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Hygrothermal Properties of Adhesively Bonded Joints and Their Correlation with Bulk Adhesive Properties

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The combined effects of heat (50°C) and humidity (95% R.H.) on the lap shear and T-peel strengths of 120°C, 150°C and 215°C service epoxy film adhesives have been characterized. Experimental results have indicated that effects of hygrothermal conditioning on lap shear and peel properties vary with exposure time and final testing temperatures and type of adhesive tested. In the cases where cohesive failure was observed in the shear and peel specimens, a correlation could be established between the bulk properties of the adhesives (tensile strength and elongation) and their adhesively bonded joint properties (shear and peel). When testing was carried out at room temperature, a general correlation between the tensile elongation and T-peel or shear could be obtained. At below freezing temperatures, lap shear strength seemed to be correlated with bulk tensile strength while peel correlated with bulk tensile elongation. At elevated temperatures, the relative contributions of bulk strength and elongation were the decisive factors as far as shear and peel strengths are concerned.

KEY WORDS Epoxy adhesives; hygrothermal properties; shear strength; peel strength; bulk properties; tensile elongation; tensile strength.

INTRODUCTION

Mechanical property degradation of adhesively bonded joints, due to moisture absorption and exposure to elevated temperature, limits the applications of epoxy-based joints to relatively moderate environments. The effect of humidity on property variations of epoxy adhesive has been attributed¹⁻⁷ to degradation of hydrogen and chemical bonds at the adhesive-adherend interface and subsequent corrosion of the adherend interface, plasticization of the epoxy resin, and finally hydrolysis of the adhesive. Heat alone causes the reduction of the adhesive rigidity and strength. However, the combination of heat and humidity has been found to be highly detrimental. Maloney *et al.*⁸ showed that exposure of epoxy-aluminum double lap shear joints to 50°C at 100% relative humidity (R.H.) resulted in gradual loss of strength with time. Similarly, Parker⁹ reported that aluminum bonded epoxy joints exhibited reduced properties upon exposure to humidities above 85% combined with temperatures above 35°C. In all cases,

the degradation mechanisms were related to water diffusion through the bond line¹⁰⁻¹² followed by interfacial corrosion, resin plasticization, and hydrolysis.

When bulk specimens of epoxy adhesives are exposed to heat and humidity the decisive degradation mechanisms are only those related to the resin itself—plasticization and hydrolysis. Furthermore, since the exposed surface-area in this case is much larger than the bond line surface-area in the case of bonded joints, the resin-induced degradation mechanisms are accelerated.

The bulk properties of adhesives play a major role in affecting the joint strength. A ductile adhesive with large elongations will lead to high joint strengths in cases where high stress concentrations appear. Elevated temperatures reduce the rigidity and tensile strength of the adhesives and consequently the joint strength. However, the result is increased strain to failure. Hence, the interaction of these two opposing effects leads to a complex dependence of adhesive joint properties on temperature. Similarly, moisture absorption may lead to increased elongations due to plasticization and concurrently to hydrolysis and degradation of the bulk adhesive. The end result in the adhesive joint is complicated and depends on the relative importance of these factors for a given adhesive and a given joint geometry.

From the practical as well as characterization points of view, it is desirable to relate the hygrothermal properties of bulk adhesives with those of the corresponding joints properties. Consequently, the objectives of the present study are aimed at extending the authors' previous study¹³ on the hygrothermal properties of bulk epoxy adhesives to their properties in lap shear and peel joints, and phenomenologically correlating the bulk and corresponding joints properties.

EXPERIMENTAL

Adhesives studied

Three groups of adhesives, with regard to their maximum service temperature (120°C—FM 73; AF 163, MB 1137; 150°C—FM 300K, MB 1515; 215°C—FM 400), have been included in the present study. Except for AF-163, all others were investigated in their bulk form and were included by the authors in an earlier study.¹³ Table I summarizes the adhesives' sources, curing conditions and glass transition temperatures.

Specimen preparation

Except for bulk specimens¹³ two types of specimens were used in the present study: single lap shear and T-peel joints.

Following storage in low temperature (−18°C), the sealed adhesives were left to thaw at room temperature. The adhesives were then cut into 12.7 × 25.4 mm and 171.5 × 228.6 mm pieces for shear and peel specimens, respectively.

Single lap shear joints were prepared according to ASTM-D-1002-72¹⁴ by bonding 101 × 25.4 × 1.6 mm pieces of unsealed chromic acid anodized 2024-T351

TABLE I
Adhesives studied and their curing conditions

Adhesive	Manufacturer	Max. Service Temp. (a) [°C]	Curing (a) [°C/h]	T _g (b) [°C]
FM 73	American Cyanamid	120	120/1	100
AF 163	3M	120	121/1	120
MB 1137	BASF	120	120/1	127
FM 300K	American Cyanamid	150	175/1	175
MB 1515	BASF	150	175/2	170
FM 400	American Cyanamid	215	175/1	190

^a According to the manufacturer's data sheets.

^b According to Ref. 1 and later measurements.

aluminum. The aluminum plates with the uncured adhesives were assembled in a special tool and were cured under pressure of 49 KPa in a temperature controlled oven for the durations and temperatures defined in Table I.

T-peel joints were prepared according to ASTM-D-1876-72¹⁵ by bonding 305 × 171.5 × 0.5 mm aluminum panels of the same type with the same treatment as mentioned above. The panels with the uncured adhesives were placed in a temperature-controlled press and were cured under the same pressure, duration and temperature as defined in Table I. The cured panels were then cut to 305 × 25.4 mm specimens.

Testing conditions and procedures

Mechanical properties of the bonded specimens were determined at a reference state-room temperature and moisture-free conditions (as received), and following exposure for 30 and 90 days to 50°C and 95% relative humidity (5 specimens were fabricated for each test).

After hygrothermal conditioning, adhesive strength was determined at -30°C, 25°C and at one elevated temperature (Th) according to the glass transition temperature of the studied adhesive. The 120°C maximum service temperature adhesives were tested at Th = 90°C, the 150°C group at Th = 150°C and the 215°C category at Th = 200°C. Specimens were conditioned at the testing temperature for 5–7 minutes prior to testing. This period of time was chosen using a thermocouple to ensure that the specimen reached the desired temperature. The loading rate was 2 mm/min for shear loading using an Instron 1185 mechanical testing machine including an extensometer.

