

Study of the Au/In Reaction for Transient Liquid-Phase Bonding and 3D Chip Stacking

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The latest three-dimensional (3D) chip-stacking technology requires the repeated stacking of additional layers without remelting the joints that have been formed at lower levels of the stack. This can be achieved by transient liquid-phase (TLP) bonding whereby intermetallic joints can be formed at a lower temperature and withstand subsequent higher-temperature processes. In order to develop a robust low-temperature Au/In TLP bonding process during which all solder is transformed into intermetallic compounds, we studied the Au/In reaction at different temperatures. It was shown that the formation kinetics of intermetallic compounds is diffusion controlled, and that the activation energy of Au/In reaction is temperature dependent, being 0.46 eV and 0.23 eV for temperatures above and below 150°C, respectively. Moreover, a thin Ti layer between Au and In was found to be an effective diffusion barrier at low temperature, while it did not inhibit joint formation at elevated temperatures during flip-chip bonding. This allowed us to control the intermetallic formation during the distinct stages of the TLP bonding process. In addition, a minimal indium thickness of 0.5 μm is required in order to enable TLP bonding. Finally, Au/In TLP joints of $\varnothing 40 \mu\text{m}$ to $60 \mu\text{m}$ were successfully fabricated at 180°C with very small solder volume (1 μm thickness).

Key words: Au/In interdiffusion, transient liquid-phase bonding, flip chip, 3D chip stacking

INTRODUCTION

Transient liquid-phase (TLP) bonding is a solder bonding process during which all solder is transformed into intermetallic compounds that have higher melting points than the solder itself (the alternative name of solid-liquid interdiffusion is sometimes used for what is fundamentally the same process).^{1,2} TLP bonding was originally investigated in the 1960s for multiple soldering operations during assembly, and its principle was explicitly elucidated by Bernstein.² Recently, TLP bonding has also attracted great attention in three-dimensional (3D) chip stacking technology.^{3–5} Indeed, TLP bonding enables repeated stacking of additional layers without remelting the joints at lower levels of

the stack, and hence can facilitate multilevel 3D interconnects.

Nowadays a major concern for TLP bonding is to lower the bonding temperature. In order to integrate a device that is sensitive to process temperature in different layers or integrate with other electronic devices which have various functions, a lower bonding temperature is always desirable. This low-temperature bonding can also reduce the process cost. If the bonding temperature needs to be lower than the melting point of the conventionally used tin (Sn)-based solder materials, indium (In) is a good solder candidate due to its lower melting point of 156°C as compared with that of Sn, 232°C. In fact, indium has been widely used as a solder in the bonding of photonic devices, where the limiting factors are temperature and thermal stress induced by the differential contraction of different materials. In addition, gold (Au) often serves as the

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underbump metallization (UBM) material since chips with a Au finish are quite common. The bonding temperature ranges from 165°C to 200°C, and the bonding pressure is between 0.3 MPa and 1.25 MPa.^{6–9} Au/In flip-chip bonding at about 150°C has also been reported,¹⁰ but it is basically controlled by solid-state interdiffusion instead of the liquid–solid reaction of the TLP process.

An excess of free indium often remains in the solder joints from the previous flip-chip bonding in the Au/In system. A large amount of liquid indium is preferred in the conventional solder bonding process because it allows good wetting of the UBM metal to form a continuous layer of intermetallic compounds. In particular, in order to create a liquid/solid interface between indium and UBM, sufficient liquid indium is needed to break up the thin AuIn₂ layer that is formed during the deposition of the Au capping layer to prevent indium oxidation.^{6–9} However, in TLP bonding, such a large amount of liquid indium is not as favorable, since it needs very thick UBM materials and a long time for complete intermetallic formation. The recently reported NiAu/In flip-chip bonding dedicated to 3D chip stacking used small solder volume (<6 μm thickness).^{3,4} However, indium was still not fully transformed into intermetallic compounds after bonding.^{3,4}

Controlling the growth of intermetallics is critical to obtain Au/In TLP joints with small solder volume. This requires good understanding of Au/In reactions at different conditions. In fact, the interdiffusion between Au and In can occur even at –50°C, and is believed to be the fastest among those of the transition metals.^{11,12} This indicates that the interval between solder deposition and subsequent dicing and bonding processes should not be too long when Au and In need to be deposited sequentially. Moreover, during the temperature ramp-up of the bonding process, a certain amount of indium will also be transformed into intermetallic compounds. Therefore, it is crucial to ensure that not too much indium reacts with Au before reaching its melting point. In order to provide sufficient liquid indium for TLP bonding, the multilayer stack needs to be optimized, i.e., the indium thickness should not be too thin. In addition, a thin diffusion barrier between Au and In may reduce the consumption of indium before melting. Recently, a very thin titanium (Ti) barrier of 1 nm to 5 nm was successfully introduced into the Pd/In system to control their reactions without changing each thickness.¹³ This method has not been studied in the Au/In system, and the suitable thickness of diffusion barrier needs to be found.

