In Situ Neutron Diffraction Study of NiTi–21Pt High-Temperature Shape Memory Alloys

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Abstract In situ neutron diffraction was used to investigate the microstructural features of stoichiometric and Ti-rich NiTiPt high-temperature shape memory alloys with target compositions of Ni₂₉Ti₅₀Pt₂₁ and Ni_{28.5}Ti_{50.5}Pt₂₁ (in atomic percent), respectively. The alloys' isothermal and thermomechanical properties (i.e., moduli, thermal expansion, transformation strains, and dimensional stability) were correlated to the lattice strains, volume-averaged elastic moduli, and textures as determined by neutron diffraction. In addition, the unique aspects of this technique when applied to martensitic transformations in shape memory alloys are highlighted throughout the paper.

Keywords High-temperature shape memory alloy · NiTiPt · Neutron diffraction · Texture

Introduction

Increasing the application temperature of shape memory alloys (SMAs) is one of the main objectives in the development of solid-state actuators for aerospace, automotive, and other industrial applications. Over the years, numerous NiTi-based alloys have been investigated with macro-

additions of Zr, Hf, Pd, Pt, and Au, and have been shown to exhibit transformation temperatures in excess of 500 °C [1–3]. In particular, Pt proved to be an effective alloying addition, raising the martensitic transformation temperature as much or more substantially than any other ternary additions [1, 4]. NiTiPt alloys are also on a short list of SMAs with *demonstrated* actuator properties in environments approaching 300 °C [5].

A series of NiTiPt alloys, ranging from 5 to 50 at.% platinum, have been studied and several formulations were shown to exhibit high work output and good dimensional stability [5–7]. Stoichiometric and Ti-rich compositions [8], in general, tend to be less dimensionally stable, but produce large transformation strains, compared to Ni-rich compositions [9]. On the other hand, the Ni-rich alloys [10–13] can be microstructurally stabilized via heat treatments to precipitate out strengthening phases; however, a decrease in strain capability is usually observed. Nonetheless, high transformation temperatures can be achieved in these alloy formulations, and the choice of a specific alloy composition is based on the application requirements (e.g., one time actuator, multi-cycle actuator, large stroke, etc.).

Of the many alloying options, NiTiPt compositions containing 21 at.% platinum have been investigated on the Ni-rich side [9], in an attempt to maximize both transformation temperature and functional capability at high temperature. These alloys are of particular interest as they provide stable high-temperature actuation behavior (martensite start temperature >300 °C) and good work output (>10 J/cm³). In contrast, the Ti-rich version has not been examined in detail. As a result, the goal of this work is to extend the alloying range of NiTi–21Pt toward stoichiometric and Ti-rich compositions (note that Pt is substituted for Ni in this ternary alloy) and perform advanced

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thermomechanical property–structure analyses to complete the investigation of the effect of stoichiometry on functional behavior.

In order to provide quantitative correlations between the alloys' microstructure and macroscopic behavior, advanced experimental methods are advantageous over conventional methods such as thermomechanical testing followed by microscopy. One such method is in situ neutron diffraction where microstructures can be examined concurrently with applied mechanical and thermal loads. This is especially important in studying SMAs due to the thermoelastic nature of the phase transformation that cannot be fully stabilized as in conventional materials, but typically evolves with temperature and stress. Neutron diffraction can provide information pertinent to phase fractions, lattice plane-specific elastic moduli, thermal expansion tensors, deformation mechanisms, textures, and other features, which can be directly linked to the macroscopic behavior. Such data are also beneficial in validating models and predictions [14–16] that cannot be obtained by other means.

Thus, the objective of this work is to examine the thermomechanical behavior of two NiTi–21Pt compositions and relate the macroscopic behavior to the ensuing microstructure via in situ neutron diffraction. The effect of stoichiometry will be discussed in the macroscopic range (i.e., dimensional stability and work output) and in the microscopic range (i.e., martensite detwinning, elastic anisotropy, and texture evolution). The unique advantages and capabilities of neutron diffraction experiments will be highlighted throughout this work.

