An In Situ HVEM Study of Dislocation Generation at Al/SiC Interfaces in Metal Matrix Composites

MARY VOGELSANG, R. J. ARSENAULT, and R. M. FISHER

Annealed aluminum/silicon carbide (Al/SiC) composites exhibit a relatively high density of dislocations, which are frequently decorated with fine precipitates, in the Al matrix. This high dislocation density is the major reason for the unexpected strength of these composite materials. The large difference (10:1) between the coefficients of thermal expansion (CTE) of Al and SiC results in sufficient stress to generate dislocations at the Al/SiC interface during cooling. In this in situ investigation, we observed this dislocation generation process during cooling from annealing temperatures using a High Voltage Electron Microscope (HVEM) equipped with a double tilt heating stage. Two types of bulk annealed composites were examined: one with SiC of discontinuous whisker morphology and one of platelet morphology. In addition, control samples with zero volume percent were examined. Both types of composites showed the generation of dislocations at the Al/SiC interface resulting in densities of at least $10^{13}$ m$^{-2}$. One sample viewed end-on to the whiskers showed only a rearrangement of dislocations, whereas, the same material when sectioned so that the lengths of whiskers were in the plane of the foil, showed the generation of dislocations at the ends of the whiskers on cooling. The control samples did not show the generation of dislocations on cooling except at a few large precipitate particles. The results support the hypothesis that the high dislocation density observed in annealed composite materials is a result of differential thermal contraction of Al and SiC. The SiC particles act as dislocation sources during cooling from annealing temperatures resulting in high dislocation densities which strengthen the material.

I. INTRODUCTION

The incorporation of 20 vol pct discontinuous SiC whiskers into a 6061 Al matrix increases the yield strength of annealed powder compacted 6061 Al alloy by more than a factor of two. This increase in strength cannot be explained directly by continuum mechanics theories. Continuum mechanics formulations developed by Piggott$^1$ and applied to the case of discontinuous Al/SiC composites by Arsenault$^2$ predict an ultimate strength of only 186 MPa for 20 vol pct SiC composite, whereas the measured value of ultimate strength for this material is 448 MPa. Arsenault and Fisher$^3$ proposed that the increased strength could be accounted for by a high dislocation density in the Al matrix which is observed in bulk composite material annealed for as long as 12 hours at 810 K.

The dislocation generation mechanism proposed by Arsenault and Fisher to account for this high dislocation density is based on the large difference (10:1) in coefficients of thermal expansion (CTE) of Al and SiC.$^4$ When the composite is cooled from elevated temperatures of annealing or processing, misfit strains occur due to differential thermal contraction at the Al/SiC interface which are sufficient to generate dislocations.

Chawla and Metzger, in an elegant investigation of Cu/W composites using etch-pitting techniques, observed a high dislocation density at the Cu/W interface which decreased with increasing distance from the interface.$^5$ They observed that if the volume fraction of W was 15 pct, the minimum dislocation density in the matrix was $7 \times 10^{11}$ m$^{-2}$ increasing to $4 \times 10^{12}$ m$^{-2}$ at the interface of W and Cu, and concluded that the dislocations were caused by the differences (4:1) in CTE of Cu and W. Recalling that the CTE difference between Al and Si is 10:1, i.e., more than twice as great as the Cu/W system, one would expect thermal stresses in Al/SiC to be sufficient to generate dislocations in this composite.

Other causes may also contribute to the high dislocation density observed in annealed Al/SiC material. Dislocations are introduced into this material during the plastic deformation processes of manufacturing, such as extrusion. During annealing, the dislocations introduced during processing may not be annihilated; they could be trapped by the SiC, resulting in a high dislocation density after annealing.

It is important to determine the origins of the high dislocation density in the composite since the strength of the composite depends on the high density. If the differential thermal contraction is the cause of the dislocations, as Arsenault and Fisher$^3$ suggest, then dislocations should be observed being generated in a composite thin foil sample on cooling from annealing temperatures in an in situ, HVEM experiment.

In situ dynamic HVEM experiments have certain advantages over other experimental techniques. The major advantage is that direct observation of a dynamic process altering a microstructure is possible while the deforming force, in this case a thermal stress, is operating. Operating at higher voltages allows penetration of thicker samples so that surface effects are minimized and bulk behavior is more closely approximated. Also, a high voltage microscope can better accommodate special stages required for in situ work because of the large pole piece region.

