The Effects of Temperature, Humidity and Aircraft Fluid Exposure on T800H/3900-2 Composites Bonded with AF-555M Adhesive *

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ABSTRACT

Fiber reinforced resin matrix composites and structural adhesives have found increased usage on commercial and military aircraft in recent years. Due to the lack of service history of these relatively new material systems, their long-term aging performance has not been well established. In this study, single lap shear specimens (SLS) were fabricated by secondary bonding of Scotch-Weld™ AF-555M between pre-cured adherends comprised of T800H/3900-2 uni-directional laminates. The adherends were co-cured with wet peel-ply for surface preparation. Each bond-line of the SLS specimen was measured to determine thickness and inspected visually using an optical microscope for voids. A three-year environmental aging plan for the SLS specimens at 82°C (180°F) and 85% relative humidity was initiated. SLS strengths were measured for both controls and aged specimens at room temperature and 82°C. The effect of this exposure on lap shear strength and failure modes to date is reported.

In addition, the effects of water, saline water, deicing fluid, JP-5 jet fuel and hydraulic fluid on both the composite material and the adhesive bonds were investigated. The up to date results on the effects of these exposures will be discussed.

1. INTRODUCTION

Fiber reinforced resin matrix composites and structural adhesives have been introduced increasingly in recent years for structural applications on military and commercial aircraft (e.g. A380, B787, YF-22). These materials offer advantages in weight savings without sacrificing strength and mechanical performance. Due to the lack of service history of these relatively new material systems, their long-term aging performance has not been well established. As part of a program to address this unknown behavior, an Aviation Safety Program (ASP) was initiated under NASA's Aeronautics Research Mission Directorate (ARMD) in 2007. As a part of ASP, the Aircraft Aging and Durability Project (AADP) was formulated to characterize, predict and develop mitigation approaches to minimize damage and degradation issues associated with aging

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effects on composites and adhesives. The focus of the AADP is aging and damage processes in "young" aircraft, rather than life extension of legacy vehicles, thus the emphasis of research is on the behavior of new composite and adhesive systems.

Previous work has demonstrated that high quality joints were bonded using a peel-ply surface treatment that serves to provide increasing surface roughness and prevents mould release agents and other materials from contaminating the surface [1-3]. In this approach, the peel-ply was co-cured as part of the top laminate ply and subsequently removed just prior to bonding. It was easily released by peeling off the adherend surface because of the non-stick nature of the carrier fabric substrate (typically a polyester or polyamide). Increasing surface roughness resulted in an increase in surface area, which allowed the adhesive to flow in and around the irregularities on the surface to form a mechanical interlocking bond. In the present study for an AADP subtask entitled "Bonded Joints for Construction and Repair", the bonding surfaces were prepared by co-curing Toray T800H/3900-2 composite prepregs with a resin impregnated wet peel-ply. Single-lap-shear (SLS) specimens were bonded using AF-555M adhesive. Processing pitfalls for producing porosity-free, high quality bonds have been reported previously [4]. The behavior of on-going aging of SLS specimens in a temperature/humidity chamber is reported herein.

Aircraft are exposed to many fluids, such as: rain, salt spray, fuel, deicing fluid and hydraulic fluid, while sitting on the tarmac and during normal maintenance operations. Environmental and chemical degradation of carbon- and glass-reinforced polymeric composites have been reported [5-7]; polymer degradation has been shown to affect flammability properties and increase the risk of pyrolysis [8]. Thus, while investigating the effects of elevated temperature and humidity, it was decided to expand the project to include the effects of exposures of the composite material and the adhesive bonds to these fluids. The expanded project involves soaking (immersion) composite and lap shear specimens in these fluids for times ranging from a few hours to 1000 hours. This report addresses the results from short exposures of these fluids on the Toray T800H/39900-2 composite and Scotch-Weld adhesive by measuring changes to the glass transition temperature (T_g) and SLS bond strength.

2. MATERIALS[†]

The materials used in this study are presented in Tables 1 and 2. Specifications of the materials in Table 1 have been reported previously [4]. All materials for the solvent study were used as received without further treatments with the exception of the saline water. Saline water was prepared by mixing 3.5 grams of Morton Sea Salt with 96.5 grams of deionized water.

[†] Use of trade names or manufacturers does not constitute an official endorsement, either expressed or implied, by the National Aeronautics and Space Administration.

Table 1. Materials used in Environmental and Solvent Study

Material Form	Material Designation	Supplier
Prepreg	T800H/3900-2	Toray
Adhesive	S/W AF-555M	3M
Wet peel ply	Hysol EA-9895	Henkel

Table 2. Solvents used in Solvent Study

Solvent	Manufacturer
Deionized Water	Milli-Q filtered water
Saline Water	3.5% (w/w) Morton Sea Salt in deionized water
Deicing Fluid	AVL-TKS
JP-5 Jet Fuel	Chevron Phillips Chemical Company
Hydraulic Fluid	Skydrol

