Investigation of adhesion features of bonded interfaces

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ABSTRACT
With the increasing application of flip-chip technology in the microelectronics industry, the adhesion strength of interfaces in the flip-chip microelectronic structures has become an important issue for manufacturing and operation. The existence of defects in the corresponding interfaces can gradually degrade the interfacial adhesion when the package is exposed to the high temperature and humidity. First, the effects of the temperature and relative humidity on the warpage of PBGA package were studied by using shadow Moire method. Then, the effects of IC package with different solder mask thickness under different temperature and humidity environment conditions on the adhesion strength were studied. The control parameters are environmental temperature, environmental humidity, soaking time and solder mask thickness. The interfacial adhesion strength was obtained by using a dynamic material testing system (MTS 810) with a set of special testing jig. By Statistical Package for the Social Science (SPSS) regression analysis, the relative equation of the interfacial adhesion strength to the humidity weight and humidity weight to the other parameters (i.e. temperature, soaking time and solder mask thickness) can be determined.

Keywords: underfill, solder mask, adhesion strength, moisture, soaking time

1. Introduction
The increasing demand for high-performance and high-density plastic packages has made flip chip technology a key solution in the microelectronics industry [1]. However, the reliability of flip chip assembly is also of concern during manufacturing and operation, due to the great difference in its interior material properties and characteristics.

The underfill used in flip chip assemblies, composed of epoxy and filler particles, mechanically couples the chip and
substrate, as well as filling around the solder joints. This can greatly increase the solder joint fatigue life by at least an order of magnitude [2]. The solder mask is a thin layer of polyamide material which is used in the substrate as the surface layer to define wettable areas for the soldering process [3]. For application in flip chip packages, the solder mask layer also serves as an adhesive between underfill and substrate, and takes a significant portion of the interconnect thickness, even though the thickness of the solder mask ranges from 20 μm to 50 μm only [4].

All polymer materials will absorb water to some extent and their mechanical behavior will reflect this interaction. The effect of the absorbed moisture is to decrease its complex dynamic modulus and decrease its glass transition temperature (T_g) [5]. For the more polar polymers, such as polyamides, the effect may be appreciable. The diffusing water disrupting the hydrogen bonding between the molecular chains of the adhesive. A decreasing of the adhesive modulus may decrease the stress concentrations in the joint. Nevertheless, since the T_g of the adhesive may have been appreciably lowered, the high temperature performance of the joint will obviously have been decreased.

During solder reflow, the high temperature will result in high thermal stresses in material interfaces due to mismatches in material coefficients of thermal expansion. In addition, the hygroscopic stresses induced through moisture absorbing will add to thermal stresses to degrade the interfacial adhesion severely [6,7]. Hygroscopic stresses in over-molded wire bond Plastic Ball Grid Array (PBGA) and molded flip chip PBGA after moisture conditioned at IPC Moisture Sensitivity Standard level 3 (IPC: The Institute of Interconnecting and Electronic Circuit) have been found to be higher than that of thermal stress at the peak temperature of reflow soldering [8].

The main mechanism of adhesion for most adhesive/substrate interfaces is described by adsorption theory. This theory states that materials will adhere due to the interatomic and intermolecular forces which are established between the surfaces of the adhesive and substrate [5]. It has been reported that the effect of moisture on the strength of adhesively bonded joints is significant due to the deterioration of the adhesive layer and the interface, the stronger interfacial bonds increased the environmental resistance of the adhesive/substrate to attack by moisture [9]. In addition, the adhesion strength for corresponding interfaces of die passivation, underfill and solder mask materials play extremely important roles in the interconnect reliability for flip chip packages [10]. In the case of thermosetting polymers, more are hydraulically stable in the presence of water. However, at relative high temperature, many of these polymers will be susceptible to hydrolysis and show appreciable loss of mechanical properties and increase the rate of adhesion strength loss.