Experimental Methods

Material

Two NiTiPt alloys, with target compositions of Ni₂₉Ti₅₀Pt₂₁ and Ni_{28.5}Ti_{50.5}Pt₂₁ (in atomic percent), were produced by vacuum induction melting of high-purity starting elements (99.95 Ti, 99.995 Pt, 99.98 Ni) using a graphite crucible. The molten materials were cast into a 25.4-mm diameter cylindrical copper mold followed by homogenization in vacuum at 1050 °C for 72 h. The ingots were then extruded at a 7:1 reduction ratio at 900 °C (designated Ext. 71 and 72).

Bulk chemical composition of the extruded material was determined using inductively coupled plasma atomic emission spectroscopy (ICP-AES) for detecting metallic elements. The chemical analyses of the two alloys are listed in Table 1. Based on the measured analyses, the stoichiometric alloy may be slightly Ni + Pt-rich (Ti-lean).

Thermomechanical Testing

Cylindrical dog bone-shaped specimens with a 3.81-mm-diameter by 14-mm-long gage section and threaded grip ends were machined from the extruded rods. The samples were subjected to two stress-free thermal cycles between 50 and 500 °C, while being mounted in the test frame, to relieve any residual stresses resulting from machining and handling and to produce an initial self-accommodated martensite structure prior to testing. Thermomechanical testing was performed using an MTS servohydraulic test frame equipped with a FlexTest™ digital controller. Strains were measured using a high-temperature extensometer with 12.7 mm gage length and $-10/+20\%$ strain range. The specimens were inductively heated at a heating rate of 20 °C/min, and temperature measurements were made using type K thermocouples spot welded to the samples.

Constant-force thermal cycling experiments were conducted in series at stresses ranging from 0 to 600 MPa (series of increasing loads on the same sample). Each sample was thermomechanically cycled twice at each stress level between the lower and upper cycle temperatures of 50 and 500 °C, respectively. The specific work output was calculated from the amount of transformation strain generated on the second cycle multiplied by the applied stress.

In Situ Neutron Diffraction Measurements

Neutron diffraction experiments were performed using the pulsed spallation source at Los Alamos Neutron Science Center (LANSCE), Los Alamos National Laboratory. In situ experiments were carried out on the Spectrometer for Materials Research at Temperature and Stress (SMARTS) [17]. SMARTS utilizes a white neutron beam with a continuous wavelength spectrum from 0.5 to ~ 4 Å. The neutrons are scattered from the sample's gage volume (using 5 mm $\times$ 5 mm slits) and collected in two fixed detector banks positioned on either side of the incident beam at $\pm 90^\circ$. The test samples are mounted on a horizontal servohydraulic load frame with the loading axis oriented at 45° relative to the incident beam. Additional details of the experimental setup can be found elsewhere [18].

Isothermal tests were conducted in the martensite phase at room temperature up to 1 GPa. Loading was done in strain-controlled mode at a strain rate of 1×10^{-4} /s, where holding was initially controlled in load (up to 400 MPa), and then switched to strain holding for the remainder of the test (up to 1 GPa and unloading). Neutron data were collected in increments of 20 MPa from 0 to 200 MPa, and in increments of 100 MPa from 200 to 1000 MPa. Constant-force thermal cycling tests were performed at 0 MPa for

Table 1 Measured composition in atomic percent determined by inductively coupled plasma atomic emission spectroscopy (ICP-AES)

ICP Run	Sample ID	Sample weight (mg)	Extrusion #	Measured composition (at.%)		
				Ni	Ti	Pt
Run1	Ni ₂₉ Ti ₅₀ Pt ₂₁	253	71	29.00	49.62	21.38
Run2	Ni ₂₉ Ti ₅₀ Pt ₂₁	"	71	29.00	49.52	21.48
Run1	Ni _{28.5} Ti _{50.5} Pt ₂₁	301.3	72	28.38	50.24	21.38
Run2	Ni _{28.5} Ti _{50.5} Pt ₂₁	"	72	28.39	50.08	21.53

Each sample was run twice

two cycles and at 400 MPa for another two cycles. In order to produce good-quality neutron spectra, hold periods of 30 min were employed at each load or temperature of interest.