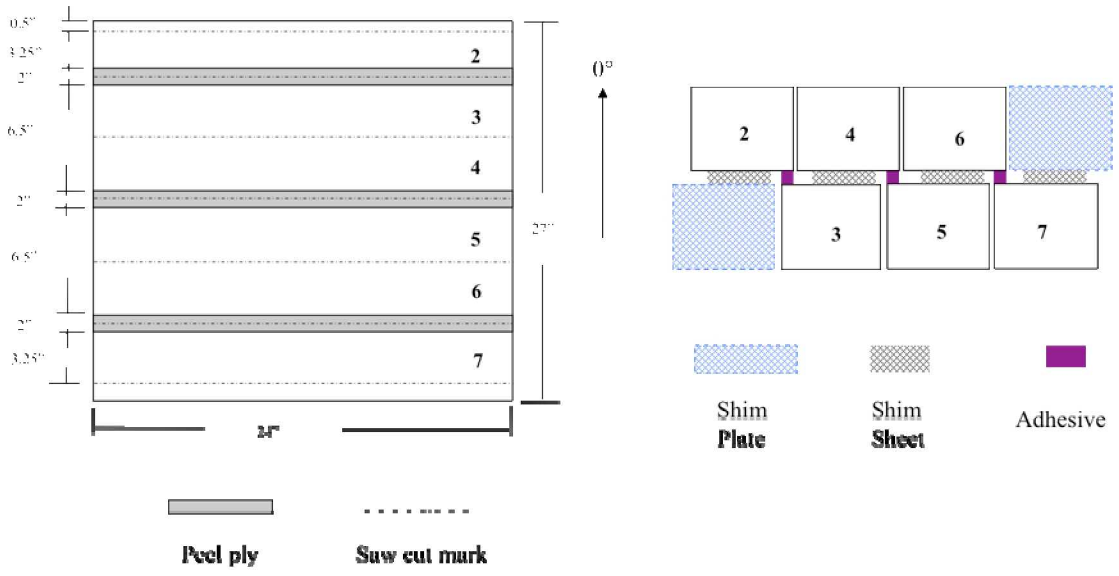
3. EXPERIMENTAL

3.1 Fabrication of composite adherends

The composite adherend panel, 61 cm by 81 cm (24” by 32”) - $[0]_{16}$, was assembled by stacking 16 plies of uni-directional T800H/3900-2 prepreg. A wet peel-ply strip of 61 cm by 5.1 cm (24” by 2”) was laid down on the top prepreg layer, perpendicular to the fiber direction and separated by a pre-determined distance as shown in Figure 1a. This assembly was cured at 177°C (350°F) for 2 hours under 690 KPa (100 psi) in an autoclave with vacuum bagging.

3.2 Bonding of adherends

Peel-ply co-cured composite adherend panels were then cut into six 61 cm by 10.8 cm (24” by 4.25”) strips along the center of the 5.08 cm (2”) wide peel-ply as indicated in Figure 1a. The peel plies were then removed to expose the co-cured surfaces. Peel-ply surfaces were used as-peeled without any additional chemical cleaning or wiping. The surfaces were visually inspected



(a) (b)

Figure 1. Schematic drawing illustrates lay-up, co-curing, trimming (Figure 1a, top view) and secondary bonding of composite/peel-ply/adhesive (Figure 1b, side view) in fabricating single-lap-shear specimens.

for residual fiber debris after the peel-plies were removed. The surface characteristics have been reported previously [4]. A 61 cm by 1.27 cm (24" by 0.5") strip of AF-555M adhesive was subsequently laid down on each exposed surface. A roller was used to help remove any trapped air by rolling and pressing down the adhesive onto the surface. The adhesive was then sandwiched by laying down the second half of the adherend panel. Shims were used to align and control the final bond-line thickness. The six-strip assembly (see Figure 1b) was then bagged and cured in an autoclave at 177°C (350°F) for 2 hours under 310 KPa (45 psi).

3.3 Preparation of the single-lap-shear (SLS) specimens

Co-cured strips measuring 61 cm by 12.7 cm (24" by 8") were cut into 2.5 cm by 12.7 cm (1" by 8") SLS specimens according to ASTM D1002-99 specifications [9]. Each bonded strip yielded 22 specimens, which were numbered as shown in Figure 2. Each specimen was given a unique identification based on Panel ID, Strips ID and Specimen number. In this way, the origin of each specimen can be traced back to the starting materials. A total of 66 specimens were fabricated from a single autoclave run.

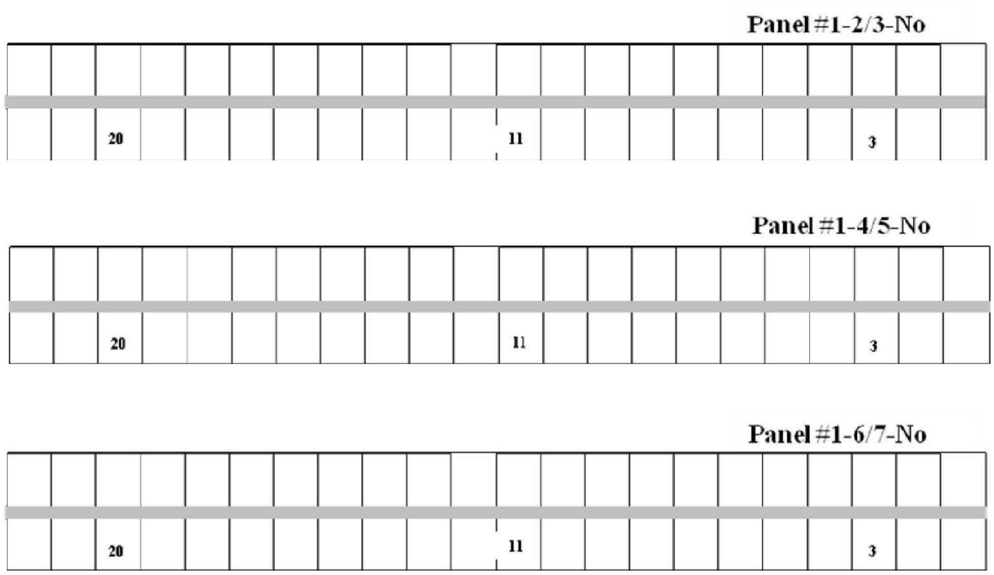


Figure 2. Schematic drawing (top view) illustrates identification of single-lap-shear specimens. Panel ID is #1, bonded strip pair IDs are 2/3, 4/5 and 6/7 (see Figure 1), and specimen IDs for each bonded strip pair is numbered 1 to 22 from right to left.

