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Superelastic response of [111] and [101] oriented NiTi micropillars

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A combination of microcompression experiments on single crystal micron-scaled pillars of NiTi of known orientations and *in situ* neutron diffraction during loading of the same NiTi but in bulk, polycrystalline form are carried out to understand the stress-induced transformation associated with superelasticity at reduced length scales. At the length scales investigated, there is evidence through this work of a fully reversible stress-induced transformation from *B2* to *B19'* NiTi that does not involve additional dislocation activity or irrecoverable strains. The orientation dependence of the elastic deformation of the *B2* phase, the onset of its transformation to the *B19'* phase, the gradient and the hysteresis in the stress-strain response during transformation, the elastic modulus of the stress-induced *B19'* phase and the onset of plasticity are quantified and analyzed in these experiments by examining the crystallography of the *B2* to *B19'* transformation. © 2010 American Institute of Physics. [doi:10.1063/1.3445262]

I. INTRODUCTION

Near equiatomic NiTi can exhibit a stress-induced or temperature-induced, reversible, thermoelastic, martensitic phase transformation between a cubic *B2*, so called austenite phase and a monoclinic *B19'*, so called martensite phase. The shape memory effect associated with the thermally induced phase transformation results in shape changes occurring against external stresses upon heating, while superelasticity, associated with the stress-induced transformation, accommodates large deformation that is mechanically reversible.^{1,2} Existing NiTi based microdevices have employed the shape memory effect for small scale actuation,^{3,4} while the use of superelasticity has remained limited even though small scale structures and devices can utilize the large reversible strain accommodation and energy absorption attributes of the effect. A possible reason for the limited application is the lack of a clear understanding of superelasticity at these reduced length scales that would enable their incorporation into new fields of use and more importantly, the lack of consistency in the results reported. This is particularly true in the case of NiTi which to date has remained the most widely used alloy for its superelastic properties. For example, while the loss of superelasticity along with an orientation independent critical stress for inducing the phase transformation was reported in some experimental studies,^{5,6} others report no such loss of superelasticity in NiTi.⁷ Thus, the need for unambiguously establishing the mechanical behavior of NiTi at the length scales considered is two-fold—first, to enhance our fundamental understanding of superelasticity in order to extend the range of applications that specifically use the stress-induced transformation at reduced length scales and second, to enhance our fundamental understanding of martensitic transformations at reduced length

scales. While previous studies have used nanoindentation,^{8–11} nano/microcompression^{5–7,12,13} to investigate shape memory and superelastic alloys, there has been no effort to completely characterize the micromechanical response of superelastic NiTi at the reduced length scales investigated. Furthermore, there has been no emphasis placed on comparing the behavior of isolated micron-sized single crystals with their behavior when consolidated in bulk polycrystalline form, where the individual grain is constrained by surrounding material. By recourse to careful microcompression experiments and *in situ* neutron diffraction during loading at a spallation neutron source, we report here on work that addresses the aforementioned shortcomings.

A previously developed test methodology in which pillars were subjected to uniaxial compression at the micron/submicron scale¹⁴ has been employed here to mechanically characterize single crystal superelastic NiTi micron-scale pillars of known orientations. A uniaxial stress state acting on a sample of known geometry helps in a simplified conversion of the measured load-displacement response from an instrumented nanoindenter to the stress-strain response of the sample. *In situ* neutron diffraction studies during loading have previously been used to study the texture, strain, and phase fraction evolution in bulk polycrystalline NiTi.^{9,15–17} When compared to (low energy) x-rays, the increased penetration of neutrons is more representative of bulk polycrystalline behavior and is typically free of surface effects. The technique is used here to acquire diffraction spectra associated with a polychromatic neutron beam with fixed detectors at a spallation neutron source from bulk polycrystalline NiTi. Such spectra are analyzed to obtain information from grains of a specific orientation relative to the loading direction and correspond to atomic scale measurements of individual grains randomly distributed in a polycrystalline matrix (when the sample has no strong starting texture). The objective of this communication is to thus use a combination of micro-

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compression experiments on single crystal micron-scaled pillars of NiTi of known orientations and *in situ* neutron diffraction during loading of the same NiTi but in bulk, polycrystalline form, to understand the stress-induced transformation associated with superelasticity at reduced length scales.

