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Studies of the Mechanical and Electrical Properties of Lead-Free Solder Joints

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Abstract

The mechanical and electrical properties of several Pb-free solder joints have been investigated that include the interfacial reactions, namely, the thickness and morphology of the intermetallic layers, which are correlated with the shear strength of the solder joint as well as its electrical resistance. A model joint was made by joining two “L-shaped” copper coupons with three Pb-free solders, Sn-3.5Ag, Sn-3.8Ag-0.7Cu, and Sn-3.5Ag-3Bi (all in weight %), and combined with two surface finishes, Cu and Ni(P)/Au. The thickness and morphology of the intermetallic compounds formed at the interface were affected by solder composition, solder volume, and surface finish. The mechanical and electrical properties of Pb-free solder joints were evaluated and correlated with their interfacial reactions. The microstructure of the solder joints was also investigated to understand the electrical and mechanical characteristics of the Pb-free solder joints.

Key Words: Pb-free solders, solder joints, intermetallic compounds, morphology, Mechanical properties, electrical properties, Sn-Ag, Sn-Ag-Cu, and Sn-Ag-Bi.

INTRODUCTION

Owing to accelerated research and development efforts in the electronics industry, universities, national laboratories, and government agencies, several promising Pb-free solders have been recently identified for replacing Pb-containing solders in microelectronic applications [1-12]. The candidates include Sn-3.5Ag, Sn-3.5Ag-0.7Cu, Sn-3.5Ag-4.8Bi, or Sn-0.7Cu (with slight variations in composition). Although a considerable amount of publications on Pb-free solders can be found in the recent literature [13, 14], it is still at the infancy stage as compared to the vast amount of the data accumulated for the Pb-containing solders over the last several decades [15-17].

In this study, we investigated the mechanical and electrical properties of several Pb-free solder joints as a function of their interfacial reactions, namely, the intermetallic compound (IMC) thickness and morphology. When a solder joint is formed in electronic assemblies, such as flip chip interconnects, ball grid arrays or surface mount packages, complicated metallurgical reactions take place between a molten solder and the metallization (or surface finish) on a substrate; dissolution/diffusion of substrate atoms into the molten solder, formation/growth of IMC at joint interfaces, solid-state interdiffusion at the growing IMC interface(s), precipitation of IMCs during solidification of a molten solder and so on. The IMC formation and dissolution of the surface finish pad are closely related which alters the microstructure of a solder joint during multiple reflow cycles or upon extended exposure at elevated temperature. Therefore, the interfacial reactions in solder joints can strongly influence their microstructure, mechanical, electrical properties and others. In this study, three Pb-free solders, Sn-3.5Ag, Sn-3.8Ag-0.7Cu, and Sn-3.5Ag-3Bi with two surface finishes, Cu and Ni(P)/Au, were chosen to form the model solder joints and to evaluate their mechanical/electrical properties in reference to their interfacial reactions.

EXPERIMENTAL

Fabrication of the Model Solder Joints

Three Pb-free solders, Sn-3.5Ag(SA), Sn-3.8Ag-0.7Cu(SAC),

Sn-3.5Ag-3Bi(SAB), and the Pb-containing, eutectic Sn-Pb solder were used in a paste form. In order to characterize the mechanical and electrical properties of solder joints, model joint samples were made by joining two “L-shaped” copper coupons (9.5 mm x 1.25 mm x 0.25 mm) with solder into a cross-shape, as shown in Fig.1. The joint geometry used here is similar to that found in a surface mount solder joint in terms of joint area and thickness. An aluminum fixture which was fabricated to hold 20 pairs of the samples was used to align/assemble them as well as to dispense solder paste between Cu coupons. All copper coupons were etched in a 10% H₂SO₄ solution for 10 min to remove any surface oxides. Some Cu coupons were plated with an electroless Ni-P (about 8 wt % P) and immersion Au. The joining operation was performed by placing the assembled fixture on a hot plate heated to 250°C, purged with nitrogen atmosphere for a set period of time (2, 12, and 52 min). The reflow time was determined by measuring the dwell time above 220°C. After the reflow, the sample fixture was quickly removed from the hot plate to a metal block to cool down the molten solder. A typical heating/cooling curve is shown for the reflow time, 2 min, in Fig. 2. Some of Pb-free solder joint samples were further annealed at 200°C for 1 or 7 days to increase their IMC thickness.

The samples were encapsulated by epoxy at room temperature and cross-sectioned using a diamond saw and metallographically polished to a final finish using 0.05 μm alumina slurry. The cross-sectioned samples were etched with a 10% HCl solution for one and half minutes to enhance the contrast in a scanning electron microscope (SEM). SEM and energy dispersive X-ray analysis (EDX) were employed to investigate the IMC growth and qualitative elemental composition.

Electrical and Mechanical Properties of Solder Joints

Electrical properties of the model joints were evaluated by measuring the four-point contact resistance of a joint sample as shown in Fig. 1. The solder joint area is approximately 50mil× 50mil (1.25mm× 1.25mm). The instrument used for electrical resistance measurement is a HP3458A multimeter with a four-point probe measurement function.

Mechanical properties of the solder joints were evaluated by measuring the joint

shear strength of a cross-bar sample in the direction as shown in Fig. 1. An Instron machine (model 4500) was employed with a special grip at a cross-head speed of 0.25 mm/min. The maximum load to fracture and a total elongation of a joint sample were recorded. The fracture surface of solder joints was also examined by SEM and EDX analyses.

RESULTS

IMC Growth and Morphology in Solder Joints

Figs.3 to 5 show SEM cross-sectional views of the interfacial reactions between Cu substrates and three Pb-free solders, Sn-3.5Ag (SA), Sn-3.8Ag-0.7Cu (SAC), and Sn-3.5Ag-3Bi (SAB), respectively. The average joint gap varies between 15 and 25 μm , even though the reflow condition was kept the same for all the samples. In SAC solder joints, the joint gap is appreciably smaller than others, indicating the wetting property of the SAC solder is better than the others.

As the reflow time increases, the IMC layer in all the joints get thicker without much changing in their morphology and thereby the joint gaps were reduced. For Cu substrates, all the interfacial IMCs were identified as $\eta\text{-Cu}_6\text{Sn}_5$, while the rod- or needle-like IMCs found in the solder matrix were identified as Ag_3Sn , shown in the accompanying EDX spectra of each Figure. The long needle or rod IMCs continue to grow and eventually connect two interfaces, as noted in Fig.4 (c) and Fig.5 (b). The average thickness of the interface IMC was measured for each solder joint as a function of reflow time, and listed in Table I.

In order to further increase the IMC thickness and thereby reduce the joint gap between two interfaces, some SAC joints were heat treated at 200°C in nitrogen for up to 7 days. Fig.6 shows a cross-sectional view of the SAC solder joints heat treated at 200°C for 1 and 7 days. The IMCs thickened significantly, occupying almost two thirds of the joint gap in the joint sample aged for 7 days. Their morphology was also changed from a round or scallop shape into a planar layer, and the second IMC phase, $\epsilon\text{-Cu}_3\text{Sn}$, was formed after heat treatment at 200°C. The corresponding IMC thickness is also reported in Table I.

Fig.7 shows the cross-sectional views of three solder joints formed between the Cu/Ni(P)/Au substrate and three Pb-free solders, SA, SAC, and SAB, respectively. Due to good wettability of a Cu/Ni(P)/Au substrate, its joint gap was generally smaller than that of the bare Cu. The IMCs formed on Cu/Ni(P)/Au substrates are also quite different from those on the Cu in term of their morphology and growth behavior. The IMCs, identified as the Ni_3Sn_4 type, are faceted, angular, or needle-like; and most of them are detached from the joint interface, except for the SAC solder joint. Note the porous structure comprised of the needle-like IMCs in front of the joint interface in the SAB, Fig.7 (c). For the extended reflow time, 52 min, the porous structure became more compact by forming more IMCs in the solder joint. At that point, the solder joints were practically filled up with the IMCs. The average thickness of IMC attached to the interface is listed as a function of reflow time for each solder in Table II. In addition, the thickness of a porous IMC layer is also reported as the value in parenthesis in Table II.

