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TEM Study of Dislocation Loops in Deformed Aluminum Films*

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We report the results of a TEM investigation of the defect structure, in particular prismatic dislocation loops, that resulted from two entirely different modes of deformation of thin films (< 1 μm thick) of aluminum

In one experiment, free-standing evaporated films, approximately 1 μm x 10 μm x 200 μm, were deformed in tension at a constant elongation rate to fracture. The tests were interrupted twice at approximately equal load increments so that EBSD data could be obtained for purposes of tracking orientation changes of individual grains. After failure, the free end of the test specimen was mounted on a silicon nitride window for examination in the TEM.

In the second experiment, Al-1%Si lines, approximately 0.5 μm x 3 μm x 800 μm, on oxidized silicon were tested under very high alternating current density, >12MA/cm^2. In such testing conditions, Joule heating and differential thermal expansion between the aluminum lines and the substrate cause cyclic thermal straining, resulting in thermomechanical fatigue (ACTMF). Tests were performed quasi *in-situ* and stopped periodically so that EBSD data could be obtained. After open circuit failure some typical extremely deformed regions were FIBed to make TEM samples.

Although the two tests are very different, we found very similar defect structures in the TEM for samples from the failure sites. In fact, this observation has provided us with an incentive to further explore the possible use of AC testing to extract mechanical properties from very small structures.

TEM examination of failed specimens from both experiments showed a very high density of prismatic dislocation loops in areas relatively devoid of forest dislocations. This observation was general throughout the ACTFM samples, Figure 1. Both the starting and failed ACTMF samples showed a low density of dislocations, and the starting ACTMF sample had few loops. The tensile samples initially had moderate loop and dislocation densities, Figure 2. The failed site at the chisel point fracture of the tensile sample showed a high density of loops and a low dislocation density, Figure 3. In the remainder of the tensile sample a lower density of loops was seen amongst tangles containing a high dislocation density, Figure 4.

The diameter of the loops in the ACTMF samples averaged 10 ± 4 nm, containing about 10^3 vacancies per loop assuming each loop to be a collapsed disc of vacancies. This dimension also gives an upper bound to the temperature reached in the thermal cycle of about 170^o C. The loops in the tensile sample were somewhat larger and averaged 13 ± 5 nm in diameter, containing about $1.7x10^3$ vacancies. The intersection of dislocations on different slip planes often creates vacancies, thus we have the most probable source. In the ACTFM experiment the thermal stresses at the Al-SiO_2 interface will provide a source for dislocations on multiple slip planes, and the temperature rise of the ACTMF allows the vacancies to diffuse and form loops. The stress in the tensile experiment applied to background of existing dislocations both within the grains and at grain boundaries will continually nucleate and cause dislocations to glide forming the tangles. Intersections between dislocations in

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these high dislocation density ($>10^{14}/m^2$) tangles will subsequently provide localized concentrations of vacancies sufficient to form the loops.

At the chisel fracture tip the glissile dislocations escape to the surface creating slip steps, which we have observed in the SEM, while the prismatic loops are sessile and remain behind. This is consistent with our EBSD observation that there is little grain rotation in the tensile samples during elongation except at the fracture site. On the other hand, in the ACTMF samples there are very few grown-in dislocations, but the thermal stress creates dislocations on multiple slip systems which glide and intersect, creating vacancies. Subsequently they continue gliding either to the surface, where significant surface deformation has been observed, or to pre-existing grain boundaries causing grain rotation, which we have observed in EBSD measurements.

These observations are thought to be the first time that a high density of prismatic loops has been reported associated with low strain rate ($\sim10^{-5}/s$) or thermomechanical fatigue deformation in Al.