3.4 SLS strength measurement

SLS strengths were measured by a MTS test frame. Details of the testing have been reported previously [4]. Failure modes were characterized according to ASTM D5573 [10].

3.5 Aging chamber

The environmental chamber used was a MicroClimate™ model MCB-1.2 manufactured by CSZ (Cincinnati Environment Chamber). This chamber has a standard temperature range of -73°C to 190°C (-100°F to 375°F) and a maximum relative humidity (RH) of 95%. The aging condition selected in this study was 82°C (180°F)/85% RH [11].

3.6 3-point Bend Specimens

The composite panel, 30.5 cm by 30.5 cm (12" by 12") - $[0]_{16}$, was assembled by stacking up 16 plies of uni-directional T800H/3900-2 prepreg and cured by vacuum press using the parameters presented in 3.1 above. The panel was cut into 60 mm X 10 mm 3-point bend specimens. Half of the specimens had a graphite fiber orientation of 0° (carbon fiber running parallel to the 60 mm dimension) and the other half had an orientation of 90° (carbon fiber running perpendicular to the 60 mm dimension). The thickness of the specimens averaged 2.70 mm.

3.7 Solvent Soak

Each specimen was placed in an individual test tube and covered with solvent and soaked for 6 hours using the soaking procedure described in ASTM Standard D 896-04 [12].

3.8 Dynamic Mechanical Analyzer Testing

The glass transition temperature (T_g) was measured using a TA Instruments Dynamic Mechanical Analyzer (DMA), Model 2980DMA and the 3-point bend accessory with operating conditions of 1Hz constant frequency, amplitude of 10 microns and ramp rate of 5 °C per minute from -50 to 250 °C. The viscoelastic parameter, Tan Delta, was used to define the T_g.

4. RESULTS And DISCUSSION

Bond-line characteristics and thickness of each specimen were documented prior to aging or testing. Due to the specimen size and limitations with our equipment, ultrasonic characterization of the bond quality was not feasible. Thus inspection of the bond quality was assessed qualitatively by examining the bond line edges under an optical microscope. A typical specimen with voids present in the bond-line is shown in Figure 3. One side of the bond-line exhibited no porosity, while the other side showed visible voids. For this study, each humidity aging time exposure test contained a mixture of specimens consisting of some that were void-free and some containing voids.

Specimens numbered 3, 11, and 20 in each bonded strip (see Figure 2) were selected to be tested at room temperature (RT) as controls before aging. Results for Panel #2 are shown in Table 3. Specimens from Strips 1 and 2 were void-free and exhibited SLS strengths which were consistently 30% above the nominal value of 35.9 MPa (5,200 psi) reported by the manufacturer [13]; while Strips 3 and 4, and 6 and 7 yielded void-containing specimens, with strength values that were 85 to 95% of this nominal value. Results from another panel (Panel #4), which yielded void-free specimens are presented in Table 4. The SLS strengths measured were consistently over 10% of the nominal strength value.

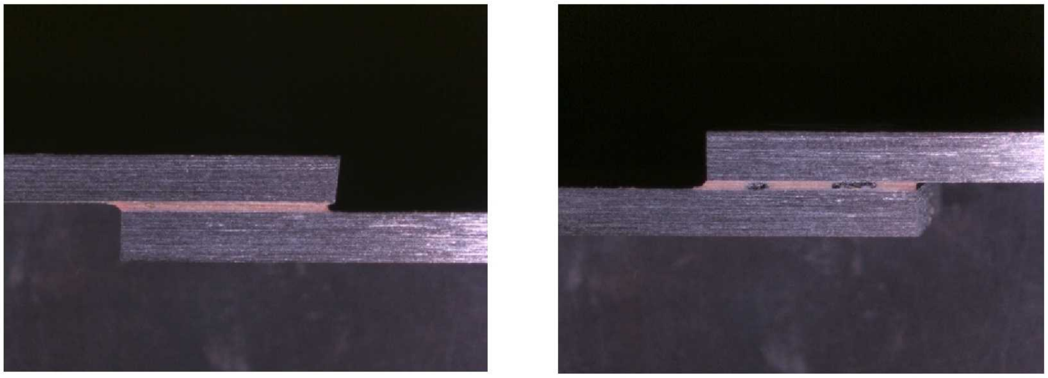


Figure 3. Visual evidence of porosity in the bond-line. Adhesive is sandwiched between top and bottom composite adherends.

It was noted that Panel #4 yielded uniform bond-line thicknesses, while Panel #2 did not. The origin of bond-line variations came from the thickness variations in the bonding adherend strips. In an autoclave run, three sets of adherend strips were bonded using a single large caul plate, which made the control of bond-line uniformity difficult.