Failure mode was determined by type of failure (Cohesive or Adhesive) and visual estimation of the total area covered by the adhesive.

Peel strength was determined at the same testing temperatures and following the same conditioning cycles as for the shear strength. Loading rate was 100 mm/min.

RESULTS AND DISCUSSION

Lap shear strengths

Table II summarizes shear strength levels of the studied epoxy adhesives exposed to hygrothermal conditions (50°C/95% R.H.) for 30 and 90 days and tested at three different temperatures (-30°C, RT, and Th).

Shear strengths of all adhesive joints measured at room temperature exhibited minor changes after 30 days of exposure except FM 400 which lost about 45% of its shear strength. After 90 days of exposure shear strength decreased by about 10–30%, while FM 400 lost about 50% of its original strength. As Table II indicates, failure mode tends to be cohesive in nature for all adhesives loaded at R.T., except for FM 400 and MB 1515. The former adhesive exhibits adhesive failure regardless of exposure time. This may be attributed to water attack at the epoxy-aluminum interface. The latter adhesive displayed peculiar behavior as its failure mode changed from adhesive to cohesive with increasing exposure time. It should be emphasized that the same general results were reported for FM 73, FM 300K and Fm 400.^{2,8,9}

The variations in shear strength at -30°C are within the standard deviation after 30 and 90 days of exposure (with the exception of MB 1515 that displayed

TABLE II
Shear strength after exposure to humidity at several testing temperatures

Adhesive	Humidity	Testing Temperature							
		25°C		-30°C			Th		Th [°C]
		Exposure Time [days]	Shear Strength [MPa]	Fail. Mode [%] ^a	Shear Strength [MPa]	Fail. Mode [%] ^a	Shear Strength [MPa]	Fail. Mode [%] ^a	
FM 73	0	39.0 ± 1.9	80 C	33.1 ± 4.0	80 C	18.3 ± 0.7	100 A	90	
	30	33.3 ± 0.6	50 C	37.9 ± 3.6	100 A	12.9 ± 0.5	100 A		
	90	30.5 ± 2.2	40 C	32.7 ± 3.7	100 A	8.4 ± 1.7	100 A		
MB 1137	0	29.1 ± 2.0	80 C	32.9 ± 1.6	100 C	19.0 ± 0.5	100 C	90	
	30	30.8 ± 1.1	100 C	31.5 ± 1.9	100 C	15.6 ± 0.8	100 C		
	90	19.8 ± 2.0	70 A	34.3 ± 1.9	75 C	12.2 ± 1.0	100 C		
AF 163	0	39.3 ± 1.3	100 C	40.2 ± 4.0	100 C	20.3 ± 2.4	10 C	90	
	30	39.9 ± 1.9	100 C	45.5 ± 0.8	100 C	19.2 ± 2.9	15 C		
	90	34.1 ± 2.4	70 C	43.3 ± 4.6	80 C	11.5 ± 1.9	100 C		
FM 300K	0	40.4 ± 3.0	90 C	31.0 ± 4.7	100 A	14.5 ± 2.2	60 C	150	
	30	35.4 ± 1.4	70 C	32.0 ± 2.6	100 A	9.8 ± 2.6	100 A		
	90	34.8 ± 2.8	50 C	35.4 ± 2.5	100 A	5.6 ± 0.9	100 A		
MB 1515	0	24.4 ± 1.8	100 A	26.2 ± 1.0	100 A	16.5 ± 0.8	100 A	150	
	30	25.5 ± 2.5	100 A	24.4 ± 1.7	100 A	10.9 ± 0.8	100 A		
	90	21.5 ± 1.5	75 C	10.2 ± 1.7	100 A	10.9 ± 0.8	100 A		
FM 400	0	36.2 ± 7.5	100 A	18.4 ± 1.4	100 A	13.0 ± 1.6	80 C	200	
	30	19.5 ± 2.6	100 A	18.9 ± 2.3	100 A	5.9 ± 1.4	100 C		
	90	18.1 ± 1.8	100 A	20.2 ± 1.5	100 A	4.5 ± 0.5	100 C		

^aFailure Mode: C = Cohesive, A = Adhesive.

60% reduction after 90 days of exposure). The changes in failure modes are relative minor, since the effect of the frozen humidity at -30°C is small.

The lowest shear properties for all adhesives were found, as expected, at the elevated temperatures (T_h). This is attributed to weakening of the adhesive as well as an enhanced moisture effect and interfacial degradation at the elevated testing temperature.

The effect of the hygrothermal environment is similar to that found when tested at RT but, generally, it is clear that the retention strength at T_h is lower than at RT which, in turn, is lower than at -30°C .

Based on the experimental characteristics, it is possible to categorize the adhesives into three groups with respect to their shear strength:

- a) Those which reach 40 MPa at RT: FM 73, AF 163, FM 300K
- b) Those which are below 30 MPa at RT: MB 1137, MB 1515
- c) FM 400 (30–40 MPa).

Shear strength varies at -30°C or at T_h . At those extremes, the retention of shear strengths with respect to hygrothermal exposure and/or testing temperature dictate different categories:

- a) High property retention adhesives: AF 163 > MB 1137 > FM 73
- b) Medium property retention adhesives: MB 1515 > FM 300K
- c) Low property retention adhesives: FM 400.

T-peel strength

Table III summarizes T-peel strength levels of the studied epoxy adhesives exposed for 30 and 90 days exposure to the hygrothermal conditions ($50^{\circ}\text{C}/95\% \text{ R.H.}$) and tested at three different temperatures (-30°C , RT and T_h).

Minor reductions in peel strengths were observed at room temperature after 30 and 90 days exposure to the hygrothermal environment. FM 300K exhibited outstanding stability compared with the other studied adhesives. Moreover, the mode of failure of the adhesives remained cohesive even after 90 days exposure, except FM 73. In the latter adhesive, the failure mode became adhesive following 90 days exposure.¹⁶ The highest reductions in peel strength at room temperature following hygrothermal exposure were observed for FM 400 and MB 1515, as was the case with shear strengths properties.

