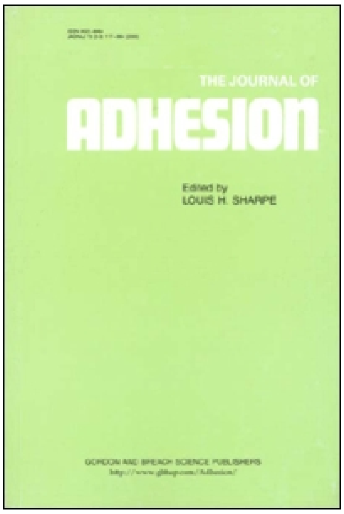

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A Study on the Properties and Structure of Polyether-Sulfone-Modified Epoxy Adhesives†

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Properties and morphology of PES-modified epoxy adhesives were studied in this paper. These systems have excellent mechanical properties and can be used as heat-resistant adhesives. SEM and EDX observations showed that the system has a two-phase structure. At lower PES content, there is a PES dispersed phase and a epoxy continuous phase. At higher PES content, there are two continuous phases. From these results, the relationship between structure and properties of this system were discussed.

KEY WORDS Epoxy adhesive; mechanical properties; morphology; phase separation; polyethersulfone (PES), structure.

INTRODUCTION

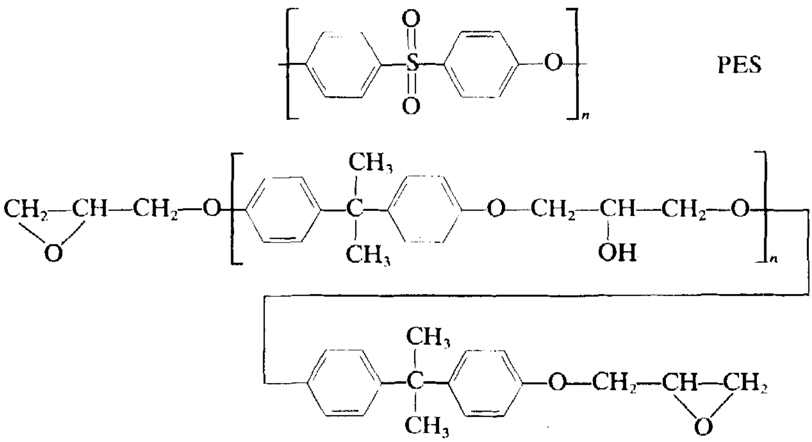
Polysulfones are a class of high-temperature-resistant thermoplastic materials, which have excellent mechanical properties and thermal stability. Adding polysulfones to epoxy resins, high mechanical strength could be obtained. Such systems have, therefore, found wide application in the fields of adhesives, coatings and composites.[1,2] This paper describes the properties and morphology of polyethersulfone (PES)-modified epoxy resins and discusses the relationship between structure and properties.

† Presented as a Poster Paper at the Tenth Annual Meeting of The Adhesion Society, Inc., Williamsburg, Virginia, U.S.A., February 22–27, 1987.

EXPERIMENTAL

1 Raw material

The chemical structures of the two resins used in the study are shown below. E-51 (Epon 828) epoxy has a viscosity of 2500 cp at 40°C, an epoxy equivalent weight of 196 g mol^{-1}, and a molecular weight of ~392. The PES has a molecular weight of ~25000. The hardener was dicyandiamide [DICY].



2 Experimental methods

(i) *Measurements of mechanical properties* Adhesives were prepared by mixing E-51 epoxy with PES previously dissolved in DMF and adding the hardener DICY. The adherends were LY 12 CZ aluminum alloy sheets.

The surface preparation of adherends was accomplished by degreasing, sanding and acid chemical etching, washing in running water and drying at 60°C.

The surface-prepared adherends were coated with the adhesives, dried at 110°C, and cured at 180°C for 3 hrs under 1 kg/sq cm pressure.

The shear strength, peel strength and non-uniform tear strength were measured according to GOST.

(ii) *SEM analysis of the fracture surface.* Adhesive solutions were heated to drive off the solvent, and cured at 180°C for 3 hrs, to produce samples. These samples were fractured at room temperature, coated with a thin layer of Au and examined by scanning electron microscope (SEM). Other samples were coated with carbon and subjected to energy dispersive X-ray (EDX) microanalysis for sulfur using SEM.

RESULTS AND DISCUSSION

Figure 1 and Figure 2 show the correlation between bond strength and concentration of PES. Epoxy adhesive containing 40–50 wt.% of PES has maximum bond strength, the maximum lap shear strength being 380 and 310 kg/sq cm, respectively, at 25°C and 150°C. The 90° peel strength is 7 kg/cm and the non-uniform tear strength is 66 kg/cm. In this system the two resins behave synergistically.

