Creep, Strength and Moisture Absorption of Adhesive Bonded FRP Joints*

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ABSTRACT

The effects of the environment on adhesive bonded single lap joints formed using XMC-3 and SMC-R50 composites were investigated. Tests were performed at temperatures of 23C and 93C with test coupons immersed in air, water, and 5% NaCl-water mixture. The weight changes of both bonded joints (XMC-3 to SMC-R50 and SMC-R50 to SMC-R50) and unbonded materials were measured. Data were also obtained showing the effects of moisture, temperature, and applied load on changes in weight, on creep deformation, lap shear strength, and separation modes of the joints.

1. INTRODUCTION

THE USE OF LIGHTWEIGHT FIBER REINFORCED PLASTICS (FRP) IN AUTOmotive applications offers a promising avenue towards achieving better fuel economy through weight reduction. In order to take full advantage of such materials, suitable methods must be found for joining them together. Both mechanical fastening and adhesive bonding may be used to join two FRP materials. Adhesive bonding is the often preferred method because it is more economical, allows more design flexibility, and eliminates the need for holes and cutouts which may result in undesirable stress concentrations.

Wide-spread application of adhesively bonded FRP joints requires a knowledge of the response of such joints to automotive environments. Recently, Wang, Sanders, and Lindholm [1] reported data on the lap shear strength, lap shear modulus, and fatigue strength of single lap joints exposed

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to automotive-related fluids. In the present investigation further data were generated shedding light on the environmental response of adhesive bonded single lap joints. Specifically, data were obtained on a) the moisture absorption characteristics of unbonded materials and single lap joints immersed in different fluids, b) the creep behavior of such joints under environmental exposure, and c) the changes in lap shear strength due to the combined effects of load, moisture, and temperature. Tests were performed with XMC-3 bonded to SMC-R50 and with SMC-R50 bonded to SMC-R50. Brief descriptions of these materials are given in Table 1. The adhesive is specified in the next section. The following parameters were measured during the tests: a) weight change as a function of time, b) creep deformation as a function of time, and c) residual lap shear strength. The measurements were performed with test coupons (specimens) exposed to air, water, and 5% NaCl-water mixture at 23C and 93C.

The experimental procedures are described in the next section.

2. EXPERIMENTAL

Two types of adhesive bonded coupons were used in the tests, the differences in the coupons being only in their dimensions (Figure 1). The

Table 1. Material formulation.

Ingredient	Type	Weight %
	XMC-3	
Continuous Glass Fibers- ±7.5°, X-Pattern	PPG XMC Strand Type 1064	50
2154 cm Chopped Glass Fibers	PPG XMC Strand Type 1064	25
Resin	PPG Selectron RS-50335 Isophthalic Polyester	21.5
Monomer	Styrene	2.4
Thickener	PPG Selectron RS-5988	0.8
Catalyst	TBPB	0.2
	SMC-R50	
2.54 cm Chopped Glass Fibers	OCF 433AB	50
Resin	OCF-E980 Polyester	32.3
Filler	Calcium Carbonate	16.1
Mold Release	Zinc Stearate	0.8
Thickener	Magnesium Oxide	0.5
Catalyst	TBP	0.3
Inhibitor	Benzoquinone	Trace

