Deformation twinning in Ni–Mn–Ga micropillars with 10M martensite

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The maximum actuation frequency of magnetic shape-memory alloys (MSMAs) significantly increases with decreasing size of the transducer making MSMAs interesting candidates for small scale actuator applications. To study the mechanical properties of Ni–Mn–Ga single crystals on small length scales, two single-domain micropillars with dimensions of $10 \times 15 \times 30 \mu\text{m}^3$ were fabricated from a Ni–Mn–Ga monocrystal using dual beam focused ion beam machining. The pillars were oriented such that the crystallographic c direction was perpendicular to the loading direction. The pillars were compressed to maximum stresses of 350 and 50 MPa, respectively. Atomic force microscopy and magnetic force microscopy were performed prior to fabrication of the pillars and following the deformation experiments. Both micropillars were deformed by twinning as evidenced by the stress-strain curve. For one pillar, a permanent deformation of 3.6% was observed and ac twins (10M martensite) were identified after unloading. For the other pillar, only 0.7% remained upon unloading. No twins were found in this pillar after unloading. The recovery of deformation is discussed in the light of pseudoelastic twinning and twin-substrate interaction. The twinning stress was higher than in similar macroscopic material. However, further studies are needed to substantiate a size effect. © 2009 American Institute of Physics.

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I. INTRODUCTION

Nonstoichiometric Ni_2MnGa magnetic shape memory alloy (MSMA) receives significant attention due to its ability to deform in response to a variable magnetic field.^{1–5} Depending on whether or not the magnetic-field-induced deformation is permanent after the removal of the magnetic field, this effect is also called magnetoplasticity (if the deformation is permanent) and magnetoelasticity (if the deformation is recovered).^{5,6} A large magnetic anisotropy and a high mobility of twin boundaries are at the origin of magnetic-field-induced deformation.

The magnetic-field-induced stress is limited by the magnetic anisotropy energy. The blocking stress was recently doubled by reducing the actuator size.⁷ This size effect was attributed to an enhancement in the energy barrier to magnetization rotation at small sample size. Experiments with a magnetic-field pulse indicated that the actuation frequency is affected by inertia and eddy currents.⁸ The inverse magneto-plastic effect, i.e., the deformation-induced change in magnetization, was utilized for power generation.^{9–12} The efficiency of MSMA-based power generation devices strongly increased with increasing actuation frequency.¹² All these phenomena indicate that the performance of MSMA-based sensors and actuators improves when reducing the size of the device. The size dependence of functional properties is the motivation to study mechanical properties of MSMA at small length scales.

Microscale deformation was studied in materials using nanoindentation,^{13,14} tension-torsion testing of wires,¹⁵ bending of thin foils¹⁶ and beams,¹⁷ microtensile testing,^{18,19} and compression tests of micropillars.^{20–24} In all cases, for smaller dimensions, an increase in flow stress or strength was reported. A large change in mechanical properties is expected when the size of an object is comparable to the characteristic length specific to a primary deformation mechanism.²⁵ For dislocation-mediated processes, the characteristic length is of the order of $1 \mu\text{m}$.²⁶ Twin boundary motion occurs by the motion of twinning disconnections.^{27–30} Disconnections are linear interfacial defects with a Burgers vector component similar to dislocations. In addition, disconnections have a step component. It is reasonable to assume that the characteristic length scale is also of the order of $1 \mu\text{m}$. Thus, a similar size effect as observed for monocrystalline micropillars of various fcc and bcc materials may also be expected for MSMA micropillars.

The overall goal of the present study is to explore the mechanical and magnetomechanical properties of Ni–Mn–Ga MSMA single crystals at small length scale. Before transitioning to the submicrometer length scale, it is important to demonstrate the experimental procedure in the “known regime” and to show that proper characterization of the deformation micromechanisms is possible. The aim of this work is (i) to prepare MSMA single crystal micropillars in a single-domain state with known orientation, (ii) to deform the pillars and measure the stress-strain curves, and (iii) to identify the deformation mode.

