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TEM-Based Analysis of Defects Induced by AC Thermomechanical *versus* Microtensile Deformation in Aluminum Thin Films

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ABSTRACT

Thin films of sputtered aluminum were deformed by two different experimental techniques. One experiment comprised passing high electrical AC current density through patterned Al interconnect lines deposited on SiO_2/Si substrates. The other consisted of uniaxial mechanical tensile deformation of a 1 μm thick by 5 μm wide free standing Al line. In the electrical tests approximately 2 x 10^7 W/cm^2 was dissipated at 200 Hz resulting in cyclic Joule heating, which developed a total thermomechanical strain of about 0.3 % per cycle. The tension test showed a gauge length fracture strain of only 0.5 % but did display ductile chisel point fracture. In both experiments, certain grains exhibited large, > 30°, rotation away from an initial <111> normal orientation toward <001>, based on electron backscatter diffraction (EBSD) measurements in the scanning electron microscope (SEM). Transmission electron microscopy (TEM) analysis of specimens from both experiments showed an unusually high density of prismatic dislocation loops. In the mechanically-tested samples, a high density of loops was seen in the chisel point fracture zone. In cross sections of highly deformed regions of the electrical test specimens, very high densities, > 10^{15}/cm^3, of small, < 10 nm diameter, prismatic loops were observed. In both cases the presence of a high density of prismatic loops shows that a very high density of vacancies was created in the deformation. On the other hand, in both cases the density of dislocations in the deformed areas was relatively low. These results suggest very high incidence of intersecting dislocations creating jogs and subsequently vacancies before exiting the sample.

INTRODUCTION

As we transition into a world of smaller dimensions – the nanoworld – it becomes increasingly more difficult to reliably measure the mechanical properties of materials with nano-dimensions (< 100 nm). New measurement tools are needed in the rapidly growing field of nanomaterials. In particular, information about mechanical properties such as elastic modulus, ultimate tensile strength, fatigue life, maximum strain, adhesion and the relation of defect structures to these properties is critical to successful development of new materials. Such information is also needed to assess integrity or reliability in many applications; for example, multilayer electronic interconnects and solder joints. The difficulty of fabricating complex systems requires the use of predictive modeling in order to achieve cost and time savings. However, modeling can correctly predict system performance only if the property data used as input are accurate at the relevant length scales. Furthermore, in heterogeneous systems it is often the localized

variation in properties that causes failure (void formation, fracture, *etc*.). Thus it is increasingly important to assess not only the "average" sample properties, but also to obtain data relating to the spatial distribution in properties.

Many existing methods for mechanical-property measurements have drawbacks: they are often destructive, not quantitative, limited to specialized geometries, require samples which are difficult to fabricate, and so on. Currently, one of the most commonly used tools for this purpose is nanoindentation [1]. However, existing nanoindentation techniques face measurement challenges as dimensions continue to shrink. In such systems, the volume sampled by nanoindentation may be too large for adequate analysis due to the large radius of the indenter tip. The lateral resolution of a typical Berkovich diamond indenter used is a few hundred nanometers and the relatively large loads applied may also be an issue.

We are pursuing nanomechanical measurements from a different point-of-view by using electrical measurements to determine the mechanical properties of thin films on a substrate. As part of this program we compared the defect structures in samples exposed to two very different types of tests. In one test thermomechanical fatigue was introduced by electrical means and in the other a uniaxial tension test was performed. The microscopic deformation behavior was evaluated in the SEM and with EBSD and is reported elsewhere in these proceedings [2]. The defect structures which remained after failure were studied by TEM.

The literature contains an abundance of TEM studies discussing defects in thin films and in thin foils prepared from bulk samples, but none from alternating current thermomechanically fatigue (ACTMF) tested Al-Si interconnect films on a substrate, or from 1 μm thick Al films fractured in tension. There have been numerous high voltage SEM studies of void formation and migration in direct current electromigration experiments [3], an entirely different type of test.

