In-situ TEM deformation of aluminium nanopillars

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Abstract. We demonstrate here that compressive and cyclic loads can be applied to individual nanostructures enabling the observation of nanoscale deformation and fatigue. We demonstrate the capability of the new NanoLAB cyclic nanotesting instrument with nanoscale mechanical testing of individual Al nanopillars with diameters of less than 350nm. The thin Al nanopillars are seen to deform via a buckling mode where single crystal Al is turned into polycrystalline Al.

1. Introduction
Following the recent trend of shrinking dimension sizes for mechanical devices such as MEMS/NEMS (micro/nano-electromechanical systems), it has been clearly shown that mechanical properties of nanostructures and nanostructured materials vary considerably from their bulk counterparts. These size-scale effects have shown that as the dimensions of an object decrease to the micron-scale and below, they have increased flow stresses and increased strength [1-7]. It is thought that both the severely limited volume and the increased surface area to volume ratio have a significant effect on the dislocation generation and propagation mechanisms which can operate.

One of the major problems in testing individual nano-objects’ mechanical properties is that nanoscale positioning is essential, linked with a method of observing and measuring their response in-situ during a test. Novel scanning probe instruments have been developed which allow a range of mechanical testing methods to be implemented in-situ in both SEM and TEM [8, 9]. Our NanoLAB instrument is based on a generic piezoelectric positioner which can be adapted with the addition of various nanotools [10]. In this work, a series of nanoscale Al pillars have been repeatedly indented in real-time in-situ in a TEM, with the aim to increase our knowledge of deformation mechanisms in aluminium single crystal nanopillars. The repeated indentation of these pillars allows a large amount of deformation to be applied to the nanopillars whilst allowing the microstructure to rearrange itself during and between indentations in order to relieve stress.

2. Experimental
Large crystals of pure aluminium (99.999%) were grown by cold rolling and a heat treatment. These samples were thinned down and electrochemically etched, in a solution of Perchloric acid and Ethanol in a 1:4 ratio, to give a finely polished edge to the samples. On this edge, figure 1(a), pillars with diameters of less than 350nm were machined using a Focused Ion Beam (FIB) microscope. The heights of the pillars were between 1 and 2µm, giving aspect ratios between 1:3 and 1:6, figure 1(b). A conductive flat punch diamond indenter was used to uniaxially compress the pillars in a 300kV JEOL 3010 microscope. A NanoLAB piezo-driven indentation drive was used to apply a local load which
has sub-nm positioning movement. Software developed for use with the indenter allows adjustment of
the direction of travel to give the correct axial alignment. A calibrated force sensor [10] was used
during the indentations and a tracking programme was used to determine the displacements from the
recorded video.

3. Mechanical Compression of Al Nanopillars

A 350nm diameter aluminium pillar was axially compressed by displacement controlled indentation of
580nm at a speed of approximately 15nms⁻¹ (figure 2(a)). The pillar was unloaded, and then two
subsequent compression cycles of applied displacement 470nm and 570nm were carried out (figures
2(b, c)). The aluminium nanopillar is observed in-situ to undergo severe mechanical deformation.
Figure 2(d) shows the load-displacement curves for the three cycles of indentation. The third cycle
shows three significant jumps in load (I, II, III), due to large changes in the microstructure of the
nanopillar under load. This can be seen in figure 3(a) to (f), which was recorded in-situ during the
compression test.

During indent 1, whilst approaching a load of 120µN, the pillar started to buckle away from the
axis due to its relatively long length. The nanopillar shown here had an initial aspect ratio of 1:4, and
was only constrained at the base. The tip of the pillar then underwent a small lateral translation across
the diamond indenter which resulted in the introduction of a slight bend in the tip of the pillar.
Subsequent indentations caused the pillar to deform further from the original axis as the applied load
was increased. The lateral deformation resulted in new grain formation and straightening of the pillar.

