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Regular Article

Investigation of the deformation behavior of aluminum micropillars produced by focused ion beam machining using Ga and Xe ions

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ABSTRACT

Sample geometries for micro-mechanical testing, e.g. micro-pillars and micro-cantilevers are primarily produced using gallium focused ion beam technology. However, the effects of the gallium ions on the mechanical properties of metals which are embrittled by liquid metal gallium are still unknown. In this work, micro-compression samples from single crystalline and ultrafine-grained aluminum are fabricated using both xenon and/or gallium ions. The different ions have little effect on the yield strength of single crystalline aluminum. However, for the ultrafine-grained aluminum, the strength is reduced with increasing Ga dose, and considerable differences in the deformation morphology and resulting microstructures are observed.

Micro-mechanical testing of structures produced using focused ion beam (FIB) machining has enabled a large number of test geometries (e.g. pillars and tension dog-bones for plasticity, and cantilevers for fracture properties) for investigating the properties of materials at small length scales. Micro-compression testing of pillars [1] is perhaps the most prominent of these techniques. This approach has been intensively used in recent years, and size effects have been explored in great detail. From this work, it has become apparent that the mechanical properties can be strongly influenced by the preparation method altering the microstructure of the sample: Shim et al. showed that the strength of molybdenum pillars prepared without FIB milling was nearly ten times higher than those prepared using FIB [2].

One issue related to FIB-prepared samples is that they are exposed to highly accelerated Ga ions which create irradiation damage at the surface. This is known to create several different types of microstructural damage to the materials: surface amorphization [3], creation of point defects [4], increased dislocation density [5], and the formation of intermetallic compounds at the surface [6]. However, the possibility of micro-scale liquid metal embrittlement by implanted Ga ions has largely been ignored, due to the lack of an alternative fabrication method for comparison.

The list of metals known to be embrittled by liquid gallium – Ag, Al, Cd, Cu, Fe, Sn & Zn [7] - contains several industrially important metals frequently investigated using FIB-machined samples, so the significance of this possible effect on mechanical properties warrants investigation. The Al—Ga system is a particularly remarkable example of liquid metal embrittlement [8]. Ga embrittles aluminum by rapidly segregating to grain boundaries, where it changes their atomic structure and weakens their bonding [9]. Using in situ transmission electron microscopy (TEM), Hugo et al. [10] found that the Ga penetration front in pure aluminum appeared as a line, which can interact with the grain boundary (GB) dislocations in order to alter its speed and profile. The grain boundary mobility is enhanced by even minor Ga additions [11]. Moreover, Mehrnoosh et al. [12] observed the formation of a new grain at a GB by TEM as a result of liquid Ga penetration. Since both of these effects should be strongly present in ultrafine-grained (UFG) aluminum, it is expected to be ideal test case for studying the mechanical properties of the specimens are affected by Ga-FIB machining.

To investigate this, aluminum samples with a range of grain sizes have been chosen, to study the embrittlement influence on mechanical properties of different microstructures. A ⟨100⟩-oriented single crystalline (SC) aluminum sample (Purity: >99.99%) was acquired from MTI Corporation (Richmond, CA, US). Polycrystalline UFG aluminum was produced at FAU Erlangen by equal channel angular pressing (ECAP), as described by May et al. [13]. This UFG material was then heated treated to produce grain sizes from 1000 nm to 2800 nm. All of the sample surfaces were conventionally prepared using standard metallographic

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techniques followed by electrolytic polishing and ion milling. The grain sizes of all the UFG aluminum samples were characterized with an electron backscatter diffraction (EBSD) technique using a FEI Quanta 200 scanning electron microscope (SEM).

Micropillars were fabricated using a Fera Xe plasma FIB and Lyra Ga FIB made by Tescan a.s. (Brno, Czech Republic). The pillars had an aspect ratio of ~3 and a target diameter of 7 μm. This size was chosen as the compromise between the sputtering rates of the two FIBs. Fluorine gas injection was applied in the Xe plasma FIB in all cases to achieve more uniform milling during the coarse and fine milling unless otherwise specified. In order to explore the degree of Ga dose influence, three different milling procedures were used to manufacture the pillars: Xenon ions only, Xe-Ga (Xenon for coarse milling, Gallium for polishing) and Gallium ions only. Initially, probe currents of 100 nA (Xe FIB) and 6.4 nA (Ga FIB - roughly equivalent to 5600 μm^3 of Ga after 2 hours coarse milling) were used to mill the rough pillar shapes. Afterwards, low current polishing was used to obtain smooth surfaces with currents ranging from 100 pA to 1 nA (both Xe and Ga FIBs). A minimum of three micropillars were manufactured using each milling method.

These pillars were then compressed using a SEM indenter system [14] (Alemnis GmbH, Thun, Switzerland) in situ in either a Zeiss DSM 962 or a Tescan Vega 3 SEM. A diamond flat punch tip with a 15 μm diameter was used. All pillars were compressed at a constant displacement rate of $2 \times 10^{-3}\ s^{-1}$ to ~10% strain. The engineering stress was calculated using the top surface area of the pillar. The yield strength, the stress at which the pillar begins to deform plastically, was determined from the onset of general yielding to account for variation in pillar geometry (apparent strain hardening due to taper) to evaluate the mechanical properties of the material. High resolution SEM images of the deformed micropillars were obtained using the Tescan Fera Xe FIB system.

