TEM observation of interfaces in a solder joint in a semiconductor device

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Abstract

Microstructure of a joint between a Pb–Sn eutectic solder and an electroless Ni–8 mass% P has been examined using transmission electron microscopy. Four layers, i.e. Ni3Sn4, Ni48Sn52, Ni2SnP and Ni–20 mass% P, are formed between the solder and the electroless Ni–8 mass% P. Among them, Ni48Sn52 and Ni2SnP were found for the first time in a solder joint. Spherical voids are formed at the interface between Ni48Sn52 and Ni2SnP, and columnar voids are formed at the interface between Ni2SnP and Ni–20 mass% P. From the analysis of the migration of the respective interfaces observed during in situ heating experiments, it is concluded that these voids are Kirkendall voids formed due to the difference in diffusivity of Ni across the interfaces. Fracture takes place at either of those interfaces during a dropping test.

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1. Introduction

In semiconductor devices, it becomes increasingly important to evaluate the reliability of not only the device itself but also the connection between the circuit board and the device terminals. This is especially true for semiconductor device packages developed in response to the needs of high-density mounting such as ball-grid arrays (BGA), which have solder balls arranged in a grid pattern on the external terminals (pads) of the package. The surface of the external terminals is metallized to provide adhesion of the solder, passivation and a diffusion barrier [1]. Such a metallization, called under bump metallization (UBM), generally consists of a multilayer structure of Cu, Ni (–P) and Au.

Many studies have been carried out on the reactions between a eutectic Pb–Sn solder and UBM using scanning electron microscopy (SEM) [2–4], X-ray microanalysis (XMA) [2–5], X-ray photoelectron spectroscopy (XPS) [5], X-ray diffraction (XRD) [3–5] and Auger electron spectroscopy (AES) [3]. Unfortunately, the spatial resolution of these methods is low. Transmission electron microscopy (TEM) provides a spatial resolution high enough for the structure of layers in a joint to be observed directly. Therefore, TEM observation should be a very important method to supplement to XMA, XPS, and AES analysis of a solder joint.

TEM observation of the solder joint on electroless Ni–P UBM has been carried out recently by Jang et al. [6], but their observation was not comprehensive enough for the detailed structure of the solder joint to be clarified. The aim of this research is to identify, by a comprehensive TEM observation, the alloyed layers formed at a solder joint on an electroless Ni–P UBM on a Cu interconnect in a plastic BGA package. Furthermore, in order to get more straightforward insight into the mechanism of evolution of microstructure of the joint during soldering, motions of the different interfaces in a solder joint have been observed directly using an in situ heating experiment in a transmission electron microscope.

2. Experimental procedures

2.1. Static observation

Samples were prepared from a device package on a printed circuit board for an interposer. The approximate dimensions and the layout of the terminals of the package are shown in Fig. 1(a). Three different types of sample, i.e. samples A, B and C, were used. Sample A, shown in
Fig. 1(b), was a sample prior to solder ball mounting, and Sample B, shown in Fig. 1(c), was the one after solder ball mounting. Sample C, shown in Fig. 1(d), was the same as Sample B but fractured by a dropping test. As shown in Fig. 1(b), the UBMs are composed of 18 μm thick Cu, 5 μm thick Ni–8 mass% P and nominally 0.56 μm thick Au films. The Cu and Ni–8 mass% P films were plated by electroless deposition. The Au film was formed by substitution plating. The temperature profile for the soldering process is shown in Fig. 2. The peak temperature is 245 °C and the entire process took approximately 7 min. The eutectic temperature of the solder, i.e. 183 °C, is exceeded for approximately 2 min.

Thin foil specimens for TEM observation were prepared using a focused ion beam (FIB) processing machine (Hitachi FB-2000). TEM observation was carried out in a
Hitachi 9000NAR at an accelerating voltage of 300 kV. Elemental analysis was also carried out on samples A and B using energy dispersive X-ray (EDX) out using an electron beam probe with 5 nm in diameter.

Although the thinned area was increased to approximately 15 µm in order to reduce noises from the surrounding areas, the results are only of semiquantitative validity.