Electrical and Mechanical Properties

Fig.8 exhibits the electrical resistance of Pb-free solder joints as a function of reflow or aging time for Cu and Cu/Ni(P)/Au substrates. Each value represents an average value of 10 measurements. The value of eutectic Sn-Pb solder joint was included as a reference. For the Cu substrate, the resistance value generally increases as the reflow time increases, except for the extended aging time of 7 days at 200°C. A similar trend is observed for Cu/Ni(P)/Au; an initial increase and then decrease for the long reflow time, 52 min. In general, the resistance value of Cu/Ni(P)/Au joints is smaller than Cu joints at an equivalent reflow condition.

Fig.9 exhibits the joint shear strength as a function of reflow time for both Cu and Cu/Ni(P)/Au substrate. Each data point represents an average value from seven to nine samples. The shear strength value was calculated by considering only an actual joint after excluding the void area in a joint. For Cu substrates, the shear strength value does not change much as a function of reflow time, except for the aged joints of SAC. As a reference, the shear strength of eutectic Sn-Pb solder joints (50.3 MPa) is included, which is somewhat higher than others. The SAB joints exhibit the highest shear strength

regardless of the reflow time. The SAC joint strength is reduced significantly after aging of 1 and 7 days at 200°C. For Cu/Ni(P)/Au, the shear strength was found to be generally less than the Cu equivalent, and it goes down as the reflow time increases.

Fig.10 exhibits the percent elongation values of solder joints as a function of reflow time and solder composition for both Cu and Cu/Ni(P)/Au. In general, the elongation value goes down as the reflow time increases for both Cu and Cu/Ni(P)/Au, except for the aged SAC on the Cu. It is also found that the elongation values with the Cu substrates are higher than the Cu/Ni(P)/Au, indicating the Cu-solder joint might be softer than the Ni-solder joint.

Fig. 11 shows SEM micrographs of the fracture surface of several solder joints on the Cu substrate as-reflowed or aged. Depending on solder alloy, the fracture surface reveals a different morphology. In SAC joints, a dimple structure is observed, while no dimple structure is seen in SA, and very small dimples observed in SAB. In the aged SAC joint, several large flat areas are noted in the fracture surface, suggesting the fracture path gone through the thick IMC layer.

The fracture surfaces on Au/Ni(P)/Cu substrates were generally different from those found on the Cu. They exhibit less dimple structure, more flat areas, and more active interaction with the IMC structure.

DISCUSSION

Intermetallic Growth and Morphology

The mechanical and electrical properties of a solder joint in electronic packages, such as BGA, flip chip or surface mount joints, are affected by many factors, such as interfacial reactions, solder composition, surface finish (or under bump metallization), solder volume, reflow conditions, and others. In this study, the major goal was to investigate the mechanical and electrical properties of Pb-free solder joints in terms of solder composition, surface finish, IMC thickness and morphology. The interfacial IMC thickness data collected from three Pb-free solders (SA, SAC, SAB) with two surface finishes (Cu, Ni(P)/Au) as a function of reflow time are plotted in Fig. 12. Depending on the surface finish, the different IMC was identified: the Cu-Sn type for Cu substrate and

Ni-Sn type IMC for Cu/Ni(P)/Au. The Cu-Sn IMC growth is gradual, following a parabolic growth rate, while the Ni-Sn IMC on Cu/Ni(P)/Au grows much faster than the Cu-Sn IMC on Cu. This observation appears to be contradictory to what has been reported in the literature that Ni-Sn IMC grows slower than Cu-Sn IMC [18-20]. However, in the present case, the enhanced growth of the Ni-Sn IMC can be understood by recognizing a smaller joint gap in Cu/Ni(P)/Au samples, as a result of good wettability of Ni(P)/Au surface finish over bare Cu. A smaller joint gap means a smaller solder volume to be saturated rather quickly with the dissolving solute atoms, such as Cu or Ni. Another explanation may be related to the porous nature of Ni-Sn IMCs observed on Cu/Ni(P)/Au, which can promote faster growth compared to the compact structure observed with Cu-Sn IMCs. In addition, it is generally known the IMC on an electroless Ni(P) grows faster than on an electrolytic Ni [20].

The interfacial Cu-Sn IMC formed on a Cu substrate has the well-known morphology of a round or scallop shape regardless of solder composition, while the Ni-Sn IMC on Cu/Ni(P)/Au exhibits different morphologies, such as angular, faceted or needle-like shape, depending on solder composition. The Ni-Sn IMCs in SA and SAB are mostly detached from the interface, while in SAC they remain adhered to the interface. This observation is consistent with the previous work, where the role of Cu, either from solder or substrate, was recognized in keeping the IMC attached [20, 21].

Effect of IMC Growth on Mechanical Properties

For Cu substrates, the shear strength of three Pb-free solder joints did not change much as a function of reflow time. This suggests the shear strength of these joints is not sensitive to the amount of IMCs formed even for an extended reflow time, such as 52 min at 235°C. However, a significant reduction in shear strength (15 to 20%) was observed with the SAC joints upon aging at 200°C up to 7 days. As noted in Fig.6, the IMCs after the aging occupied almost two thirds of the whole joint gap. The shear strength values of Cu joints obtained in this study are in a reasonable agreement with the previously reported, although they were measured by different testing methods and sample geometries [22, 23].

The shear strength of Ni(P)/Au joints is lower than the Cu, which also decreases

steadily with reflow time, suggesting the amount of Ni-Sn IMCs influence more strongly on the mechanical properties.

The percent elongation data confirms the similar trends observed with the joint shear strength. That is, the Cu joints possess a larger elongation than Au/Ni(P). And the percent elongation of Ni(P)/Au joints steadily decreases as reflow time, indicating the amount of IMCs formed significantly affecting their joint properties. However, one exception is noticed with SAC joints on a Cu substrate, where more elongation is observed with the aged joints comprising of thicker IMCs.

Effect of IMC Growth on Electrical Resistance

The electrical resistance of Pb-free solder joints, as shown in Fig.8, appears to follow a general trend for both Cu and Cu/Ni(P)/Au substrates: an initial increase as a function of reflow time and then a decrease in the final stage. The initial increase of the electrical resistance can be explained in terms of intermetallic growth. That is, more IMC growth would bring a higher electrical resistance, since the electrical resistance of IMCs is usually higher than that of a Sn-rich, Pb-free solder: $17.5 \mu\Omega\text{-cm}$ for Cu_6Sn_5 [24], $28.5 \mu\Omega\text{-cm}$ for Ni_3Sn_4 [24], and $7.7 \mu\Omega\text{-cm}$ for Sn-3.5Ag [19]. However, the decrease of electrical resistance in the final stage seems to be complex to understand. A few explanations for this decrease include formation of the second Cu-Sn IMC, Cu_3Sn , which has a lower resistance than Cu_6Sn_5 : $8.9 \mu\Omega\text{-cm}$ for Cu_3Sn [24], and growth of large Ag_3Sn needles or plates connecting or shorting two joint interfaces under an extended reflow condition. In addition, annealed solder joints are expected to contain less defects such as vacancies and dislocations, and consequently, the electrical scattering with defects is reduced and so the electrical resistance. Overall, it is demonstrated that electrical resistance of Pb-free solder joints seems to be better than Pb-containing solder joints, or at least the equivalent under a similar reflow condition.

IMC Growth and Fracture

As shown in Fig.11, fracture during the shear test of Cu joints took place inside the solder matrix near the interface for a short reflow time regardless of solder

composition. As the reflow time increases, the mixed mode of fracture between solder matrix and IMC was detected, but still the major fracture occurred in the solder matrix. However, for the aged SAC joints with thick IMC layers, the fracture occurred mostly within the IMC.

In Au/Ni(P)/Cu joints, the fracture path was influenced strongly by both reflow time and solder alloy. In SA and SAC joints, the fracture path has changed from solder (2 min reflow) to solder + IMC (12 min) to IMC (52 min), while in SAB the fracture went through the boundary between IMC and the substrate, indicating a very weak interface between the IMC and substrate.

