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On the plasticity of small-scale nickel–titanium shape memory alloys

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Focused ion beam machined compression pillars created from [1 1 1], [0 0 1] and [2 1 0] NiTi demonstrate that orientation plays a dominant role in determining dislocation flow stress in stress-induced martensite. This is in contrast to bulk NiTi in which martensite strength is primarily dictated by precipitate size. Post-mortem transmission electron microscopy and Laue microdiffraction measurements reveal respectively dense dislocation structures and stabilized martensite consistent with bulk observations in heavily deformed NiTi.

Keywords: Plastic deformation; Shape memory alloys (SMA); Compression test; Transmission electron microscopy (TEM); Micropillars

Nickel–titanium (NiTi) is of particular interest because of its unique strain recovery behavior. Under the appropriate conditions, NiTi is capable of recovering up to approximately 8% strain upon removal of the applied load (i.e. pseudoelasticity) or upon the application of heat (i.e. shape memory behavior) [1,2]. Essentially, NiTi experiences a thermoelastic, stress-induced martensitic phase transformation, whereby the original crystal lattice experiences a shear-like phase transformation that is reversible.

Widespread use of NiTi is limited because mechanical behavior is extremely sensitive to composition, microstructure and processing history. For example, applied strain recovery associated with the martensitic phase transformation for various microstructures degrades significantly before 16 cycles [3], and high-cycle fatigue shows relatively low fracture toughness values [4]. It has been theorized that an increased propensity for defect nucleation exists at the austenite–martensite interface, which hinders phase transformation recovery [3]. However, martensitic stabilization associated with cold working of NiTi has also proven to be useful by generating a two-way shape memory effect; when processed correctly, dislocations can be used to direct the formation of the martensite variants, allowing a spontaneous shape recovery under thermal cycling in the absence of applied stress [5]. While dislocations are well known to influence or inhibit the martensitic phase transformation in NiTi, the fundamental relationship between the two is not well understood.

In order to gain deeper insight into the mechanisms associated with NiTi plastic deformation, the effect of size scale on the compressive flow stress of stress-induced martensite is investigated in this study. Focused ion beam (FIB) machined compression pillars created from aged [1 1 1], [0 0 1] and [2 1 0] Ti–50.9 at.% Ni single crystals, as well as pillars cut from individual grains in solutionized polycrystalline NiTi were tested. For the NiTi used in this study, the austenite parent phase (B2) is stable at room temperature, and the martensite phase (B19') is induced upon loading [6,7].

However, to better understand the mechanisms involved, additional microstructural analysis was first performed on previously deformed samples. In a prior study by the current authors, martensite flow stress values in aged [1 1 1] NiTi compression pillars ranging in diameter from 2 μm to 200 nm were shown to be unaffected by pil-

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lar size [7]. This was partly expected due to randomly distributed, semicoherent Ti_3Ni_4 precipitates 10 nm in diameter, with interparticle spacing of the same order. Because the precipitate spacing was significantly smaller than the smallest diameter tested (below 200 nm), these precipitates probably dominated the stress needed for dislocation motion. However, despite the known influence of these precipitates, preliminary testing of a 1 µm diameter [2 1 0] NiTi pillar surprisingly demonstrated a reduced martensite flow stress at approximately one-third the average value of the [1 1 1] from [7]. This observed orientation dependence is in stark contrast with bulk [8] and micro/nanoindentation NiTi studies [9,10], which show that precipitate size and coherency strongly influence martensite flow stress and dominate over single-crystal orientation.

Figure 1 displays the stress–strain behavior and a scanning electron microscope (SEM) image of a heavily deformed [1 1 1] NiTi pillar approximately 2 µm in diameter. The initial loading/unloading cycles of the pillar at low strains (<6%) demonstrate pseudoelasticity, as evidenced by the flag-shaped hysteresis loops in the stress–strain graph (Fig. 1a). This is a signature of the recoverable austenite-to-martensite phase transformation, which dominates deformation at low strains. Upon further loading the pillar transforms to martensite, which proceeds to deform primarily elastically until the curve exhibits a large burst in displacement at a stress of approximately 3000 MPa. Such bursts have been demonstrated in other load-controlled micropillar studies, and have been shown to correlate to dislocation events [11,12]. Because the focus of this study is the martensite plastic deformation, only pillars taken to high strains (>10%) were investigated. SEM imaging of the

