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Internal Microstructure Investigation of Tin Whisker Growth Using FIB Technology

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The problem of tin (Sn) whiskers has been a significant reliability issue in electronics for the past several decades. Despite the large amount of research conducted on this issue, a solution for mitigating the growth of whiskers remains a challenge for the research community. Whiskers have unpredictable growth and morphology, and a study of a whisker's internal structure may provide further insights into the reason behind their complex growth. This study reports on the internal microstructure and morphology of complex-shaped Sn whiskers grown from an electroplated bright Sn layer on brass substrates exposed to ambient and 95% humid environment. The variables analyzed include surface and microstructure conditions of the film, and morphology and internal microstructure of the Sn whiskers using scanning electron microscopy with focused ion beam technology. Experimental results demonstrated that the whiskers with more complex morphology grow primarily from surfaces exposed to a controlled environment, and some of them have traits of polycrystalline growth rather than only single crystalline, as usually known.

Key words: Sn whisker microstructure, polycrystalline, morphology, scanning electron microscopy (SEM), focused ion beam (FIB)

INTRODUCTION

Electronic systems consist of many electronic components that undergo common electroplating processes. The components are metal or alloy coated for corrosion resistance, better outside appearance, solderability, and increased electrical conductivity.[1,2] For years, the alloy of choice for electroplating electronic components has been tin-lead (Sn-Pb) alloy.[2,3] However, since the Reduction of Hazardous Substances (RoHS) directive came into effect in Europe and Asia on July 1, 2006, requiring the removal of any lead content from electronic devices as a result of the toxic nature of Pb, a number of additional research studies for comparable electroplating alternatives have been conducted globally.[3] Because of its excellent conductivity and resistance to oxidation and corrosion, pure Sn was selected as an alternative choice for coating electronic components.[3,4] However, pure Sn has the tendency to spontaneously grow electrically conductive Sn whiskers during storage. A Sn whisker is usually accepted as a pure single-crystal Sn with filament or hair-like structure grown directly from the electroplated surface. Growth of metal whiskers on electroplated surfaces has been an industrial issue and unique metallurgical phenomenon for many years.[4–9] A metal whisker is highly conductive, and can cause short circuits in electronic components, which is a very significant reliability problem.[4,8] Damage caused by metal whisker growth is reported in highly critical applications such as aircraft, spacecraft, satellites, biotechnology products, military weapons systems, and recently automotive parts.[4,8] It is very hard to predict the period for the growth of whiskers, which can vary from hours to years.[4] They are also naturally very strong, grow by addition of material at their base rather than at their tip, and appear to be a local response to

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developed compressive residual or external stress in the film.[10–12] Literature reports that whisker formation and growth is hypothesized to be a multistage process including: whisker nucleation; growth part I, where atoms are added to the whisker; and growth part II, where long-range diffusion is required for enough atoms to be present to result in the observed whisker lengths.[13] Xu et al.[14] were among the first to report cross-section results of Sn whisker growth from matte Sn (grain size 2 μm to 3 μm) and satin bright Sn (grain size 1 μm) using a focused ion beam (FIB). The FIB images showed that the root of the tin whisker grain starts right above the already formed copper-tin intermetallic.[14] Similar microstructural behavior was also reported by Baudry and Kerros.[15] However, these FIB studies focus more on the evolution of the microstructure of the film and its relationship to the root of the whisker growth; the inner microstructure of the actual whisker is not discussed. Further, Lee and Lee linked Sn whisker growth with compressive stress as a function of storage time, stating that the stress was attributed to the formation of an irregular intermetallic compound (IMC) at the Sn grain boundaries.[12] Many additional theories have been proposed on whisker growth, including dislocation loops,[16,17] helical dislocations,[18] recrystallization,[19–21] grain boundary diffusion,[11,22] grain boundary fluid flow,[23] and residual stress gradients in Sn film.[24] Even so, the whole process is still not well understood, and the metal whiskering problem remains a significant reliability issue for the green electronics industry, especially as electronic devices become smaller in size. Careful examination and characterization of whisker cross-sections will provide additional insight into the process of whisker formation and growth. This paper reports on the internal microstructure and morphology of complex-shaped Sn whisker growth from an electroplated bright Sn layer on brass substrates exposed to ambient and 95% humid environment. The analyzed variables include surface and microstructure conditions of the film, as well as morphology and internal microstructure of the Sn whiskers using scanning electron microscopy (SEM) and FIB technology. The results provide detailed insights into the inner microstructure of complex-shaped Sn whiskers formed on Sn electroplated surface.