Extensive studies on the effects of moisture absorption and solder reflow on the delamination and warpage of flip chip PBGA package, and the adhesion features of underfill/die passivation/die and the reliability of solder bumps have been made [11-19]. However, information on hygroscopic and thermo-mechanical behaviors of the underfill/substrate adhesion is still insufficient, especially as related to the thickness of solder mask.

This current paper studies of the effects of temperature, relative humidity, solder mask thickness and absorption time on the moisture weight gain and adhesion strength for the button shear test specimen. In addition, the relationship between the moisture weight gain and adhesion strength was also investigated. The research results can be helpful to identify improvements required in the adhesion performance of underfill/solder mask/substrate interface.

2. Experiment

2.1 Materials and specimens
The specimen used in this study is a button shear test joint, as illustrated in Fig. 1. It consists of three different materials, (1) the underfill composed of 60% epoxy and 40% silica filler, (2) a photosensitive type polyamide solder mask, and (3) an epoxy-coated FR-4 substrate with copper plating on both sides.

![Diagram of button shear specimen](image)

Fig. 1. The dimension of the button shear specimen.

FR-4 substrates measuring 160 mm × 160 mm were first cleaned and etched, followed by coating with solder mask and curing at 150 °C for 30 minutes. Then, the FR-4 substrate with solder mask was sectioned into 70 mm × 13 mm strips which were subsequently transfer molded with the underfill. Finally, specimens were cured at 165 °C for 4 hours. Three types of specimen with different thicknesses of the solder mask layers, 20 μm, 40 μm and 60 μm, were chosen for comparing the effect on shear strength. The underfill button is a truncated cone with an angle of 15.8 degrees and a height of 6 mm. The top and bottom diameters of the button are 9 mm and 11.3 mm, respectively, with a nominal bonded area of 100 mm².

Before the experiments proceeded, the specimens were screened by an eddy current film thickness detector (LH-330, Seiko Instruments Co., Ltd.) with accuracy of 0.1 μm. A maximum 3 μm error of the solder mask thickness was acceptable.

2.2 Preconditioning and simulating soldering temperature cycle

After curing, the specimens were dry by baking at 125 °C for 24 hours and then placed in a microprocessor-controlled environmental chamber for moisture preconditioning, it was shown in Fig. 2. Four preconditioning levels, 30 °C/30%RH, 30 °C/60%RH, 60 °C/30%RH and 60 °C/60%RH, were chosen for each type of specimen respectively. Moisture weight gain measurements were carried out at 24, 48, 72, 120 and 168 hours using an electronic balance with an accuracy of 10⁻³ mg. The sample of five parts was used at each test condition. Subsequently, the specimens were retrieved from the chamber and exposed to solder reflow simulation. Then, the button shear test was performed at room temperature.
2.1. Shadow Moiré [20]

Shadow moiré provides whole-field maps of out-of-plane displacement, and suits for the warpage measurement of PCB and plastic IC packages. Figure 3 is a typical arrangement for shadow moiré contouring. The CCD camera and light source are located at the same perpendicular distance, \( L \), from the grating. The governing equation of the out-of-plane displacement can then be expressed as

\[
W = \frac{g}{\tan \alpha + \tan \beta} N_z \tag{1}
\]

where \( g \) is the grating pitch, \( N_z \) is the moiré order, and \( W \) is the axial distance from grating plane to object. \( \alpha \) and \( \beta \) are the incidence angle and viewing angle, respectively. Because

\[
\tan \alpha + \tan \beta = \frac{L}{D} \tag{2}
\]

where \( D \) is the distance between the CCD camera and the light source, the z-component of displacement is then found to be

\[
W = \frac{gL}{D} N_z \tag{3}
\]

Normal viewing is preferred to avoid foreshortening and distortion of the image. With this arrangement, a 30° angle of illumination is used. The warpage measurement system consists of the environment oven, optical measurement system and positioning & mounting module as shown in Fig. 4. A quartz glass reference grating (100 lines/mm) is positioned directly above the PBGA package, and the distance between them is precisely adjusted to 5% less than the Talbot Distance, until a good
image is obtained.

