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# TEM and SEM Analysis for Formation Mechanism of Tin Whiskers

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**Abstract.** Close observation with a transmission electron microscope (TEM) and a scanning electron microscope (SEM) was performed for the growth process of tin (Sn) whiskers on lead (Pb)-free Sn-plating. Whiskers were formed on a Sn layer plated on Cu/polyimide flexible substrate. The whisker was found to be of a single crystal and have a characteristic "Y"-shaped grain boundary structure at its root. The growth process of a curling whisker was successfully observed in a continuous way in SEM. TEM observation revealed that the curling whisker had a single crystallographic orientation irrespective with its external shape. these microstructures indicate that the rearrangement of dislocations plays an important role in the growth process of whiskers.

## Introduction

Due to restriction of hazardous substances for environmental concerns, removal of lead (Pb) from electric and electronic devices and components is needed. However, whiskers grow up spontaneously from a surface of Pb-free plating and solder, and they might cause a serious problem such as short circuit. There have been a lot of researches on the growth of Sn-whiskers in order to understand the origin of whisker growth and to establish a technique of prevention form the whisker growth [1-4]. These studies have shown that the whisker growth is initiated by compressive stress inside the Sn layer. The whiskers are classified into several types according to the origin of compressive stress;

(1) external stress type; caused by stresses due to factitious actions such as pressing, bending, scratching etc.
(2) internal stress type; caused by stresses due to not-factitious actions such as difference in thermal expansion and/or formation of precipitates, etc.
(3) surface corrosion type; caused by stresses due to the formation of oxide-phase regions
(4) welding type; caused by stresses in the lamellar structure of (Sn+Al) eutectic formed after welding
(5) others.

Recently a difference in stress in the Sn-layer is thought to be an important factor for whisker growth rather than a compressive pressure [5,6] . But the detail mechanism of growth is not clarified yet so far.

In this study, close observation of Sn whiskers with a transmission electron microscope (TEM) and a scanning electron microscope (SEM) was performed for the growth process of whiskers. The growth mechanism is discussed for whiskers of external stress type with a special attention to the grain boundaries in the Sn-layer.

## Experimental

The following two specimens were used in the present experiments.

*Sample-1*; Sn/Cu/polyimide(PI)

A flexible electrical connector was prepared. The pin-plates of the connector were made of copper (Cu) plated polyimide (PI) flexible substrates on which a pure Sn-layer about 10 μm thick was electrodeposited. They were inserted into a connector set and kept at room temperature for 11 days, as shown in Fig. 1.

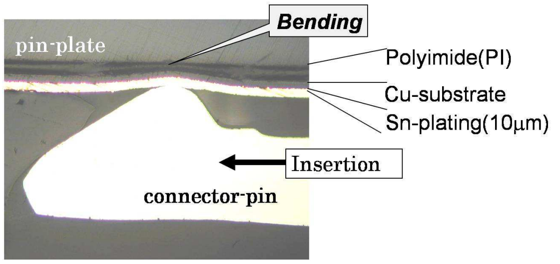


Fig. 1 Cross sectional optical microscope image of a fluxible connector-set It should be noted that the pin-plate is bent by a mechanical contact.

*Sample-2*; Sn/Fe-42Ni

A substrate of Fe-42Ni 0.2 mm thick was prepared [7]. A matte pure Sn layer about 10 μm thick was electroplated on the substrate. A specially designed device was made for in-situ observation with a SEM as illustrated schematically in Fig. 2. The device with the sample was set in a SEM (JSM-5300LV / a digital image capture (JEOL)), and observation was carried out continuously at room temperature for 39 days at the longest.

Thin foil specimens for TEM were made using a focused ion beam (FIB) mill with a micro-sampling unit (FB-2000K, Hitachi). TEM observation was carried out with JEM-2000EX/T (JEOL) and Technai-20F (Philips).