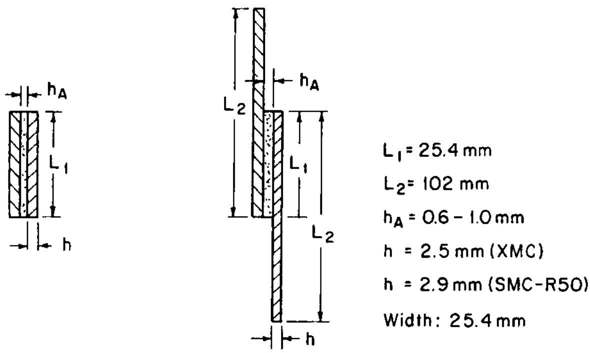

Figure 1. *Description of test coupons.*

"small" coupons consisted of two 25.4 mm wide and 25.4 mm long adherends bonded together over the entire 25.4 mm^2 surface area. The "large" coupons consisted of 25.4 mm wide and 102 mm long adherends bonded with an overlap length of 25.4 mm. The thicknesses of the adherends were nearly the same for both coupons, having the nominal values of 2.5 mm and 2.9 mm for the XMC-3 and SMC-R50, respectively. The adhesive was Goodyear Pliogrip 6040/6041, ranging in thickness from 0.6 to 1 mm. A modified isocyanate base prepolymer (Pliogrip 6040) was mixed with a polyol base curative in a 3:1 ratio by weight. Goodyear Pliogrip 6036 primer, consisting of 2.5 percent isocyanate, 2.5 percent color dye, and 95 percent methylene chloride, was wiped on the bonded areas before the adhesive was applied. Additional information regarding the properties of this adhesive may be found in Reference 1.

In addition to bonded coupons, simple unbonded (i.e. no adhesive) XMC-3 and SMC-R50 specimens were also used in the tests. The widths and lengths of these were the same as those of the "small" bonded coupons (25.4 mm).

The "small" bonded and unbonded coupons were used only for measuring moisture absorption characteristics. The "large" coupons were used in the creep tests and in the lap shear strength measurements. Weight changes of "large" coupons were also recorded after 30 days of environmental exposure. Before placing the coupons into the environmental chambers for the moisture absorption studies or before mounting them on the creep frames, the coupons were dried in an oven at 65C. During drying, the weights of the specimens were monitored. Drying was concluded when the weight loss became stabilized and no more weight loss was observed.

Moisture absorption studies were conducted by exposing the small coupons to the appropriate environmental conditions and by weighing them periodically on a Mettler Analytical Balance. The coupons were exposed to a) humid air, b) distilled water, and c) 5% NaCl-distilled water solutions. At 23C and 50 percent relative humidity the coupons were kept in an air conditioned, temperature controlled room. At 93C (air) the coupons were placed in a heated chamber with the temperature regulated within ± 1C. The moisture content of the 93C air corresponded to 50 percent relative humidity at 23C. Accordingly, at 93C the air was practically dry, the relative humidity being about 0.1 percent. The distilled water and salt water were kept in temperature controlled chambers. The temperatures of these chambers were maintained within ± 1C.

In addition to the small coupons, large coupons were also exposed to the above fluids. The weights of the large coupons were not recorded periodically, but only after 30 days' exposure.

The data are presented in terms of the percent weight change which is defined as

$$M = \frac{\textit{weight of coupon—weight of dry coupon}}{\textit{weight of dry coupon}} \times 100 \qquad (1)$$

Upon receiving the coupons, the "base line" ("as received") lap shear strength was measured with a Baldwin Universal Tester using a 5337 N/min loading rate. During the 93C tests the test section was surrounded by a heater. The baseline data thus obtained are given in Table 2.

Creep tests were performed as follows. After drying, the weight of each coupon and its dimensions were recorded. The coupon was then mounted on the creep frame. The applied load was either 10, 20, or 30 percent of the baseline strength given in Table 2. During the creep tests the coupons were exposed to the appropriate environment. For tests in 23C air at 50 percent relative humidity the creep frames were located in an air conditioned, temperature controlled room. For tests in 93C air, the coupons were surrounded with a heater. At 93C, the relative humidity of the air around the coupons was practically zero. Creep tests were also performed with coupons immersed in distilled water and in 5% NaCl-distilled water solutions. These liquids were kept in a heated container surrounding the test coupons. The temperature of the liquid was kept within ± 1C.

During creep tests the deformation of the bonded joint was measured with a modified Martens Optical Extensometer mounted at the two edges of the joints. Thus, the extensometer recorded the deformation of the bonded section of the coupon. The initial deformation on loading was recorded. The deformation as a function of time was measured over a 30 day period, or until the coupon failed, whichever came first.

Table 2. Baseline ("as received") lap shear strengths of adhesive bonded single lap joints.

	Strength (MPa)	
	23C	93C
XMC-3 to SMC-R50	6.55	3.89
SMC-R50 to SMC-R50	6.11	2.12

Coupons surviving the 30 day creep tests were weighed and their lap shear strengths (residual strengths) were measured at 23C with a Baldwin Universal Tester using a 5337 N/min loading rate. The residual strengths of large coupons immersed for 30 days in different fluids (but not loaded) were also measured.

3. RESULTS

The data on moisture absorption characteristics, creep behavior, and residual strengths are presented in this section.

