

## Application News

AGX™-V2 Precision Universal Testing Machine

### Peel Test of Plating Film on Resin Under Varied Temperature Environments

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#### User Benefits

- ◆ The adhesion of plating under temperature environments from -60 °C to 250 °C can be measured by using the jig and thermostatic chamber introduced in this article.
- ◆ It is possible to measure the adhesion of the plating as described in JIS H 8630, JIS C 6481.

#### Introduction

Parts produced by applying platings to the surface of resins are generally used in applications ranging from decorative parts in the automotive industry to electrical devices and the electrical and electronic/semiconductor fields. Since technologies for applying platings to hard-to-plate resins have also been required for the printed boards of automotive components and advanced electronic equipment in recent years, a wide-range of research and development projects are underway on topics including new resin materials, plating chemicals, and plating production processes.

To ensure that plating films fully demonstrate their functions, films must secure adhesion without peeling under their intended use environments. At present, the main methods for evaluating the adhesion of plating are the thermal cycle test and the heat shock test, but these cannot be considered quantitative evaluation methods. This article introduces an example in which a peel test was conducted using a universal testing machine, and the adhesion of the plating under various temperature environments was quantified.

#### Test Sample

Fig. 1 shows the dimensions and cross section of the sample used in the measurement. Polybutylene terephthalate (PBT), which is a hard-to-plate resin, was used as the substrate. As a method for plating hard-to-plate resins, first, a thin plating seed layer is formed on the resin substrate as a conductive layer, and a plating of the specified thickness is then applied by an electroplating method. In this experiment, films with a thickness of approximately 0.6 μm were formed using two different methods of applying the plating seed layer (sputtering, which is a physical method, and electroless plating using a chemical reaction). After film-forming, a copper plating film with a target thickness of 25 μm was formed on the respective samples by the electroplating method.

In the measurements, the plating layer was scribed with a cutter on a 10 mm part in the center of the 30 mm wide sample as the peel area.

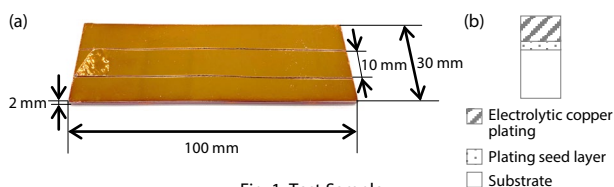


Fig. 1 Test Sample  
(a) Dimensions, (b) Cross section

Table 1 Sample Information

|                                 |                                                |
|---------------------------------|------------------------------------------------|
| Sample dimensions               | : Width 30 mm × length 100 mm × thickness 2 mm |
| Substrate                       | : Polybutylene terephthalate (PBT)             |
| Plating                         | : Copper                                       |
| Seed layer film-forming methods | : Sputtering<br>Electroless plating            |

#### Instruments and Test Conditions

The peel test was carried out using a universal testing machine and a thermostatic chamber. Fig. 2 shows views of the test, and Table 2 shows the instrument configuration.

The peel length is 60 mm or more. The adhesion of the plating was calculated from the average test force of the data taken from the length between the 10 mm and 60 mm after the start of peeling. (Fig. 3). The test width and speed of the test conditions were set referring to JIS H 8630, JIS C 6481, and ASTM B 533-85. Table 3 shows the test conditions.

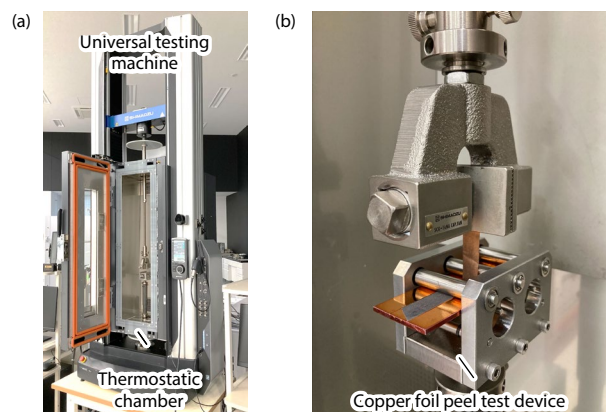


Fig. 2 Instruments

- (a) Universal testing machine, Thermostatic chamber  
(b) Copper foil peel test device

