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Technical Report

Cross-Sectional Transmission Electron Microscopy Sample Preparation of Soldering Joint Using Ultramicrotomy

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Solder/electroless nickel immersion gold (ENIG) joint sample which is comprised of dissimilar materials with different mechanical properties has limited the level of success in preparing thin samples for transmission electron microscopy (TEM). This short technical note reports the operation parameters for ultramicrotomy of solder joint sample and TEM analysis results. The solder joint sample was successfully sliced to 50~70 nm thick lamellae at slicing speed of 0.8~1.2 mm/s using a boat-type 45° diamond knife. Ultramicrotomy can be applied as a routine sample preparation technique for TEM analysis of solder joints.

Key Words: Sn-Ag solder, Electroless nickel immersion gold plating, Interfacial reaction layer, Ultramicrotomy, Transmission electron microscopy

INTRODUCTION

Many studies have reported the interfacial reaction of Sn-Ag/electroless nickel immersion gold (ENIG). Various analysis techniques, such as scanning electron microscopy, electron probe microanalyzer, and transmission electron microscopy (TEM), have been used to examine interfacial reaction. Among them, TEM equipped with analytical attachments offers the most complete tool for characterizing the formation of intermetallic compounds (IMCs) at the interface. However, solders are typically soft and ductile, while IMCs tend to be quite hard and brittle, which has limited the level of success in preparing TEM samples.

We utilized ultramicrotomy since it might offer a reasonable and relatively quick alternative in this case. Ultramicrotomy has been used for the preparation of TEM specimens from biological materials and extending a range of materials including polymers, coating layer, particles, and even metals (Becker & Bange, 1993; Glanvill, 1995; Howell et al., 1995). TEM sample making to section hard materials by ultramicrotomy are not new. Besides, it has been traditionally considered that the mechanical damage during sectioning by diamond knife might hinder unambiguous specimen characterization. However, this technique also has many advantages such as no ion beam irradiation damage, no chemical mixing, no differential thinning rate and the ease of preparation of many serial sections with large, thin areas of uniform thickness in a relatively short time (Quintana, 1997). The increasing acceptability of ultramicrotomy in materials science is evidenced by a number of recent paper giving results for different hard materials (Hwang & Suganuma, 2003).

MATERIALS AND METHODS

The solder joint sample was prepared using conventional Sn-3.5wt.%Ag solder and Cu plate with ENIG surface finish. The solder balls were bonded to the ENIG substrate in a reflow process using rosin mildly activated (RMA) flux in a reflow machine (RF-430-N2; Japan Pulse Laboratory Co., Ltd., Japan) with a maximum temperature of 255°C for 1 minute.

TEM cross-sectional sample was prepared by ultramicrotomy (MT 7000; RMC Boeckeler, USA). The solder joint sample was fixed directly to the 'Flat Specimen Clamp' without embedding in epoxy resin (Fig. 1). The trimming process to provide a suitable cutting area was carried out slowly and very

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carefully because the glass knife for trimming was broken repeatedly due to a high hardness of solder joint sample. After trimming process, the sample was cut into 50~70 nm thick slices with 0.8~1.2 mm/s slicing speed using a boat-type 45° diamond knife (Drukker International, The Netherlands) filled with distilled water. The sections floating on the distilled water surface were collected onto 200 mesh copper grid. The microstructure and chemical composition were observed by TEM (JEM-2100F; JEOL Co., Ltd., Japan) equipped with energy dispersive X-ray spectrometer (EDS).

RESULTS AND DISCUSSION

Fig. 2 shows a typical low magnification TEM micrograph of as-sectioned slices from Sn-3.5Ag/ENIG joint sample prepared by ultramicrotomy technique. These slices tended to crack and bend during sectioning but this did not become a problem in characterization such as size measurement, and determination of crystal structure, crystallinity and chemical composition. The observing area was as large as a few tens of micrometers.

Fig. 3 shows a high magnification TEM micrograph of a sectioned slice and the nano-beam electron diffraction (NBED) patterns obtained from interfacial reaction layers. Based on the TEM image contrast, it was apparent that the interfacial reaction layers between a predominant phase

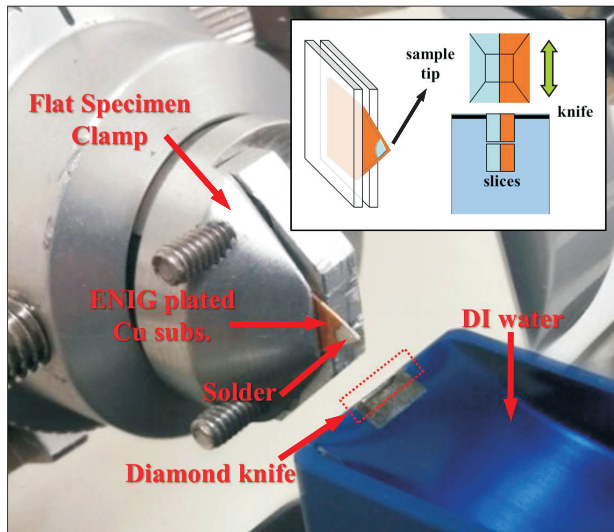

Fig. 1. Sample holder of ultramicrotome. The solder joint sample was fixed directly to the 'Flat Specimen Clamp' without embedding in epoxy resin. The interface of solder/ENIG plated substrate was aligned perpendicular to the diamond knife edge.