Table 3. RT strength values for Panel #2 control specimens

Strip ID	Specimen ID	Bond-line thickness, mil	SLS strength, MPa	% of nominal strength**
1/2	3	1	46.8	130
	11	1	46.6	130
	20	1	47.6	133
3/4	3	9	41.5	116
	11*	13	30.5	85
	20*	10	34.0	95
6/7	3*	10	33.0	92
	11*	7	32.8	91
	20*	8	30.2	84
Avg.			38.1 ± 7.4	106

*Specimens with visible voids on the bond-line edge.
**Nominal strength 35.9 MPa (5,200 psi) from the supplier

4.1 Aging plan

The 3-year aging plan is presented in Table 5. Specimens were selected according to the measured RT strengths of controls from each bonded adherend strip. Bond-lines were photographed and characterized by 3 categories: Void-free, those containing 1-2 visible voids and those containing 3-5 visible voids. It is important to note that the voids are only visible along the bond edges. Specimens were too small for ultrasonic inspection of voids within non-visible bonded area. Thus it was not possible to assess overall bond quality for each specimen. At least five specimens in each aging group (i.e. exposure time) were randomly selected from these three categories and were also selected such that they were not derived from a single co-cured panel. Specimens were aged at 82°C (180°F) and 85% RH with pre-determined aging times. Prior to placing the specimens in the aging chamber, they were labeled, baked-out at 66°C (150°F) for 1 hour to eliminate moisture, and weighed. Control specimens were wrapped in a plastic bag and "aged" in desiccators at RT. A total of 160 specimens were required to conduct this study. Tests of control and aged specimens were conducted at RT and 82°C (180°F). The test intervals were more frequent in the first year. Specimen weights after aging were recorded and fracture surfaces were examined after testing.

Table 4. RT strength values for Panel #4 control specimens

Strip ID	Specimen ID	Bond-line thickness, mil	SLS strength, MPa	% of nominal strength*
2/3	3	1	39.7	111
	11	1	40.7	133
	20	1	35.9	100
4/5	3	1	42.1	117
	11	1	45.6	127
	20	1	44.3	123
6/7	3	1	39.5	110
	11	1	44.4	124
	20	1	39.8	111
Avg.			41.3 ± 3.0	115

*Based on nominal strength 35.9 MPa (5,200 psi) from the supplier

Table 5. Planned three-year aging schedule in 82°C/85% RH chambers and completed tests

Year	Planned aging time, days	Completed aging time, days	No. specimens	No. controls	No. specimens tested at RT	No. specimens tested at 82°C
1	0	0	0	10	5	5
	30	35	12	0	6	6
	90	95	12	0	6	6
	180	125	12	10	11	11
	270	185*	12	0	6	6
	360		12	10	11	11
2	540		12	10	11	11
	720		12	10	11	11
3	900		12	10	11	11
	1080		12	10	11	11
Total			108	70	89	89

* Specimens were not included in this report because the environmental chamber failed and the actual number of days of exposure is unknown

4.2 Results of control specimens "aged" in desiccators

The proposed three-year aging plan was revised after viewing the 95-day specimens results. Because the average strengths for the 35-day and 95-day specimens were substantially reduced compared to the controls, the plan was modified and the remaining specimens were removed from the environmental chambers and tested on a monthly basis.

Results of control specimens aged up to 125 days in desiccators are tabulated in Table 6. Specimen grouping for each aging interval consisted of 6 specimens that were a mixture of void-free and specimens with voids. Strengths at RT and 82°C (180°F) for each aging time were measured and average strengths and strength retentions calculated. Elevated temperature (82°C) average strengths are provided in Tables 6 and 7 for completeness of the study; however, only the RT nominal strength was provided by the supplier. Therefore, all comparisons are based on this value.

Table 6. Strengths of control specimens aged in desiccators at RT

Specimen ID	No. of voids in bone-line	Bond-line thickness, cm (mil)	Days aged	Test temp, °C	SLS strength, MPa	Failure mode**	Avg. strength, MPa	% RT strength retention*
A0-1	0	0.018 (7)	0	RT	37.3	80 CF, 20 TLC	37.8 ± 2.7	105
A0-2	1-2	0.028 (11)			33.8	80 CF, 20 TLC		
A0-3	3-5	0.025 (10)			38.6	95 CF, 5 TLC		
A0-4	3-5	0.043 (17)			38.0	95 CF, 5 TLC		
A0-5	3-5	0.020 (8)			41.4	95 CF, 5 TLC		
A0-6	0	0.031 (12)	0	82	31.1	50 CF, 50 TLC	30.4 ± 2.0	85
A0-7	1-2	0.038 (15)			33.1	60 CF, 40 TLC		
A0-8	1-2	0.010 (4)			30.4	80 TLC, 20 CF		
A0-9	3-5	0.028 (11)			27.6	70 TLC, 30 CF		
A0-10	3-5	0.008 (3)			29.7	80 TLC, 20 CF		
A180-1	0	0.031 (12)	125	RT	41.1	85 CF, 10 TLC, 5 LFT	36.6 ± 6.7	102
A180-2	1-2	0.033 (13)			42.0	90 CF, 5 TLC, 5 LFT		
A180-3	1-2	0.025 (10)			30.4	70 CF, 20 TLC, 10 AS		
A180-4	3-5	0.025 (10)			28.3	70 CF, 20 AS, 10 TLC		
A180-5	3-5	0.028 (11)			41.1	90 CF, 5 TLC, 5 LFT		
A180-6	0	0.031 (12)	125	82	31.1	60 TLC, 40 CF	31.8 ± 0.8	88
A180-7	1-2	0.031 (12)			32.4	60 TLC, 40 CF		
A180-8	1-2	0.041 (16)			31.1	75 CF, 15 TLC, 10 AS		
A180-9	3-5	0.038 (15)			32.4	85 CF, 15 TLC		
A180-10	3-5	0.025 (10)			n/a	60 TLC, 40 CF		

*Based on nominal strength 35.9 MPa (5,200 psi) from the supplier

**Failure modes: CF – cohesive, TLC – thin layer cohesive, LFT – light fiber tear, FT – fiber tear, AS – adhesive starvation. Numerical values depict percentages.