During the indentations, loads of ~100µN were reached before activating significant plasticity, in
the form of dislocation flow, which was sufficient to reduce the applied load (figure 2(d)). In the load-
displacement curve for indent 3, the three significant jumps (reduction) in load (I-III, figure 2(d)) can
be associated with significant rapid microstructural events. These appear to be the collective rearrangement of dislocations to form low and high angle grain boundaries to reduce stress (see §4, 5). After the first two of these events (jumps I and II), there is a subsequent small initial increase in the load, before dislocation activity re-establishes and the load again begins to decrease. At a total displacement of 960nm the load, and therefore the compressive stress in the pillar again begins to build up. A significant lateral displacement of material, corresponding to Jump III, occurs to relieve this stress.

4. Nanopillar Deformation

Under the uniaxial compression a bowing of the pillar was first seen with Bragg contours moving down the pillar. A zone of high compressive stress formed at the tip of the pillar which was in contact with the diamond indenter. To release compressive stress, the pillar deformed by bending the tip over and straightening the remaining, shortened, pillar (figure 4). On unload, after indent 2, and with the loss of contact with the indenter, some time-depdendant recovery was seen with the pillar straightening itself by approx. 75nm. During the subsequent indentation there was an increase in the size of the sideways displaced material with increasing indentation depth. Within the displaced material, TEM contrast was seen to change in discrete areas, due to locally different Bragg scattering and thickness. This is also representative of the low angle lattice tilt expected during low angle sub-grain formation. As the applied force reaches a maximum the severe lattice distortion leads to dislocation rearrangement and the formation of new grains (figure 4), generating a polycrystalline pillar tip. Figure 4(b) shows grains protruding from the surface of the deformed pillar after testing.
5. Deformation Mechanisms
To release stress in the bent pillar, dislocation flow occurs within the zone of highest compressive stress at the tip. Dislocations are also seen to propagate in the less stressed main shaft of the pillar. Rapid jumps in displacement are observed as the new grains are formed within the deformed volume. Post mortem evaluation in the SEM shows a three dimensional arrangement of these grains as a protrusion away from the original axis of the pillar, figure 4(b). The grain formation enables straightening of the remainder of the main shaft of the pillar which now contains less material. The constraint at the base of the pillar leads to dislocation pile-up at the pillar base and the creation of a high-angle grain boundary which stops further dislocation flow into the substrate, figure 3(e-f). In other pillar investigations dislocations are seen to flow up the shaft towards the deformed area, as well as from the deformed area towards the base, to reduce stress.

The deformation in these 350nm diameter pillars differ from deformation mechanisms seen in wider, stiffer, pillars, such as slip bands and shearing [1-3]. Plastic flow due to localised slip has been seen in single crystal aluminium alloy pillars (140-180nm diameters) resulting in ‘mushrooming’ [7]. This would have the effect of spreading the load over a greater area, thus reducing the applied stress, as seen in this experiment; however the formation or growth of grains was not observed. The localised slip seen to cause the ‘mushrooming’ was explained due to the taper of the pillar.

The growth of existing grains under deformation has been seen in aluminium thin films [11] and grain rotation was seen in nanoscale grained aluminium [12], however the formation of new grains through deformation has not been previously reported by in-situ experiments. Thin film single crystals have previously been observed to form new grains after ex-situ nanoindentation [13]. The grains formed here during the in-situ compression of the nanopillars also appear to have undergone grain rotation as the deformation progressed.

6. Conclusions
During in-situ real-time uniaxial compression tests of nanopillars undertaken in the TEM microscope, it is seen that rapid microstructural changes in the Al nanopillars can be directly related to features in the force-displacement curves. The mechanisms of deformation were analysed and Al nanopillars are seen to reduce stress by grain formation and re-arrangement in a three dimensional manner. As the diameter and length of the Al pillars are clearly important factors in the deformation mechanisms it would be prudent for future work to investigate deformation mechanisms as a function of geometry.

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