EXPERIMENTAL PROCEDURES

Flat, rectangular-shaped Copper Development Association nomenclature (CDA) 360 brass coupons with dimensions of 5.08 cm × 2.54 cm × 0.1 cm and area of approximately 13 cm^2 were used as substrates. The coupons were cut using an abrasive water-jet machine, wet-polished with #1000 silicon carbide (SiC) paper, wet-polished with sodium bicarbonate to remove any residual grit, rinsed with distilled water, and treated with 10% concentrated sulfuric acid prior to plating. Each coupon was taped on its back side with clear platers tape so that the plating was applied to one side only. The electroplating station was equipped with a high-power (HP 6235) supply connected in series to a digital volt–ohm meter (VOM) to precisely measure the applied cell current. A direct-current (DC) motor with an attached impeller was used to provide fluid agitation in the chemical bath during plating. All coupons were plated in 250 mL pure bright Sn bath (with approximately 0.5 μm grain size) at room temperature, with a Sn anode of 99.9% metal purity, and the current was set to 0.5 A according to the manufacturer's operating requirements. The deposition layer thickness of 5 μm was achieved by plating time and current density following literature recommendations.[7] Following the plating procedure, each sample was posttreated in a distilled water bath and neutralized in potassium hydroxide (KOH) with concentration of 0.010 mol/L. Each plated sample was dried with an air fan and placed inside an environmental chamber with 95% humidity. Five samples of the same kind were observed for a total aging period of 6 months. Additionally, for comparison, another set of the same samples were observed under regular ambient environment. Each sample was analyzed by SEM operated at 5 kV and 30 pA. The cross-sections were prepared using a dual-beam FIB. Prior to milling, a thin platinum (Pt) layer was deposited on the electroplated surface including the whisker's surface where cross-sectioning was performed. The samples were milled at a 52° tilt angle with a 30-kV gallium (Ga) ion beam operating at current of 30 pA. Initial trench milling of the sample was done at 20 nA, and the final face milling was done at 1 nA to 3 nA. Extra FIB images were taken with the Ga-ion beam at current of 11 pA. All samples were examined at the coupon center over an area of 20 μm × 20 μm. The metal composition of each sample was investigated by SEM using energy-dispersive spectroscopy (EDS). All measurements were performed with accelerating voltage of 15 kV, probe current of 1150 pA, and acquisition time of 30 s.

RESULTS AND DISCUSSION

SEM analysis of electroplated Sn surfaces exposed to 95% humidity for several weeks showed that they were populated with growth of very complex-shaped whiskers that had a composite geometry composed of straight, twisted, and bent parts in one single whisker. Figure 1a–c presents a few examples of whiskers with complex shapes formed on the electroplated surfaces over time. Previous studies report similar morphology of complex whisker growth when exposed to a controlled environment.[24–27] However, Jadhav et al.[28] report that a similar complexity of whisker growth is not present under a vacuum environment. It is further suggested that the presence of oxygen, water vapor, or other gas may play a role in the