Fig. 3. Schematic of shadow moiré.

Fig. 4. Warpage measurement system.
2.3 Button shear test

The button shear test is widely used to measure the interfacial adhesion strength between the molding compound and lead frame [21]. In this experiment, it was used to obtain the debonding load of underfill from FR-4 substrate with solder mask layer. Shear tests were carried out on a dynamic material testing system with a set of special testing jig. A constant loading rate of 0.1mm/s with a loading height of 2.75 mm was adopted. The loading height is the perpendicular distance from the bonded plane to the point on the button where the force was applied, as shown in Fig. 5.

![Fig. 5. The schematic of button shear test.](image)

3. Experimental Analysis

3.1 Analysis of variance (ANOVA)

Analysis of variance is the statistic method used to interpret experimental data. This method was developed by Sir Ronald Fisher in 1930s [22]. ANOVA is a decision tool for detecting any differences in average performance of groups of items tested.

In most experimental situations, the variation due to the mean has no practical value. The variation due to the mean does not affect the calculations for the error variation, and neither does it affect the calculations for the factor effects. In addition, the percent contribution is a function of the sums of squares for each significant item. The portion of the total variation observed in an experiment attributed to each significant factor and interaction is reflected in the percent contribution. In this paper, therefore, the methods of excluding the mean and the percent contribution were used in ANOVA to complete the calculations.

Three-way full factorial ANOVA entails three controlled factors, equations of degrees of freedom and sums of squares (variation) for total, individual factors, interaction of factors and error are shown in Table 1 [23].

There were 8 sets of ANOVA have been made for analyzing the main and interactive effects of temperature, relative humidity, solder mask thickness and absorption time on moisture weight gain and debonding force for specimens. The arrangements for the factors and levels are tabulated in Table 2.

Table 1. Analysis of variance

<table>
<thead>
<tr>
<th>Source</th>
<th>df</th>
<th>SS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Treatments</td>
<td>abc-1</td>
<td>(N \sum_{hij} (\bar{y}<em>{hije} - \bar{y}</em>{egee})^2)</td>
</tr>
</tbody>
</table>
### 3.2 Linear regression analysis

Regression is used to study relationships between measurable variables. Linear regression is used for a special class of relationships, namely, those that can be described by straight lines, or by generalizations of straight lines to many dimensions. Generally, regression analysis consists of many steps. To study a relationship between a number of variables, data are collected on each of a number of units or cases on these variables. In the regression models studied here, one variable takes on the special role of a response variable, while all of the others are viewed as predictors of the response. The model generally will also specify some of the characteristics of the failure to provide exact fit through hypothesized error terms. Then, the data are used to obtain estimates of the unknown parameters. The analysis to this point is called aggregate analysis, since the main purpose is to combine the data into aggregates and summarize the fit of a model to the data. In addition, the phase of a regression analysis is called case analysis, in which the data are used to examine the suitability and usefulness of the fitted model for the relationship studied. The results of case analysis may lead to modification of the original prescription for a fitted model, and cycling back to the aggregate analysis after modifying the data or assumptions.

### 4. Results and Discussions

The existence of defects in the corresponding interfaces can gradually degrade the interfacial adhesion when the package is exposed to the high temperature and humidity. Then, the effects of IC package with different solder mask thickness under different temperature and humidity environment conditions on the adhesion strength were studied. The size of experimental specimen is determined according to the standard of JESD22-A104-B. The control parameters are environmental temperature,
environmental humidity, soaking time and solder mask thickness. The interfacial adhesion strength was obtained by using a dynamic material testing system (MTS 810) with a set of special testing jig. By ANOVA and SPSS regression analysis, the relative equation of the interfacial adhesion strength to the humidity weight and humidity weight to the other factors can be determined.