2.2. In situ observation

A specimen used for the in situ experiment is shown in Fig. 1(e). On a Si substrate, consisting of a 500 nm thick Ti layer and a 400 nm thick Cu layer were deposited successively by RF sputtering, followed by coating with an approximately 2 µm thick electronless Ni–8 mass% P. Onto this UBM a eutectic Pb–Sn approximately 10 µm in thickness was electrolytically deposited. Again, foil specimens for in situ heating experiment in a TEM were prepared using a FIB technique as described previously. In order to suppress the evaporation during melting, the whole specimen was coated with a polymerized hydrocarbon, the thickness being approximately 400 nm [7].

The foil specimens were examined in a Hitachi HU-1000D high-voltage electron microscope operated at an accelerating voltage of 1000 kV. Reactions between the Ni–P UBM and a eutectic Pb–Sn solder just above the eutectic melting point of the eutectic solder (183 °C) were observed and recorded continuously on a video tape.

3. Results

3.1. Static observation

3.1.1. Sample prior to solder ball joining (Sample A)

Fig. 3(a) shows a typical cross-sectional TEM micrograph of Sample A (i.e. before solder ball joining) at low magnification. Here, the topmost layer marked by W is a
protective tungsten layer applied to prevent damage to the sample during FIB thinning and has nothing to do with the structure of Sample A. Immediately underneath the tungsten layer is an approximately 0.5 \( \mu \text{m} \) thick layer, denoted by A. This layer was identified as polycrystalline Au by electron diffraction. Underneath the Au layer is Ni–8 mass% P layer (denoted by N) with relatively uniform contrast, and the corresponding diffraction pattern is shown in Fig. 3(b). This diffraction pattern is indexed to be Ni. Fig. 3(c) shows a dark-field image, at a higher magnification, produced by using part of the diffraction rings in Fig. 3(b), and the white spots as indicated by arrows in Fig. 3(c) correspond to single grains of Ni crystals. EDX analysis showed that the composition of this thin layer was approximately Ni–8 mass% P. Since the diffraction pattern shown in Fig. 3(b) shows no evidence of Ni\(_3\)P, it is reasonable to assume that P is soluble in the Ni matrix. The cross-sectional structure is shown schematically in Fig. 9(a).

### 3.1.2. Sample after solder ball joining (Sample B)

Fig. 4 shows the solder joint of Sample B (i.e. after the solder mounting) at a very low magnification, together with the diffraction patterns. The whole area from the eutectic Pb–Sn solder all the way down to the Cu substrate is thinned uniformly and part of the printed circuit board survives. Fig. 5 shows the interface between the eutectic Pb–Sn solder and the Ni–P UBM at a higher magnification. A typical example of elemental analysis by EDX near the interface (not the same area as shown in Fig. 5) is shown in Fig. 6. Au was not detected at any point by EDX elemental analysis.
analysis. This indicates that Au, which sat on top of the Ni–P layer before soldering, diffused into the solder and/or Ni–P layer during the mounting of a eutectic solder ball. Furthermore, Pb was detected only in the Pb–Sn eutectic solder.

Figs. 5 and 6 suggest that, in Sample B, four layers exist between the Pb–Sn eutectic solder and the Ni–P plating. The diffraction patterns from the respective layers are shown in Fig. 7. The detail of the analysis of the diffraction patterns are summarized in Table 1. The phases of the four layers were identified by EDX analysis and diffraction as follows:

Layer 1. Around a few micrometer thick, the grain size is around 1 \( \mu m \). Diffraction from layer 1 (Fig. 7(a)) is identified as \( \text{Ni}_3\text{Sn}_4 \). The result of EDX analysis gives a composition of 47 mass% Ni and 53 mass% Sn. This composition is lower in Sn than that of the ideal \( \text{Ni}_3\text{Sn}_4 \) (i.e. 72 mass% Sn). However, the EDX result obtained in an FIB-fabricated TEM specimen is only of semiquantitative validity. Thus, it can be said that the EDX result and diffraction analysis are in a reasonable agreement with each other. Furthermore, since neither Pb nor P is detected, it may be concluded that layer 1 consists of \( \text{Ni}_3\text{Sn}_4 \) alone.