CONCLUSIONS

The mechanical and electrical properties of Pb-free solder joints, made of three solder alloys (Sn-3.5Ag, Sn-3.8Ag-0.7Cu and Sn-3.5Ag-3Bi) and two surface finishes (Cu and Au/Ni(P)), were investigated in terms of reflow time, IMC thickness and morphology. The following conclusions are drawn:

- 1). The Cu-Sn IMCs on Cu with a small solder joint gap grow gradually with a round or scallop morphology regardless of solder composition.
- 2). The Ni-Sn IMCs on Ni(P)/Au with a small solder joint gap grow rapidly with an angular, faceted, or needle-like morphology, depending on solder composition. The Ni-Sn IMCs in SA and SAB are mostly detached from the interface, while in SAC they are attached to the interface.
- 3). The shear strength of Pb-free solder joints on Cu is not much affected by the duration of reflow time or IMC thickness. A significant reduction in the shear strength with Cu substrates (15 to 20%) is observed when the IMCs occupy about two thirds of a joint gap.
- 4). The shear strength of Ni(P)/Au joints is much lower than that of Cu joints and also decreases steadily with reflow time, or IMC thickness.
- 5). The percent elongation of Cu joints is larger than that of Ni(P)/Au. And it does not change much with IMC thickness, while that of Ni(P)/Au is more affected by the IMC growth.
- 6). The electrical resistance of Pb-free solder joints increases initially and then decreases

for an extended reflow or aging time for both Cu and Ni(P)/Au. This trend can be understood in terms of various IMCs formed at the interface or in the solder joint.

7). The fracture path observed with Cu joints is mostly inside the solder near the IMC interface for a short reflow time, but it occurs through IMC for an extended reflow or aging. The fracture with Ni(P)/Au joints was more influenced by the presence of IMC.

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Table I. IMC Thickness on a Cu Substrate as a Function of Solder Alloy
and Reflow Time

Solder Composition (wt %) (m. p.)	Reflow Temp (°C)*	Reflow Time (min)**	IMC Thickness (mm)	IMC Aged, 200°C, 1day	IMC Aged, 200°C, 7day
96.5Sn-3.5Ag (221°C)	235°C	2	2.2		
		12	2.85		
		52	5.43		
95.5Sn-3.8Ag-0.7Cu (217°C)	235°C	2	2.3		
		12	3.5	5.6	12.9
		52	6.4		
93.5Sn-3.5Ag-3Bi (214°C)	235°C	2	2.93		
		12	4.07		
		52	5.67		

* Reflow temperature refers to the peak temperature measured in the sample fixture during reflow.

** Reflow time is defined as the time spent above 220°C estimated from the heating profile.

**Table II. IMC Thickness on Cu/Ni(P)/Au as a Function of Solder Alloy
And Reflow Time**

Solder Composition (wt %) (m. p.)	Reflow Temp (°C)*	Reflow Time (min)**	IMC Thickness (mm)
96.5Sn-3.5Ag (221°C)	235°C	2	1 (9.3)***
		12	2 (9.3)
		52	3 (13.3)
95.5Sn-3.8Ag-0.7Cu (217°C)	235°C	2	4.0
		12	4.0
		52	10.9
93.5Sn-3.5Ag-3Bi (214°C)	235°C	2	0.8 (5.7)
		12	2 (8.3)
		52	3.5 (12.3)

* **Reflow temperature refers to the peak** temperature measured in the sample fixture during reflow.** Reflow time is defined as the time spent above 220°C estimated from the heating profile.*** The thickness of a porous IMC layer detached from the interface, but located near the interface.

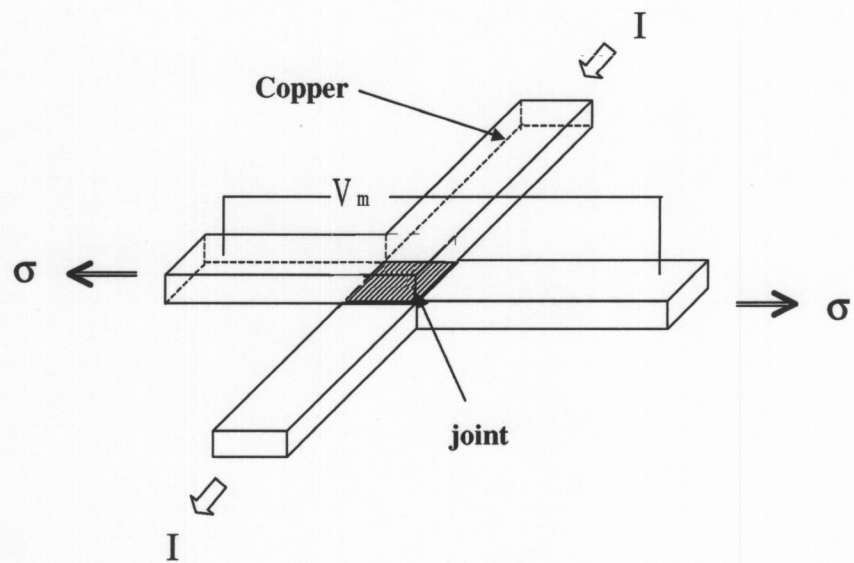


Fig.1. Schematic diagram of a model joint made of two copper coupons of “L-shape” for electrical and mechanical evaluation.

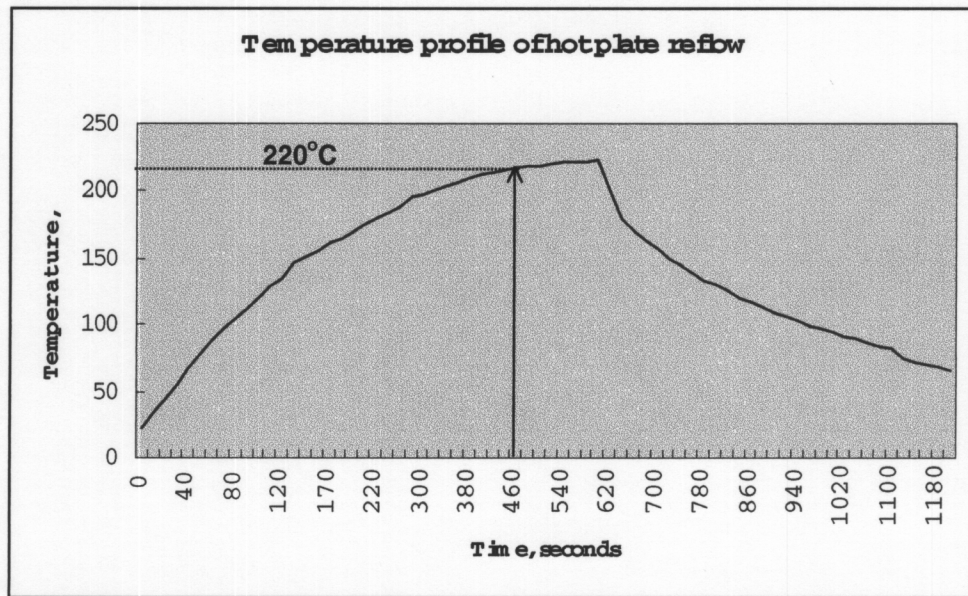
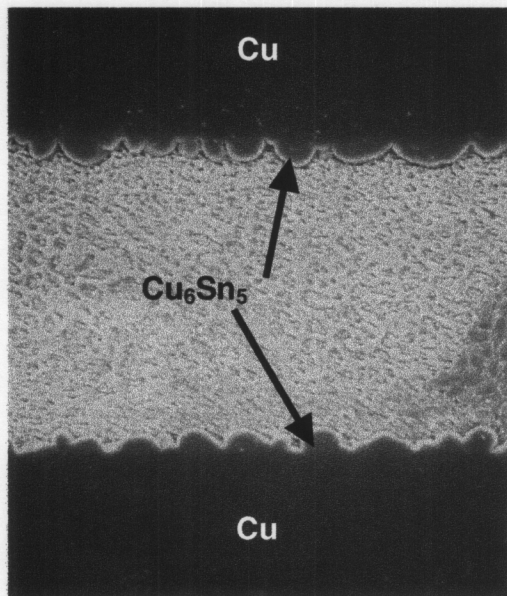
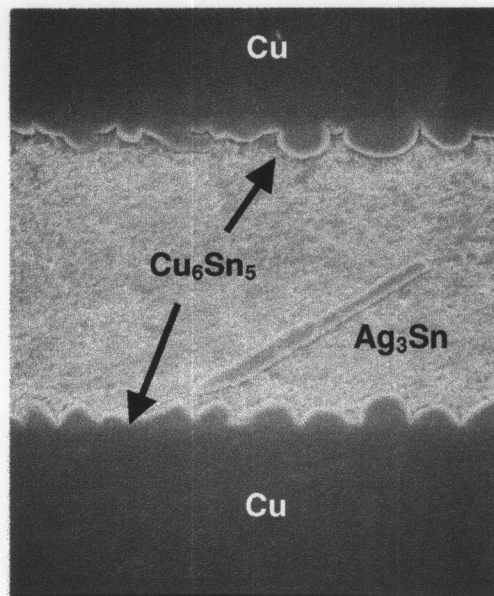


