Neutron diffraction studies and multivariant simulations of shape memory alloys: Empirical texture development–mechanical response relations of martensitic nickel–titanium

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Abstract

Mechanical responses and texture developments were observed in situ during the creation of multiaxial stress states in polycrystalline NiTi parallelepiped specimens, achieved via sequential compression along unique principal axes. For all of the compression stages, regardless of initial texture, the two major texture components behaved similarly: (1 0 0) poles aligned with, while (0 1 1) poles oriented perpendicular to, the loading direction. The effective critical resolved shear stress needed to induce significant reorientation, however, was reduced through prior loading. In the macroscopic responses, prior transverse direction loading resulted in widening of the reorientation plateau, a reduction of the effective Young’s modulus, and substantial alteration of effective Poisson’s ratios during axial direction straining. Additionally, these empirical results are presented in a manner conducive to the verification of shape memory alloy micromechanics and continuum mechanics constitutive models.

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1. Introduction

Shape memory alloys (SMAs), one of a class of smart or active materials, can recover large strains when heated above their transformation temperatures (shape memory effect) or upon load removal (superelasticity) due to reversible solid-state phase transformation and crystallographic reorientation. In addition to shape memory behaviors, other characteristics of the phase transformation, such as hysteresis, latent heat release and change in electrical resistance, enable SMAs to have a variety of applications in numerous industries, including medical, aerospace, defense, consumer technology, automotive and telecommunications. Among many alloy systems exhibiting shape memory behaviors, nickel–titanium (NiTi, Nitinol) is the most prevalent in commercial applications due to its large recoverable strain, superior fatigue performance and biocompatibility.

Nitinol is commercially available in various forms: bar, tube, rod, wire, sheet, ribbon and thin film. In all of these conditions, even in one- and two-dimensional forms such as wire or thin film, crystallographic texture development plays a very important role and can lead to drastic changes in the alloy’s thermomechanical responses. Through calculation, it has been shown that recoverable deflection sensitivity to film texture differs between major alloy systems \cite{1}, agreeing with the work in Refs. \cite{2–4}. Thin NiTi plates have also been fabricated via cold rolling, and dependence of deformation or recrystallization texture on annealing...
temperature and rolling reduction were found in addition to directional dependence of transformation [3]. Related studies on rolled sheets have shown similar results [5–9]. More recently, digital imaging correlation has been used to examine these relationships [10]. In addition to examining films, plates and sheets, relationships between microstructure and the tensile responses of NiTi wires have been reported [11], as well as texture-dependent effects in other NiTi forms: tension–compression asymmetry in both single crystals and polycrystalline rods [12], cyclic behaviors of rectangular single-crystal specimens [13], and fatigue properties of hot-rolled and cold-drawn rods [14].

Among the previous work, the experimental research focused mostly on measuring austenite texture at various points in the processing paths while observing transformation strain, and in several of the studies, pre-load martensite texture and subsequent stress–strain responses were also characterized [11–14]. Utilizing Spectrometer for Materials Research at Temperature and Stress (SMARTS) [15] at Los Alamos Neutron Science Center (LANSCE), the evolution of internal elastic strains, crystallographic orientation, volume fraction, coefficient of thermal expansion, and strain during in-situ thermomechanical loading of bulk NiTi systems has been monitored [16–20]. However, due to the configuration of SMARTS, the study of the texture evolution was limited to two specimen orientations (i.e. diffraction vectors along perpendicular and parallel sample directions), hence complete orientation distribution functions (ODFs) could not be obtained from the collected data.

In the current study, in addition to recording pole peak intensity evolutions along an axial and transverse specimen direction in situ using SMARTS, complete ODFs of the 54.8 wt.% NiTi specimens were measured ex situ via neutron diffraction using High Pressure Preferred Orientation Diffractometer (HIPPO) [21] at critical points of the loading sequences. Broadly speaking, these experiments were designed to meet four primary objectives:

1. to explore the effects of loading along either a transverse or axial direction of a parallelepiped specimen;
2. to allow for comparison of behaviors of specimens with unique initial textures subjected to the same loading condition;
3. to observe the creation of multiaxial stress states such that the macro- and microscale behaviors associated with such a process may be correlated;
4. to collect the data in such a manner that their presentation not only furthers fundamental understanding of the material, but also facilitates verification of SMA constitutive models.