Layer 2. Several hundred nanometre thick, the grain size is around 1 \( \mu m \). Diffraction from layer 1 (Fig. 7(a)) is identified as \( \text{Ni}_3\text{Sn}_4 \). The result of EDX analysis gives a composition of 47 mass% Ni and 53 mass% Sn. This composition is lower in Sn than that of the ideal \( \text{Ni}_3\text{Sn}_4 \) (i.e. 72 mass% Sn). However, the EDX result obtained in an FIB-fabricated TEM specimen is only of semiquantitative validity. Thus, it can be said that the EDX result and diffraction analysis are in a reasonable agreement with each other. Furthermore, since neither Pb nor P is detected, it may be concluded that layer 1 consists of \( \text{Ni}_3\text{Sn}_4 \) alone.

Layer 3. Around 200 nm thick. In addition to Ni and Sn, P is detected. The composition is 5 mass% P, 27 mass% Sn and 68 mass% Ni. Diffraction pattern as shown in Fig. 7(c) consists of Debye rings superimposed with networks of spots. This indicates that layer 3 consists of fine polycrystals and coarser crystal grains. The size of the selected area aperture used in taking the diffraction patterns is approximately 200 nm. It is, therefore, reasonable to assume that the size of the coarse crystals is also around 200 nm or larger. From these networks of diffraction spots, the coarse crystal is identified as a ternary intermetallic compound \( \text{Ni}_2\text{SnP} \), as shown in Fig. 7(c) and (c'). On the other hand, the Debye rings from the polycrystalline structure could not be identified from the JCPDS-ICDD cards.

Layer 4. Several hundred nanometres thick. Sn is no longer detected but a high level of P and Ni is detected by EDX. The composition is approximately Ni–20 mass% P. The diffraction pattern consists of many Debye rings, as shown in Fig. 7(d). The intensities of the rings show strong anisotropy, which indicates that layer 4 has a strong texture. It was found impossible to index all the Debye rings by assuming a mixture of \( \text{Ni}_3\text{P} \) and \( \text{Ni}_2\text{P} \). According to the phase diagram of the Ni–P system [9], Ni–20 mass% P corresponds to a mixture of \( \text{Ni}_{12}\text{P}_3 \) and \( \text{Ni}_3\text{P} \). In this case, according to the JCPDS-ICDD cards, \( \text{Ni}_{12}\text{P}_3 \) has so many Debye rings that it was practically impossible to identify crystal structures of layer 4.

In Fig. 5, layer 4 (Ni–20 mass% P) appears brighter than the others. Fig. 8 shows the microstructure near layer 4 at a higher magnification. Within layer 4, there exist many white
stripes perpendicular to the joint interface. Furthermore, in layer 3 (Ni$_2$SnP), chains of white dots exist near the interface between layers 2 (Ni$_{48}$Sn$_{52}$) and 3 (Ni$_2$SnP). From these observations, the cross-sectional structure of Sample B can be shown schematically as in Fig. 9(b).

### 3.1.3. Sample after fracture (Sample C)

Fig. 10(a) and (b) shows electron micrographs, at low and high magnifications, respectively, of the external terminal side of Sample C. Main fracture took place parallel to the eutectic solder/UBM interface and the solder balls were lost during the dropping test. The vertical crack shown in Fig. 10(a) is a secondary fracture. Fig. 10(b) shows that the main fracture must have taken place at either an interface between layers Ni$_{48}$Sn$_{52}$ and Ni$_2$SnP or an interface between Ni$_2$SnP and Ni–20 mass% P. From Fig. 8 it is evident that, near these interfaces, many voids are formed. Thus, it is natural to assume that the cracks have initiated at and propagated through these voids.

## 3.2. In situ observation

Fig. 11(a) and (b) compares microstructure of an interface before and after joining by the in situ heating experiment. Since virtually no reaction takes place between the electroless Ni–8 mass% P and the underlying UBM consisting of Ti and Cu, the absolute positions of the interfaces between the eutectic Pb–Sn and the electroless Ni–8 mass% P can be determined very accurately. It is clear that, after joining, the electroless Ni–8 mass% P layer reduces its thickness by 0.4 mm.