Table 7. Strengths of control specimens aged in desiccators at RT

Specimen ID	Days aged in desiccators	Bond-line characteristics	Test temp, °C	Avg. strength, MPa	% Strength retention*
A0	0	Void-free	RT	37.3 ± 0	104
			82	31.1 ± 0	87
A180	125		RT	41.1 ± 0	114
			82	31.1 ± 0	87
A0	0	Void-containing	RT	37.9 ± 3.0	106
			82	30.2 ± 2.3	84
A180	125		RT	35.5 ± 7.1	99
			82	32.0 ± 0.8	89

*Based on nominal strength 35.9 MPa (5,200 psi) from the supplier

As previously observed, SLS strength values were independent of bond-line thickness. These results were further re-grouped for clarity by specimens with and without voids in Table 7. Void-free fresh specimens (i.e., specimens A0) exhibited RT average strength of 37.3 MPa (5,459 psi), which was 104% of the nominal 35.9 MPa (5,200 psi) from the supplier. An average strength of 30.2 MPa (4,380 psi) was measured at 82°C (180°F) and represented 84% retention of the RT value. Void-free specimens aged in the desiccators for 4.2 months (125 days) yielded an average strength of 41.1 MPa (5,954 psi) and 31.1 MPa (4,505 psi) at RT and 82°C (180°F), and represented strength retentions of 114% and 87%, respectively. As expected, RT aging in desiccators had no apparent effect on void-free specimens as both strength and strength retention at RT and 82°C (180°F) were unchanged.

Void-containing specimens aged in desiccators behaved similarly to those void-free specimens reported above (see Figure 6). Apparently the presence of voids along the bond-line edge was not an effective discriminator in this aging condition. It was also noted that the standard deviations were large in all cases for reasons that were not clear; +/-10% was a reasonable deviation for the adhesively bonded specimens

4.3 Results of strength retention for specimens aged in humidity chambers

Results of specimens aged at 82°C/85% RH up to 4.2 months (125 days) are tabulated in Table 8. Typically the specimens exhibited an increase in weight of about 1% due to moisture uptake.

Table 8. Strengths of specimens aged in humidity chambers

Specimen ID	No. of voids in bone-line	Bond-line thickness, cm (mil)	Days aged	Test temp, °C	SLS strength, MPa	Failure mode**	Avg. strength, MPa	% RT strength retention*
A30-1	0	0.018 (7)	35	RT	47.2	95 CF, 5 TLC	37.5 ± 6.3	104
A30-2	0	0.003 (1)			37.7	70 CF, 30 TLC		
A30-3	0	0.031 (12)			38.3	95 CF, 5 LFT		
A30-4	1-2	0.031 (12)			34.1	95 CF, 5 TLC		
A30-5	3-5	0.031 (12)			30.3	75 CF, 25 TLC		
A30-6	0	0.010 (4)	35	82	27.8	80 CF, 20 TLC	26.8 ± 5.2	75
A30-7	0	0.018 (7)			30.8	75 CF, 25 TLC		
A30-8	0	0.038 (15)			32.1	75 CF, 25 TLC		
A30-9	1-2	0.028 (11)			24.2	70 CF, 15 TLC, 15 AS		
A30-10	3-5	0.013 (5)			19.3	75 AS, 15 CF, 15 TLC		
A90-1	0	0.033 (13)	95	RT	36.8	55 CF, 40 LFT, 5 AS	30.7 ± 5.4	86
A90-2	0	0.020 (8)			35.6	70 CF, 20 TLC, 10 LFT		
A90-3	0	0.031 (12)			24.5	90 LFT, 10 FT		
A90-4	1-2	0.010 (4)			30.3	60 TLC, 40 CF		
A90-5	3-5	0.028 (11)			26.5	60 LFT, 40 CF		
A90-6	0	0.033 (13)	95	82	24.9	60 TLC, 40 CF	23.7 ± 2.9	66
A90-7	0	0.003 (1)			23.9	80 TLC, 20 CF		
A90-8	0	0.046 (18)			23.4	65 TLC, 35 CF		
A90-9	1-2	0.023 (9)			19.3	65 TLC, 25 CF, 10 AS		
A90-10	3-5	0.020 (8)			27.3	60 CF, 40 TLC		
A180-1	0	0.038 (15)	125	RT	28.8	85 CF, 15 FT	29.6 ± 4.3	82
A180-2	0	0.003 (1)			37.0	50 CF, 50 TLC		
A180-3	0	0.031 (12)			29.8	90 CF, 5 TLC, 5 FT		
A180-4	3-5	0.028 (11)			28.5	95 CF, 5 FT		
A180-5	3-5	0.025 (10)			30.0	70 CF, 30 TLC		
A180-6	0	0.043 (17)	125	82	23.7	50 CF, 50 TLC	23.4 ± 1.5	65
A180-7	0	0.010 (4)			25.2	65 TLC, 35 CF		
A180-8	0	0.031 (12)			22.9	65 TLC, 35 CF		
A180-9	1-2	0.020 (8)			24.9	60 TLC, 40 CF		
A180-10	3-5	0.036 (14)			22.3	40 CF, 40 TLC, 20 AS		

