Adhesion Evaluation of Adhesiveless Metal/Polyimide Substrate for MCM and High Density Packaging

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Abstract

Directly metallized polyimide films are being widely used as advanced high-density packaging substrates. Adhesion of metal to polyimide is a key performance requirement of the film. Excellent initial adhesion and good retention after severe process steps and reliability stresses are required. In this paper, the adhesion performances of films with different types of polyimide and tie layer from multiple vendors have been evaluated. The interface failure mechanisms are also examined by SEM micrograph and XPS survey scan. Peel strength and its retention behavior are characterized in term of tie layer materials, environmental stresses, and surface analysis results. The objective of this work is to select the proper base material for high-density flexible multilayer substrates, which technology is under developing by FCPT.

Key words:
Metal/Polyimide Adhesion, Reliability, Flex Substrate, and High Density Packaging.

1. Introduction

It is well recognized that polyimide flex substrate is able to achieve the highest wiring density of any printed circuit and has favorable mechanical and electrical properties. New materials and processes have enabled the construction of ultra-thin and high-density adhesiveless polyimide flex for fine-line geometry that looks especially promising for IC packaging applications. The absence of adhesive means that the distinctive characteristics of polyimide can be exploited to the full. Metallization of polyimide film by direct sputter deposition followed by electroplating has been most commonly used for extremely high-density, fine-line applications such as BGA, CSP, and MCM since the metallization has smooth surface and thin thickness for promoting fine-line etching.

Adhesion of metal to polyimide is a key performance requirement of polyimide flex films. Although good initial adhesion is essential, adhesion retention after thermal and humidity stresses may be more important since any of these stresses have the potential of reducing adhesion to cause process yield losses and product reliability issues. The common technique to improve metal/polyimide adhesion and its reliability is to deposit a thin tie metal layer (such as nickel/copper alloy and chromium) first on plasma modified polyimide film prior to the copper deposition.

Adhesion degradation after thermal and humidity stresses can occur for a number of reasons. Copper diffusion can promote adhesion loss at elevated temperatures and can be inhibited by coating a barrier layer of metal – tie layer. Oxygen diffusion through polyimide film to the metal/polyimide interface plays a critical role in promoting degradation too. Adhesion of Cr/polyimide interface is degraded significantly upon exposure to high temperature and humidity environment due to the hydrolysis of polyimide. Catastrophic adhesion loss has been linked to moisture induced oxidation of chromium interfaces based on studies using radioactively tagged water.

In this paper, the researchers have evaluated the adhesion of five types of polyimide films with different types of tie layer from multiple vendors. The interface failure mechanisms are also examined by SEM micrograph and XPS survey scan. Peel strength and its retention behavior are characterized in terms of tie layer materials, environmental stresses, and surface analysis results. The objective of this work is to select the proper base material for high-density flexible multilayer substrates, which technology is under developing by FCPT.
different types of base films that have different Cu/tie metal/polyimide configurations from multiple material vendors. The objective is to compare adhesion between metal and polyimide after flex circuitry fabrication and the adhesion retention after environment stresses. To accomplish this objective, an additive flex circuitry process was employed to define 20µm thick copper strips on the films. The 90° peel strength test was used to determine the initial adhesion and its retention after three common IC packaging reliability tests; High Temperature Storage, Thermal Shock, and Pressure-Cook Test. Analyses of fracture surfaces exposed in peel test were done by Scanning Electron Microscope and X-ray Photoelectron Spectroscopy.

2. Experiments

In this study, a total of five different types metal/polyimide base films provided by multiple vendors have been evaluated. All the base films have seed Cu/tie metal/polyimide configurations as shown in Table 1.

Table 1. Polyimide flex films.

