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Preparation of Ni-Mn-Ga micropillars using focused Xe-ion beam milling for magnetic actuation on microscale

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Abstract:
Magnetic shape memory alloys are promising candidates for actuation on microscale. We investigate the possibility of using focused Xe-ion (as an alternative to Ga-ion) beam milling (Xe-FIB) for microfabrication on Ni-Mn-Ga magnetic shape memory single crystals. We started with the most basic structures such as micropillars. Xe-FIB working conditions of 30 kV/100 nA results in large curtaining effect thus for micrometre sized structures a two-step milling procedure was used with the final polishing conditions of 30 kV/100 pA. Pillars down to 1 micrometre size were produced; preparation of 10 x 10 micrometres square-base pillar for magnetomechanical testing is illustrated in individual steps. By the combination of mechanical testing and optical microscopy we demonstrate that the Xe-FIB milling does not significantly change the ability of martensite twin boundaries to propagate through the fabricated pillars.

Keywords: ferromagnetic shape memory, focused ion beam, Ni-Mn-Ga, magnetic actuation, microscale fabrication

Introduction

Large energy density, fast and large actuation strain, and the possibility of remote operation by magnetic field make the Ni-Mn-Ga magnetic shape memory (MSM) alloys promising candidates for actuation on microscale [1-5]. Various micro-magneto-mechanical systems (MMMS) can be designed in analogy to micro-electro-mechanical systems (MEMS). There were a few successful demonstrations of thin foil MSM actuation [1-4], however the effort towards true microsystems using Ni-Mn-Ga has been less successful in the past. The fabricated microstructures such as micropillars either could not actuate due to large twinning stress [4] or actuation was not reversible or was far from theoretical uniaxial strain [1]. The main reason may be that the typical microfabrication technology was focused Ga-ion beam (Ga-FIB) milling technique. Due to deep penetration of Ga-ions into material and sensitivity of the Ni-Mn-Ga alloy to composition and defects the material is highly influenced by the Ga-ion milling. Consequently the actuating-relevant properties, such as very low twinning stress, deteriorate.

Xe-FIB milling is an alternative technique for microfabrication. Its advantages are lower ion penetration depth, much higher milling rate compared to Ga ions (due to the higher currents) and the fact that the implanted Xe is inert in the material [6-9]. In our work we investigate the possibility of microfabrication using Xe-FIB milling on Ni-Mn-Ga MSM single crystals. We start with the microfabrication of the most basic structures such as micropillars.

