Tensile shape memory behavior of Ni_{50.3}Ti_{29.7}Hf₂₀ high temperature shape memory alloys

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The effects of heat treatment on the shape memory characteristics of a polycrystalline Ni_{50.3}Ti_{29.7}Hf₂₀ alloy were studied via thermal cycling under stress and isothermal stress cycling experiments in tension. It was revealed that transformation temperatures could be increased above 100 °C with aging at temperature above 500 °C and in particular were stabilized against stress-free thermal cycling after aging at 500 °C. Recoverable strain of ~5% was observed for the as-extruded samples and decreased to ~4% after aging due to the formation of non-transformable precipitates. The aged alloys demonstrated near perfect shape memory effect under tensile stresses as high as 700 MPa and perfect superelasticity at temperatures up to 230 °C. Finally, the tension-compression asymmetry observed in NiTiHf alloys was discussed.

Introduction

Shape memory alloys (SMAs) have attracted considerable interest for many applications in biomedical, civil, automotive and aerospace industries [1-6] due to their unique capabilities to recover large deformations, generate high stress, and high damping capacity [7-10]. It is known that the martensitic transformation in SMAs can be induced by thermal cycling and by applied stress. But the final mechanical and functional properties are also dependent on the deformation modes, structure of the transforming phases, martensite morphology, microstructure (including precipitate characteristics), and crystallographic orientation [2].

Furthermore, the stress-strain behavior of NiTi is asymmetric with stress state [11-16]. The tension-compression asymmetry of SMAs is well established experimentally, which can be attributed to the difference in deformation mechanisms with applied stress direction and the unidirectional nature of twin deformation [12-14]. NiTi demonstrates smaller recoverable strain, higher critical stress to induce martensitic transformation (σ_{SIM}), and steeper transformation stress-strain slopes in compression than in tension [11, 15, 17, 18]. For instance, the σ_{SIM} is ~200

MPa under compression while it is greater than 300 MPa in tension for $\text{Ni}_{50.8}\text{Ti}_{49.2}$ after 400 °C-1.5h aging [13]. In another study on textured $\text{Ni}_{50.8}\text{Ti}_{49.2}$ after 500 °C-15h aging [19], it was revealed that the recoverable strain was two times larger in tension than in compression. Also, the critical stress for transformation is found to be higher in tension as compared to torsion during the deformation of equiatomic NiTi [20].

Microstructural investigations on deformed equiatomic NiTi alloys confirmed that the deformation mechanism for martensite is different in tension and compression [12, 21]. After tensile deformation, $\langle 011 \rangle$ type II twinning is more frequently observed and two neighboring martensite variants become (001) twin related to each other. This (001) twin boundary is highly mobile under tension and as a result, two self-accommodated variants can reorient into one variant that is favorable to the applied stress. In addition, no significant plastic deformation is observed inside the twin bands, while dislocations are decorated along the variant interfaces. On the other hand, a high density of dislocations is observed inside twin bands and variant accommodated areas after compressive deformation. Boundaries between two martensite plates containing $\langle 011 \rangle$ type II twins are not twin related to each other, resulting in no interfacial movement in compression. Thus, deformation proceeds by continuously increased compressive load and results in a high stress-strain slope in the transformation region, where plateau type deformation is typically observed in tension.

NiTi binary alloys exhibit excellent shape memory properties, but due to low transformation temperatures (TTs) they cannot be employed at temperatures above 100 °C, therefore, ternary elements such as Hf, Zr, Pd, Pt and Au are added to NiTi to increase the TTs. Amongst all elements, Hf and Zr are the most practical alloying additions to develop high temperature shape memory alloys (HTSMAs) since the precious metal additions are prohibitively expensive for most practical applications [2, 4]. NiTiHf alloys demonstrate excellent dimensional stability at high stress levels, perfect superelastic behavior at high temperatures, and good cyclic stability after appropriate heat treatments [22-24]. Most investigations have been focused on the effects of precipitates on the shape memory properties [22, 23], orientation dependence [25, 26], and microstructural characterization [27] on $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ alloys. With the exception of [23] these studies have focused on the shape memory properties of NiTiHf alloys under compression and to the author's knowledge there has been only a single study that has experimentally addressed the

tension-compression asymmetry of aged $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ alloys [23]. In that study by Bigelow et al., the transformation strain was 3.74 % in tension and 2.24% in compression during thermal cycling under 500 MPa [23], clearly demonstrating a strong tension-compression asymmetry.

