BLOWING-OUT EFFECT OF EPOXY COMPOSITES FLAME-RETARDED BY DOPO-POSS AND ITS CORRELATION WITH AMIDE CURING AGENTS

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An interesting phenomenon, called the "blowing-out effect", has been observed in flame retarded epoxy resins loaded with a novel polyhedral oligomeric silsesquioxane containing 9,10-dihydro-9-oxa-10-phosphaphenanthrene-10-oxide (DOPO-POSS). Model of the blowing-out effect is shown in Fig. 1 [1,2]. In order to further understand the reasons behind and the factors that influence the blowing-out effect, we have investigated the epoxy resins (diglycidyl ether of biphenol A: DEGBA) cured by both oligomeric polyamide 650 (PA650) and 4,4′-diaminodiphenylsulphone (DDS), respectively, with DOPO-POSS as a flame retardant. The epoxy composites with DOPO-POSS showed different flame retardant properties depending on the amides used. The results of UL-94 tests show that the DEGBA/DDS with DOPO-POSS exhibits a blowing-out effect through vigorous emission of pyrolytic gases, but the DEGBA/PA650 does not (Fig. 2). Moreover, only 2.5 wt. % DOPO-POSS imparts to the epoxy resin DEGBA/DDS a LOI value of 27.1 % and UL-94 V-1 rating. In contrast, 10 wt. % DOPO-POSS in the DEGBA/PA650 results in a LOI value of 25.9 % and a UL-94 V-1 rating. The details of fire behavior, such as the values of TTI, HRR, p-HRR, COPR, and CO_2PR have been tested using a cone calorimeter. DOPO-POSS in the DEGBA/DDS causes a lower value of p-HRR and longer TTI than in the DEGBA/PA650. The DEGBA/DDS with even as little as 2.5 wt. % DOPO-POSS easily forms a compact char. However, the DEGBA/PA650 with DOPO-POSS does not char until 10 wt. % DOPO-POSS. The thermal stability and pyrolytic gases of the two kinds of epoxy resins were detected by TGA-FTIR under a nitrogen atmosphere. DOPO-POSS performs better in accelerating charring in the DDS curing system compared with the PA650 curing system. It is postulated that for the

DEGBA/DDS/DOPO-POSS, fast and dense charring and accumulating of pyrolytic gases in the char contribute to the blowing-out effect. By contrast, the aliphatic chains of the PA650 are easy to break down and produce combustible gases, so are uneasy to form a crosslinking structure in the condensed phase until enough DOPO-POSS is added. These results may be very helpful for investigation of the conditions under which the blowing-out effect in epoxy resins can be caused by synergy of phosphorous (DOPO) and silicon (POSS).

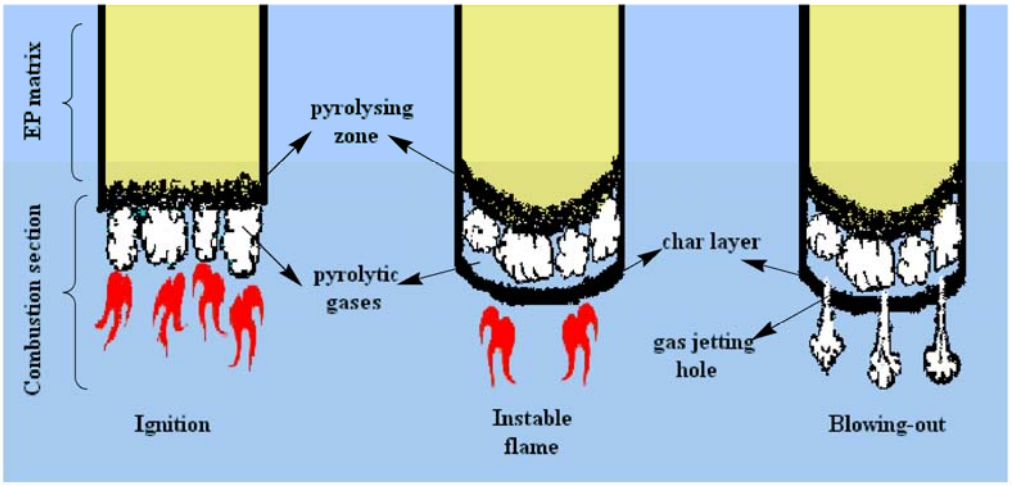

Fig. 1. The model of the blowing-out effect.

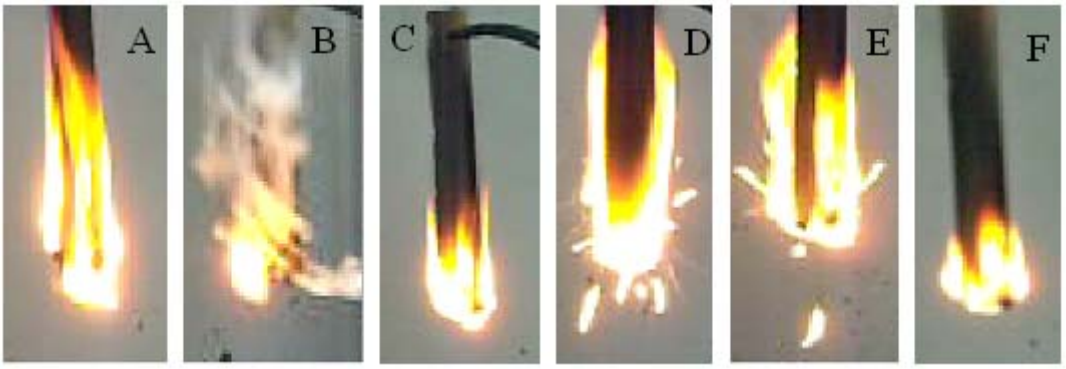

Fig. 2. (A) DGEBA/DDS, (B) DGEBA/DDS/2.5 wt. % DOPO-POSS, (C) DGEBA/DDS/10 wt. % DOPO-POSS, (D) DGEBA/PA650, (E) DGEBA/PA650/2.5 wt. % DOPO-POSS, and (F) DGEBA/PA650/10 wt. % DOPO-POSS.

References

[1] Zhang WC, Li XM, Yang RJ. Polym Degrad Stab 2011;96:2167-2173.

[2] Zhang WC, Li XM, Li LM, Yang RJ. Polym Degrad Stab 2012;97:1041-1048.