Diffraction spectra were analyzed using the Rietveld method implemented in the general structure analysis system (GSAS) code [19]. The data were analyzed to determine lattice strains and crystallographic texture as a function of load and/or temperature. Texture data are represented using raw spectra and inverse pole figures (IPFs) constructed from the individual parallel and perpendicular diffraction vectors using generic mapping tools and SMARTSware software [20]. The IPFs describe the distribution of specific macroscopic directions, e.g., the loading axis, in the coordinate systems of the individual crystallites comprising the polycrystalline sample and are reported in multiples of random distribution (MRD) plotted on the irreducible stereographs. A single peak fitting method was also used to compute (*hkl*) lattice strains from the individual reflections. The lattice strains were obtained from the change in interplanar spacing ($\Delta d_{hkl}/d_{hkl}$) where the initial d_{hkl} is taken as the interplanar *d*-spacing at 0 MPa at the respective temperature before deformation.

Results and Discussion

Macroscopic Behavior

The strain–temperature responses measured during ex situ constant-force thermal cycling are shown in Fig. 1. For clarity, only the second cycles are plotted at each stress level for each alloy composition. There are significant differences in the behavior between the two alloys particularly with regard to the generation of strains during cycling under load (both recoverable and irrecoverable strains). The recoverable strains, i.e., transformation strains, are measured as the strain recovery obtained on heating from the austenite start (A_s) to the austenite finish (A_f) temperature. The irrecoverable strains, i.e., residual strains, are measured as the final strain at the lower cycle temperature (in the martensite phase) minus the initial

strain before starting the cycle. For a schematic representation of how these values are measured, a detailed diagram can be found in Ref. [21].

Since these tests were performed in series, the second cycles plotted in Fig. 1 were not shifted and represent the absolute strain evolution. It is evident that the Ti-rich Ni_{28.5}Ti_{50.5}Pt₂₁ alloy produced higher transformation strains, resulting in a slightly higher work output, when compared to the stoichiometric Ni₂₉Ti₅₀Pt₂₁ alloy. It is also seen that the strain evolution and the residual strains, which are indicative of dimensional instability, are also higher on the Ti-rich version, making it a less dimensionally stable alloy. A summary of transformation strains, residual strains, and work outputs are shown in Fig. 2. The stoichiometric Ni₂₉Ti₅₀Pt₂₁ alloy follows an increasing trend in transformation strains up to the tested 600 MPa stress, whereas the Ti-rich Ni_{28.5}Ti_{50.5}Pt₂₁ strain peaked at 300 MPa. The trend in residual strains is similar for both alloys, where the magnitudes increase with the increasing applied stress. It should be noted that these alloys exhibited significant work outputs, greater than 10 J/cm³ at transformation temperatures near 300 °C (Fig. 2a).

The transformation temperatures as a function of applied stress were determined from the constant-force thermal cycling data using the tangent intercept method as outlined in ASTM Standard F2082–06 [22]. The transformation temperatures shown in Fig. 3 exhibited near-linear stress dependence, with A_f temperature–stress slopes of ~ 0.18 and 0.28 °C/MPa for the stoichiometric and Ti-rich compositions, respectively. Thermal hysteresis, measured as ($A_f - M_s$), increased with the increasing stress from 9 °C (at 100 MPa) to 32 °C (at 600 MPa) for the Ni₂₉Ti₅₀Pt₂₁ alloy, and from 19 °C (at 100 MPa) to 36 °C (at 400 MPa) for the Ni_{28.5}Ti_{50.5}Pt₂₁ alloy.

