the stress plateau was not noticeable in 0.24 mm wire. This could be the effect of heating temperature on the Martensite reorientation process. Interestingly, as shown in Fig. 9, when the preconditioned temperature at 120-130°C, the stress plateau was observed for all wire diameters.

In short, for all wires except the 0.24 mm wires that were preconditioned at 70-80°C range, the critical stresses were at about the same value. The difference between the maximum and the minimum reported values for both starting and finishing critical stress (σ_{crsT} and σ_{crfT}) was between 5 and 25 MPa. It was also observed that as the wire diameter increased, the starting critical stress (σ_{crsT}) increased. However, a similar conclusion cannot be taken for the finishing critical stress (σ_{crfT}).

Table 4 Starting and finishing critical stresses determined from isothermal tensile test (σ_{crsT}, σ_{crfT}) for the five different wire diameters (0.10-0.24 mm) at two preconditioning procedures

	Preconditioning temperature (70-80°C)		Preconditioning temperature (120-130°C)	
Diameter, mm	σ_{crs}	σ_{crf}	σ_{crs}	σ_{crf}
0.10	100	140	136	146
0.12	115	150	142	150
0.15	120	130	146	154
0.19	125	135	140	180
0.24	NA	NA	172	176

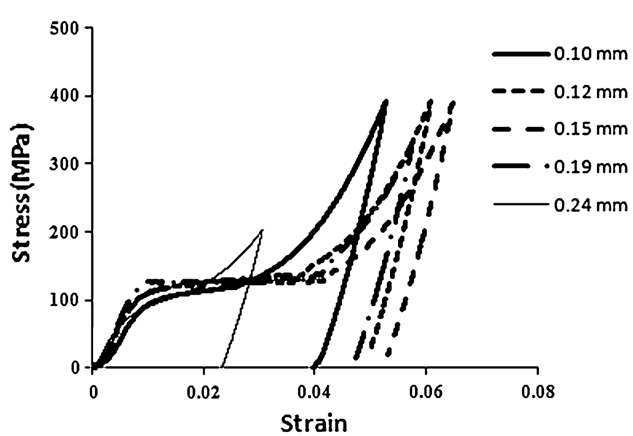

Fig. 8 Stress vs. strain response of Nitinol wires preconditioned between 70 and 80°C

3.4 Comparison of Constant Stress and Uniaxial Tensile Test Results

The comparison between the critical stresses (σ_{crsC} and σ_{crfC}) achieved from the constant stress experiment and the uniaxial tensile test (σ_{crsT} and σ_{crfT}) both at heating range of 70-80°C is presented in Fig. 10 and 11. Note that 70-80°C was the heating range in the constant stress experiment while it was the preconditioning heating range in the uniaxial tensile test. In Fig. 10, the starting critical stresses (σ_{crsC} and σ_{crsT}) obtained from constant stress experiment and uniaxial tensile test were found to be similar in most wires; the largest difference was 16 MPa for 0.12 mm wire. The comparison of 0.24 and 0.29 mm wires was not plotted in Fig. 10 and 11 because the plateau was not observed in the preconditioning temperature for those diameters. It was observed from Fig. 11 that there was a slight difference in the finishing critical stress (σ_{crfC} and σ_{crfT}) obtained from both test methods for 0.10 mm. The inaccuracy of reading where the unrecovered strain merges to zero in the

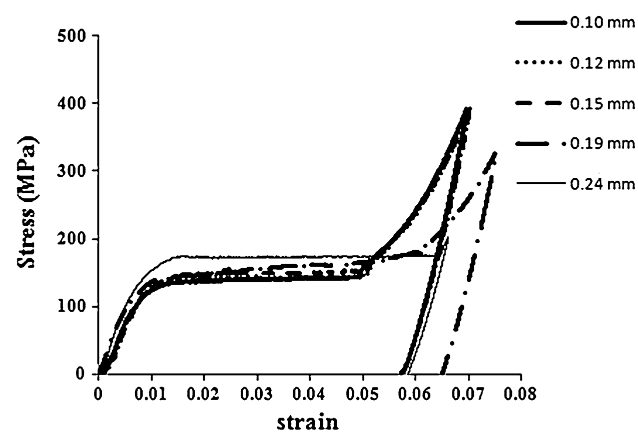

Fig. 9 Stress vs. strain response of Nitinol wires preconditioned between 120 and 130°C

