Development of Novel Intermetallic Joints using Thin Film Indium Based Solder by Low Temperature Bonding Technology for 3D IC Stacking

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Abstract

Low temperature bonding technology was developed using In-alloy on Au at a low temperature below 200 °C forming robust intermetallics (IMC) joints with high remelting temperature (>300°C), so that after bonding, the IMC joints can withstand the subsequent processes without any degradation. Process parameters on the solder joint were optimized extensively in bonding and annealing process (temperature, time, and pressure). The joint fabricated at an optimal condition, which is 180°C for 45sec followed by annealing at 120°C for 12hrs, was evaluated in terms of microstructure and compositional observations by means of scanning electron microscope (SEM) and transmittance electron microscope (TEM). As a result, it was confirmed that the joint was completely occupied with the Au-In based IMC phases. And the re-melting temperature was measured as above 400°C by using Differential Scanning Calorimetry (DSC) and Thermo-Mechanical Analysis (TMA). This IMC joint showed a high bonding shear strength (>20MPa) and a low electrical resistance (<100mΩ). Based on this study, the 3 stacked dice with 8x8 mm² dies with ~1700 I/Os of 80um solder bumps were fabricated in a chip to chip stacking method. It showed uniform bonding all over the die in each layer and the high bonding strength of ~40 MPa and passed the 3 times reflow test at 260 °C. The IMC joint reliability was examined. After going through the multiple reflows at 260°C, the bonded samples exhibited no delaminating and no changes in the bonding strength and the electrical resistance.

Introduction

3D integration has been a mainstream to be developed by the industries and the research institutes in order to make electronic products with multi-functions, high performances and a small form factor for the different applications [1-2]. In case of the extended applications, such as Bio, MEMS, Optical, RF devices, the integration process is required to be done at a low temperature below 200°C so as not to degrade the device performance. The current integration method is using somewhat higher temperature around 300°C or even higher for bonding and interconnecting the different devices or wafers in the vertical fashion [3-5]. A high bonding temperature may degrade the performance and the sensitivity of the Bio, MEMS, Photonics and RF devices. Therefore, a low temperature bonding at less than 200°C is a must for vertically integrating the different systems such as multifunctional devices into a System in Package (SiP).

Low temperature bonding method allows stacking the devices vertically with reduced thermal damage and stresses due to the lower processing temperature. Since the joints form robust intermetallics (IMC) interconnects with high remelting temperature after bonding the second layer to the first layer below 200°C, the next third layer can be stacked up subsequently. Indium layer with Ni, Cu or Au has been used for the low temperature bonding in 3D integrations [6-8], since the In has a low melting temperature (~156°C) [9]. However, because of the fast interdiffusion between the In and the pad metals (Cu, Ni, Au) even at a room temperature, thin In layer is susceptible to get transformed shortly to an alloy layer with higher melting temperature causing the bonding temperature to be raised by much higher than 30°C. In the end, bonding with thin In layer could be happened higher than 200°C. Therefore, they have been using thicker In layer or a kind of barrier layers, such as Ti layer, was tried to be inserted to prevent the excessive interdiffusion [10]. Another important issue in this technology is to choose the right composition of the solder and the under bump metallurgy (UBM) to form nothing but IMC having a high remelting temperature, because In atoms with additional elements tend to form ternary eutectic phase which has even lower melting temperature than In binary eutectic point [11]. Hence we need to avoid forming the ternary eutectic phase by designing the suitable layer combination.

Figure 1 Schematics of stacked dice fabricated by low temperature bonding

In this study, thin In based solder layers were chosen for the low melting temperature solder and bonded to thin Au layer to form Au-In based IMC after bonding. The layer structure was designed based on the ternary phase equilibrium considering the phase combinations. The effects of processing parameters of bonding and annealing on the bonding shear strength were studied through the design of experiment (DOE) to get the optimal condition. The IMC
joints were extensively investigated in terms of re-melting temperature, microstructure observations, mechanical and electrical properties. Au-In based IMC joints were applied to the 3 layers C2C die stacking as shown in Fig. 1. The test vehicles were consisted of ~1700 I/Os of 80µm solder bumps with 200µm pitch. The die was 8 x 8 mm² with 200µm thickness. The 3 layer stacked dice were examined in-depth.

