In Situ TEM Study of Dislocation Nucleation and Escape in a FIB Structured 500nm Thick Al Single-Crystal Wire

S.H. Oh [1,2] M. Legros [3], D. Kiener [1], G. Dehm[1,4]

1. Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Austria
2. Division of Electron Microscopic Research, Korea Basic Science Institute, Daejeon, Korea
3. CEMES-CNRS, Toulouse, France
4. Department Materials Physics, University of Leoben, Austria

Gerhard.dehm@mu-leoben.at
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"Smaller is stronger" does not only hold true for nanocrystalline materials but also for single crystals. It is argued that this effect is caused by geometrical constraints on dislocation nucleation and/or motion in (sub-)micron sized crystals [1,2]. Here we report the first in situ TEM tensile tests of a submicron single crystal Al wire directly providing insight into source controlled dislocation plasticity [3].

A nominally 500 nm thick Al film was grown epitaxially on a (001)-oriented NaCl single crystal substrate and subsequently covered by an ~8 μm thick layer of polyimide. Then, the sample was immersed in deionized water to remove the NaCl substrate obtaining a single crystal Al film on polyimide. A piece of Al/polyimide stripe with ~3 mm length and less than ~2 mm width was cut along the <100> directions and fixed with conventional superglue on a rectangular Cu support (Fig. 1A) which can be fixed in a conventional TEM straining stage. FIB was used to thin a 100 x 100 μm^2 wide electron transparent window into the polyimide without thinning the Al film. Subsequently, a wire pattern was cut by FIB into the Al film (Fig. 1B). Additionally, a side notch was made by FIB to concentrate stress and strain in the ~10 μm long and ~0.5 μm wide wire. During incremental tensile loading the polyimide substrate prematurely cracked at a strain of ~40 % perpendicular to the tensile axis at the notch center, probably because of FIB-induced embrittlement of the polymer. This exposed a free-standing Al wire in-between both fractured sides of the polyimide substrate (Fig. 2).

Single-ended dislocation sources built up by dislocation interaction and became the dominant deformation mechanism (Fig. 2). A total of six dislocation sources were detected in the course of straining from $\varepsilon \approx 40$ % to 160 %. Dislocation sources were activated in an alternating manner along the diagonal directions of the crystal. Each strain increment governed one or two single-ended sources to emit 2-3 dislocations towards the central neck. The other sources remained inactive until the next load increments. The projection corrected source lengths (L) are in the range of 50-150 nm. The local shear stresses (τ) required to activate the single-armed source can be estimated by $\tau \approx 0.09G\boldsymbol{b}/L \cdot \ln(L/\boldsymbol{b})$ to values between 28 and 70 MPa, where G = 26 GPa is the shear modulus, and $|\boldsymbol{b}|$ = 0.286 nm the amplitude of the Burger's vector. These values significantly exceed the flow stress of a few MPa reported for bulk single crystal Al in literature.

Although many dislocation activities were observed in the course of deformation up to $\varepsilon \approx 160$ %, the general notion is that, for a maintained strain rate of around 10^{-4} s^{-1}, the dislocation density remained statistically constant due to the continuous exhaustion of mobile dislocations by the surfaces (Fig. 3). Thus, the present in situ TEM straining study confirms that the dislocation escape rate is indeed comparable to the nucleation rate, continuously exhausting mobile dislocations throughout the deformation. For a given strain rate, the

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dislocation density fluctuates around a level similar to its initial value indicating a loss of work hardening at small sample dimensions.