In this study, we investigated the intermetallic formation in Au/In films at both room and elevated temperatures. This also enabled us to analyze the kinetics of Au/In reaction in the solid and liquid states. Furthermore, we studied the impact of a diffusion barrier on Au/In interdiffusion at different

temperatures in order to fabricate Au/In TLP joints with very small solder volume (1 μm thickness).

EXPERIMENTAL DETAILS

Au/In interdiffusion was tested on unpatterned p-Si (100) with 250-nm-thick SiO₂ and 20-nm-thick Ti adhesion layer. Au and In of 99.9999% purity were evaporated sequentially to form a stack of Au/In/Au in a vacuum of 10^{–6} Pa. Table I lists the details of the multilayer stack used for this study. Film thickness was monitored with a quartz-crystal oscillator during evaporation. The sheet resistance of films was measured by a four-point probe, and the intermetallic formation was characterized by x-ray diffraction (XRD). In order to study the growth rate of intermetallic compounds, a 10-μm-thick indium film was bonded to a 0.6-μm-thick Au film on a Karl Suss FC6 flip-chip bonder. Bonding was performed at 100°C to 180°C with varying time. Samples (5 mm × 5 mm) were immediately quenched into water after bonding, and then put into epoxy for subsequent polishing to expose the cross-section (X-section). Intermetallic thickness was measured by scanning electron microscopy (SEM) with a backscattered electron detector.

Test samples containing either Ø40 μm or Ø60 μm bumps were prepared for flip-chip bonding. The pitch was 100 μm and 125 μm, respectively. The bonding process was also done on the aforementioned flip-chip bonder at 180°C. The bonding pressure was 1 MPa, and the bonding time ranged from 90 s to 200 s. TLP joints were connected in a daisy chain in order to measure the electrical connection yield, which was carried out by using a HP4156 probe station. A full daisy chain consisted of 2240 and 1380 joints for the Ø40 μm and Ø60 μm bumps, respectively. Two types of UBM material were used in our flip-chip bonding experiments. One was electroplated thick Au, and the other was evaporated thin Au in combination with electroplated copper (Cu). Cu was also chosen as the UBM material because of its common use in electronic packaging. Since a thin Ti diffusion barrier was introduced in the multilayer structure, indium was deposited by evaporation instead of electroplating and eventually released by lift-off. After bonding, some of the samples were sent for shear testing to measure the bonding strength, while the others were used for electrical measurements and X-section SEM investigations.

Table I. Details of the Film Stack Used for Sheet Resistance and XRD Measurements

	Au/In/Au	Au/Ti/In/Au
Thickness (nm)	400/1000/100 500/1000/500 160/1000/40	160/10/1000/40

RESULTS AND DISCUSSION

This section consists of two subsections. The first subsection presents some fundamental understanding on Au/In interdiffusion. This fundamental understanding allows us to develop a realistic Au/In TLP bonding process with small solder volume, for which the experimental data will be shown in the second subsection.

Kinetics of Au/In Interdiffusion

Sheet resistance measurements combined with XRD demonstrate the evolution of intermetallic phase in the sandwiched Au/In/Au films. The top layer of Au was used to protect the indium from oxidation. Since an increase in film resistance due to indium oxidation has been ruled out, we can exclusively correlate the change in sheet resistance with intermetallic compound formation. Figure 1 shows the sheet resistance evolution of two specimens at room temperature, where the nominal thickness of each layer in the Au/In/Au films is 400 nm/1000 nm/100 nm and 500 nm/1000 nm/500 nm, respectively. The thickness of indium is the same for these two samples, whereas the thickness of Au is different. Apparently, the speed of sheet resistance evolution depends strongly on the stack. For the sample with a thinner Au layer, there is a plateau in the sheet resistance curve and it took about 30 days to reach this value. The XRD spectrum (Fig. 2), which was captured on the 30th day after deposition, does not show any reflection peaks of either Au or In, while almost all the reflection peaks can be attributed to AuIn_2 . This indicates that Au and In have been fully transformed into AuIn_2 after 30 days. Indeed, the sheet resistance was quite stable afterwards and the XRD spectrum did not change over time. For the sample with thicker Au layers, the sheet resistance increased with time and it took more than 140 days to reach a plateau. Correspondingly, the XRD spectra (Fig. 3), which were captured on the 30th