<table>
<thead>
<tr>
<th>Vender</th>
<th>Polyimide</th>
<th>Tie Metal</th>
<th>Seed Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Kapton® 50µm</td>
<td>Ni/Cu, 180Å</td>
<td>Cu 2000Å</td>
</tr>
<tr>
<td>B</td>
<td>Kapton® 50µm</td>
<td>Cr 200Å</td>
<td>Cu 2000Å</td>
</tr>
<tr>
<td>B</td>
<td>Apical® 50µm</td>
<td>Cr 200Å</td>
<td>Cu 2000Å</td>
</tr>
<tr>
<td>C</td>
<td>Upilex®-A 50µm</td>
<td>Cr 200Å</td>
<td>Cu 2000Å</td>
</tr>
<tr>
<td>C</td>
<td>Upilex®-B 50µm</td>
<td>Cr 200Å</td>
<td>Cu 2000Å</td>
</tr>
</tbody>
</table>

An additive flex circuitry process was employed to build up copper peeling strips on the base films. The film was annealed first at 150°C for 2 hours under vacuum. Then, photoresist was applied to define the peeling strip patterns in width of 3mm and 10mm, followed by electrolytic plating to build up copper thickness to 20±1µm. Finally, the photoresist was striped and the seed Cu and tie metal were etched to have the copper strips.

The adhesion was determined by the 90° peel test using Instron-4502 tensile tester and Series IX V5.24 testing software. All peeling was measured at a rate of 50mm/min cross-head speed with 100 N load cell. The peel length was 20mm for each sample with a data acquisition rate of 5 per second. An average of the collected data was calculated as the peel strength of sample. Both 3mm and 10mm peel strips were used and mounted on ceramic backing plates. An average peel strength at each test points was determined from three samples.

Three reliability stress tests were selected to evaluate adhesion stability based on the possible adhesion failure mode discussed above and common industry requirement for IC packaging. These tests were High Temperature Storage (HTS), Thermal Shock (TS) with preconditioning, and Pressure Cook Test (PCT). Table 2 provides an overview of these stress tests and conditions.

X-ray Photoelectron Spectroscopy (XPS) and Scanning Electron Microscope (SEM) were used to analyze the two fracture surfaces exposed in peel test. XPS spectra were collected on a Physical Electronics Model Quantum 2000 Scanning ESCA System using rastered monochromatic Al Kα x-ray beam. ESCA survey spectra (0 eV to 1100 eV) were acquired with an energy resolution of 1.0 eV per channel for each sample in order to determine the elements present and their approximate concentrations. AMRAY 1850 Field Emission SEM was used to view the topography of the surfaces with 1000X-10000X magnification.

3. Results

3.1. Initial Adhesion

The initial peel strengths of all different films with 20µm copper are shown in Figure 1-A. Three of five films have good initial adhesion about 100g/mm. The films are Cr/Kapton®, Ni-Cu/Kapton®, and Cr/Upilex®-B. The Cr/Apical® shows fair initial adhesion of about 85g/mm. The worst one is Cr/Upilex®-A which initial adhesion is just over 50g/mm.

