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Quantitative Studies of microstructural phase transformation in Nickel–Titanium

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A new experimental approach is used to quantify the full field heterogeneous strain accommodation associated with martensitic phase transformation in Nickel–Titanium at the microstructural length scale. Local deformation measures were matched with underlying microstructural characteristics obtained by EBSD, providing point-by-point correlation between transformation extent and microstructure. It was found that a single grain can accommodate both a heavily deformed martensite phase and an austenite phase, and that the transformation does not progress in an ordered fashion but rather can skip over grains or portions of grains in an extremely heterogeneous manner. The martensite front was diffuse at the microstructural length scale, and formed a cross-hatch structure in advance of the stable band. Strain accommodation was strongly heterogeneous at the grain level; while on average the martensite band accommodated approximately 10% strain, some individual regions accommodated up to 20% strain while regions of retained austenite remained at 1% strain.

1. Introduction

Shape memory alloys (SMAs) are increasingly employed in high performance systems. However, the role of microstructure on phase transformation in these polycrystalline materials—while known to be critically important to their function—is not well understood. SMAs exhibit two unique properties arising from a solid-to-solid, diffusionless phase transformation: the shape memory effect and superelasticity [1]. Both properties allow SMAs to recover large strains with little residual deformation and stem from a complex phase transformation between an austenite phase and one or more martensite variants. Superalastic deformation in Nickel–Titanium, which is examined here, proceeds with a transition from a parent B2 structure directly to a single variant, or twinned variant pair, of a B19 structure accommodating the applied strain. Recovery occurs upon unloading through reverse transformation to the thermodynamically stable austenite phase. Theoretically, the lath structure of martensite variants developed within a polycrystalline austenite matrix is extremely complex in both the superelastic and shape memory cases. Such variant configurations have been extensively modeled (for a review of microstructural models, see [2]), but experimental confirmation of these fine microstructures has proven difficult and fundamental questions about small scale transformation remain unanswered.

There is an extensive body of work characterizing the heterogeneity involved with the martensitic transformation at the macroscopic length scale (for a thorough review, see [3]), but few measurements have been made of transformation at length scales on the order of the grain size or smaller. Multiple techniques have shown that at the macroscale, Lüders-like band structures of localized martensite nucleate and propagate to accommodate strain in a specimen under tensile loading at relatively low strain rates [4–8]. Initially, these martensite bands were regarded as monolithic structures composed purely of favorable martensite variants with a sharp transition—defined by a crystallographic habit plane—between the transformed and untransformed regions. However, recent investigations [9,10] have shown that the bands are not monolithic, nor comprised solely of favorable martensite variants. Rather, the martensite band at the microscale consists of a complex array of interacting variants and has no clearly defined boundary with the untransformed material. Martensite will transform at the microstructural length scale in advance of the macroscopic front, and areas within the martensitic band can remain in the parent austenite structure despite massive transformation in the surrounding areas. Such observations, however, have thus far been qualitative in describing martensite formation within a polycrystalline structure. In situ TEM and synchrotron XRD studies [11–13] have resolved sub-granular local strain and crystal structure in polycrystalline superelastic NiTi specimens. These measurements provided...
detailed quantitative mechanical information tied to microstructure, but were limited to measuring deviatoric lattice strains in the austenite phase and inferring martensite transformation in areas where the austenite diffraction pattern could not be resolved. High-resolution, full-field measurement of accommodated strains in austenite, martensite, and two-phase regions over a representative polycrystalline structure is needed for the validation of micromechanics-based constitutive models, and warrants further detailed investigation for both the scientific interest in SMAs and the impact such models have on their development and use.