Experimental Procedure
For the study on design of experimental (DOE), In based solder metallization was deposited on a Au layer in an 8" Si wafer for the top die with e-beam evaporation chamber. 0.05µm Au was coated to prevent the In based layer surface to get oxidized. And then the coated Si wafer was diced into 2x2 mm² size for bonding. For the bottom substrate, thin Au/Ti films were deposited on Si wafer and diced to 3x3 mm² of a die size. The bonding condition was varied as a function of the bonding time and pressure and the subsequent annealing condition was also investigated in terms of time and temperature. Table I shows the parameters used for the DOE study. To find out the optimal condition, the response surface model was utilized and total 30 numbers of experiments were carried out to complete the DOE study. And the shear strength was measured as a response to determine the optimal condition. The bonding was repeated 3 times to see the repeatability.

Table I Parameters for DOE design

<table>
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<th>Factor</th>
<th>Name</th>
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<th>High Actual</th>
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<td>sec</td>
<td>Numeric</td>
<td>20</td>
<td>60</td>
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<tr>
<td>B</td>
<td>BPress</td>
<td>MPa</td>
<td>Numeric</td>
<td>3</td>
<td>6</td>
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<tr>
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<td>degC</td>
<td>Numeric</td>
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<td>120</td>
</tr>
<tr>
<td>D</td>
<td>ATime</td>
<td>Hrs</td>
<td>Numeric</td>
<td>5</td>
<td>12</td>
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</table>

The test vehicles for the die stacking were fabricated on 8" Si wafers with SiO₂ coating. On the front side, 1µm Au/0.1µm Ti layers were sputtered on SiO₂/Si and patterned to form circles with 100µm diameter. Then the wafer was back-grinded to 200µm thickness. The back side was coated by SiO₂ for the passivation layer. And 1µm Au/0.1µm Ti layers for the under bump metallurgy were deposited in the sputter chamber, followed by the wet etching process to pattern circular pads with 100 µm in diameter. The 20µm dry film was laminated on the back-side and then developed to open the solder bump. In-based solder and Au films were sequentially deposited on top of Au in the evaporation chamber. Then the dry-film was striped for the lift-off process. Figure 2 shows the optical image of the die consisted of ~1700 I/Os with 80 µm solder bumps and <200 µm pitch. Table II lists the test vehicle dimension.

Figure 2 Optical microscope image of solder bumps and pads on the top surface of test vehicle

Table II Test Vehicle Dimensions

<table>
<thead>
<tr>
<th>Die (Chip) Size</th>
<th>~ 8x8 mm²</th>
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<tr>
<td>Wafer Size</td>
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<tr>
<td>Chip Thickness</td>
<td>200 µm</td>
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<tr>
<td>Solder Bump Diameter</td>
<td>80 µm</td>
</tr>
<tr>
<td>Pitch</td>
<td>&lt;200µm</td>
</tr>
<tr>
<td># of I/O</td>
<td>~1700</td>
</tr>
<tr>
<td># of Stacked Chip</td>
<td>3 Layers</td>
</tr>
<tr>
<td>Joint Thickness</td>
<td>2.5 µm Solder</td>
</tr>
<tr>
<td></td>
<td>1-2 µm UBM</td>
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</table>

Results and Discussions

Solder Design
In order to bond a thin In film on Au pad below 200°C, a diffusion barrier layer is needed in between In and Au layers, since the Au diffusion into In layer is fast enough to form Au-In intermetallics even at a room temperature. For our study, thicker In layer makes the processing time longer or the temperature elevated, which it is not right way to go for. So we need to use a kind of diffusion barrier layer in between In and Au layers for minimizing the interdiffusion before...
bonding. On the other hand, this diffusion barrier layer should not be interrupting the interfacial reaction between the In and the Au layer in bonding that will create intermetallics completely after bonding. In this study, thin diffusion barrier layer was inserted in between In and Au layers to inhibit from fast Au diffusion. Thin diffusion barrier layer may react with Au at the interface forming thin intermetallics, which can act as a diffusion barrier temporarily before bonding so that In layer is able to be preserved. Therefore, this In layer bump was able to form a uniform joint at a low temperature.

