The authors report on a relatively new alloy, Ni_{54}Ti_{45}Hf, that exhibits strengths more than 40% greater than those of conventional NiTi-based shape memory alloys — 2.5 GPa in compression and 1.9 GPa in torsion — and retains those strengths during cycling. Furthermore, the superelastic hysteresis is very small and stable with cycling. Aging treatments are used to induce a very high density of Ni_{4}Ti_{3} precipitates, which impede plasticity during cycling yet do not impart substantial dissipation to the reversibility of the phase transformation. Pairing compression testing with high-energy synchrotron X-ray diffraction and aberration-corrected electron microscopy provides an in-depth look at the structure-property relationships of this alloy. Specifically, it is found that a combination of small, untwinned retained martensite laths, and dislocations on the austenite-martensite interfaces primarily strengthen the alloy as opposed to dislocation networks. Furthermore, some combination of nanoprecipitation and interface dislocations is responsible for the remarkably low mechanical hysteresis exhibited by this material.

1. Introduction

In 1958, extraordinary behaviors were discovered in the equiatomic shape memory alloy (SMA) nickel-titanium (NiTi, also known as “Nitinol”). In SMAs, phase transformation between an austenite phase composed of a high-symmetry crystal structure and a martensite phase of low crystal symmetry can lead to remarkable strain recovery capabilities. Specifically, deformations imposed upon martensite can be fully recovered when thermally transforming back to austenite, giving the material an apparent “shape memory effect.” These same deformations may also be recovered while the material is still under load, giving rise to actuation. Similarly, SMAs can recover seemingly inelastic strains by deforming above the austenite finish temperature to the extent martensite is stress-induced. When the load is removed, the martensite phase reverts to its original austenite crystal structure and shape, a phenomenon known as superelasticity. These recoverable strains may approach 13% in single crystals. Scientists and engineers have since imagined numerous applications enabled by three functional behaviors of near-equiatomic compositions of NiTi: shape memory effect, actuation, and superelasticity.

Concurrent to the discovery of these unique SMA behaviors, it was documented that the structural alloy Ni_{55}Ti_{45} (in atomic percent) was paramagnetic and exhibited very high hardness and outstanding resistance to cavitation erosion, and stress and crevice corrosion. Decades later, a NASA search for bearing materials that could withstand shock and vibration loads during space launch showed that Ni_{55}Ti_{45} exhibits a higher static load capacity to resist denting than conventional bearing alloys because of its comparably high hardness and relatively low stiffness. Lower stiffness allows for reduced stresses at contacts via increased contact area (low stiffness), while retaining comparable high hardness allows the same stress levels to be elastically accommodated. The net effect is that bearing components made from this material can withstand much higher loads without plastic deformation. Ni_{55}Ti_{45} was also found to react favorably to lubrication, unlike other alloys with high titanium content, and it even outperforms bearing steels in
spiral orbit tribometer tests with oils representative of those used in spacecraft mechanisms. For these reasons, Ni$_{55}$Ti$_{45}$ alloys are especially desirable materials for tribology applications, evidenced by the targeted deployment of Ni$_{55}$Ti$_{45}$ bearing elements for the International Space Station’s environmental control system.

Recently, Dellacorte et al. showed that substituting just 1 at% Hf for Ni in Ni$_{54}$Ti$_{45}$Hf$_1$ significantly improved strength and wear resistance in rolling contact fatigue tests. This small alloying addition of Hf in place of Ni was initially made to reduce the solvus temperature, as quench-cracking had been observed in the binary composition when rapidly cooled from solid-solution temperatures. As a result of these demonstrated successes, there has been a growing interest in these materials and a drive to understand the mechanisms responsible for their remarkable behavior. In the remainder of this article, we proceed to document the structure-property relationships of this alloy. Specifically, in situ X-ray diffraction, electron microscopy techniques, and mechanical testing are used in tandem to study the deformation mechanisms and microstructures of the material in response to compression loadings. The results are considered in the light of an established theory for structure-property relationships of SMAs.