3.1 Moisture Absorption Characteristics

The results of the moisture absorption tests are shown in Figures 2–5. Each point in these figures is the average of five data points. In general all five points were within ± 15 percent.

At 23C both bonded (XMC-3 to SMC-R50 and SMC-R50 to SMC-R50) and unbonded (XMC-3 and SMC-R50) coupons seemed to approach asymptotically the same maximum moisture content (Mm) when immersed in the same fluid. During the two month test period Mm was reached only in air. In water and in the 5% NaCl-water mixture the maximum moisture contents were not attained. However, the Mm values can be estimated by extrapolating the data, giving 0.18, 1.5, and 2.0 percent for air, 5% NaCl-water, and water, respectively.

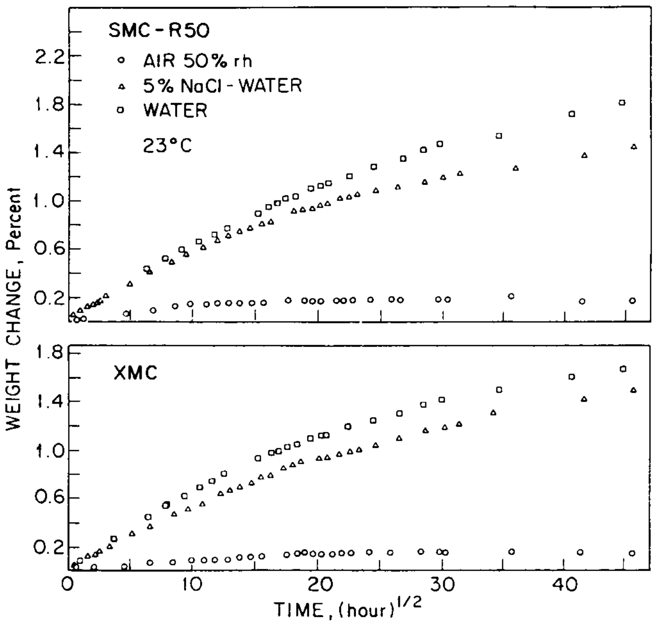

Figure 2. Weight change as a function of immersion time of XMC and SMC-R50 (no adhesive).

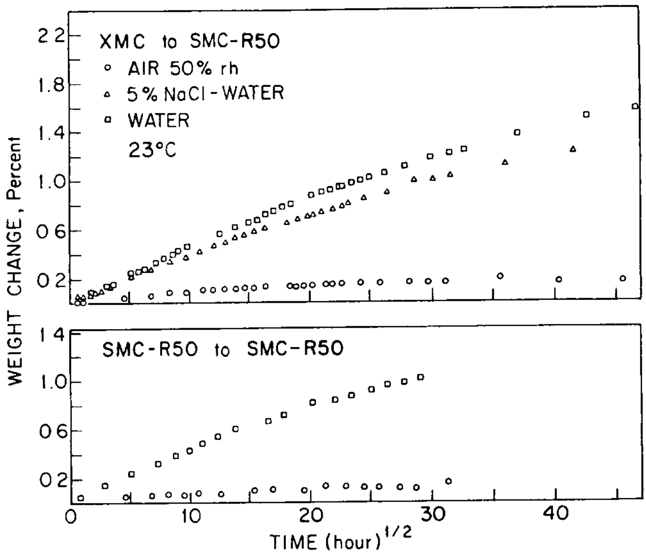

Figure 3. Weight change as a function of immersion time of adhesive bonded single lap joints.