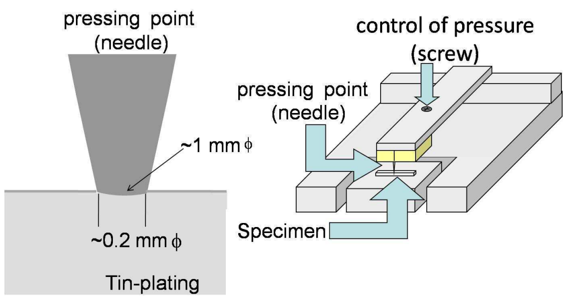


Fig. 2 Schematic drawing of a device for in-situ observation of whisker growth in SEM

## Results and Discussion

*Sample-1*; Sn/Cu/polyimide(PI)

Fig. 3 shows a SEM image of a pin-plate of connector after removing from a connector-set. On the center of pin-plate there is a hollow formed by a mechanical contact. It can be seen that whiskers are formed preferentially in a limited rectangular region and some whiskers are lined up normal to the direction of insertion. The inhomogeneous distribution of whiskers are thought to reflect the stress distribution introduced by the bending of pin-plate associated with its insertion, and the lining-up of whiskers might result from their growth along micro-cracks on the surface of pin-plate.

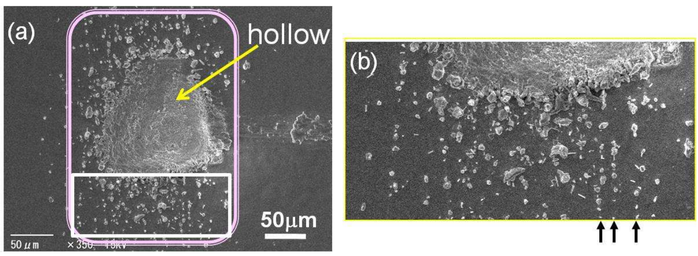


Fig. 3 SEM images of pin-plate of Sn/Cu/PI
The pin-plate was kept for 11 days at room temperature after inserted in a flexible connector set.
(a) Whiskers sre formed in the rectangular region.
(b) Enlarged image; Whiskers are lined-up, as shown with arrows

Fig. 4 shows a scanning ion microscope (SIM) image taken with a FIB mill. There formed are whiskers of various shape-types; filamentary, columnar and nodular. A columnar whisker marked with an oval was picked up and thinned to be a TEM thin foil specimen. Fig. 5 shows a cross-section TEM-image of the whisker and a region near the root of it. One can see that the whisker is made of a single crystal of $\beta$-Sn. The whisker lies on a vertical grain boundary in a Sn-layer so that a characteristic "Y" shape grain-boundary structure is formed. The formation of "Y"-shaped boundary

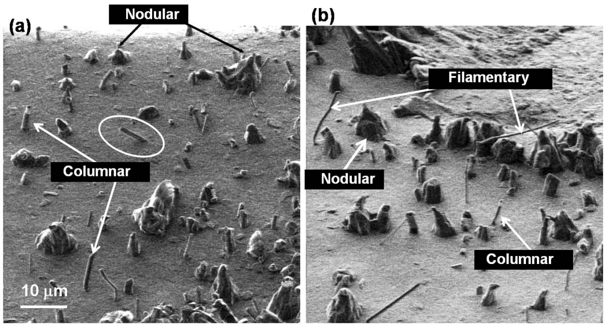


Fig. 4 FIB-SIM images of whiskers on a Sn layer on Cu/PI
Various types of whiskers are observed.
The whisker in a circle was picked up and observed with TEM as shown in Fig. 5