EXPERIMENT

AC tests were carried out on non-passivated, single-level structures composed of patterned and etched Al-1Si lines sputtered onto thermally oxidized silicon, using conventional processing parameters. We used a NIST test pattern originally designed for electromigration and thermal conductivity measurements. Testing was conducted on a 4-point probe station using 100 Hz sinusoidal alternating currents with zero DC offset. Tests were conducted continuously until the lines became electrically open. Current densities (rms) applied to individual lines ranged from 11 to 16 MA/cm^2. For the sample whose microstructure is presented here, testing was done with an AC current density of 12.2 MA/cm^2 at 100 Hz, The line was 800 μm long by 3.3 μm wide and 0.5 μm thick. In this test approximately 2 x 10^7 W/cm^2 was dissipated at 200 Hz resulting in cyclic Joule heating. Based on prior work [4], when a current density of 12.2 MA/cm^2 was applied, the base specimen temperature, as monitored using a thermocouple attached directly to the die, indicated a rise of < 10 K during testing. However, the low frequency AC signals led to temperature cycling superimposed onto the base die temperature, with an amplitude of approximately 100 K, at a frequency of 200 Hz, corresponding to a power cycling input into the line which developed a total thermomechanical strain of about 0.3 % per cycle. Based on the TEM observations of loop diameters, discussed later, an upper

bound on the temperature rise of ~170 ^{o}C [5] can be established. For more details of the AC test see [2]. The uniaxial tension tests were conducted on sputtered Al test sections 200 µm long, 1 µm thick and 5 µm wide at room temperature (RT). The elongation rate was approximately 10^{-4}/s. The tension test showed a gauge length fracture strain of only 0.5 % but did display ductile chisel point fracture.

Specimens for TEM were prepared from severely deformed regions after the ACTMF electrical test by focused ion beam (FIB) preparation of sections along the test line. In the tensile tests, the free fractured end from the samples was mounted on a Si_3N_4 window by simply pressing the non-tested end down firmly along the outer frame.

The TEM experiments were carried out in a microscope with a LaB_6 gun at 200 kV. Images were recorded on a 1K x 1K CCD camera mounted below the viewing chamber. Sample manipulation was done using side entry holders with single-tilt, double-tilt and tilt-rotate capabilities. The fracture tip samples pressed onto the Si_3N_4 window grids were very fragile and usually studied using the single tilt holder, which is better designed for loading more delicate samples.

EBSD studies of samples from both experiments were done quasi *in-situ*. A summary of the ACTMF experiment is presented elsewhere in these proceedings [2]. Grain orientation maps from a few samples in the tensile experiments were obtained prior to pulling and also during the deformation by stopping a few times prior to fracture and acquiring maps from the complete test section with EBSD. After fracture we could only get data from the part of the sample that was still fixed to the deformation jig.

RESULTS

TEM examination of failed specimens from both experiments showed a very high density of prismatic dislocation loops in areas relatively devoid of forest dislocations, see Figures 1 and 2. Figure 1 is typical of a severely deformed grain after failure in the AC test. The average diameter of the loops is 8 ± 4 nm. Each loop contains about 10^3 vacancies assuming a loop to be a collapsed disc of vacancies. The loop density is about 3.3 x 10^{15}/cm^3, which compares with ~ 10^{15}/cm^3 reported for Al sheets quenched from 600 ^{o}C into iced brine [6]. The vacancy concentration is about 10^{-4}, which also compares favorably with the prior TEM results from quenched Al sheets [6]. The loop diameter of 8 nm also suggests the maximum temperature reached during the thermal cycle is less than 170 ^{o}C [5]. Figure 2, from the tip at a site of chisel point fracture in a tensile sample, shows somewhat larger loops, about 14 ± 5 nm in diameter. The loop density here is ~ 7 x 10^{14}/cm^3. This is an order of magnitude increase over that in undeformed samples. There is also a decrease in average loop diameter after fracture. Initially the average loop diameter was 23 ± 10 nm, but after fracture it was ~ 14 ± 4 nm. Calculations using these numbers yield an initial vacancy concentration of 5 x 10^{-5} while after fracture at the chisel point tip it increased to about 10^{-4}. This latter result is similar to that obtained from the ACTMF tested samples. As expected, the dislocation density in the Al tensile specimens increased from the initial state of ~ 2 x 10^{13}/m^2, (see Figure 3), to ~10^{15}/m^2 after fracture, (see Figure 4).