At -30°C , peel strengths of FM 73, AF 163, FM 300K and FM 400 degraded after 30 and 90 days of conditioning. Distinctively, MB 1137 and MB 1515 exhibited small increases in T-peel strength. Similar to RT results, the failure mode was found to be cohesive except for FM-73 which demonstrated adhesive failure.

At T_h the same general response to hygrothermal environment was observed as was the case at RT and -30°C .

TABLE III
Peel strength after exposure to humidity at several testing temperatures

Adhesive	Humidity	Testing temperature						Th [°C]
		25°C		-30°C		Th		
		Exposure time [days]	Peel strength [KN/m]	Fail. mode [%] ^a	Peel strength [KN/m]	Fail. mode [%] ^a	Peel strength [KN/m]	
FM 73	0	8.7 ± 0.5	100 C	7.5 ± 0.4	100 C	5.3 ± 1.6	100 C	90
	30	6.1 ± 0.4	50 C	5.5 ± 0.3	100 C	5.4 ± 0.3	100 C	
	90	4.6 ± 0.2	80 A	5.7 ± 0.3	50 C	4.7 ± 0.4	100 A	
MB 1137	0	5.5 ± 0.8	100 C	4.2 ± 0.5	100 C	3.8 ± 0.4	100 C	90
	30	5.9 ± 0.3	100 C	5.0 ± 0.5	100 C	3.6 ± 0.3	100 C	
	90	4.8 ± 0.5	100 C	5.4 ± 0.2	100 C	4.0 ± 0.2	100 C	
AF 163	0	5.6 ± 0.5	100 C	4.0 ± 0.4	100 C	6.7 ± 0.7	100 C	90
	30	5.2 ± 0.5	100 C	3.9 ± 0.4	100 C	4.7 ± 0.5	100 C	
	90	4.8 ± 0.3	100 C	2.6 ± 0.2	100 C	5.1 ± 0.4	100 C	
FM 300K	0	2.5 ± 0.4	100 C	1.0 ± 0.3	100 C	3.8 ± 0.5	100 C	150
	30	2.0 ± 0.3	100 C	1.3 ± 0.1	100 C	3.6 ± 0.5	100 C	
	90	2.0 ± 0.1	100 C	0.8 ± 0.1	100 C	2.9 ± 0.2	100 C	
MB 1515	0	1.6 ± 0.5	100 C	0.7 ± 0.2	100 C	0.5 ± 0.1	100 C	150
	30	1.0 ± 0.4	100 C	0.7 ± 0.1	100 C	0.7 ± 0.3	100 C	
	90	0.9 ± 0.1	100 C	0.8 ± 0.2	100 C	0.9 ± 0.1	100 C	
FM 400	0	3.3 ± 0.1	100 C	3.2 ± 0.3	100 C	1.6 ± 0.1	100 C	200
	30	1.9 ± 0.8	100 C	2.1 ± 0.3	100 C	1.0 ± 0.3	100 C	
	90	2.6 ± 0.1	100 C	1.9 ± 0.2	100 C	0.8 ± 0.4	100 C	

^a Failure Mode: C = Cohesive, A = Adhesive.

Generally, elevated temperatures and adhesive plasticization due to humidity absorption result in softening of adhesives. This, in turn, may enhance their inherent bulk peel strength and, in contradiction, degrade the adhesive-adherend interfacial peel strength. The relative contribution of these two opposing effects on the T-peel strengths varied and depended on the specific adhesive studied.¹⁷ Based on the adhesive characteristics, it is possible to categorize the adhesives into three groups with respect to their peel strength:

a) Toughened adhesives characterized by high peel strength: FM 73 > MB 1137 = AF 163

b) Untoughened adhesives: FM 300K > MB 1515

c) Particulate filled adhesive-FM 400 with moderate peel strength.¹⁸

Correlation between adhesive bulk and joint properties

The adhesive bulk response at various testing temperatures to hygrothermal conditioning has been previously studied.¹³ Experimental results indicated that testing temperature had a pronounced effect on the adhesive tensile modulus and strength, while the effect of moisture content varied with respect to adhesive type. The moduli of the film adhesives, having a wide range of glass-transition

temperatures (T_g), have been related to both moisture level in the adhesive and testing temperature. This has been accomplished by employing a dimensionless temperature, which incorporates the wet and dry T_g and the testing, as well as reference temperatures. The strength properties have shown a higher degree of scatter using the above mentioned dimensionless temperature.