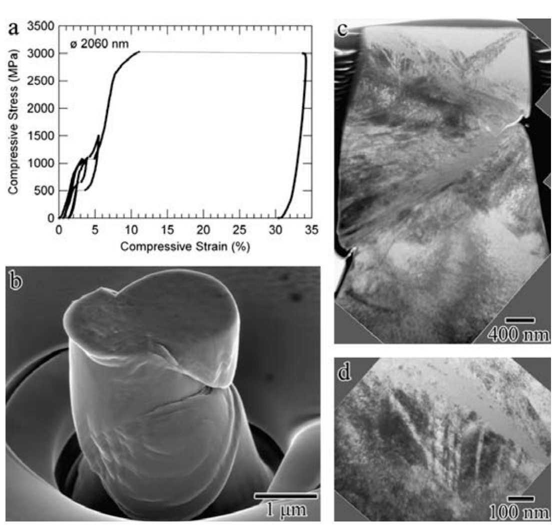

Figure 1. Compression behavior and electron microscopy images of a heavily deformed [1 1 1] NiTi pillar approximately 2 µm in diameter taken from a previous study [7]. (a) Stress–strain curve demonstrating pseudoelasticity in the first two loading cycles, and upon further loading a large burst in displacement at a stress of approximately 3000 MPa, after which the strain is not recovered. (b) SEM image showing massive slip primarily along three slip planes. (c) Bright-field TEM micrographs of a cross-sectional sample overlapped together to display the entire pillar. (d) Higher-magnification TEM image taken from the upper left corner of the pillar. TEM images show distinct slip traces and pockets of twins, believed to be residual martensite.

pillar (Fig. 1b) shows massive slip deformation, which is representative of all pillars tested to large strains. Interestingly, saw-tooth features are also observed on the surface. Such triangular features were infrequently observed, and are probably caused by the stabilized martensite variants formed during the phase transformation, previously observed in NiTi samples [13].

Post-deformation, the pillar shown in Figure 1b was sectioned via FIB into a transmission electron microscopy (TEM) sample, in a manner similar to that outlined in Frick et al. [12]. Figure 1c shows bright-field TEM micrographs overlapped together to display the entire pillar, while Figure 1d shows a higher-magnification image of the upper left pillar corner. Both Figure 1c and d illustrate distinct slip traces and pockets of twins, believed to be residual martensite. Although the phase transformation is pseudoelasitic, dislocation-assisted stabilized martensite is often observed in heavily deformed NiTi [10]. Compression-induced martensite is known to take the form of two twin-related martensite plates, formally termed correspondence variant pairs (CVPs) [14,15]. From Figure 1d, the width of the CVPs range from approximately 15 to 30 nm. Additionally, Figure 1c and d show dense dislocation tangles throughout the pillar. These results are not expected to significantly vary over the size scales tested in this study, as near 200 nm diameter NiTi pillars compressed to approximately 20% strain during in situ TEM testing also demonstrated residual dislocations and stabilized martensite [16]. It is important to note that Figure 1b and c also demonstrate large slip traces through the entire pillar diameter, between 30° and 40° relative to the applied load. Therefore, although the dispersed Ti_3Ni_4 precipitates and CVP martensite structure heavily influence dislocation motion, once the flow stress was reached, dislocations propagated across the width of the sample.

Similar results shown in Figure 2 were obtained for a 2.8 µm diameter NiTi pillar tested by Laue microdiffraction at the MicroXAS beam line of the Swiss Light Source [17]. This sample was cut from the same parent crystal as the previous example in Figure 1, and therefore has the same nominal composition and precipitate microstructure. The [2 1 0] orientation was chosen because preliminary results demonstrated a much lower martensitic flow stress than that observed in [1 1 1] NiTi [7]. Discrete spatial diffraction measurements along the pillar length before testing (Fig. 2a) and after testing (Fig. 2b) demonstrate the presence of austenite phase throughout. However, after compression, the upper one-fifth of the pillar revealed the presence of the martensite phase (Fig. 2c). Thus, Laue diffraction measurements of [2 1 0] NiTi coincide with TEM imaging of [1 1 1] NiTi, demonstrating stabilized stress-induced martensite. It is important to note that due to pillar taper, stress values will nominally depend on spatial location along the length of the pillar [12], being largest at the top. Using the diameter at the pillar top, the maximum stress measured during compression was approximately 900 MPa, approximately one-third the average flow stress value observed for the [1 1 1] NiTi pillars [7]. Figure 2d and e display the $(0 2 \bar{2})$-peak of the austenite phase before and after testing, respectively. Before testing a mild anisotropic broad-