*Based on nominal strength 35.9 MPa (5,200 psi) from the supplier

**Failure modes: CF – cohesive, TLC – thin layer cohesive, LFT – light fiber tear, FT – fiber tear, AS – adhesive starvation

Specimen groupings for each aging interval consisted of 5 specimens that were a mixture of void-free and specimens with visible voids along the bond-line edges. Strengths at RT and 82°C (180°F) for each aging time were measured and the average strength and strength retention values were calculated. These results were further re-grouped for clarity by specimens with and without voids, with the results presented in Table 9.

Table 9. Avg. strengths of specimens aged in humidity chambers: void-free vs. void-containing

Specimen ID	Days aged	Bond-line characteristics	Test temp, °C	Avg. strength, MPa	% Strength retention*
A30	35	Void-free	RT	41.0 ± 5.3	114
			82	30.2 ± 2.2	84
A90	95		RT	36.2 ± 0.8	101
			82	24.1 ± 0.8	67
A180	125		RT	31.9 ± 4.5	89
			82	23.9 ± 1.1	67
A30	35	Void-containing	RT	32.2 ± 2.6	90
			82	21.8 ± 3.4	61
A90	95		RT	28.4 ± 2.7	79
			82	23.3 ± 5.6	65
A180	125		RT	29.3 ± 1.1	82
			82	23.6 ± 1.8	66

*Based on nominal strength 35.9 MPa (5,200 psi) from the supplier

Percent strength retention measured at RT in Table 9 was plotted in Figure 4 for comparison between void-free and void-containing specimens. Strength retention for the void-free specimens was clearly better than those containing voids. Aged void-free specimens exhibited 100% strength retention up to 3.2 months (95 days) in the humidity chambers and 90% strength retention after 4.2 months (125 days). On the other hand, the void-containing specimens exhibited 90% strength retention after only 1.2 months (35 days) of aging; then rapidly dropped to 80% retention afterwards.

Percent strength retention measured at 82°C (180°F) in Table 9 was plotted in Figure 5. In this case, strength retention for the void-free specimens is only marginally better than for those containing voids. Aged void-free specimens maintained 85% strength retention up to 1.2 months (35 days), and then began to diminish. For the specimens containing voids, the strength retention was noted to drop immediately and reached 60% level at 1.2 months (35 days) of aging. The void-containing specimens were more susceptible to moisture ingression and consequently began to degrade quicker. After 35 days of aging, strength retention for both the void-free and void-containing specimens remained at ~65% up to 4.2 months (125 days) of aging.

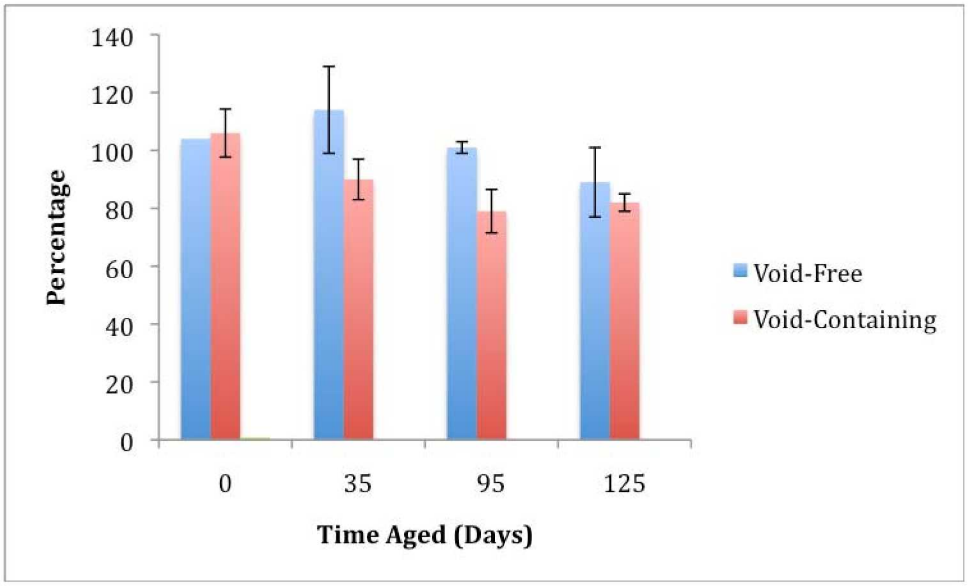


Figure 4. Percent strength retention measured at RT for void-free vs. void-containing specimens aged in 82°C/85% RH chambers up to 125 days.