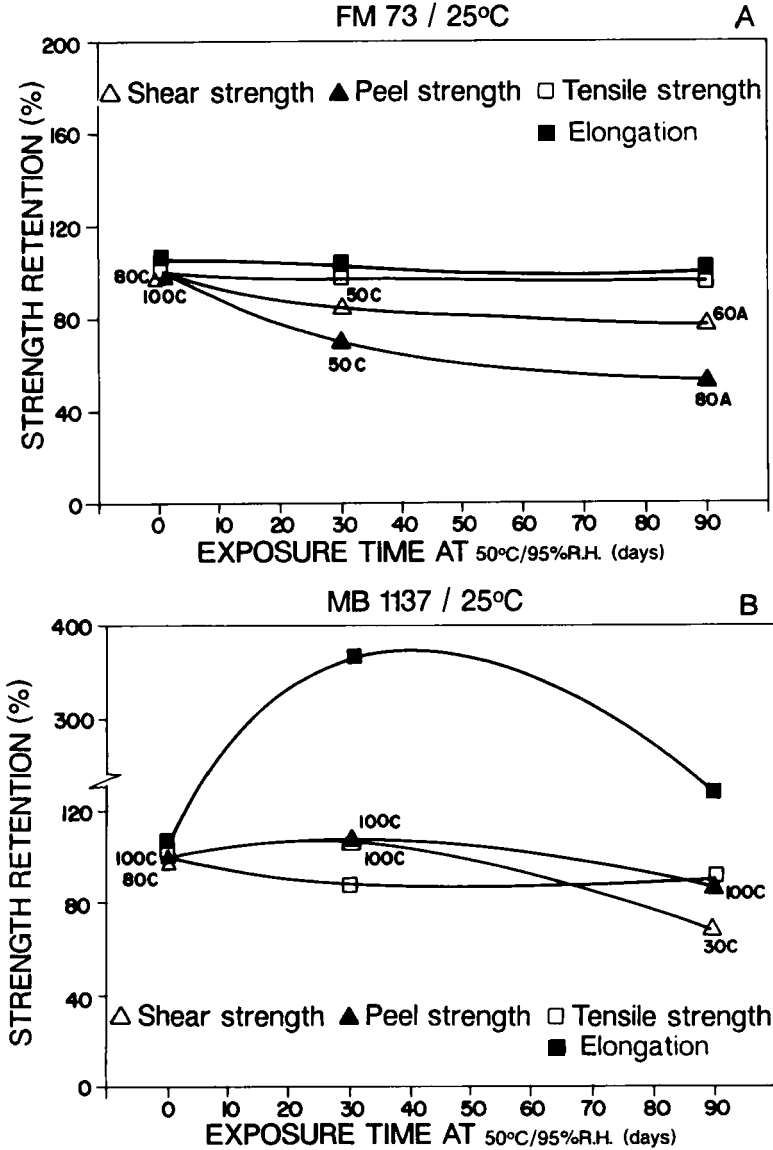


FIGURE 1 Retention values (%), measured at 25°C of adhesive joint strengths, failure mode (% , A-adhesive C-cohesive) and bulk properties as a function of exposure time to 50°C/95% R.H. A: FM 73, B: MB 1137, C: AF 163, D: FM 300K, E: MB 1515, F: FM 400.

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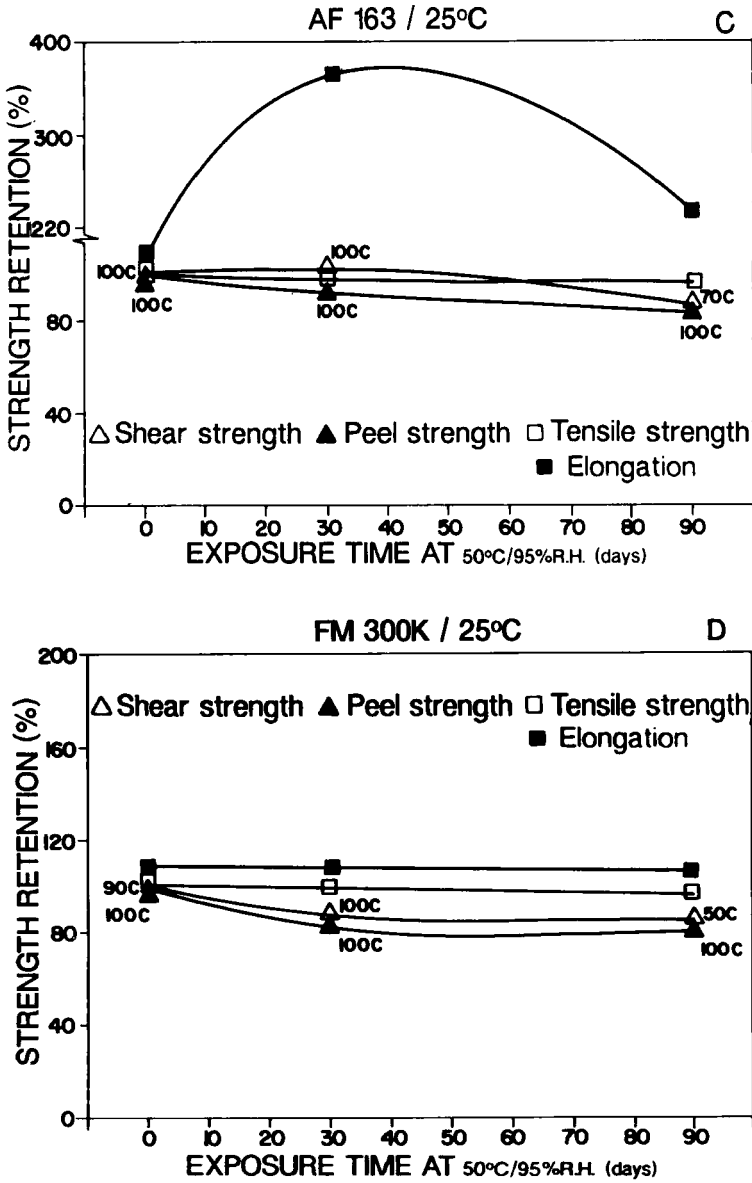


FIGURE 1—(continued)

Tables II and III summarize the lap shear and T-peel adhesive joint strengths of the same high performance epoxy film adhesives studied in the previous research.¹³ The adhesives were characterized over the same hygrothermal conditions.

An attempt was made to correlate the mechanical properties as characterized in the adhesive bulk state to those obtained using adhesively-bonded lap shear and