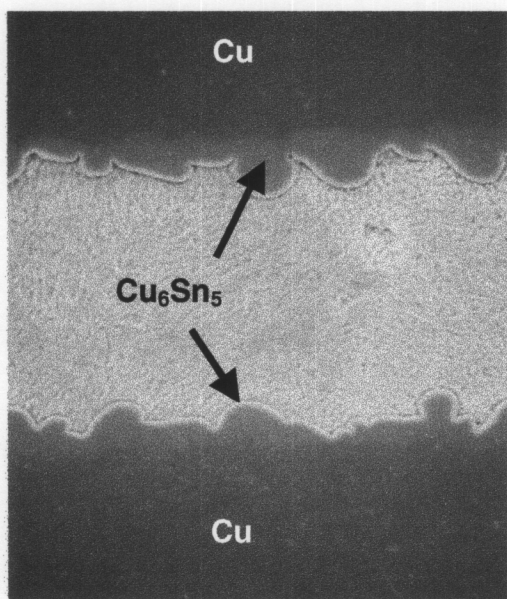
Fig.2. Temperature profile of a sample reflow fixture on a hot plate heated to 250°C for 2 min under nitrogen atmosphere.



a

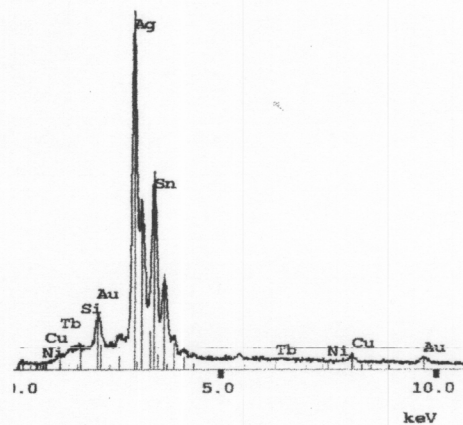
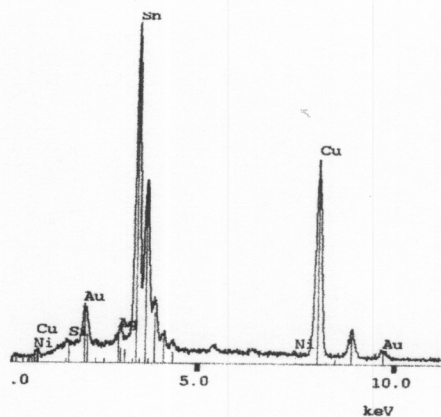


b



c

10 μm



d

Fig.3. Cross-sectional views of interfacial reaction series with Sn-3.5Ag on Cu substrate at 235°C: (a) 2, (b) 12, (c) 52 min, and (d) EDX spectra.

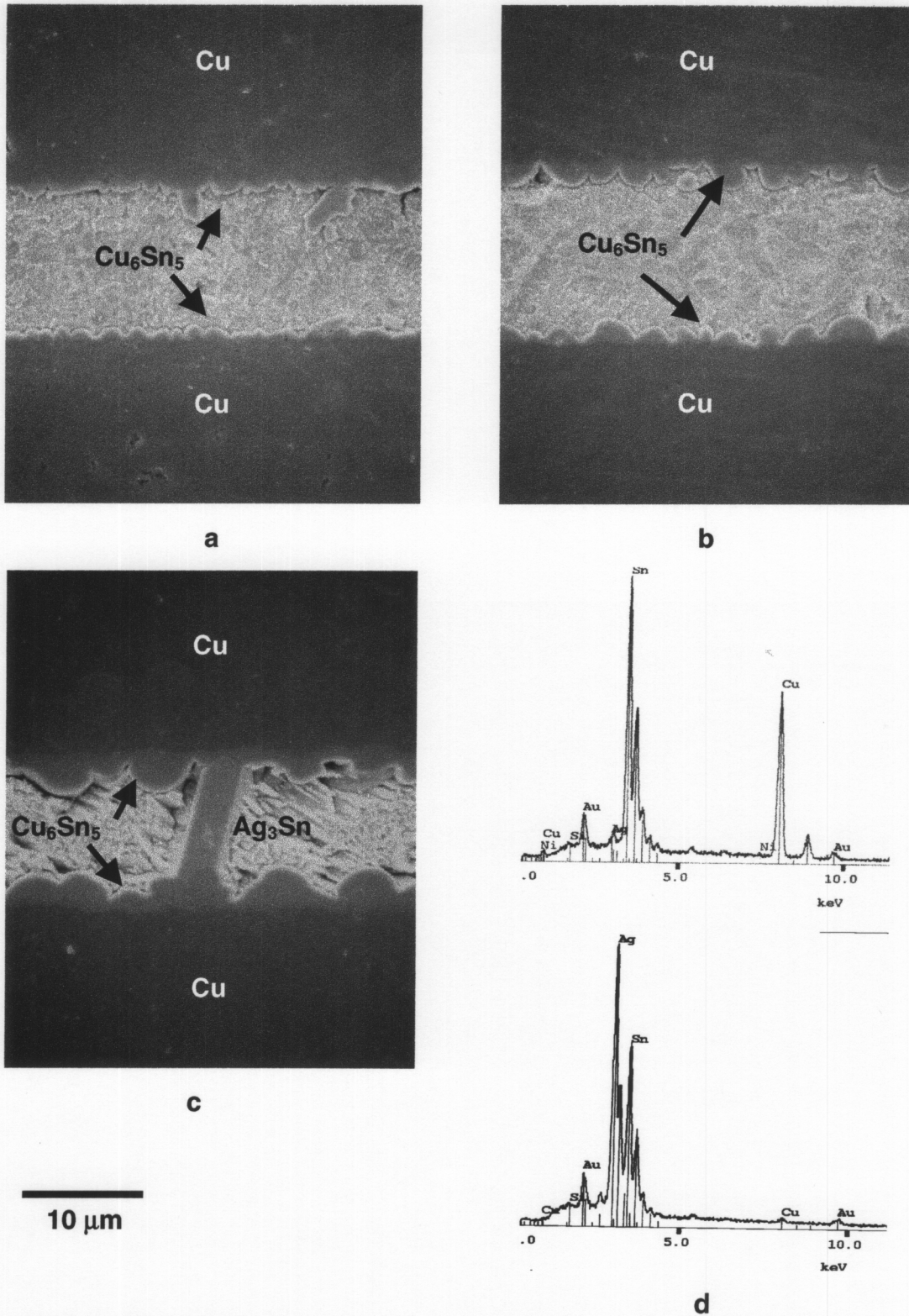
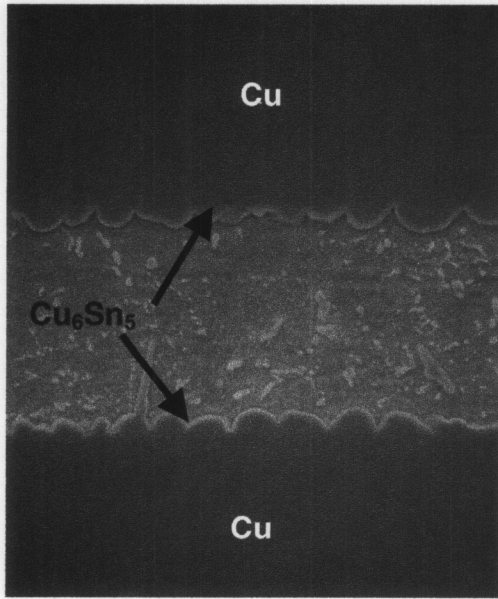
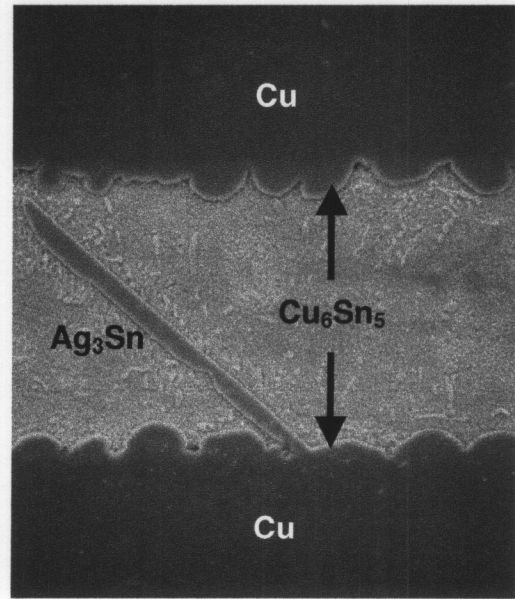