2. Experimental Section

2.1. Sample Preparation

The majority of the material studied in this investigation came from a master heat of Ni$_{54}$Ti$_{45}$Hf$_1$ (at%) that was vacuum-induction-melted (VIM) using a cold crucible technique and cast into an ingot 30 mm in diameter and 600 mm long. The ingot was homogenized at 1050°C for 24 h, then sealed inside a mild steel can and hot-extruded into 11.5 mm diameter rods at 1000°C. To demonstrate the insensitivity of the observed behavior to processing technique, the data in Figure 1b was generated from a master heat of pre-alloyed Ni$_{54}$Ti$_{45}$Hf$_1$ powder. Hot isostatic pressing (HIP) was performed on the powder at 103 MPa for 4 h at a temperature of 982°C to form a solid ingot. The ingot was then cut into sample blanks, which were solution-treated at 1000°C for 2 h and water-quenched.

Final samples, produced from both the VIM and powder metallurgy ingots, were machined using a CNC lathe, then aged at 400°C for 30 min. Compression cylinders 1 mm in diameter and 2 mm long were used for synchrotron diffraction experiments. Cylinders 5 mm in diameter and 8 mm long were used to generate Figure 1b. Two cylindrical dog-bone-shaped sample geometries, 50 mm in length, were used for compression and torsion testing (Figure 1a,c). Compression samples had 5 x 14 mm gage sections, tapered from 9.5 mm diameter grip ends using a 4.8 mm radius. Torsion samples had 1 x 1 mm gage sections, tapered from 6.3 mm diameter grip ends using a 9.5 mm radius.

2.2. Microscopy

Microstructure characterization was carried out using scanning electron microscopy (SEM) and scanning transmission electron microscopy (STEM). Bulk microstructure observations were made using an FEI Quanta 200 SEM in backscatter mode. STEM samples were trenched and thinned to electron transparency using an FEI Helios DualBeam focused ion beam (FIB). An FEI CM200 TEM was used for initial observations and to obtain selected area diffraction patterns. Aberration-corrected STEM was performed on an FEI Titan^3 80–300 at 300 kV, allowing for atomic resolution analysis of tested samples.

2.3. Mechanical Testing

Compression tests used to generate Figure 1 were performed using an MTS 810 servo-hydraulic load frame managed with an
MTS FlexTest SE digital controller. Load was measured with a 100 kN load cell. Strain within the gage section was measured with a clip-on extensometer with a 12.7 mm gage length. Mechanical loading was controlled in strain control at a rate of 1 × 10−4 s−1 between load limits equivalent to 0 and 2 GPa engineering stress.

The torsion test was performed using a Mark-10 electromechanical load frame fitted with a 5.7 N-m torque cell. Surface strains were measured using a GOM ARAMIS digital image correlation system configured with stereo five-megapixel cameras, 50 mm C-mount lenses and focusing optics, polarizers, and blue light band-pass filters. The strains reported are the average strains over the portion of the gage section of the sample that was in view of both cameras, excluding the edges of the analyzed regions. The test was run at a constant twist rate of 0.2° s−1 and the sample was incrementally cycled six times to a higher twist each cycle, starting with 10° and increasing to 35° in increments of 5°.

2.4. Synchrotron Diffraction Experiments

In situ high-energy X-ray diffraction compression experiments were performed on the A2 station of the Cornell High Energy Synchrotron Source (CHESS). Monochromatic (50.3 keV), unfocused X-rays were selected from the high-energy wiggler spectrum using a double-crystal silicon <111> monochromator and defined by slits to an incident beam size of 0.6 mm wide by 0.5 mm tall. During the experiment, the sample gage was centered in the X-ray beam and the gage volume was irradiated. The Bose 3330 25 kN load frame was rotated about the sample center through 130°, which has a texture-free intensity nearly 80% greater than the next most intense (hkl) reflection, which has a texture-free intensity nearly 80% greater than the next most intense (hkl) reflection. This masking allowed other reflections to be more easily observed in a given exposure, as the detector only had a 12-bit effective dynamic range once background was subtracted.