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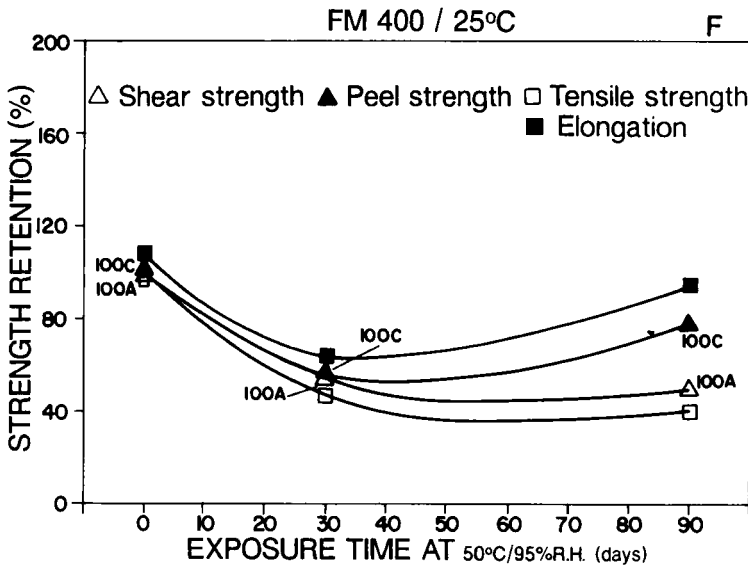
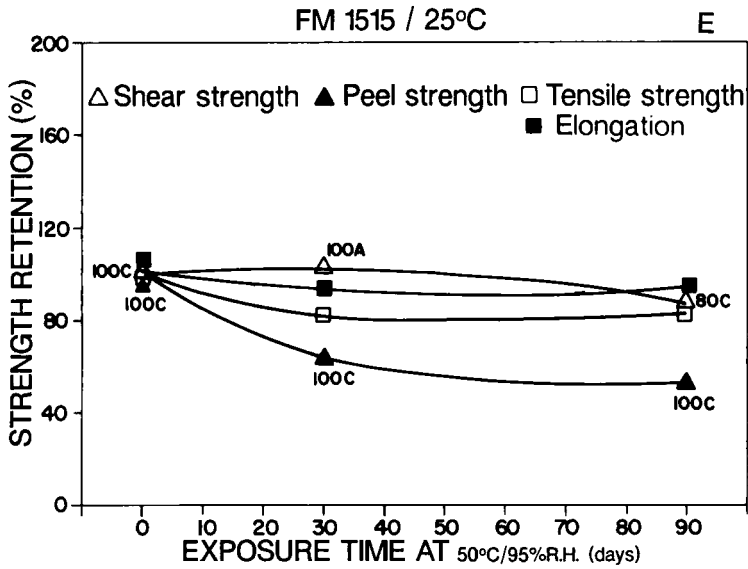


FIGURE 1—(continued)

peel joints. Figures 1, 2 and 3 display the tensile strength and elongation of the bulk adhesives together with the respective shear and peel strengths for various testing temperatures and hygrothermal exposure times. For the purpose of comparison, properties were normalized with respect to R.T. at unexposed conditions. Furthermore, the mode of failure was characterized by cohesive, C, or adhesive, A. To establish the bulk-joint correlation, properties determined in

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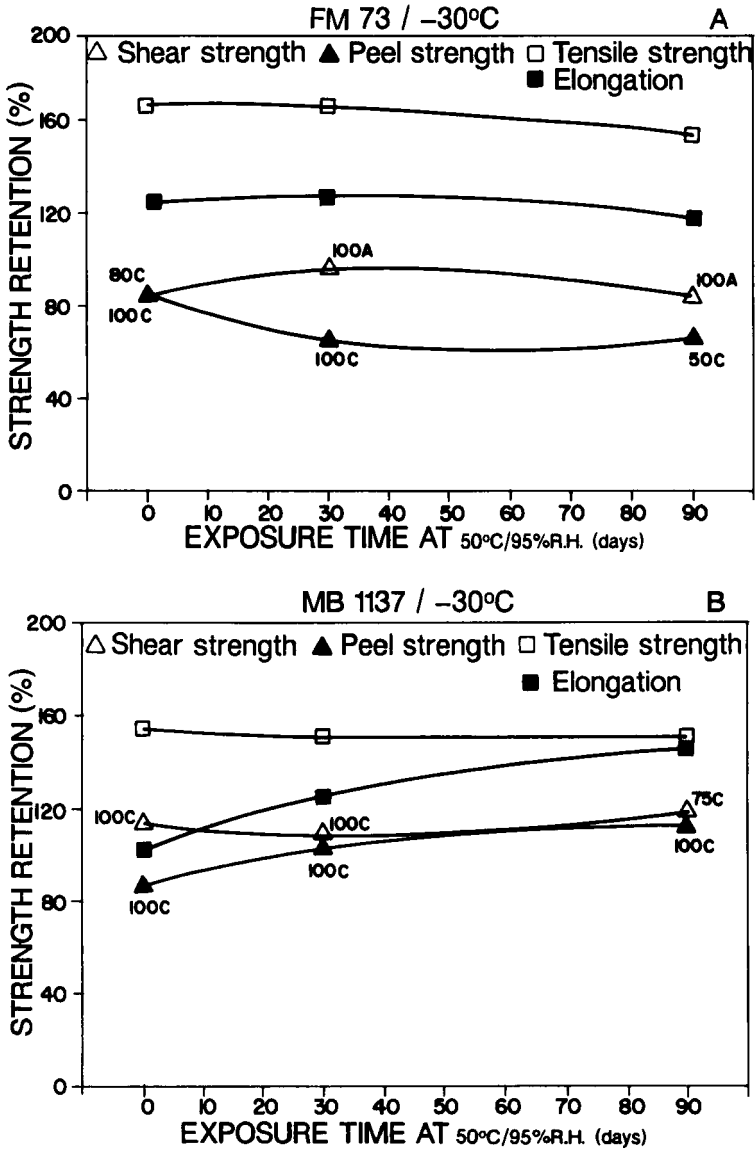


FIGURE 2 Retention values (%), measured at -30°C, of adhesive joint strengths, failure mode (%), A-adhesive, Cohesive) and bulk properties as a function of exposure time to 50°C/95% R. H.: A: FM 73, B: MB 1137, C: AF 163, D: FM 300K, E: MB 1515, F: FM 400.

specimens that failed adhesively were excluded. Only cohesively failed joints were taken into consideration since a correlation, if one exists, is meaningful only in those cases.

Figure 1A indicates that the magnitude of the changes in FM 73 bulk properties at RT are smaller when compared with those of adhesive joints. However, they