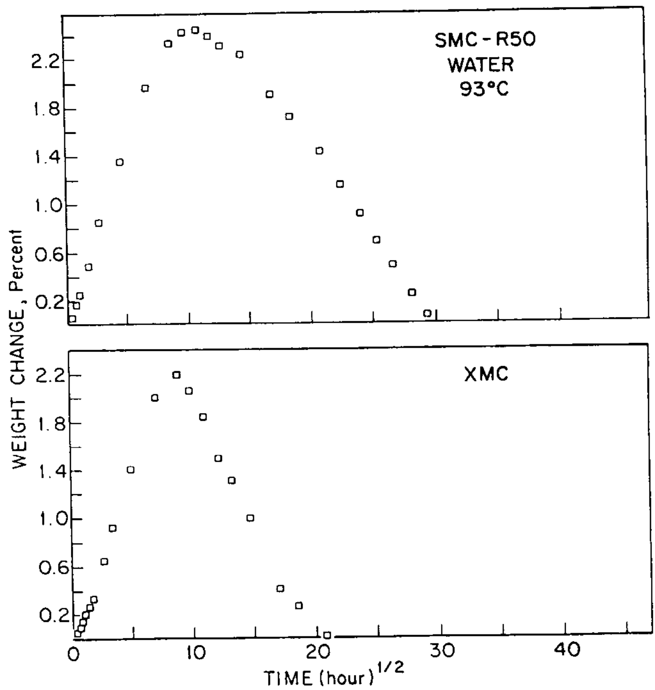

Figure 4. Weight change as a function of immersion time of XMC- and SMC-R50 (no adhesive).

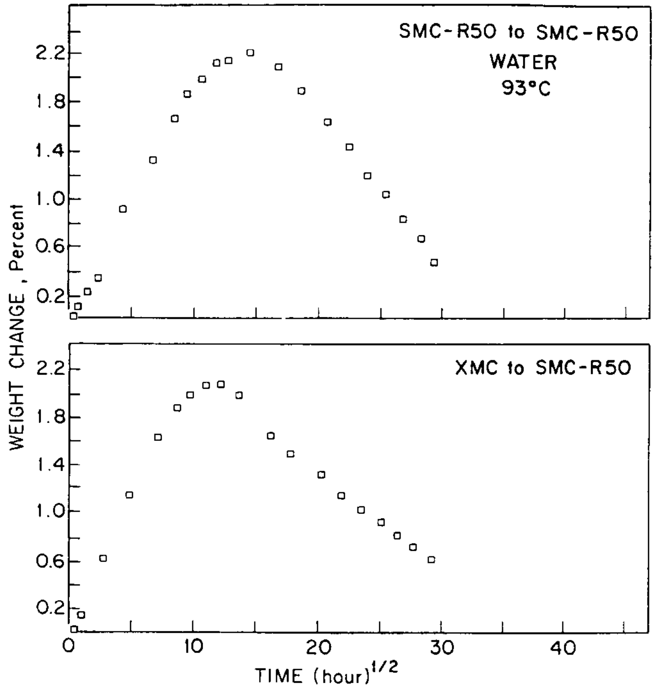

Figure 5. Weight change as a function of immersion time of adhesive bonded single lap joints.

At 93C (immersion in water) the maximum moisture levels were not approached asymptotically (Figures 4,5). Here the weight increased for about the first 100 hours, and then decreased at a rapid rate. After 20 to 30 days the weight of the coupons became actually less than the dry weight. This indicates that the material deteriorated and resin was lost during the exposure. Similar behavior has been observed previously with different types of SMC materials [2,3]. The fact that both the bonded and unbonded coupons behaved similarly at 93C suggests that the degradation was mostly in the material (XMC-3 and SMC-R50) and was not caused by the adhesive.

The *rate* of weight change depends on the thickness of the material. Although the thicknesses of unbonded XMC-3 and SMC-R50 materials were slightly different, the data in Figure 2 indicate that, under the same conditions, moisture was gained by both materials at nearly the same rates. This observation, and the fact that the maximum moisture levels were also the same, implies that the moisture transport characteristics of unbonded XMC-3 and SMC-R50 materials are similar.

The bonded coupons were thicker than the unbonded ones. Therefore, the weight changes in Figures 2 and 3 can not be compared directly.

The effects of load, fluid, and temperature on the moisture transport in adhesive bonded single lap joints are illustrated in Table 3. The results in this table again show that the types of adherends forming the bonds did not cause appreciable differences in the weight change. Furthermore, up to 20 percent (of baseline) load, the applied load did not significantly increase the moisture uptake. Even with an applied load of 30 percent above the baseline value the weight increase was small. More significant are the weight changes caused by the different types of fluids and by differences in temperature. The data in Table 3 are consistent with those in Figures 2–5. After 715 hours of exposure, at 23C the weight change is lowest in air and highest in water. At 93C the weight changes in air were small, manifesting materials degradation during the 715 hours of exposure, in agreement with the trend observed with water at 93C (Figure 5). The data for immersion of SMC-R50 to SMC-R50 bonded joints in water at 93C do not show such low values of weight change. The limited data do not offer a safisfactory explanation for this phenomenon.