II. EXPERIMENTAL METHODS

The NiTi (nominal composition 56.05 wt % Ni) alloy used in this study was fabricated using vacuum induction melting and vacuum arc remelting. The alloy was first solutionized at 1273 K for 1 h, oil quenched, and then aged at 673 K for 1 h followed by an ice water quench. The martensite start, martensite finish, austenite start, and austenite finish temperatures were 243 K, 213 K, 272 K, and 283 K, respectively. Large (70 μm) grains in the sample (average grain size approx. 30 μm) with [101] and [111] orientations in the parent austenitic state were identified using electron back scattered diffraction. Cylindrical micropillars were fabricated using an FEI 200 focused ion beam instrument. The [101] oriented pillar was 12.6 μm in diameter and 26.4 μm in height and the [111] oriented pillar was 9.4 μm in diameter and 31.4 μm in height. The microcompression studies were performed using an instrumented Hysitron Triboindenter at the Center for Integrated Nanotechnologies (CINT) at Los Alamos National Laboratory (LANL). A review of the factors influencing microcompression studies is available^{18,19} and was appropriately considered. The uniaxial compression studies on the pillars were carried out in displacement control at a constant displacement rate between 13 and 17 nm s^{-1} . This corresponds to an initial strain rate of $5 \times 10^{-4} \text{ s}^{-1}$. A 30 μm diameter flat diamond punch was used as the compression platen. The pillars were mechanically cycled (10 load-unload cycles with each cycle going to higher loads up to the loads reported in this work) to homogenize and remove any instabilities associated with the transformation while ensuring that the nonrecoverable strain was minimal.²⁰ Additionally, cylindrical compressive specimens (6.35 mm in diameter and 19.05 mm in gauge length) with threaded ends were fabricated by electrical discharge machining. *In situ* neutron diffraction measurements were performed in “time-of-flight” mode on the Spectrometer for Materials Research at Temperature and Stress (SMARTS) at LANL. SMARTS, a third generation neutron diffractometer, using a pulsed polychromatic neutron source, has two banks of detectors positioned opposite to each other. The loading axis of the sample formed an angle of 45° with the incident neutron beam, allowing measurements of the spacing of lattice planes in directions both along and normal to the loading axis of the sample. Additional information on the diffraction setup can be found elsewhere.²¹ A total of 51 load-unload compressive cycles were completed at ambient temperature, each between 0% to -4.0% in strain, with neutron spectra obtained at various strain levels during selected cycles. For intermediate cycles where no neutron data were collected, the crosshead displacement rate was $\pm 6 \text{ mm min}^{-1}$, while for neutron diffraction cycles, it was $\pm 0.2 \text{ mm min}^{-1}$. Optical microscopy showed an average grain size of 30 μm ,

FIG. 1. [101] oriented austenite NiTi pillar (a) before and (b) after deformation. The magnifications are appropriately selected to show adequate clearance for the nanoindenter tip in (a) and slip lines associated with plasticity in (b).

which ensured that there were a sufficiently large number of grains in the gauge and diffracting volumes for measurements representative of bulk polycrystalline behavior.

III. RESULTS AND DISCUSSION

A representative [101] oriented pillar used in this study is shown in Fig. 1(a) and the same pillar, following plastic deformation, is shown in Fig. 1(b). Figure 1(a) is appropriately magnified in order to show regions around the pillar that have been removed in order to apply large compressive strains to the pillar without the nanoindenter tip making contact with the material surrounding the pillar. Figure 1(b), on the other hand, is appropriately magnified to show the absence of buckling, brooming, or otherwise local deformation^{14,22} in the pillar following plastic deformation and is indicative of the uniaxiality of the stress applied by the nanoindenter tip. Furthermore, slip lines are also evident in the deformed pillar in Fig. 1(b). The stress-strain response of the [111] oriented pillar is shown in Figs. 2(a) and 2(b) while the stress-strain response of the [101] oriented pillar is shown in Fig. 2(c). Figures 2(a) and 2(b) represent different maximum loads to which the [111] oriented pillar was loaded to and unloaded and are separated here for clarity. Figure 2(a) is more representative of stress-strain responses previously reported in the literature⁷ and does not represent a complete transformation of austenite to martensite during loading as observed in the stress-strain response corresponding to higher applied loads

FIG. 2. Microcompression stress-strain response of (a) [111] oriented NiTi pillar mechanically cycled to 570 MPa, (b) 1480 MPa, and (c) [101] oriented NiTi pillar.

in Fig. 2(b). Similar to previously reported behavior in superelastic NiTi,^{16,17} while some irrecoverable strain remained upon unloading in the initial cycles, it was absent in the subsequent stress-strain responses reported here.