nonuniform growth of whiskers such as those seen in Fig. 1, perhaps by surface oxide that retards or modifies the whisker growth.[28] In this study, the authors are interested in observing what is really the inner morphology of these complex-shaped whiskers, which is most likely stimulated by the influence of humidity. From the SEM scans in Fig. 1, it seems that each whisker growth starts with a smaller-scale hillock, which later grows into long extrusions, i.e., whiskers. It is interesting to note that the example in Fig. 1a has no straight parts along the body of the whisker, while in Fig. 1b, c long extrusions with growth up to 216 μm in length (Fig. 1c) have been formed. After some time, the direction of the growth of this straight part changes, and an additional straight (Fig. 1b) and convoluted part (Fig. 1c) grew in different direction, forming an end whisker with very complex shape. One question to ask is: What exactly causes this change of growth direction? Is it controlled by the root of the whisker and a result of some changes taking place in the realignment of grains in the microstructure of the Sn film, or simply an environmental influence that causes the tip of the whisker to take a different growth direction? Previous studies reporting on real-time monitoring of hillock and whisker growth suggested that two mechanisms such as stress-induced deformation by dislocation or by grain growth processes with grain boundary sliding in the film work together to produce the complicated evolution of whiskers seen on the surface.[28] This may not be the only reason, as whisker growth is very unpredictable, but the complex morphologies and rotation observed in whiskers would be most likely the result of plastic deformation taking place as well in the film. Furthermore, the complex morphology could suggest that Sn whiskers of this kind could be polycrystalline in nature and not single crystalline as widely accepted by the research community for the past several decades.[4] To further investigate the polycrystalline nature and define what is happening to the inner microstructure of the whisker with respect to the film, cross-sectional analyses were performed using FIB. The convoluted whisker in Fig. 1c and the film right below the whisker were cross-sectioned. The presented results are averages of five samples with similar convoluted whiskers observed in this study. Figure 2 presents a cross-section of the film right below the complex-shaped whisker growth in Fig. 1c. The cross-section of the electroplated surface at the root of the whisker shows formation of nonuniform IMCs at the film–substrate interface, and the growth density is not uniform on the entire surface (Fig. 2). From the same figure, it seems that the whiskers extrude exactly at the location where the Cu-Sn IMCs are uniformly concentrated. This behavior has been seen in studies of growth of whiskers with simpler shape.[14,29,30] The same Fig. 2 shows the existence of a clearly defined grain boundary between grains that extrude into whisker (Fig. 2b). Only some grain regions develop into whiskers. A cross-section of the inner microstructure of the straight and the convoluted parts of the Sn whisker in Fig. 1c shows distinct boundaries between sections along the body of the whisker (Fig. 3a, b). A higher-magnification image of the cross-sectioned area (Fig. 3a) between the straight and the convoluted parts of the whisker shows the presence of lateral growth along the body of the whisker. Lateral growth is also seen in Fig. 4, which shows magnification of the tip of the convoluted part from the same whisker reported previously in Fig. 1c, and the presence of distinct boundaries between regions are observed. Visual inspection of the inner microstructure further suggests that these complex-shaped whiskers are of polycrystalline nature. Recent transmission electron microscopy (TEM) results on the complex nature of hillocks revealed that, regardless of the complex surface formed, the hillock is still of single-crystalline structure; however, the study did not report on the nature of the complex-shaped whisker.[31] Further, the complex growth and rotation of grains has been attributed to the following factors: reorientation of the underlying grain boundaries, rotation of the underlying grains by formation of subgrain boundaries seen with TEM, or dislocations in the film.[28,31] Looking closely at the inner microstructure of the complex whisker, the authors believe that the dislocations in the material, nonuniformity in the stress surrounding the grains connected to nonuniform IMCs present, and reorientation of the underlying grain boundaries feeding material at the root greatly accelerated by humidity contribute to the formation of the complex-shaped whisker that could be polycrystalline in nature. The authors believe that complex-shaped whisker growth is greatly stimulated by humidity, as the presence of humidity accelerates oxidation of Sn compared with ambient conditions. Tin oxides dissolve preferentially along the grain boundaries of Sn and can act as impurities. The presence of impurities or any external content can cause realignment of grains and initiation of complex-shaped whiskers with parts that have nonuniform growth direction. This process can take place inside the morphology of the whisker over time and not just inside the film itself. A study reporting composite whisker formation of Cr and Sn suggests that whiskers are highly nonuniform structures in terms of chemical composition.[32] Additionally, debris and contamination could also contribute to the multiple elements present in the whisker growth, which can lead to polycrystalline growth. However, to accurately confirm the polycrystalline nature of these complex-shaped Sn whiskers, TEM analyses need to be performed in future studies. This study has provided a detailed inner microstructure of the whisker itself, which gives additional insights into the shape and orientation of complex whiskers formed under humid environment.