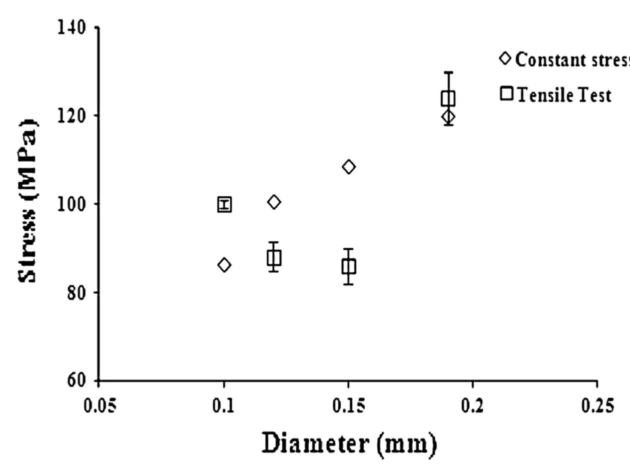

Fig. 10 Comparison of starting critical stress determined from constant stress experiment and uniaxial tensile test for Nitinol wire diameters of 0.10, 0.12 0.15, and 0.19 mm

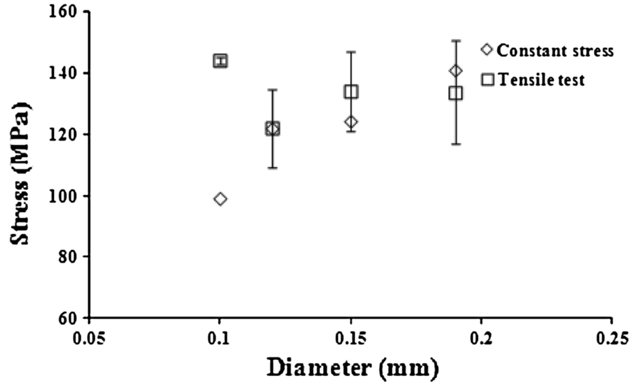

Fig. 11 Comparison of finishing critical stress determined from constant stress experiment and tensile test for Nitinol wire diameters of 0.10, 0.12, 0.15, and 0.19 mm

constant stress experiment may contribute to this difference and so the standard deviation of ±15 MPa can be considered for critical stresses that were achieved from constant stress experiment.

In constant stress experiment for 0.10-0.19 mm wires at the heating range of 70-80°C, the strain response is due to the transformation from the detwinned Martensite (initial phase) to the Austenite during heating cycle and transformation of Austenite to twinned Martensite and then detwinning process during cooling cycle (Ref 28, 37). This cyclic phase transformation can produce up to 5% recoverable strain at stress levels higher than or equal to the critical stress, which accommodate the detwinning process during cooling cycle to the room temperature. For 0.24 and 0.29 mm wires, the detwinning process did not take place at 70-80°C due to an incomplete transformation from detwinned Martensite to Austenite. Neither the stress plateau in tensile test nor the sharp drop in the amount of the unrecovered strain was observed. Ongoing work is being performed to study the microstructures of different phases (twinned and detwinned martensite) occurring in Nitinol wires due to different thermal conditions by x-ray diffraction (XRD) method.

3.5 Effect of Heating Temperature on Strain Response of Nitinol Wires

It is evident from the above discussion in Table 4 that temperature has a significant effect on the strain response of Nitinol alloy (Table 4). This effect of temperature was different for different wires due to the difference in the heat transfer rate of each wire. It is observed in this work that increasing the temperature increased the material transformed from the detwinned Martensite phase to the Austenite phase at 120-130°C.