Fig. 12 shows change of microstructure of an interface on further heating (carried out on a different specimen). It is clear that Ni–20 mass% P (layer 4) grows significantly on further heating. Vertical curtain-like contrasts are due to artefacts caused by FIB thinning and have nothing with the actual microstructure. It is noted that points as A and B are fixed, so that, again, the absolute positions of the respective interfaces can be determined accurately.

## 4. Discussion

### 4.1. Structure of the Ni–P film deposited in electroless plating (Sample A)

The composition of the electroless deposited Ni–P plating film has been studied using XRD by Zieheke et al. [10] and Brenner [11], and using electron diffraction by Graham et al. [12,13]. They concluded that this Ni–P must consist of Ni$_3$P microparticles and Ni matrix and that Ni$_3$P particles are too small to be detected by XRD. Mei and Callery [5] have shown that a P-rich phase exists near a Ni–P/Au interface before soldering. Puttlitz [14] has shown that a Ni$_3$P-rich layer exists at the Ni–P/Au interface before soldering. In the present study, however, no definite evidence for the existence of fine Ni$_3$P particles was obtained. According to the phase diagram [9], Ni–8 mass% P consists of a mixture of Ni and Ni$_3$P. That Ni$_3$P was not detected even by TEM observation suggests...
that electroless plated Ni–8 mass% P is in a non-equilibrium condition.

4.2. Alloy layers formed by solder ball mounting (Sample B)

It was confirmed that four layers were formed between a Pb–Sn eutectic solder and a Ni–P plating during mounting of the solder balls. Table 2 summarizes the intermetallic compounds observed so far at interfaces between a eutectic Pb–Sn (or Sn) and Ni (or electroless Ni–P). General consensus is that Ni₃Sn₄ is the major intermetallic compound and that Ni₃Sn₂ and Ni₅Sn can be formed, depending on the condition under which the reaction takes place. In the present study, Ni₃Sn₄ is observed but neither Ni₃Sn₂ nor Ni₅Sn was detected. Instead, Ni₄₈Sn₅₂ and a ternary intermetallic compound Ni₃SnP are found to exist at the Pb–Sn solder/Au/electroless Ni–P interface. It is also noted that, although Ni₄₈Sn₅₂ is not cited by Masasalski et al. [9], it has been identified by Bhargava and Schubert [8]. Both Ni₄₈Sn₅₂ and Ni₃SnP are very thin (less than 500 nm), which must have made it very difficult to identify by methods other than TEM.

4.3. Sample after fracture (Sample C)

Lau and Pao [19] have analysed fracture surfaces of a solder joint after a dropping test and inferred that coarsening of grains of layers formed at the solder/Ni interface reduces the strength of the alloy layers and/or the interfaces. The present study shows definitely that fracture takes place at
interfaces between layers Ni₄Sn₅₂ and Ni₂SnP or between Ni₂SnP and Ni–20 mass% P. Many voids are formed in Ni₂SnP and Ni–20 mass% P. Mei and Callery [5] have detected high concentrations of P and Ni at an interface fractured in a dropping test. This is in good agreement with the present observation.

4.4. Formation mechanism of voids

Fig. 13 summarizes the results obtained by the present in situ heating experiments. The reaction can be summarized as follows.

(1) At the early stage of heating, Ni₃Sn₄ (layer 1) is formed by the solid-state reaction between Ni and Sn (Fig. 13(a)). Throughout the reactions, Pb does not participate in the reaction at all. On heating Ni₃Sn₄ starts growing. Eutectic solder/Ni₃Sn₄ interface keeps going up and both Ni₃Sn₄/layer 4 and layer 4/Ni(–8 mass% P) interfaces keep going down. In other words, interdiffusion of both Sn and Ni takes place. Sn must be supplied from the eutectic solder to the Ni₃Sn₄/layer 4 interface and Ni must be supplied from Ni–8 mass% P to the eutectic solder/ Ni₃Sn₄ interface across Ni₃Sn₄ (Fig. 13(b)). As a result, the concentration of Ni in the electroless Ni–8 mass% P adjacent to the eutectic solder/Ni(–8 mass% P) interface is reduced. Inversely, P is enriched in this area.