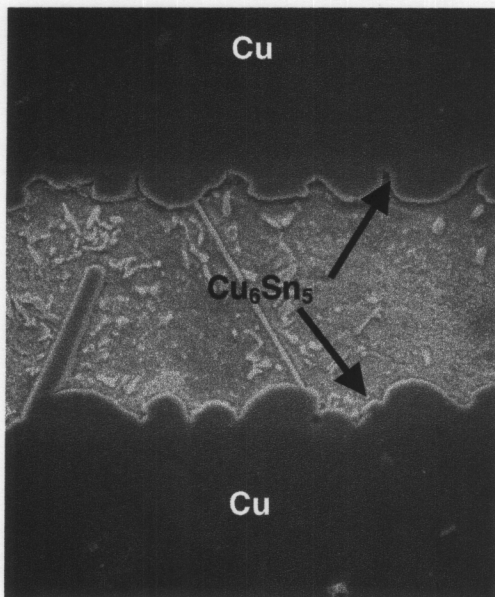
Fig.4. Cross-sectional views of interfacial reaction series with Sn-3.8Ag-0.7Cu on Cu substrate at 235°C:(a) 2, (b) 12, (c) 52 min, and (d) EDX spectra.



a

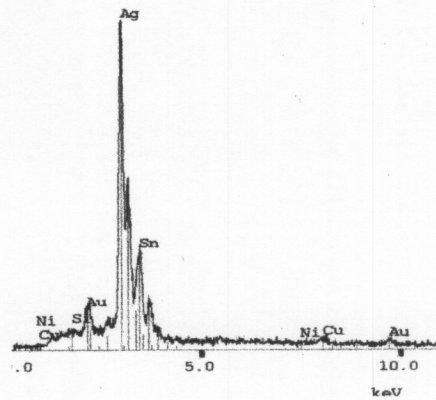
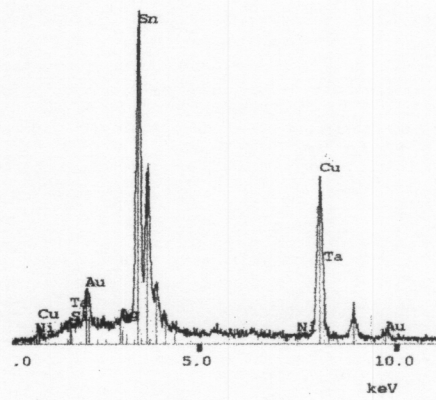


b



c

10 μ m



d

Fig.5. Cross-sectional views of interfacial reaction series with Sn-3.5Ag-3Bi on Cu substrate at 235°C:(a) 2, (b) 12, (c) 52 min, and (d) EDX spectra.

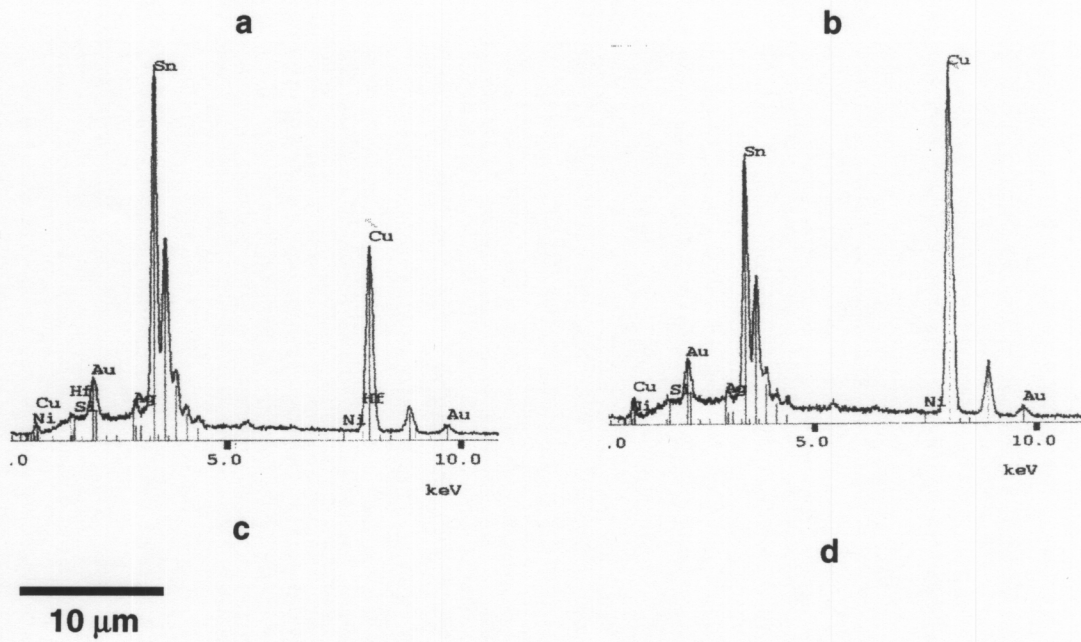
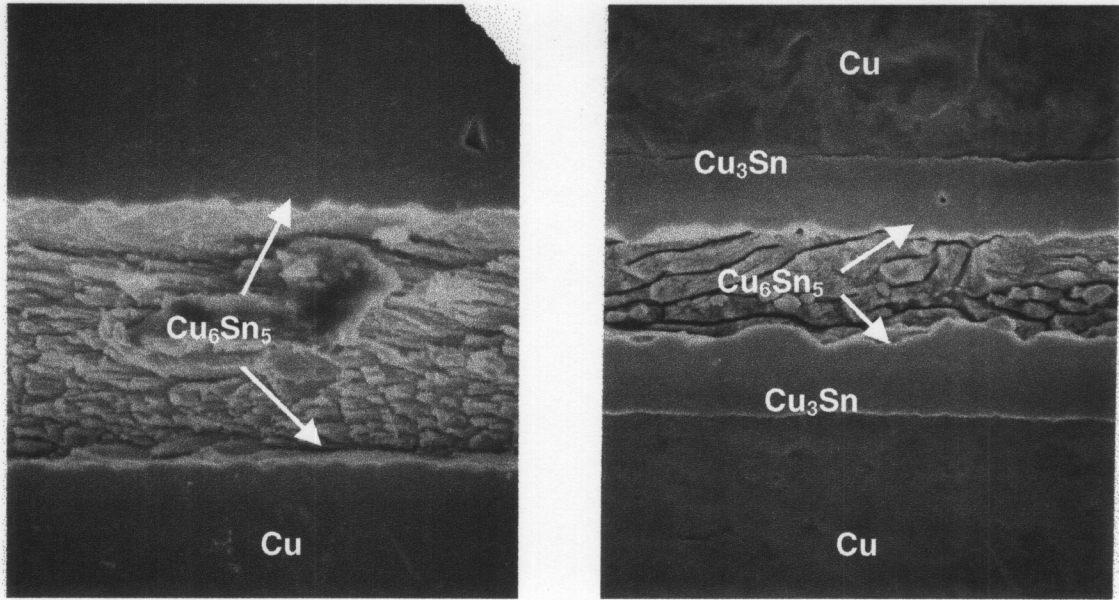


Fig.6. SEM cross-sectional views of Sn-3.8Ag-0.7Cu joints on Cu, aged at 200°C: (a) 1 day, (b) 7 days, (c) and (d) EDX spectra for IMCs.

