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Superelasticity in a New BioImplant Material: Ni-rich 55NiTi Alloy

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Abstract With the drive towards minimally invasive procedures, the medical industry is looking towards ‘avant-garde’ materials, with 50NiTi currently being the prime choice for many critical components/applications. This paper examines a new Ni-rich NiTi alloy that exhibits superelasticity (SE) and shape memory (SM) properties. Superelastic (SE) properties of 55NiTi* [all compositions are quoted in atomic% throughout the paper. The reader should note the following conversions: *50NiTi (at.%)≈55NiTi (wt.%)* and *55NiTi (at.%)≈60NiTi (wt.%)*] are studied here as a function of heat-treatment between 400–800°C, and compared with the corresponding response of 50NiTi*, with an aim to develop and optimize thermal treatment procedures to maximize recoverable elastic strains. While optimal tuning of the SE properties in 50NiTi necessitates cold working in conjunction with specific heat treatment/aging, 55NiTi does not require cold work to achieve its optimal SE behavior. Moreover, it can be heat treated to produce strong, stable SE and SM response from the same ingot, with transformation temperatures being a strong function of heat treatment. The main difference between the two alloys is that Ni–Ti alloys with Ni content greater than 50.6 at.% are sensitive to heat treatment; aging in these materials leads to precipitation of several metastable phases. The initial work focuses on SE properties relevant to biomedical use, such as: plateau stress, recoverable strains and strength, as a function of heat treatment and microstructure.

Keywords Ni-rich Nitinol · Superelasticity · Heat-treatment · Precipitates

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Introduction

The commercial success of ‘superelastic’ Nitinol was essentially triggered by the medical industry’s drive towards minimally invasive procedures that demanded non-conventional materials [1–4]. Nitinol, among many contenders, proved to be the vanguard due to its unique superelastic (SE) properties. Indeed, the success of Nitinol alloys was due to the ability to optimize the superelastic ‘window’ around the constant body temperature of 37°C [1, 2]. One of the most important parameter that governs the transformation temperatures in Ni–Ti alloys is the Ni composition. Even an increase in Ni composition by 1%, from 50.5 to 51.5 at.%, decreases the M_S (martensite start temperature) to below 100 K (in solution-treated condition) [5]. Such high sensitivity to Ni composition is highly undesirable in many applications. However, changes in both the transformation temperatures and the superelastic properties of NiTi may be achieved through thermo-mechanical treatments, specifically, either through cold work or aging heat treatments or even a combination of both [6, 7]. In near-equiatomic (cold-worked) NiTi alloys, the high density of dislocations constrain the martensitic transformation that distorts the lattice, whereas the resistance in Ni-rich NiTi alloys is provided by the introduction of both dislocations and precipitates. This leads to a variation in the transformation temperatures and hence the variation of superelastic ‘window’ in which the material can exhibit a stable superelastic effect. Additionally, this resistance also leads to multiple transformation paths, from B2 (austenite)—R-phase—B19’ (martensite) phase [6]. Aging leads to the rearrangement of the dislocation networks [6, 8] in 50NiTi or precipitation and grain growth in Ni-rich NiTi alloys [9]. Earlier aging studies done on Ti-50.6Ni indicates that the aging temperature controls the size and distribution of the Ti_3Ni_4 precipitates, which in turn determine the extent of recoverable strains in superelasticity [6]. The combination of both cold-work and aging heat-treatment on Ti-50.6Ni was seen to increase the plateau stress and also the stability of the superelastic curves [6, 10]. The combination of cold work and aging also eliminates the preliminary step of solution treatment, thus being economical in some cases. Moreover, in Ni-rich Nitinol alloys that exhibit precipitation hardening, the Ni content of the matrix is progressively reduced upon aging at higher temperatures and larger times, with excess Ni being partitioned into the Ni-rich precipitates (such as Ti_2Ni_3 or $TiNi_3$), thus providing an excellent handle to control the transformation temperatures [2].