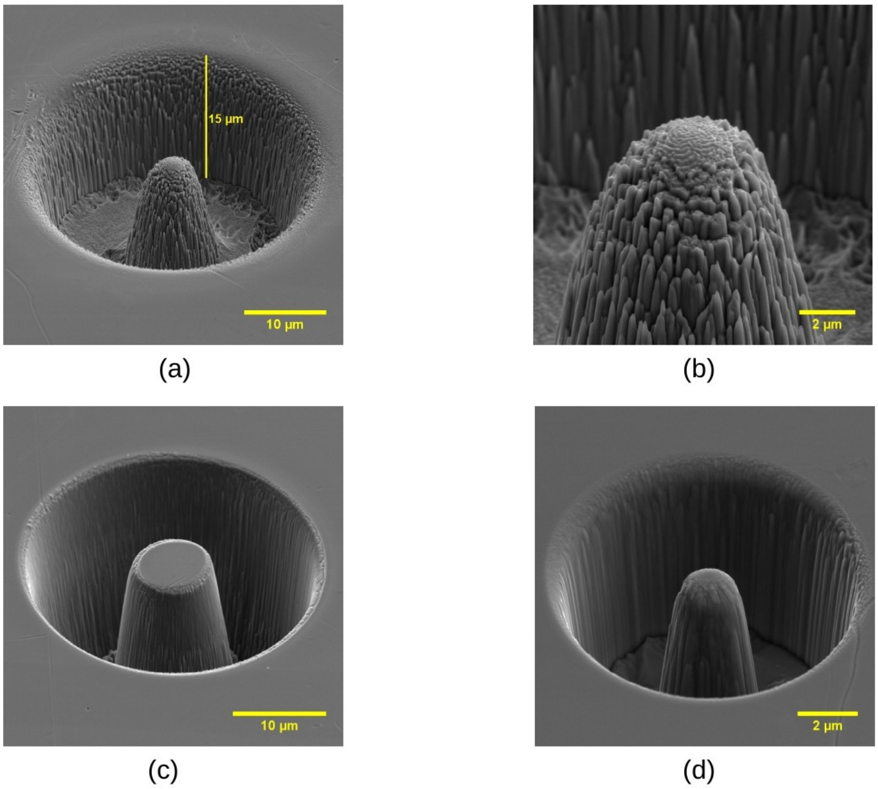
Experiment

The Xe-FIB milling was performed on {100} oriented $Ni_{50}Mn_{28}Ga_{22}$ single crystals using fully integrated Xe plasma source FIB with scanning electron microscope (FIB-SEM) TESCAN FERA3 GM. Prior microfabrication of pillars, a set of testing objects with various FIB working conditions was milled. After these initial experiments we selected 30 kV/1 µA to 300 nA as optimum setting for initial milling, and for final polishing the optimum setting was 30 kV/30 nA.

SEM observations were done using secondary electrons imaging (Everhart-Thornley SE detector). The polarized-light optical observations were made using ZEISS Axio Imager Z1m light optical microscope. Uniaxial compression to induce the motion of martensite twin boundaries was applied using in house-made loading device with screw-based loading mechanism.

Results

Pillar of about 2.5 micrometres in the top diameter and height of 15 µm produced using 30 kV/100 nA working conditions is shown in Fig. 1a. Detailed look in Fig. 1b reveals a significant curtaining effect. Due to its small scale, the curtaining might be no issue for larger structures (~ 100 micrometres) and can be reduced by lowering the FIB working current. That of course goes on the cost of milling speed and thus a balance must be always found between the milling speed and surface quality.



The effect of lowered FIB working current can be

Fig. 1. a) Pillar d_{top} = 2.5 µm fabricated with 30 kV/100 nA ion beam working conditions, b) detail of (a), c) pillar d_{top} = 7.5 µm fabricated with 30 kV/100 pA ion beam working conditions, d) pillar d_{top} = 1 µm fabricated with 30 kV/100 pA ion beam working conditions.

seen in Fig. 1 c, d, where pillars of the top diameter d_{top} = 7.5 µm (height 20 µm) and d_{top} = 1 µm (height 5.5 µm) were fabricated using 30 kV/100 pA working conditions. The d_{top} = 1 µm pillar simultaneously illustrates that structures down to micrometre scales can be produced in our setup.

Apart of the artefacts such as curtaining, the FIB milling is known to produce two types of surface damage: i) surface amorphization ii) point defects in the material [6-9]. Xe-ions are known to penetrate the material less than Ga-ions and another advantage of Xe is that it is inert. The basic test of surface damage was made by observation of interaction of martensite twin boundaries with the microfabricated pillars.

The polarized-light microscopy observation of martensite twin boundaries propagating through the array of micropillars of various sizes is shown in Fig. 2. The propagation of twin boundaries is clearly seen in pillars of intermediate size (>10 micrometres) and in pillars of “large” diameters (100 micrometres). The boundaries propagating in the pillars follow those observed in the bulk. Such an easy propagation of the boundaries through the pillars indicates that the surface stress due to the ion damage must be small, presumably less than nucleation stress (~3 MPa).

In other words the surface damage due to the Xe-FIB milling does not seem to be large since i) the twin boundaries can be actually observed in pillars and FIB-milled surfaces using polarized light, ii) the twin boundaries easily propagate through the pillars. However, tests in magnetic field show some influence and removing the FIB-affected surface e.g. by carefully controlled electroetching may be critical for successful actuation in magnetic field [10, 11].