Fig. 1 shows representative engineering stress-strain curves from SC and UFG Al micropillars produced using only Xe or Ga ions. All of the stress-strain curves show a nearly elastic loading behavior followed by apparent strain hardening in the plastic deformation process due to the pillars' taper. Close inspection of the flow curves in the plastic deformation regime reveals that an intermittent serrated flow behavior is observed in all the curves. This is more pronounced in the single crystal due to the continuous generation of new dislocations, since the dislocation density is limited by surface annihilation [15]. From Fig. 1, it can be seen that the yield stress increases with decreasing grain size in accordance with the Hall-Petch effect. Comparing pillars produced by different ions, the Xe pillars show significantly higher yield strength and slightly more apparent strain hardening than the corresponding curves recorded for pillars prepared with Ga. The higher apparent strain hardening observed in stress-strain curves of Xe pillars is attributed to the larger taper angle (~5°) resulting from the larger spot size of the Xe FIB. This is significantly larger than the typical taper (~2°) of pillars made using Ga FIBs.

Fig. 2 shows the variation in yield strength between the three different FIB techniques on the various aluminum samples. In the case of the UFG polycrystalline aluminum, all the Xe pillars show the highest yield strength while the Ga pillars show the lowest. It appears that FIB milling with Ga ions leads to a reduction of the mechanical strength with the increasing of Ga implantation dose. Furthermore, the magnitude of this reduction in strength appears to increase with decreasing grain size (or increasing number of grain boundaries).

In the case of the SC pillars, any effect of the different ion species appears less significant – Fig. 2. In contrast to the polycrystalline results, for the SC Al, it is seen that the pillars made by Ga FIB are slightly stronger – possibly indicating that Ga ions introduce more point defects and dislocation loops than Xe ions for similar ion energies. However, the possible effect of the fluorine gas injection on the deformation of the Xe-prepared pillars was also investigated. It is shown in Fig. 2 that the injection of fluorine gas has slightly lowered the yield strength of single crystal aluminum pillars. This suggests that the difference in strength between the Xe and Ga SC Al pillars might be due to the fluorine gas injection during milling, which may modify the pillar surface and affect dislocation nucleation sites. Since the fluorine gas prevents redeposition of aluminum, this may provide a more pristine surface which aids dislocation nucleation.

To consider the possible mechanisms that might result in the variation in strength between the different FIB methods, it is instructive to examine the morphology of the deformation of the pillars. Fig. 3 shows high resolution secondary electron (SE) micrographs of the salient features of the deformed micropillars of SC and UFG aluminum fabricated using the two extreme cases: pure Xe FIB and pure Ga FIB. For the ⟨100⟩-oriented SC Al, the morphology of the deformed pillars is comparable: slip offsets consistent with the $\{111\}\langle 1\bar{1}0\rangle$ slip system are clearly observed in all samples. The black spots observed on SC Ga pillar are probably due to contamination.

However, the UFG Al samples show considerable differences in the deformation morphology. For all grain sizes, stronger slip markings can be observed on the surfaces of the Xe pillars than on the Ga pillars. Instead, in the Al pillars produced using Ga, the deformed grains protrude from the pillar surface, and increasing the gallium dose appears to result in more grains “popping out” during deformation. This was also observed in situ during deformation. The Ga ions appear to weaken

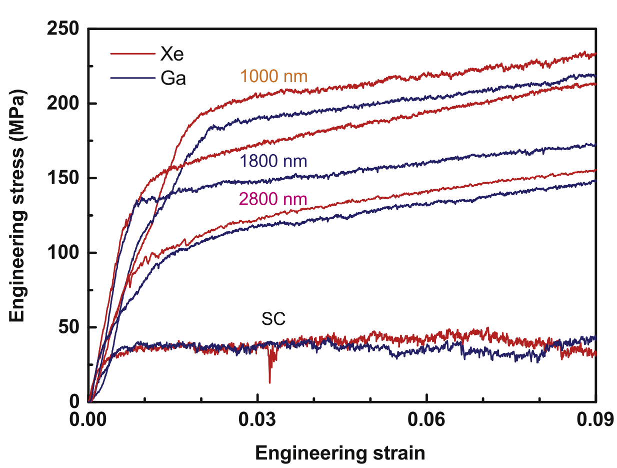

Fig. 1. Representative stress-strain curves from SC and UFG Al micropillars with grain sizes ranging from 1000 nm to 2800 nm produced by Xe and Ga FIB techniques.

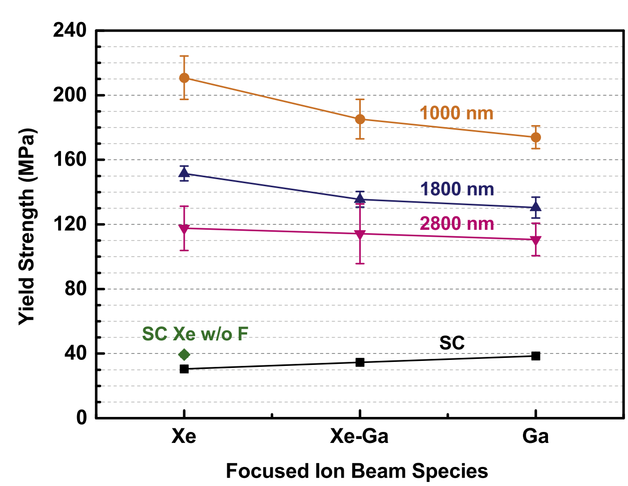

Fig. 2. The average yield strength of SC and UFG Al fabricated by three FIB techniques: Xe, Xe-Ga (Xenon for coarse milling, Gallium for polishing) and Ga; the green diamond shows the yield strength of Xe produced pillars without fluorine gas injection (N.B. the error bars for SC pillars lay inside the symbols: ±2 MPa). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

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