Early studies on Nitinol encompassed alloys with different compositions, ranging between 50–56 at.% NiTi [11, 12]. However, due to difficulty in forming and working with Ni-rich alloys (around 54–56 at.% NiTi) that exhibited extreme brittleness and high notch sensitivity, machining and forming these materials into near-net shapes for practical applications became difficult and impractical [13, 14]. Since the development of hot rolling methods for 55NiTi and special fabrication techniques, a number of patents have emerged that exploit some unique properties of the alloy, such as: high hardness, non magnetic character, immunity against most corrosive agents, high strength and toughness [14, 15]. A decade ago, it was shown that under special heat treatment conditions, both the SM and SE effects could be produced in the alloy from the same ingot [15], without any additional mechanical processing. Since then, the interest in the development of Ni-rich 55NiTi alloys has seen a resurgence, although very little work exists on the mechanical properties of these alloys [14, 16].

The current work addresses the superelastic properties of 55NiTi that are crucial for any development towards medical device applications. The main difference between 50NiTi and 55NiTi is that Ni–Ti alloys with Ni content greater than 50.6 at.% are sensitive to heat treatment; aging in these materials leads to precipitation of several metastable phases [9]. Therefore, this initial work focuses on SE properties such as plateau stress, recoverable strains and strength as a function of heat treatment and microstructure.

Table 1 Aging schedule for 50NiTi and 55NiTi alloys

Alloys	Aging schedule
50NiTi: HT-1	400, 500, 600, 700°C: 1 h
55NiTi: HT-1	400, 500, 600, 700, 800°C: 1 h
55NiTi: HT-2	500, 600, 700°C: 5 h
55NiTi: HT-3 (Step aging)	500°C (5 h)+400°C (1 h), 600°C (5 h)+500°C (1 h), 700°C (5 h)+600°C (1 h)
55NiTi: HT-4	ST+600°C (24 h), ST+700°C (24 h), ST+800°C (24 h)

Experimental Procedure

Materials and Methods

Equiatomic Nitinol (Ti-50Ni) in the form of 1 mm thick rolled sheet was obtained from *Nitinol Devices and Components, Inc. (NDC, Fremont, CA)*. The austenite finish temperature is 14°C, and therefore, the material was in superelastic-austenitic condition at room temperature (RT ~ 22°C).

Ni-rich Nitinol, Ti-55Ni alloys were obtained from *Nitinol Technologies, Inc. (Edgewood, WA)* in the form of ~3 mm plates that were hot-rolled at 950°C. Numerous dog bone tensile specimens of a 25 mm gage length and measuring 6.4 mm width × 3 mm thickness were cut from this stock and aged according to the schedule given in Table 1. For the solution-treatment (ST), the specimen was held at a temperature of ~1,100°C for 1 h in evacuated quartz tubes and then quenched into water. A minimum of two samples (often 3–4) were tested at each test condition, and typical stress-strain data are presented for comparison.

Microstructure

The microstructures of as-received 50NiTi and 55NiTi are shown in Figs. 1(a) and 2(a), respectively. The microstructure of 50NiTi sheet was similar to that observed by Robertson et al. [17], with an average grain size less than 10 μm. The as-received (AR) 55NiTi contained precipitates of Ti_2Ni_3 and $TiNi_3$ in various morphologies. The major secondary phase was the $TiNi_3$ precipitate and was typically in an elongated, needle shape [see Fig. 1(b)], whereas, Ti_2Ni_3 precipitates occurred in lenticular morphologies and were only occasionally observed, see Fig. 1(d). It has been known that NiTi alloys containing more than 50.6 at.% Ni decompose on aging through the precipitation of Ti_3Ni_4, Ti_2Ni_3 and $TiNi_3$, in that order, with Ti_3Ni_4 and Ti_2Ni_3 being metastable phases [9, 18]. Typically, Ti_2Ni_3 and subsequently $TiNi_3$ form at higher aging temperatures and times [6]. Ti_3Ni_4 is usually formed at lower aging temperatures (~400–500°C) and shorter times, with the upper temperature limit for the formation of a particular precipitate being a function of Ni composition in the Nitinol alloys [18]. Due to the hot rolling of 55NiTi, the precipitates have been deformed severely and extensive dislocations in $TiNi_3$ precipitates can be seen in Fig. 1(b).