Microscopic Response

Phase Identification and Initial Texture

Neutron diffraction spectra acquired in the martensite phase at room temperature and in the austenite phase at 450 °C in the nominally unloaded condition (~ 2 MPa)

Fig. 1 Macroscopic strain–temperature responses of $\text{Ni}_{29}\text{Ti}_{50}\text{Pt}_{21}$ and $\text{Ni}_{28.5}\text{Ti}_{50.5}\text{Pt}_{21}$ (at.%) in tension

Fig. 2 **a** Transformation and residual strain behavior of $\text{Ni}_{29}\text{Ti}_{50}\text{Pt}_{21}$ and $\text{Ni}_{28.5}\text{Ti}_{50.5}\text{Pt}_{21}$ (at.%). **b** Specific work output as a function of applied stress

are shown in Fig. 4. The spectra were refined using the Rietveld method where a best fit was obtained using *Pmcm* orthorhombic space group for B19 martensite and *Pm* $\bar{3}m$ cubic space group for B2 austenite. The best fit lattice parameters for both alloys at the two temperatures are listed in Table 2. It is noted that the uncertainty in the lattice parameter measurements ignores systematic uncertainty such as specimen misalignment and center of mass and only represents statistical estimations based on the standard deviation from the lattice parameter fits. Moreover, being associated with this neutron technique, the absolute lattice parameters determined can be altered by neutron penetration, and thus the average lattice parameters from multiple refined spectra are reported in Table 1.

Both alloys in the as-extruded condition showed no presence of secondary phases, such as P-phase precipitates, within the instrument resolution, as expected in

these near-stoichiometric and Ni-lean alloys. Since the alloys were hot extruded with a dynamically recrystallized microstructure, the starting texture was close to random with a texture index of ~ 1.2 , where 1 corresponds to a random polycrystalline sample. Comparing Figs. 4a and b, it is evident that the martensitic diffraction peaks are broader in the stoichiometric $\text{Ni}_{29}\text{Ti}_{50}\text{Pt}_{21}$ alloy implying significantly more internal stresses due to size or strain broadening possibly from pre-precipitation effects or clustering of atoms. While the target composition was stoichiometric, e.g., 1:1 Ni + Pt:Ti, the actual chemical analyses indicate that the alloy is slightly Ti-lean, which would eventually lead to the precipitation of P phase or precursor precipitate phases [11, 12].

In addition to identifying phases and initial textures, the essentially stress-free lattice parameters are very useful in predicting the austenite-to-martensite transformation strain

Fig. 3 Transformation temperatures determined from the second cycle during constant-force thermal cycling

Fig. 4 Neutron diffraction patterns for $\text{Ni}_{29}\text{Ti}_{50}\text{Pt}_{21}$ and $\text{Ni}_{28.5}\text{Ti}_{50.5}\text{Pt}_{21}$ (at.%) with diffracting lattice planes parallel to the loading axis with no externally applied load. **a, b** Orthorhombic martensite at room temperature and **c, d** cubic austenite at 450 °C. The measured data are indicated by cross marks in red and the calculated profile (Rietveld

refinement) is indicated by the solid green line. The tick marks below the profile pattern indicate the reflections used in the refinement. The lower magenta curve indicates the difference between measurement and refinement (Color figure online)

and martensite variant conversion strains as outlined in [23, 24]. They can also be used to calculate the austenite/martensite interfaces and single-crystal recoverable strains using the crystallographic theory of martensite as outlined in [25–27]. Undeniably, lattice parameters can be obtained via conventional X-ray diffractometers, but when

additional information is required as related to depth of penetration (bulk versus surface), greater sampling volume, d -spacing range, and in situ experimentation of bulk material, neutron diffraction provides unparalleled advantages (to some extent, synchrotron sources can also be used for this purpose).