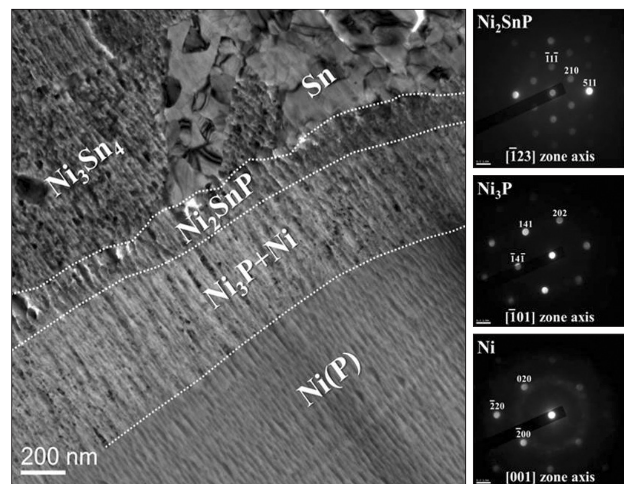

Fig. 3. High magnification TEM micrograph of a sectioned slice and NBED patterns obtained from interfacial reaction layers. Although there was wrinkling, ultramicrotomy provides contamination free sections with large, thin area of uniform thickness, which is suitable for the TEM study using electron diffraction and X-ray microanalysis.

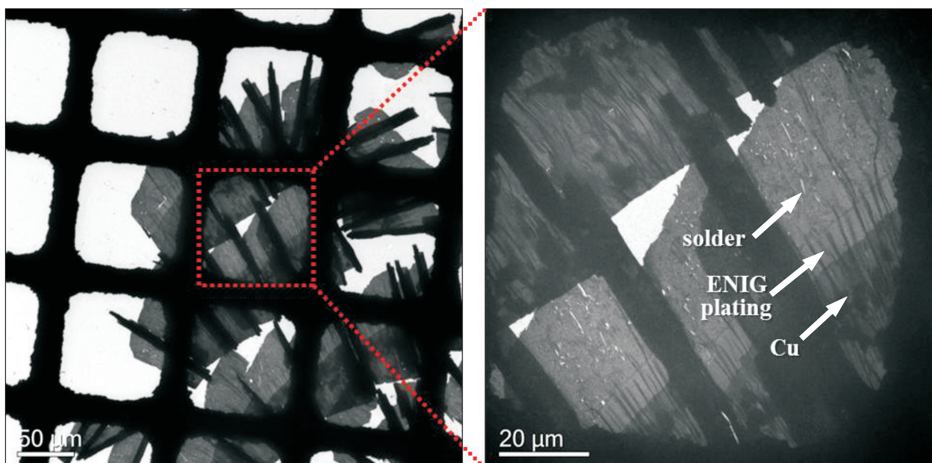

Fig. 2. Low magnification TEM micrographs of as-sectioned slices from Sn-3.5Ag/ENIG joint sample prepared by ultramicrotomy technique. The sections were collected onto 200 mesh copper grid. The observing area was as large as a few tens of micrometers.

of Ni_3Sn_4 IMC and the electroless Ni (P) substrate were composed of two more reaction layers rather than one. The lower layer next to the electroless Ni (P) exhibited a light gray contrast with black spots, while the upper layer next to the Ni_3Sn_4 IMC showed a dappled-gray contrast. Although there was wrinkling in the sections due to the mechanical stress during sectioning by diamond knife, it did not affect the TEM study using electron diffraction and X-ray microanalysis. Identification of phases formed was performed by using NBED and EDS analysis. Diffraction pattern indexing confirmed each layer which was distinguished by the difference in TEM image contrast: the Ni_3Sn_4 IMC layer, Ni_2SnP layer, and final layer composed of Ni_3P and Ni nano-crystallites. Detailed TEM analysis results of Sn-Ag/ENIG solder joint sample prepared by ultramicrotomy were reported elsewhere (Kang et al., 2008, 2011).

CONCLUSIONS

In summary, ultramicrotomy technique is suitable to prepare thin sections of Sn-Ag solder/ENIG joint samples. Even though there is a risk of breaking diamond knife during sample preparation due to the high hardness of metal component in the sample, ultramicrotomy enables the relatively rapid preparation of thin sectioned slices for the TEM analysis. In addition, this technique provides contamination free sections with large, thin areas of uniform thickness.

CONFLICT OF INTEREST

No potential conflict of interest relevant to this article was reported.

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