Another major difference between 50NiTi and 55NiTi is evident upon observing their microstructure in solution-treated (ST at 1,100°C and water-quenched) condition. While 50NiTi shows a typical twinned martensitic microstructure [see Fig. 2(b)], 55NiTi does not exhibit any twinned microstructure, i.e., it formed cubic austenite phase [see Fig. 2(c)]. Moreover, upon furnace cooling (FC) of the materials from 1,100°C, 50NiTi formed only the austenitic

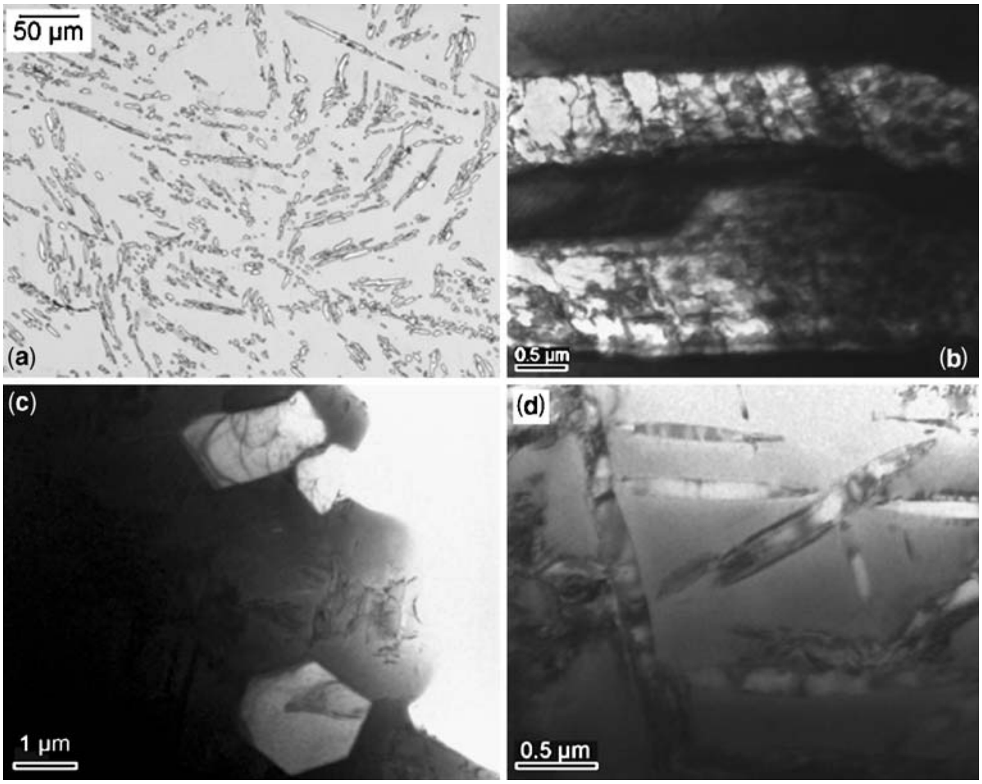


Fig. 1 (**a**) Optical micrograph of AR 55NiTi. TEM micrographs of (**b, c**) $TiNi_3$ and (**d**) Ti_2Ni_3 present in the AR structure. Notice the large deformation evident in $TiNi_3$ precipitates due to initial hot-rolled plate

phase, whereas 55NiTi formed an extensive network of $TiNi_3$ precipitates, as shown in Fig. 2(d). The furnace-cooled 55NiTi also exhibited shape-memory characteristics (M_s= 16°C, M_f=0°C, A_s=38°C, A_f=57°C). $TiNi_3$ precipitate formed both on the grain boundaries that exhibited blocky morphology and in the grain interior in the form of long elongated needles of different lengths. In the grain interior, lenticular martensite was also observed along with the $TiNi_3$ precipitates.