As shown in Fig 6, with the change in storage condition of temperature, relative humidity, time and different solder mask thickness, every experiment does five times with the same condition and get the average shear force data.

First, the effects of the temperature and relative humidity on the warpage of PBGA package were studied by using shadow Moire method. The results reveal that the PBGA package has the absolute warpage with 12.3 μm through 168hrs and in 60℃/60%Rh condition as illustrated in Fig. 7. The results shown that the temperature, relative humidity and soaking time have significant effect on the moisture weight gain of package, thus we utilized the ANOVA and SPSS regression analysis to discuss the effect of each parameter.

![Fig. 6. Average debonding forces with respect to the solder mask thickness and preconditioning conditions (soaking time: 168h)](image-url)
4.1 The effects of temperature, relative humidity and soaking time on the moisture weight gain.

Temperature and relative humidity to investigate the variation of moisture weight gain and debonding force for test specimens. Meanwhile, ANOVA method was used to complete the analyses.

As can be seen in Table 3, when the solder mask thickness is a constant, the most significant factor for the moisture weight gain of adhesively bonded joint is the soaking time. In addition, as the solder mask thickness increases, the effects of the soaking time and relative humidity on the moisture weight gain become greater. On the contrary, the effect of temperature decreases gradually.

Table 3. Analysis of variance on the moisture weight gain and debonding force with the variation of solder mask thickness

<table>
<thead>
<tr>
<th>Solder mask thickness</th>
<th>20µm</th>
<th>40µm</th>
<th>60µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture weight gain</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Factor</td>
<td>Effect (%)</td>
<td>Effect (%)</td>
<td>Effect (%)</td>
</tr>
<tr>
<td>St</td>
<td>63.84</td>
<td>70.505</td>
<td>79.119</td>
</tr>
<tr>
<td>T</td>
<td>23.52</td>
<td>9.935</td>
<td>5.756</td>
</tr>
<tr>
<td>RH</td>
<td>5.37</td>
<td>9.77</td>
<td>10.765</td>
</tr>
<tr>
<td>Error</td>
<td>7.27</td>
<td>9.79</td>
<td>4.36</td>
</tr>
<tr>
<td>Debonding force</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>T</td>
<td>57.745</td>
<td>54.775</td>
<td>49.3</td>
</tr>
<tr>
<td>St</td>
<td>17.13</td>
<td>25.51</td>
<td>34.3</td>
</tr>
<tr>
<td>RH</td>
<td>15.345</td>
<td>11.15</td>
<td>7.24</td>
</tr>
<tr>
<td>Error</td>
<td>9.78</td>
<td>8.56</td>
<td>9.16</td>
</tr>
</tbody>
</table>

$S_t$: Soaking time

T : Temperature

RH : Relative humidity

The saturated moisture concentration and moisture diffusivity of the solder mask material is significantly higher than the
underfill and substrate material. And, moisture diffusivity of these materials exhibits Arrenhius behavior [1, 2]. The concentrated of moisture at the interface plays a crucial role to degrade the adhesion strength. Moisture mainly diffuses through the underfill and solder mask material into the underfill/solder mask and solder mask/substrate interfaces of the button shear test specimen.

Due to the test specimens were pre-baked at 125°C for 24 hours before preconditioning in an environmental chamber, the moisture desorption was nearly complete. Therefore, basing on the diffusion equation the moisture absorption rate at the start of preconditioning is greater comparatively. In addition, the thermal energy will accelerate the moisture diffusing, particularly at long times of exposing in high relative humidity environment. The effects of temperature on moisture weight gain become more significant than those of relative humidity.

4.2 The effects of temperature, relative humidity and solder mask thickness on the moisture weight gain.

During 24 hours, the percentage contribution of individual factors and interaction effects on the total variance of the moisture weight gain is shown in Table 4. It indicates that the effect of relative humidity solder mask thickness was the largest, main effects of factors relative humidity and temperature were approximately the same. Because of the solder mask was coated on whole surface of the substrate, the effect of its thickness on moisture weight gain is appreciable with its high moisture diffusivity.

