Preconditioning of epoxy film adhesives for bond strength improvement

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The moisture level of two commercial overaged epoxy film adhesives has been controlled by drying under vacuum and/or exposure to humid atmosphere. Shear and peel bond strengths of the conditioned adhesives were evaluated. Predrying of the uncured adhesive under vacuum (3–5 mm Hg) at room temperature is shown to be very effective for bond strength enhancement. Additional humidifying/drying cycles show the same effect but some irreversible degradation occurs and only partial improvement of adhesive bond strength is achieved.

Key words: adhesives; environmental testing; adhesive strength; epoxy resins

Experience among end-users of film adhesives has indicated inherent difficulties in B-stage epoxy-based materials due to the presence of the curing agent in the adhesive films. The curing reaction proceeds in the B-stage epoxy, even at low temperatures, sometimes to the point where the material is rendered unusable. The reversible or irreversible mechanism causing the degradation of adhesives due to the penetration of water into the precured polymer network has been reviewed by Comyn[1,2], Parker[3] and others.

In the present investigation, the effect of drying and humidifying cycles on two epoxy film adhesives (following storage for different durations in a deep freezer at − 18°C) was evaluated to determine the effect of absorbed or desorbed moisture (in the uncured film adhesive) on the ultimate shear and peel bond strength.

Experimental details

The substrate used was 2024 T351 aluminium etched by the FPL (Forest Products Laboratory) method. A thin layer of BR-127 primer (Bloomingdale Department of American Cynamid) was spray-applied and cured (30 min at room temperature (RT) and 1 h at 120°C).

Supported film epoxy adhesives FM-73 and FM-300K (Bloomingdale Department of American Cyanamid), which were stored for different durations in a deep freezer at − 18°C, were cured according to the manufacturer's recommendations.

The film adhesives were passed through humidifying (100% relative humidity (RH)) and drying (3–5 mm Hg) cycles. At each cycle the moisture gain or loss was determined by weighing and the effect of sorbed moisture was quantified by measurement of the lap shear and T-peel strengths.

Tensile lap shear specimens were prepared according to ASTM D-1002-72 and T-peel specimens according to ASTM D-3167-73T. Bondline thickness was 0.10 ± 0.03 mm.

Bond strength was determined using a 5 ton Instron machine (cross-head speed 2 mm min^{-1}) at 25 and 105°C. The mode of failure (adhesive or cohesive) was evaluated by visual inspection.

Results and discussion

The initial lap shear and T-peel strengths of the epoxy film adhesives which were stored for 6–42 months at − 18°C show a marked degradation from the expected values at both test temperatures (see Table 1). As can be seen from Fig. 1, a significant enhancement in bond strength was achieved by the drying process before cure. The bond strength increased as drying time increased up to 2.5 h (∼ 0.3% moisture loss), but extra drying caused some inconsistency and degradation. This phenomenon may be due to the removal of permeated moisture at the initial drying, but extra drying causes removal of residual solvents or diluents which reduces flow in the subsequent cure step[4]. The main outgassing product from the uncured film adhesive was identified as water by infrared spectroscopy[5].

The effect of drying/humidifying cycles on bond strength is shown in Table 1 and Fig. 2. The shear strength deteriorated significantly as the moisture content increased (see Fig. 2). Redrying resulted in consistently higher bond strength. However, samples which absorbed more than

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Table 1. Effect of drying/humidifying cycles on the strength of various film adhesives

Preconditioning	FM-73 (1.5 years storage)			FM-300K (3.5 years storage)		
	Lap shear strength (MPa) [44]*		T-peel ($N\ mm^{-1}$) [7.9]*	Lap shear strength (MPa) [46.7]*		T-peel ($N\ mm^{-1}$) [1.9]*
	RT	105°C	RT	RT	105°C	RT
No preconditioning	28.6 ± 0.7	5.2 ± 0.4	8.0 ± 0.2	23.6 ± 0.2	19.8 ± 1.2	0.9 ± 0.0
First drying cycle†	31.9 ± 0.3	7.7 ± 0.1	7.9 ± 0.0	26.1 ± 0.1	24.9 ± 0.4	2.6 ± 0.0
First humidifying cycle‡	27.5 ± 0.3	7.3 ± 0.7	6.3 ± 0.0	25.4 ± 0.7	20.5 ± 0.6	1.5 ± 0.0
Second drying cycle†	31.1 ± 0.6	11.0 ± 0.6	6.2 ± 0.0	23.2 ± 0.9	21.2 ± 0.5	2.0 ± 0.1

Values given are the average of five test specimens ± standard deviation
The specimens showed cohesive bond failure in all cases
*Values in square brackets are those given in the manufacturer's data
†3–5 mm Hg, 2.5 h, ∿ 0.3% weight loss
‡∿ 0.3% weight gain

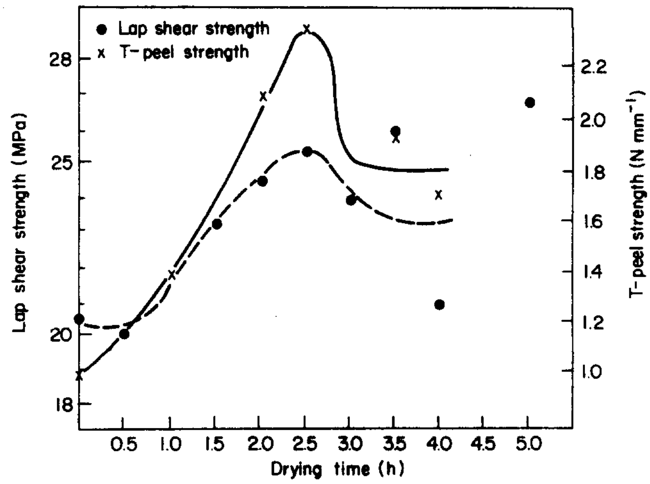

Fig. 1 Lap shear and T-peel strength *vs* drying time for FM-300K after two years storage. Maximum strength values occurred at 0.3% weight loss

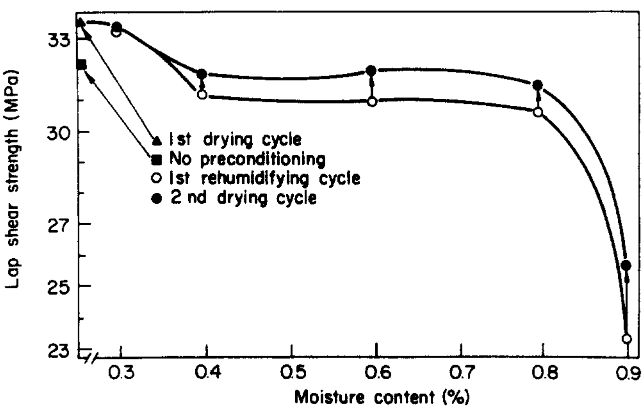

Fig. 2 Lap shear strength *vs* moisture content for FM-73 after 6 months storage

0.2–0.3% moisture did not return to their original (after first drying cycle) strength. This may be attributed to the irreversible mechanism occurring at higher moisture level. Table 1 also shows that humidifying cycles (∿ 0.3% weight gain) caused degradation of bond strength, *ie* RT T-peel and RT, 105°C lap shear, in most cases. Drying cycles improved the bond strength in most cases.

The phenomena described above reappeared in all tested adhesive batches which were stored for different periods[5]. The results presented in Table 1 and Figs 1 and 2 are quite typical. Therefore preconditioning of overaged B-staged adhesives under 3–5 mm Hg (gauge) vacuum for controlled periods is recommended to remove moisture and to regain bond strength.

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