Heat Treatment: Aging Schedule

Following the procedure recommended by [15], the as-received (AR) 55NiTi material was aged between 400 and 800°C. Various aging schedules that were followed are summarized in Table 1. While HT-1 and HT-2 were single-step aging at various temperatures for 1 and 5 h, respectively, HT-3 consisted of step-aging treatment. Finally, solution-treatment (at 1,100°C) followed by aging between 600 and 800°C for 24 h was accomplished. To compare the superelastic properties of 55NiTi against the popular 50NiTi, annealing of 50NiTi sheet was accomplished according to the schedule indicated in Table 1.

Testing and Characterization

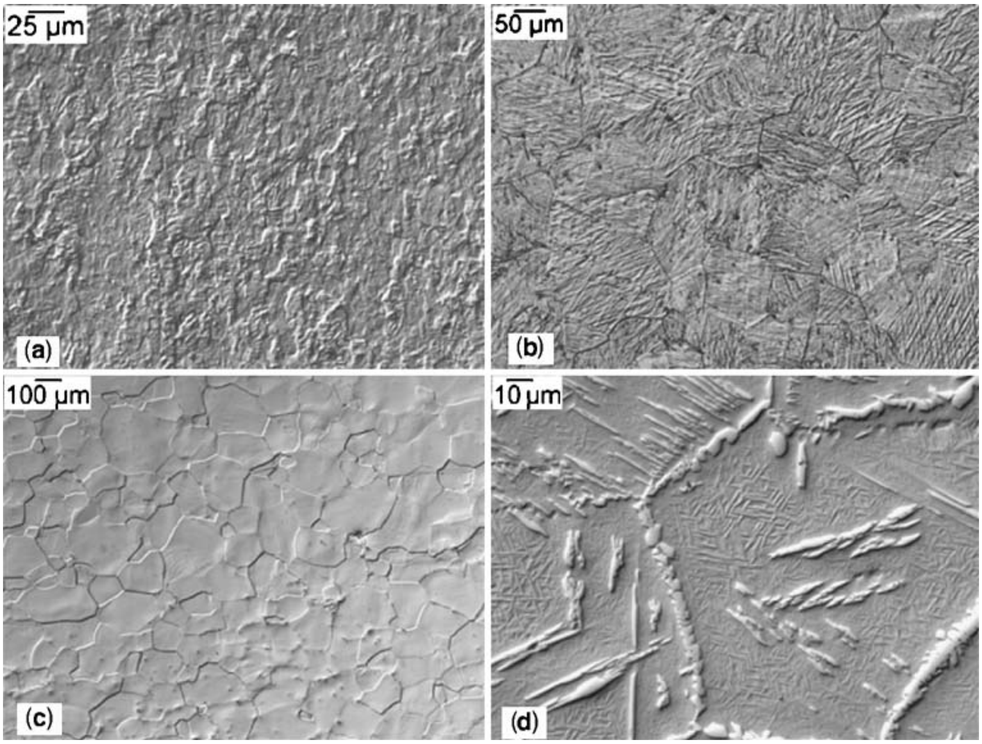
Tensile testing of both 50NiTi and 55NiTi and compression testing of 55NiTi was done using a standard servo-hydraulic load frame at a quasi-static strain rate of ~10^{-3}/s. Metallographic studies were done on specimens that were mechanically polished to diamond finish and then etched with HF+ HNO_3+deionized water. In addition, 3 mm diameter disk specimens measuring 0.1–0.2 μm in thickness were prepared for TEM studies on a 120 kV Philips EM420. Prior to the TEM observation, they were electropolished using the twin jet method that utilized electrolyte of composition 20% H_2SO_4+80% Methanol at 10°C and 15–20 V.

