INFLUENCE OF ZINC BORATE ON FIRE PROTECTION AND THERMAL DEGRADATION OF INTUMESCENT COATING CONTAINING A NOVEL CAGED BICYCLIC PHOSPHATE

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Fire protection and thermal degradation of intumescent epoxy coatings with a novel caged bicyclic phosphate (Trimer) [1], APP and zinc borate (ZB) were investigated. The experimental results as follows: compared to the coatings with ZB, No.1 coating shown the best initial fire protection property up to 300s due to the earliest expansion (Fig 1, Fig 2 and Table 1), however, at a longer time a plateau was achieved for No.2 and No.3 coatings and their final fire protection time was increased about 100% respectively when compared with that of No.1 coating. Compared to calculated TGA curve of No.3 coating in N\textsubscript{2}, the experimental one changed slightly (Fig 3 (a) and Table 1), the experimental residue at 700°C was increased by 1.5% on the base of calculated one. Moreover, the difference was obviously enlarged in air (Fig 3 (b) and Table 1), particular at temperature higher than 550°C, significant improvement was observed in thermo-oxidative stability of coating with the addition of 5%ZB. The final residue of No.3 coating was 38.2% at 700°C compared to 27.0% for No.1 coating and the beneficial effect of ZB was clearly demonstrated as well. It is of interest to notice that the degradation curve of Trimer/APP abviously changed from 480°C by incorporating ZB and the final residue increased from 14.6% to 47.8% (Fig 4). The $^{31}$P solid-state NMR spectrum (Fig 4) shown a high peak at -30ppm, which is attributed to B-O-P bonds [2]. This result further confirmed that the interaction taken place between Trimer/APP and ZB which lead to the formation of borophosphate. Above mentioned results clearly indicated that an appropriate ZB may yield synergistic effect on improving the thermal stability of coating with Trimer/APP at high temperature, which could contribute to enhance the fire protection property of coating.
Fig 1. Fire protection curves of coatings.  Fig 2. Image of (a) No.1 and (b) No.3 coatings at 30s in the fire protection test.

Fig 3. TGA curves of No.1 and No.3 coatings in (a) N₂ and (b) air.

Fig 4. TGA curves of Trimer/APP, ZB and Trimer/APP/ZB in air.

Fig 5. $^{31}$P solid-state NMR of Trimer/APP/ZB treated at 600°C in air.

Table 1. Fire protection and TGA data of coatings

<table>
<thead>
<tr>
<th>Samples</th>
<th>Fire protection time (s)</th>
<th>N₂, 10°C/min</th>
<th>Air, 10°C/min</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>250°C</td>
<td>300°C</td>
<td>350°C</td>
</tr>
<tr>
<td>No.1</td>
<td>80</td>
<td>200</td>
<td>530</td>
</tr>
<tr>
<td>No.3</td>
<td>70</td>
<td>121</td>
<td>1030</td>
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Reference