Discussion of the data as they pertain to these objectives follows in this paper, exploring the empirically observed ties between crystallographic orientations and mechanical behaviors. In concurrent work, the final objective is realized as these data are used to verify the simultaneous mechanical response and texture development predictions of a mainstream, multivariate, micromechanics model for the first time [22], namely the simplified multivariate model [23]. Previously, to the best of the authors’ knowledge, only virgin austenite texture measurements had been empirically and numerically compared for the purpose of model calibration. To make these data applicable not only to micromechanical models, but also to phenomenological constitutive models, the in situ deformation processes were repeated ex situ such that the crystallographic data are complemented by triaxial macroscopic flow curves and effective Poisson’s ratios, results that would not have been possible via neutron diffraction measurements alone.

2. Experimental details

2.1. Specimen preparation

A cast cylindrical ingot of 54.8 wt.% NiTi was purchased from Special Metals (now SAES Smart Materials, New Hartford, NY). Parallelepiped specimens 8 mm × 8 mm × 20 mm were electrical discharge machined such that their axial direction (1-direction as defined in Fig. 1) coincided with the cylindrical axis of the ingot. Specimens in this state, prior to any loads being applied, are hence referred to as “virgin” specimens. The stress-free transformation temperatures of this composition have been reported as $M_f \approx 316$ K and $A_f \approx 363$ K [24], though they are not critical in the ensuing discussion except to note that the material was fully martensitic at room temperature.

2.2. Neutron diffraction measurements

In situ neutron diffraction measurements were performed using the SMARTS instrument at the Manuel Lujan Jr. Neutron Scattering Center of Los Alamos National Laboratories. SMARTS has been discussed elsewhere [15] and only a brief description will be given here. Fig. 2 shows a schematic of the specimen and the diffraction configuration with the diffraction plane in the plane of the paper (in-plane or top view). SMARTS accepts a pulsed white beam of neutrons generated through spallation reactions in a tungsten target and moderated by a water moderator at 283 K. The incident neutron beam impinges on a specimen and is scattered in all directions. Two detector banks consisting of 196 He-filled tubes are
The compression experiments were performed under load control using a custom-built horizontal load frame oriented at 45° relative to the incident beam. The detectors on either side of the specimen (Fig. 2) recorded data with diffraction vectors parallel ($Q_{\parallel}$, −90° bank) and perpendicular ($Q_{\perp}$, +90° bank) to the applied load. The entire diffraction pattern (effective range 0.5–3.5 Å) was recorded simultaneously in each detector.

Three specimens were loaded at room temperature (~298 K) in the beam’s path. The macroscopic strain was determined concurrently with the neutron diffraction measurements using an extensometer that spanned the irradiated region. Diffraction patterns were recorded for roughly 40 min at incrementally increasing loads. The highest instantaneous strain rate was about 1.5 × 10^{-5} s^{-1} and the average strain rate was approximately 10^{-6} s^{-1}, accounting for the hold time. Loading conditions applied to each specimen are shown in Table 1 (Specimens 1–3).