3. Results and Discussion

3.1. Mechanical Testing

Mechanical testing results for Ni54Ti45Hf1 are shown in Figure 1. Figure 1a compares the room-temperature compression response of the aged and unaged samples processed by VIM + hot extrusion. The aged sample was cycled five times and the unaged sample was cycled ten times, between load limits of 0 and 2 GPa. Both samples exhibited minimal plastic strain accumulation after the first cycle. The unaged material appears to exhibit elastic-plastic behavior in the first cycle, with linear-elastic behavior upon repeated loading/unloading after the first cycle, indicating normal elastic-plastic work-hardening of the material. This mechanical behavior suggests that in the unaged condition, room temperature is above the martensite desist (Mf) temperature for the material.

Contrarily, the aged sample exhibited a fully recoverable superelastic response approaching 4% engineering strain and strengths over 2.5 GPa. More than 40% greater load carrying capacity than commercially available NiTi alloys.14 Thus, in the aged condition, room temperature is below the Mf temperature of the material. This thermodynamic change is most likely due to the depletion of nickel from the matrix as the nickel-rich Ni4Ti3 phase precipitates.15–17 This additional toughness provides a greater ability to absorb applied loads or deformations without permanent damage to the material.

Figure 1b shows compression responses for the powder metallurgy processed material in the aged condition measured at three unique temperatures. With increasing temperature, the stress required to initiate macroscopic superelasticity increased according to Δσ/stress = 3.5 MPa/°K. This value was calculated from the data shown in Figure 1b, noting that the onset of inelastic deformation during loading (as measured by greater than 5% deviation from the initial loading modulus) shifted from 945 to 1130 MPa as the test temperature increased from 22 to 75°C. Note that the actual Clausius-Clapeyron relation for this material is likely non-linear, as a larger increase in transformation stress was observed in increasing from 22 to 50°C than from 50 to 75°C. Hence, the value we report here should not be interpreted to be more than a first-order approximation. Still, using this value, in addition to the transformation stress at room temperature, we can extrapolate the martensite start and austenite finish temperatures to approximate values of 24 K/−249°C and 61 K/−212°C, respectively. These transformation temperature approximations explain why we could not measure transformation temperatures using differential scanning calorimetry (DSC), even with liquid nitrogen cooling. Furthermore, because the transformation temperatures were not able to be measured, it is probable that the transformation would not occur at these low temperatures without an applied load due to kinetic arrest, as has been observed in other SMA systems.18 Torque testing of the aged, hot-extruded material, shown in Figure 1c, demonstrated an analogous superelastic response in shear, exceeding 8% fully recoverable shear strain. Maximum engineering shear strengths of 1.9 GPa were observed in torsion according to the usual calculation made at the outer radius of the gage sections: τ = T/r, where r is the engineering shear stress, T is the applied torque, r is the radius of the gage section, and JG is the torsion constant for the gage cross-section (again, test to failure not shown).
3.2. Synchrotron Diffraction Experiments

Results from in situ synchrotron diffraction experiments are shown in Figure 2. X-ray area detector patterns, calculated by summing all of the area detector images collected for all sample rotations at each load step normalized by the number of images that were summed, are shown for the initial state (−10 MPa), the peak load (−2290 MPa), and unloaded after 10 cycles in Figure 2a–c, respectively. Figure 2a also includes labels for some of the cubic Debye–Scherrer rings and azimuthal direction, η. Corresponding diffraction spectra calculated by integrating the summed and normalized X-ray area detector patterns are provided in Figure 2d–f, respectively. Select reflections are labeled for the various phases present. The initial mean lattice parameter for the B2 phase, found using the Pawley method\[^{19}\] for fitting the diffraction spectrum shown in Figure 2d, was 3.0067 ± 0.0004 Å.