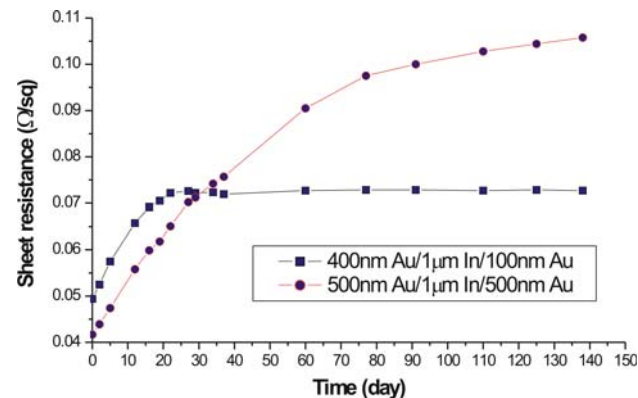


Fig. 1. Sheet resistance evolution of two Au/In/Au (400 nm/1000 nm/100 nm, and 500 nm/1000 nm/500 nm) sandwiched films at room temperature. It should be noted that the thickness of Au and In is the nominal thickness and is not calibrated.

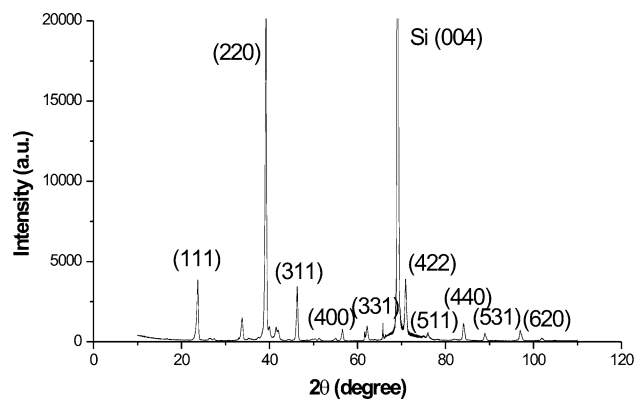


Fig. 2. XRD spectrum of the Au/In/Au (400 nm/1000 nm/100 nm) film captured on the 30th day (corresponding to Fig. 1) after deposition, showing that indium was fully transformed into AuIn_2 .

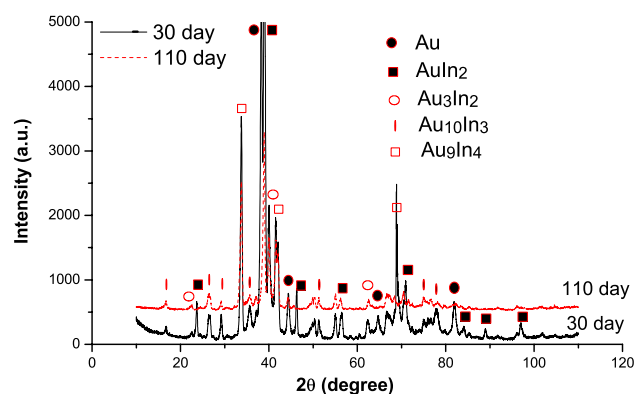


Fig. 3. XRD spectra of the Au/In/Au (500 nm/1000 nm/500 nm) film captured on the 30th and 110th days (also corresponding to Fig. 1), respectively, after deposition, indicating that indium was completely transformed into complex intermetallic compounds and that the phase evolution continues with time.

and 110th days after deposition, respectively, show that the phase evolution continues. First, indium peaks disappeared in the spectrum of the 30th day, indicating that all indium was transformed into intermetallic compounds. Second, Au peaks can still be found in this spectrum since there is an excess of Au in the stack for AuIn_2 formation. However, the remaining Au continues to react with AuIn_2 and will eventually be completely transformed into intermetallics. Hence the Au peaks either diminish or overlap with that of intermetallic compounds such as AuIn_2 after 110 days. For the same reason, some of the AuIn_2 peaks that appeared in the 30-day spectrum also later disappeared. Finally, it is worth mentioning that, according to literature, AuIn_2 is normally the first intermetallic compound to be formed. It will remain stable if there is sufficient indium in the system.¹⁴ Otherwise AuIn_2 will react with the excess Au to form new Au-rich intermetallics. Indeed, the phase formation in the second sample is complex, where at least AuIn_2 , Au_9In_4 , and $\text{Au}_{10}\text{In}_3$ coexist (see also Fig. 3).