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FIG. 1. Inclined SEM micrograph of both Ni-Mn-Ga micropillars before load application. Both micropillars have dimensions of $10 \times 15 \times 30 \mu\text{m}^3$. Cartesian coordinates are defined as indicated, i.e., x , y , and z parallel to the short, intermediate, and long edges of the pillars.

II. EXPERIMENTAL

Two Ni-Mn-Ga micropillars with dimensions $10 \times 15 \times 30 \mu\text{m}^3$ were fabricated near the edge of a $\text{Ni}_{51.5}\text{Mn}_{27}\text{Ga}_{21.5}$ (numbers indicate at. %, and the composition error is about 0.5%) single crystal with faces parallel to $\{100\}$ using dual beam focused ion beam (FIB) machining. For the composition range of this sample, both 10M (pseudotetragonal) and 14M (pseudo-orthorhombic) martensite were reported.³¹ Before micropillar fabrication, the sample was polished on two adjacent faces using a $1 \mu\text{m}$ diamond slurry to a surface roughness of 10 nm. The sample was then “trained” via a thermomechanical treatment at 150°C . During heating and cooling, a constant load of $15 \pm 3 \text{ MPa}$ was applied in the short sample direction. This was done to orient the crystal lattice with its shortest lattice parameter c parallel to the shortest edge of the sample. The crystallographic a directions aligned predominantly parallel to the mid- and long sample edges. The micropillars were prepared in an orientation such that the crystallographic c direction was perpendicular to the loading direction of the micropillar deformation experiments. Before machining, the sample was characterized with atomic force microscopy (AFM) and magnetic force microscopy (MFM) to ensure that the pillars had the correct orientation. Cartesian coordinates are defined on the micropillar with the x , y , and z directions parallel to the short, intermediate, and long edges of the micropillars (Fig. 1). Thus, the loading direction was the z direction. The crystallographic c direction was parallel to the Cartesian x direction.

For fabrication of the micropillars, a dual beam FIB/scanning electron microscope (SEM) (Zeiss Leo 1540 XB) operated with a Ga^+ ion source at 30 keV was applied. Since the sample edge was rounded, a sharp $\sim 100 \mu\text{m}$ long edge was milled using a 10 nA beam current. Subsequently, the micropillars were fabricated close to this edge using an ion current of 2 nA for coarse milling and a milling current of 100 pA for final polishing. All four sample sides were fabricated with individually adjusted tilt angles on the order of 3°

FIG. 2. *In situ* SEM micrograph of micropillar 1 being compressed by a truncated cone-shaped microindenter. Note that there are image distortions due to charging of the diamond tip.

to minimize sample taper. Moreover, after tilting the sample by 90° the micropillar top faces were also polished. These efforts were taken to ensure a homogenous stress distribution during loading.

Samples were loaded using an indenter (UNAT, AS-MEC) equipped with a flattened conical diamond tip mounted in an SEM (Zeiss LEO 440 Stereoscan), as shown in Fig. 2. The flat punch had a diameter of $20 \mu\text{m}$. Thus, precise positioning using *in situ* SEM observation was required in order to properly cover the microcompression samples. Again, great care was taken to ensure alignment between sample surface and flat punch.

Sample loading was performed in displacement controlled open loop mode with initial strain rates of $\sim 2 \times 10^{-4} \text{ s}^{-1}$. For micropillar 1, a 30 s creep segment was added after reaching the maximum displacement, while micropillar 2 was unloaded just after reaching the maximum displacement. The load-displacement data, as well as consecutive SEM images, were recorded during the test.

A Veeco Dimension 3100 AFM system was used to characterize the surface relief and magnetic domain structure of each micropillar. The surface relief was imaged in tapping mode using a Veeco MESP ferromagnetic tip coated with CrCo. For MFM, the tip was magnetized so that the magnetic moment of the tip was parallel to the tip axis and perpendicular to the sample surface.

