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Microstructure and shear strength of a Au–In microjoint

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Abstract

Two types of Au–In microjoints, i.e. Au/In/Au in which In foil was used and Au/In, were prepared by either solid state interdiffusion (SSID) or solid–liquid interdiffusion (SLID) bonding for single lap tensile test. Deposition of the Au and In thin films was carried out by thermal evaporation on a polyethylene terephthalate (PET) substrate. It is found that the shear strength of the Au/In microjoints is higher than that of Au/In/Au using In foil. It is also observed that the fracture mode of Au–In microjoints depends on the types of In used. Failure of the Au/In microjoints appeared to be along the joint–substrate interface, whereas it occurred within the In foil for the other type of specimens. Examination of the Au/In microjoints by glancing angle X-ray diffraction reveals the presence of the two major constituent phases, Au_7In_3 and Au, as well as other intermetallics $AuIn_2$, $Au_{10}In_3$, and Au_9In_4 in small amount. On the other hand, only the intermetallic $AuIn_2$ and pure In were observed in the Au/In/Au microjoints, where the total thickness of In is much higher than that of Au. © 2000 Elsevier Science Ltd. All rights reserved.

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1. Introduction

Indium (In)-containing alloy solders show a longer fatigue life, better mechanical properties and reliability than conventional tin (Sn)-containing alloys as solder interconnections for electronic packaging [1–4]. As a result, indium and its alloys are considered to be used in the surface mount technology for various microelectronic devices in the future. From the processing point of view, indium has lower melting point, 156°C, than that of tin, 232°C, thus the joining temperature can be reduced further. To take advantage of this merit and also improve the service life of a In-containing microjoint, a new bonding technique, called solid-liquid interdiffusion bonding (SLID) [5,6], has recently been introduced in electronic packaging industry.

The SLID process uses a multilayer of high-melting and low-melting materials as a bonding preform and is carried out at temperatures above the melting point of the low-melting material. Subsequently metallurgical transformation takes place between the high- and low-melting materials and the low-melting material is gradually consumed, resulting in a microjoint which contains only intermetallics and/or the high-melting material. By properly controlling the film thickness and processing conditions, it is possible to form a strong microjoint free from the low-melting material. This technology will allow the system designers of electronic packaging to reverse the conventional soldering hierarchy of lower and lower process temperatures of subsequent manufacturing steps.

Since the technology relies on the formation of an intermetallic, it is important to understand the phase formation during joining and its influence on the mechanical properties of the microjoints as well. In this study, both transmission electron microscopy and glancing angle X-ray diffraction are used to study the microstructure and phase formation of the Au–In microjoints. In addition, the effect of In thickness on the shear strength of the microjoints is presented and discussed.

2. Experimental

Two types of Au–In microjoints, i.e. Au/In/Au and Au/In, schematically shown in Fig. 1, were prepared, in which the former contains an In foil of 20 μm thick, while the latter uses an In thin film of 2 μm thick. Specimens containing the In foil were made by depositing a thin layer of Au about 5 μm thick, using thermal evaporation, on a polyethylene terephthalate (PET) substrate of dimensions 5×10×0.2 mm for use in a single lap tensile test. The base pressure for Au evaporation was 8×10^{-6} torr and rised up to 2×10^{-5} torr during deposition. The In foil was then sandwiched between

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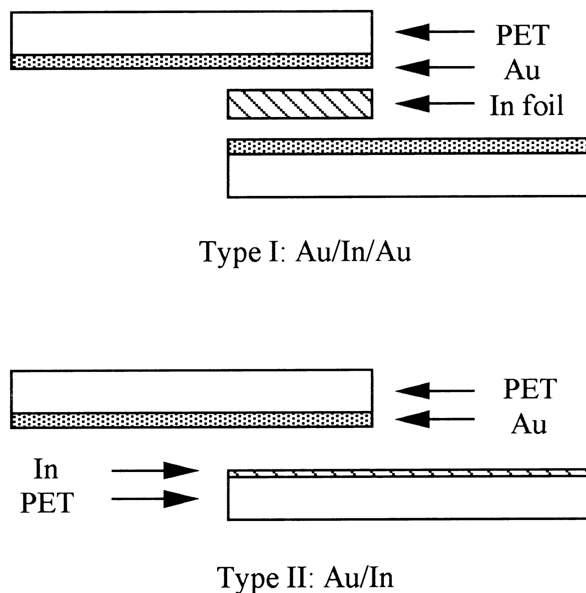