NiTiHf SMAs are becoming a promising candidate for use in variety of applications in the aerospace, automotive and energy industries where they will be subjected to bending and/or multiaxial loads. Thus, it is important to study the shape memory behavior of NiTiHf under different stress states. In this study, shape memory properties and superelastic behavior of $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ have been investigated in tension. The alloys were aged at selected temperatures for three hours to study the effects of precipitation on tensile behavior. Additionally, the results are compared to the previous shape memory characteristics reported for this material under compression and aspects of the observed tension-compression asymmetry discussed.

Experimental Procedures

An ingot with aim composition $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ was induction melted and cast into a 1 inch diameter copper chill mold. The ingot was homogenized at 1050 °C for 72h in vacuum, then extruded at 900 °C with 7:1 area reduction followed by air cooling to room temperature. $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ alloys transform from cubic B2 austenite to monoclinic B19' martensite. This condition is referred to “as-extruded” throughout the text. Samples were additionally heat treated for three hours at selected temperatures varying from 300 to 800 °C followed by a water quench. Dog bone shaped tensile specimens with a gauge length, thickness and width of 12 mm, 3 mm and 2 mm, respectively, were cut using an electro discharge machine from the as-extruded and aged samples. A Perkin Elmer Pyris 1 DSC with heating/cooling rate of 10 °C/min was used to determine the transformation temperatures. The thermo-mechanical experiments were conducted using an MTS Landmark servohydraulic test frame with custom tension grips. The level of applied force was measured by a 100 kN capable load cell and the axial strain was measured with an MTS high temperature extensometer with a gauge length of 12 mm. Heating of the sample was achieved by conduction through the tension grips at a rate of 10 °C/min and cooling was achieved by flowing liquid nitrogen through copper coils wrapped around the grips resulting in a cooling rate of 5 °C/min. Temperature was controlled using a PID driven Omega temperature controller.

Results

DSC responses of the $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ alloy, heat treated at selected temperatures from 300 to 800 °C for a duration of 3 hours, are shown in Figure 1a. Each sample was thermally cycled three times in the DSC to determine the stability of the transformation peaks. Figure 1b compares the martensite start temperature (M_s), austenite finish temperature (A_f), and thermal hysteresis of the aged samples. Transformation temperatures (TTs) were determined by using the tangent line method and thermal hysteresis was calculated as the difference between A_f and M_s . The M_s is below 100 °C after aging at low temperatures (< 500 °C). After an initial drop at 400 °C, TTs increased with aging temperature and M_s reached a maximum temperature of 150 °C after 600 °C-3h aging. In $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$, the formation of H-phase precipitates depleted Ni from the matrix and increased TTs [27]. Finally, TTs decreased with further increase in aging temperature which can be attributed to the lower volume fraction of H-phase precipitates. Thermal hysteresis decreased from 35 to 25 °C as the aging temperature increased from 300 to 500 °C and then increased moderately with aging temperature reaching a maximum value of 50 °C for 700 °C-3h aged condition. Three hours aging at 500 °C and 550 °C resulted in moderate increases in transformation temperature, minimum hysteresis, and good thermal stability for the transformation peaks. Thus, these two aged conditions were selected along with the as-extruded condition for more detailed analysis of the shape memory properties of the $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ alloy under tensile stress.

Figure 1: (a) DSC responses and (b) transformation temperatures (M_s and A_f) and thermal hysteresis of $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ (determined from the first thermal cycle) as a function of aging temperature.

Figure 2 represents the thermal cycling behavior of $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ under applied tensile stress for the as-extruded, 500 °C-3h and 550 °C-3h aged conditions. All the samples were loaded in the fully austenitic condition and then thermally cycled between a temperature below M_f and a temperature above the A_f under a constant tensile stress. After a thermal cycle was completed, the sample was loaded to the next stress level and the thermal cycle was repeated. Consistent with the DSC response at zero load, the TTs increased with aging temperature and for a particular condition increased with stress level.

Figure 2a shows the thermal cyclic behavior under tensile stress of the as-extruded alloy. The M_s increased from 75 to 165 °C as the applied stress was increased from 100 to 600 MPa. The transformation (recoverable) strain was 1.9% at 100 MPa and increased to 4.7% at 500 MPa. Near perfect shape memory effect with a small irrecoverable strain of 0.2 % was observed at 500 MPa. However, a transformation strain of 4.8% with an irrecoverable strain of 0.7% was observed during thermal cycling under 600 MPa. Furthermore, thermal hysteresis increased with stress, growing from 33 °C under 100 MPa to 63 °C under 500 MPa.