EBSD measurements showed a rotation of about 30°, from near the <011> to near the <112>, of the tensile axis in grains at the fracture tip This is as would be expected from classical single crystal tensile deformation of face centered cubic metals.

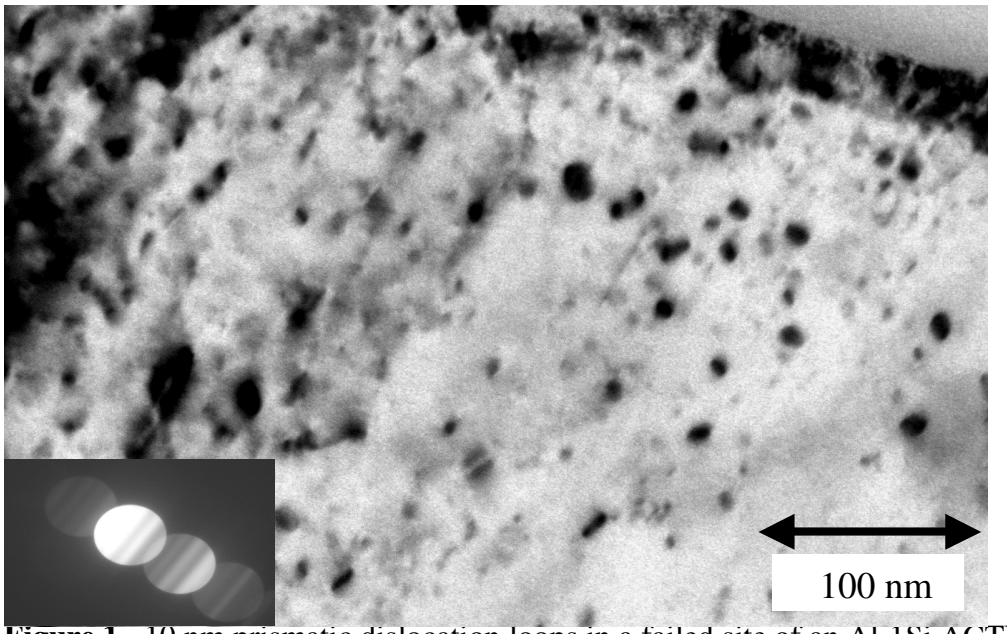

Figure 1.~10 nm prismatic dislocation loops in a failed site of an Al-1Si ACTMF sample.