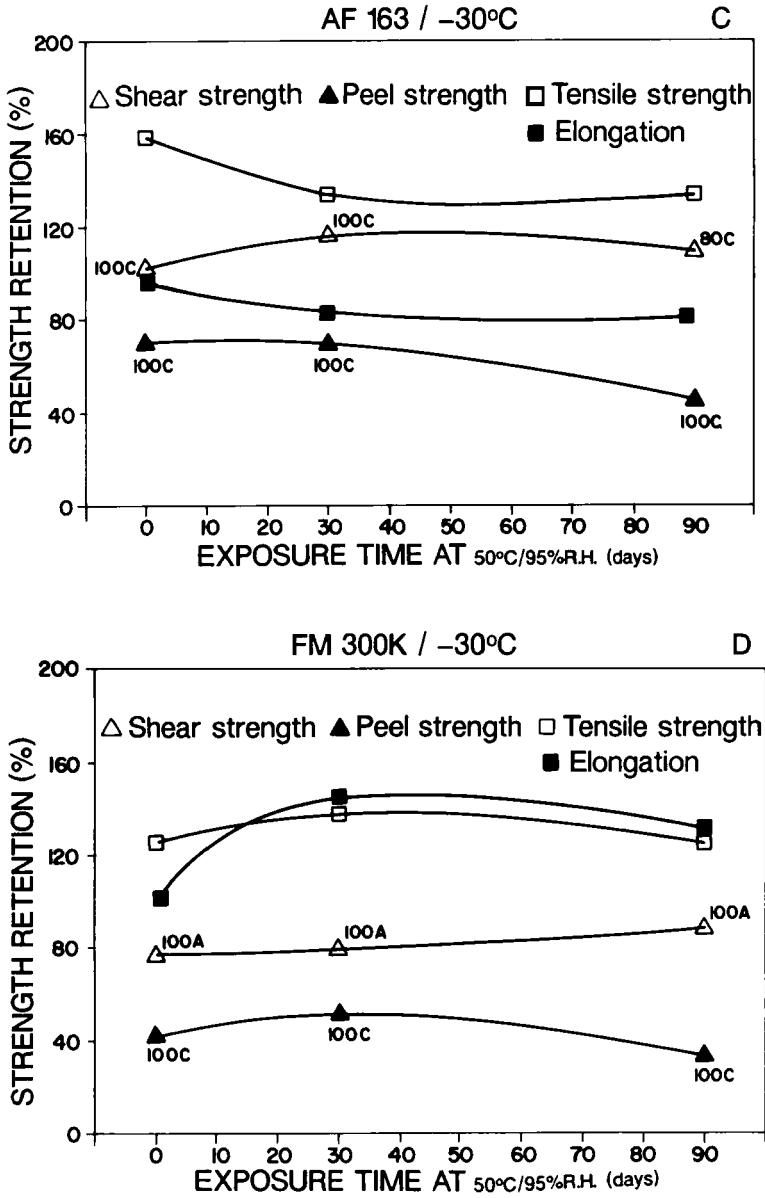


FIGURE 2—(continued)

follow the same trend. As exposure time to hydrothermal conditions was prolonged, property retention decreased.

MB 1137 exhibited a somewhat different behavior. As observed in Figure 1B, an initial increase in shear and peel properties occurred after 30 days of hydrothermal conditioning, corresponding to an equivalent increase in elongation. Subsequently, joint strength decreased at longer exposures, with shear

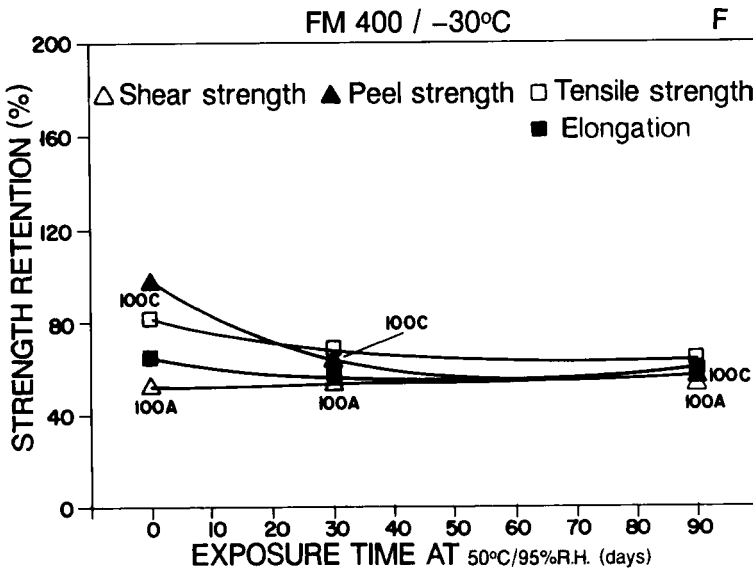
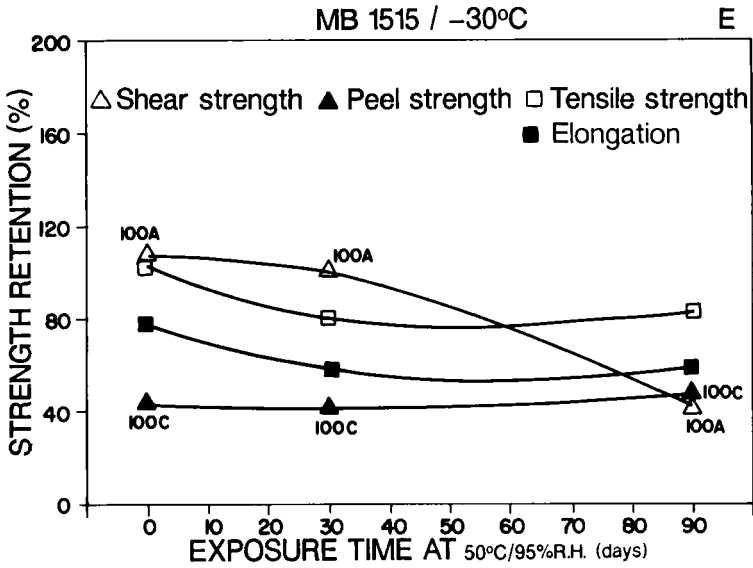


FIGURE 2—(continued)

strength affected mainly by weakening of the interface. In this case, the plasticization effect of humidity resulted in initially enhanced shear and peel values.

AF 163, as shown in Figure 1C, demonstrated a similar increase in tensile elongation after one month of exposure to heat and humidity. In this case, a smaller increase in shear strength was obtained with almost no effect on peel strength.