Results and Discussion

As-received (AR) vs. Solution-treated (ST)

The tensile stress-strain curves of as-received (AR) and solution-treated (ST) 50NiTi and 55NiTi are shown in Fig. 3. The ST material of both compositions are extremely brittle and exhibit strengths of ~350–400 MPa. The AR 50NiTi exhibits typical superelastic behavior with plateau strains between 1–7% coupled with a ductility of ~25%, whereas, 55NiTi displays a non-flat 'plateau behavior' between 1–4%. The reason for the positive slope of the 'plateau' (strictly speaking, 55NiTi does not exhibit a zero-slope plateau, but for the purpose of comparison, the term 'plateau' will be used in this context throughout the paper) is due to the presence of the precipitates that resist the stress-induced transformation, and as a result, progressively

Fig. 2 Optical micrographs of (**a**) AR 50NiTi sheet, (**b**) solution-annealed (1,100°C) and water-quenched 50NiTi, notice the twinned martensite microstructure, (**c**) solution-annealed (1,100°C) and water-quenched 55NiTi and (**d**) solution-annealed (1,100°C) and furnace-cooled 55NiTi. Notice the $TiNi_3$ precipitate formation both on the grain boundary and grain interior



higher stresses are necessary to drive the transformation to completion [19–21]. Similar to the well known compression-tension asymmetry in 50NiTi [22–27], 55NiTi also exhibits a large asymmetry in tension-compression behavior, as shown in Fig. 3(a). The strength levels in both AR and ST condition for 55NiTi reach nearly ~2,500 MPa under compression. Additionally, the superelastic 'plateau' is evident in the AR 55NiTi material compression curve, although the slope in compression is greater than the slope in tension.

Heat-treatment (HT-1): Compression and Tension

The compression stress-strain curve for 55NiTi heat-treated according to the HT-1 schedule is shown in Fig. 4 along with the AR and ST compression data. The variation in the overall stress-strain behavior due to different aging temperatures is not significant. The major observations are that the 'plateau' disappears for the specimens aged at 800°C (1 h), while only a small portion of the curve for 700°C exhibited the 'plateau.' Compared to the AR material, however, the ductility and strength levels have increased to about ~30% and 3 GPa, respectively.

The tensile stress-strain curves for 55NiTi and 50NiTi for HT-1 aging conditions are shown in Fig. 5(a) and (b), respectively. Similar to the compression behavior in 55NiTi, the tensile 'plateau' is clearly evident in 55NiTi aged between 400 and 600°C, barely visible at 700°C, and completely disappears at 800°C. Additionally the 'plateau' start-stress increases as the aging temperature is increased from 400 to 700°C. Moreover, the typical strain range for the 'plateau' was ~1–5%, with reversible strain up to 3–4%. It may be possible to optimize the reversible elastic-strains up to 6% in these Ni-rich 55NiTi alloys [15]. Another interesting property of these alloys is that they are extremely hard. For example, depending on the aging temperature and time, the hardness of the SE 55NiTi can be varied between 40–70 R_C (Rockwell C hardness).

As we will see in the subsequent section, the aging treatment given after solutionizing treatment does not provide enough ductility and the concomitant precipitation

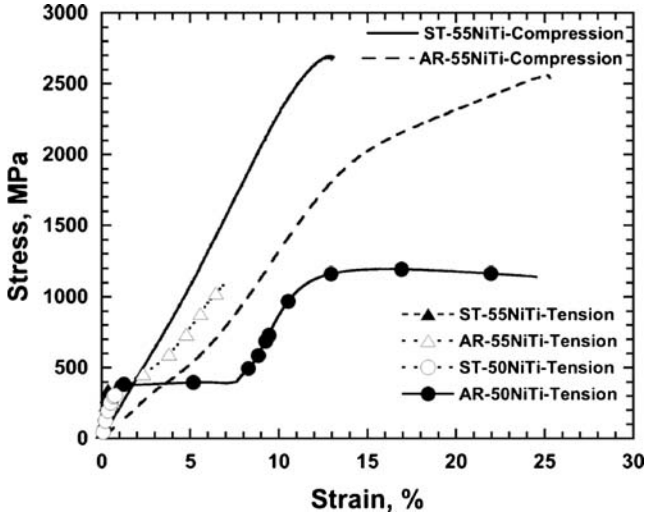


Fig. 3 Quasi-static tension stress-strain curves for 50NiTi and 55NiTi for AR and ST heat-treatment conditions. Compression curves for AR and ST 55NiTi are also included