3.2 Creep and Residual Strength

The creep deformations are presented in Figures 6–10. The deformations upon loading and the numerical values of the deformations over a 715 hour period are listed in Reference [4].

Table 3. The effects of load, fluid, and temperature on moisture transport in adhesive bonded single lap joints.

Fluid	Load, % of Baseline	Weight change after 715 hours, %	
		XMC-3 to SMC-R50	SMC-R50 to SMC-R50
Air, 23C	0	0.24	0.196
	10	0.24	0.165
	20	0.23	0.163
	30	0.30	0.167
Air, 93C	0	0.029	0.045
	10	−0.039	0.003
	20	−0.064	−0.031
	30	0.006	−0.040
Water, 23C	0	1.38	1.25
	10	1.50	1.30
	20	1.45	1.27
	30	1.44	1.35
Water, 93C	0	−0.014	1.85
	10	N.S.	1.93
	20	N.S.	N.S.
	30	N.S.	N.S.
5% NaCl-Water, 23C	0	1.13	—
	10	1.46	—
	30	1.40	—

N.S. No coupon survived 715 hours.

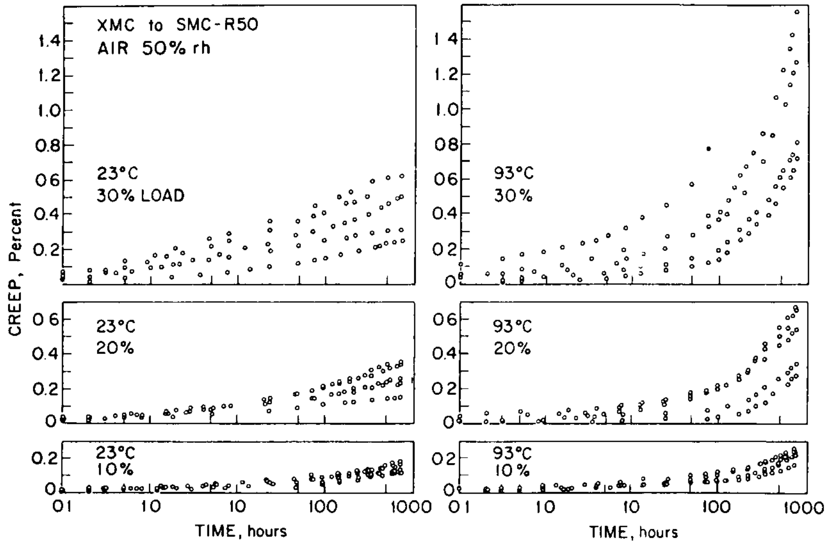

Figure 6.* *Creep deformation as a function of time. Load levels indicated are percent of baseline value. Solid circle indicates test coupon failed.

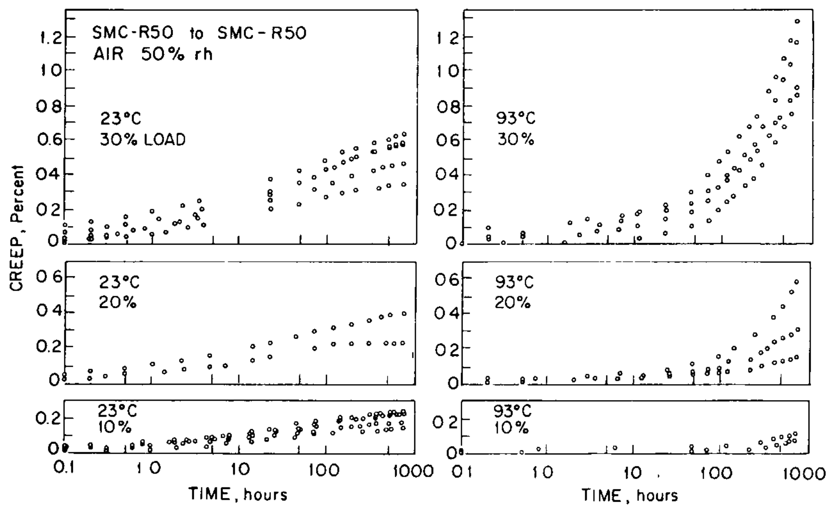

Figure 7.* *Creep deformation as a function of time. Load levels indicated as percent of baseline value.