2. Material and methods

Full-field, quantitative measures of deformation at the microstructural length scale were captured through a method combining scanning electron microscopy and digital image correlation (SEM–DIC). Digital image correlation is a non-contact optical technique used to determine full-field displacements on the surface of a sample with sub-pixel resolution, where gray-scale images of the surface are compared to track the movement of material subsets (for details see [14,15]). This tracking typically requires the application of a random high-contrast pattern of appropriate feature size to the sample surface. The full-field displacement maps can then be used to determine surface strains by a local application of continuum strain definitions. DIC was initially developed for use with traditional optical imaging, and extensive work has been performed to remove the spatial distortions of typical optical elements. When applying DIC to scanning electron micrographs, far more complex distortions are caused by the rastering and time dependent nature of SEM image capture, the complicated ‘shape’ of electron focusing lenses, and other factors. To remove the spatial and temporal distortions from the micrographs used in this study, custom scripts were created using [16] as a starting point. This process enables SEM–DIC to reach sub-pixel resolution on the order of that achieved in traditional optical DIC systems.

Dogbone-shaped specimens were cut following ASTM E345 from 0.480 mm thick superelastic NiTi (50.8 at% Ni) received from Nitinol Device Corporation. The specimen tensile axes were oriented parallel to the sheet rolling direction. Specimens with nominal gage dimensions of 4.5 mm × 18 mm were encapsulated in quartz and backfilled with argon (0.9999 purity). Specimens were heat treated at 900 °C for 1 h followed by a water quench, resulting in a mean grain diameter of ~70 μm. Heat treated specimens were mechanically ground and polished to a mirror finish, and their microstructure was characterized by Electron Backscatter Diffraction (EBSD). Samples were then patterned for SEM–DIC using a custom-built apparatus (Fig. 1a) modeled after [17].

In situ tensile tests were performed in a FEI Dualbeam Quanta 3D equipped with a Kammrath and Weiss tension/compression stage. The samples were loaded under displacement control at a nominal strain rate of 5 × 10⁻⁵ s⁻¹. Images of the patterned surface were captured periodically and concurrently with grip displacement and load cell data. High-resolution, quantitative strain fields were calculated using displacements generated by the SEM–DIC procedure and are shown in Fig. 2 (for details on the methodology please see [18–20]). The EBSD crystallographic data was aligned with the SEM–DIC measured strain field using a pattern of platinum markers deposited via Focused Ion Beam. The discrete averaged DIC strain field data of three distinct microstructural neighborhoods, one of which is shown in Fig. 2a–f, are matched with the macroscopic stress–strain curve, as shown in Fig. 2.

3. Results and discussion

SEM–DIC was used to track the evolution of strain in both austenite and martensite phases as martensite bands progressed through the polycrystalline microstructure. At a globally applied stress of ~450 MPa, a single macroscopic martensite band nucleated near the center of the specimen and then propagated though the gage section under a nominally constant stress of 400 MPa. The microscale strain maps shown in Fig. 2b–e show what appears to be the progression of a single martensite front. At

![Fig. 1.](image-url)
Fig. 2. Experimentally measured strain fields during stress-induced martensitic transformation in superelastic NiTi. The macroscopic stress strain curve was generated using grip displacement. Stress dips in the curve occurred during pauses in grip displacement required to capture in situ images. Each highlighted point represents a spatial average of an equally sized area of interest (left, center and right) in the gage section of the sample. The images (a)–(f) below are spatial strain maps in the right area of interest captured during band propagation through the area. Images (a)–(e) were captured from 0.015–0.04 macroscopic axial (grip) strain and show the local microstructural strain progressing from an average of 0.01 in image (a) to 0.1 in image (f). Image (f) is a map of the residual strain in the microstructure after the sample is completely unloaded. Please see online edition for color images.
the microstructural length scale, the front was diffuse and formed a crosshatch-like structure in advance of the stable band, where stability is defined as less than ±0.25% change in local strain value from the previous measurement. This diffuse cross-hatched region extended approximately 150 μm beyond the stable region of the band. The areas that first transformed in the diffuse band as the front progressed tended to attain the highest local strains when transformation in those areas saturated. When a region was subsumed by the stable macroscopic band, strain accumulation in that region ceased and local strain levels remained nominally constant (within the aforementioned ±0.25%) as loading continued and the macroscopic band propagated past the region.

