

## Assessment of Indium Joint Strength

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The purpose of research is to investigate indium joint strength with known indium oxide thickness. The indium joint strength was obtained by measuring the maximum load of indium solder with the comparison of the wetting angle. After the oxide thickness was grown at different temperatures in air, it is provided for the joint strength measurement. The indium joint strength with different oxide thicknesses was tested at a high strain rate enough for the interfacial failure by tensile loading. This investigation will be very useful to characterize the indium solderability at different environment in terms of the quantitative joint strength.

**Key words:** indium joint strength, indium solderability, indium characterization, indium oxide thickness

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### 1. INTRODUCTION

There are strict restrictions against the use of lead as a solder alloy in the human life and environmental concern, even if Pb provides many technical advantages. Instead, a relatively large number of Pb-free solders, such as Sn-Au, Sn-In, Sn-Ag, Sn-Bi, have been proposed with the expected levels of electrical and mechanical performance<sup>[1]</sup>. Indium as an elemental composition in Pb-free solder alloy has a significantly lower melting temperature of 156 °C, which is much lower than the melting temperature of Sn (232 °C). The melting temperature is the most important factor from a manufacturing prospective because thermoset polymers used in microelectronics packaging should not be degraded during the soldering operation. Currently, the highest exposed temperature of thermoset polymers is about 230 °C for 90 sec. during board assembly and reflow of solder balls. Even if indium has been used as one of elements that is major constituents, it was not well characterized in the mechanical joint strength at different environmental conditions.

100In is very ductile and has high thermal conductivity (80 W/mK). It also has a good wettability on many surfaces including ceramics, glass and quartz in low process temperature (slight above melting point of 157 °C). Besides, 100In is a good candidate solder for the fluxless solder sealing, which is free from the residue after post reflow.

To assess the joint strength of indium with different thickness existence of indium oxide layer at air ambient and differently controlled environmental reflow condition, indium

joint strength by single lap shear test and indium wetting angle measurement have been performed.

The aim of this research is to develop a soldering environment to meet acceptable indium joint strength for industrial requirements.

### 2. EXPERIMENTAL

Indium samples were cut from indium sheet (purity: 99.999%, Goodfellow) by razor blade. The samples were electrochemically polished in a 3:1 ethanol/nitric acid bath at 0 °C. and were washed for 2 minutes in distilled water, followed by immediately stored in nitrogen environment. The indium samples were annealed at the given different temperature for 2 hours on the hot plate. Four different oxide thicknesses were measured at the indium samples, which were grown at four different temperatures (25, 145, 160, 200 °C) on a hot plate. The oxide thickness was measured by NBTC' EP3 Ellipsometer.

For the single lap shear test, the indium sample was prepared in 3.5 by 3.5 mm and 0.64 mm in thickness and bonded with the silicon substrate by hot, press. As shown in Fig. 1, the silicon substrate is 6.5 mm wide and 28 mm in length. The bonded area on the silicon substrate is deposited with titanium (0.04 μm) and Au (0.5 μm) respectively.

Indium was reflowed on the Au layer between a pair of silicon substrates for single lap shear test. This indium reflow for bonding was performed on the hot plate using a specially designed fixture for this test. The reflow condition of indium is around 230 °C for 20 minutes in an air ambient environment.

Single lap shear tests were performed using the Bose ELF3200 system operated by Wintest software. The strain

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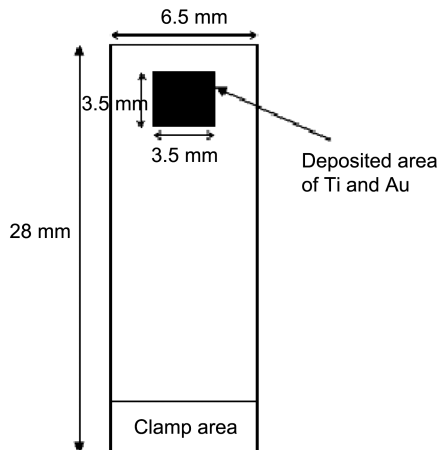


Fig. 1. Top view of bonding substrate.