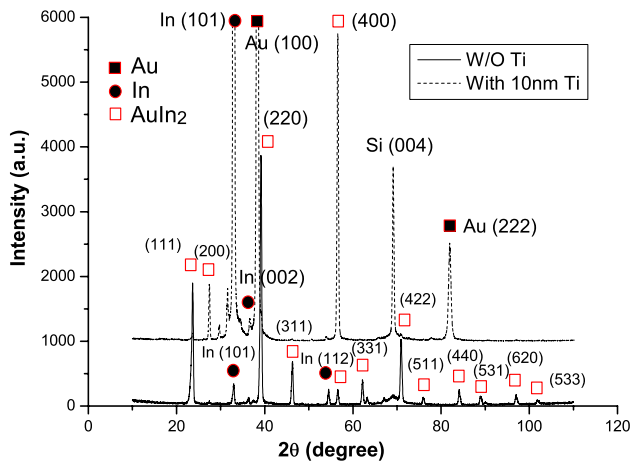


Fig. 4. XRD spectra of the Au/In/Au (160 nm/1000 nm/40 nm) and Au/Ti/In/Au (160 nm/10 nm/1000 nm/40 nm) films captured after phase stabilization (there was no Ti barrier between the indium and the thin Au top layer that was used to protect the indium from oxidation), indicating that Au/In interdiffusion is largely slowed down by introducing a thin Ti barrier.

Since Au/In interdiffusion occurs at room temperature, how to control the intermetallic formation becomes interesting. As mentioned in the introduction, we now introduced a thin Ti diffusion barrier between the Au and In. The multilayer stacks investigated were Au/Ti/In/Au (160 nm/10 nm/1000 nm/40 nm) and Au/In/Au (160 nm/1000 nm/40 nm). Here the thickness of Au also became thinner than the previous two samples in order to stabilize the phase formation quickly. As expected, the time to reach a stable sheet resistance value was about 26 h for the stack with a 10-nm-thick Ti barrier layer, while it was ~ 2 times longer for the stack without this barrier. Furthermore, as shown in the XRD spectra (Fig. 4) captured after phase stabilization, the Au peaks were very strong in the sample with the Ti barrier. In contrast, these reflection peaks cannot be distinguished for the sample without the Ti barrier, indicating that all the Au has reacted with indium to form AuIn₂. Since there is still excess pure indium left in both samples, indium peaks are present in both XRD spectra. However, for the sample without the Ti barrier, the intensity of the indium peaks is much smaller than for the sample with the Ti barrier. All this suggests that the 10-nm-thick Ti barrier can effectively slow down Au/In interdiffusion at room temperature.

A thin Ti barrier, on the other hand, does not block the intermetallic formation at high temperatures. This point is shown by comparing the XRD spectra of the Au/Ti/In/Au (160 nm/10 nm/1000 nm/40 nm) film before and after a 200°C anneal for 3 min in N₂ ambient (Fig. 5). Interestingly, the strong Au peaks that are present in the XRD spectrum before the anneal disappear after the anneal. This is attributed to the fact that there is only a limited amount of Au in this stack and all the Au has been transformed

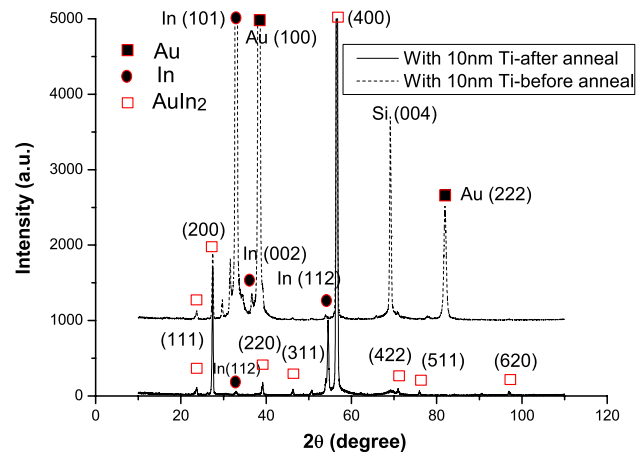


Fig. 5. XRD spectra of the Au/Ti/In/Au (160 nm/10 nm/1000 nm/40 nm) film before and after a 200°C anneal for 3 min in N₂ ambient, showing that a thin Ti barrier does not block Au/In reaction at high temperature.

