

## Interfacial Microstructure and Joint Properties of Copper Direct Bond Inserted by the Thin Film of Indium

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### 1. Introduction

Recently, in the field of the electronics packaging, as high performance and compactness of electronic equipments advances, the demand of the high density assembly technology is increasing [1], [2]. Moreover, to prevent environmental pollution, Pb-free and flux less are required in the solder joint process at the same time. Direct metal bonding is considered as a joining process satisfying these demands. However, the bonding method, which normally requires ultimate clean and flat joint surface, or alternatively needs high process temperature and large pressure, is not feasible to the electronic assembly.

In this paper, the method is investigated that low-melting-point-alloy as an insert material is utilized to completely diffuse and react to the base metals. and so, high strength interface was obtained because the low-melting-point-alloy layer is completely consumed. The schematic outline of the process is shown in Fig.1. So far we confirmed the influence, of the insert material and the joining parameters, on the Cu/Cu joint, and clarified that the shear strength over 30MPa was obtained when either the Sn-In eutectic or indium was used as an insert material [3], [4]. As for the joint interface, by the SEM observation of the cross-section and the EDX line analysis, the formation of voids and the existence of Cu contents at the reaction layer were confirmed. However, the composition of the Cu-In alloy in a reaction layer has not been clarified.

Accordingly, energy diffusion x-ray (EDX) analysis and a transmission electron microscopy (TEM) study were conducted to determine the microchemical and microstructure in a reaction layer.

### 2. Materials and procedure

The oxygen-free copper rods of  $\phi 3\text{mm} \times 2\text{mm}$  and  $\phi 5\text{mm} \times 5\text{mm}$  were prepared as the joint specimen. Removed the natural oxidation film on the surface ( $R_a \leq 100\text{nm}$ ), the vacuum deposition of indium was conducted to form the thin film of the  $1\mu\text{m}$  thickness. So the total inserted In film thickness becomes  $2\mu\text{m}$ . Joining was carried out by fixing a couple of specimens with In-deposited surfaces facing, and applying the load of 90MPa by the jig, heating to 523K in  $\text{N}_2+\text{H}_2$  (5:1) atmosphere, and making In completely diffused and reacted.

The sample used for the TEM observation was prepared by grinding from the Cu side of the  $\phi 3\text{mm} \times 2\text{mm}$  sample to the vicinity of the joint, and using the FIB (Focused Ion Beam) method at the center of the joint.

### 3. Experimental results and discussion

#### 3.1 Microanalysis and observation of the joint interface

The TEM observation result at the reaction layer cross-section is shown in Fig. 2. In this observation result, fine voids of approximately 10nm ranges and are scattered in the region near the Cu side of a reaction layer. These fine voids also have been found in the other part near the opposite Cu side. The line which ties the fine voids forms a boundary, and the size of the crystal grain in both reaction layers are different. The thickness of reaction layer near the Cu is  $0.15\text{--}0.3\mu\text{m}$ , and the size of the crystal grain is in  $0.3\text{--}0.7\mu\text{m}$ . On the other hand, the thickness of a reaction layer between the boundaries is  $1.5\mu\text{m}$ , and the crystal grain between these is  $0.5\text{--}1\mu\text{m}$  in size. The ruggedness of the line which ties these fine voids is 100nm or less, and the size almost agrees to the surface roughness of the original Copper surface. Moreover, this line is guessed to be an original surface of Cu, considering that the film thickness of the inserted