(2) Ni₃Sn₄/layer 4 interface is enriched in P to such an extent that the ternary intermetallic compound Ni₂SnP (layer 3) is formed (Fig. 13(c)). The growth of layer 3 is very sluggish. Also, according to elemental analysis shown in Fig. 6, Sn does not exist in layer 4. This indicates that diffusion of Sn in Ni₂SnP (layer 3) is slow. By contrast, Ni₃Sn₄ (layer 1) keeps growing even after the formation of

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**Table 2**

Summary of the previous observation on intermetallic compounds formed as a reaction between Ni or Ni–P with Sn or Sn–Pb

| Ni₃Sn₄ | Ni₃Sn₂ | Ni₃Sn | Techniques | Reference |
|-------|-------|-------|------------|-----------|
| ∙     | SEM, XRD, XMA [18] |
| ∙     | TEM [6] |
| ∙     | SEM [17] |
| ∙     | XRD [15] |
| ∙     | SEM, XMA, XRD [4] |
| ∙     | SEM, XMA, XRD, AES [3] |
| ∙     | SEM, TEM [16] |
| ∙     | SEM, XMA [2] |
| ∙     | XPS, XRD, XMA [5] |

○, formed as the major phase; ○, formed but not as the major phase; SEM, scanning electron microscopy; XRD, X-ray diffraction; XMA, X-ray microanalysis; TEM, transmission electron microscopy; AES, Auger electron spectroscopy; XPS, X-ray photoelectron spectroscopy.

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**Fig. 11.** Comparison of microstructure of an interface before (a) and after (b) joining. The underlying layers consisting of Ti and Cu remained at the original positions, so that the absolute positions of the interfaces between the eutectic Pb–Sn and the electroless Ni–8 mass% P can be determined very accurately.

**Fig. 12.** Evolution of microstructure of the interface on further heating. Ni–20 mass% P (layer 4) grows significantly. A high density of vertical linear contrasts are due to artefacts caused by FIB thinning and have nothing with the actual microstructure.

**Fig. 13.**
Ni$_2$SnP. That is, Ni can diffuse easily across Ni$_2$SnP (layer 3). In order for Ni$_3$Sn$_4$ (layer 1) to keep growing, Ni must be supplied all the way from the electroless Ni–8 mass% P. Thus, the content of Ni in the electroless Ni–8 mass% P just adjacent to Ni$_2$SnP (layer 3) keeps decreasing. If the supply of Ni from the underlying original Ni–8 mass% P is not sufficient, Kirkendall voids are formed, which grow along the diffusion path of Ni in layer 4 (Ni–20 mass% P) for some reasons which are not understood well at the moment (Fig. 13(d)).

(3) On further heating, spherical voids are formed in Ni$_2$SnP (layer 3). This can be explained by assuming that diffusion of Ni in Ni$_2$SnP (layer 3) is slower than in Ni$_3$Sn$_4$. This difference in diffusivity of Ni gives rise to imbalance of supply of Ni at the Ni$_3$Sn$_4$/Ni$_2$SnP interface, which leads to formation of Kirkendall voids near the Ni$_3$Sn$_4$/Ni$_2$SnP interface in Ni$_2$SnP (Fig. 13(e)).

5. Conclusions

The following results were obtained from TEM observation of microstructure of a solder joint in a semiconductor package.

1. An FIB technique is very useful to prepare TEM thin foil specimens from the solder joint interface.
2. Four different layers, i.e. Ni$_3$Sn$_4$, Ni$_{48}$Sn$_{52}$, Ni$_2$SnP and Ni–20 mass% P were formed between a eutectic Pb–Sn solder and the electrolytic Ni–8 mass% P plating during mounting of the solder balls. Among them, Ni$_{48}$Sn$_{52}$ and Ni$_2$SnP were detected for the first time in the present study.
3. Spherical voids are formed at the interface between Ni$_{48}$Sn$_{52}$ and Ni$_2$SnP, and columnar voids are formed at the interface between Ni$_2$SnP and Ni–20 mass% P. These voids are Kirkendall voids formed due to the difference in diffusivity of Ni across the interfaces.
4. Fracture takes place at either of those interfaces where many voids are present.

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