III. RESULTS

The engineering stress-strain curves for both micropillars upon loading and unloading are shown in Fig. 3. They were calculated based on the measured load-displacement data and the sample dimensions determined precisely after FIB fabrication. For micropillar 1, twin boundary motion sets in at 60 MPa as indicated by a sudden reduction in the slope of the stress-strain curve. Twinning is discontinuous

FIG. 3. Deformation curves for micropillar 1 (solid line, larger loop) and micropillar 2 (dotted line, smaller loop). The arrows indicate the onset of twinning events. At some large twinning events, the slope of the stress-strain curve appears negative because deformation advances so fast that the feedback loop control reduces the applied load to minimize overshooting of the displacement. Besides the linear elastic recovery, there is a nonlinear strain recovery of about 1% for both micropillars. The recovery is assumed to stem from pseudoelastic twinning. The permanent strains are 3.6% for pillar 1 and 0.7% for pillar 2).

and causes numerous serrations (arrows). On average and over the entire twinning regime, there is significant hardening during plastic deformation. After compression to 7% strain, significant strain recovery sets in upon unloading, leaving a permanent strain of 3.6%. For micropillar 2, twin boundary motion sets in at 50 MPa. About 2.5% strain of the applied compressive strain of 3.2% is recovered after unloading, leaving only 0.7% permanent strain.

Following loading and unloading, AFM and MFM measurements were performed on each micropillar. Figures 4(a) and 4(b) are the height image and magnetic image of the yz face of micropillar 1. Slightly inclined ridges and valleys are visible on the top end of micropillar 1. The areas marked with boxes in Fig. 4 are magnified in Figs. 5(a) and 5(b) and show the twinned region in greater detail. A schematic of the twin structure found in Figs. 5(a) and 5(b) with the c direction labeled is provided in Fig. 5(c). Figure 5(d) is a $3\ \mu\text{m}$ long profile across the ridges and valleys along the line in Fig. 5(a). The profile shows linear slopes at an angle φ_b

FIG. 4. (a) AFM height image and (b) MFM magnetic image of the yz face of micropillar 1. The scan encompasses most of the top area of the micropillar. The left and right edges of the scan leave out approximately $1\ \mu\text{m}$ of material on each side. The top edge of the scan shows the top end of the micropillar. Twins are visible on the upper section of both images. The boxed area is shown in higher magnification in Fig. 5.

FIG. 5. Detailed view of the boxed area in Fig. 4. (a) AFM height image of the surface relief and (b) MFM magnetic image. The strong contrast indicates out-of-plane magnetization, while the weak (neutral) contrast indicates in-plane magnetization. The direction of magnetization is parallel to the crystallographic c direction. Every ridge [bright line in (a)] and valley [dark line in (a)] matches with a change in magnetic pattern in (b). (c) A schematic of the MFM magnetic image where the c direction is labeled for each magnetic domain. (d) A $3\ \mu\text{m}$ profile of the surface with a surface relief angle φ_b between twins of $\varphi_b = 3.5^\circ \pm 0.2^\circ$. The position of the cross section (d) is marked with a white line in (a). The zero position of the cross section corresponds to the x on the white line.

$= 3.5^\circ \pm 0.2^\circ$. Each change in slope of the surface relief corresponds to a change in the magnetic image alternating between strong and weak contrasts. Dark and bright areas in the MFM image indicate that the c direction is perpendicular to the surface.^{31–35} Dark areas indicate that the magnetic moment is going into the surface while bright areas indicate that the magnetic moment points out of the surface. In the bands with soft (or neutral) contrast, the c direction is parallel to the sample surface as indicated with a white bar in Fig. 5(b).

Figure 6 shows the AFM height image (a) and the MFM magnetic image (b) of the yz face of micropillar 2. The surface relief is completely flat and consists of a single crystal-

FIG. 6. (a) AFM height image and (b) MFM magnetic image of the yz face of micropillar 2. The scan encompasses most of the micropillar. The left and right edges of the scan leave out approximately $1\ \mu\text{m}$ of material on each side. The top edge of the scan shows the top end of the micropillar. The surface is completely flat with no evidence of twinning.