By taking a local average of strain values in the region shown in Fig. 2e, which was fully inside the macroscopic martensite band, it was determined that ‘complete’ transformation accommodated an average of 10% longitudinal strain. However, this strain was accommodated remarkably heterogeneously. Some sub-grain areas experienced strains of up to 20%, while others essentially remained near 0% strain despite the large amounts of transformation that could encompass their boundary. Comparison of strain and EBSD data indicates that areas accommodating larger strains developed primarily along grain boundaries while the low strain regions generally lay in grain interiors. The fact that the larger strains appear to have a microstructural association with barriers to dislocation motion (i.e. grain boundaries) may indicate that they are primarily caused by additional plastic deformation either within or closely associated with the martensitic transformation. The exact mechanisms and details of this heterogeneity, including specific variant activation, the interaction between plastic deformation and martensitic transformation, and the role of intergranular constraint on progression or cessation of transformation, require detailed analysis of the strains surrounding particular microstructural features at higher resolution; this is currently being pursued.

The high resolution deformation maps in Fig. 2a–f yield an amount of data comparable to over 80,000 submicron strain gages distributed across each area of interest. While the analysis of these images gives new insight into the spatial heterogeneities of deformation, this spatial consideration of strain does not elucidate whether these strains are the result of single deformation mode, or multiple deformation modes acting simultaneously in the microstructural neighborhood. To gain insight into the relative activity of deformation mechanisms, we consider in Fig. 3 the evolution of the strain distribution. Initially (Fig. 3a), a single Gaussian distribution centered at approximately 1% strain indicates a single phase of primarily elastically deformed austenite. As the macroscopic martensite band progresses across the field of view (Fig. 3b), there is a transition from the elastic austenite distribution, which remains centered at the 1% strain value, to a martensite peak centered at 6–7% and a higher strain peak that likely represents plastic deformation. When the macroscopic martensite band has fully passed through the field of view (Fig. 3c), the distribution of strains continues to show a large population with a peak at nominally 8% associated with saturated martensite, but also shows secondary peaks at higher strain levels. These secondary peaks are much lower in intensity than the primary martensite peak and extend the strain distribution of the saturated band into ranges beyond those for martensitic transformation alone. Given the annealed state of the sample and the existence of residual deformation corresponding to regions of high strain (Fig. 2f), a reasonable postulate is that these high-strain deviations represent plastic deformation of the transformed martensite. How this plasticity interacts with martensitic transformation in subsequent cycles will be addressed in...
future work, as will the identification of the variants actively accommodating strain in the early stages of transformation.

4. Conclusions

The experimental technique and results shown here are a quantitative evaluation of the complex interactions between microstructure and martensitic transformation that are fundamental to SMA behavior. Transformation heterogeneity at the microscale has been characterized by a new experimental approach combining scanning electron microscopy and digital image correlation, and directly correlated to the material microstructure obtained by EBSD. It was found that a single grain can accommodate both a heavily deformed martensite phase and an austenite phase, and that the transformation does not progress contiguously but rather can skip over grains or portions of grains in an extremely heterogeneous manner. The martensite front was diffuse at the microstructural length scale, and formed a cross-hatch structure in advance of the stable band. Nearly all of the deformation that a microstructural neighborhood experienced, including transformation or recovery (within the limits of detection), took place only at the phase front, rather than within the already transformed band. The strain accommodation was strongly heterogeneous at the grain level; while on average the martensite band accommodated approximately 10% strain, some individual regions accommodated up to 20% while the retained austenite regions remained at 1% strain for the duration of the test. Additional studies using SEM–DIC at higher resolution are underway to investigate in greater detail the mechanisms responsible for the heterogeneous deformation observed in these alloys.

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