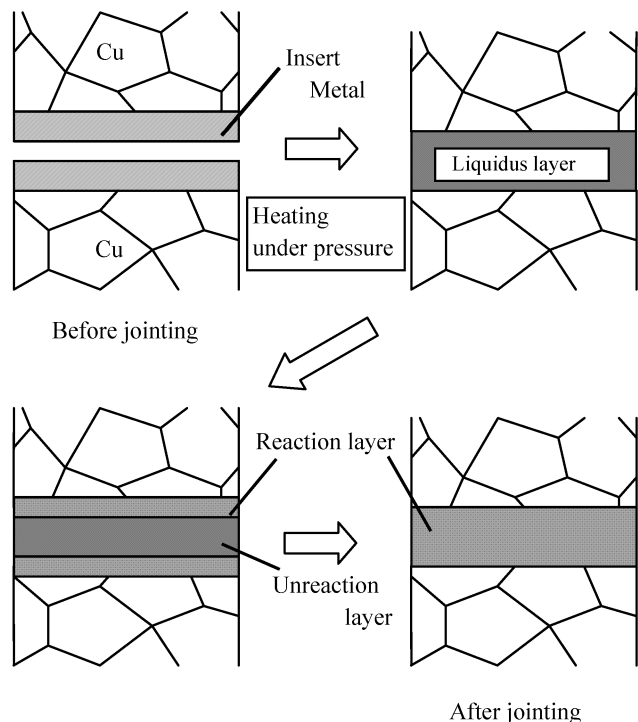


Fig.1 Bonding process currently assumed.

indium film is 2 $\mu\text{m}$ , and the space in the indium film was crushed by the load during joining.

As the reaction mechanism is guessed by the above observation result, the diffusion reaction of liquid indium to solid Cu is predominant in a reaction layer (reaction layer A thereafter) on the Cu side compared with the surface of original Cu, while the dissolution reaction of solid Cu to liquid indium is predominant in the reaction layer (reaction layer B thereafter) between Cu surfaces. It is supposed that a reaction layer is formed by these mutual reactions. Moreover, voids of approximately 1 $\mu\text{m}\times 0.2\mu\text{m}$  in size are observed in reaction layer B along the grain boundary. This is thought to be formed by the volume shrinkage when the reaction layer coagulates.

### 3.2 Evaluation by EDX analysis and electron diffraction

The EDX analysis and the electron diffraction were carried out. Analysis points are designated as (a) for the part near reaction layer A, (b) for the reaction layer B, (c) for the reaction layer B close to A, and (d) for the center part in B, respectively. The beam diameter of the EDX is approximately 2nm $\phi$ , and the beam diameter at the electron diffraction is 10nm $\phi$ . The quantitative analysis result in the reaction layer by the EDX analysis is shown in Table1. In the intermetallic compound in a reaction layer, the ratio of Cu and In is almost 52:48 in weight. It is found that the compositions are nearly the same for the crystal part (reaction layer B) at the center of a reaction layer, and for Cu near the crystal part (reaction layer A). This fact is because that the thermal equilibrium was reached due to a long joining time, although concentration gradient is large enough at the initial stage of the reaction.

By using analysis software (Pattern doctor: Nippon Steel Corporation) on the electron diffraction pattern at each EDX analysis position, both reaction layer A and reaction layer B are identified as Cu<sub>11</sub>In<sub>9</sub> (The lattice parameter: a=12.814, b=4.354, c=7.353, and  $\beta$ =54.49 [5].

### 4.Results

To clarify the characteristic of the joint interface in the Cu/Cu joint using indium as an insert material, EDX and electronic diffraction analysis by the TEM observation were carried out. As a result, the following conclusions were obtained.

- 1) The reaction layer formed during joining consists of the area in a reaction layer A (grain diameter 0.15-0.3 $\mu\text{m}$ ) and a reaction layer B (0.5-1 $\mu\text{m}$ ), and is supposed to be an original surface of Cu by the reason of the spill over of voids approximately 10nm in size at the interface. It is thought that the diffusion reaction of melt indium to Cu is predominant in this reaction layer A, while the dissolution reaction of Cu to melt In is predominant in reaction layer B, and a reaction layer is formed by these two mutual reactions.
- 2) Both diffusion reaction areas and the dissolution reaction areas in a reaction layer were the intermetallic compounds of Cu-In according to the EDX quantitative analysis on the TEM image, and the ratio was almost 52:48 in weight % in either area.
- 3) The existence of Cu<sub>11</sub>In<sub>9</sub> phase was identified according to the EDX quantitative analysis and the electron beam diffraction pattern.