Numerous in situ HVEM heating stage investigations of Al have been performed. Hale et al.$^6$ and Caillard and Martin$^7$ investigated dislocation motion during creep at
elevated temperatures using HVEM. Kivilahti et al.8 observed an Al-2 pct Mg alloy in situ during recovery processes at elevated temperatures recording dislocation interactions on videotape. Shimotomori and Hasiguti9 observed in situ prismatic punching of dislocation loops at second phase precipitates in an Al-1.3 pct Li alloy. Electron irradiation of Al at elevated temperatures has been extensively studied using HVEM.10,11

There have been several non-dynamic, non-in situ TEM investigations of dislocations about particles in a metal matrix. Weatherly12 observed multiple slip mode dislocation tangles around silica in Cu, and concluded they were caused by differential thermal contraction of the two materials on quenching. Ashby et al.13 observed dislocations about pressurized silica-Cu, noting a critical size dependence for dislocation generation. Williams and Garmong14 reported a high incidence of dislocations at the Ni/W interface in this directionally solidified eutectic composite.

Calculations of the dislocation density in Al/SiC due to thermal stresses predict high dislocation densities. The misfit strain which develops at the circumference of a 1 μm diameter SiC particle due to differential thermal contraction during cooling is approximately 1 pct. The plastic strain at one-half the interparticle spacing, obtained from Lee et al.,15 ranges from 1 to 2 pct. The dislocation density can be simply calculated from the following equation:

\[ \varepsilon_p = \rho L \beta. \]  

where \( \varepsilon_p \) is the plastic strain (1 pct), \( \rho \) is the dislocation density (m\(^{-2}\)) generated, \( L \) is the average distance moved by the generated dislocations, which was taken to be 1/2 the inter-whisker spacing, \( \beta \), is 2 μm, and \( \beta \) is the Burgers vector of Al. The \( \rho \) obtained is 1.8 \( \times \) 10\(^{13}\) m\(^{-2}\).

Consideration of another type of dislocation described by Ashby16 predicts additional dislocations in the matrix. These dislocations are called "geometrically necessary" dislocations by Ashby, and occur in order to allow compatible deformation of a system with geometrical constraints such as hard particles which do not deform as the surrounding ductile matrix. These geometrically necessary dislocations are required if the deformation takes place without the formation of voids about the hard particles. Slip dislocations are a function of the material properties of the system, and are not dependent on the microstructural constraints. According to Ashby, the density of geometrically necessary dislocations, \( \rho^G \), is given by:

\[ \rho^G = \frac{4\gamma}{\lambda^G \beta}. \]  

where \( \lambda^G \) is the "geometrical slip distance" analogous to the slip distance in pure crystals. For platelet particles, \( \lambda^G \) is equal to the length of the plate and \( \gamma \) is the shear strain. For a 1 pct shear strain and \( \lambda^G = 4 \mu m \), \( \rho^G \) equals approximately 3 \( \times \) 10\(^{13}\) m\(^{-2}\). Taking these dislocations into account results in a further addition to the predicted dislocation density in the Al matrix.

The purpose of this investigation was to determine if dislocation generation occurs at the Al/SiC interface on cooling a composite from annealing temperatures, and to determine if the observed densities of dislocations generated during cooling are in agreement with densities predicted by theoretical calculations.

III. SAMPLE PREPARATION AND EXAMINATION PROCEDURE

An ion milling technique was required for the production of TEM samples due to the SiC in the Al matrix.

These two types of composite and the 0 vol pct control were machined into rods (12 mm in diameter, 4 cm long), annealed for 12 hours at a solutionizing temperature of 810 K, and furnace cooled. After annealing, slices of 0.76 mm thickness were cut by electric discharge machining (EDM) at 80 to 100 V. Deformation damage from EDM is estimated to extend 0.20 mm beneath the surface.17,18 The slices were fixed to a brass block with double-sided tape and surrounded by brass shims, then mechanically thinned on a rotating water flooded wheel covered with 400 then 600 grit paper to remove the EDM damage and reduce the thickness to approximately 0.127 mm. Final thinning was carried out using argon ion plasma bombardment, operating at 6 kV, and ion current of 50 micro amperes and a sample inclination of 15 deg to the ion beam. For these operating parameters the projected range, or average distance the argon ion travels into the foil, is only 20 nm.19,20 Dupuoy21 conducted an in situ ion thinning experiment on Fe and Al-Ag specimens using a 3 MV microscope. Dislocation arrangements and microstructures in Al-Ag and Fe were not altered by ion thinning even though some point defects are introduced into the near surface region of the sample by ion bombardment. Therefore, it can be concluded that ion-milling does not introduce or remove dislocations in the TEM foils.

The 1100 grade Al control samples were prepared from the as-received wrought rod in the same manner as the composite samples, except electro-polishing was employed instead of ion-thinning.

The thinned samples were then observed in the HVEM operating at 800 kV with a beam current of 2.3 μA. A double tilt, side-entry, furnace type heating stage was used to heat the specimen. While being observed in the microscope, the samples were heated to 800 K and held for 15 minutes, then cooled to ambient temperature. Subgrains exhibiting dislocations in contrast were chosen for observation. During heating and cooling, thermal drift of the stage and thermal expansion and contraction of the sample caused the chosen subgrain to move. In order to maintain the