Evaluation of Grain Boundary Grooving by Atomic Force Microscopy (I)

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1. Introduction

Grooving at grain boundary is a process of capillary-driven evolution of surface topography in the region where grain boundary emerges at the free surface of materials. Engineering point of view, especially, nano-scale process of electrical materials, the precise measurement of the grain boundary groove topography to determine surface energy and grain boundary energy becomes important. Mullins described the thermal grooving mechanism for the sufficiently small grooves less than submicron in which surface self-diffusion is the mechanism responsible for the groove growth.[1]

The original Mullins model is based on thermodynamics such as free energy minimization at plat surface in which the dihedral angle at the root of grain boundary is kept constant and it value is determined by Young's equation.[2]. The Mullins's model are assumed that the free surface is isotropic, the only driving force for surface diffusion being the gradient of surface curvature. Accordingly, it is important that the surface in the vicinity of a grain boundary exhibits positive curvature, convex morphology. Recently, geometrical method to determine the dihedral angle on curved surface was also reported.[3]

Kinetic point of view, grooving rate depends on surface diffusion behavior such as radius of curvature, vacancy concentration, temperature. If the materials are under-irradiated, especially heavy deformed, number of defects like void interstitial atom and vacancy is increased. This kind of defect, especially, increased vacancy concentration should influence grain boundary grooving behaviors. Although, thermodynamical approach about grain boundary grooving is relatively well described by Mullins and his colleagues, the grooving rate after irradiation is less studied. Hence, the objective of this study is to find an approach method to study the effect on irradiation on grain boundary grooving. Emphasis is on the determination of grain boundary energy and phase boundary energy.

2. Experimental Method

Composite materials with copper and silver filaments were prepared by sintering at 900°C with copper powders and silver filaments and heavy extrusion at 750°C followed by cold drawn to 1.5 mm. After drawn, the specimen was hot isostatically pressed at 750°C at a pressure of 100 MPa to get full density. The extruded bundled wire was then swaged and drawn to achieve its final size of a 2 mm x 3 mm rectangular cross section. The specimen was annealing for simulating gamma heating effect in HANARO. Surface morphology was evaluated by atomic force microscopy. Microstructure was observed by field emission scanning electron microscopy (JSM 6700F).

3. Results and Discussion

Fig. 1 is the microstructure change with annealing temperature. As shown in Fig. 1, polycrystalline filaments tend to be broken their morphology which become a faceted sphere, separately.
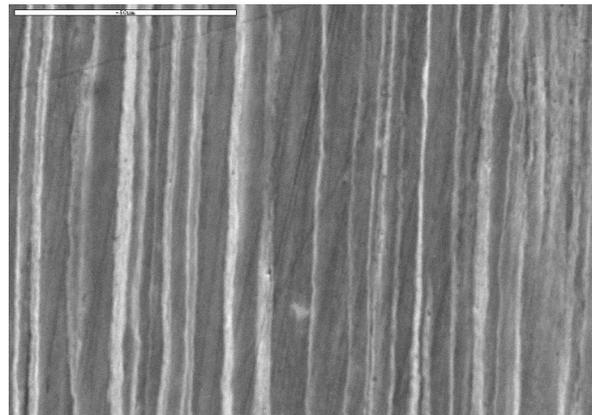
Fig. 1. SEM image of copper-silver composites

Fig. 2 is surface morphology and histogram of the surface of as-received copper-silver composites observed by atomic force microscopy. Comparing Fig. 1 and 2, silver filaments homogeneously aligned in copper filaments which is well agreement with previous results.[4] Fig. 3 is line profiles of surface roughness following green and red lines on Fig. 2. Since anisotropic surface angle is observed in interfacial boundary, whereas, isotropic surface angle is observed in grain boundary. Accordingly, the green line is scanned on same phase and the red line is scanned on different phases, respectively. Although several size of roughness were observed, the roughest surface may be the phase boundary or grain boundary like silver-silver grain boundary in green lines and silver-copper brain boundary in red line in Fig. 3. In this study, Mullins

model was applied to evaluate interface energy and grain boundary energy of copper and obtained. The ratio of grain boundary energy of silver to silver free surface energy is 1.977. Since free surface energy of silver at room temperature is estimated as about 1143.6 erg/cm^2, that is estimated by a linear relationship with temperature [4], the grain boundary energy of silver of the copper-silver composites is 2260.9 erg/cm^2. It is difficult to accurately determine free surface energy of metals at room temperature, however, it is clear that a technique with atomic force microscopy is one of plausible method to quantitatively evaluate grain boundary grooving behavior.

heating of HANARO. It is estimated that the ratio of grain boundary energy of silver to silver free surface energy is 1.977. The free surface energy and grain boundary energy of silver of the copper-silver composites are 1143.6 and 2260.9 erg/cm^2, respectively. A technique with atomic force microscopy is one of plausible methods to quantitatively evaluate grain boundary grooving behavior.

References

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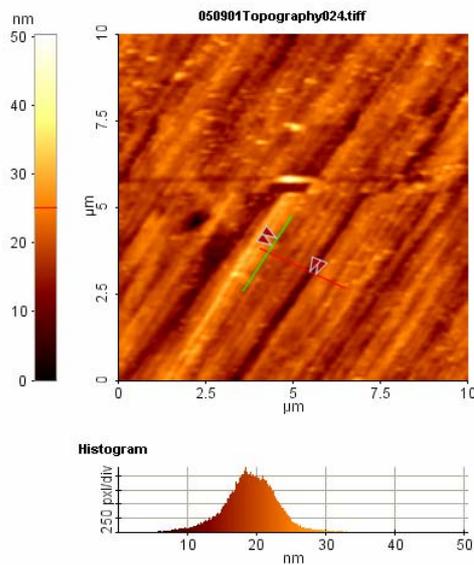
Fig. 2 Surface morphology observed by atomic force microscopy.
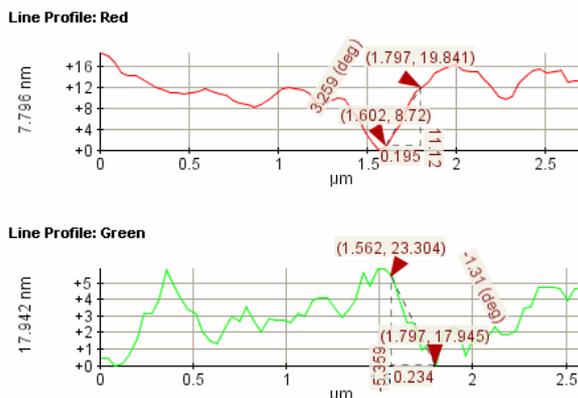
Fig. 3 Surface roughness profiles of copper-silver interface (top) and silver-silver interface (bottom).

4. Summary

Atomic force microscopy was applied to study the grain boundary grooving behavior of copper-silver composites at the simulated condition of gamma