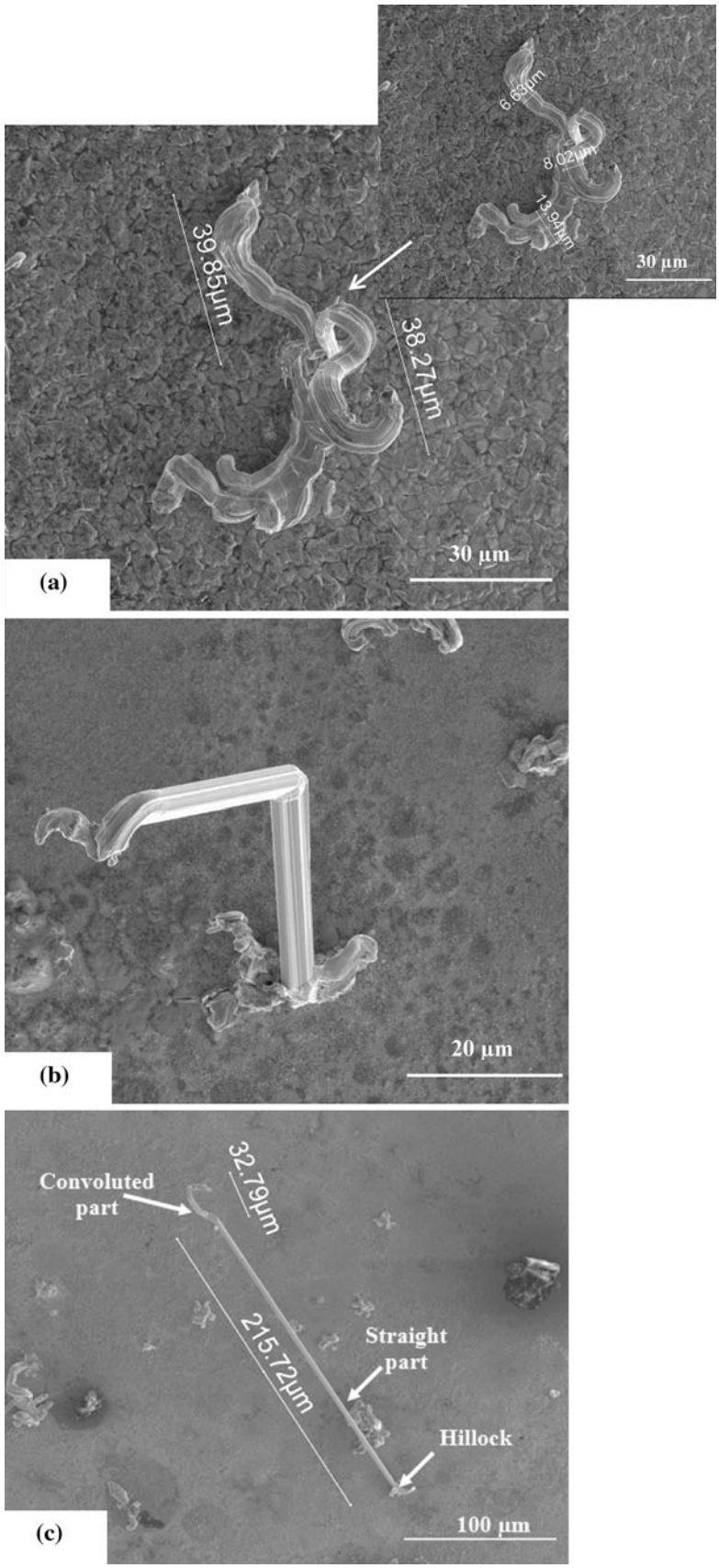

Fig. 1. (a–c) SEM scans of whisker growth with various morphologies from electroplated Sn surfaces.

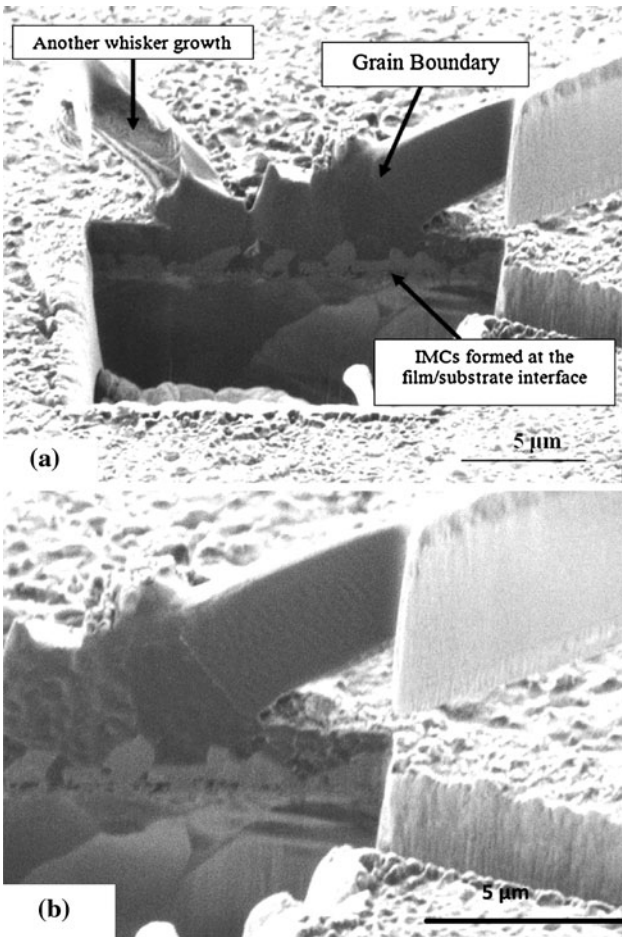

Fig. 2. (a) FIB cross-section of the film microstructure right below the growth of complex-shaped Sn whiskers. (b) Higher magnification showing the grain boundary on the right side of scan under (a).

