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Effects of focused ion beam milling and pre-straining on the microstructure of directionally solidified molybdenum pillars: A Laue diffraction analysis

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White beam Laue micro-diffraction was performed on directionally solidified, single-crystal Mo pillars in the as-grown state, after focused ion beam (FIB) milling and after pre-straining. The Laue diffraction peaks from the as-grown pillars are very sharp and show no broadening, similar to those from single-crystal Si wafers. Significant broadening and streaking of the peaks occurred after FIB milling and pre-straining, indicative of the damage these treatments induce in the nearly perfect crystal structure of the directionally solidified Mo pillars.

Keywords: Focused ion beam; X-ray diffraction; Single-crystal; Micropillars; Directional solidification

Specimen size effects on mechanical properties, e.g., smaller is stronger, have attracted much attention since Uchic et al. [1] introduced the technique of focused ion beam (FIB) milling to fabricate micropillars, followed by compression in a nanoindentation system to obtain stress–strain curves as a function of pillar size. Recently, an alternative technique to produce and test micropillars was introduced by Bei et al. [2], who utilized a directionally solidified (DS) NiAl–Mo eutectic grown at different rates to obtain Mo fibers of various sizes embedded in a NiAl matrix [3], followed by etching of the matrix to expose Mo pillars for compression. The DS pillars were found to yield at stresses close to the theoretical strength independent of pillar size (in the range 350–1000 nm) [2], reminiscent of whisker behavior [4]. When the DS pillars were pre-strained before compression, their strengths decreased dramatically and the stress–strain curves became stochastic and exhibited a size dependence [5]. A substantial decrease in the strengths of the DS pillars was also observed after FIB milling [6]. Since both pre-straining and FIB milling are expected to introduce defects (e.g., dislocations) in the initially defect-free DS pillars, their characterization is an important first step in understanding effects on mechanical properties.

White beam Laue micro-diffraction has proven to be a powerful technique to evaluate the defect structure of micropillars. When performed in situ, it allows the observation of microstructural evolution during compression [7–9]. In the case of FIB-milled pillars, even before deformation starts, the diffraction peaks often show broadening which, especially in the smaller pillars, can be of relatively high intensity [10]. Such broadening is indicative of deviatoric strains [11], which can be ascribed to elastic strain gradients or orientation differences (rotations) in the probed volume. Moreover, defects such as a small-angle grain boundary [12], a twin, or misorientations between pillar base and body have been revealed in some of the FIB-milled pillars prior to deformation [10], as well as surface nanograins in single-crystal nanoporous gold pillars [13]. In contrast, Laue diffraction of the DS Mo pillars showed no broadening [14]. Energy scans in combination with a differential aperture technique (backscatter mode) revealed the presence of elastic strains in the embedded Mo fibers which were relaxed when the NiAl matrix was etched to expose Mo pillars [14]. These results were obtained by analyzing individual diffraction spots from different DS pillars in a forest of Mo pillars.

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Here, we report that white beam Laue micro-diffraction (transmission mode) can be performed on single, exposed, DS Mo pillars in the as-grown (0% pre-strained), at 2% and 11% pre-strained conditions, as well as in the DS plus FIB-milled condition. The NiAl–Mo eutectic was directionally solidified and pre-strained as described previously [5]. All fibers had an axial direction parallel to the [1 0 0] crystallographic axis. To obtain single pillars for analysis from a forest of DS pillars, a special specimen preparation procedure was developed which will be briefly described here.

Thin (~300 μm) disks were cut from the DS and pre-strained materials by electro-discharge machining, and mechanically ground and polished to a wedge shape. At the thin end its thickness was ~20–50 μm, and consisted of ~10–30 rows of ~1 μm fibers. Within this region, islands containing 7 fibers in a hexagonal pattern (Fig. 1a) were obtained by removing the surrounding material by FIB milling. Next the NiAl matrix was etched, exposing 7 pillars in each island with aspect ratios of ~2.5–3.0 (Fig. 1b). To free the central pillar, the 6 pillars surrounding the central one were bent with a micro-manipulator in a high-resolution scanning electron microscope (SEM), leaving behind isolated exposed pillars (Fig. 1c and d). The operations used for getting a wedge shape introduced uncertainties concerning the crystal orientation of the sample. Therefore as a final step, the sample was aligned in the SEM so that the pillar axis corresponds with the vertical axis. This entire procedure was repeated on multiple wedges to produce for analysis 13 pillars of the as-grown eutectic, 2 pillars after 2% pre-strain (2%-P1 and 2%-P2) and 3 pillars after 11% pre-strain (11%-P1, 11%-P2, and 11%-P3). Additionally, two of the free-standing as-grown pillars were FIB-milled down from ~1 μm to ~560 and 680 nm diameter (FIB-P1 and FIB-P2), resulting in a slightly tapered geometry (Fig. 1e). For comparison, three more pillars were produced directly from the wedge shapes by FIB milling without any etching (FIB-P3, FIB-P4 and FIB-P5), reducing the Mo fiber diameter from ~1.1 μm to 820, 930 and 700 nm, respectively. For those three pillars the final aligning step before FIB milling could not be realized, as the fibers were still embedded in the matrix. This resulted in a misalignment between the FIB beam and the fiber longitudinal axis (crystallographic (1 0 0) axis) of about 10°. The Mo crystal orientation was measured to be close to the [6 1 0] direction for all three pillars. A 30 keV gallium ion beam was used for the FIB milling, with the following sequential step-downs in current: 6500, 700, 300, 40, and 10 pA. These parameters are similar to those typically reported in the literature and summarized in Ref. [6].