The reorientation process in Martensite phase occurs due to the movement of twin boundaries toward the direction of the applied stress, which form detwinned Martensite (Ref 24, 29). If this process is incomplete, unrecovered strain will be generated in the wires since the final phase of the material is not detwinned Martensite (Ref 29, 36). It appears that in the heating cycle of wire diameters of 0.10-0.19 mm in the range of 70-80°C, the whole portion or approximately high volume amount of material was transformed from detwinned Martensite to Austenite. Consequently during cooling the material was transformed from Austenite to twin Martensite due to the absence of high amount of stress. High amount of unrecovered strain was generated since there were different phases before heating the wire and after cooling back to the room temperature.

On the other hand, it was observed that for 0.24 and 0.29 mm wires, the transformation of detwinned Martensite to Austenite was not complete at the heating range of 70-80°C. Consequently some volumes of detwinned Martensite are not transforming to Austenite during the first heating cycle and will remain as detwinned Martensite after cooling to the room temperature. While the portion of initial detwinned Martensite transformed to twinned Martensite after a cooling cycle to the room temperature. Lower unrecovered strains were generated due to the small differences between the initial and final phases.

By increasing the heating range to 120-130°C, the residual fraction of detwinned Martensite transformed completely to Austenite during heating. Therefore, similar to smaller diameter (0.10-0.19 mm) at lower heating range, high values of unrecovered strain were observed between the first and second thermal cycles for all wire diameters since no untransformed detwinned Martensite remained in the wires.

The presence of stress plateau in uniaxial tensile test depends on the amount of twinned Martensite in the wires before loading. Since the purpose of preheating in tensile test was to ensure complete formation of twinned Martensite in the wires, the preheating temperature plays an important role. Based on the above discussion for 0.10-0.19 mm wires at the heating range of 70-80°C, considerable fraction of twinned Martensite was formed after cooling cycle. Therefore, the stress plateau was observed in these wires during the tensile test. In contrast, for larger wire diameter at a lower heating range the stress plateau was not observed. It is conjectured that the non-existence of stress plateau is due to a negligible fraction of twinned Martensite but a high fraction of residual detwinned Martensite. In summary, increasing the heating temperature to the range of 120-130°C generated a high fraction of twinned Martensite after cooling resulting in distinctive stress during the tensile test.

4. Conclusion

The effect of wire diameters on the strain response of Nitinol wires was studied. The critical stresses, i.e., the stress level for preventing the presence of the unrecovered strain between the first and second thermal cycles, were determined by the constant stress and uniaxial tensile tests. The strain response of Nitinol wires was found different for wire diameter in range of 0.10-0.29 mm. For wire diameters of 0.10-0.19 mm, decreasing the wire diameter decreased the critical stresses. Because of the effect of preconditioning temperatures, this trend was not very obvious for critical stresses obtained from the tensile tests. For 0.24 and 0.29 mm wires heated to 70-80°C, the effect of critical stress on the strain response of Nitinol wires was not observed. The comparison of critical stresses achieved from constant stress experiment and the uniaxial tensile test showed consistent results except in the 0.10 mm diameter (inconsistency in this diameter can be due to the difficulties in reading of accurate temperature of this wire in constant stress experiment). It was observed that the range of heating temperature during heating cycle of constant stress experiment and preconditioning of uniaxial tensile test can affect the results in a great extent due to the amount of transformed initial phase (detwinned Martensite) to Austenite and consequently the amount of twinned Martensite formed after cooling to the room temperature.