Table 2 Instrument Configuration

|                      |                                                            |
|----------------------|------------------------------------------------------------|
| Testing machine      | : AGX-50NV2D                                               |
| Load cell            | : 50 N                                                     |
| Jig                  | : Copper foil peel test device                             |
| Thermostatic chamber | : Refrigerator thermostatic chamber, air-cooled type TCR2A |
| Software             | : TRAPEZIUM X-V                                            |

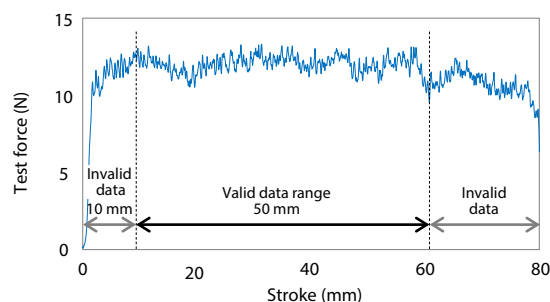


Fig. 3 Range of Data Used in Calculation of Adhesion

Table 3 Test Conditions

|                             |                                                             |
|-----------------------------|-------------------------------------------------------------|
| Test speed                  | : 25 mm/min                                                 |
| Set atmosphere temperatures | : -50 °C, -25 °C, 0 °C, 25 °C, 50 °C, 75 °C, 100 °C, 125 °C |
| Number of tests             | : N = 5 (at each set atmosphere temperature)                |
| Valid data range            | : 10 mm to 60 mm from start of peeling                      |
| Peel width                  | : 10 mm                                                     |

## ■ Test Results

Prior to the peel test, the relationship between the film thickness and adhesion of the plating was investigated. The film thickness in the central part of the samples was measured nondestructively by using the X-ray fluorescence method. Although the target film thickness of the plating in film-forming for this test was 25  $\mu\text{m}$ , the measured values of the film thickness spanned a range from 15  $\mu\text{m}$  to 30  $\mu\text{m}$ . As the result of a room-temperature peel test of those specimens, Fig. 4 shows an adhesion-plating film thickness graph. The correlation coefficient of the film thickness and adhesion was 0.86, indicating that adhesion has a strong correlation with the film thickness of the plating. Thus, it can be said that measuring the plating film thickness and evaluating adhesion using specimens with approximately the same film thickness are important in quantitative evaluations of adhesion.

To evaluate adhesion when the temperature environment was changed, a peel test was carried out using samples with measured plating film thicknesses from 20  $\mu\text{m}$  to 25  $\mu\text{m}$ . As an example of the results, Fig. 5 shows the test force-stroke graphs of samples prepared by the two different film deposition methods. Fig. 5 (a) shows the test results for the specimens when film-forming was done by the sputtering method, and (b) shows the results for the specimens prepared by electroless plating.

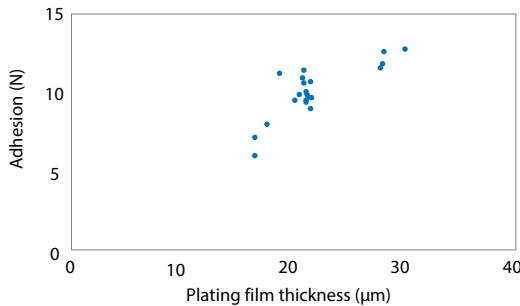


Fig. 4 Adhesion-Plating Film Thickness Relationship (Sputtering)