Table 2 Lattice parameters of Ni₂₉Ti₅₀Pt₂₁ and Ni_{28.5}Ti_{50.5}Pt₂₁ (at.%) in the martensite (at 35 °C) and austenite (at 450 °C) conditions

Alloy composition (at.%)	Crystal structure	Temperature (°C)	Lattice parameters (Å)				
			<i>a</i>	<i>b</i>	<i>c</i>	$\alpha = \beta = \gamma$	
Ni ₂₉ Ti ₅₀ Pt ₂₁	B19: orthorhombic	35	2.774	4.672	4.447	90	
	B2: cubic	450	3.087			90	
Ni _{28.5} Ti _{50.5} Pt ₂₁	B19: orthorhombic	35	2.751	4.703	4.451	90	
	B2: cubic	450	3.093			90	

Isothermal Deformation and Plane-Specific Young's Moduli

The macroscopic stress–strain responses obtained during uniaxial tensile loading at room temperature are shown in Fig. 5. These curves were generated during the in situ neutron diffraction experiments (in strain control), and the stress relaxations are due to the ~30-min hold periods required for diffraction data acquisition. Both alloys exhibited similar initial elastic response with a Young's modulus of 61 GPa, though it must be acknowledged that inelastic deformation may occur concomitantly at low stresses, causing deflation of the measured value, as observed in similar alloys [28]. At higher loads, the onset of non-linearity is apparent at stresses above 200 MPa, where the stress “plateaus” are indicative of inelastic deformation, with the primary mechanism being variant reorientation and detwinning along with contributions from plasticity [29, 30]. In this case, the stress plateaus are not flat, as observed in binary NiTi [31], but have a relatively steep hardening rate, indicating increased resistance to detwinning and possibly a greater contribution from plasticity or other irrecoverable processes with increasing

Fig. 5 Room-temperature macroscopic stress–strain responses of Ni₂₉Ti₅₀Pt₂₁ and Ni_{28.5}Ti_{50.5}Pt₂₁ (at.%) in tension

strain. The Ti-rich alloy displayed a longer plateau and slightly lower hardening rate providing more detwinning strain at a comparable stress level, which is consistent with the larger transformation strains observed in Fig. 2.

The corresponding lattice strains as a function of applied stress, determined from the single peak fitting method, are

Table 3 Martensite elastic moduli in directions perpendicular to *hkl* planes as determined by in situ neutron diffraction measurements and the correlation (*R*) of the fit used to determine the moduli. For comparison, the macroscopic moduli are also listed

<i>hkl</i>	d-spacing (Å)	Ni ₂₉ Ti ₅₀ Pt ₂₁		Ni _{28.5} Ti _{50.5} Pt ₂₁	
		<i>E</i> _{hkl} (GPa)	<i>R</i>	<i>E</i> _{hkl} (GPa)	<i>R</i>
Macro	–	61	–	61	–
(011)	3.199	206.3	0.981	(max) 257.8	0.985
(002)	2.197	115.6	0.953	138.7	0.994
(111)	2.094	114.6	0.985	99.2	0.997
(120)	1.785	(min) 57.8	0.952	(min) 55.1	0.988
(102)	1.721	171.1	0.955	138.3	0.993
(121)	1.654	67.1	0.977	75.3	0.995
(030)	1.556	(max) 227.7	0.976	173	0.998
(013)	1.398	59.5	0.961	132.3	0.988
(122)	1.385	178.9	0.933	88.3	0.996
(032)	1.270	112.9	0.89	218.6	0.886

shown in Fig. 6. The data presented here are from diffracting planes perpendicular to the loading direction. The measured lattice strains represent the average strain in all martensite variants with orientations that satisfy the Bragg condition in the direction normal to the hkl plane. From Fig. 6, lattice strains are partitioned between different lattice directions highlighting the stiffer and more compliant directions. Both alloys exhibited similar partitioning and to some extent the deviation from linearity also occurred at stresses above 200 MPa. On initial loading (the elastic region), neutron data were collected at more than ten points for better statistics in order to determine the volume-averaged elastic moduli (E_{hkl}) as shown in Table 3. The fits are shown in Fig. 6 where the data were linearly fitted up to 200 MPa where the onset of the stress plateau was macroscopically observed.