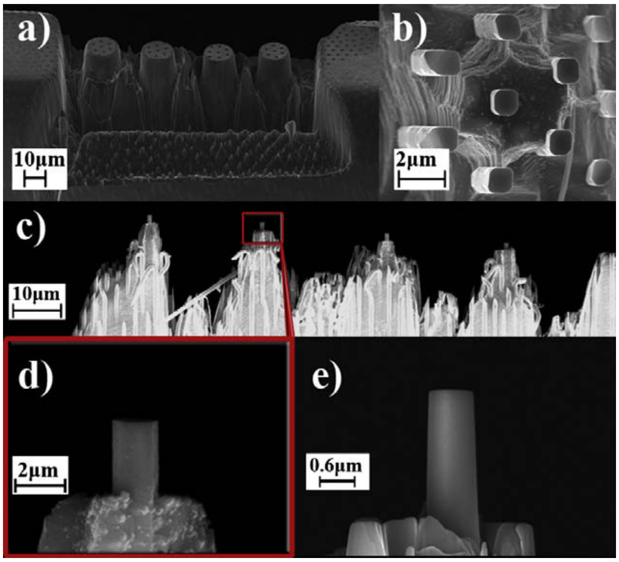

Figure 1. SEM pictures of (a) sample containing 4 FIB-milled islands; (b) top view of one of the island containing hexagonal arrangement of seven Mo fibers after etching the NiAl matrix; (c) side view of exposed Mo pillars after the surrounding six pillars were bent with a micro-manipulator; (d) magnified view of highlighted pillar in (c); and (e) magnified side view of a FIB-milled pillar.

White beam Laue micro-diffraction was performed in transmission at the MicroXAS beam line of the Swiss Light Source with a micro-focused X-ray beam having an energy distribution ranging from 5 to 23 keV and a beam size less than 1 μm in the focal plane. Further details of the experimental technique are given in the online material of Ref. [7]. Depending on the pillar orientation relative to the direction of the incident beam, the Laue diffraction patterns contained 1–6 diffraction spots with sufficient intensity for peak shape analysis.

In order to locate the pillars, two-dimensional (2D) spatially resolved measurements were performed with a step size of 300 nm. Figure 2a shows the shape of a (0 1 −1) peak from one of the DS Mo pillars. For comparison, the (−3 −1 −1) intensity obtained from a 100-μm thick single-crystal Si wafer measured under the same conditions is shown.

For the Mo pillar, the approximate full-widths at half-maximum (FWHM), defined in units of radial and azimuthal angles, $\Delta 2\theta$ and $\Delta\psi$, are 0.04 and 0.05°, respectively. In this study the radial angle is defined as the angle between the scattering vector and the direct beam, and the azimuthal angle corresponds to the angle between the projection of the scattering vector onto the charge coupled device (CCD) detector plane and the horizontal axis of the CCD plane. Note that these definitions are different from the one used in Ref. [11]. The corresponding values for the Si wafer are 0.06 and 0.06°, respectively. In other words the peak width obtained for the Mo pillars is within the resolution of the experimen-

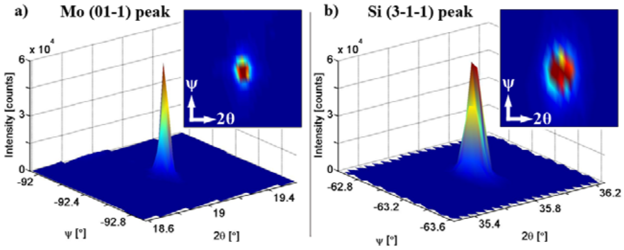

Figure 2. Typical shapes (3D and 2D) of Laue diffraction peaks from (a) DS Mo pillar, and (b) single-crystal Si wafer (100-μm thick). Both peaks are plotted in units of radial (2θ) and azimuthal (ψ) angles. In the 2D plots, the dark red color represents the intensity at half-maximum.