The response of the crystallites whose poles were parallel to the applied load were recorded in the $Q_{\parallel}$ bank (−90°). Due to the symmetry of the initial crystallographic texture about the cylinder axis (1-direction, see Section 3.1), the response of Specimen 1’s lattice in the transverse directions are assumed to be equivalent and recorded in the $Q_{\perp}$ bank (90°). Specimen 1 was loaded in the 1-direction from the virgin state, while Specimens 2 and 3 were compressed from the virgin state in the 2-direction ex situ prior to in situ deformation in the 1-direction. The transverse direction pre-straining process broke the initial axial fiber symmetry of the texture, hence the lattice response of these specimens in the 2- and 3-directions can no longer be considered equivalent during 1-direction compression. Thus, Specimens 2 and 3 were deformed in situ with the 2- and 3-direction in the diffraction plane (the plane comprising of $Q_{\parallel}$ and $Q_{\perp}$), respectively, so that both transverse directions were monitored independently. Pole peak intensity evolutions were measured in situ in SMARTS. To more completely capture texture development, ODFs were measured ex situ in HIPPO after the completion of each deformation stage. Because of the necessary aspect ratio of the specimen geometry and the configuration of SMARTS, in situ measurements during pre-straining in the 2- and 3-directions were not possible.

### 2.3. Neutron data analysis

Several single peaks (hkl) within each diffraction pattern collected as a function of strain on SMARTS were analyzed using the RAWPLOT subroutine of the General Structure Analysis Software (GSAS) [25]. Within the context of this paper, we are concerned with the development of the integrated intensity of each peak and its relation to the evolution of the crystallographic texture with deformation.

Determination of the crystallographic texture from diffraction data collected on HIPPO using the spherical harmonics expansion within GSAS is now well established [21]. However, it should be noted that neither HIPPO nor the analysis technique (spherical harmonics expansion) are well suited to the sharp textures observed in these samples and the pole figures should be considered as qualitative representations of the actual specimen crystallography.

### 2.4. Ex situ measurement of principal axis strains

In situ deformation precludes the use of strain gages to measure the transverse strains, as the lead wires would

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**Table 1**

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Pre-load path</th>
<th>In situ loading path</th>
<th>Ex situ loading path</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Direction</td>
<td>$\varepsilon_{\text{unload}}$ (%)</td>
<td>Direction $\varepsilon_{\text{max}}$ (%) $\varepsilon_{\text{unload}}$ (%)</td>
</tr>
<tr>
<td>1</td>
<td>2</td>
<td>−4.3</td>
<td>1</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>−3.0</td>
<td>1</td>
</tr>
<tr>
<td>3</td>
<td>2</td>
<td>−3.3</td>
<td>1</td>
</tr>
<tr>
<td>4</td>
<td>2</td>
<td>−3.3</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>2</td>
<td>−3.3</td>
<td></td>
</tr>
</tbody>
</table>

* The strain gage in the loading direction slipped relative to the specimen surface prior to reaching the maximum applied load.
cause contamination peaks. Thus, to monitor the triaxial mechanical responses and calculate the effective Poisson’s ratios, which are essential components of SMA constitutive models, principal axis strains were simultaneously recorded while the loadings were repeated ex situ. The loading of Specimen 4 mimicked that of Specimen 1; a virgin specimen was compressed in the 1-direction. Specimen 5 compressions repeated the same paths as had been applied to Specimens 2 and 3; virgin specimens were compressed in the transverse direction (2-direction) and subsequently compressed in the axial direction (Table 1).

3. Results and discussion

In the ensuing presentations, pole figures for only one of the two pre-textured specimens (Specimen 2) are shown and discussed, as the texture developments observed in Specimen 3 were very similar and the trends and major deformation modes identical [26].