Fig. 1. Schematic of the two types of specimen geometry for single lap tensile test.

two Au-coated PETs and joining was carried out in air at 250°C for 5 min. The other type of specimens was produced by hot pressing the Au-coated PET with an In-coated PET together in air at temperatures spanning from 100 up to 250°C and duration from 1 to 30 min (see Table 1). The In thin films were prepared also by thermal evaporation and the chamber pressure was maintained at 5×10^{-4} torr during deposition.

The shear strength of the Au–In microjoints was evaluated by single lap tensile test. The fracture morphology of the tested specimens was examined by a JEOL 5400 scanning electron microscope operated at 20 KeV. Intermetallic phase formation within the microjoints was investigated by glancing angle X-ray diffraction using Cu K_α radiation with incident angle fixed at three different values 0.1°, 0.5° and 1.0°.

Microstructure of the microjoints was characterized by a Zeiss 902A energy filtering transmission electron microscope (TEM) operated at 80 KeV. Thin sections about 100 nm thick were made by a Reichert Ultracut E ultramicrotome for TEM observation.

3. Results and discussion

3.1. Shear strength of the Au–In microjoints

A single lap joint loaded in tension is the most common test geometry for evaluating adhesive joints. If the stress concentrations arising from the differential straining of the bonded substrates and from the eccentricity of the loading path can be neglected, the ultimate shear stress, τ_{uss}, of the adhesive joint is related to the applied tensile load, F, by $\tau_{uss} = F/A$, where A is the bonded area.

The test result of the Au–In microjoints produced using different geometry and under different joining conditions is given in Table 1, in which has been included the shear strength of pure In and the PET substrate. It is found that on the average the Au/In microjoints exhibit higher shear strength than those of Au/In/Au which contain In foil. The shear strength of the Au/In microjoints prepared below the melting point of In, which undergo solid state interdiffusion, and above the melting point of In, which undergo solid–liquid interdiffusion, is quite similar. It is also noted that the duration of hot pressing shows insignificant effect on the measured shear strength. Nevertheless, the shear strength of the Au/In/Au microjoints is apparently higher than that of the pure In, likely due to a difference in the defects content in the In [6]. Fig. 2 shows the fracture surface of the In foil in a Au/In/Au microjoint in which failure of the single lap specimen occurred within the In. Cavities and stretching in the In due to shear deformation can be easily observed.

The most striking outcome of the mechanical test is the change of failure modes in the microjoints with the types of In used for joining. It is obtained that failure of the single lap specimens containing In foil occurred within the In layer, whereas it took place at either the Au–PET or the In–PET interfaces when In thin films

Table 1
The processing conditions, measured shear strength and observed phases in the Au–In microjoints

Types of specimen geometry	Hot pressing conditions		Shear strength (MPa)	Observed phases	Remarks
	Temp (°C)	Time (min)			
I	250	5	1.0 ± 0.14	In, $AuIn_2$	SLID bonding
II	250	1~5	1.5 ± 0.51	Au_7In_3, Au_9In_4, Au	SLID bonding
	200	5~30	1.13 ± 0.19	Au_7In_3, Au_9In_4, Au,	
	150	5~30	1.25 ± 0.50	Au_7In_3, $AuIn_2$, Au,	SSID bonding
	100	5~30	1.13 ± 0.64	Au_7In_3, $AuIn_2$, $Au_{10}In_3$, Au	
In			0.8 ± 0.10		Foil
PET			3.2 ± 0.18		Sheet