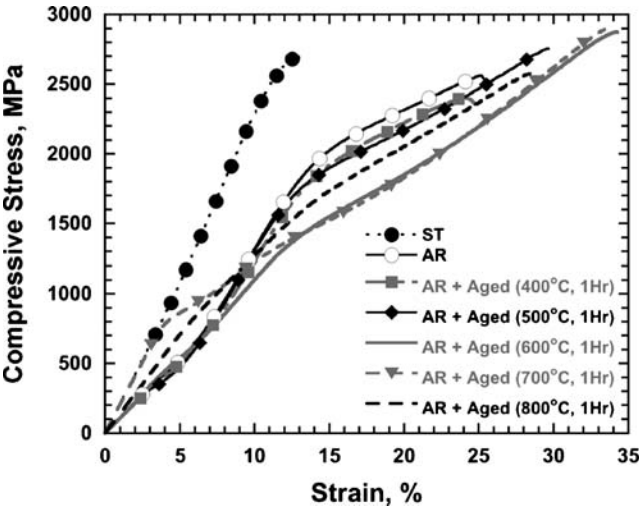


Fig. 4 Compression stress-strain curves for 55NiTi heat-treated according to the HT-1 schedule indicated in Table 1

hardening is also not enough to provide adequate strength in order to encourage any superelasticity in the matrix for the HT-4 heat-treatment condition. This would suggest that solution treating and cold-working, as necessary parameters to tune the superelasticity, are obviated in comparison to 50NiTi, where they are very crucial. This would render the process of using these alloys very economical, since the essential SE or SM properties can be tuned using aging heat-treatments only (in addition to perhaps additional preliminary hot-rolling steps).

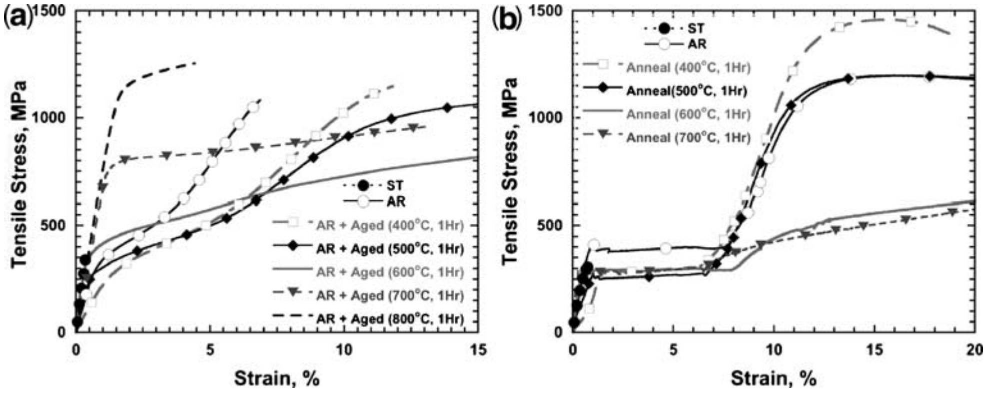
The effect of HT-1 annealing of 50NiTi on its stress-strain behavior is shown in Fig. 5(b). The first manifestation of annealing is the decrease of plateau stress from ~400 to ~250–300 MPa. Additionally, the ductility due to annealing at 600 and 700°C was seen to increase from 25% in the AR condition to nearly 60% (the data in Fig. 5(b) is shown only until 20%), whereas, the tensile strength drops from 1,200 MPa to below 1,000 MPa (see [28]). The plateau strains are essentially unchanged, *i.e.*, between 1–6.5%. An interesting observation is that 50NiTi specimens annealed at 400 and 500°C exhibited an early plateau (at a low stress level of ~50 MPa) in a very small strain range, indicating the formation of R-phase that was subsequently followed by the larger plateau at ~250 MPa, indicating the formation of B19’ martensite phase.