**Design of Experiments**

Based on the previous work [12], we could manage to bond uniformly with In alloy solder at 180°C at lowest. In this study, the temperature was fixed as 180°C, and the time and the pressure in bonding were optimized together with the annealing temperature and time as listed in Table I. After carrying out 30 numbers of experiments, all the joins were tested for the bonding shear strength. Using these testing results, the data were analyzed by means of ANOVA (Analysis of Variance) method. It was found that higher bonding force, higher annealing temperature, and a longer annealing time made a stronger bonding strength. And a certain point in between the minimum and maximum values in bonding time showed the highest bonding strength as shown in Fig. 3. As a result, the optimal condition was obtained, 40 sec. as a bonding time, 6MPa as a bonding pressure, 120°C as an annealing temperature, and 12 hours as an annealing time. With this parameters, the Au-In based IMC joint is expected to have 15.5 MPa as calculated.

**Figure 4** SEM micrograph of the cross sectional view of the Au-In based IMC joint fabricated with optimized condition

**Figure 5** SEM images of fractured surfaces in (a) Chip side and in (b) Substrate side

**High Temperature Stability of Au-In based IMC Joints**

To confirm whether this IMC joint will withstand in a subsequent higher temperature reflow process, the joints were reflowed repeatedly three times using a standard Pb-free reflow profile with 260°C peak temperature. After 3 times reflows, there was not noticeable change found in the microstructures and the shear strength values. Figure 6 exhibits the SEM image of the cross sectional view of IMC joints after 3 times reflows. The IMC composition was almost constant regardless of post processes, such as reflows for SMT. It means that the IMC joints did not change much even after multiple reflows. Moreover, the bonding strength was
also consistently obtained with 20~30 MPa after reflows with being broken along the IMC joins. It proves that the Au-In based interconnects are stable and robust even at high temperature processes around 260°C and enables to be processed for the vertical integration by stacking.

Figure 6 SEM micrograph of the cross sectional view of the Au-In based IMC joint fabricated after 3 times reflow tests at 260°C

Mechanical and Electrical Characteristics of Au-In based IMC Joints

The elastic modulus and the hardness of the Au-In based IMC joint were evaluated by means of a nano-indentation method. Since the IMC joint was too thin to be recognized under the optical microscope for placing the tip right into the IMC joint of the cross section of the samples, the measurement was carried out along on the fractured surfaces on each side of chip and substrate. As a result, elastic modulus was about 81 GPa, which is a bit higher than Young’s modulus of Au (78 GPa)[13]. However, this is even lower than that of Cu$_6$Sn$_5$ (112.6 GPa) or that of Cu$_3$Sn (132.7 GPa) [14]. Lower modulus is better for reducing the bonding and thermal stresses accumulated in stacking process. The hardness was measured as 1~2 GPa.

Figure 7 Schematic of test vehicle for the electrical resistance measurement

Different type of test vehicle was fabricated to measure the electrical resistance of the Au-In based IMC joint in Kevin’s measurement method. Two bumps with 300μm diameter were formed far away 800μm each other. Figure 7 depicts the schematics of the test vehicle used. Just after bonding before annealing, the IMC joint resistance was almost 50mΩ and did not change further as the annealing process is going on as shown in Fig. 8. In addition, it was found out that the resistance was not degraded even after 3 times annealing.

Figure 8 IMC joint electrical resistances as a function of annealing time at 120°C

3 Layer Die Stacking by Chip to Chip Method

8 mm x 8 mm sized dice with 200 μm thickness were fabricated for the die stacking. The 400 um substrate in thick with UBM (Au and Ti) was used as the first layer. The second and the third layers were 200 μm thick and consisted of In solder and UBM metallization on the back side and Au and Ti on the front side.

For C2C stacking, a bonding process at 180 °C for 45sec with 6MPa was repeated to stack up 2 layers on the substrate. And then the samples were annealed at 120 °C for 12 hours. Figure 9 demonstrates SEM images of the cross sectional view of the 3 layer stacked die with Au-In based IMC joints. All the bumps were uniformly bonded to the UBM pads. Firstly formed IMC joint for the second layer stacking endured the subsequent stacking process of the third layer.