into AuIn₂. Indium peaks remain in the spectra because there is excess indium in the system. Similar results are also observed when the thickness of the Ti barrier is increased to 20 nm (data not shown), indicating that even a 20-nm-thick Ti barrier does not inhibit the formation of Au/In intermetallic compounds at high temperatures.

It should be noted that a few reflection peaks in the XRD spectra could still not be identified for sure; these might correspond to Au₇In₃ (Fig. 3) and Au₉In₄ (Figs. 4 and 5). Moreover, it is hard to identify the pure Ti and Ti-contained compounds in the XRD spectra because either the amount of Ti is very small or the peak positions are very close to those of Au/In intermetallics. In fact, it would be interesting to study the effect of Ti on intermetallic compound formation in detail in order to understand whether the Au-In-Ti isothermal sections will be available, as reported in the ternary Ti-Ag-Cu system.¹⁵

Up to now we understand that, to some extent, we can control the Au/In reaction to make our TLP bonding easier: on the one hand, we use a thin Ti barrier to slow down Au/In interdiffusion at lower temperatures to achieve a larger process window; on the other hand, we still ensure intermetallic formation at higher temperatures for good TLP bonding.

In order to understand the fundamental kinetics of the Au/In reaction, we now study the growth rate of intermetallic compounds at different temperatures. Unlike the aforementioned sandwiched sample, here we bonded a 10- μ m-thick indium film to a thin Au film by applying a pressure of 0.7 MPa, and immediately quenched the sample in water after bonding. Since there is sufficient indium in the system, only the AuIn₂ intermetallic compound will be formed.^{9,14} This approach avoids the complexity of having to validate the diffusion laws when multiple intermetallic phases coexist. The measured

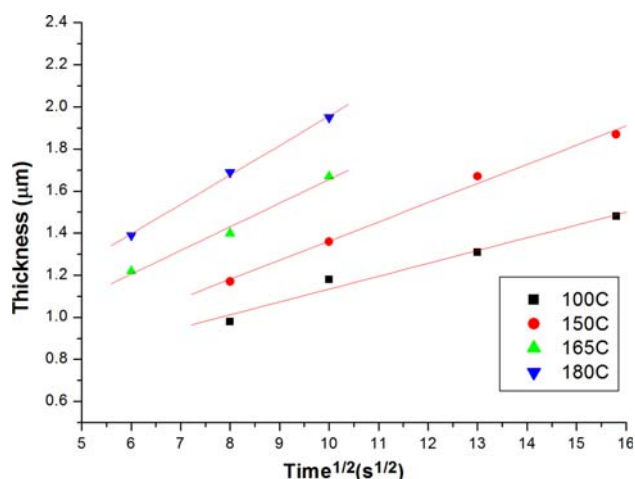


Fig. 6. The thickness of AuIn_2 versus the square root of the Au/In bonding time, indicating diffusion-controlled growth.

thickness of AuIn_2 versus the square root of the bonding time is shown in Fig. 6, revealing a linear relationship. This suggests that the kinetics of Au/In reaction follows a parabolic law and that the growth of AuIn_2 is controlled by diffusion. The intercepts ($t = 0$) of these lines with the vertical axis of Fig. 6 vary from $0.46 \mu\text{m}$ to $0.56 \mu\text{m}$. Since all samples were cooled very quickly after bonding, AuIn_2 of such thicknesses mainly grew during the temperature ramp-up of the bonding process. Thus we believe that a minimal indium thickness will be required in order to enable TLP bonding using this Au/In system. The minimal indium thickness can be estimated by $X_{\text{In}} = (\gamma_{\text{AuIn}_2} \times X_{\text{AuIn}_2}) / (0.33\gamma_{\text{Au}} + \gamma_{\text{In}})$. The density of Au and In is known, while it is not available for AuIn_2 in the literature. From our blanket film test, it is found to be approximately 12.1 g/cm^3 . If the thickness of AuIn_2 is taken as $0.56 \mu\text{m}$, the minimal indium thickness is estimated to be about $0.5 \mu\text{m}$.