A subset of the X-ray data – centered at ω = 7.5° and η = 342.5° – was integrated over 5° in ω and η and compared.

Figure 2. Summed X-ray area detector patterns a) at −10 MPa (with several of the cubic Debye–Scherrer rings and azimuthal direction (η) labeled), b) at −2290 MPa (peak) applied engineering stress, and c) unloaded after 10 compression cycles to −2290 MPa engineering stress. d–f) Integrated summed diffraction spectra corresponding to a–c), respectively, with reflections labeled for different phases present. g–i) Comparison between summed spectra and region integrated over 5° in ω (where ω is the rotation of the sample) and η, centered at ω = 7.5° and η = 342.5°, corresponding to d–f), respectively. Evidence for retained B19° martensite after cycling is shown in i).
with the summed and normalized spectra. The angle \( \omega \) indicates the rotation of the sample about its loading axis — the choice of \( \omega = 0^\circ \) was arbitrary since the sample was cylindrical. The angle \( \eta \) is a rotation from horizontal on the X-ray area detector, as shown in Figure 2a. These comparison diffraction profiles corresponding to initial, peak, and unloaded states are plotted in Figure 2g–i, respectively. From the integrated subset of data, reflections consistent with B19\(^\prime\) monoclinic martensite ([103/201] and [022]) planes can be identified, indicating the presence of retained martensite in the unloaded state after 10 cycles. These monoclinic \((hkl)\) reflections overlap with cubic B2 \((hkl)\) reflections in the fully summed and normalized data, resulting in a convoluted, slightly asymmetric peak-broadening effect (Figure 2i). This convolution of peaks, together with a low volume fraction of martensite, posed great challenges to complete analysis of the retained martensite phase fraction, as Rietveld refinement of both phases was numerically unstable. Further insight as to why the total summed diffraction pattern was ambiguous with respect to martensite was gained by considering the morphologies observed with electron microscopy, especially the size of the retained martensite structures.

3.3. Electron Microscopy

Microstructure investigations were performed using both SEM and TEM techniques to complement the X-ray experiments. Samples for both SEM and TEM observations were cut using a Struers Accutom-50 high speed saw and mechanically polished using an Allied MultiPrep parallel polishing system to a grit size of 1200. They were then placed in a vibratory polisher with 0.05 \( \mu m \) colloidal silica for 24 h to remove any residual damage layer from polishing. Transverse section backscatter SEM micrographs of polished bulk samples of the hot-extruded, aged Ni\(_{54}\)Ti\(_{45}\)Hf\(_{1}\) material are shown in Figure 3 in both the untested state and after 10 load cycles. The initial state microstructure, shown in Figure 3a, contains regions of both smaller and larger grains, ranging approximately 5–40 \( \mu m \) in diameter. After 10 compression load cycles to 2290 MPa, the resulting microstructure appears to show similar grains, only with distinct surface features that suggests the presence of martensite. Because they are uniquely oriented within each grain, they are inconsistent with potential polishing damage. This observation is more evident in examining uncycled and cycled microstructures are shown at higher magnification in Figure 3c and d, respectively. The white particles approximately 1 \( \mu m \) in diameter are consistent with HfO\(_2\), which was determined using X-ray energy dispersive spectroscopy (not shown).

Results from TEM-level investigations of a FIB-fabricated thin foil sample prepared from the 10-cycle-tested sample are shown in Figure 4. Several bright, lath-like features can be seen traversing low-angle grain boundaries in the dark-field STEM image shown in Figure 4a. The fine streaks vary in width from 10 to 100 nm and form at an angle approximately 45\(^\circ\) to the loading direction, which is horizontal with respect to the images of
Figure 4. a) Dark-field STEM image of the aged, 10-cycle-tested Ni_{54}Ti_{45}Hf_{1} alloy showing martensite streaks crossing low-angle grain boundaries. b) Higher magnification micrograph showing a martensite lath within the matrix containing a very high density of Ti_{3}Ni_{4} precipitates on the size scale of 10–30 nm. c) TEM selected area diffraction pattern of a matrix region exhibiting the characteristic $\{321\}$ type reflections, indicated by the arrows, confirming the rhombohedral structural characteristic of the Ti_{3}Ni_{4} precipitates.