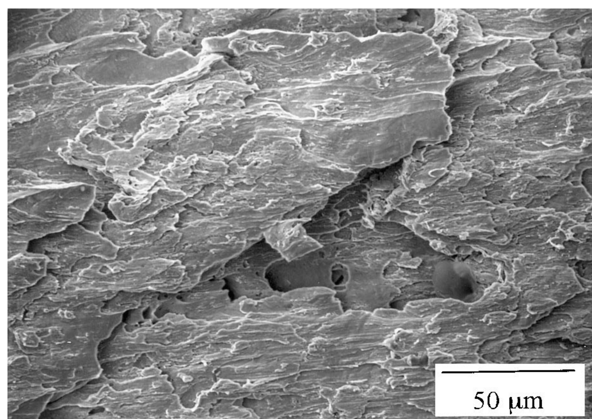

Fig. 2. A SEM micrograph of the fracture surface of a Au/In/Au microjoint where failure occurred within the In.

were used. Since the result of X-ray diffraction (see below) indicates that the thin film In was nearly consumed, only intermetallic phases and some remnant Au would be left in the microjoints. The shear strength of both Au and the various Au–In intermetallic compounds is expected to be higher than that of pure In, as reflected from the melting point of the phases [8]. As a result, phase transformation, via interdiffusion, of the low-melting In and high-melting Au into the various Au–In intermetallics has resulted in a strong Au–In microjoint. The real strength of the Au/In microjoints is, however, not known due to poor joint–substrate adhesion.

3.2. Analysis of the phase formation by X-ray diffraction

The result of single lap tensile test demonstrates the importance of layer thickness, which will eventually determine the phases formed upon joining, on the shear strength of Au–In microjoints. Typical X-ray spectra of a Au/In microjoint hot-pressed at 100°C for 15 min, in which delamination of the microjoint occurred along the In–PET interface are shown in Fig. 3. In addition to the peaks from PET and clay, both of which were used as an internal standard, intermetallic phases Au_7In_3, $AuIn_2$, and $Au_{10}In_3$, and pure Au are observed. The intensity of the Au (111) peak increases with the incident angle due to an increased X-ray interaction volume at higher incident angle. It is clear from the X-ray diffraction that the low-melting constituent In has been completely consumed and reacted with Au to form a variety of intermetallic phases after joining; thus a strong Au/In microjoint free from pure In is obtained. The depletion of In in the Au/In microjoint is also reflected in the color change of the In layer from bright to dark gray before and after joining, while viewing through the transparent PET substrate.

A summary of X-ray diffraction result of the various specimens prepared for the single lap tensile test is given in Table 1. From the table it can be seen that Au_7In_3 and Au are the common phases observed in all the Au/In microjoints, and indeed they are the two major constituent phases detected in the X-ray spectra. This can be rationalized by the fact that the atomic composition of Au_7In_3 is close to the thickness ratio of Au (5 μm)