The diffusivity D , which is determined by $X = 2\sqrt{Dt}$, is plotted against the reciprocal of the temperature in Fig. 7. The Arrhenius relation is well satisfied in two temperature ranges: between 100°C and 150°C , and between 150°C and 180°C . Consequently, the temperature dependence of D can be expressed as follows:

$$D = 6.43 \times 10^{-6} \exp\left(-\frac{0.46}{kT}\right) \text{ cm}^2/\text{s} \quad (T > 150^\circ\text{C}),$$

$$D = 1.20 \times 10^{-8} \exp\left(-\frac{0.23}{kT}\right) \text{ cm}^2/\text{s} \quad (T \leq 150^\circ\text{C}),$$

where k is the Boltzmann constant and T is the thermodynamic temperature in Kelvin. The activation energy here is 0.46 eV and 0.23 eV for the higher and lower temperature ranges, respectively. Temperature-dependent activation energy was also observed in the Cu/Sn system.^{16,17} This may relate

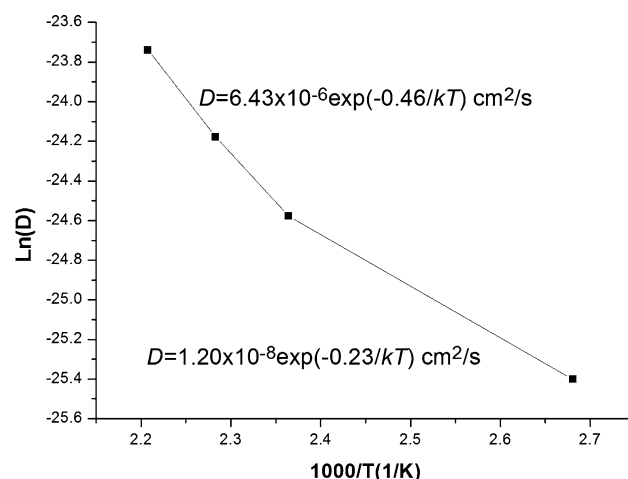


Fig. 7. Arrhenius plot of the diffusivities for the Au/In reaction and the activation energy calculated from the slopes of the lines.

to the different boundary conditions and diffusion mechanisms.^{17–19} Bulk diffusion is believed to be dominant at higher temperatures that can create a liquid/solid interface, while solid-state diffusion at low temperatures might be controlled by grain-boundary diffusion.

Our activation energy of 0.46 eV is close to the one reported by Liu and Chuang, 0.41 eV , which was obtained from similar soldering reactions of liquid In on Au.²⁰ In addition, our activation energy of 0.23 eV is the same as the one reported by Bjontegaard et al.,¹¹ whereas it is lower than the one reported by Shohji and Hasumi (0.45 eV and 0.52 eV),^{21,22} respectively, although all these values were obtained from the solid-state reaction. It should be noted that all the aforementioned activation energies were obtained from diffusion-controlled growth. In fact, an interface reaction-controlled growth mode was observed by Yost with an activation energy of 0.61 eV .²³ A similar growth mode was also reported by Millares and Pieraggi at temperatures below 100°C , with an activation energy of about 0.32 eV .²⁴ However, when the temperature was in the range of 100°C to 150°C , the interfacial reaction only controlled the growth of AuIn_2 at the early stage. The subsequent growth of intermetallic compounds became diffusion controlled.

Microstructure of Au/In TLP Bonding

The understanding on Au/In interdiffusion enables us to optimize the stack in microbumps and to carry out TLP bonding at 180°C . First, a $1\text{-}\mu\text{m}$ -thick indium was used in our flip-chip bonding, which is much thinner than the conventional thickness of solder medium. Then a 10-nm -thick Ti barrier was deposited between the Au and In to reduce the amount of intermetallic compound formed at room temperature and during the temperature ramp-up. Finally, a 10-nm - or 40-nm -thick Au capping layer was used to protect the indium from oxidation. Such an approach enables

