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In situ Laue diffraction of metallic micropillars

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ABSTRACT

Laue micro-diffraction performed on metallic micropillars prior to deformation revealed the presence of strain gradients and planar defects in samples made by focused ion beam (FIB) milling. In situ Laue micro-diffraction shows that such pre-existing gradients can play a role in the determination of the first activated slip system, and thus leading to un-expected geometrical strengthening. Lattice rotations resulting in the formation of substructures are observed at stresses well below the strength of the pillars usually defined as the stress at 5% strain.

1. Introduction

Size effects in plasticity resulting from the reduction in specimen size have received much attention in recent years since conventional theories do not explicitly incorporate geometrical length-scale dependencies. In order to circumvent the coupled effects of strain gradients, as are encountered in nano-indentation and wafer curvature testing, uni-axial micro-compression testing is a technique to study the effects of geometric size [1–4]. In these experiments pillars with diameters of the order of tens of microns down to 100 nm are compressed using a nano-indenter outfitted with a flat punch indenter. The results show a general trend of increasing strength with decreasing diameters. Rationale for such behavior is greatly debated, with emerging theories including dislocation exhaustion and stochastic, scale-free dislocation mechanisms [5–7]. Micropillars are typically fabricated using focused ion beam (FIB) machining from a thin film or bulk specimen. It is well-known that in metals FIB causes damage due to the Ga implantation [8]. It is often assumed that such damage is restricted to a small surface region and that the FIB procedure does not alter the mechanical properties [5,9,10], whereas other propose models for FIB induced hardening [11,12]. Computational simulations are currently being carried out in order to explore the origin of the observed geometric length scale size effect [13–15]. Up till now only macroscopic mechanical data and SEM observations are available as experimental input parameters for such simulations whereas very little is known about the evolving microstructure during compression. Furthermore it is assumed that the geometrical boundary conditions of the testing technique do not promote self-organization and multiplication of dislocations [5,16,17], which for example would lead to crystal rotation. To shed light on the initial microstructure and on possible FIB damage we have performed ex-situ Laue micro-diffraction on Si pillars made both by FIB milling and by deep reactive ion etching. We furthermore present an in situ time resolved Laue micro-diffraction experiment that captures the changes in microstructure during deformation of Au micropillars [18]. The dynamics of the Laue patterns show that the initial strengthening seen in the smaller pillars can be explained by plasticity starting on a slip system that is geometrically not predicted but selected because of the character of pre-existing strain gradients within the sample. Moreover as the plasticity proceeds, significant rotation of the crystal is observed, which implies strain hardening that is also suggested by the evolving peak topologies.

2. Experimental setup

The presented experiments were done with a custom designed in situ micro-compression device (MCD) [18] at the MicroXAS beam line of the Swiss Light Source (SLS) (see Fig. 1a). Micro-compression and white beam micro-focused X-ray diffraction are combined to probe the microstructure as a function of deformation strain. Force and displacement are measured with a single axis 1D Triboscope transducer from Hysitron Inc. The positioning of the flat punch compression tip above the sample is performed with the help of two high resolution optical microscopes positioned in two orthogonal view axes that are perpendicular to the compression axis. A smooth touchdown procedure is carried out by a sub-nm resolution piezo stage that stops the approach once a given transducer displacement

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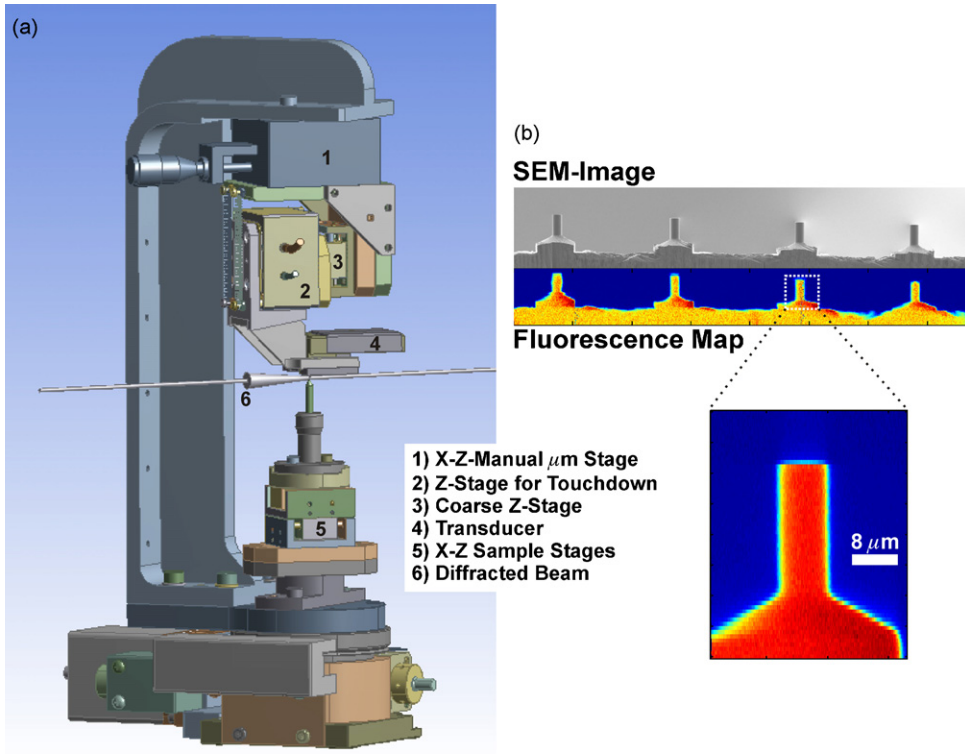