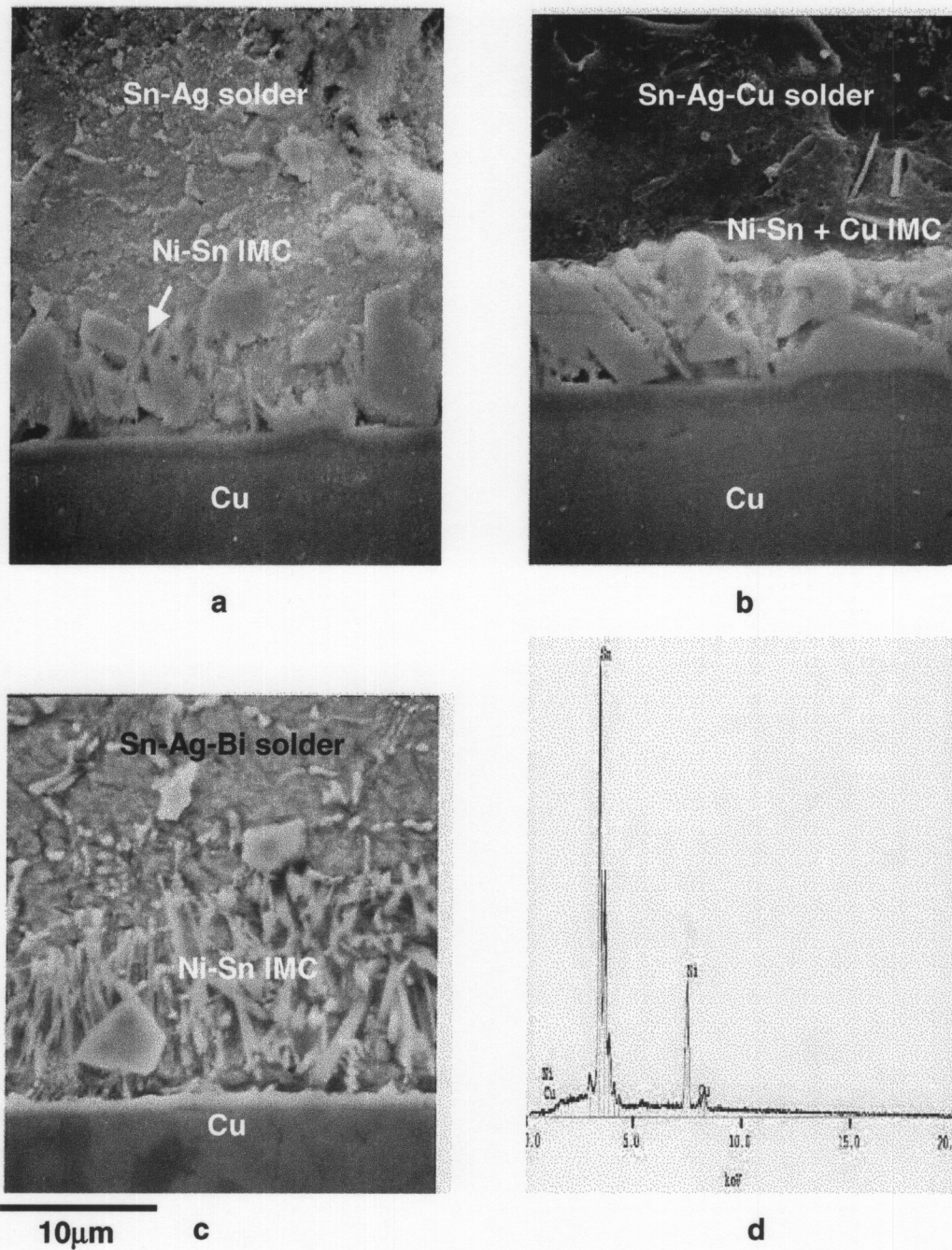
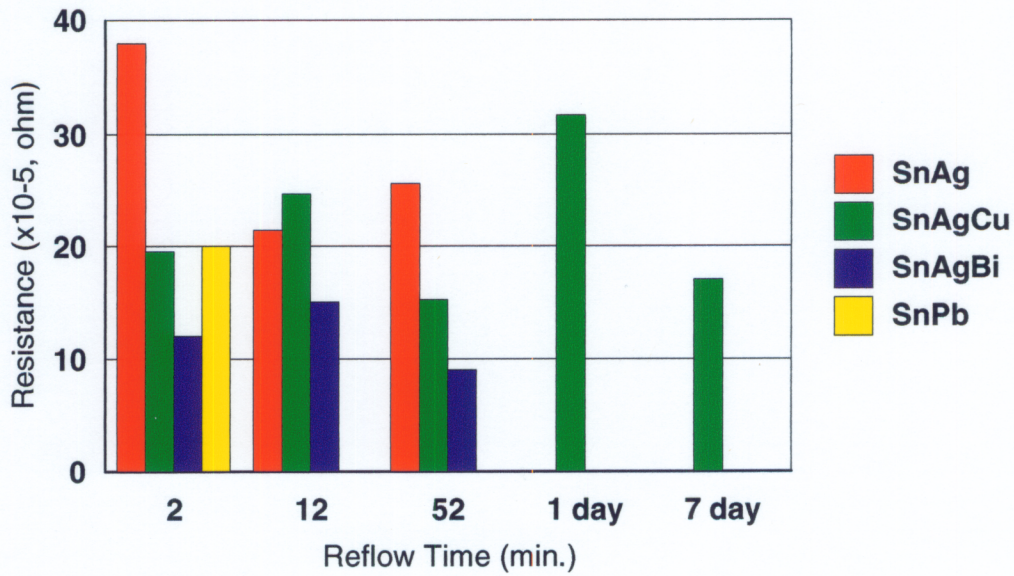


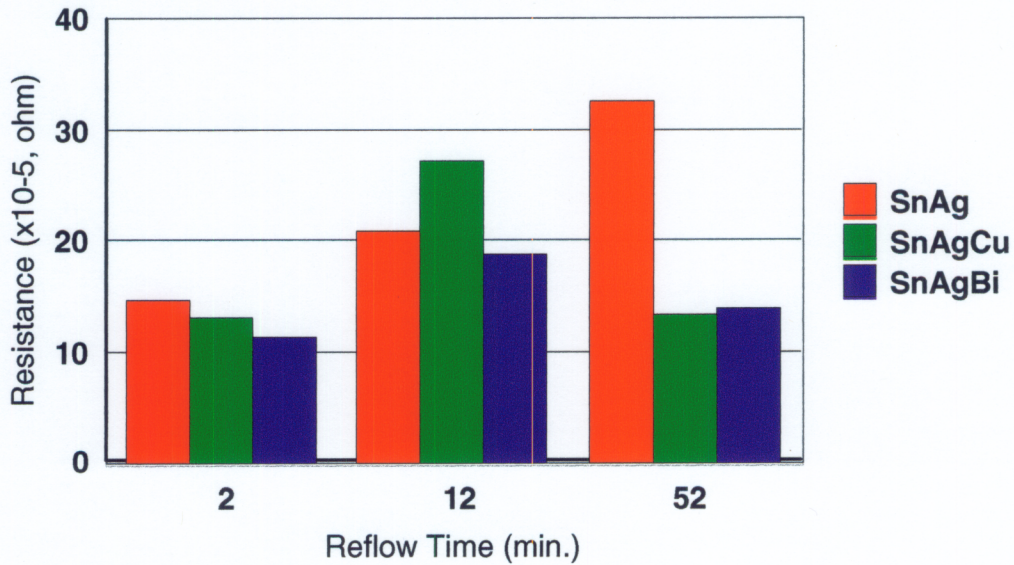
Fig.7. SEM cross-sectional views of interfacial reactions of Cu/Ni(P)/Au joints with three Pb-free solders reflowed for 2 min, at 235°C:(a) SA, (b) SAC, (c) SAB, and (d) EDX spectrum of IMCs formed at interface in SA and SAB.