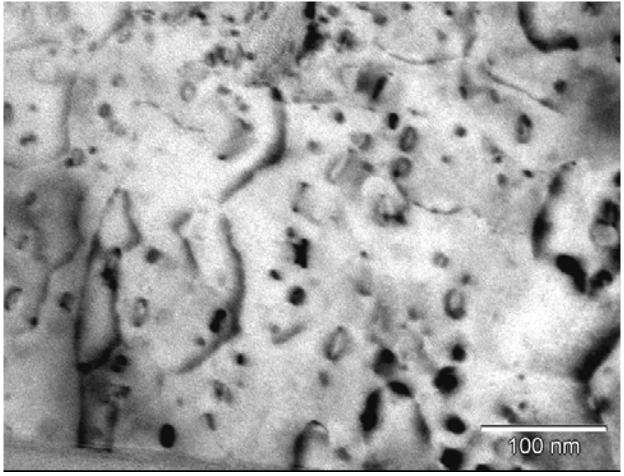

Figure 1. ~40 nm prismatic dislocation loops in a failed site of a ACTMF sample.

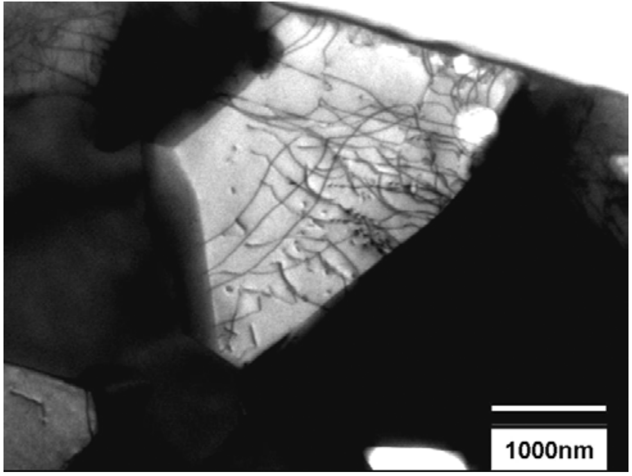

Figure 2. Typical dislocation and loop structure in a tensile sample prior to testing.

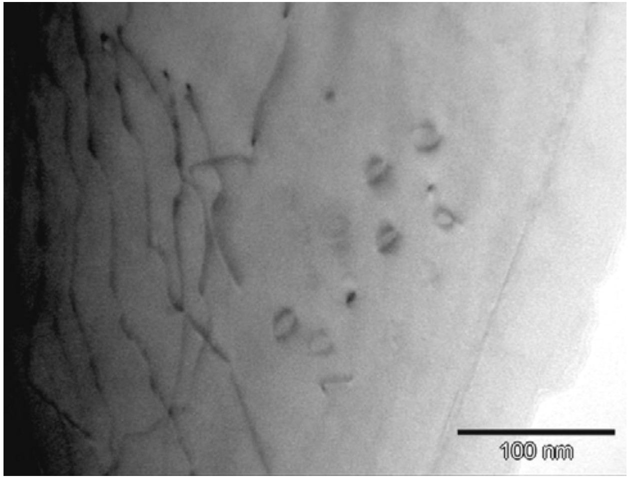

Figure 3. ~80 nm prismatic dislocation loops at the chisel point fracture in a tensile sample.

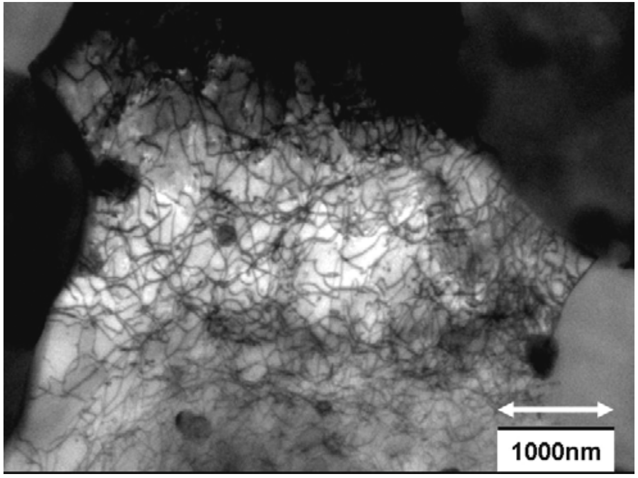

Figure 4. Dislocation loops and tangles in the bulk of a tensile sample after pulling to failure.