A New Method for the Measurement of Internal Stress in Coatings

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Introduction

Internal stresses develop in organic coatings during the cooling process after thermosetting or curing. Internal stresses in thermosetting coatings is thermal internal stress as a consequence of the difference in the linear expansion coefficients of the coating and the substrate. Internal stresses have been thought for a long time to influence the characteristics of coatings, such as adhesive strength or the susceptibility to form cracks. Internal stress in organic coatings has been measured by the bending beam technique (BBT). In the BBT method, internal stresses are calculated using the elastic constants of coatings and substrate. The values obtained include essentially the error due to the method of calculation and the preparation of the sample. Furthermore, internal stresses obtained by BBT are the values created by the bending operation. In this investigation, a new method for measuring internal stress is proposed, called the FSB (film stretch back) method, based on the direct measurement of stress in detached film. In this method internal stresses and internal strains are identified directly and simultaneously. Using this method, on an thermosetting epoxy-phenolic coatings system, internal stress and related physical properties are discussed.

Experimental

Preparation of the sample

The epoxy-phenolic coatings used were Epikote 1009 containing different amounts of Bisphenol A type resole. These coatings were applied to tinplate and stoved under the conditions that are shown in Table 1. In order to obtain a uniform film, the method of spin coating was used. The thickness of the film was measured by a mechanical thickness meter using detached films.

Table 1: Phenolic contents and coating conditions of test materials

<table>
<thead>
<tr>
<th>Condition</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phenolic content (%)</td>
<td>5</td>
<td>15</td>
<td>30</td>
<td>50</td>
<td>70</td>
</tr>
<tr>
<td>Thermosetting temp. (°C)</td>
<td>200</td>
<td>200</td>
<td>185, 200, 215</td>
<td>200</td>
<td>185, 200, 215</td>
</tr>
<tr>
<td>Thermosetting time (min)</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Thickness of coatings (μm)</td>
<td>15</td>
<td>15</td>
<td>8.3, 15, 24.5, 34</td>
<td>15</td>
<td>15</td>
</tr>
</tbody>
</table>

Procedure of FSB method

The procedure for measuring internal stress by the FSB method was as follows and is illustrated schematically in Figure 1(a)–(e) and 1(f).

Figure 1(a)–1(e): Schematic illustration of the procedure for measuring internal stress by means of the FSB method
Figure 1(f): Measure the stress-strain curve

A schematic illustration of the stress-strain curve obtained by the tensile test is shown in Figure 1(f). Internal stress and internal strain correspond respectively to load and strain at break point in the stress-strain curve. Because the initial length of coating films which were subjected to tensile test was 100 mm, internal strain is equal to \( \varepsilon \). Internal stress \( P \) is calculated using the following formula.

\[
P = 9.80665 \times 10^{-6} \cdot P_{ob} / (W \cdot d) \quad \text{[MPa]}
\]

where \( P_{ob} \) is load [kg] at break point, \( W \) is width [cm] of specimen, \( d \) is thickness [cm] of the film.

Measurement of adhesion strength

Adhesive strength of coatings to tinplate was measured by 90° peeling test as shown in Figure 2.

Physical properties obtained by the FSB method

In this method, internal stress and internal strain are measured directly and simultaneously. Because the region before break point corresponds to the region of elasticity, the elastic modulus \( E \) of the coatings is obtained by the following equation.

\[
E = P / (\varepsilon / 100 - \varepsilon) \quad \text{Equation 1}
\]

In the case of thermosetting coatings, above the \( T_g \) of coatings there is no stress present because in the rubber-like state the relaxation processes counteract any stresses that develop. On cooling to a temperature below the \( T_g \), the stresses increase because, in the glassy state, coatings contract at a rate greater than that of the substrate. The internal stress can be calculated from Equation 2.

\[
P = E (\alpha_c - \alpha_m) (T_g - T_m) \quad \text{Equation 2}
\]

where \( \alpha_c \) and \( \alpha_m \) are the linear expansion coefficients of the coating and substrate metal, respectively, \( T_g \) is temperature at the measurement. The term \( (\alpha_c - \alpha_m) (T_g - T_m) \) in Equation 2 relates to the shrinkage of coatings. Then \( \varepsilon \) is expressed as follows.

\[
\varepsilon = 100 (\alpha_c - \alpha_m) (T_g - T_m) \quad \text{Equation 3}
\]

Therefore the linear expansion coefficient of the coating \( \alpha_c \) is calculated from Equation 4.

\[
\alpha_c = \alpha_{md} + 10^4 \cdot \varepsilon (T_g - T_m) \quad \text{Equation 4}
\]