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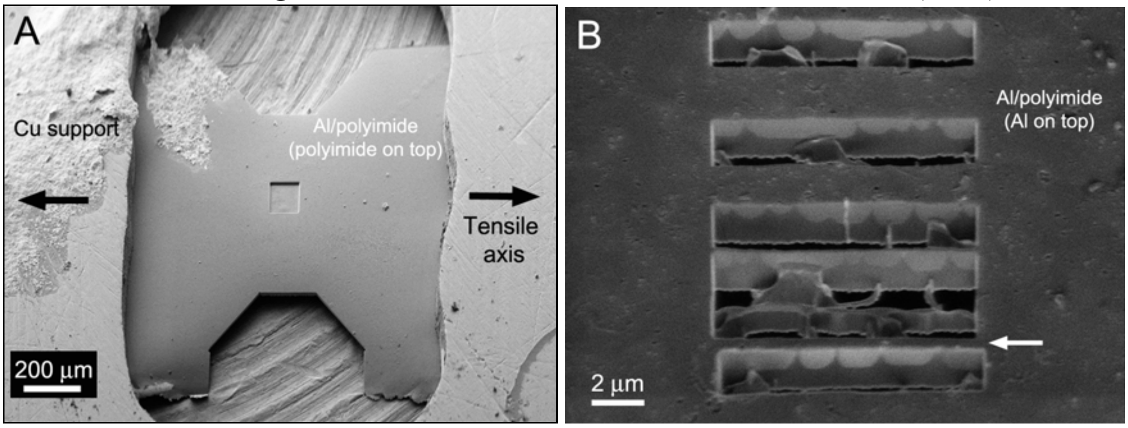

Figure 1. (A) SEM image showing the side grooves and the electron transparent window made in the tensile testing sample by FIB. (B) SEM image of the parallel wires shaped within the electron transparent window by FIB. The wire observed during TEM straining, marked by a white arrow in the image, was 10 µm long and 0.5 µm wide.

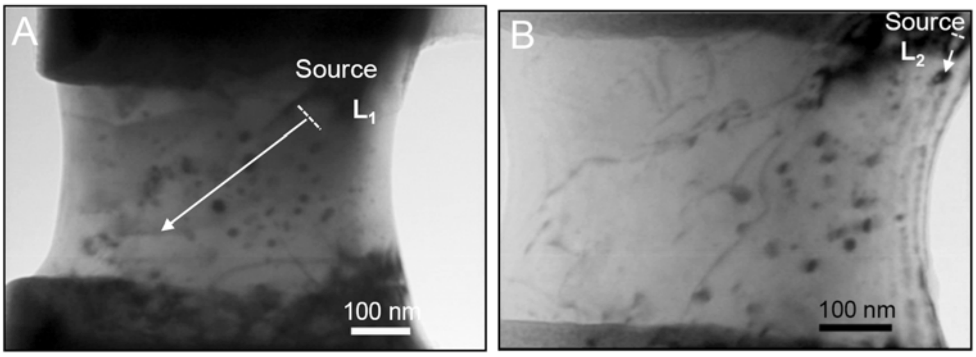

Figure 2. An example of dislocation sources observed at different strain stages. Single-ended sources with sizes of (A) 135 ± 15 nm at ε ≈ 80 % and (B) of 52 ± 15 nm at ε ≈ 140 %.

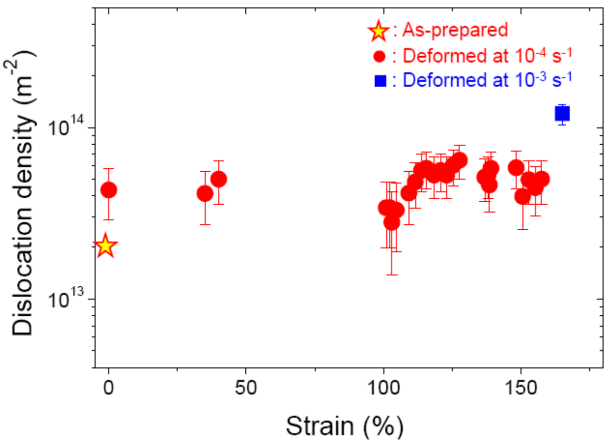

Figure 3. Dislocation density (error bars give the standard deviation) measured from TEM images taken at various two-beam conditions as a function of the axial strain. Within a good approximation, the dislocation density remains constant in the range of ~5 × 10^{13} m^{-2} during straining at $\dot{\varepsilon} = 10^{-4}\,s^{-1}$. For comparison, the initial as-prepared Al film had a dislocation density of ~2 × 10^{13} m^{-2}. A steep increase of the strain rate by one order of magnitude ($\dot{\varepsilon} = 10^{-3}\,s^{-1}$) causes the dislocation density to rise by a factor of 2. A pre-crack followed this density increase and precluded further reliable measurements.

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