3.1. Virgin austenite and martensite texture

The pre-load lattice parameters of the martensite phase as determined from the Rietveld refinement are $a = 2.908$ Å, $b = 4.677$ Å, $c = 4.126$ Å, and $\gamma = 97.95^\circ$ with the $P 1 1 21/m$ space group. Fig. 3c shows the observed pre-load $(1 \ 0 \ 0)$, $(0 \ 1 \ 0)$, $(0 \ 0 \ 1)$ and $(0 \ 1 \ 1)$ pole figures representing the initial texture of the material. Strong pole alignment is indicated by higher peak multiples of random distribution (MRD). Two major texture components are apparent, namely $(1 \ 0 \ 0)$ and $(0 \ 1 \ 1)$ fibers aligned with the ingot axis (specimen 1-direction). As seen through the
coordinate system shown in Fig. 3a, the (1 0 0) fiber is associated with the (0 1 0), (0 0 1) and (0 1 1) density on the rim of these pole figures. Similarly, the (0 1 1) fiber is associated with (1 0 0) density on the rim and (0 1 0) and (0 0 1) density spread in a continuous ring at \( \Theta \approx 45^\circ \), using Kocks Euler angle designations [27] (Fig. 3b). The relationships of these texture components to the known correspondence variants of NiTi are given in Ref. [22], though it should be noted for the subsequent discussion that (0 1 1) and (0, -1, 1) poles both contribute to the (0 1 1) fiber. Fiber symmetry is observed for each of these components in the virgin material. These textures are consistent with temperature-induced transformation products originating from the virgin material. These textures are consistent with temperature-induced transformation products originating from the virgin material. These textures are consistent with temperature-induced transformation products originating from the virgin material. These textures are consistent with temperature-induced transformation products originating from the virgin material. These textures are consistent with temperature-induced transformation products originating from the virgin material.

3.2. Development of texture during deformation

3.2.1. Axial-direction compression of a virgin specimen

The texture observed in Specimen 1 after 1-direction compression such that \( \varepsilon_{\text{load}}^{1}\text{-direction} = -3.5\% \) is shown in Fig. 3d. Comparison with the virgin specimen pole figures (Fig. 3a) highlights the development of the crystallographic texture. During compression, the (1 0 0) fiber parallel to the straining direction strengthens considerably, with a concomitant decrease in the (0 1 1) fiber. Correspondingly, the (1 0 0) pole density on the rim of the pole figure and (0 1 0) and (0 0 1) pole density at \( \Theta \approx 45^\circ \) also decrease considerably after this deformation.

Fig. 4 shows the development of the (1 0 0) and (0 1 1) diffraction peak intensities recorded in situ in both the \( Q_{\parallel} \) (1-direction) and \( Q_{\perp} \) (2-direction) detectors during this straining process. The single peak diffraction intensities correspond to the integrated pole densities over the acceptance area of the detectors. These (1 0 0) and (0 1 1) diffraction peaks were chosen to highlight the crystallographic response of the material both because they are intense and well resolved, and because they represent the two primary texture components of the virgin material. The intensities are normalized to an initial value of 1 for ease of comparison. The development of the intensities is consistent with the change in orientations measured ex situ and also those previously observed of polycrystalline, thermally induced, NiTi martensite [16]. That is, (1 0 0) poles originally aligned in transverse directions reorient to align with the loading (1-) direction, signified by the increase to \( \sim 1.75 \) in the 1-direction normalized intensity and depletion to nearly 0 in the 2-direction. Concurrently, (0 1 1) pole population depletes in the loading direction to nearly 0 and remains relatively unaffected in the 2-direction.

By examining a stereographic projection along the [0 1 1] direction relative to the diffraction beam and applied load orientations, Dunand et al. showed that these reorientation processes are well described by (1, 1, 1) Type I twinning, and furthermore cannot be described by (0.7206, 1, 1) Type II twinning [16]. They also calculated the Schmid factor \( F = |\cos(\alpha)\cos(\beta)| = 0.21 \) for this twinning mode [16]. Having verified this result, here it is employed, along with the observation that substantial variant reorientation initiates when the compressive load is around 250 MPa (Fig. 4), to calculate the effective critical resolved shear stress (CRSS) needed for major reorientation to begin: \( \text{CRSS}_{\text{effective}} = F \times \sigma_{\text{reorient}} = 52.5 \text{ MPa} \). The reorientation stress was measured by observing the transition from constant pole populations to, within the deformation range of this measurement (0-5% strain), linearly increasing and decreasing populations. It has been shown for monoclinic martensite that these linear relations are indicative of the inelastic deformation being dominated by variant reorientation and not slip processes [30].