Figure 4a and b. A higher magnification annular dark-field (ADF) STEM image is shown in Figure 4b. This image was acquired at a camera length of 220 mm, resulting in strong diffraction contrast. A single high-intensity lath is shown within the matrix, containing a high density of Ti_{3}Ni_{4} precipitates approximately 10–30 nm in size. Selected area electron diffraction measurements of a matrix region confirm the rhombohedral structural characteristic of the Ti_{3}Ni_{4} precipitates, evidenced by the characteristic $\{321\}$ type reflections that are indicated by the arrows in Figure 4c.

Further investigation of the lath-like features using high-angle annular dark-field (HAADF) STEM, shown in Figure 5, confirmed that they were retained B19' martensite structures. Figure 5a shows an isolated martensite lath within the B2 matrix. The region within the square inset is shown at higher magnification in Figure 5b, where the atomic structure of both the [001]B2 and [100]B19' zone axes can be viewed simultaneously. The martensite lath is a single variant, and the patchy contrast results from the high density of Ti_{3}Ni_{4} precipitates present throughout. Fast Fourier transforms confirming the structures

Figure 5. a) HAADF STEM image showing an isolated martensite lath within the B2 matrix. The region identified by the square inset is shown at higher magnification in b) where the martensite atomic column structure can be observed. The patchy contrast arises from the Ti_{3}Ni_{4} precipitates present throughout. c), d) Fast Fourier transforms (FFTs) of the B2 and B19' regions in b), confirming structures and showing a slight lattice rotation between the regions.
of the B2 and B19′ regions are shown in Figure 5c and d, respectively. There appears to be a slight lattice rotation between the two regions.

An additional FIB foil was fabricated to determine whether a relationship existed between the martensite laths observed using STEM and the grain surface texture seen using SEM. An appropriate grain in the aged, 10-cycle-tested transverse section sample chosen for this purpose is shown using secondary electron SEM imaging in Figure 6a. A 2 μm tall layer of platinum was deposited to protect the surface structure, shown in Figure 6b, prior to following standard FIB-foil trenching and thinning procedures.

Once the sample was thinned to electron transparency, it was imaged using ADF STEM, shown in Figure 6c. The imaged region contains remnants of the surface platinum layer. Five parallel martensite laths can be seen intersecting at an angle with the surface. The loading direction is vertical in the case of Figure 6c. The spacing between the laths appears to be consistent with SEM observations of the target grain. This result confirms that the surface features seen in the cycled material (Figure 3) result from the presence of fine laths of retained martensite. This retained martensite is the primary source of unrecovered strain after the first mechanical cycle of the aged material in Figure 1a — not all of the material transforms back to the cubic austenite phase after the first mechanical cycle. In elastic-plastic alloys, and likely the “unaged” condition in Figure 1a, this work-hardening would occur via dislocation mechanisms and lead to a loss in ductility. However, in this aged condition, significant plasticity in the form of dislocation networks or forests is not observed. However, it is puzzling how martensite would be retained without pinning from dislocations, thus we probed further into the structure of the deformed material in search of dislocations.

Further microstructure observations made using STEM are shown in Figure 7. Figure 7a shows an array of coarsened Ti₃Ni₄ precipitates along a low-angle grain boundary above a martensite lath in the lower left corner. Figure 7b shows larger precipitates within the bulk of the grains, away from any boundaries. Two different martensite lath variants can also be seen intersecting in the upper region. While dislocations in the bulk are challenging to identify and characterize because of the immense strain fields produced by the high density of Ti₃Ni₄ precipitates, they can occasionally be seen interacting with larger precipitates. An example of this is shown in Figure 7c. This is the only identifiable dislocation content in the B2 matrix that was resolved, in spite of attempts at diffraction contrast imaging with various B2 reflections.