In order to compensate for potential compliance issues associated with the nanoindenter tip making initial contact with the top of the pillar, we do not report individual moduli for the two orientations but report their ratios. The ratio of the elastic moduli in the [111] and [101] directions from Figs. 2(b) and 2(c) is 1.20. The elastic modulus using single crystal stiffness constants $C_{11}=162$ GPa, $C_{12}=129$ GPa, and $C_{44}=35$ GPa,²³ was 97 GPa in the [111] direction and 77 GPa in the [101] direction. Alternatively, the elastic modulus using single crystal stiffness constants $C_{11}=137$ GPa, $C_{12}=103$ GPa and $C_{44}=34$ GPa,²⁴ was 95 GPa in the [111] direction and 78 GPa in the [101] direction.

Thus, the ratios of the moduli are 1.25 and 1.21, respectively, and agree well with the value of 1.20 determined in this work.

The stress for inducing the martensite phase from austenite, σ_{SIM} , for the [111] oriented pillar is determined to be 502 MPa using an arbitrarily selected 0.2% strain offset from the austenite elastic response, while it is 206 MPa for the [101] oriented pillar. The 0.2% offset method is adopted given the gradual transition of the stress-strain response from elastic to stress-induced phase transformation behavior that is macroscopically similar to elastic to plastic transitions in metallic alloys. The results from the microcompression experiments indicate a strong orientation dependence of the critical stress required for the onset of the phase transformation as the [101] oriented pillar exhibits a lower σ_{SIM} when compared to the [111] orientation. This difference in σ_{SIM} can be attributed to the preferential selection and formation of martensite correspondent variant pairs (CVPs) along the individual orientations,²⁵ which are additionally influenced by the distribution and size of the Ni_4Ti_3 precipitates which arise from the aging treatment used in this work.²⁶ Previous experimental studies,^{26–28} have established the [111] direction to be a hard orientation in compression for the austenite to martensite phase transformation, implying a higher σ_{SIM} . This is observed despite the role of Ni_4Ti_3 precipitates in assisting the phase transformation along the [111] orientation.²⁶ For the [101] oriented pillar, this is not the case as no significant differences were observed in σ_{SIM} in samples that were peak aged and over aged.²⁵ Furthermore, due to the presence of Ni_4Ti_3 precipitates, the [111] oriented pillar is expected to exhibit a sharper transition from elastic deformation to the onset of the phase transformation when compared to the [101] orientation,²⁹ as observed in this work. While we note reasonable agreement between values for σ_{SIM} between this work and others for the [101] oriented pillar²⁵ and for the [111] oriented pillar,²⁶ and disagreement between this work and others⁷ (which can be attributed to the lack of precipitates given the absence of an aging treatment in the latter case), we urge caution in these kinds of comparisons. This is due to the sensitivity of transformation temperatures to stoichiometry and residual stresses which directly impacts the determination of σ_{SIM} across samples with different processing histories given the Clausius–Clapyeron stress-temperature equivalence¹ in martensitic phase transformations.

The aforementioned discussion is supported by neutron diffraction results presented in Fig. 3, obtained from the same bulk polycrystalline alloy. Here we recognize the issues in comparing results from single crystals (microcompression testing) with those from a polycrystalline specimen (neutron diffraction testing). Recently, the role of grain-scale intergranular stresses³⁰ in the consolidated polycrystalline bulk response has been assessed and comparisons made with the single crystal coefficient of thermal expansion tensor. Due to the strong evolving texture in these alloys and the ability of these alloys to accommodate mismatch by detwinning, variant conversion/reorientation, and stress-induced transformation such comparisons are possible in a limited way as is applied here. Here neutron diffraction spectra are merely

FIG. 3. (Color online) Sections of neutron diffraction spectra at various stresses acquired from NiTi during compression loading. The spectra shown here are from diffracting grains oriented with their (111) and (101) plane normals parallel to the loading direction. The entire spectra were normalized so that (111) peaks have the same peak intensity.