3.2.2. Transverse-direction compression of virgin specimens

Fig. 3e shows the observed texture of Specimen 2 after transverse-direction compression such that \( \varepsilon_{\text{load}}^{2}\text{-direction} = -4.3\% \) after 2-direction compression. In contrast to the texture observed after 1-direction straining of a virgin specimen (Fig. 3d), the (1 0 0) pole density is reduced in the 1-direction and almost completely depleted in the 3-direction, with a concomitant increase in the 2-direction. The population of (0 1 1) poles is now concentrated in the unloaded directions (1 and 3) and nearly void in the load direction (2). While these textures are different from that observed after 1-direction loading (Fig. 3d), they are similar in that the (1 0 0) poles have aligned themselves with the compression direction, while the propensity of (0 1 1) poles to align perpendicular to the load is again evident. Thus, the dominance of (1,1,1) Type I twinning during compressive loading is shown to be independent of the chosen compression axis (i.e. transverse to or aligned with the parallelepiped axis).
Recall that in the virgin specimen (Fig. 3c), the densities of the (0 1 0), (0 0 1) and (0 1 1) poles of the (1 0 0) fiber were randomly distributed perpendicular to the 1-direction. It is seen in these textures (Fig. 3e) that after transverse-direction compression, (0 1 0) and (0 1 1) poles have predominantly reoriented from the 2-direction to the 3-direction, while the band of (0 0 1) pole density on the rim has reduced almost uniformly. The different response of the (0 0 1) poles seems contradictory and may be an indication of the limitations of the measurement and analysis techniques. It is clear, however, that the (0 1 1) fiber rotates such that the (1 0 0) poles in the 3-direction reorient to align with the compression, while the (0 1 0) and (0 0 1) poles initially positioned at $\Theta = 45^\circ$ and, $\Psi = 90^\circ$ or $270^\circ$ circumvolve to $\Theta = 45^\circ$, $\Psi = 0^\circ$ or $180^\circ$. This reorientation is uniquely accomplished by a $180^\circ$ rotation of the (0 1 1) poles about the (1, 1, 1) plane normal, or a reflection in the (1, 1, 1) plane (again Type I twinning). Thus, (0 1 1) pole density at the center of the pole figure is relatively unaffected in both figures.

3.2.3. Axial-direction compression of transversely pre-loaded specimens

Fig. 3f shows the observed texture at $\varepsilon_{\text{max}}^{\text{unload}} = -5.3\%$ after 1-direction compression following the aforementioned 2-direction compression. The strong concentration of (1 0 0) pole density in the 1-direction after these deformations is a manifestation of the realignment of (1 0 0) poles with the specimen axial-direction. Indeed, this fiber is stronger than observed in the virgin specimen and of the same order of magnitude as was observed after 1-direction compression of the virgin specimen (Fig. 3d). A trace amount of (0 1 1) pole density in the 1-direction and residual lobes of (0 1 0) and (0 0 1) pole density at $\theta = 45^\circ$, $\Psi = 0^\circ$ and $180^\circ$, remnants of the transverse-direction pre-straining, indicate that the reorientation is not complete after this deformation process.

The developments of the (1 0 0) and (0 1 1) peak intensities during straining in the 1-direction after transverse-direction straining are shown in Fig. 5. Once again, the initial diffracted peak intensities are normalized to one for ease of comparison. These evolutions confirm the observations in the pole figures; that is, the most pronounced reorientation is the alignment of (1 0 0) poles with the 1-direction, evidenced by an increase of over six times their initial intensity, with corresponding depletion of these poles in the 2- and 3-directions. Concurrently, but less dramatically, the (0 1 1) pole population grows in the 2-direction to nearly three times its initial intensity while remaining relatively unaltered in the 2-direction, and all but completely depleting in the 1-direction. Here, the transition from constant to linearly varying peak intensities is observed to initiate around 200 MPa, resulting in an effective CRSS of 42 MPa.