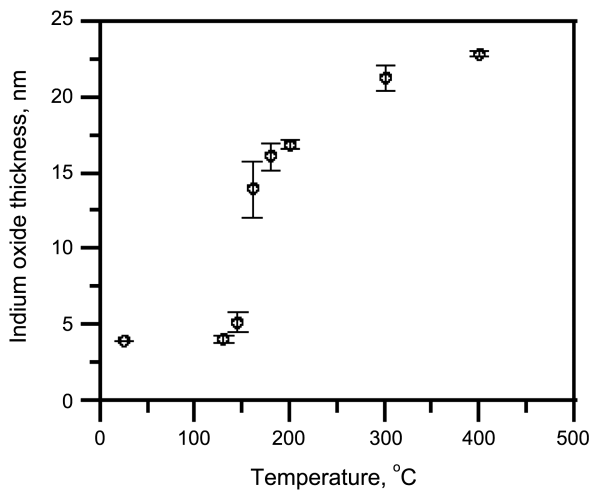


Fig. 2. Variation of indium oxide thickness with temperatures at ambient condition.

rate applied for the shear test is high enough to cause interfacial failure between indium and the silicon substrate<sup>[2-3]</sup>. The maximum joint strength ( $\tau_{ult}$ ) of each indium sample with different oxide thicknesses was obtained by dividing the applied maximum tensile load,  $F$ , by the bonded area,  $A$ . ( $\tau_{ult} = F/A$ )

The indium samples for wetting angle measurement were prepared by melting on the Au deposited silicon wafer substrate on the hot plate (200 °C for 2 min.) at both an air ambient condition and in a glove box<sup>[4]</sup>. The atmosphere condition of glove box can be controlled by nitrogen and hydrogen gas. The glove box can be operated at  $O_2 < 0.1$  ppm,  $H_2O < 1$  ppm, and  $H_2 = 1.5\%$ . After the wetting was formed, wetting angles were measured by Wyco surface profiler. The wetting angle was determined at the interface of indium and the Au layer on the substrate.

Figure 2 shows that the indium oxide growth suddenly increases at indium melting temperature (156 °C). Initial indium oxide thickness is about 4 nm at room temperature

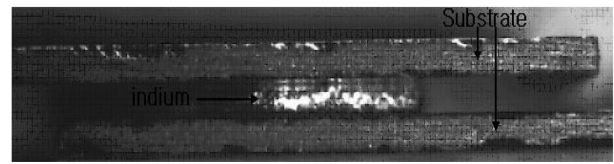


Fig. 3. The shape of indium shear test sample.

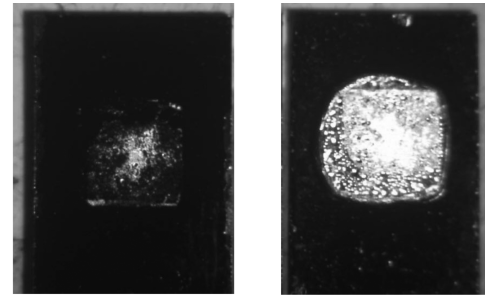


Fig. 4. A pair of test sample after joint failure test.

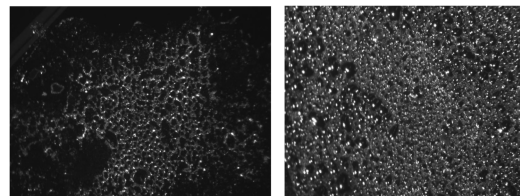


Fig. 5. Magnified view (40 X) of the failure site in Fig. 4.

and it is about 17 nm at 200 °C after 2 hours in an air.

Figure 3 shows real shear test sample before testing. Indium with 4 nm oxide thickness was reflowed to the substrates at 230 °C for more than 5 min in an air.

### 3. RESULTS AND DISCUSSION

After the joint failure test by single lap shear, the failure mode was dominated by interfacial failure between indium and the substrate as shown in Fig. 4. The magnified view of Fig. 4 by 40 times shows the void trace formed at the interface between indium and the substrate in Fig. 5.

Figure 6 shows one of representative curves which is tested for indium with a 4 nm oxide thickness. The joint strength is 1.35 MPa with the standard deviation of 0.2 MPa at a strain rate of 0.5 mm/sec. Figure 7 shows the joint strength variation with the increase of initial oxide thickness at the air ambient reflow condition. At an air ambient reflow condition of 230 °C, indium joint strength is decreased to below 1.0 MPa due to the void existence and thicker oxide layers.

Pure indium (4 nm oxide thickness) wetting shapes at an ambient condition and reduced environment ( $O_2 < 0.1$  ppm,  $H_2O < 1$  ppm, and  $H_2 = 1.5\%$ ) are compared with each other in Fig. 8. The indium wettings are formed on the Au-coated

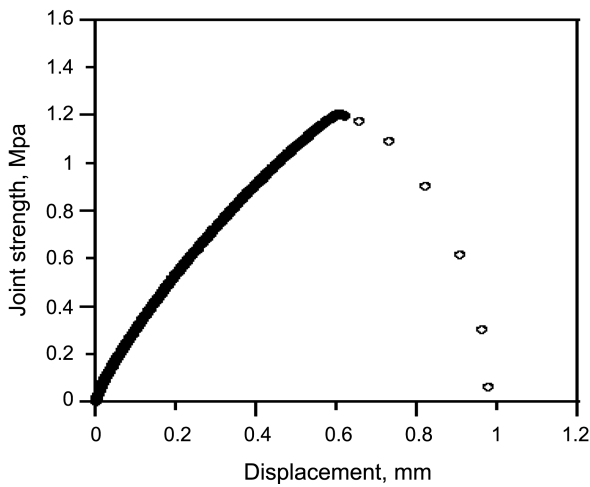


Fig. 6. Indium joint strength at 230 °C reflow.