Heat-treatment (HT-2, 3, 4): Tension

Aging of the AR 55NiTi was also accomplished for 5 h single-step and (5+1 h) two-step heat-treatments at different temperatures. The corresponding tensile stress-strain results are shown in Fig. 6(a) and (b), respectively. Aging for longer times typically reduced the ‘plateau’ start stress and the tensile strength, while an increase in ductility by 2–5% was observed. However, the post-‘plateau’ work hardening rate was similar to the specimens aged for 1 h, see Fig. 6(a). In the two-step aged specimens, while the ‘plateau’ start stress was reduced, the tensile strength remained nearly the same compared to 5 h single-step aging (HT-2). Additionally, the slope of the ‘plateau’ region increased under the two-step heat-treatment; see Fig. 6(b).

Figure 7(a) shows the stress-strain data for compression and tension loading of 55NiTi specimen that were solutionized (solution annealed at 1,100°C for 1 h and subsequently water quenched) and then aged for 24 h at 600, 700 and 800°C. The tensile curves [see the inset in Fig. 7(a)] indicate that the material is extremely brittle with low strength and hence not preferable for any SE applications. It was, however, in these aging conditions, that the largest asymmetry between tension and compression in 55NiTi was observed. The compression results indicate good ductility of nearly 20% and strength levels in excess of 2 GPa. As pointed in the earlier section and also shown elsewhere [9], the precipitation products are dependent on the aging temperature and time. In the case of 55NiTi, aging at 600, 700 and 800°C for 24 h leads to the precipitation and growth of Ti_3Ni_4+Ti_2Ni_3, Ti_2Ni_3+$TiNi_3$, and $TiNi_3$ precipitates, respectively. Optical micrographs of 55NiTi, which were solutionized and then aged for 24 h for 600, 700, and 800°C are shown in Fig. 7(b), (c), and (d), respectively. In Fig. 7(b) and (c) the precipitates grow to span the whole grain, while

Fig. 5 Tensile stress-strain curves under HT1 heat-treatment condition for (**a**) 55NiTi and (**b**) 50NiTi



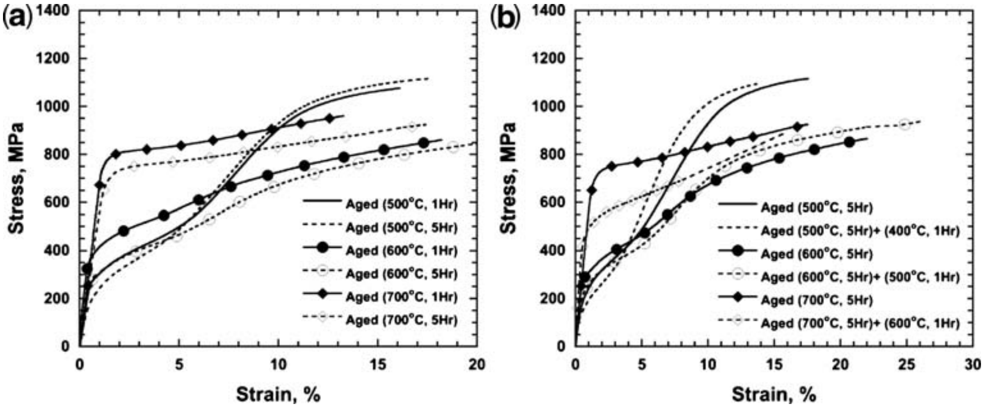


Fig. 6 Tensile stress-strain curves of 55NiTi aged according to HT-2, 3 heat-treatment conditions (**a**) Single-step and (**b**) Two-step HT

in Fig. 7(c), blocky grain-boundary and needle-like grain-interior $TiNi_3$ precipitates and precipitate-free-zones (PFZ) near these grain boundaries were observed.

Conclusions

Preliminary studies have shown many promising features of 55NiTi as a candidate for future medical applications. It has been shown that 55NiTi possess some unique properties such as, high strength and very high hardness (can be varied between 25–70 Rockwell C hardness) along with low thermal conductivity and low modulus [15]. More importantly, 55NiTi allows extremely fine surface finish [15] that is very important for medical devices as the susceptibility of failure in these devices is higher for surface defects that arise due to non-optimal surface finish than bulk volume defects (such as scratches or pitting during electropolishing). In addition, 55NiTi is non-magnetic and immune to most corrosive agents [15]. Above all, it has been shown in this work and elsewhere [14–16] that both SE and SM properties may be obtained from the same ingot without resorting to cold-work or solutionizing treatments or a combination of both, which are usually very expensive. A range of SE and SM properties may be obtained by strictly controlling the microstructure through various aging conditions only. The lone shortcoming in these alloys is that the range of recoverable superelastic strains is about 60–75% less than conventional 50NiTi due to the presence of large precipitates that typically resist the stress-induced