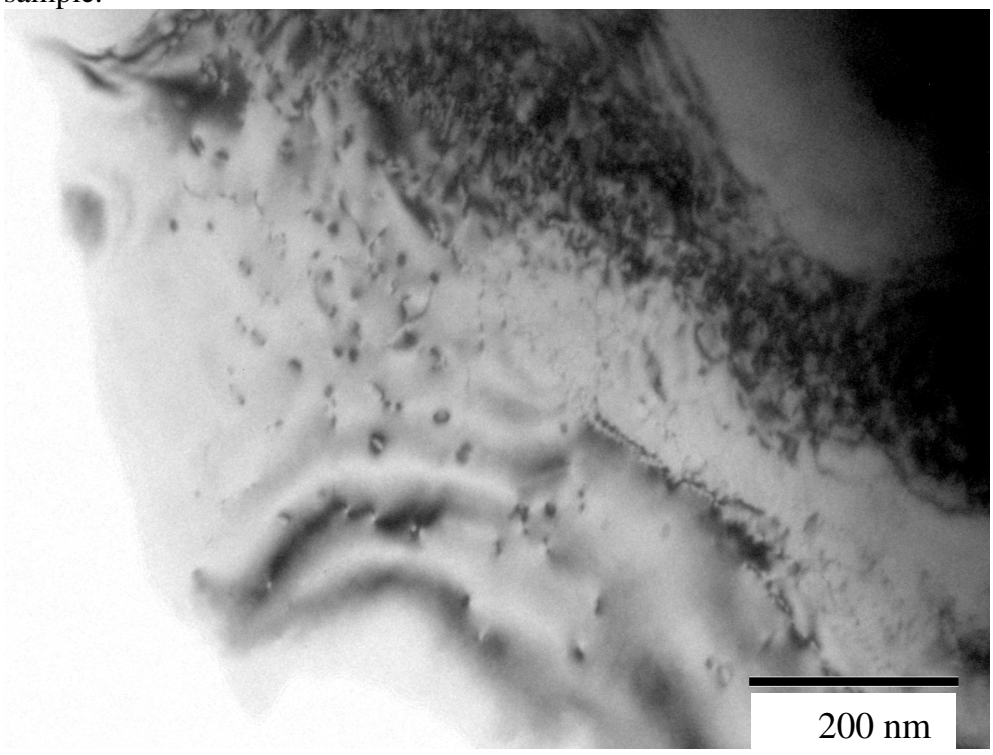

Figure 2. ~15 nm prismatic dislocation loops at the chisel point fracture tip in a tensile sample.

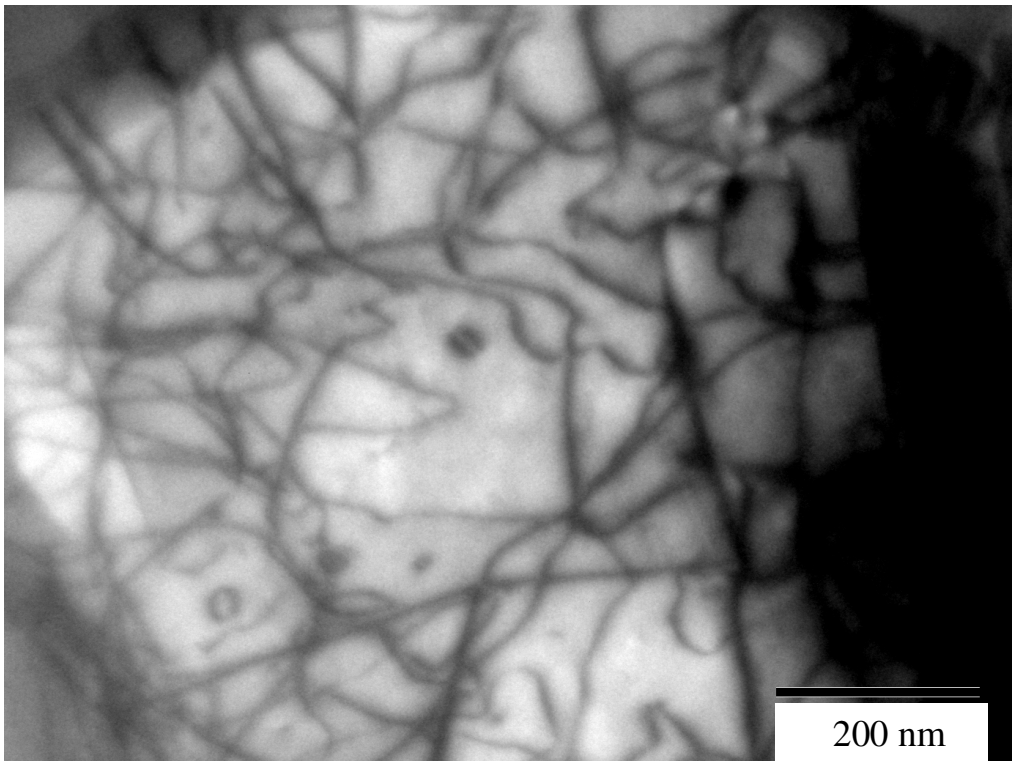

Figure 3. Typical dislocation and loop structure in a tensile sample prior to testing.