Figure 2: Strain-temperature response of $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ in the (a) as-extruded, (b) 500 °C-3h aged, and (c) 550 °C-3h aged conditions under applied tensile stress.

The load-biased shape memory behavior of $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ after 500 °C-3h aging is shown in figure 2b. It is clear that the strength of the material was improved after aging at 500 °C-3h, because near perfect dimensional stability with negligible irrecoverable strain was demonstrated

at 500 MPa and only a small residual strain of 0.1% was obtained at 700 MPa. Recoverable strains of 1.4%, 3.4% and 3.9% were achieved during thermal cycling under 100, 500 and 700 MPa, respectively and the thermal hysteresis was less than 25 °C for stress levels between 100 MPa and 600 MPa. Finally, M_s was shifted to temperatures above 100 °C, making this a good candidate material for high temperature applications.

Figure 2c illustrates the load-biased shape memory behavior of $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ aged at 550 °C-3h. The TTs were the highest among the three conditions examined in detail. For example, M_s was 157 °C at 100 MPa increasing to 200 °C at 500 MPa. Furthermore, perfect dimensional stability with a recoverable strain of 3.7% was achieved at 500 MPa. Thermal hysteresis was about 25–30 °C for stress levels up to 500 MPa.

Isothermal tensile stress-strain responses of the as-extruded, 500 °C-3h and 550 °C-3h aged $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ at selected temperatures above A_f are illustrated in figure 3. Full recovery was not exhibited in the as-extruded condition after unloading from 5% applied strain, however, the residual strain was fully recovered upon heating of the sample to 300 °C. As anticipated, the critical stress to induce martensitic transformation (σ_{SIM}) increased with test temperature for all cases. In the as-extruded condition, the σ_{SIM} increased from 500 MPa at 160 °C to 680 MPa at 200 °C, but the value decreased after each stress cycle (Figure 3a). This could be attributed to the presence of retained martensite and plastic deformation, which assists the formation of stress induced martensite in each successive stress cycle.

Perfect superelasticity with 4% recoverable tensile strain at temperatures of 190–210 °C was observed in the 500 °C-3h aged alloy as shown in Figure 3b. The σ_{SIM} increased from 530 MPa at 190 °C to 730 MPa at 230 °C and an applied stress of 1000 MPa was reached after 4% deformation. A negligible irrecoverable strain of 0.05% was observed after 3% deformation at 230 °C. Figure 3c illustrated that aging at 550 °C-3h resulted in perfect superelastic behavior at temperatures between 180 °C and 220 °C. For this condition, σ_{SIM} was 400 MPa at 180 °C and increased to 640 MPa when the test temperature was raised to 220 °C.

Figure 3: Stress-strain response of the $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ alloy in the (a) as-extruded, (b) 500 °C-3h, and (c) 550 °C-3h conditions.

The stress-strain curves were more stable and the dissipation of energy (hysteresis) was lower after aging in contrast to the as-extruded condition. The mechanical hysteresis was determined as the stress difference between loading/unloading curves in the middle of the transformation regime. The mechanical hysteresis was strain dependent and increased from ~280 MPa after 3% deformation to ~380 MPa after 5% strain at 180 °C for the as-extruded condition. In contrast, the mechanical hysteresis was in the range of 160-220 MPa and 200-280 MPa for 500 °C-3h and 550 °C-3h aged conditions, respectively. It is worth noting that the dissipation energy during thermal and stress induced transformation were well correlated. The as-extruded samples demonstrated high thermal and mechanical hysteresis, whereas, low temperature and stress hysteresis were observed after 500 °C-3h aging.

Discussion

Figure 4a compares the recoverable and irrecoverable strains of $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ alloys observed in tension from the current study and compression from the literature [22]. It is clear that recoverable strain increases initially with applied stress, which is attributed to an increase in the volume fraction of favorable martensite variants activated with stress. Then, the recoverable strain saturates and eventually decreases with further increase in stress due to plastic deformation. For instance, the recoverable strain for the as-extruded sample in tension increased from 1.9% under 100 MPa and saturated at a maximum value of 4.7 % at 500 and 600 MPa. However, 0.2 % irrecoverable strain was observed at 500 MPa increasing to 0.7 % at 600 MPa.