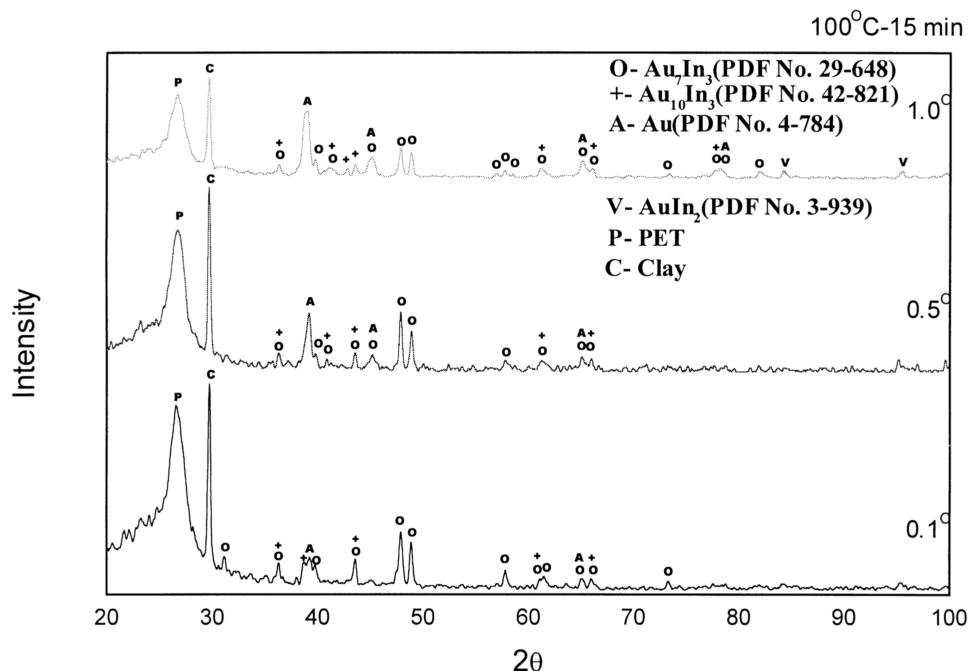

Fig. 3. Typical X-ray spectra of the Au/In microjoint hot-pressed at 100°C for 15 min, recorded at different incident angles.

and In (2 μm) before joining. In addition, two other intermetallic phases, $Au_{10}In_3$ and $AuIn_2$, were observed for the specimens prepared using solid state interdiffusion bonding, i.e. hot pressing below the melting point of In. For those specimens produced above the melting point of In, i.e. undergoing solid liquid interdiffusion bonding, the intermetallic phase Au_9In_4 was observed in the X-ray spectra. The two phases, Au_7In_3 and Au_9In_4, have similar atomic composition, and are designated as γ' and γ in the Au–In binary phase diagram [8]; however, the latter is stable only at high temperature. This result illustrates that the SLID process can form intermetallics which are different from those existed in the specimens prepared by the SSID process. When the In foil was used to form the microjoints, i.e. In is a major constituent in the microjoint, it is observed that the X-ray spectra show peaks mainly from In, with small peaks from the intermetallic $AuIn_2$.

3.3. *Transmission electron microscopy of a Au/In/Au microjoint*

Reactions in the Au–In diffusion couples is reported to be the fastest formation of intermetallics among transition metals and is characterized by an activation energy of 0.23 eV [9]. Consequently, preparation of thin sections for TEM observation by conventional ion milling method has been found to be invalid because ion beam induced phase transformation can readily occur. To overcome this difficulty, ultramicrotomy was utilized in the current study to prepare thin sections of the microjoints for TEM investigation.

A cross-section TEM micrograph of the Au/In/Au microjoint after the single lap tensile test is shown in Fig. 4a, which consists of a region of equiaxial grains and a portion of fractured segments. As a result of poor adhesion, the PET substrate delaminated from the Au/In/Au microjoint during ultramicrotomy. It has been demonstrated that a brittle material such as $AuIn_2$ will shatter and form periodic cracks during ultramicrotomy [10]. From the selected area diffraction (SAD) pattern shown in Fig. 4b, of an interfacial region, it is obtained that the equiaxial grains are pure In, and the fragments are identified to be $AuIn_2$. This result is the same as the conclusion of Millares et al. [11], where it is reported that the $AuIn_2$ intermetallic is the major phase in the reaction zone of the bulk Au–In diffusion couple.