used to identify the evolving martensite texture and the orientation of preferentially transforming grains relative to the loading direction, given that a source of spallation neutrons was used with fixed detectors and an incident polychromatic beam. The texture evolution in the austenite phase during the phase transformation is captured by comparing the relative

intensities of (111) and (101) peaks with increasing stress. These peaks correspond to two sets of diffracting grains, one oriented with their [111] directions along the loading axis and the other oriented with their [101] directions along the loading axis, in uniaxial compression. The spectra are normalized so that (111) peaks have the same maximum intensities at various stresses. Thus, the reduction in intensity of the (101) peaks with increasing stress implies a preferential transformation of [101] oriented grains compared to [111] oriented grains for the same stress state. The diffracting intensity ratios for these two grain orientations with stress (MPa) are 0.73 (at -230 MPa), 0.64 (at -380 MPa), 0.61 (at -440 MPa), and 0.55 (at -480 MPa) and is a direct measure of this preference when relative changes are compared. This is consistent with σ_{SIM} being lower for [101] when compared to [111] from the microcompression results.

Adapting from literature,³¹ and using a previously established convention for labeling CVPs,³² the transformation strains associated with austenite transforming to martensite as well as martensite variant conversion strains along $\langle 111 \rangle$ and $\langle 101 \rangle$ (austenite basis) are calculated and reported in Table I. The strains are calculated using $a=3.0010$ Å for austenite, and $a=2.8759$ Å, $b=4.1531$ Å, $c=4.5515$ Å, and $\beta=94.91^\circ$ for martensite, as determined from Rietveld refinement of neutron spectra obtained in this work. The variant conversion strains are only shown for possible variant conversions equivalent to a $(11\bar{1})$ type I twinning operation as observed in the neutron diffraction spectra in this work and also previously for other NiTi alloys.^{17,32} Recognizing that the neutron spectra are representative of grains under different stress states arising from intergranular interactions, anisotropy and stress-strain compatibility, and still only observing martensite texture evolution equivalent to $(11\bar{1})$ type I twinning in the spectra, we proceed in the following to only consider possible variant conversions associated with $(11\bar{1})$ type I twinning in our analyses of data from the NiTi pillars. The combinations are thus limited to conversions to variants 1, 1', 2, 2', 3, 3', 4, and 4' as shown in Table I. Since the strains reported in Table I use the cubic austenite as their

TABLE I. B_2 to B_{19}' transformation and B_{19}' variant conversion strains (%) calculated along $\langle 111 \rangle$ and $\langle 101 \rangle$ directions of the parent B_2 phase.

Variant	Transformation strain (%)		Variant conversion	Variant conversion strain (%)	
	$\langle 111 \rangle_{B_2}$	$\langle 101 \rangle_{B_2}$		$\langle 111 \rangle_{B_2}$	$\langle 101 \rangle_{B_2}$
1	-2.8	-3.9	3' $\rightarrow$ 1	-10.3	-11.1
1'	-2.8	2.4	6' $\rightarrow$ 1	0	0
2	7.5	2.4	5' $\rightarrow$ 1'	-10.3	0
2'	-0.5	-3.9	4 $\rightarrow$ 1'	0	4.5
3	-0.5	7.2	6 $\rightarrow$ 2	10.3	0
3'	7.5	7.2	4' $\rightarrow$ 2	10.3	4.5
4	-2.8	-2.1	3 $\rightarrow$ 2'	0	-11.1
4'	-2.8	-2.1	5 $\rightarrow$ 2'	0	0
5	-0.5	-3.9	5 $\rightarrow$ 3	0	11.1
5'	7.5	2.4	6' $\rightarrow$ 3'	10.3	11.1
6	-2.8	2.4	5' $\rightarrow$ 4	-10.3	-4.5
6'	-2.8	-3.9	6 $\rightarrow$ 4'	0	-4.5