Example of microfabrication of 10 µm x 10 µm x 50 µm square-base pillar for testing in magnetic field is shown in individual steps in Fig. 3. First the pillar was FIB-milled (300 nA) to rough dimensions, Fig. 3a, b. It exhibited a wedged shape (taper angle) due to the Gaussian profile of the ion beam and redeposition effect. The wedged shape pillar was further polished from front, back, left and right side using smaller (30 nA) probe current in order to remove the taper angle effect and acquire curtain-free sides. This “side-polishing” was processed in steps (each surface at a time) with different position and rotation of the sample in relation to the ion beam.

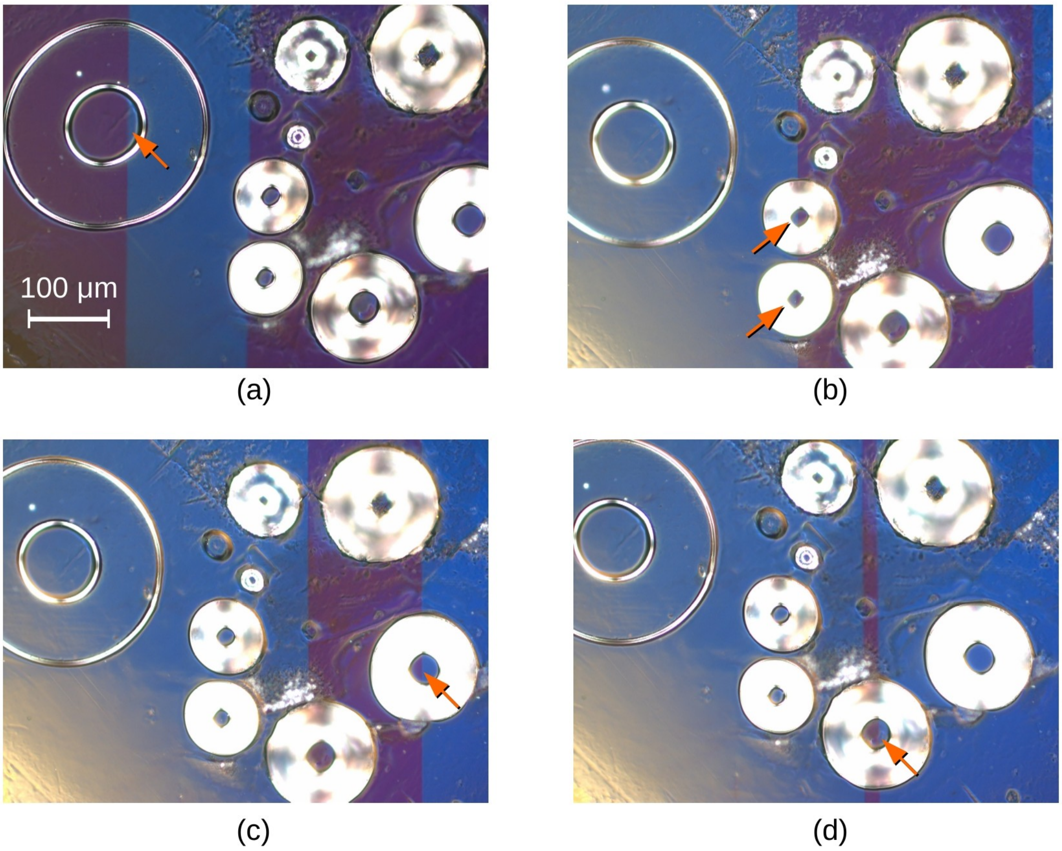


Fig. 2. Polarized light microscopy on array of pillars in sample loaded in uniaxial compression. Initial state is in (a), gradual propagation of twin bounadaries in (b), (c), final state in (d). Pillars with just transversing twin boundaries are marked by red arrows.

The pillar geometry was nearly cuboid one after the polishing of sides, Fig 3c. The final step was polishing of the pillar top (10 nA), Fig. 3d. Top and overall view of the final pillar is shown in Fig. 3e and f, respectively. For magnetomechanical testing of microfabricated pillars, see Refs. [10] and [11].