Fig. 4c shows the microstructure of a region, which is expected to be under the maximum shear stress during the single lap tensile test, of the In about 10 μm away from the In–$AuIn_2$ interface. As a result of severe shear deformation, the polycrystalline In has transformed from a random orientation near the $AuIn_2$ to a texture near the center of the single lap specimen at which the

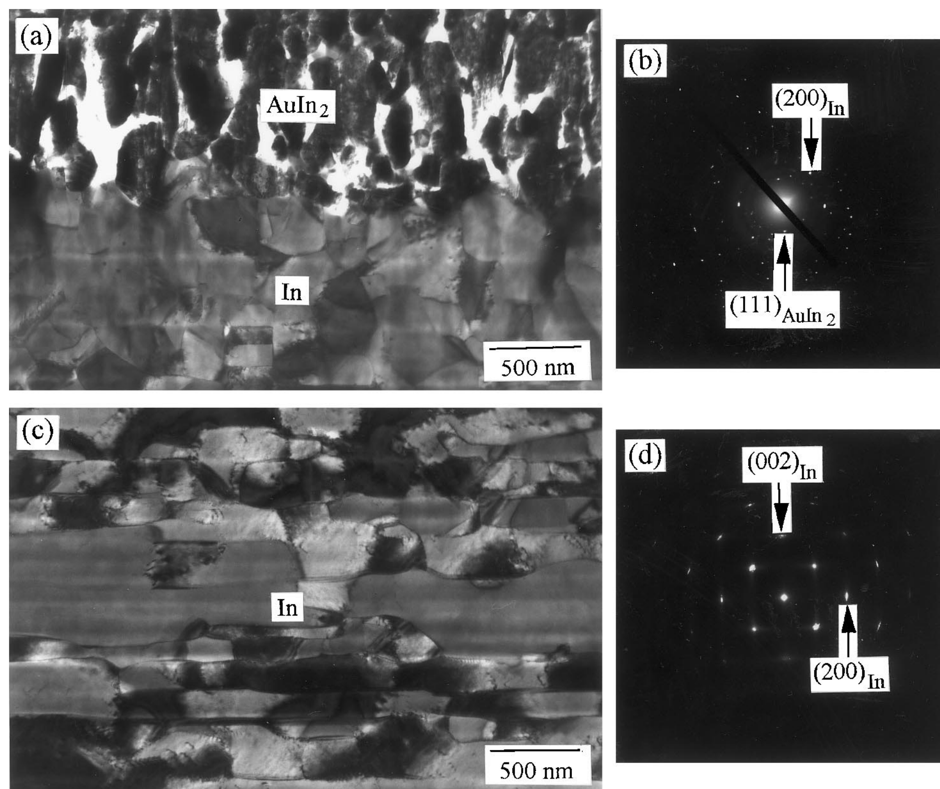

Fig. 4. (a) A cross-section TEM micrograph of the Au/In/Au microjoint after single lap tensile test; (b) the SAD pattern of an interfacial region in (a); (c) the microstructure of pure In about ~10 μm away from the In-$AuIn_2$ interface; (d) the SAD pattern of an area in (c).

shear stress reaches a maximum value. The existence of texture is illustrated both by the presence of a rectangular cell structure where high dislocation density can be readily seen in the bright field image of Fig. 4c and by the pseudo single crystal SAD pattern of Fig. 4d. The zone axis in the SAD pattern of Fig. 4d is close to the [001] direction of In that has a body-centered tetragonal crystal structure. The deformation texture consists of {110} plane oriented parallel to the substrate surface with ⟨111⟩ direction, which is the primary slip system for a body-centered cubic metal [7], aligned in the maximum shear stress.

4. Conclusions

It has been demonstrated that the shear strength of a Au–In microjoint evaluated by single lap tensile test can be improved by decreasing the thickness of the In layer and avoiding the presence of unalloyed In in the end materials. This can be achieved for example by replacing in the process In foils by In thin films.

The reaction and phase formation in the Au–In system was examined either by glancing angle X-ray diffraction or transmission electron microscopy. In the Au/In configuration where the film thickness of Au is higher than that of In, Au_7In_3 and Au are the two major phases observed in the microjoints, in addition to other intermetallics $AuIn_2$, $Au_{10}In_3$, and Au_9In_4 in small amount. On the other hand, only $AuIn_2$ and In are detected in the Au/In/Au microjoints in which In foil was used.

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