Testing at higher stresses would be expected to result in a diminishing recoverable strain due to rapidly increasing plastic deformation.

It is clear from Figure 4a that the recoverable strain at small tensile stresses, such as 100 MPa, was low for all sample conditions due to the fact that the stress was not sufficient to activate significant favorable martensite variants and instead mostly self-accommodated martensite plates formed during the martensitic transformation. Tensile strains after three hours aging at 500 and 550 °C were almost identical for both aging conditions but lower than the strains of the as-extruded sample. Formation of non-transformable precipitates after aging decreased the volume fraction of transforming material and at least in part was responsible for the smaller tensile strain. Finally, the recoverable strains in tension were generally higher than ones earlier observed in compression [22], consistent with previous experimental results by Bigelow et al. [23] and theoretical calculations by Stebner et al. [25].

Figure 4: (a) Recoverable (solid symbols) and irrecoverable (open symbols) strains and (b) thermal hysteresis of $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ as a function of tensile (this study) and compressive stresses [22].

Thermal hysteresis of the $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ alloy under tension is illustrated in Figure 4b and compared to the thermal hysteresis under compression [22]. Temperature hysteresis depends primarily on the dissipation of energy through friction, defect generation during transformation, interaction between the interphases, lattice compatibility between phases, and strength of the material. It is clear from Figure 4b that thermal hysteresis decreased after aging in both tension and compression. All else being equal, this is probably due to precipitation hardening, which

reduces the amount of defect generation and plasticity that occurs during the transformation. Thermal hysteresis increased rapidly with applied stress in the as-extruded condition due to the lower strength of the alloy, where the applied stress was sufficient to generate defects during the phase transformation. The thermal hysteresis shows similar trends in tension and compression, which suggests that the main mechanism behind the hysteresis is mostly independent of stress-state.

It is well known that SMAs are anisotropic materials and that shape memory properties are highly orientation dependent in single crystal form and the behavior of polycrystalline SMAs can be controlled by texture formation [2, 25, 28, 29]. It has been reported in [13, 14, 19] that [111]-oriented NiTi alloys are characterized as soft under tension, since they exhibit large transformation strains and low critical transformation stress levels. Conversely, [001]-oriented NiTi is considered a hard orientation in tension since transformation strains are smaller and the stress to induce transformation is significantly higher under tension. However, [111] is hard and the [001] orientation is soft under compression. More specifically, it has been demonstrated that the theoretical maximum resolved shear stress factor (RSSF) is ~ 0.4 in tension and 0.27 in compression while recoverable strain is 5-8% in tension and 3% in compression along the [111] orientation of NiTi. Furthermore, the RSSF has its largest value along the [111] and [011] orientations under tension while the maximum RSSF is along the [001] orientation under compression for NiTi. Thus, it is clear that the mechanical behavior of NiTi single crystals highly depends on the loading direction.

Theoretical transformation strains for selected orientations of NiTiHf were previously calculated by energy minimization theory [30] in compression [26] and are compared to similar calculations in tension in Table 1. The theoretical strain is determined for the $\langle 011 \rangle$ type I twin mode, which is most frequently observed in $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ alloys [22, 26, 27]. In all three cases, the theoretical strains are greater under tension than compression. This is consistent with several different analytic polycrystal calculations performed by Stebner et al. [25], which also indicate that transformation strains, regardless of texture, are greater in tension than compression.

Table 1: Maximum theoretical strains of single crystalline and experimental strains of polycrystalline NiTiHf alloys in tension and compression

NiTiHf <011> type I twinning						
	Compression [22, 26]			Tension [this study]		
	RSSF	CVP strain	$\epsilon_{\text{experimental}}$	RSSF	CVP strain	$\epsilon_{\text{experimental}}$
[001]	0.14	0.97%		0.16	2.92%	
[011]	0.48	8.00%		0.50	9.95%	
[111]	0.33	5.25%		0.40	8.10%	
polycrystalline			3.5%			4.7%

In summary, the tension-compression asymmetry in NiTiHf can be attributed to the unidirectional nature of shear strain across the martensite habit planes and martensite ordering. It is known that during martensitic transformation, only one specific martensite variant group is selected that provides the largest possible strain along the loading direction. The martensite corresponding variant pair (CVPs) with the largest RSSF are activated and then oriented along the shear direction with transformation. If the same material loaded in the opposite direction, it is possible that the RSSF of those CVPs are not the largest ones along the transformation direction, hence, they are not energetically favored where other CVPs can be activated. Another possible contributing factor to the different transformation strains in tension and compression can be the change in the mobility of martensite variant interfaces. It has been reported that the martensite variant boundaries are twin related and can move easily as transformation proceeds under tension in NiTi [12, 21]. However, the junction between the twin related martensite variants is not mobile under compression, resulting in the generation of lattice defects during transformation.