Figure 1. Initial peel strengths and their retention after environmental stresses.
Both SEM and XPS were used to characterize the two fracture surfaces - the metal side and polyimide side exposed in peel test. SEM micrographs reveal highly uneven metal side and polyimide side surfaces for all films except Upilex®-A which shows very smooth fracture surfaces. Figures 2-A and 2-B show two fracture surfaces of Ni-Cu/Kapton® as an example. On the surface of metal side, the raised areas indicate that a thin layer of polyimide is carried over with metal strip during peel test. On the surface of polyimide side, the depressed areas indicate locations where polyimide was transferred to the metal surface. Elemental compositions of the surface base on XPS survey scans confirm that there is a thin layer (approximately 100 Å) polyimide on the metal surface after peel test. In Table 3, the atomic concentration shows evidence of polyimide on both fracture surfaces and no evidence of metal on polyimide surface. Hence, the peel fracture is cohesive in the polyimide just below a thin plasma modified surface region. For the Upilex®-A, XPS analysis shows on evidence of polyimide on the metal surface after peel faces of Ni-Cu/Kapton® as an example. SEM micrographs reveal highly uneven metal side and polyimide side surfaces for all films except Upilex®-A which shows very smooth fracture surfaces. Figures 2-A and 2-B show two fracture surfaces of Ni-Cu/Kapton® as an example. On the surface of metal side, the raised areas indicate that a thin layer of polyimide is carried over with metal strip during peel test. On the surface of polyimide side, the depressed areas indicate locations where polyimide was transferred to the metal surface. Elemental compositions of the surface base on XPS survey scans confirm that there is a thin layer (approximately 100 Å) polyimide on the metal surface after peel test. In Table 3, the atomic concentration shows evidence of nitrogen on both fracture surfaces and no evidence of metal on polyimide surface. Hence, the peel fracture is cohesive in the polyimide just below a thin plasma modified surface region. For the Upilex®-A, XPS analysis shows on evidence of polyimide on the metal surface so that the peel fracture is cohesive in the metal/polyimide interface.

Table 3. Surface atomic concentration of the fracture surfaces of Ni-Cu/Kapton® film.

<table>
<thead>
<tr>
<th>Description Location</th>
<th>Cu</th>
<th>Cr</th>
<th>Si</th>
<th>C</th>
<th>N</th>
<th>O</th>
<th>Others</th>
</tr>
</thead>
<tbody>
<tr>
<td>Peeled at initial 0 hours</td>
<td>Cu side</td>
<td>0.3</td>
<td>--</td>
<td>0.3</td>
<td>94.2</td>
<td>5.8</td>
<td>0.9</td>
</tr>
<tr>
<td></td>
<td>PS side</td>
<td>--</td>
<td>10.5</td>
<td>0.6</td>
<td>37.5</td>
<td>1.3</td>
<td>28.7</td>
</tr>
<tr>
<td>Peeled at HTS 1000 hours</td>
<td>Cu side</td>
<td>0.3</td>
<td>12.0</td>
<td>3.8</td>
<td>40.1</td>
<td>0.9</td>
<td>12.6</td>
</tr>
<tr>
<td></td>
<td>PS side</td>
<td>0.3</td>
<td>15.1</td>
<td>3.8</td>
<td>40.1</td>
<td>0.9</td>
<td>12.6</td>
</tr>
</tbody>
</table>

3.2. Adhesion Retention after HTS test

Figure 1-B shows peel strength retention of films after exposed to high temperature of 150°C up to 1000 hours. Cr/Upilex®-A gives us the best adhesion stability compared to others. It remains about 70% adhesion after 1000 hours test. The retention of Cr/Kapton® and Cr/Upilex®-B are fairly OK to have about 50% of their original values. Ni-Cu/Kapton® and Cr/Apical® shows significant adhesion degradation to 30% and 10% of initial. All films exhibit a sharp initial adhesion drop followed by a slowing slope except for Cr/Upilex®-A which keeps pretty consistent drop slope.

The fracture surfaces of 1000 hours samples were analyzed by SEM and XPS. The researchers found both cohesive and adhesive failure modes depended on the film types. The result is listed as follows:

- Ni-Cu/Kapton® Adhesive
- Cr/Kapton® Cohesive
- Cr/Apical® Adhesive
- Cr/Upilex®-A Adhesive
- Cr/Upilex®-B Cohesive

SEM micrographs of adhesive failed interfaces are given in Figures 2-C and 2-D that show very smooth fracture surfaces on both metal and polyimide side of Ni-Cu/Kapton® peeled sample after 1000 hours HTS test. In Table 3, the atomic concentration shows significant copper at both metal and polyimide sides that confirms the SEM result.