Fig. 1. (a) Schematic of the in situ micro-compression device (MCD), (b) four 8 μm metallic micropillars imaged by scanning electron microscopy (SEM) and by X-ray fluorescence. The inset shows the fluorescence signal from one pillar which was scanned with a step size of 300 nm.

threshold is attained. The results presented here are obtained in a load controlled mode with loading rates of 1–3 μN/s and a single crystal diamond flat punch tip with an end diameter of 22 μm. The samples were illuminated in transmission geometry with a polychromatic X-ray beam with an energy distribution ranging from 2 to 22 keV. The X-ray beam is focused with a set of Kirckpatrik–Baez mirrors, yielding a beam size of typically 0.8–2 μm in the focal plane and an angular divergence of 0.2 mrad. The samples are positioned with sub-micron precision in the X-ray beam using an X-ray fluorescence detector. This is demonstrated in Fig. 1b, which presents a row of Ni micropillars with a diameter of 8 μm visualized with X-ray fluorescence in comparison with a conventional scanning electron microscopy (SEM) image. The inset of Fig. 1b demonstrates the high precision that can be achieved using this technique. Diffracted X-rays are recorded on a two-dimensional Photonic Science FDI-VHR 150 charged coupled device (CCD) with a pixel size of 31 μm. The detector parameters such as tilt and sample-to-detector distance are calibrated using a least-square refinement of a Laue pattern from a strain-free single crystal Si wafer. The angular width of Si diffraction spots is about 0.06°, which is a good measure for the experimental resolution of the complete setup. Recorded Laue patterns were indexed using the triplet method, as described by Tamura et al. [19]. This allows determining the crystallographic orientation of the probed volume with high accuracy. During in situ testing the diffraction peaks evidence peak asymmetries, collective peak movements and peak splitting. Peak asymmetries are due to lattice curvatures; e.g. strain gradients and provide therefore information about the perfection of the single crystal. Statistically stored dislocations (SSD) results in symmetric peak broadening; an initial content of geometrically necessary dislocations (GND) cause a lattice curvature resulting in peak streaking [20]. Peak streaking is evidenced by an asymmetric peak shape that has a maximum (major) and minimum (minor) peak width. Collective changes in peak positions are a signature of lattice rotations. A peak split is a signature of a plastically bent portion of the crystal, which has broken up into smaller disoriented sub-volumes that each diffract with slightly different angles. The misorientation angle can easily be calculated from the Laue peaks and the diffraction geometry.

This paper reports first on examples of initial defects found in focused ion beam (FIB) machined micropillars, followed by a section that relates the pre-existing defects to the plastic response. Finally an example is given of a micropillar with no significant initial defects. It is shown that such a pillar in general deforms according to the slip system with the highest critically resolved shear stress.

3. Results

3.1. Pre-existing defects in micropillars

Single crystal Si micropillars (1 and 2.7 μm) having a ⟨0 0 1⟩-orientation were either produced by FIB (2.7 μm) or by a special etching technique (1 μm) [21]. Final Ga^+ currents of 50 pA where used to prepare the FIB-synthesized samples. Using Pt markers below the Si pillars the samples could be located. The Laue patterns of the Si pillars show mainly reflections from the {3 3 1}, {1 1 3} and {2 4 2}-families of lattice planes for an incoming beam parallel to a ⟨0 1 1⟩-direction. Fig. 2 summarizes the results for the Si pillars. For the etched pillar a Laue pattern with an inset of the SEM image (a) is shown together with a truncated contour plot (b) and a 3D intensity profile (c) of the (1 −3 3)-reflection. All reflections of the etched pillar show peak shapes similar to the peak profiles of a Si wafer reference sample. For the FIB-Si pillar an SEM picture (d) and the contour plots of the (1 −3 3) (e) and the (1 3 3)-reflection (f) are shown together with a 3D profile of the (1 −3 3)-peak (g). Laue spot streaking was observed for all recorded reflections of the FIB-prepared pillar. The derived streaking direction can be interpreted