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References

1. T. Duerig, A. Pelton, and D. Stockel, An Overview of Nitinol Medical Applications, *Mater. Sci. Eng. A*, 1999, **273–275**, p 149–160
2. T.K. Podder, A.P. Dicker, P. Hutapea, and Y. Yu, A Novel Curvilinear Approach for Prostate Seed Implantation, *Med. Phys.*, 2012, **39**, p 1887–1892
3. B. Konh, N.V. Datla, M. Honarvar, T.K. Podder, A.P. Dicker, Y. Yu, and P. Hutapea, Design Exploration Study of a Shape Memory Alloy Actuated Surgical Needle, *J. Intelligent Mater. Syst. Struct.*, 2014 (in press)
4. B. Konh, N.V. Datla, and P. Hutapea, Feasibility of SMA Wire Actuation for an Active Steerable Cannula, *J. Med. Devices*, 2014 (under review)
5. N.V. Datla, B. Konh, M. Honarvar, T.K. Podder, A.P. Dicker, Y. Yu, and P. Hutapea, A Model to Predict Deflection of Bevel-Tipped Active Needle Advancing in Soft Tissue, *Med. Eng. Phys.*, 2014, **36**(3), p 285–293
6. N.V. Datla, B. Konh, J.J. Koo, D.K. Choi, Y. Yu, A.P. Dicker, T.K. Podder, K. Darvish, and P. Hutapea, Polyacrylamide Phantom for Self-actuating Needle-Tissue Interaction Studies, *Med. Eng. Phys.*, 2014, **36**, p 140–145
7. B. Konh, M. Honarvar, and P. Hutapea, Application of SMA Wire for an Active Steerable Cannula, *ASME Conference on Smart Materials, Adaptive Structures and Intelligent Systems*, Snowbird, UT, 2013
8. M. Honarvar, B. Konh, N.V. Datla, S. Devlin, and P. Hutapea, Size Effect on the Critical Stress of Nitinol Wires, *ASME Conference on Smart Materials, Adaptive Structures and Intelligent Systems*, Snowbird, UT, 2013
9. N.V. Datla, M. Honarvar, T.M. Nguyen, B. Konh, K. Darvish, Y. Yu, A.P. Dicker, T.K. Podder, and P. Hutapea, Towards a Nitinol Actuator for an Active Surgical Needle, *ASME Conference on Smart Materials, Adaptive Structures and Intelligent Systems*, Stone Mountain, GA, 2012
10. W. Huang, On the Selection of Shape Memory Alloys for Actuators, *Mater. Des.*, 2002, **23**, p 11–19
11. A. Saigal and M. Fonte, Solid, Shape Recovered "Bulk" Nitinol: Part I-Tension-Compression Asymmetry, *Mater. Sci. Eng. A*, 2011, **528**, p 5536–5550
12. J.F. Dyet, G.W. Watts, D.F. Ettles, and A.A. Nicholson, Mechanical Properties of Metallic Stents: How do These Properties Influence the Choice of Stent for Specific Lesions?, *Cardiovasc. Intervent. Radiol.*, 2000, **23**, p 47–54
13. C.B. Churchill and J.A. Shaw, Shakedown Response of Conditioned Shape Memory Alloy Wire, *SPIE 6929, Behavior and Mechanics of Multifunctional and Composite Materials*, M.J. Dapino, Z. Ounaies, Eds., San Diego, CA, 2008, 6928
14. N. Troisfontaine, P. Bidaud, and M. Larnicol, Optimal Design of Micro-actuators Based on SMA Wires, *Smart Mater. Struct.*, 1999, **8**, p 197–203
15. I. Mihalcz, Fundamental Characteristics and Design Method for Nickel-Titanium Shape Memory Alloy, *Periodica Polytech Ser. Mech. Eng.*, 2001, **45**(1), p 75–86
16. L.C. Brinson, One-Dimensional Constitutive Behavior of Shape Memory Alloys: Thermomechacnical Derivation with Non-constant Material Functions and Redefined Martensite Internal Variable, *J. Intell. Mater. Syst. Struct.*, 1993, **4**(2), p 229–242
17. V. Waitz, T. Antretter, F.D. Fischer, and H.P. Karnthaler, Size Effects on Martensitic Phase Transformation in Nanocrystalline NiTi Shape Memory Alloys, *Mater. Sci. Technol.*, 2008, **24**(8), p 934–940
18. Y.Q. Fu, S. Zhang, M.J. Wu, W.M. Huang, H.J. Du, J.K. Luo, A.J. Flewitt, and W.I. Milne, On the Lower Thickness Boundary of Sputtered NiTi Films for Shape Memory Application, *Thin Solid Films*, 2006, **515**, p 80–86