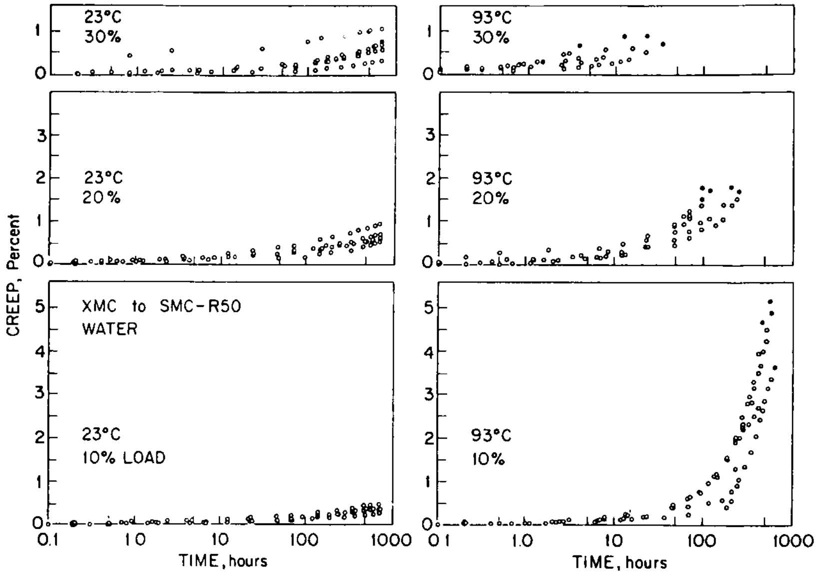

Figure 8. *Creep deformation as a function of time. Load levels indicated are percent of baseline value. Solid circles indicate test coupon failed.*

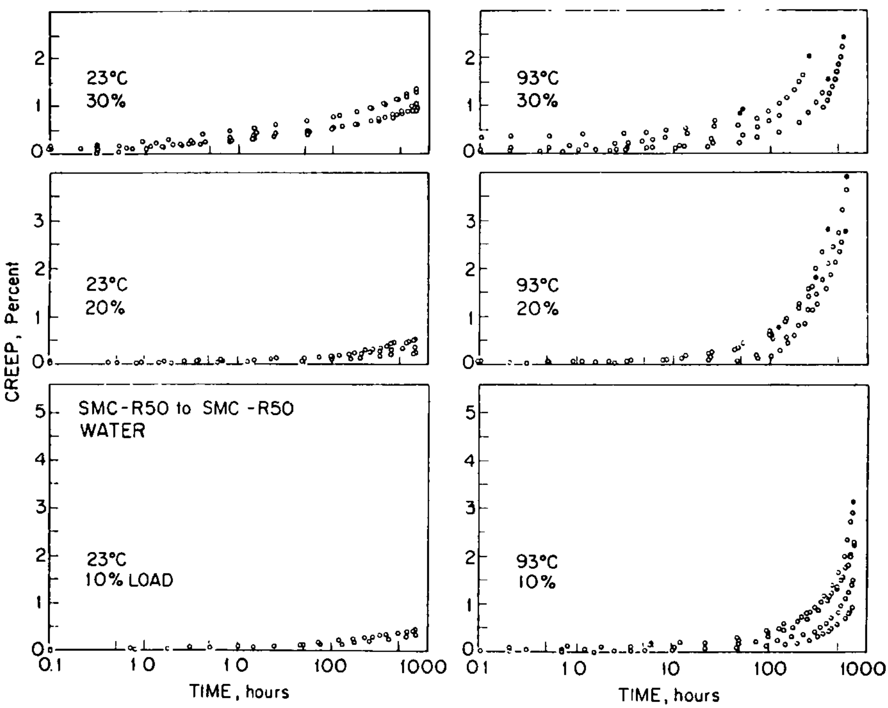

Figure 9. *Creep deformation as a function of time. Load levels indicated are percent of baseline value. Solid circles indicate test coupon failed.*