basis, compressive strains arising in a $[111]$ oriented pillar would favor conversion to variants 1, $1'$, and 4. While other variants can form and exist, the complete transformation of austenite to martensite due to the high applied loads and the development of strong texture, as observed from analyses of the neutron spectra, suggests that this is extremely unlikely. From Table I, we now consider the strains associated with austenite transforming to martensite variants 1, $1'$, and 4 and note that all variants are associated with -2.8% strain. From Fig. 2(a), the strain that can be attributed to phase transformation of the $[111]$ oriented pillar is -2.1% (as determined by again using a 0.2% strain offset from the elastic stress-strain responses of austenite and martensite). The difference can be attributed to Ni_4Ti_3 precipitates that do not generate recoverable strains as well as not accounting for the non-orthogonal angle of the monoclinic martensite crystal in reporting strains with respect to a cubic basis. A similar determination for the $[101]$ oriented pillar results in conversion to variants 1, $2'$, 4, and $4'$ that generate strains of -3.9% , -3.9% , -2.1% , and -2.1% , respectively. The larger number of resulting variants (both in terms of the variants that are crystallographically possible and given the need to accommodate the disparity in the range of the magnitude of their resulting strains) for the $[101]$ oriented pillar when compared to the $[111]$ oriented pillar is consistent with a larger hysteresis in the stress-strain response for the $[101]$ oriented pillar since the hysteresis typically arises from energy dissipative processes associated with increased interface area (in this case variant boundaries).³³ From Fig. 2(c), the strain that can be attributed to phase transformation of the $[101]$ oriented pillar is 0.9% . This is lower than the strains associated with any of the martensite variants that can be expected to form and points to the existence of a criterion for the compatibility of individual CVPs (given the larger number of possible variants in this case as described previously). The difference between the predicted strains and the measured strains would necessarily imply that the martensite variants formed in the $[101]$ oriented pillars can still reorient with additional stress. This is indeed the case as evidenced by larger differences between subsequent stress-strain cycles for the $[101]$ oriented pillar when compared to the $[111]$ oriented pillar in Figs. 2(c) and 2(b), respectively.

On examining the lattice correspondence between the austenite and martensite phases, martensite variants 1, $1'$, and 4 that are expected to form during compression loading of the $[111]$ oriented pillar form with normals to the martensite (110) planes nearly (the deviation arises due to the non-orthogonal monoclinic angle) aligned with the loading axis. The elastic modulus using the single crystal stiffness constants determined from *ab initio* calculations reported³⁴ and validated^{30,35} is 168 GPa in the martensite $[110]$ direction. In calculating the elastic modulus, attention was paid to appropriately considering the coordinate system used in that work.³⁴ A similar analysis for the $[101]$ oriented pillar results in 1, $2'$, 4, and $4'$ variants forming with normals to (010) and (211) planes aligning parallel to the loading direction. The elastic moduli in the $[010]$ and $[211]$ directions are 111 GPa and 101 GPa, respectively. These predicted values are higher than the unloading martensite modulus of 69 GPa deter-

FIG. 4. Yielding of stress-induced martensite in (a) $[111]$ and (b) $[101]$ oriented NiTi pillars.

mined from Fig. 2(b) for the $[111]$ oriented pillar and 35 GPa determined from Fig. 2(c) for the $[101]$ oriented pillar. The reason for choosing the unloading modulus rather than the loading modulus was to reduce inelastic contributions to the modulus. We attribute this difference to martensite detwinning, variant conversion, growth, and coalescing processes that are occurring in the pillars since the maximum transformation strain has not been attained as described previously. Previous nanoindentation and neutron diffraction experiments have shown evidence of such a deflation of the measured modulus.⁹ We also note the increased gradient during the stress-induced transformation (the region of the stress-strain curve that is used to determine the transformation strain in Figs. 2(b) and 2(c) for the $[101]$ oriented pillar when compared to the $[111]$ pillar. This observation is consistent with more elastic strain energy that has to be stored during transformation of the $[101]$ oriented pillar when compared to the $[111]$ oriented pillar. The increased stored elastic energy can be directly attributed to the larger number of variants that can form in the case of the $[101]$ oriented pillar and the lower modulus measured due to detwinning and variant conversion processes.