Figure 9 SEM micrographs of the cross sectional view of 3 layer stacked dice with 200um thickness consisted of 1700 I/Os of 80 um solder bumps and with <200 μm Pitch
The shear strength and tensile pull test of the IMC joints in the stacked dice were measured. Since the Si die with 200 µm can be easily broken when being pushed by shearing tool, we used 400 µm thick die instead for the shear test. The second IMC joint between 2nd and 3rd layers were sheared first with holding the first IMC joint firmly, followed by the first IMC joint testing. It was shown that 60MPa in the first joint and 55MPa in the second joint were obtained respectively. It is thought that the higher bonding strength in the first joint was attributed to bonding processes experienced by two times. Stronger bonding strength in the first IMC joints was observed in the tensile pull test as well. We used the jig for fixing top and the bottom surfaces of the stacked die and then installed this to Intron for pulling. Then most of failure occurred between the 2nd and the 3rd layers as a mixture failure mode of both inside IMC joint and the UBM interface. It indicates that the first IMC joint is somewhat stronger than the second IMC joint. However, the strength of the 2nd IMC joint was almost 43–57 MPa. Those mechanical strength values were high enough to be comparable with other IMC joints [8,10,15].

Due to the size limitation of the platinum crucible containing the samples in DSC equipment, the stacked die was not able to be put for the re-melting temperature measurement. So instead, an indirect approach was tried to measure the transformation temperature with Thermal Mechanical Analyzer (TMA), which detects the dimensional change of the stacked die with increasing the temperature. The whole body of the stacked die will expand as the temperature is raised. However, when the IMC joints melt, then the die would be collapsed down resulting in the negative slope in the TMA curve. Then we can regard this temperature as a re-melting temperature. Hence, it is seen that the re-melting temperature of the IMC joints in the 2 layers and 3 layers stacked dice is higher than 400°C.

The stacked samples have gone through the 3 times reflow tests at 260 °C to investigate the high temperature durability. There was not significant degradation in the IMC joints detected by observing the cross sectional view of the interfaces. And the IMC composition did not vary further. It means that the IMC joints on both interfaces would be stable enough to withstand the next following process steps accompanying with the high temperature. The IMC joint strength was evaluated by means of a shear test and a tensile pull test. The shear strength value goes down a little bit in each interface after reflows as shown in Fig. 10. Fractured surface observation reveals that the interface failure between UBM and the Si die took in part in the fracture, indicating the decrease in the shear strength may be caused by weakened interfaces, not only due to the IMC joint. Therefore, it is confirmed that the IMC joint itself remains strong even after the multiple reflow tests at 260 °C.

Therefore, the die stacking with low temperature bonding was demonstrated to be promising with the high temperature robustness by eliminating the concerns of the thermal issues in stacking.

Conclusions

Novel IMC joints consisted of Au-In based was developed by means of a low temperature bonding below 200°C. The process was optimized by DOE and the optimal condition was obtained. The Au-In based IMC joint showed reliable interconnects property in stacking processes as well as the subsequent high temperature reflow processes. Some important results are summarized as in the following.

1. The thin In based solder in between Au formed Au-In based IMC, of which re-melting temperature was higher than 300°C measured by DSC. Au-In based joints were survived after reflowing three times at 260°C, which means that the Au-In based IMC joints are robust enough to do the stacking process for 3D IC integration.
2. The optimal process condition to obtain the highest bonding strength possible was analyzed and bonding at 180°C for 45sec with 6MPa, and then followed by annealing at 120°C for 12hrs was achieved as a result of DOE study.
3. Reliability data such as 20~30 MPa of the bonding shear strength and <100mΩ of the electrical resistance were observed in the Au-In based IMC interconnects. Those values are comparable with other IMC joints such as CuSn IMCs.
4. C2C stacking with 3 layers of dice was carried out by means of a low temperature bonding with Au-In based IMC joints at 180°C. During the stacking process, Au-In based IMC joints were stable and successfully stacked up the next layer without damaging the IMC joints formed in the previous stacks. Also, the 3-layer stacked dice were able to go through the reflow processes with a peak temperature of 260°C without degradation.

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