High-resolution STEM was also used to determine the retained martensite lattice parameters, which were not discernable with X-ray methods due to the average B2 peak broadening effect that dominated observations/measurements of most martensite reflections (as previously discussed and shown in Figure 2i). Using STEM, the monoclinic structure cannot be fully characterized through information from the [100] zone axis seen in Figure 5b, which provides only two of the lattice parameters. Data from the [010] zone axis is necessary to obtain the third lattice parameter and the monoclinic angle. An additional FIB foil was fabricated from an adjacent site, trenched

![Figure 6](https://www.advancedsciencenews.com/)

**Figure 6.** a) Secondary electron SEM micrograph showing the grain from which the TEM sample was extracted from the aged, 10-cycle-tested Ni₄Ti₆Hf₁ alloy using focused ion beam (FIB) methods. b) Selected area after depositing protective platinum layer, before trenching adjacent regions. c) ADF STEM micrograph of corresponding thinned foil, with preserved surface morphology showing parallel martensite laths consistent with spacing in SEM observations.
at a 90° angle relative to the previous foil. Using the new thinned foil, the [010] zone could be reached within a feasible tilt range in STEM.

Figure 8a shows a high-resolution HAADF STEM image of the austenite-martensite interface of a lath captured within the foil. The B2 matrix was tilted to a [110] zone axis, which caused a [010] zone axis of B19\textsuperscript{0} to be simultaneously visible. Both (b) and (c) in Figure 8 show higher magnification HAADF STEM images of the [100]B19\textsuperscript{0} and [010]B19\textsuperscript{0} structures, respectively, together with structural models for comparison and confirmation of the structure in the lower left corners. By pairing orthogonally acquired images with scan distortion correction software,\textsuperscript{[20]} the monoclinic lattice parameter ratios, ratios of cubic versus monoclinic parameters, and the monoclinic angle could be measured. The undeformed B2 lattice parameter measured with X-rays (again, 3.0067 ± 0.0004 Å) was used as a reference for the ratios measured with STEM to calculate the martensite lattice parameters. Mean lattice parameters for retained B19\textsuperscript{0} in the aged, tested material were found to be $a = 2.82 \pm 0.06$ Å, $b = 4.08 \pm 0.03$ Å, $c = 4.53 \pm 0.03$ Å, and $\beta = 97° \pm 1°$.

Using the latest developments in the crystallographic theory of martensite,\textsuperscript{[21–23]} which mathematically describe the ability for the austenite and martensite phases to share a common crystallographic plane without any distortion of the interface, the geometric compatibility of the austenite and retained martensite phases can be quantitatively described by the values of the first cofactor condition $\lambda_2 = 0.9595$ and the second cofactor conditions (CC2) for Mode A twinning $= 2.5 \times 10^{-4}$, Mode B twinning $= 5.7 \times 10^{-3}$, Mode C twinning $= 5.3 \times 10^{-3}$, and Mode D twinning $= 2.0 \times 10^{-4}$ (twinning mode designation follows\textsuperscript{[24]}). A $\lambda_2$ value not near unity (where near unity usually refers to agreement to the hundredths, thousandths, or better) indicates very poor austenite-martensite geometric compatibility;\textsuperscript{[25]} a single variant of martensite should not be able to exist next to a single austenite crystal with a straight, elastically strained interface given this $\lambda_2$ value. This result likely explains the curved and stepped nature of the atomically resolved interface in Figure 8a.