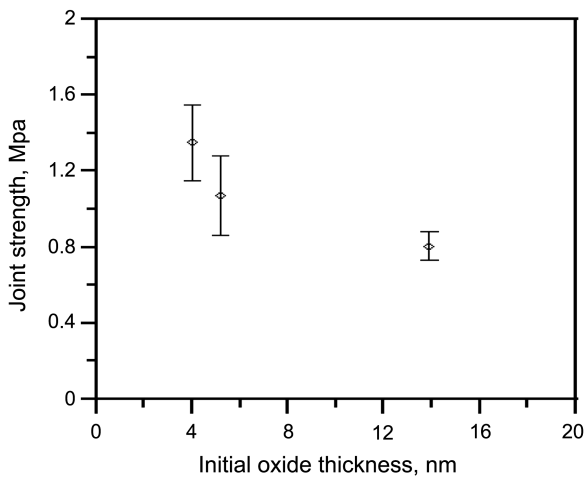


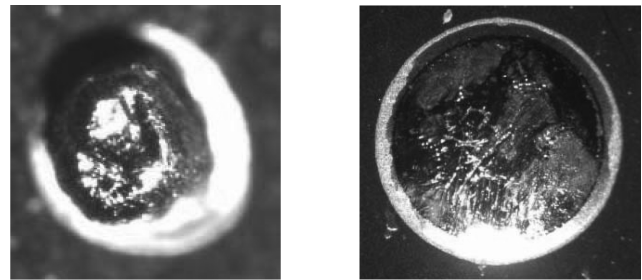
Fig. 7. Indium joint strength at air ambient reflow condition.

substrate. The wetted indium at ambient condition is just melted on the Au layer in Fig. 8(a), but the wetted indium in a reduced environment shows the pronounced spreading after its wetting, as shown in Fig. 8(b). The indium wetted in a reduced environment shows better wetting than that at air ambient condition.

Wetting angle measurement were attempted for indium samples with different oxide thickness at air ambient condition<sup>[5]</sup>. The wetting angles were close to 90 degrees, which is difficult to determine their wetting angles by Wyco surface profiler. However, in a controlled environmental glove box condition, the wetting angles increased with increasing of indium oxide thickness, as shown in Fig. 9.

**4. SUMMARY**

Pure indium’s solderability has been investigated by single lap joint shear test and wetting angle measurement. The



(a) Ambient environment (b) Reduced environment

Fig. 8. Comparison of wetting shape at different environments.

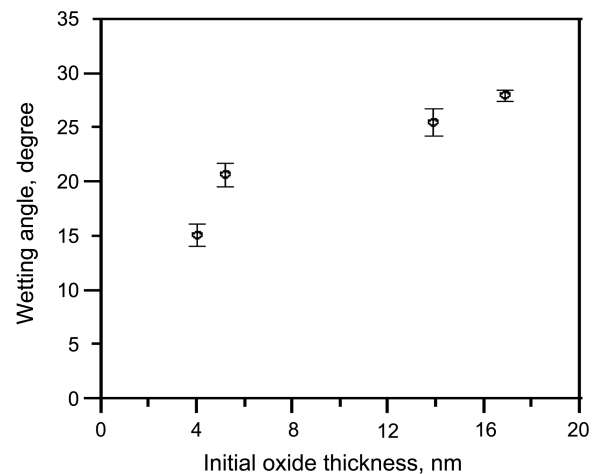


Fig. 9. Variation of wetting angle in a reduced environment.

indium oxide thickness was measured at different temperatures. The joint strength of indium at air ambient condition is controlled by the void existence and oxide thickness. Good wettings are observed at the controlled environmental condition ( $H_2O < 1$  ppm,  $O_2 < 0.1$  ppm, and  $H_2 = 1.5\%$ ). The effect of indium oxide thickness on joint strength will be investigated at the controlled environmental condition.

In the consideration of importance of indium as one of the major constituent solder elements, the present assessment of indium’s solderability with different oxide thickness will greatly contribute the determination of appropriate and acceptable environmental conditions for indium soldering in the packaging industry.

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