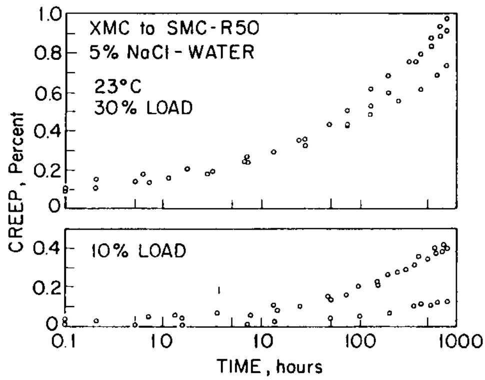

Figure 10. *Creep deformation as a function of time. Load levels indicated are percent of baseline value.*

Figures 6–10 illustrate the effects of material, fluid, temperature, and applied load on the creep behavior. The type of adherends used in forming the bonded joints (XMC-3 to SMC-R50 or SMC-R50 to SMC-R50) did not have a significant influence on the creep. The type of fluid, the temperature, and the applied load strongly affected the creep. The creep was lowest in air, and was higher in water and in salt water. The creep also increased with temperature and with applied load. At 23C none of the joints separated in any of the fluids. At 93C only one of the joints separated. This occurred at a 30 percent load level. However, during water immersion (93C) all but 3 joints separated before the end of 715 hours. The smallest deformation at which separation occurred was about 0.8 percent.

The results in Figures 6–10 also show that there was a sharp increase in the deformation rate after about 100 hours. The increase occurred somewhat sooner at higher loads.

The changes in lap shear strength due to load and environmental exposure are summarized in Table 4 and in Figures 11 and 12. The lap shear strength decreased appreciably (by about 50%) only in water and in 5% NaCl-water at 93C. At all other conditions the lap shear strength was unchanged, or even increased slightly. Improvements in lap shear strength of SMC materials exposed to various fluids were also observed by Wang et. al [1]. They attributed the beneficial effects of fluids to plasticization.

An inspection of the coupons revealed that the joints separated either due to delamination of the composite, due to separation along the interface, or due to a combination of both (Table 5). Generally, delamination was the major cause of separation when the coupons were exposed to 23C and 93C humid air. At 23C delamination was also the cause of separation for coupons immersed in water and in 5% NaCl-water. At 93C the separation along the interface occurred first when the coupons were immersed in water and in 5% NaCl-water.

Table 4. Changes in lap shear strengths of loaded and unloaded, adhesive bonded single lap joints after 715 hours of environmental exposures. Data shown are the ratio of the average residual strength (S) to the baseline strength (S_B) given in Table 2.

Fluid	Load % of Baseline	Residual Strength, S/S_B	
		XMC-3 to SMC-R50	SMC-R50 to SMC-R50
Air, 23C	0	0.99	1.03
	10	1.16	0.98
	20	1.00	1.00
	30	1.17	1.02
Air, 93C	0	1.20	0.92
	10	1.13	1.21
	20	1.11	1.20
	30	1.06	1.09
Water, 23C	0	1.20	1.17
	10	1.11	1.18
	20	1.09	1.02
	30	1.06	1.07
Water, 93C	0	0.64	0.51
	10	N.S.	0.40
	20	N.S.	N.S.
	30	N.S.	N.S.
5% NaCl Water, 23C	0	1.20	—
	10	1.0	—
	30	0.96	—
5% NaCl-Water, 93C	0	0.69	—

N.S. No coupon survived 715 hours.

4. SUMMARY

The following general conclusions can be reached on the basis of the data generated in this program.

1. The moisture absorption characteristics of XMC-3 and SMC-R50 materials are nearly the same.
2. At 23C both bonded joints and the unbonded coupons asymptotically approached maximum saturation levels, the values of these levels being 0.18, 1.6, and 2.0 percent, respectively for air (50% r.h.), water, and 5% NaCl-water.
3. At 93C the weight changes of both bonded joints and unbonded coupons increased and were followed by a decrease, indicating material degradation and subsequent material loss.