There is a large anisotropy in the elastic response of both alloys, but slightly higher in the Ti-rich alloy. This suggests that the mismatch stresses due to higher anisotropy may favor additional load transfer and detwinning, resulting in higher transformation strains as shown previously. In addition to determining the volume-averaged elastic moduli, this kind of data is particularly valuable in determining the elastic constants from a single sample, as was outlined in previous studies [29, 30]. Moreover, these data are also used in the validation of first principle calculations of elastic constants such as those determined by Hatcher et al. [32] and Wagner and Windl [33].

Thermal Strains

Direction-specific thermal strains were determined only for the $\text{Ni}_{29}\text{Ti}_{50}\text{Pt}_{21}$ alloy. This was accomplished by thermal cycling in the absence of an external applied stress and measuring the lattice spacing at incremental temperatures.

The lattice strains of the stoichiometric $\text{Ni}_{29}\text{Ti}_{50}\text{Pt}_{21}$ alloy were measured along directions normal to various B19 and B2 planes (Fig. 7). While a single-valued martensitic coefficient of thermal expansion (CTE) is measured macroscopically ($12.3 \times 10^{-6}/^\circ\text{C}$), the atomic-level thermal strains exhibited large anisotropy encompassing expansion (e.g., $(111)_{\text{B19}}$, $(102)_{\text{B19}}$, and $(120)_{\text{B19}}$), contraction (e.g., $(011)_{\text{B19}}$ and $(030)_{\text{B19}}$), and near-zero thermal expansion (e.g., $(013)_{\text{B19}}$). This anisotropy arises from the crystal symmetry (or lack thereof), and when using linear fits through the initial data, the lattice-specific CTEs were determined to range from +41 to $-12 (\times 10^{-6}/^\circ\text{C})$ as shown in Eq. 1:

$$A = \begin{pmatrix} \alpha_{011} \\ \alpha_{111} \\ \alpha_{120} \\ \alpha_{102} \\ \alpha_{121} \\ \alpha_{030} \\ \alpha_{013} \end{pmatrix} = \begin{pmatrix} -9.057 \\ 41.901 \\ 15.417 \\ 27.874 \\ 16.14 \\ -12.87 \\ 3.6993 \end{pmatrix} \cdot \frac{10^{-6}}{^\circ\text{C}}. \quad (1)$$

While it is possible that the internal stress distribution between grains, dictated by the elastic anisotropy, influences these measurements, the fact that the measurement was made with a polychromatic neutron beam, wherein diffracting grains in the gage volume that meet the Bragg condition have different neighbors, makes this unlikely. In addition, the result is consistent with earlier work by Qiu et al. on NiTi [34], and more recent work by Monroe et al. [35] suggesting that CTE anisotropy, including negative values, is actually a common attribute in materials that undergo reversible martensitic transformations and that the negative and positive thermal expansion directions can be predicted.

The B2 lattice strains (Fig. 7b), on the other hand, exhibited little to no anisotropy along the $\langle 100 \rangle$, $\langle 110 \rangle$,

Fig. 6 Lattice strains corresponding to applied stress levels indicated in Fig. 5 of $\text{Ni}_{29}\text{Ti}_{50}\text{Pt}_{21}$ and $\text{Ni}_{28.5}\text{Ti}_{50.5}\text{Pt}_{21}$ (at.%) alloys associated with several B19 reflections perpendicular to the loading direction

Fig. 7 Lattice strain evolution as a function of temperature in **a** B19-phase $\text{Ni}_{28.5}\text{Ti}_{50.5}\text{Pt}_{21}$ (at.%) during heating and **b** B2-phase $\text{Ni}_{28.5}\text{Ti}_{50.5}\text{Pt}_{21}$ during cooling