Conclusion

We found the following advantages of using the focused Xe-ion beam milling for fabrication of micropillars:

i) both pillars with size of hundreds micrometres as well as small micrometre-sized pillars can be prepared in the same setup.

ii) the surface damage introduced by Xe-ions in not large as confirmed by the free propagation of twin boundaries through the Xe-FIB milled surfaces.

iii) the microfabricated pillars actuate with strain close to theoretical 6% value as described in the continuing works [10, 11].

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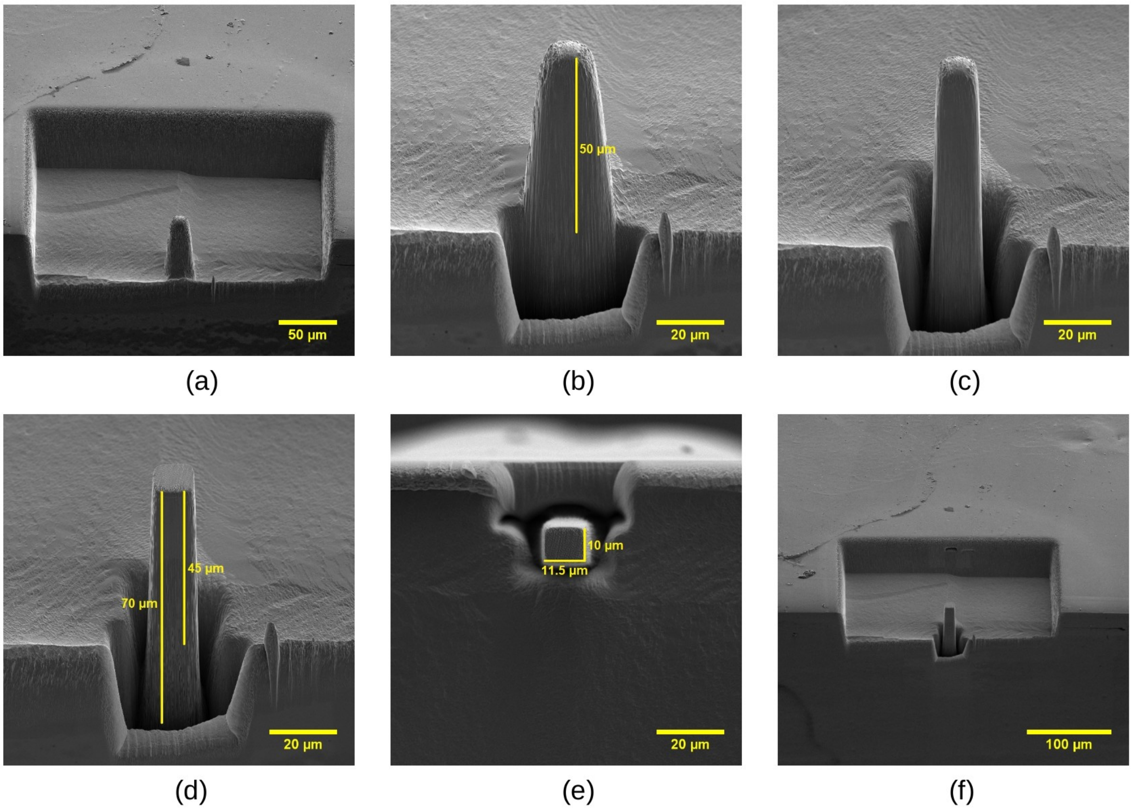

Fig. 3. Xe-FIB microfabrication of 10 μm x 10 μm x 50 μm square-base pillar in individual steps: a) Initial “rough” milling with 30 kV/300 nA ion beam working conditions, b) the detail of (a) after additional front side milling (30 kV/30 nA), c) the pillar after fine polishing of front, back, left and right side (30 kV/30 nA), d) the pillar after following fine polishing (30 kV/10 nA) of its top side, e) top view of the final pillar, f) overall view of the final pillar.

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