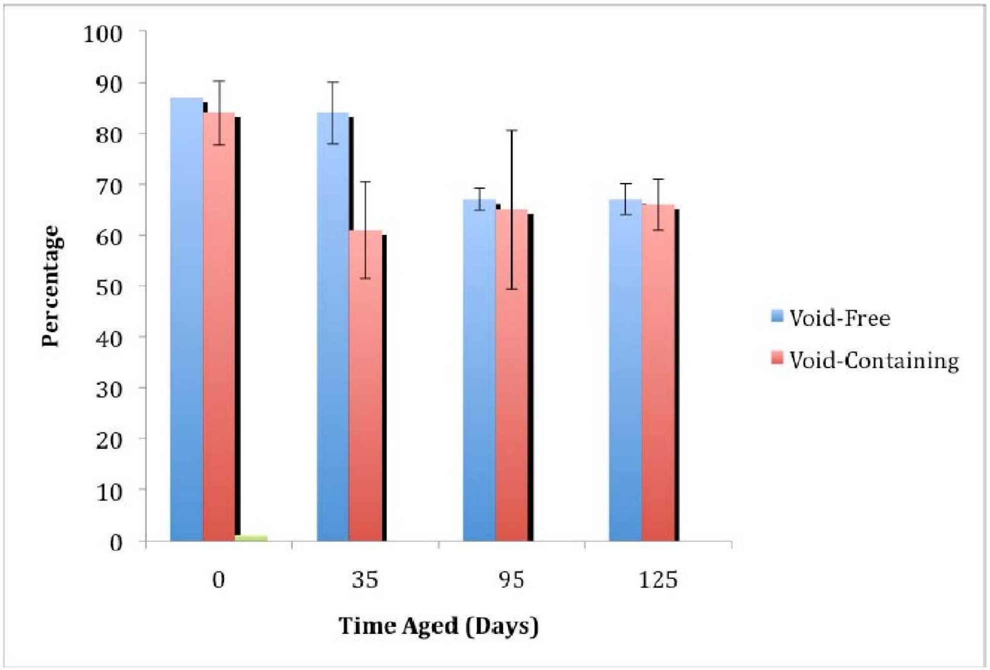


Figure 5. Percent strength retention measured at 82°C for void-free vs. void-containing specimens aged in 82°C/85% RH chambers up to 125 days.

Percent strength retention in Figure 5 was comparable between void-free and void-containing specimens aged after 35 days, with strength retention values leveling off in spite of the bond-line quality. This observation raised the possibility that voids were actually entrapped in the bulk of the "void-free" specimens but were not visibly detectable. As discussed, these specimens were only examined optically along the bond-line edges as shown in Figure 3.

The poor strength retention observed in this study requires further investigation to determine if bond quality, excessive exposure temperature, or a combination of both were responsible. Since the bonded area of these specimens was too small for ultrasonic inspection, this issue will be addressed in future work by fabricating specimens with larger bond areas. Additional SLS specimens will be fabricated, aged and tested following ASTM D5868 [14] which specifies a 6.5 sq-cm (1 sq-in) shear area versus the 3.2 sq-cm (0.5 sq-in) (ASTM D1002) used in the current study. This larger bonded surface area will retard moisture ingression and allow for bond quality investigation by non-destructive techniques such as c-scan. In addition, a lower aging temperature of 71°C (160°F) under 85% RH will be investigated.

4.4 Failure modes of SLS specimens

Fracture surface failure modes were characterized according to ASTM D5573 [10]. For specimens aged up to 125 days, the failure modes were dominated by cohesive failure and thin-layer cohesive failure.

4.5 Solvent Exposure Results

At the time of this writing, solvent exposures had been conducted for only 6 hours. The specimens were placed in a container with sufficient liquid so as to immerse the samples completely. Tables 10 and 11 list the measured thermal transitions (i.e. T_gs) for the 0° and 90° 3-point bend specimens using the vicoelastic parameter, tan δ , as measured by DMA; the graphs of the DMA runs are shown in Figures 6 and 7. The higher temperature transitions are due to the T_g of the epoxy resin, the lower temperature transitions (~150-160 C) are not fully understood at this time, but may be emanating from the thermoplastic polymeric toughener used in the resin formulation. Due to the measurement technique, the 0° specimens are configured in a manner so that the properties of the fiber dominate the behavior of the specimens, likewise the 90° specimens are configured such that the properties of the matrix resin dominate the behavior. The shift to higher T_gs for both the 0° and 90° specimens were surprising considering the short exposure time to the solvent. In this study, the water exposure had little effect; however, prolonged exposure to water is known to result in significant drops in tensile and flexural properties due to the degradation of the fiber-matrix interface [6]. The T_gs for the 0° (fiber property dominated) saline, jet fuel, deicer and hydraulic fluid exposed specimens increased between 1 and 6 °C over the control and water exposed specimens. The 90° (resin property dominated) specimens, showed a similar pattern; however, the increase in T_g was more pronounced with the increase being between 4 and 12 °C.