19. C.P. Frick, S. Orso, and E. Arzt, Loss of Pseudoelasticity in Nickel-Titanium Sub-micron Compression Pillars, *Acta Mater.*, 2007, **55**, p 3845–3855
20. Y. Chen and C.A. Schuh, Size Effects in Shape Memory Alloy Microwires, *Acta Mater.*, 2011, **59**, p 537–553
21. J.M. San Juan, L.M. No, and C.A. Schuh, Superelasticity and Shape Memory in Micro- and Nano-meter Scale Pillars, *Adv. Mater.*, 2008, **20**, p 272–278
22. H.S. Liu, Jr, and L. Mishnaevsky, Martensitic Transformation in Nanostructured Nitinol: Finite Element Modeling of Grain Size and Distribution Effects, *Comput. Mater. Sci.*, 2013, **76**, p 27–36
23. L. An, W.M. Huang, Y.Q. Fu, and N.Q. Guo, A Note on Size Effect in Actuating NiTi Shape Memory Alloys by Electrical Current, *Mater. Des.*, 2008, **29**, p 1432–1437
24. D.W. Norwich and A. Fasching, Study of the Effect of Diameter on the Fatigue Properties of NiTi Wires, *J. Mater. Eng. Perform.*, 2009, **18**(5), p 558–562
25. S. Civjan, E.F. Huget, and L.B. DeSimon, Potential Applications of Certain Nickel-Titanium (Nitinol) Alloys, *J. Dent. Res.*, 2012, **54**(1), p 89–96
26. S. Leclercq and C. Lexcellent, A General Macroscopic Description of the Thermomechanical Behavior of Shape Memory Alloys, *J. Mech. Phys. Solids*, 1996, **44**(6), p 953–980
27. Y. Liu and J. Van Humbeeck, Luders-Like Deformation Associated with Martensite Reorientation in NiTi, *Scr. Mater.*, 1998, **39**(8), p 1047–1055
28. M.H. Elahinia, M. Hashemi, M. Tabesh, and S.B. Bhaduri, Manufacturing and Processing of NiTi Implants: A review, *Prog. Mater. Sci.*, 2012, **57**, p 911–946
29. Y. Liu and Z. Xie, Progress in Smart Materials and Structures, chap. 3, *Detwinning in Shape Memory Alloy*, P.L. Recee, Ed., Nova Science Publishers Inc, Singapore, 2007, p 29–65
30. R. Delville, B. Malard, J. Pilch, P. Sittner, and D. Schryvers, Transmission Electron Microscopy Investigation of Dislocation Slip During Superelastic Cycling of Ni-Ti Wires, *Int. J. Plast.*, 2011, **27**, p 282–297
31. Y. Liu, Z.L. Xie, J. Van Humbeeck, and L. Delaey, Effect of Texture Orientation on the Martensite Deformation of NiTi Shape Memory Alloy Sheet, *Acta Metall.*, 2009, **47**(2), p 645–660
32. D.C. Lagoudas and S.G. Shu, Residual Deformation of Active Structures with SMA Actuators, *Int. J. Mech. Sci.*, 1999, **41**(6), p 595–619
33. S. Daly, G. Ravichandran, and K. Bhattacharya, Stress-Induced Martensitic Phase Transformation in Thin Sheets of Nitinol, *Acta Mater.*, 2007, **55**(10), p 3593–3600
34. H. Sayyaadi, M.R. Zakerzadeh, and H. Salehi, A Comparative Analysis of Some One-Dimensional Shape Memory Alloy Constitutive Models Based on Experimental Tests, *Sci. Iranica*, 2012, **19**(2), p 249–257
35. J.M. McNaney, V. Imbeni, Y. Jung, P. Papadopoulos, and R.O. Ritchie, An Experimental Study of the Superelastic Effect in a Shape-Memory Nitinol alloy Under Biaxial Loading, *Mech. Mater.*, 2003, **35**, p 969–986
36. D.C. Lagoudas, *Shape Memory Alloys Modeling and Engineering Application, Chap. 2*, Springer, New York, 2008, p 64–75
37. Y. Liu and D. Favier, Stabilisation of Martensite Due to Shear Deformation Via Variant Reorientation in Polycrystalline NiTi, *Acta Mater.*, 2000, **48**, p 3489–3499
38. M. Iijima, M. Ohta, W.A. Brantley, A. Naganishi, T. Murakami, T. Muguruma, and I. Mizoguchi, Transformation Behavior of Nickel-Titanium Orthodontic Wires Under Tensile Load, *Dent. Mater. J.*, 2011, **30**(3), p 398–403