Figure 5 shows the relationship between M_s and applied tensile and compressive [22] stresses in $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ alloys, which are extracted from stress-biased thermal cycling experiments. M_s linearly increases with stress, following the Clausius-Clapeyron (CC) relation where the slope of the stress- M_s temperature plots are proportionally related to the enthalpy of transformation and inversely correlated to transformation strain and equilibrium temperature. The difference in M_s values, particularly extrapolated back to zero stress, could be attributed to slight composition

variations of the alloys during processing, since the tensile data from this study and the compressive data from [22] are from two different heats of material.

The CC slope was 5.4 MPa/ °C in tension and 12.5 MPa/ °C in compression for the as-extruded sample. After 550 °C-3h aging, M_s shifted to higher temperature and the CC slope in compression decreased to 7.5 MPa/ °C while it increased to 8.5 MPa/ °C in tension. The change in CC slopes could simply be explained by the asymmetry of the transformation strain for tension and compression. The as-extruded material exhibited a maximum tensile strain of 5.5 % while the maximum compressive strain was 2.4%, resulting in higher CC slope in compression as compared to tension. In comparison to the as-extruded samples, the maximum transformation strain decreased under tensile loading, while it increased under compression stress, after aging at 550 °C. Consequently, the CC slope for the aged samples increased in tension and decreased in compression. After aging at 550 °C-3h the transformation strain and CC slopes under compression and tension, depicted in Figures 4 and 5, are similar. It should be noted that the recoverable strains and thermal hysteresis are also very close in tension and compression after 550 °C-3h aging. Thus, it would seem that the presence of the precipitate phase after 550 °C-3h aging of the $Ni_{50.3}Ti_{29.7}Hf_{20}$ seems to diminish anisotropy in the alloy.

Finally, it is worth comparing the work output, which is the product of the recoverable strain and applied stress. The maximum work output of 29 J cm^{-3} was achieved under tension in the as-

extruded condition and decreased to 27.1 and 18.5 J cm⁻³ after 500 °C-3h and 550 °C-3h aging, respectively. This is attributed to formation of non-transformable phases after aging, which decreased the transformation strain. However, aging increased the dimensional stability of the alloy and maybe a necessary tradeoff for applications involving multiple cycles. The maximum work output for Ni_{50.3}Ti_{29.7}Hf₂₀ under compression is 12 J cm⁻³, 11.5 J cm⁻³ and 16.5 J cm⁻³ for as-extruded, 500 °C-3h and 550 °C-3h conditions [22], respectively. The lower work output in compression is simply attributed to the lower transformation strain in compression.

Conclusions

The shape memory properties of Ni_{50.3}Ti_{29.7}Hf₂₀ can be tailored by aging. Transformation peak temperatures, determined by DSC, were stable after 500°C-3h aging and M_s reached a maximum value of 150 °C after 600 °C-3h aging. Polycrystalline Ni_{50.3}Ti_{29.7}Hf₂₀ demonstrated superior shape memory effect and superelasticity in tension compared to analogous behavior in compression determined from the literature. A recoverable strain of ~5% with the maximum work output of 29 J cm⁻³ was achieved by thermal cycling the as-extruded alloy in tension at 600 MPa. Aging at 550 °C for 3h resulted in material with perfect dimensional stability during stress-biased thermal cycling with a recoverable strain of 3.7% under 500 MPa and a work output of ~18.5 J cm⁻³. The CC slopes were on the order of 5-9 MPa/ °C under tensile stress and perfect superelastic behavior with 4% recoverable strain was demonstrated over a temperature window of 180–230 °C for both 500 °C-3h and 550 °C-3h aged conditions. The asymmetry in recoverable strain under tension and compression can be attributed to ordering of martensitic transformation, the unidirectional nature of habit plane and activation of different number of CVPs.

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