FIG. 7. Schematic cross section of the ac twin of micropillar 1. The twin sequence is CAC (the letters indicate which crystallographic direction is perpendicular to the surface) and the surface relief angle is ϕ_b . From the left boundary marking a ridge, the right boundary marking a valley, and the intermediate twin being of type A follows that the twin boundaries are inclined to the lower left.

lographic domain with c direction normal to the surface, indicative of a twinless region. The magnetic domains form a maze pattern with 180° domain walls. The magnetic moments point out of the surface in bright areas and into the surface in dark areas.

IV. DISCUSSION

The stress needed to induce twinning in the micropillars is larger than measured for a bulk sample taken from the same monocrystal, which was only 1 MPa.^{36,37} This increase in stress may be partly due to gallium implantation during the fabrication of the micropillars with the Ga^+ ion beam.^{38–40} Depending on the accelerating voltage, Ga^+ ions can be found 10–20 nm from the surface at low voltages³⁸ and up to 1000 nm at high voltages.⁴⁰ Another possibility for the high twinning stress is a size effect for twinning induced plasticity similar to the dislocation nucleation controlled size effect encountered in micropillar studies of fcc metals.^{20,24} Recently, Dehm *et al.*⁴¹ observed that twinning in single crystal Au thin films scales inversely with film thickness, indicating that the stress to nucleate twins may also depend on the sample dimensions. Finally, flow stresses in micro-compression testing were found to depend on the lateral stiffness of the loading system showing higher stress values for lateral stiff systems like the one used in this study.^{42,43} Which of these effects actually contribute to the high twinning stress of the pillars remains to be clarified at this point.

The twinning mode occurring in micropillar 1 can be found from the alternation of the c direction between in plane and out of plane across twin boundaries and the surface relief angle of $3.5^\circ \pm 0.2^\circ$.³³ The alternating c direction disqualifies ab twinning in pseudo-orthorhombic (14M) martensite in this case because the c direction does not change across a boundary of ab twins. The angle of $3.5^\circ \pm 0.2^\circ$ narrows the twinning mode to ac twinning in pseudotetragonal (10M) martensite because the relief angles for ac and bc twinings in 14M martensite are 6.5° and 2.7° .³³ Figure 7 is a schematic cross section of the twins in Fig. 5(d) where the twin boundary direction and the twinning sequence is CAC (the letters indicate which crystallographic direction is perpendicular to the surface). Twin boundaries in 10M martensite correspond to the $\{110\}$ planes of the cubic austenite

FIG. 8. Side view schematic of ac twins in the micropillar 1 where there is a surface relief on the front and back sides of the micropillar and the twin boundaries (dashed lines) are angled downward and to the right. The sample before compression is shown with dotted lines.

phase. Therefore, the boundary must extend into the pillar on a plane which forms an angle close to 45° with the z direction (Fig. 7). From the analysis of the lattice directions and the positions of the relief ridges and valleys, it follows that the twinning planes are inclined downwards into the pillar. Figure 8 is a schematic cross section of micropillar 1 perpendicular to the y direction where the surface imaged with AFM/MFM is on the left. The twin boundary traces on the “back side” of the pillar are positioned approximately 10 μm below the traces on the front side. On the front surface, the twin traces are located about 5–10 μm below the top face of the pillar. Therefore, the twins do not enter the base material but extend fully across the micropillar and form traces on the back surface.