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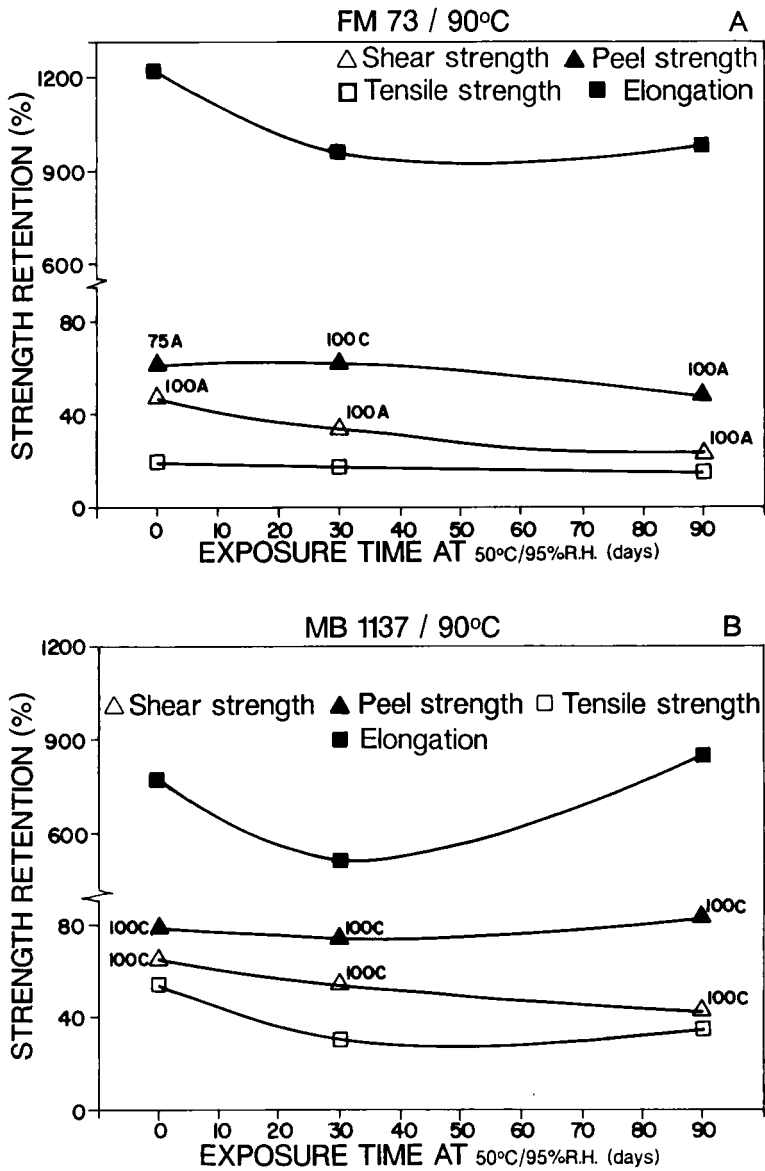


FIGURE 3 Retention values (%), measured at Th, of adhesive joint strengths, failure mode (%), A-adhesive, C-cohesive) and bulk properties as a function of exposure time to 50°C/95% R.H.: A: FM 73/90°C, B: MB 1137/90°C, C: AF 163/90°C, D: FM 300K/150°C, E:MB 1515/150°C, F: FM 400/200°C.

The two 150°C service adhesives— FM 300K and MB 1515 (Figures 1D and 1E, respectively) showed similar behavior as far as tensile elongation is concerned. However, in this class of materials peel strength, and partially shear strength, could be correlated with tensile strength of the bulk adhesive. As seen, a minor decrease in tensile strength at RT upon exposure to heat and humidity could be

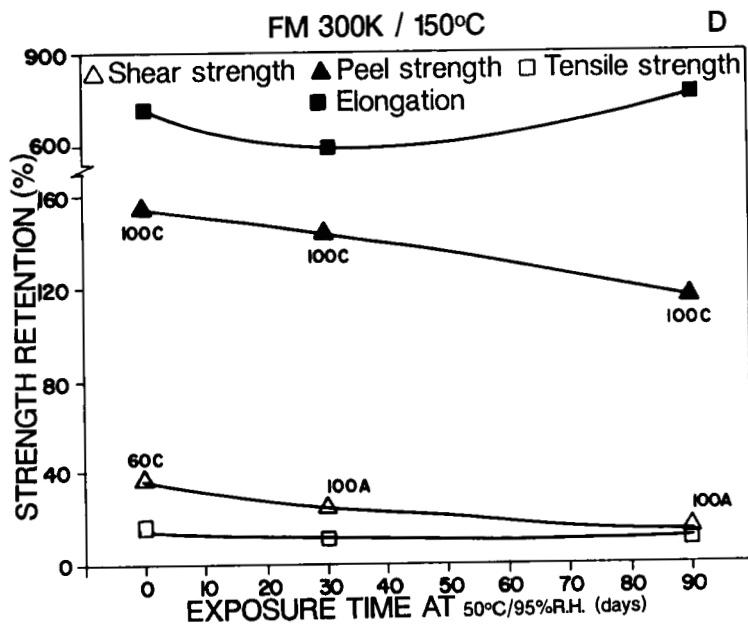
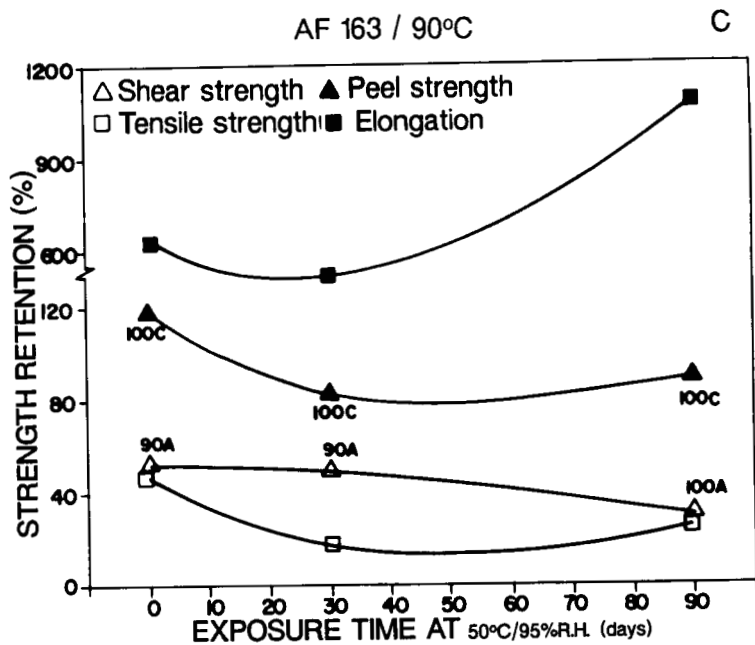