Cu Substrate



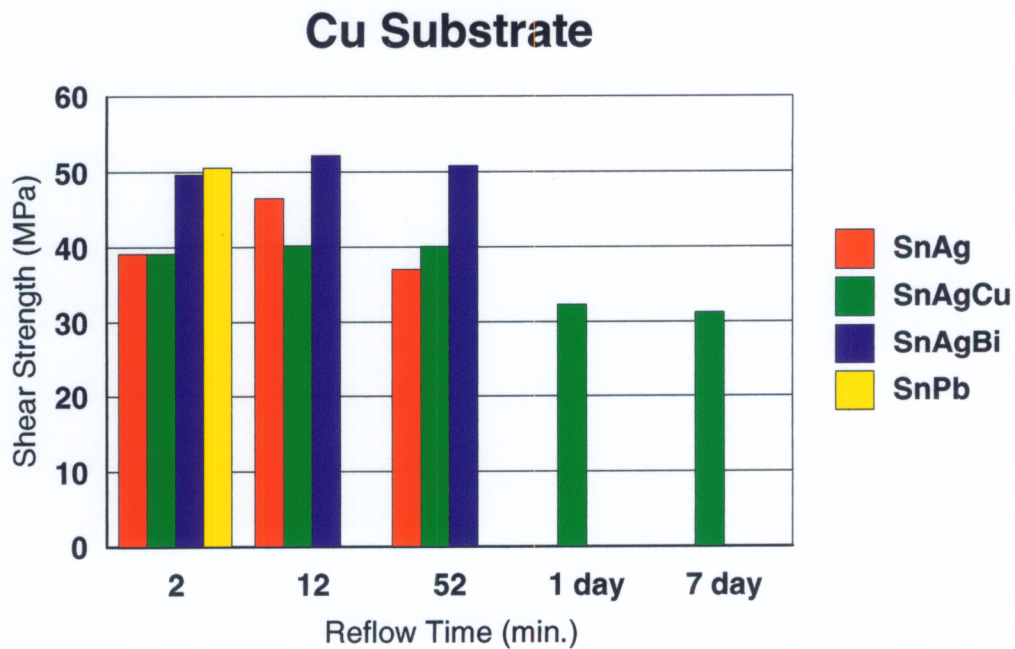
a

Cu/Ni/Au Substrate

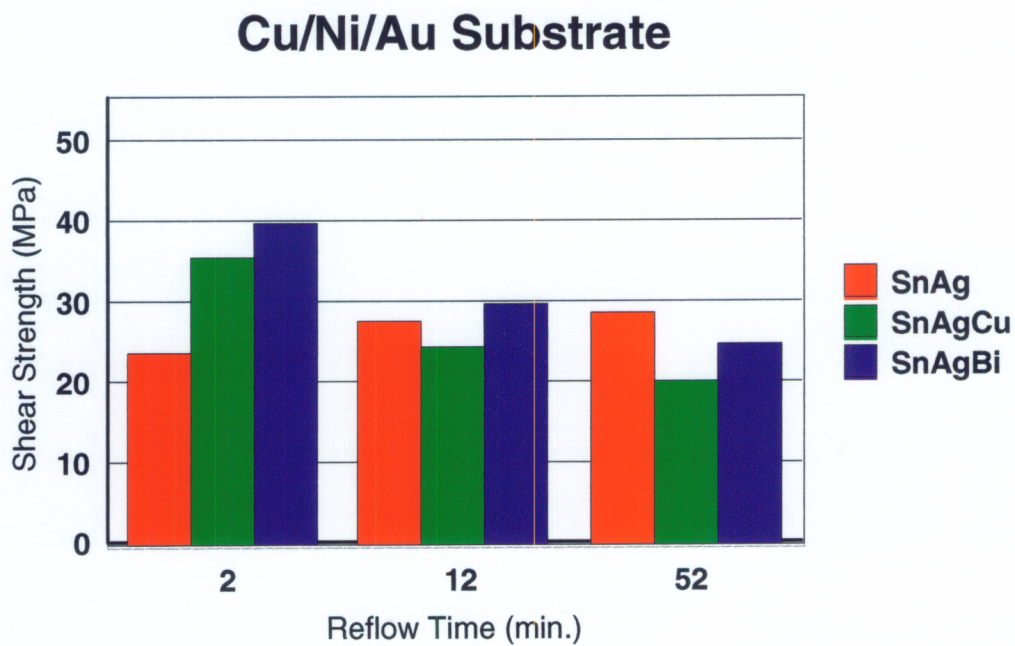


b

Fig. 8. Electrical resistance of Pb-free solder joints as a function of reflow time: (a) Cu substrate, and (b) Cu/Ni(P)/Au substrate.



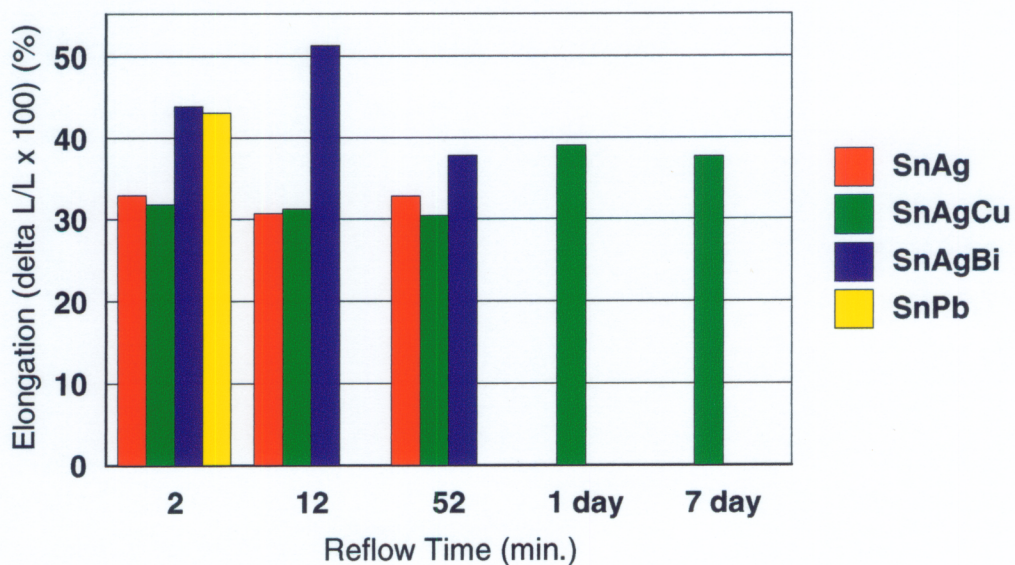
a



b

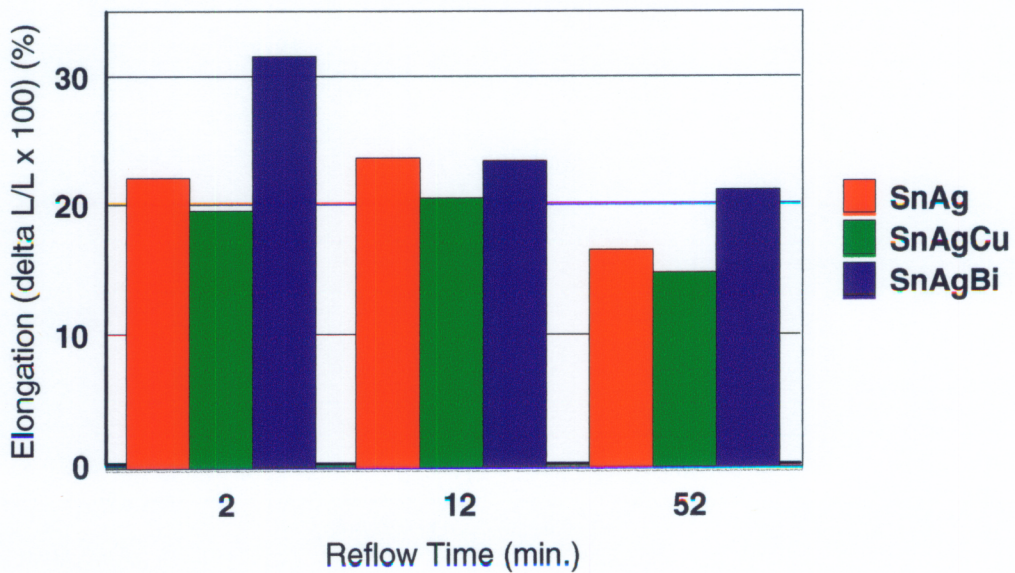
Fig. 9. Shear strength of Pb-free solder joints as a function of reflow time: (a) Cu substrate, and (b) Cu/Ni(P)/Au substrate.