Dislocation plasticity has not yet been reported for Ni–Mn–Ga and is unlikely to occur because of the large Burgers vector of lattice dislocations. Therefore, the serration in the deformation curve of micropillar 2 (black arrow in Fig. 3) and the stress plateau indicate that deformation took place via twinning. The unloading curve shows almost complete strain recovery and the AFM results (Fig. 6) confirm that no twins remained in the pillar. Figure 9 shows what might have occurred in micropillar 2 during loading and unloading. The twins may have formed at lower heights than in micropillar 1. In particular, if the twins formed at a height slightly smaller than the pillar width yet high enough such that the twin travels a large distance through the pillar before extending into the base material, then twinning contributes significantly to deformation. However, due to the inclination of the twinning plane, the twins would not intersect with the surface of the micropillar on both yz surfaces. Rather, the twins may extend into the base material. Within the base material, the stress diminishes quickly. Therefore, the twins would not grow to large size but end closely below the bottom end of the micropillar. Upon unloading, the image force on the disconnections (or twinning dislocations) drives the disconnections out of the micropillar resulting in “detwinning.” This effect is called “pseudoelastic twinning” and was described by Garber (in Russian) and reviewed by Kosevich and Boiko.⁴⁴

FIG. 9. Side view schematic of micropillar 2 at maximum load before unloading. The micropillar deformed via twinning. The twin boundaries (dashed lines) start at the micropillar and extend into the bulk. The pillar before compression is shown with dotted lines.

Micropillar 1 exhibits a similar amount of strain recovery as micropillar 2. This strain is likely due to twinning in the lower part of the pillar. These twins respond pseudoelastically in the same way as those in micropillar 2. Thus, twinning may have occurred over most of the height of micropillar 1, but only those twins that extend fully across the pillar remained. For these twins, the disconnections escape through the side walls thereby eliminating the driving force for strain recovery.

It is currently not fully clear what is the origin of the permanent deformation of 0.7% of micropillar 2. There might be twins present which are not resolved with the AFM/MFM methods. Another possibility is that the micropillar was slightly punched into the bulk material. This might also be the case for micropillar 1, for which the detected amount of twins does not fully account for the permanent strain of 3.6%.

Within the strain range covered by both deformation experiments, the stress of micropillar 1 is about 50% higher than the stress of micropillar 2. Various causes may contribute to this variation. Nonuniformities in concentration are often due to crystal growth and may lead to significant variation in structure, transformation temperature, and thus in mechanical properties.^{45–47} Different amounts of nanotwins may have been present at the beginning of the deformation experiments. These nanotwins may have caused microplasticity, which would affect the slope of the linear region. Variations in process conditions during ion beam milling may lead to different levels of gallium implantation which will also affect the mechanical properties. Finally, the deformation process is inherently inhomogeneous, i.e., twinning occurs at discrete locations, not evenly spread over the entire sample. As discussed above, twinning might occur at different heights in the pillar which can result in permanent twinning if the twinning plane extends entirely through the micropillar

and in pseudoelastic twinning if the twinning plane extends into the base material. These variations affect not only recovery of twins but also the twinning stress and the hardening rate. To answer which of these phenomena effectively contributes to the variations in the mechanical behavior of the two micropillars is beyond the scope of this study and requires further work.

V. CONCLUSIONS

The conclusions of this study are as follows.

- Ni–Mn–Ga single-domain micropillars were prepared with dual beam FIB milling from a trained single crystal.
- The micropillars were deformed in uniaxial compression and the stress-strain curves were measured. The stress required to induce twin boundary motion is higher in FIB fabricated micropillars than in bulk crystals.
- Deformation in Ni–Mn–Ga micropillars occurs through crystallographic twinning. In one situation, the twinning mode was identified as *ac* twinning in pseudotetragonal 10M martensite.

Two pillars were tested whereof one pillar showed large permanent (i.e., plastic) deformation, whereas the other pillar showed almost completely recovered strain upon unloading. The permanent deformation is attributed to twinning where twins extend across the entire micropillar. Pseudoelastic deformation may be due to twinning where twins impinge from the pillar into the substrate and travel back to the surface upon unloading, leading to strain recovery. Though the twinning stress of the micropillars is significantly higher than that of similar bulk samples, the present results do not provide sufficient evidence for claiming a size effect. This would need a systematic variation in pillar dimensions and is beyond the scope of this study.

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