Figures 4(a) and 4(b) show the respective stress-strain responses of the $[111]$ and $[101]$ oriented pillars loaded beyond the onset of plasticity in the stress-induced martensite. The yield stress in Fig. 4(a) was determined to be 1670 MPa for the $[111]$ oriented pillar and 1140 MPa for the $[101]$ oriented pillar in Fig. 4(b) using a 0.2% offset strain from the elastic stress-strain response of the stress-induced martensite. The difference in yield stresses for the two pillars is not

FIG. 5. (Color online) Section of neutron diffraction spectra acquired in the nominally unloaded condition before and after 50 mechanical cycles (i.e., at the start of cycle 1 and cycle 51). The spectra shown here are from diffracting grains oriented with their (111) and (101) plane normals along the loading direction. The entire spectra were normalized so that (101) peaks have the same peak intensity. The peak at 1.765 Å is due to the steel from the extensometer knife edge and does not influence the analysis since the normalization considered peak heights and not areas.

exactly clear. A possible reason is the variant reorientation that occurs during loading of martensite in the [101] oriented pillar and is responsible for a lower apparent elastic modulus may produce favorable orientations of the martensite variants for slip. Another possible reason is stress that may develop due to the larger number of variant boundaries in the [101] oriented pillar compared to the [111] pillar. Nevertheless this orientation dependent difference in the yield stress has implications for the generation of irrecoverable strain during mechanical cycling in bulk polycrystalline samples. On comparing normalized austenitic neutron spectra at the start of the stress-induced transformation before and after 50 cycles in the nominally unloaded condition in Fig. 5, we note a reduction in intensity in the (111) peaks when compared to the (101) peaks. This implies that there are fewer austenite grains oriented with the (111) planes normal to the loading direction when compared to the (101) planes. Given that the single crystal pillar experiments point to higher stresses for both the onset of the transformation and plasticity for the [111] oriented pillar, the reduction in the (111) peak intensity can be attributed to residual or retained martensite³⁶ that results from the inability of [111] oriented grains to generate large strains or reorient upon transformation when compared

to the [101] oriented grains (even though the former was not observed in our [101] oriented pillar experiments due to the early onset of plasticity).

IV. SUMMARY

Thus, for the first time the complete micromechanical response of superelastic NiTi is examined by performing careful microcompression experiments on single crystal pillars of known orientations. Specifically, the orientation dependence of elastic deformation of austenite, the onset of its transformation to martensite, the gradient, and the hysteresis in the stress-strain response during transformation, the elastic modulus of the stress-induced martensite, and the onset of plasticity of the stress-induced martensite are analyzed in separate experiments. A majority of the results were explained by recourse to a quantitative determination of strains associated with austenite grains transforming to martensite CVPs or twinning in martensite. Comparison is also made with neutron diffraction experiments on a bulk polycrystalline superelastic sample in order to justify calculation of the strains and examine the role of intergranular interactions on superelastic behavior. At the length scales investigated, there is evidence through this work for a fully reversible stress-induced transformation from austenite to martensite (that does not involve additional dislocation activity). Such use of a reversible martensitic phase transformation can thus be made to accommodate mismatch in material microstructures and nanostructures (e.g., composites or multilayered materials) or superelasticity in structures and devices at reduced length scales (e.g., grippers, medical implants, etc.).

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¹K. Otsuka and X. Ren, *Prog. Mater. Sci.* **50**, 511 (2005).

²K. Bhattacharya, *Microstructure of Martensite* (Oxford, New York, 2003).

³Y. Fu, H. Du, W. Huang, S. Zhang, and M. Hu, *Sens. Actuators, A* **112**, 395 (2004).

⁴M. Kohl, *Shape Memory Microactuators* (Springer, Berlin, 2004).

⁵C. P. Frick, B. G. Clark, S. Orso, P. Sonnweber-Ribic, and E. Arzt, *Scr. Mater.* **59**, 7 (2008).

⁶C. P. Frick, S. Orso, and E. Arzt, *Acta Mater.* **55**, 3845 (2007).

⁷D. M. Norfleet, P. M. Sarosi, S. Manchiraju, M. F. Wagner, M. D. Uchic, P. M. Anderson, and M. J. Mills, *Acta Mater.* **57**, 3549 (2009).

⁸C. P. Frick, T. W. Lang, K. Spark, and K. Gall, *Acta Mater.* **54**, 2223 (2006).

⁹S. Rajagopalan, A. L. Little, M. A. M. Bourke, and R. Vaidyanathan, *Appl. Phys. Lett.* **86**, 081901 (2005).

¹⁰G. A. Shaw, J. S. Trethewey, A. D. Johnson, W. J. Drugan, and W. C. Crone, *Adv. Mater.* **17**, 1123 (2005).