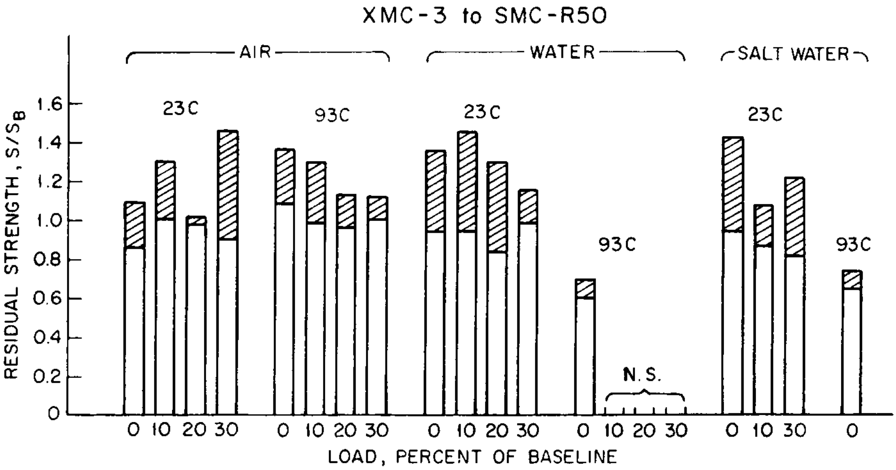

Figure 11. The residual strength (S) of loaded and unloaded, adhesive bonded single lap joints after 715 hours of environmental exposures. S_B is the baseline strength given in Table 2. Shaded areas represent spread in data. N.S. indicates no coupon survived 715 hours.

4. The applied load had only a small and in most cases negligible effect upon the weight changes of adhesive bonded single lap joints.
5. The creep deformation of adhesive bonded single lap joints was influenced by the applied load, the fluid, and the temperature, but was independent of the adherends used in forming the joints.
6. The rate of deformation increased significantly after 100 hours.
7. The coupons survived 715 hours of creep testing under all conditions,

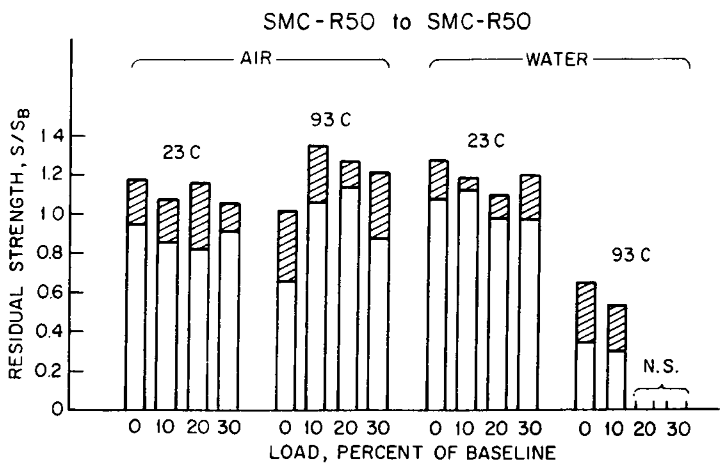

Figure 12. The residual strength (S) of loaded and unloaded, adhesive bonded single lap joints after 715 hours of environmental exposures. S_B is the baseline strength given in Table 2. Shaded areas represent spread in data. N.S. indicates no coupon survived 715 hours.

Table 5. Separation modes of adhesive bonded single lap joints at various test conditions.

Fluid	Load % of Baseline	Separation Mode: XMC-3 to SMC-R50	Separation Mode: SMC-R50 to SMC-R50
Air, 23C	0 to 30	delamination	delamination
Air, 93C	0 to 30	delamination	delamination
Water, 23C	0 to 30	delamination	delamination and adhesive
Water, 93C	0	delamination and adhesive	delamination and adhesive
Water, 93C	10–30	adhesive	adhesive
5% NaCl Water, 23C	0–30	delamination	—
5% NaCl Water, 93C	0	delamination and adhesive	—

except in water at 93C, where nearly all joints separated before the end of the 715 hours creep test. The lowest deformation at which separation occurred was about 0.8 percent.

8. The lap shear strength of the adhesive bonded single lap joints did not change appreciably during the 715 hours of load and environmental exposure, except in 93C water and in 93C 5% NaCl-water mixture. Under these two conditions the lap shear strength decreased by about 50 percent.
9. In general, joints separated due to delamination when they were exposed to humid air at 23C and 93C or immersed in water and in 5% NaCl-water at 23C. Separation occurred along the interface for coupons immersed in 93C water and in 93C NaCl-water.

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