3.3. Macroscopic mechanical response

3.3.1. In situ deformation of specimens

Fig. 6 shows the experimentally determined flow curves for Specimens 1–3, the in situ specimens. Consistent with the observation of higher effective CRSS being required to initiate substantial reorientation in the virgin specimen (52.5 MPa as opposed to 42 MPa), a larger applied stress is indeed also required to observe significant reorientation effects in the macroscopic stress–strain response, indicated by the point of deviation from the initial linear portion of these responses. Note that it has been shown that some reorientation also takes place in conjunction with elastic...
deformation during the initial application of stress [16,19], so while the initial part of these responses are linear in nature, thus lending themselves to an “effective” Young’s modulus being construed for use in many SMA constitutive models, this is not a measure of solely elastic deformation. The “effective” qualifier was analogously invoked in describing CRSS, as the critical stress to describe the onset of variant reorientation is not solely governed by the same physical mechanisms as in traditional slip. It is of value to observe these effective measures though, as they provide phenomenological insight to changes in SMA responses and have been found to be natural calibration parameters for phenomenological models simulating shape memory behaviors [31,32]. Here we see that the effective Young’s modulus decreases due to pre-texturing, though interestingly more so for the specimen pre-loaded to unload $e_{22} = -4.3\%$ as opposed to $e_{22} = -3.0\%$. This pattern suggests that increasing the amount of transverse-direction pre-strain directly corresponds with a decrease of the effective Young’s modulus in subsequent axial-direction compression.

Widening of the reorientation plateaux of the pre-strained specimens relative to the virgin specimen is also obvious. Recall that during the deformation of the virgin specimen shown here, an ~1.75 times increase in the $(1\ 0\ 0)$ pole population was observed in the 1-direction, while a greater than six times normalized peak intensity growth of the same poles was seen in the pre-loaded specimen. With this in mind, the observation that more macroscopic deformation correlates with larger amounts of variant reorientation is logical. Furthermore, it follows that pre-texturing a specimen in a direction perpendicular to the subsequent loading direction is a means to widen the reorientation plateau, i.e. achieve greater strains from the material, at least for a single cycle.

3.3.2. Ex situ deformation of specimens

Table 2 shows the measured strains in three directions and the corresponding effective Poisson’s ratios resulting from ex situ loading of Specimens 4 and 5. For Specimen 4, these are the unload strains, but in the straining of Specimen 5, all three strain gages did not remain in contact with the specimen surface throughout the complete deformation process. Thus the strains reported for this specimen were measured at the highest loading for which all three directional measurements were obtained. The applied stress–triaxial strain responses observed of both specimens are depicted in Fig. 7.

Some of the values reported in Table 2 are initially counterintuitive without consideration of the microstructure of the material. Perhaps these most obvious of these are the effective Poisson’s ratio values approaching 1 and 0 during 1-direction compression of Specimen 5. Considering the pre-loading of the specimen in conjunction with twinning dominating the non-elastic deformation, however, these values make sense. First, behaviors in the direction that is perpendicular to the load in each deformation process (3-direction) are examined. Recall from Section 3.2.2 that it was shown that noticeable variant reorientation occurred in the 3-direction during 2-direction compression. This elicits the development of substantial 3-direction strain (1.98%). Now recall that during subsequent compression in the 1-direction, texture in the 3-direction was relatively unaltered as the 3-direction remained perpendicular to the load (Fig. 5), thus the accumulation of additional 3-direction strain is also relatively minuscule (0.68%). To

<table>
<thead>
<tr>
<th>Specimen ID</th>
<th>Load direction</th>
<th>$\varepsilon_{11}$ (%)</th>
<th>$\varepsilon_{22}$ (%)</th>
<th>$\varepsilon_{33}$ (%)</th>
<th>$\nu_{12}$</th>
<th>$\nu_{21}$</th>
<th>$\nu_{23}$</th>
<th>$\nu_{13}$</th>
</tr>
</thead>
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<tr>
<td>4</td>
<td>Axial, 1-direction</td>
<td>$-4.15$</td>
<td>$2.66$</td>
<td>$1.83$</td>
<td>$0.64$</td>
<td>$0.44$</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5 (Pre-strain)</td>
<td>Transverse, 2-direction</td>
<td>$1.08$</td>
<td>$-3.26$</td>
<td>$1.98$</td>
<td>$0.33$</td>
<td>$0.61$</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Axial, 1-direction</td>
<td>$-3.38$</td>
<td>$3.29$</td>
<td>$0.61$</td>
<td>$0.97$</td>
<td>$0.18$</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