### References

- [1] I.Ohnuma, Y.Cui, X.J.Liu, Y.Inohara, H.Ohtani, R.Kainuma, and Kishida, *Phase Equilibria of Sn-InBased Micro-Soldering Alloys*, Journal of

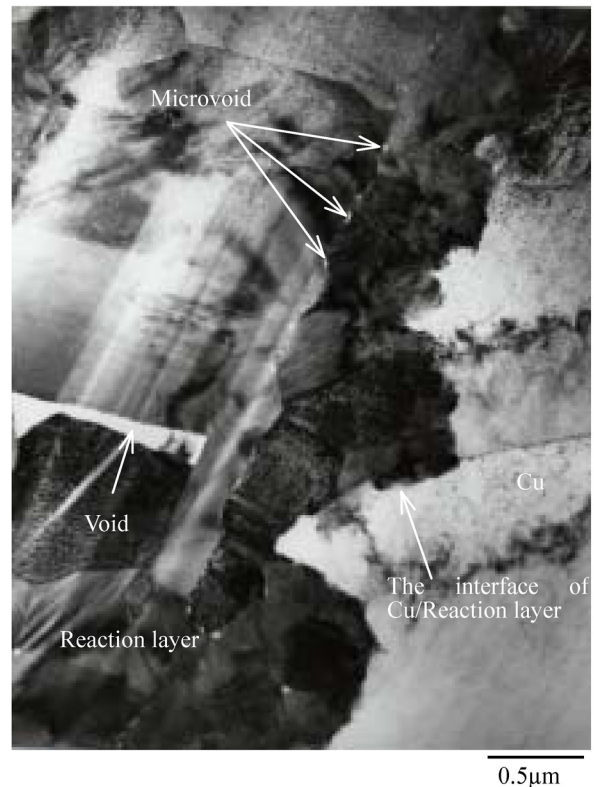


Fig.2 TEM micrographs of the cross section across bonding interface.

Table1 Results quantitative of EDX analysis for the reaction layer.

| Measurement Point | Element & Line | Weight Percent | Atomic Percent |
|-------------------|----------------|----------------|----------------|
| a                 | Cu Ka          | 53.67%         | 67.67%         |
|                   | In Ka          | 46.33%         | 32.33%         |
| b                 | Cu Ka          | 50.45%         | 64.78%         |
|                   | In Ka          | 49.55%         | 35.22%         |
| c                 | Cu Ka          | 52.92%         | 67.01%         |
|                   | In Ka          | 47.08%         | 32.99%         |
| d                 | Cu Ka          | 52.04%         | 66.22%         |
|                   | In Ka          | 47.96%         | 33.78%         |
| Average           | Cu Ka          | 52.27%         | 66.42%         |
|                   | In Ka          | 47.73%         | 33.58%         |

- Electronic Materials, vol.29, No.10, 2000, p243-246.
- [2] Yi-Chia Chen, Chin C.Lee, *Indium-Copper Multilayer Composite for Fluxless Oxidation-Free Bonding*, Thin Solid Films, 283(1996), p243-246
- [3] T.Yamada, H.Akamizu, K.Yasuda, K.Fujimoto, *Study on the Flux-free Direct Bonding and the Interface Characteristics Using Thin Film of Low Melting Temperature Alloys*, (Mate2003), Yokohama, p169-174.
- [4] K.Taniguchi, M.Shimoda, T.Goto, K.Yasuda, K.Fujimoto, *Copper Direct Bonding Process Using Thin Film of Indium*, MES2003, p172-175
- [5] H.Okamoto, *Comment on Cu-In*, Journal of Phase Equilibria, 15(2), 1994.