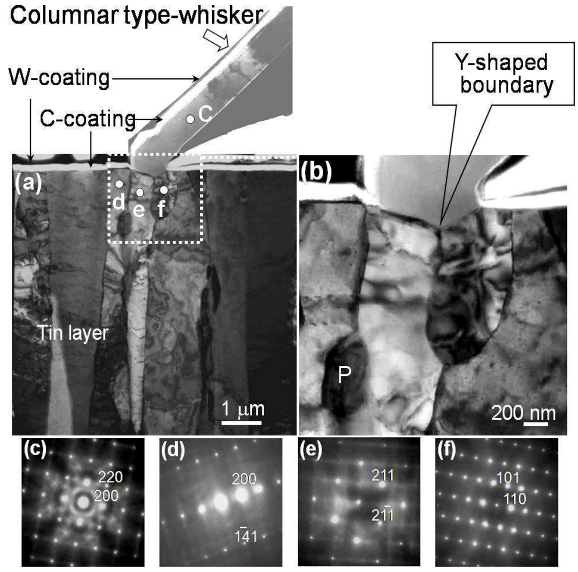


Fig. 5 TEM image of a whisker grown on Sn/Cu/PI
Electron diffraction patterns indicate that the whisker is of a single crystal and the crystal grains do not have a particular relationship in crystallographic orientation with one another.
(b) Enlarged image of the region of the dotted rectangle in (a).
The whisker has a characteristic "Y"-shape grain boundary structure.
Domain P in (b) is a precipitate of $Cu_6Sn_5$ .

structure is a common phenomenon among all types of whiskers. It can be noted that the size of Sn-grains is a little smaller under the whisker in comparison with that in the region nearby. This seems to indicate as if a whisker grew up at the most stressed region. But it is not the case. It is already known that a whisker can start to grow first from a region without grain boundaries [7]. Such a distribution of grain boundaries as in Fig. 5 results from a rearrangement of dislocations that were inserted by deformation in association with application of compression. A whisker sprouts from a stress relieved position in a highly stressed region. In this connection, it was reported that the grain size of Sn grains decreases first and then increases with growth of whiskers [7]. The changes in grain size result from deformation and recrystallization, respectively. The mechanism of whisker growth will be described in detail elsewhere.

*Sample-2*; Sn/Fe-42Ni

In many cases, microstructures have been observed for a specimen with well-grown whiskers so far. Microstructural changes in the early stage should be observed to clarify the growth process. Fig. 6 shows an example of the in-situ observation of whisker growth in SEM. One can see that a whisker grows with continuous curling. It should be noted that the whisker come up a little far from the position at which a compressive stress is applied with a needle. This clearly indicates that a whisker does not sprout at the position with a stronger stress. This phenomenon reveals again that a whisker does not come up by pushing by the compression applied, but by mass-transport to the region of lower stress.

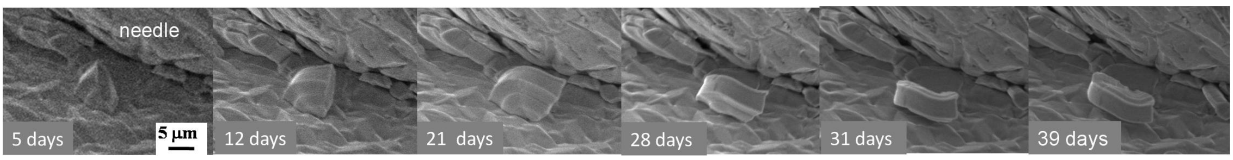


Fig. 6 Series of SEM images of growing whisker
The Sn plate was set to a devise illustrated in Fig 2 and put in SEM.
The curling whisker starts to grow at a certain distance from the position at which a stress is applied with a needle.

Fig. 7 shows a cross sectional TEM image of the curling whisker. It should be noted that there is no grain boundary observed in the whisker and it has a single crystallographic orientation irrespective to its external shape. The origin of bending was discussed by several investigators [8,9] and considered to be due to uneven stress. It might be true, but the microstructure without grain boundaries cannot be expected from their growth model. In order to form a curl whisker of a single crystal, the growth orientation should be tilted in accordance to the bending of whisker. The tilting in orientation is explained by a slipping on the grain boundary at the root of whisker or spontaneous recrystallization in the root-region of whiskers [10]. Behavior of dislocations is very important in the formation mechanism of whiskers in either case.

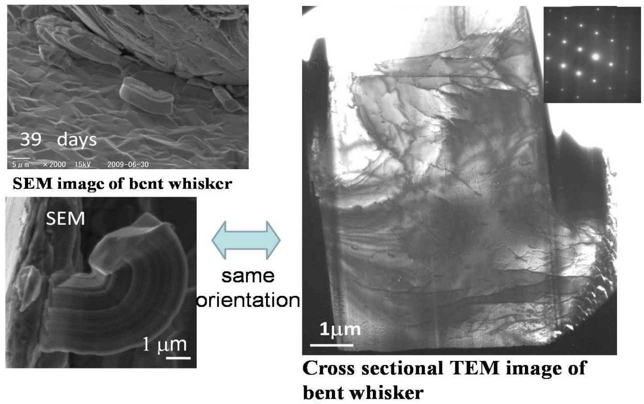


Fig. 7 Cross sectional TEM image of a curling whisker
A curling whisker grown for 39 days was picked up with a microsampling unit and thinned with an FIB mill.
It should be noted that there is no grain boundary in the whisker.