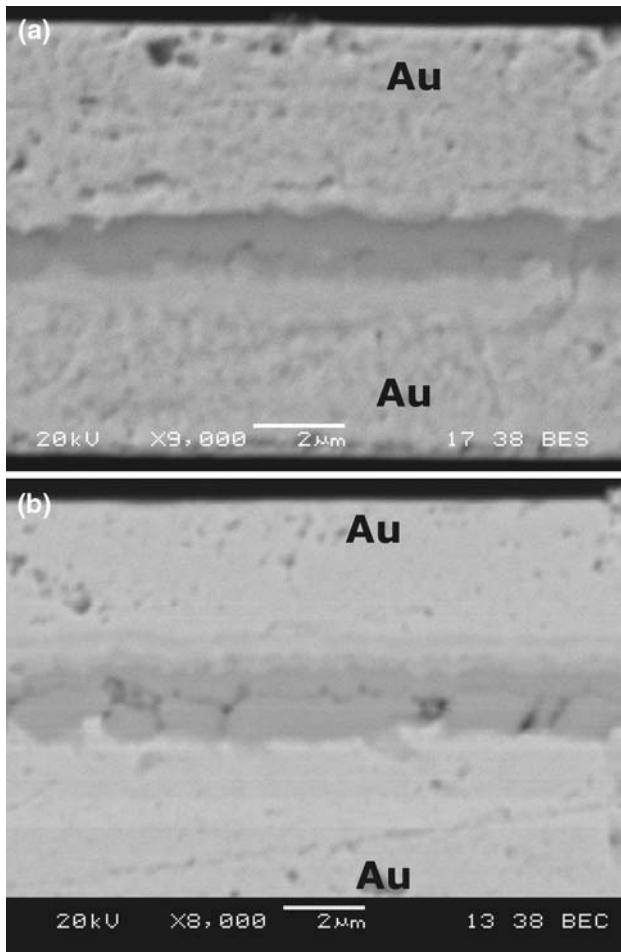


Fig. 8. Cross-sectional SEM of a Au/In/Au joint after flip-chip bonding. The stack of bumps in the upper chip was Au/Ti/In/Au (5000 nm/10 nm/1000 nm/10 nm), and was Au (5000 nm) in the lower chip. These two chips were placed face to face, and were bonded at 180°C with a pressure of 1 MPa for (a) 100 s and (b) 169 s, respectively.

us to have sufficient pure indium before reaching its melting temperature on the one hand, and not to leave any pure indium after the bonding process on the other hand.

Figure 8 shows a X-section of a Au/In/Au joint after bonding, where the initial stack of the upper and lower chips was Au/Ti/In/Au (5000 nm/10 nm/1000 nm/10 nm) and Au (5000 nm), respectively. The joint interface exhibits high quality, although sometimes cracks inside the intermetallics are still visible. The shear strength obtained from such samples is generally higher than 8.0 MPa. Nevertheless, the thickness of intermetallics increases with time, indicating that the phase evolution continues. According to the designed proportion of Au and In, the α_1 phase (a highly Au-rich intermetallic) will eventually be formed to connect the remaining Au layers on each side, which can enhance the adhesion strength to substrates. The α_1 phase is very stable and has been observed by Waelti et al.²⁵ in the Ni/In/Au system after annealing at 160°C for 1000 h.

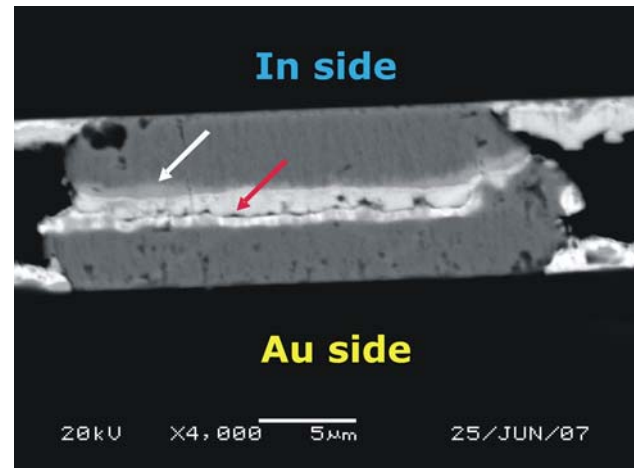


Fig. 9. Cross-sectional SEM of CuAu/In/AuCu joint bonded at 180°C/1 MPa/150 s. The stack of bumps in the upper chip is Cu/Ti/Au/Ti/In/Au (5000 nm/20 nm/160 nm/10 nm/1000 nm/40 nm), and is Au/Ti/Cu (500 nm/20 nm/5000 nm) in the lower chip. The thickness of Au and In layers is adjusted to achieve the proportion of indium of 35.2 wt.% so that the equilibrium composition is always in the solid-solution region.