Fig. 8 Inverse pole figures (IPFs) for martensitic $\text{Ni}_{29}\text{Ti}_{50}\text{Pt}_{21}$ and $\text{Ni}_{28.5}\text{Ti}_{50.5}\text{Pt}_{21}$ (at.%) from diffracting planes perpendicular to the loading direction at **a** 0 MPa and **b** after two thermomechanical

cycles at 200 MPa. The maximum and minimum MRD values are recorded within each IPF

$\langle 111 \rangle$, and $\langle 210 \rangle$ directions as would be expected for a cubic lattice structure. In this case, the macroscopic austenite CTE (from extensometry) was measured to be $14.5 \times 10^{-6}/^{\circ}\text{C}$, while the average lattice CTE was found to be $17.5 \times 10^{-6}/^{\circ}\text{C}$. In principle, these data are very useful in determining the thermal expansion tensors as was demonstrated in previous studies [34, 35]. Lattice-specific thermal strains can also be used to produce alloys with

specific orientations or texture with tailored CTE as shown by Monroe et al. [35].

As demonstrated above, the neutron diffraction technique with a polychromatic neutron beam is particularly suited to the study and determination of thermal strains, especially in non-cubic structures. In this case, all lattice directions are sampled at the same time from a single polycrystalline sample. This is much more efficient than

the alternative of making multiple single crystals (if achievable) and probing each one individually.

Constant-Force Thermal Cycling

Macroscopic actuator properties are often obtained using constant-force thermal cycling experiments as shown in Fig. 1. By incorporating in situ neutron diffraction, it is possible to identify the preferred martensite variants/orientation responsible for the generated strains by examining the texture evolution with cycling. The corresponding texture, represented by the inverse pole figures (IPFs) for both alloys, is shown in Fig. 8, which uses multiples of random distribution (MRD) scale. Only the IPFs from the lattice planes perpendicular to the loading direction, which represent the axial direction/strains, are shown, while the lattice planes parallel to the loading direction represent the radial direction/strains due to Poisson's effect. Starting from a self-accommodated texture (Fig. 8a), both alloys select the (010)_{B19} preferred orientation on the first cycle, similar to previously reported work on NiTiPd [36], NiTi [30], and NiTiHf [18]. The pole densities increase from a magnitude of 2.5 at 0 MPa to ~6.9 for the stoichiometric alloy and 11.9 for the Ti-rich alloy at 200 MPa. The difference in magnitude between the two alloys suggests additional reorientation, which manifests as higher macroscopic transformation strains in the Ti-rich composition.

It is also noted that the pole intensity evolves with cycling, increasing from 6.98 to 7.08 and from 11.93 to 12.80 for the stoichiometric and the Ti-rich alloys over two thermomechanical cycles, respectively. While the intensity changes in the stoichiometric alloy are insignificant (within the instrument noise resolution), the increase is much greater for the Ti-rich alloy from cycle 1 to cycle 2, which is an indication of dimensional instability, as the texture continues to evolve with cycling. The IPFs are advantageous in understanding the bulk textures, preferred orientations, volume fractions of individual orientations, and other features that cannot be obtained using conventional methods.

Concluding remarks

Two NiTiPt high-temperature shape memory alloys with target compositions of Ni₂₉Ti₅₀Pt₂₁ and Ni_{28.5}Ti_{50.5}Pt₂₁ (in atomic percent) were investigated using in situ neutron diffraction. Actuation properties including transformation strains, specific work output, and residual strains were evaluated along with phase-specific microstructural features. It was found that the Ti-rich alloy exhibited higher transformation strains and also higher residual strains when

compared to the stoichiometric alloy. Neutron diffraction revealed higher propensity for reorientation and detwinning in the Ti-rich alloy and the texture evolved more with cycling, which support the macroscopic observations. Phase- and lattice-specific elastic moduli and coefficient of thermal expansion were determined, and it was found that the B19 martensite exhibited large anisotropy in both strains (elastic and thermal), while the B2 austenite phase was quantitatively isotropic. Moreover, some of the B19 martensite thermal strains exhibited negative thermal expansion (contraction).

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