Careful evaluation of the atomic plane continuity captured in the STEM images of the interface further reveals several examples of dislocations near the interface, some of which are shown in Figure 9. Dislocations with clearly different Burgers vectors and significant edge component are indicated by the white and red dislocation symbols in Figure 9. An example of
one such interfacial dislocation is exemplified by the horizontally compressed image of Figure 9b, which allows the extra half-plane of the dislocation to be more clearly evaluated, along with the Burgers circuit. This analysis indicates that there is only a single set of (220) B2 planes associated with this (and the other) dislocations that have been analyzed. This closure failure indicates that the interfacial dislocations could be either $\frac{1}{2}$[110] B2 or $\frac{1}{2}$[111] B2 (or $\frac{1}{2}$[111] B2) mixed dislocations for this viewing direction. The Burgers circuit analysis is inconsistent with dislocations of the type $a<100>$, which are the dislocations usually associated with plasticity in NiTi with B2 crystal structure.\[26,27\] These dislocations could nonetheless help compensate the elastically incompatible single-variant martensite and B2 matrix, and also provide the mechanism for retaining martensite. The near-zero CC2 conditions for martensite twins indicate that straight martensite-austenite interfaces of many different martensite twin fractions could form if the first cofactor condition were close to unity; although, it should be noted that such interfaces are not theoretically possible for Mode A and Mode D twins, regardless of the twin fractions given these austenite and martensite structures.

The ramifications of near-zero CC2 but $\lambda_2$ not near unity are not theoretically proven; however, we have observed in a Ni$_{50.3}$Ti$_{29.7}$Hf$_{20}$ alloy that nanoprecipitation lowers one of the CC2 conditions while $\lambda_2$ does not really improve, but that the change in the cofactor conditions coincides with reduced hysteresis.\[28\] The fact that we observe a curved, stepped, dislocated, and single-variant martensite-austenite interface together with very small hysteresis (in fact, nearly zero in torsion, see Figure 1c), while simultaneously having a poor first cofactor condition and many near-zero second cofactor conditions, seems to indicate that perhaps microstructures that satisfy the second cofactor condition but not the first can still show remarkably low hysteresis. Such a conclusion, however, is not definitive from this work, as we have not uniquely deconvoluted such an effect from the role and short-range mechanisms of the nanoprecipitation in this alloy. In fact, we hypothesize that the coupling of the mechanisms is a necessary condition for remarkably low hysteresis in an alloy with $\lambda_2$ far from unity. The latter condition necessitates plasticity on interfaces for geometric accommodations, yet the plasticity requires nanoprecipitation to accommodate the plasticity in a manner that does not inhibit the reversibility of the phase transformation. Deeper understanding of the microstructure mechanisms controlling hysteresis in these alloys is desired, as hysteresis is an important characteristic for any alloy designed for fatigue, as it is a measure of the damage (i.e., dissipated work) each time the material is cycled. In theory, an alloy with zero hysteresis should have infinite life in fatigue loading – the mechanical behaviors should be perfectly reversible.

4. Conclusions

Ni$_{54}$Ti$_{45}$Hf$_{1}$ has been studied using a combination of mechanical testing, X-ray synchrotron diffraction, and electron microscopy methods. The alloy exhibits strengths more than 40% greater than commercially available NiTi, as well as a fully recoverable macroscopic superelastic response exceeding 4% in compression and 8% in torsion. These properties, together with high hardness and resistance to Brinell-type damage observed by other researchers,\[10,13\] make the alloy highly desirable for further development for tribological applications, and we postulate that the increased mechanical toughness that we observed in static loading could also carry into higher rate deformations, providing great advantages for high-rate impact resistance, such as in armors. Furthermore, the very low mechanical hysteresis exhibited by this alloy in spite of the presence of interface dislocations and a high volume fraction of precipitates suggests that the hysteresis of SMAs can be tuned to be small through plasticity and precipitation, even when the austenite and martensite phases cannot share a common, undistorted crystallographic plane across an interface.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

In situ x-ray diffraction, Martensite, Precipitates, Scanning transmission electron microscopy, Shape memory alloy

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