The X-section of CuAu/In bonded to AuCu is shown in Fig. 9. In this case, indium not only reacted with Au, but also reacted with Cu (indicated by the white arrow). Nevertheless, the Cu/In reaction only occurred on the indium side (upper chip), where the Au layer is about three times thinner than on the Au side (lower chip). In addition, the Au/Cu reaction due to solid-to-solid interdiffusion cannot be distinguished on the thicker Au side. This indicates a slow reaction rate between the Au and Cu layers, which has been reported elsewhere.²⁶ The Cu/In reaction on the indium side may result from the fact that liquid indium quickly reacted with the underlying Au layer. Once this thin Au layer was fully consumed, indium then reacted with the adjacent Cu layer to form CuIn intermetallics. In contrast, some excess Au still remained in the joint on the Au side and hence the Cu/In reaction did not occur there. At first sight, this phenomena is beyond our expectations because firstly the growth rate of AuIn intermetallic compounds is about four times faster than that of CuIn intermetallics,² and secondly there is a Ti layer with a total thickness of 30 nm on the indium side (10 nm between the In and Au, and another 20 nm between the Au and Cu) which will further slow down the reaction rate there. However, there was still some remnants of Au on the Au side until all the indium is transformed into intermetallics. Since only a thin AuIn₂ layer lies between the In (upper chip) and Au (lower chip), we believe that the preformed AuIn₂ is also an effective dissolution barrier for liquid indium to dissolve Au.²⁷

It should be noted that there are still some cracks or voids at the joint interface (indicated by the red arrow). The amount of voids can be reduced either by increasing the initial thickness of the indium or

by decreasing the thickness of the Au capping layer. Both of these approaches help to break up the pre-formed AuIn₂ layer quickly and make intimate contact between the liquid In and Au. This will facilitate the formation of a continuous layer of intermetallics, and hence reduce the number of defects at the joint interface. A thinner Au capping layer is thus preferred when we use a small solder volume. Indeed, the interface of Au/In/Au in Fig. 8, where the thickness of the Au capping layer is 10 nm instead of 40 nm, is much better.

It is worth mentioning that all the indium was transformed into intermetallics in our microbump. This point was confirmed by XRD measurements, where Au₁₀In₃ and Cu₉In₄ were also identified in addition to the majority AuIn₂. Consequently, the melting temperature of the joints is only determined by the intermetallics, typically being higher than 451°C. This is significantly different from NiAu/In microjoints, as reported previously,^{3,4} in which excess pure indium still remained in the joints. The electrical connection yield was 100% for the daisy chain containing 1380 bumps with a diameter of 60 μm.

One may worry about the reliability of TLP joints, which are brittle in nature. In particular, the shear strength is generally not high in the Au/In system.^{3,4,12} However, the poor mechanical properties can be improved by introducing a polymeric underfill. A proper underfill can also reduce the local stress arising from repeated mechanical, electrical, and thermal cyclings. Moreover, we found that AuIn₂ is less brittle than Cu₃Sn and Cu₆Sn₅ based on nano-indentation tests, indicating less chance of being a site of crack nucleation in the Au/In TLP joints. In fact, we recently also found that TLP joints are very resistant to electromigration (EM).²⁸ Therefore, we believe that the reliability of Au/In TLP joints will be acceptable for 3D applications.

CONCLUSIONS

The Au/In reaction was systematically studied across a wide temperature range. It was found that the kinetics of AuIn₂ phase formation is diffusion controlled. The activation energy of the Au/In reaction is 0.46 eV at temperatures above 150°C, while it is 0.23 eV below that temperature. Moreover, a thin Ti barrier can strongly slow down Au/In interdiffusion at room temperature, while it does not block intermetallic compound formation at elevated temperatures suitable for TLP bonding. This ensures a large process window for Au/In flip-chip bonding. In addition, a minimal indium thickness of 0.5 μm is required in order to enable TLP bonding. Finally, Au/In TLP joints of Ø40 μm to 60 μm were successfully fabricated at low temperature and with 1-μm-thick solder material. This will facilitate multilevel 3D interconnects.

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