**Summary**

We have studied the microstructures in Sn-layers for the formation mechanism of whiskers. In this paper, some of the experimental results on whiskers of an external stress type were briefly described and summarized as follows.

(1) A whisker is of a single crystal of $\beta$-Sn.
(2) A whisker has a characteristic "Y"-shaped grain boundary structure at the root. The crystal grains under a whisker were small in size compared with that in the region nearby. These are considered to result from rearrangement of dislocations during the growth.
(3) The growth process of a whisker was successfully observed in a continuous way in a SEM.
(4) A curling whisker has a single crystallographic orientation irrespective to the external shape. The tilting of growth direction is explained by slipping of crystal grain on the boundary or spontaneous recrystallization.

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**References**

[1] B.-Z. Lee and D. N. Lee: Acta Metallur. Vol.46 (1998), p.3701.
[2] G. T. Galyon and L. Palmer: IEEE Trans. Electro. Packag. Manuf. Vol. 28 (2005), p. 17
[3] K. S. Kim, C. H. Yu, S. W. Han, K. C. Yang, J. H. Kim: Microelectronics Reliability Vo. 48 (2008), p. 111.
[4] N. Jadhav, E. J. Buchovercky, L. Reibold, S. Kumar, A. F. Bower and E. Chason: IEEE Trans. Electro. Packag. Manuf. Vol. 33 (2010), p. 183.

[5] C. Xu, Y. Zang, C. Fan and A. Abys: IEEE Trans. Electr. Packag. Manuf. Vol. 28 (2005), p.31.

[6] K. Kato, H. Akahoshi, M. Nakamura, T. Terasaki, T. Iwasaki, T. Hashimoto ans A. Nishimura: IEEE Trans. Electr. Packag. Manuf. Vol. 33 (2010), p.165.

[7] N. Kuwano, A. Jinnouchi, S. Horikami, N. Hirokado and H. Sosiati; Mater. Trans. Vol. 51, (2010), p.1741.

[8] J. Smetana: IEEE Trans. Electro. Packag. Manuf. Vol. 30 (2005), p. 11.

[9] C.-M. Chen and Y.-J. Chen: Mater. Lett., Vo. 63 (2009), p.1517.

[10] N. Kuwano : unpublished.

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10.4028/www.scientific.net/AMR.545

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**DOI References**
[1] B. -Z. Lee and D. N. Lee: Acta Metallur. Vol. 46 (1998), p.3701.
doi:10.1016/S1359-6454(98)00045-7

[2] G. T. Galyon and L. Palmer: IEEE Trans. Electro. Packag. Manuf. Vol. 28 (2005), p.17.
doi:10.1109/TEPM.2005.847443

[3] K. S. Kim, C. H. Yu, S. W. Han, K. C. Yang, J. H. Kim: Microelectronics Reliability Vo. 48 (2008), p.111.
doi:10.1016/j.microrel.2007.01.088

[4] N. Jadhav, E. J. Buchovercky, L. Reibold, S. Kumar, A. F. Bower and E. Chason: IEEE Trans. Electro. Packag. Manuf. Vol. 33 (2010).
doi:10.1109/TEPM.2010.2043847

[6] K. Kato, H. Akahoshi, M. Nakamura, T. Terasaki, T. Iwasaki, T. Hashimoto ans A. Nishimura: IEEE Trans. Electr. Packag. Manuf. Vol. 33 (2010), p.165.
doi:10.1109/TEPM.2010.2045384

[7] N. Kuwano, A. Jinnouchi, S. Horikami, N. Hirokado and H. Sosiati; Mater. Trans. Vol. 51, (2010), p.1741.
doi:10.2320/matertrans.MJ201021