Table I shows the resistance of PES modified epoxy adhesives to liquid media compared to a polysulfone (P1700)-modified material. This system displays better solvent resistance, oil resistance and boiling water resistance than the general polysulfone [P1700] modified epoxy adhesive.

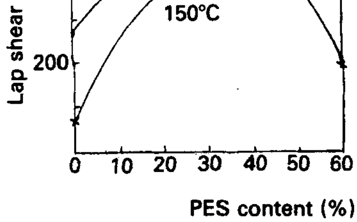


FIGURE 1 Lap shear strength *vs* PES content.

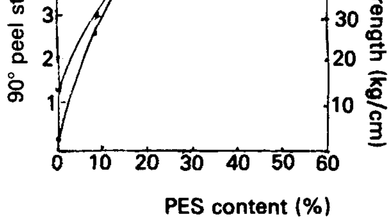
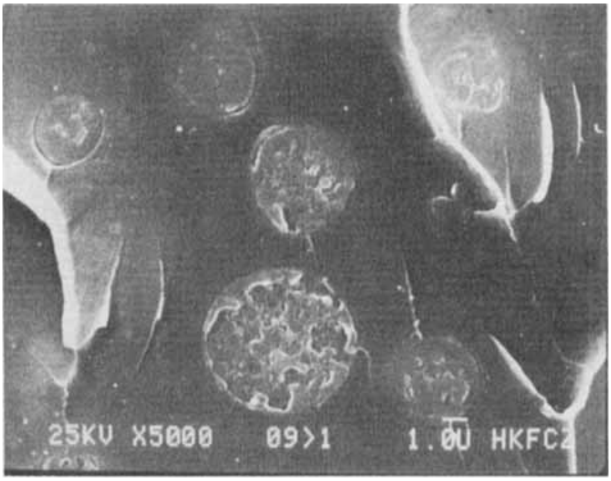


FIGURE 2 90° peel, non-uniform tear strength *vs* PES content.

TABLE I
Effect of liquid media on bond strength

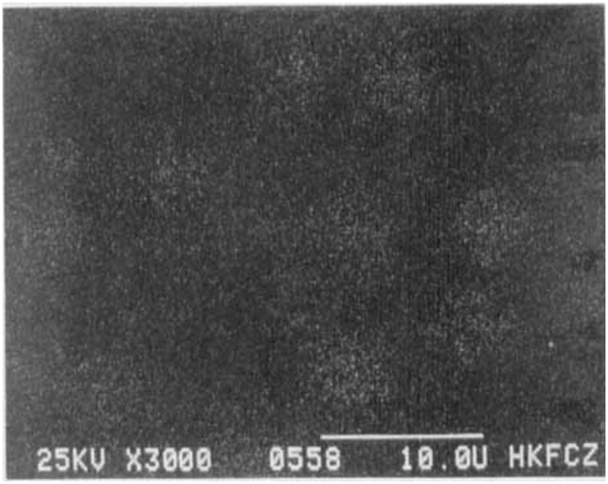
Media	Immersion time (days)	Average lap shear strength (kg/sq cm)	Lap shear strength retention (%)	Polysulfone (P1700) E-51 adhesives retention (%)
Ethyl acetate	30(R.T.)	357	98.0	81.6
Kerosene	30(R.T.)	362	99.5	71.5
Alcohol (95%)	30(R.T.)	347	95.0	88.2
	2 hrs	362	99.4	79.3
	6 hrs	353	97.0	75.0
Boiling water	48 hrs	335	92.0	55.0
	168 hrs	295	81.0	—

Figure 3 shows the morphology of the fracture surface of PES-modified epoxy resin samples. At lower PES content (Figure 3a) the system contains a dispersed phase appearing as globules. In order to identify the PES rich phase, EDX microanalysis was used to determine the distribution of sulfur in the fracture surface of the samples. These globules show a significantly higher concentration of sulfur than the surrounding matrix (Figure $3a_0$). It is clear that these globules also contain cross-linked epoxy resin. At 20% PES content

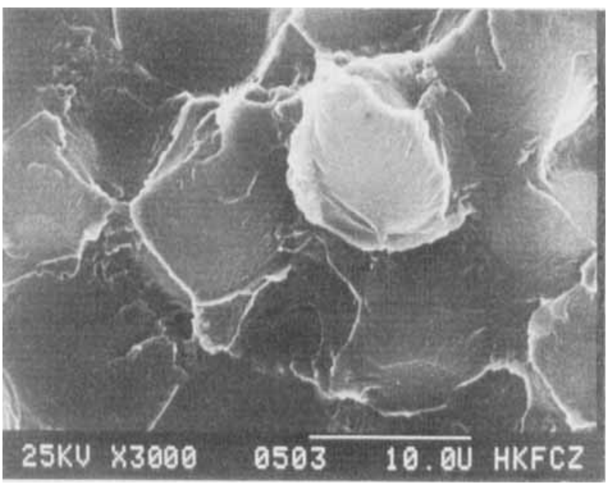


(a)