¹¹C. R. Rathod, S. Rajagopalan, and R. Vaidyanathan, Proceedings of the International Conference on Shape Memory and Superelastic Technolo-

- gies, 2003 (SMST Society, Inc., Menlo Park, 2004), p. 331.
- ¹²J. San Juan, M. L. N6, and C. A. Schuh, *Adv. Mater.* **20**, 272 (2008).
 - ¹³J. San Juan, M. L. N6, and C. A. Schuh, *Nat. Nanotechnol.* **4**, 415 (2009).
 - ¹⁴M. D. Uchic and D. M. Dimiduk, *Mater. Sci. Eng., A* **400–401**, 268 (2005).
 - ¹⁵V. B. Krishnan, R. M. Manjeri, B. Clausen, D. W. Brown, and R. Vaidyanathan, *Mater. Sci. Eng., A* **481–482**, 3 (2008).
 - ¹⁶C. R. Rathod, B. Clausen, M. A. M. Bourke, and R. Vaidyanathan, *Appl. Phys. Lett.* **88**, 201919 (2006).
 - ¹⁷R. Vaidyanathan, M. Bourke, and D. Dunand, *Metall. Mater. Trans. A* **32**, 777 (2001).
 - ¹⁸D. Kiener, C. Motz, and G. Dehm, *Mater. Sci. Eng., A* **505**, 79 (2009).
 - ¹⁹H. Zhang, B. E. Schuster, Q. Wei, and K. T. Ramesh, *Scr. Mater.* **54**, 181 (2006).
 - ²⁰S. Miyazaki, T. Imai, Y. Igo, and K. Otsuka, *Metall. Trans. A* **17**, 115 (1986).
 - ²¹M. A. M. Bourke, D. C. Dunand, and E. Ustundag, *Appl. Phys. A: Mater. Sci. Process.* **74**, s1707 (2002).
 - ²²X. Shi, G. Saada, and P. Veyssi6re, *Philos. Mag. A* **73**, 1419 (1996).
 - ²³O. Mercier, K. N. Melton, G. Gremaud, and J. Hagi, *J. Appl. Phys.* **51**, 1833 (1980).
 - ²⁴T. M. Brill, S. Mittelbach, W. Assmus, M. Mullner, and B. Luthi, *J. Phys.: Condens. Matter* **3**, 9621 (1991).
 - ²⁵K. Gall, H. Sehitoglu, Y. I. Chumlyakov, and I. V. Kireeva, *Acta Mater.* **47**, 1203 (1999).
 - ²⁶H. Sehitoglu, I. Karaman, R. Anderson, X. Zhang, K. Gall, H. J. Maier, and Y. Chumlyakov, *Acta Mater.* **48**, 3311 (2000).
 - ²⁷K. Gall and H. Sehitoglu, *Int. J. Plast.* **15**, 69 (1999).
 - ²⁸S. Miyazaki, S. Kimura, K. Otsuka, and Y. Suzuki, *Scr. Metall.* **18**, 883 (1984).
 - ²⁹K. Gall, H. Sehitoglu, Y. I. Chumlyakov, I. V. Kireeva, and H. J. Maier, *ASME J. Eng. Mater. Technol.* **121**, 28 (1999).
 - ³⁰S. Qiu, V. B. Krishnan, S. A. Padula II, R. D. Noebe, D. W. Brown, B. Clausen, and R. Vaidyanathan, *Appl. Phys. Lett.* **95**, 141906 (2009).
 - ³¹T. Saburi and S. Nenno, Proceedings of the International Conference on Solid-Solid Phase Transformations, Pittsburgh, 1981 (AIME, Warrendale, 1982), p. 1455.
 - ³²D. Dunand, D. Mari, M. Bourke, and J. Roberts, *Metall. Mater. Trans. A* **27**, 2820 (1996).
 - ³³R. J. Salzbrenner and M. Cohen, *Acta Metall.* **27**, 739 (1979).
 - ³⁴M. F. X. Wagner and W. Windl, *Acta Mater.* **56**, 6232 (2008).
 - ³⁵S. Qiu, B. Clausen, S.A. Padula II, R.D. Noebe and R. Vaidyanathan, *Acta Mater.* (submitted).
 - ³⁶S. A. Padula II, S. Qiu, D. Gaydos, R. D. Noebe, G. Bigelow, A. Garg, and R. Vaidyanathan, *Metall. Trans. A* (submitted).