

# Synchrotron X-ray Microdiffraction Analysis of Whisker Growth in Sn Films

W. Liu,<sup>1</sup> G.E. Ice,<sup>1</sup> B.C. Larson,<sup>1</sup> P.J. Bush,<sup>2</sup> I. Boguslavsky<sup>3</sup>

<sup>1</sup>Oak Ridge National Laboratory (ORNL), Oak Ridge, TN, U.S.A.

<sup>2</sup>State University of New York at Buffalo (UB), NY, U.S.A.

<sup>3</sup>National Electronics Manufacturing Initiative, Inc. (NEMI), Herndon, VA

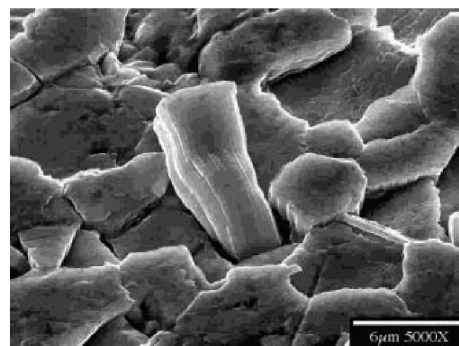
## Introduction

Grain growth in Sn films is an example of anomalous grain growth and an area of long-standing interest and vital importance to the semiconductor industry. For example, efforts to replace Pb-based solders with Sn are underway, despite the known tendency of Sn films to develop whiskers that can lead to catastrophic failure of electrical components. The nature of whisker development has been the subject of much debate, and various mechanisms have been proposed. Possible mechanisms include residual stresses, special grain boundaries, special grain orientations, or combinations of these factors [1-3]. Recent measurements at the APS polychromatic 2-D microprobe illustrated the ability to measure the local strain and grain orientation of whiskers and near-whisker regions [4]. The next step is to study the near-whisker region in three dimensions, including characterizing the local grain boundaries, elastic stress, and whisker root environment.

The ability of the 3-D polychromatic x-ray microscope to accurately measure the local grain orientation and elastic and plastic deformation tensor, as well as the grain-boundary type, provides for a stringent test of whisker growth theories. Here we characterize the local orientation and elastic strain in whiskers on a thin (~20- $\mu\text{m}$ -thick) Sn film.

## Methods and Materials

Thin Sn films with many whiskers were selected for examination. Scanning electron micrograph (SEM) images at various magnifications were used to select regions of particular interest (Fig. 1). The 3-D grain structure was evaluated by differential aperture microscopy on the polychromatic x-ray microscope located at beamline station 34-ID-E at the APS. The samples were placed in the x-ray microbeam sample holder and positioned to an interesting region by comparing the optical microscope image to the SEM image. Once an interesting region was centered on the beam, the x-ray microbeam was used to make a 3-D map of the grain orientation and strain through the Sn film.



*FIG. 1. SEM of a Sn film showing whiskers projecting from the film surface. Often the growth of whiskers is accompanied by subsidence from neighbor grains.*

## Results

In virtually all Sn samples observed, the Laue spots were sharp, and the angles between reflections were well defined by the centroid of the spots. This made it relatively simple to accurately determine the orientation of the grains and the elastic strain from the angles between the reflections [5, 6]. Typically, a good angular match to four reflections is sufficient to determine orientation. This approach has been widely and successfully applied to a number of materials systems, including electronic interconnects; polycrystalline Ni, Al, and Cu; and high-temperature superconducting, Sn, and diamond films.

Figure 2 shows a false color image of two adjacent slices through the Sn film. Whiskers can be seen projecting out of the film near the left and center of the image. From these and similar images, we conclude that the orientations of whiskers can be different, whiskers can grow on top of grains with distinct orientations, and the orientation of the whiskers can change abruptly during growth (Fig. 3). We can also determine the grain boundary types surrounding each whisker. Additional information that can be obtained includes the local strain

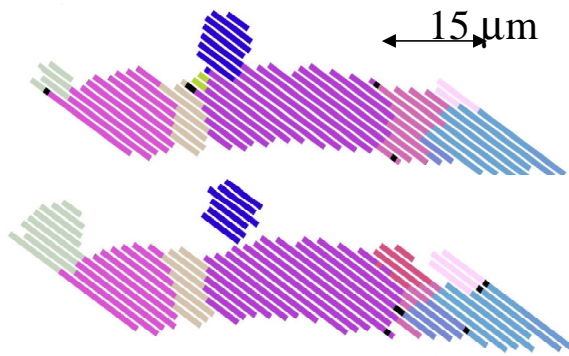


FIG. 2. Adjacent slices —  $2\ \mu\text{m}$  apart — through the surface of a Sn film. Whiskers are clearly visible, projecting out of the surface of the film near the left edge and center of the images. The grain size of this sample is about  $2\text{-}3\ \mu\text{m}$ . Samples with slightly larger grains will greatly simplify analysis of the grain-boundary networks. Color image: X-ray diffraction map showing adjacent slices through a thin Sn layer with three whiskers (gray, blue, pink) projecting from the surface.

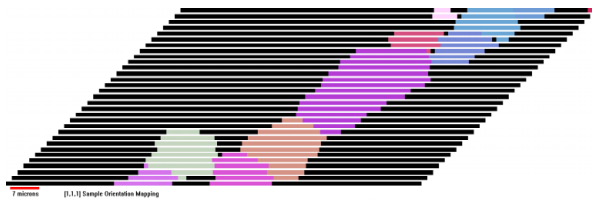


FIG. 3. Image of a whisker showing a grain boundary in the whisker.

tensor distribution and the local plastic deformation tensor.

Although the data are still being analyzed, measurements like these are certain to provide powerful tests of various theoretical models for Sn whisker growth.

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### References

- [1] R. Kawanaka, K. Fujiwara, S. Nango, and T. Hasegawa, *Jpn. J. Appl. Phys.* **22**(6), 917-922 (1983).
- [2] B.Z. Lee, D.N. Lee, *Acta Metall.* **46**, 3701 (1998).
- [3] U. Lindborg, *Acta Metall.* **24**, 181 (1976).
- [4] N. Tamura, A.A. Mac Dowell, R. Spolenak, B.C. Valek, J.C. Bravman, W.L. Brown, R.S. Celestre, H.A. Padmore, B.W. Batterman, and J.R. Paterl, *J. Synchrotron Radiat.* **10**, 137-143 (2003).
- [5] B.C. Larson, W. Yang, G.E. Ice, J.D. Budai, and J.Z. Tischler, *Nature* **415**, 887 (2002).
- [6] J.S. Chung and G.E. Ice, *J. Appl. Phys.* **86**, 5249-5256 (1999).