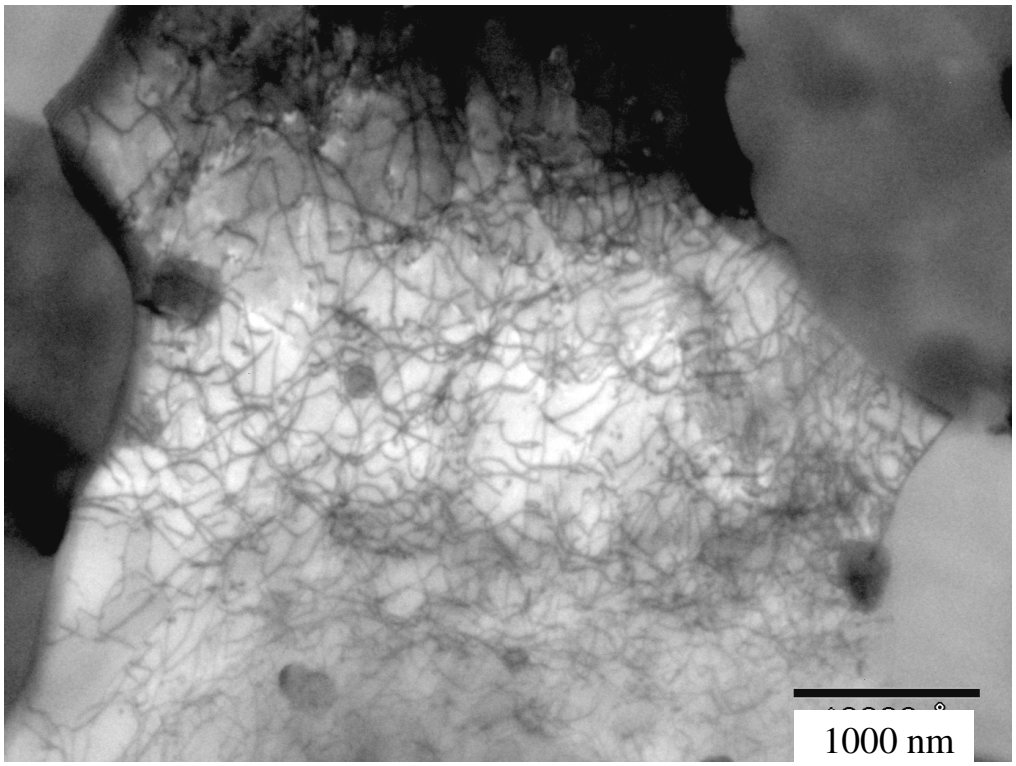

Figure 4. Dislocation loops and tangles in the bulk of a tensile sample after deformation.

DISCUSSION

TEM studies of defects in sputtered aluminum films, deformed using two very different experimental techniques, show striking similarities. In both ACTMF and tension tests very high concentrations of prismatic dislocation loops were observed in locations generally void of dislocations. In both instances we propose that a large number of dislocations on multiple glide systems intersected and created vacancies which subsequently coalesced to form the loops. The loops are prismatic on (111) planes with a ½[110] Burgers vector normal to the plane of the loops and were not very mobile at the temperatures of the experiments, approximately 170 ^{o}C for the ACTMF experiment and RT for the tension experiment. Thus the loops remained while the dislocations continued to slip to either to free surfaces or to grain boundaries resulting in the formation of dislocation free zones with a high density of loops. EBSD analysis of both experiments showed large grain rotation in the deformation zones most likely caused by a large number of dislocations intersecting grain boundaries and increasing the degree of misorientation across a boundary. The vacancy concentrations in the deformed samples are consistent with those in prior reports on quenched samples and suggest that a concentration of 10^{-4} may be an upper limit to that which can remain in aluminum at room temperature. The unique observations that we have made in samples from both our experiments are the presence of a very high density of prismatic dislocation loops and the absence of dislocations. In the AC tested samples this observation is general throughout the whole of the deformed regions. In the fractured sample this observation is restricted to fracture tip areas.

The similarities in the defect microstructures after testing, namely the high density of prismatic dislocation loops, suggest a similar deformation mechanism proceeded in both experiments and that AC electrical thermomechanical fatigue testing might be used to measure the mechanical properties of constrained dimension metal structures.

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