Table 10. Glass Transition Temperature Values for 0° Specimens

Chemical Conditions	**T_g (°C)**
Control	219
Water	217
Saline	224
Jet Fuel	224
Deicer	224
Hydraulic Fluid	226

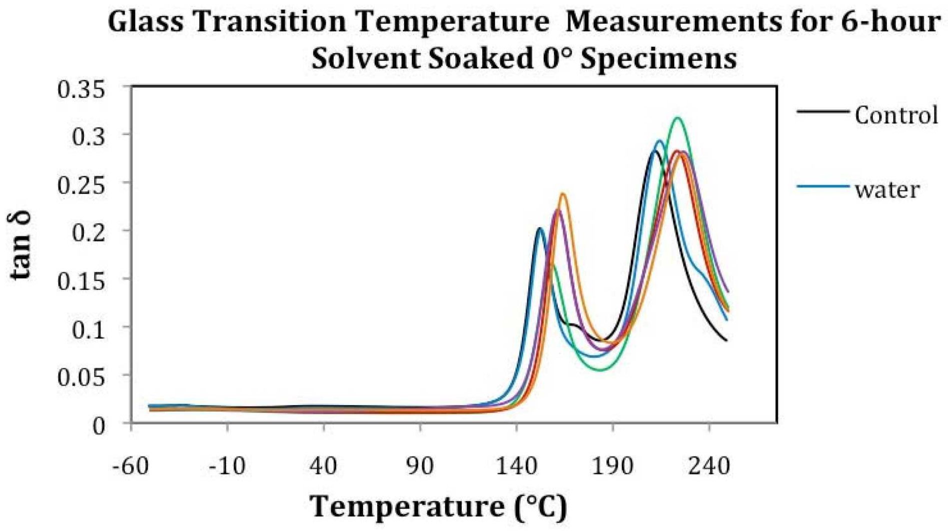


Figure 6. Graph of the viscoelastic parameter vs. temperature for the fiber property dominated, 0° specimens.

Table 11. Glass Transition Temperature Values for 90° Specimens

Chemical Conditions	**T_g (°C)**
Control	227
Water	230
Saline	241
Jet Fuel	240
Deicer	241
Hydraulic Fluid	240

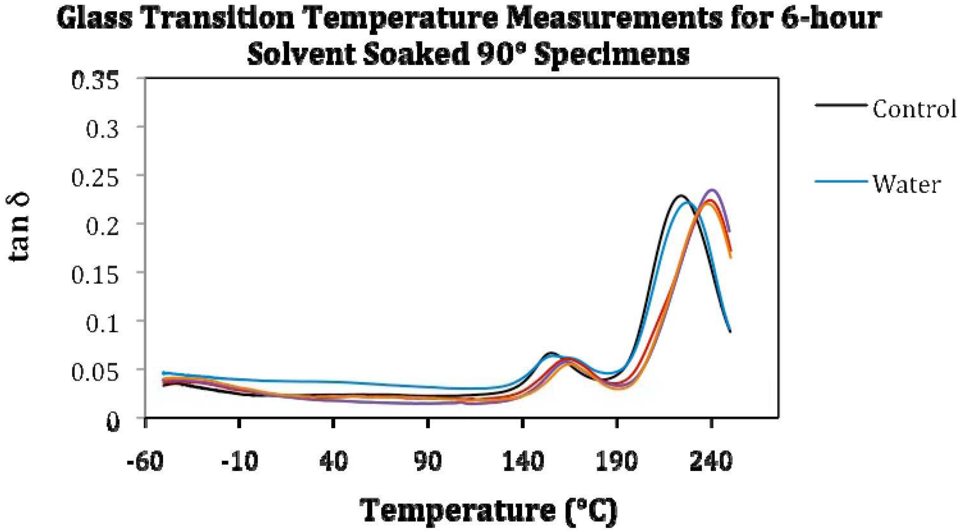


Figure 7. Graph of the viscoelastic parameter vs. temperature for the resin property dominated, 90° specimens.

5. SUMMARY

SLS specimens were fabricated using state-of-the-art carbon fiber/epoxy laminates and AF-555M adhesive. The adhesive bond was cured in an autoclave under 310 KPa (45 psi) at 177°C (350°F) for 2 hours. Fresh specimens, with either void-free or void-containing bond-lines, yielded RT SLS strengths which were consistently >10% higher than the nominal 35.8 MPa (5,200 psi) reported by the manufacturer, and had 85% strength retention when measured at 82°C (180°F).

A three-year aging plan was initiated in 82°C (180°F)/85% RH chambers. Due to an unanticipated decrease in the measured SLS after the thirty and the ninety-day results the plan was modified to incorporate a monthly inspection, thereafter. Specimens were removed at specific aging times and SLS strengths measured. Bond-line characteristics and thickness, and specimen weights before and after aging were documented. Each aging time was composed of a mixture of void-free and void-containing specimens for a total of five specimens. Control and aged specimens were tested under RT and 82°C (180°F) conditions.

For the controls stored in desiccators, both RT and 82°C (180°F) strength values remained unchanged after 125 days for both void-free and void-containing specimens. For the aged specimens in humidity chambers, RT strength retention for the void-free specimens was clearly better than for those with voids. Void-free specimens exhibited 100% strength retention with up to 3.2 months (95 days) of aging and 90% retention after 125 days of aging. On the other hand, specimens containing voids exhibited 90% strength retention after only 1.2 months (35 days) of aging; subsequently, strength diminished more rapidly.

The 82°C (180°F) strength retentions for the void-free specimens were only marginally better than those with voids. Void-free specimens maintained 85% strength retention up to 1.2 months (35 days) of aging before diminishing.

The poor strength retention behavior after aging at 82°C (180°F) and 85% RH observed in this study requires further investigation to determine if porosity in the bulk of the specimens is the cause of degradation of properties. Also, the exposure temperature may be too high for the adhesive.

After just a 6-hour exposure to commonly used aircraft solvents, the T_gs of the laminates increased between 1 and 11 ° C demonstrating the effect of the solvents on the carbon epoxy composite. An increased T_g is indicative of increased stiffness and/or brittleness in the epoxy matrix. Future work will address solvent effects on the adhesive.

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