Cu Substrate



a

Cu/Ni/Au Substrate



b

Fig.10. Percent elongation of Pb-free solder joints as a function of reflow time: (a) Cu substrate, and (b) Cu/Ni(P)/Au substrate.

Fracture Surface of Solder Joints on Cu Substrate

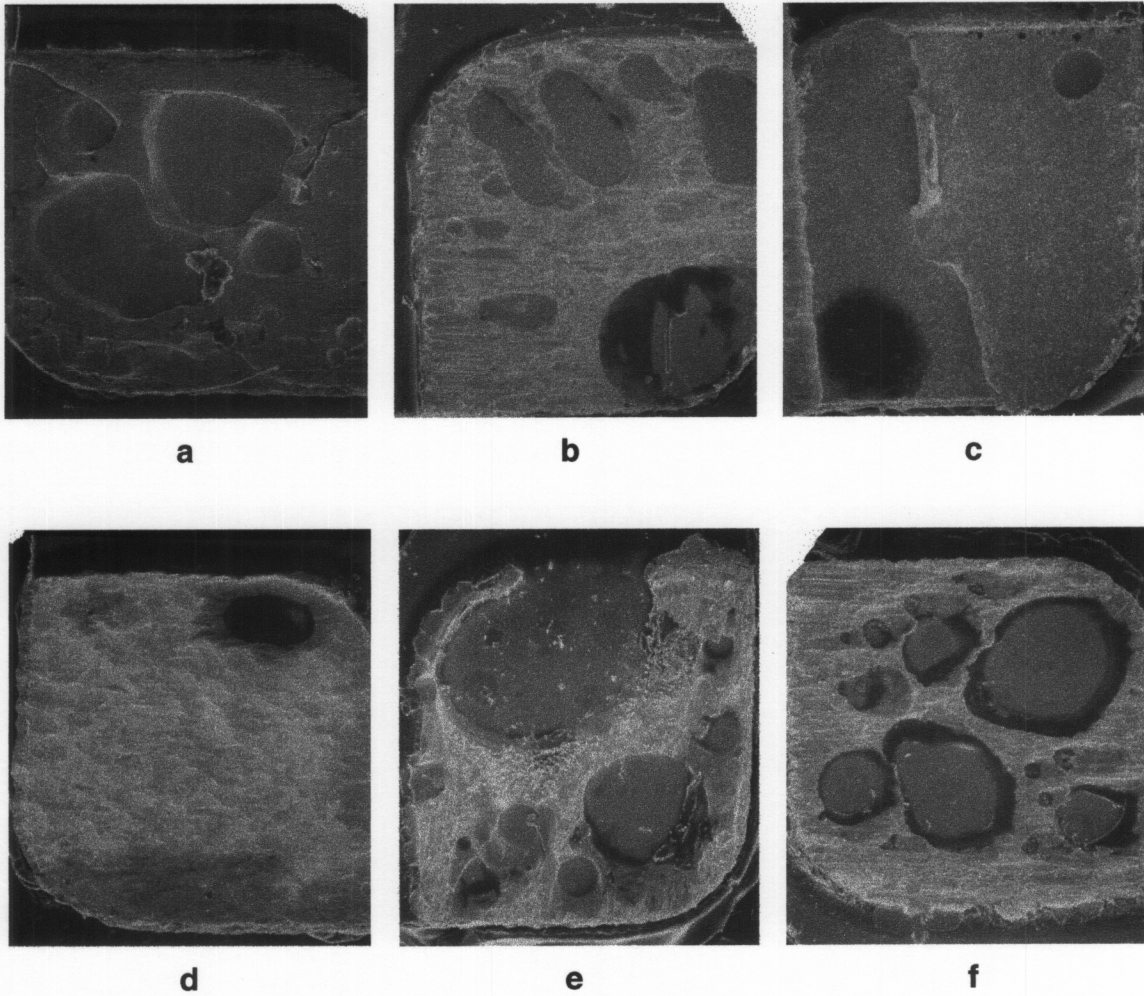


Fig.11. SEM micrographs of the fracture surface of Pb-free solder joints with Cu substrate: (a) SA, (b) SAC, (c) SAB, and (d) Sn-Pb reflowed for 52 min at 235°C, (e) SAC aged for 1 day, and (f) SAC aged for 7 days at 200°C.

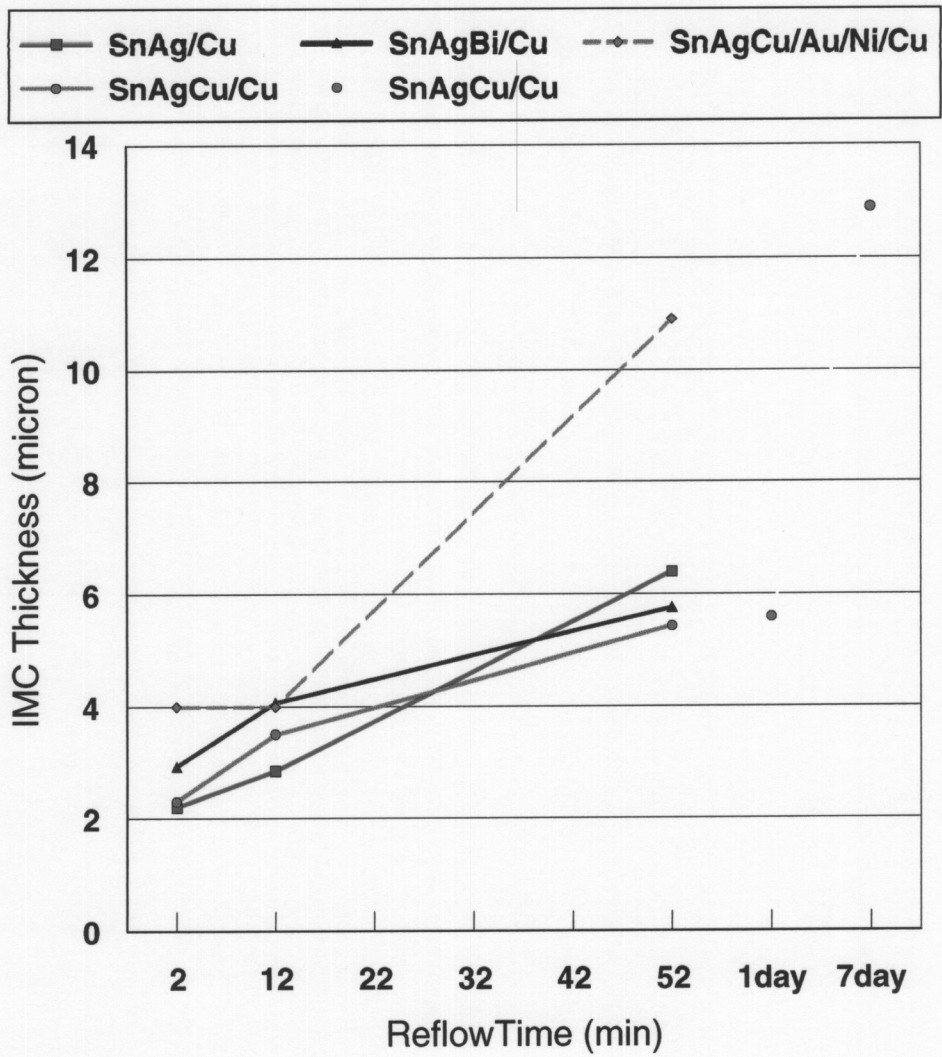


Fig.12. Growth of IMC in Pb-free solder joints as a function of reflow (at 235°C) or aging (at 200°C) time and substrate finish.