FIGURE 3—(continued)

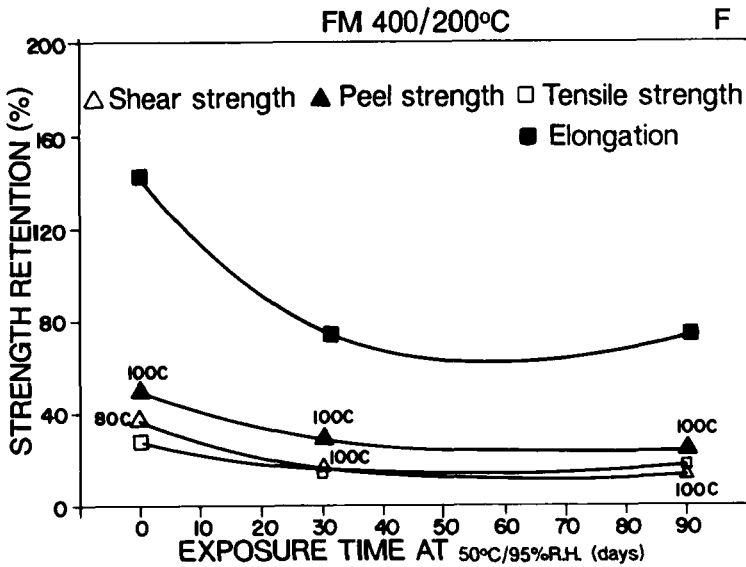
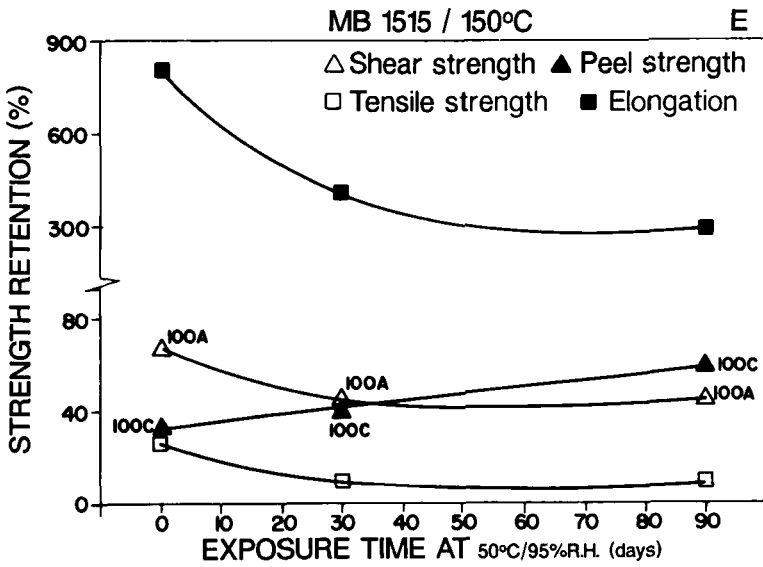


FIGURE 3—(continued)

associated with a relatively small reduction in both shear and peel strengths of FM 300K and a relatively larger one in the case of MB 1515.

The high-temperature-cured adhesive FM 400 in Figure 1F degraded markedly when exposed to heat/humidity. This was manifested both in bulk as well as in joint properties. In this adhesive a decrease in elongation is accompanied by an

equivalent decrease in peel strength. Since shear failure was adhesive, no effort was made to correlate bulk and shear properties.

Figure 2 depicts the property retention levels of the adhesives as obtained from shear and peel joints and from adhesive bulk specimens at -30°C .

The 120°C curing adhesives (FM 73, AF 163, MB 1137) demonstrated an increase in shear strength compared with RT values, associated with an increase in tensile bulk strength. Furthermore, only minor degradation in joint properties was observed due to hygrothermal exposure.

With respect to peel strength, FM 73 showed a decrease in properties compared with RT but there was no correlation with its tensile elongation. Distinctively, MB 1137 and AF 163 demonstrated a clear correlation between their peel strength variation with exposure time and their elongation to break.

The shear failure mode of the 150°C adhesives at -30°C had been found to be adhesive in nature. Thus, they were excluded from the comparative investigation. Peel failure mode at -30°C which was cohesive, though exhibiting lower absolute values compared with those at RT, showed the same general trends as the tensile elongation. Similar reponse was obtained with FM 400.

Testing at 90°C of the 120°C curing adhesives following hygrothermal exposure gave rise to distinct behavior as illustrated in Figure 3. In all three adhesives the tensile elongation increased substantially. However, the weakening effect of the bulk adhesive strength counterbalanced the elongation increase. Consequently, peel strength decreased with temperature compared with RT, but remained relatively at the same level with increasing hygrothermal exposure time. Whenever shear failure was cohesive (MB 1137) the same trend was observed. The 150°C service adhesives exhibited somewhat different behavior when tested at 150°C (Figure 3). Though their tensile strength was highly degraded, the relatively high increase in elongation resulted in very high peel strength in the case of FM 300 with moderate values in the case of MB 1515. At 150°C most of the shear failure modes were observed to be interfacial.

FM 400 that was tested at 200°C demonstrated a relatively large decrease in tensile elongation and strength. This was also exhibited in their respective peel strengths.

CONCLUSIONS

Correlations between adhesive bulk properties (tensile elongation and strength) and the corresponding adhesive joint properties (peel and shear strengths) were established. The correlations were observed, provided the failure modes were cohesive in nature. The correlations depended on the class of adhesive studied (120 , 150 and 215°C service temperature) and loading temperatures.

In cases where testing was performed at room temperature, lap shear and T-peel strengths could be correlated with bulk tensile elongation. At -30°C , lap shear properties indicated a correlation with bulk tensile strength, while T-peel could be correlated with the respective bulk tensile elongation. Elevated

temperature results indicated that the relative contributions of the bulk tensile strength and bulk tensile elongation affected the shear or peel strengths of the joints.

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