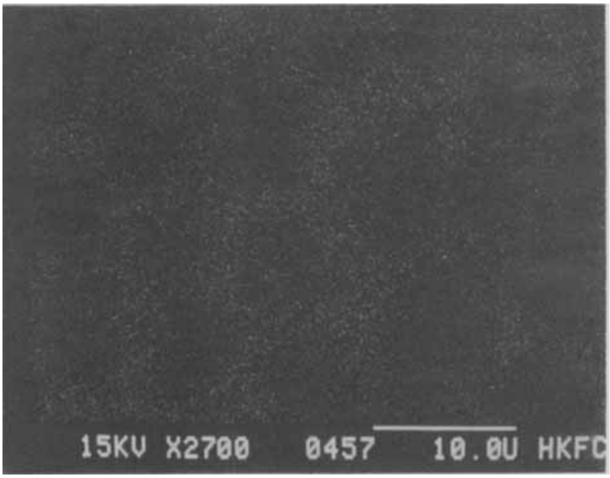
FIGURE 3 Morphology *vs* PES contents.



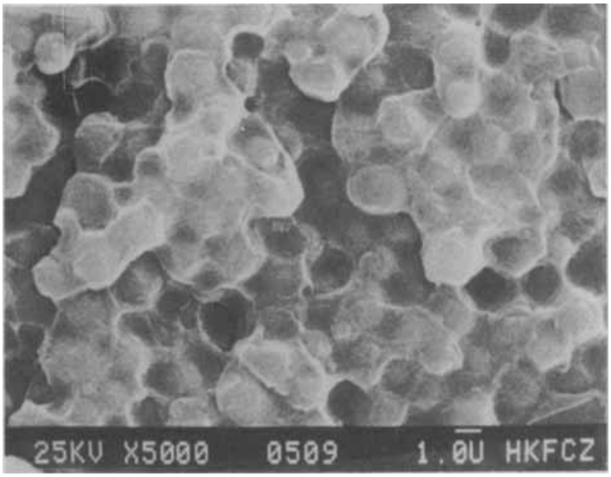
(a_0)
FIGURE 3 (*contd*)



(b)
FIGURE 3 (*contd*)



(b_0)
FIGURE 3 (*contd*)



(c)
FIGURE 3 (*contd*)

(Figure 3b), the globules disappear and the system exhibits a new morphology of two continuous phases (Figure $3b_0$). PES forms the more continuous phase and the epoxy forms a cellular structure. With increasing PES contents, the sizes of the epoxy cellular structure decrease rapidly, with the cellular structure disappearing at 60% PES content (see Table II).

TABLE II
Relationship between morphology and PES content

PES content wt.%	Morphology	Average domain diameter (μm)
0	a homogeneous phase	
10	a dispersed PES phase	2
20	two continuous phases	8
30	two continuous phases	2
40	two continuous phases	1
50	two continuous phases	0.5
60	a homogeneous phase	0

It has been noted that some cracks appear around the globules (Figure 3a). These cracks are similar to those in some CTBN-modified epoxy resins. In this respect, the PES globules probably act in a similar way as the rubber globules in CTBN-modified resins, in as much as the presence of the globules appears to benefit the bond strength of PES-modified epoxy adhesives.

At higher PES content (Figure 3b, Figure 3c), the phase boundary is not clear. This means that the compatibility of the two resins in phase boundary is much better. PES is a linear thermoplastic polymer, whereas the cured epoxy resin is a network polymer. On the basis of the morphology and tne composition, it is suggested that a snake-cage structure was formed, the cured epoxy resin forming cages and PES forming snakes. From the difference of the solubility parameters ($\Delta\delta = 3.3(\text{cal/cm}^3)^{1/2}$), PES and epoxy resin are chemically incompatible polymers, phase separation taking place in the curing reaction. But the snake cage structure can limit phase separation[3,4] and improve the bond strength.

CONCLUSIONS

1. PES-modified epoxy adhesives have high bond strength at room and high temperature, and excellent resistance to liquid media.

2. At lower PES content (<20%) the system contains a PES dispersed phase, which acts in a similar way as the rubber globules in CTBN-modified epoxy resins.

3. At higher PES content (≥20 wt.%), the system exhibits a morphology of two continuous phases. PES forms the more continuous phase, epoxy resin forms a cellular structure. The size of the cellular structure depends on the compositions, and a snake-cage structure was probably formed.

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