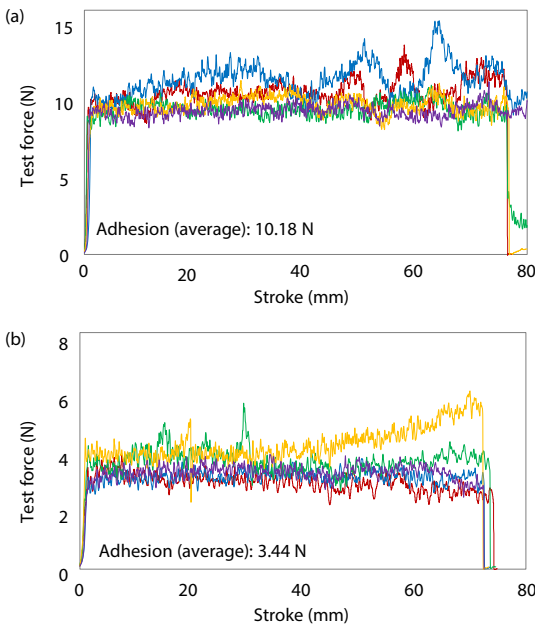


Fig. 5 Test Force-Stroke Graphs (Room Temperature)  
(a) Sputtering, (b) Electroless plating

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Table 4 shows the test results (average of  $N = 5$  tests) for the various temperatures, and Fig. 6 shows the relationship between the plating adhesion and thermostatic chamber temperature for each seed layer. When the seed layer was applied by sputtering, the adhesion of the plating showed a decreasing tendency as the temperature increased, and with electroless plating, there was almost no change from  $-50\text{ }^{\circ}\text{C}$  to  $75\text{ }^{\circ}\text{C}$ , and a decreasing tendency was observed at  $100\text{ }^{\circ}\text{C}$  and higher.

Table 4 Test Results (Average Values)

| Temp.<br>( $^{\circ}\text{C}$ ) | (a) Sputtering  |                                                                  | (b) Electroless plating |                                                                  |
|---------------------------------|-----------------|------------------------------------------------------------------|-------------------------|------------------------------------------------------------------|
|                                 | Adhesion<br>(N) | Difference<br>(vs. $25\text{ }^{\circ}\text{C}$ baseline)<br>(%) | Adhesion<br>(N)         | Difference<br>(vs. $25\text{ }^{\circ}\text{C}$ baseline)<br>(%) |
| -50                             | 12.33           | 21.10                                                            | 4.25                    | 23.63                                                            |
| -25                             | 11.19           | 9.85                                                             | 4.07                    | 18.27                                                            |
| 0                               | 10.82           | 6.21                                                             | 3.68                    | 7.00                                                             |
| 25                              | 10.18           | -                                                                | 3.44                    | -                                                                |
| 50                              | 10.23           | 0.43                                                             | 4.45                    | 29.43                                                            |
| 75                              | 9.57            | -6.08                                                            | 4.31                    | 25.39                                                            |
| 100                             | 9.35            | -8.19                                                            | 3.19                    | -7.32                                                            |
| 125                             | 8.99            | -11.71                                                           | 2.39                    | -30.49                                                           |

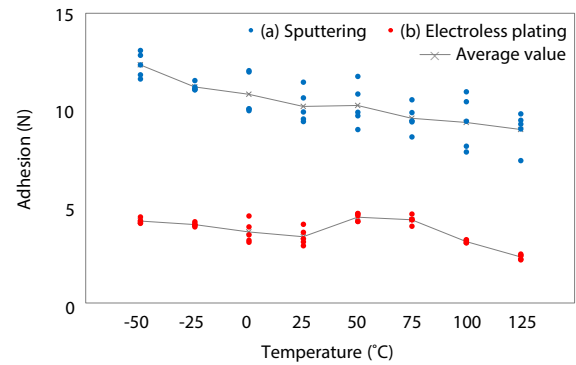


Fig. 6 Adhesion-Temperature Relationship

## ■ Conclusion

A peel test of specimens prepared by applying copper platings to a hard-to-plate resin was conducted in a thermostatic chamber using an AGX-V2 precision universal testing machine. It was found that the film thickness of the plating affected plating adhesion, and adhesion showed temperature dependence, tending to decrease at high temperatures exceeding  $100\text{ }^{\circ}\text{C}$ .

Evaluation of adhesion under a wide range of temperature environments, assuming different use environments, is an effective test method for various types of development, including the development of film-forming methods for plating, resin materials, plating chemicals, and plating production process.

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