![Fig. 7. Measurements of the strains along the three principal axes of (a) Specimen 4 during 1-direction compression, (b) Specimen 5 during 2-direction compressive pre-straining, and (c) Specimen 5 during subsequent 1-direction compression.](image-url)
fully comprehend these effective Poisson’s ratio values, these observations relative to the behaviors of the directions that alternate between being loading direction and perpendicular to the loading direction (1- and 2-directions) must be considered. In Sections 3.2.2 and 3.2.3, consider-
able texture development was observed for each of these directions during both deformation processes. Hence it would be expected that applying axial strain along a direc-
tion that was previously transverse would cause significant strain development in both the new loading direction and the previous loading direction, and this was indeed observed. It now logically follows that since the texture development, and therefore strain development, in the 3-
direction was nearly saturated in the first deformation process, strain development is dominated by the 1- and 2-
directions in the second deformation process, and as a conse-
quence $v_{12}$ dwarfs $v_{13}$.

One result in Table 2 is still unclear, even through consider-
ation of these complex, inelastic, physical mechanisms: $v_{13}$ is smaller than $v_{12}$ for 1-direction straining of Specimen 4. Given the (1 0 0) and (0 1 1) fiber symmetries observed of the virgin specimen 1-direction (Fig. 3), an isotropic dilation would be expected during 1-direction deformation. This empirical evidence suggests, however, that very slight anisotropy (so slight it cannot be reasonably measured within the resolution of the experimental technique) can dramatically affect the dilation; that despite being cut in the same orientation relative to the ingot there was sam-
ple-to-sample texture variance; that one of the strain gages was not properly mounted or calibrated; that there is some mechanism other than the specimen’s crystallography affecting the transverse response; or some combination of these effects. The empirical evidence does not support a decisive conclusion. There is also an asymmetric dilation when straining a virgin specimen in the 2-direction (pre-
load of Specimen 5), as $v_{23}$ is significantly greater than $v_{21}$. In this case, however, the asymmetric response is expected and logical as (1 0 0) and (0 1 1) fibers were observed to be strongly aligned with the 1-direction axis and less pronounced along the 2- and 3-directions in the virgin material (Fig. 3c), i.e. the initial texture around the 2-direction axis is not symmetric.

4. Conclusion

Several correlations between texture development and mechanical responses were observed and verified for iso-

thermal deformation of martensite in polycrystalline NiTi. The propensity of (1 0 0) poles to align with, and (0 1 1) poles perpendicular to, the direction of compressive load-
ing was shown to be independent of choosing an axial or transverse loading direction relative to a parallelepiped geometry. In addition, the creation of a complex stress state was clearly observed by sequentially compressing specimens along their transverse and axial directions both in situ and ex situ, and through subsequent analysis, the inelastic nature of twinning was evident. In the realm of understanding the entirety of complex stress states, how-
ever, these data only begin to open the door. As work con-

tinues on this material system, the value of a verified and validated micromechanical model becomes more evident. Experiments such as these will always be necessary, but their cost—in terms of both money and time—is high. Complementing them with trusted simulations has proven to be an effective way to produce data more rapidly, and also to decouple more exactly the contributions of each physical mechanism to overall responses in other material systems [27]. Thus, the data set that has been presented has been collected with the micromechanical, multivariant modeling community in mind. In Ref. [22], the crystal mechanics driving the behaviors observed here are further explored through verification of the simplified multivariant model.

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