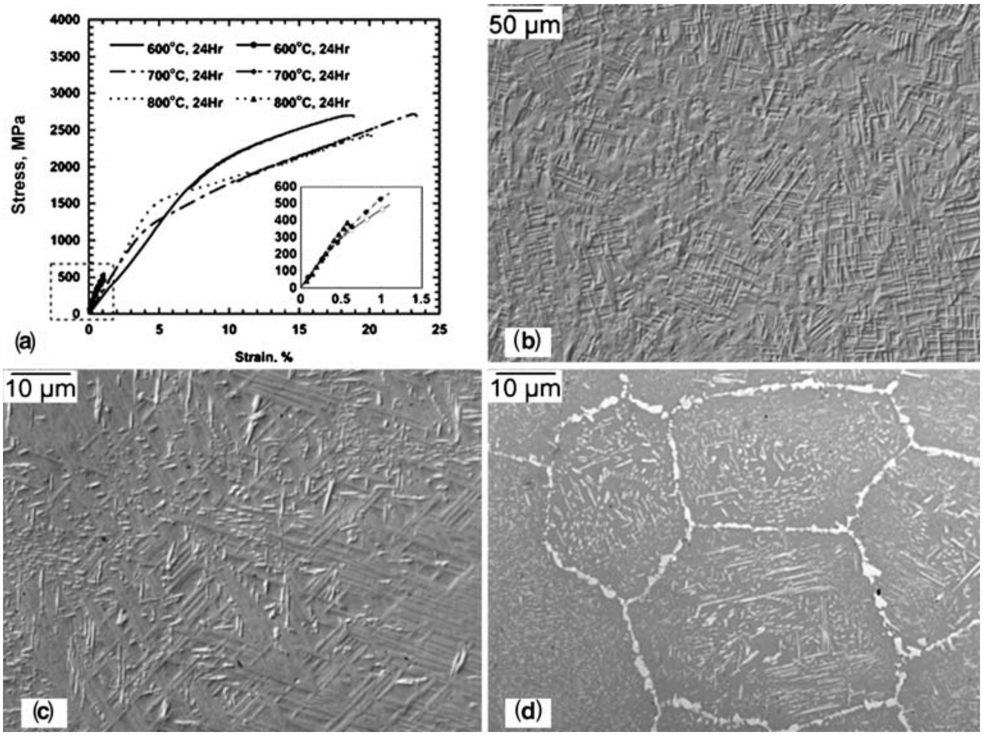


Fig. 7 (**a**) Tension and compression stress-strain curves for solution-treated (ST)+aged (for 24 h) 55NiTi. The tension data is enlarged and shown in the inset. Optical micrographs of 55NiTi that were solutionized and then aged for 24 h for (**b**) 600°C, (**c**) 700°C, (**d**) 800°C. In (**b**) and (**c**), due to the presence of very large precipitates that span the whole grains, differential interference contrast imaging was used to reveal the microstructure. Ti_3Ni_4 and Ti_2Ni_3 precipitates at 600°C (24 h), and Ti_3Ni_4 and Ti_2Ni_3 precipitates at 700°C (24 h) are precipitated on aging; (**d**) is a standard bright field image. Notice the presence of blocky grain boundary and needle-like grain interior $TiNi_3$ precipitates and precipitate-free-zones (PFZ) near these grain boundaries

martensitic transformation. However, given the fact that the superelastic window in these alloys can range between −150 and 600°C [15], and combined with the afore-mentioned properties, this preliminary work encourages further study of 55NiTi for exploitation in critical medical device applications and elsewhere. Currently, work is underway to optimize the SE and SM properties of 55NiTi in order to wheedle maximum reversible strains in these alloys.

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