According to the ANOVA, it can be found that the most significant factor for the moisture weight gain of adhesively bonded joint is still the solder mask thickness, shown as Table 4. However, temperature turns into the next significant factor instead of the relative humidity. In this case, the effect of the relative humidity on the moisture weight gains decreases with the increase of soaking time because the moisture absorption of test specimens is getting saturated.

With regard to the thickness issue, the moisture absorption of solder mask can be considered one dimensional for relatively thin layer [3]. That is, the moisture is considered to diffuse predominantly through the larger surfaces of the solder mask, and diffusion through the edges is considered negligible. As for the underfill, however, moisture diffusion through the edges may become significant.

| Table 4. Analysis of variance on the moisture weight gain and debonding force with the variation of soaking time |
|--------------------------------------------------|-------|-------|-------|-------|
| Soaking time | 24hr | 72hr | 120hr | 168hr |
| Factor | Effect (%) | Effect (%) | Effect (%) | Effect (%) |
| SMt | 72.76 | 71.28 | 68.06 | 68.03 |
| RH | 13.89 | 12.32 | 10.91 | 10.35 |
| T | 8.29 | 11.92 | 15.26 | 16.94 |
| Error | 5.06 | 4.48 | 5.77 | 4.68 |
| T | 45.74 | 47.81 | 52.41 | 60.17 |
| SMt | 33.64 | 31.04 | 28.67 | 20.85 |
| RH | 16.68 | 14.61 | 13.86 | 12.94 |
| Error | 3.94 | 6.54 | 5.06 | 6.04 |
4.3 The effects of temperature, relative humidity, solder mask thickness and soaking time on the debonding force.

To investigate how the factors of the solder mask thickness and soaking time influence the strength of the joints, the ANOVA analyses are employed in this experiment and the results are shown in Table 3 and 4, respectively.

In Table 4, it demonstrates that the most significant factor for the debonding force of adhesively bonded joint is temperature. Besides, the debonding force increases with the increase of the solder mask thickness, soaking time and relative humidity in order. Furthermore, as the solder mask thickness decreases, the difference between the effects of temperature and soaking time on the debonding force also increases. However, with regard to the factor of relative humidity, the change in its effect on the debonding force is slight with the varied solder mask thickness.

In Table 4, the ANOVA results indicate that the effect of temperature on the debonding force is the largest, followed by the solder mask thickness and relative humidity when the soaking time is 24 hours. In addition, the effect of temperature on the debonding force enlarges as the soaking time increases. However, the effects of the solder mask thickness and relative humidity on the debonding force decrease with the soaking time increases.

The influence of the variety of parameters on the adhesion strength is a problem of great complexity. For a bonded component with polymeric materials, it is vulnerable to absorb moisture to some extent and their mechanical behaviors will follow to change. The absorbed moisture may decrease its complex dynamic modulus and decrease its glass transition temperature by disrupting the bonding between the molecules of the adhesive and/or hydrolyzing the molecules of the adhesive to decrease their molecular weight.

In addition, segmental motions of the polymer molecules increase with the increase of temperature. This permits diffusing moisture to penetrate more readily. Furthermore, the oxides and hydroxides formed with weaker mechanical properties may also increase while exposure to higher temperature. During solder reflow, the mismatches in material coefficients of thermal expansion will result in high thermal stresses because of subjecting the high temperature. The combination of thermal stresses with hygro stresses will apparently deteriorate the adhesion strength to the already weakened material interfaces by lowering the free energy barrier of interfacial molecular bonds.