3.3. Adhesion Retention after PCT test

Figure 1-C shows the adhesion degradation curves of films after exposed to severe temperature/humidity condition – 121°C/100%RH under 15psi. One can see that Cr/Upilex®-A has the slowest drop slope and remains 65% of its initial after 168 hours, followed by Cr/Kapton® and Cr/Upilex®-B which have about 55% adhesion retention after 168 hours. The adhesion of Ni-Cu/Kapton® degrades significantly to 20% at the end. Cr/Apical® performs the worst and has a sharp drop to 10% just after 48 hours.

SEM and XPS surface analysis reveal that the fracture surfaces could be either metal/polyimide interface (adhesive) or polyimide itself (cohesive) for different types of film. They are as follows:

- Ni-Cu/Kapton® Adhesive
- Cr/Kapton® Cohesive
- Cr/Apical® Adhesive
- Cr/Upilex®-A Adhesive
- Cr/Upilex®-B Cohesive

3.4. Adhesion Retention after TS test

The adhesion retention of films after exposed to liquid to liquid temperature shock with level 3 precondition are given in Figure 1-D. After preconditioning and 500 cycles of thermal shock, Cr/Upilex®-A shows highest adhesion retention about 93%, followed by Cr/Upilex®-B that has very good retention of 85% too. Cr/Kapton® and Ni-Cu/Kapton® still have good retention of 66%. Cr/Apical® film has worse, but fair adhesion retention of 47%.

The results of SEM and XPS surface analysis for the fracture surfaces are as follows:

- Ni-Cu/Kapton® Mixed adhesive/cohesive
- Cr/Kapton® Cohesive
- Cr/Apical® Adhesive
- Cr/Upilex®-A Adhesive
- Cr/Upilex®-B Cohesive
4. Summary and Conclusion

In this work, the adhesion behavior of five types of polyimide flex substrates was evaluated in terms of initial peel strength after an additive process and peel strength retention after severe environment stress tests. The base substrates were 50µm polyimides with 0.2µm seed Cu/tie metal layer. Subsequent additive process was done to build up the copper peel strip to 20µm in thickness. The 90° peel test was employed to measure the peel strength. SEM and XPS were used to analyze the fracture surface for determining the interface failure mode.

Table 4 provides the over rank of adhesion performance for all evaluated films and the failure mode of peeling interfaces. Both Cr/Kapton® and Cr/Upilex®-B have good initial adhesions and good stability after stresses. Cr/Upilex®-A has very good adhesion retention, but its initial adhesion is quite low. Ni-Cu/Kapton® and Cr/Apical® have huge adhesion degradation after stresses, especially after the high temperature and humidity PCT test, even their initial adhesion are good or fair. SEM and XPS analysis of fracture surfaces have found both adhesive and cohesive failure mechanisms. When the film has significant adhesion loss after stresses, the failure mode would change most likely from cohesive at initial to adhesive after stress.

Table 4. Adhesion performance and failure mode of evaluated metal/polyimide films.

<table>
<thead>
<tr>
<th>Vendor Polyimide</th>
<th>Tie Metal</th>
<th>Overall Rank of Adhesion Performance</th>
<th>Failure mode of the peeling interfaces</th>
</tr>
</thead>
<tbody>
<tr>
<td>A Kapton®</td>
<td>NoCu</td>
<td>Good</td>
<td>Good</td>
</tr>
<tr>
<td>B Kapton®</td>
<td>Cu</td>
<td>Excellent</td>
<td>Excellent</td>
</tr>
<tr>
<td>B Apical®</td>
<td>Cu</td>
<td>Excellent</td>
<td>Excellent</td>
</tr>
<tr>
<td>C Upilex®-A</td>
<td>Cr</td>
<td>Excellent</td>
<td>Excellent</td>
</tr>
<tr>
<td>C Upilex®-B</td>
<td>Cr</td>
<td>Excellent</td>
<td>Excellent</td>
</tr>
</tbody>
</table>

This work has provided the information required for properly selection of adhesiveless metal/polyimide materials depending on the specific application.

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References


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