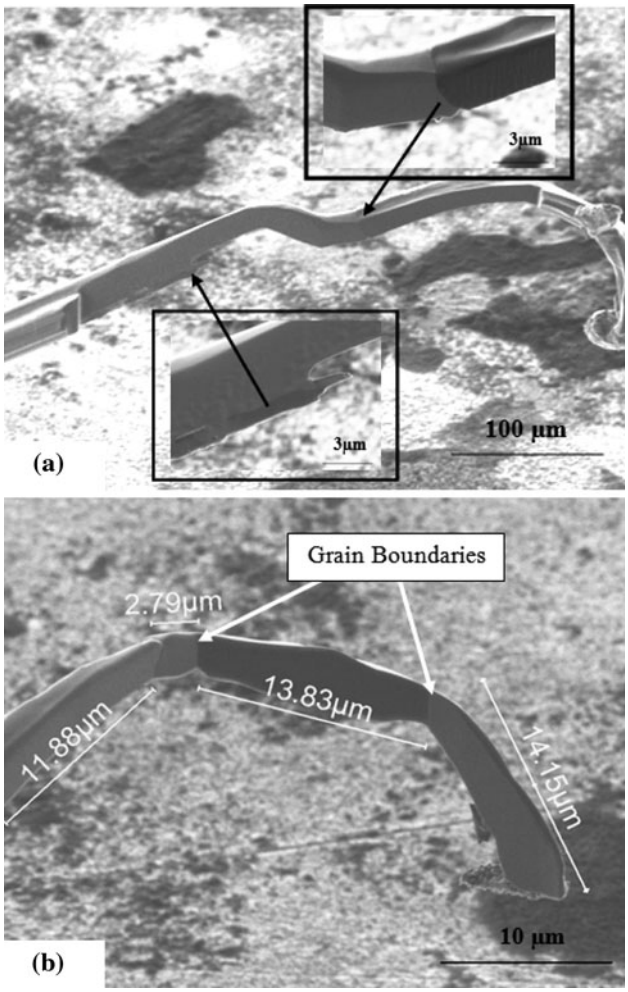

Fig. 3. (a, b) FIB cross-section of convoluted Sn whisker growth showing the polycrystalline structure of the whisker.

CONCLUSIONS

This study reports on the inner microstructure of complex-shaped Sn whiskers grown from electroplated bright Sn surfaces that could also be of polycrystalline nature. Visual inspection of the FIB cross-section of the whisker shows highly distinct boundaries between segments along the body of the whisker, which suggests polycrystalline nature. Additional TEM results are necessary to validate the presence of polycrystalline whiskers. Complex-shaped whiskers are believed to usually grow in controlled environments in this study, i.e., the humid environment. Additionally, it will be valuable to investigate how the change in the direction of the growth of the whisker takes places, why some grains develop into convoluted long whiskers while others remain at the hillock stage over the same time period, and whether a matte Sn film with larger grains will form whiskers of the same nature as reported herein. Many theories and assumptions concerning what causes the change of growth direction reported in this study by the author and

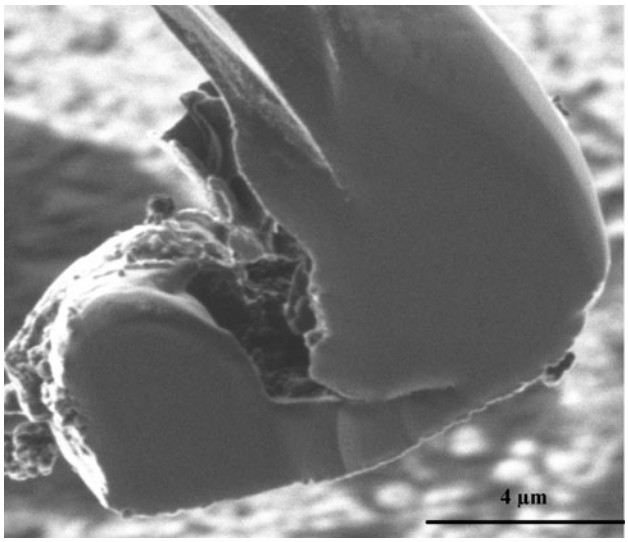

Fig. 4. Magnified SEM image showing the convoluted section of the whisker in Fig. 1c.

from literature review will be useful for quantification and experimental validation in future work.

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