4.4 Multiple-regression Analysis

By using analysis of variance to analyze the moisture weight gain and all factors (relative humidity, temperature, solder mask thickness, and soaking time) that we found there is linear relation existent between them, the result shows all factors affect the moisture weight gain significantly, so we try to find the relative equation between moisture weight gain and all factors that we use the SPSS 11.5 to apply multiple regression analysis (through the SPSS analysis the equations can be expressed as dimensionless equation), then the outcome is an linear equation between the moisture weight gain and all factors as demonstrate in Eq. 1, also we found the relative equation between the moisture weight gain and debonding force as demonstrate in Eqn.2,
\[
\frac{W}{W_0} = 1.064(RH) - 0.640(T) + 0.570(SM_t) + 0.165(S_t) - 0.164 \quad \cdots \cdots (1)
\]

where RH is the Relative humidity, T is the Temperature, SM_t is the Solder mask thickness, S_t is the Soaking time

\[
\frac{F}{F_0} = -13.62 \times \frac{W}{W_0} + 14.63 \quad \cdots \cdots (2)
\]

where

\[
\frac{F}{F_0} : \text{Normalized debonding force}
\]

\[
\frac{W}{W_0} : \text{Normalized moisture weight gain}
\]

Three preconditioning conditions for the adhesively bonded joints were employed to determine the compatibility of the Eq. (1) and Eq. (2), as shown in Table 5. Five specimens were used in each test. The experimental results of the average of moisture weight gains and debonding forces with corresponding storage conditions are put into Eq. (1) and Eq. (2), respectively. Compared the values of W/W_0 and F/F_0 from the results of this experiment with those calculated from the Eq. (1) and Eq. (2), it can be found that the maximum error is 10.78%, as shown in Table 6. Thus, these dimensionless equations can offer easily to estimate the influences of storage conditions on the moisture weight gains and the degradation of interfacial strength for the adhesively bonded joints.

| Table 5. Three preconditioning conditions for the adhesively bonded joints |
|-----------------|---|---|---|
|                | A  | B  | C  |
| Relative humidity | 45% | 45% | 45% |
| Temperature     | 45℃ | 45℃ | 45℃ |
| Solder mask thickness | 30μm | 50μm | 50μm |
| Soaking time    | 42 hr | 84 hr | 126 hr |

| Table 6. Results of the regression and experiments |
|-----------------|---|---|---|---|---|---|
|                | A  | B  | C  | A  | B  | C  |
| Sample Condition | W_0 (g) | 4.1493 | 4.1854 | 4.1854 | F_0 (N) | 369 | 466.8 | 466.8 |
|                 | W (g) | 4.1781 | 4.2840 | 4.3113 | F (N) | 337.8 | 321.6 | 280.2 | 312.9 | 344.9 | 314.0 |
| Error (%)       | 7.96 | 6.75 | 10.78 |
5. Conclusions

This study is using the experiment data and the interaction influence of ANOVA's and regression analysis factors, the conclusions as following:

1. In initial condition by changing the solder mask thickness, the shearing force is higher by the solder mask thickness, in the same situation of temperature and moisture and during the storage time of 168 hours, the shearing force is lower by the solder mask thickness, the wet gain is increase by the thickness but the shearing force is decrease by the thickness.

2. The effects of temperature, relative humidity and solder mask thickness on the moisture weight gain are dominate by solder mask thickness.

3. The temperature factor dominate the debonding force, the results influence the high end soaking time levels are higher than low end soaking time levels, the influence effect is inverse by the solder mask thickness.

4. The effects of temperature, relative humidity and solder mask thickness on the debonding force, the temperature factor is dominate the results, during the storage time is 24 hour, the results of parameters of temperature and solder mask thickness are almost the same, however, during to 168 hours, the results have obvious different.

5. This experiment results are using interaction of ANOVA's parameters, the maximum error is 9.78% and the minimum is 3.94%.

6. By using analysis of variance to analyze the moisture weight gain and all factors that we found there is linear relation existent between them, so we can apply regression analysis to find the relative equation between the moisture weight gain and all factors, also the relative equation between the moisture weight gain and debonding force can be found.

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7. References


