

Comparison of Autoclave and Out-of-Autoclave Composites^{*}

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ABSTRACT

The National Aeronautics and Space Administration (NASA) Exploration Systems Mission Directorate initiated an Advanced Composite Technology Project through the Exploration Technology Development Program in order to support the polymer composite needs for future heavy lift launch architectures. As an example, the large composite dry structural applications on Ares V inspired the evaluation of autoclave and out-of-autoclave (OOA) composite materials. A NASA and industry team selected the most appropriate materials based on component requirements for a heavy lift launch vehicle. Autoclaved and OOA composites were fabricated and results will highlight differences in processing conditions, laminate quality, as well as initial room temperature thermal and mechanical performance. Results from this study compare solid laminates that were both fiber-placed and hand-laid. Due to the large size of heavy-lift launch vehicle composite structures, there is significant potential that the uncured composite material or prepreg will experience significant out-life during component fabrication. Therefore, prepreg out-life was a critical factor examined in this comparison. In order to rigorously test material suppliers recommended out-life, the NASA/Industry team extended the out-time of the uncured composite prepreg to values that were approximately 50% beyond the manufacturers out-time limits. Early results indicate that the OOA prepreg composite materials suffered in both composite quality and mechanical property performance from their extended out-time. However, the OOA materials performed similarly to the autoclaved composites when processed within a few days of exposure to ambient “shop” floor handling. Follow on studies evaluating autoclave and OOA aluminum honeycomb core sandwich composites are planned.

Keywords: Composites, Out-life, Out-of-Autoclave

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1. INTRODUCTION

Composite structures for heavy-lift launch vehicles are projected to be the largest composite structures ever fabricated in aerospace applications. Some of these large composite shelled structures are projected to be larger than 9 meters in diameter and greater than 10 meters in length. Currently, access to processing large composite structures in large autoclaves is limited since, there are not autoclaves large enough to process 9-10 meter composite shells. Therefore, OOA composites are an excellent alternative to autoclaved composite structures.

Recent advances in composite automated manufacturing technologies will be required to mitigate out-life (OL) and tack-life of prepreg for large composites. In order to gage the performance of traditional composite prepreps and slit tape used in automated fiber placement manufacturing, a comparative study examined composite performance as a function of prepreg OL. Several intermediate modulus carbon fiber matrix composites were fabricated and tested. Four polymer matrix carbon fiber composites were evaluated for this comparative study: two autoclave carbon fiber epoxy composites (IM7/8552-1 and IM7/977-3) and two OOA carbon fiber epoxy composites (IM7/MTM45-1 and T40-800b/5320). The Hexcel[®] product IM7/8552-1 was fabricated using both hand layup and fiber placement processes.

This study focused on solid laminate construction and tests performed provided an initial comparison of three factors:

- Autoclave and OOA composites
- Fresh and OL composites
- Hand-layup and fiber-placed (FP) composites

Prepreg tack was measured to verify vendor's tack-life suggestions.¹⁻⁴ While tack is strongly associated with humidity and temperature, each prepreg system exhibited tack-life as suggested by the manufacturers. Composite performance was based on laminate quality, thermal and mechanical properties. In addition to thermography, acid digestion and void volumes were compared for all four composites. Glass transition temperatures (T_g) were very close to those reported by each material supplier. Reduction in T_g was also noted after hot-wet conditioning. Mechanical response focused on room temperature, resin dominated properties: short beam shear, compression, open-hole compression, and fracture toughness.

2. EXPERIMENTAL

COMPOSITE PROCESSING

All composite systems (IM7/8552-1, IM7/977-3, IM7/MTM45-1, and T40-800b/5320) were flat 32 ply quasi-isotropic produced from 145gsm uni-tape prepreg. The OOA prepreg were partially impregnated. The ply structure was (0,0,45,45,90,90,-45,-45,-45,-45,90,90,45,45,0,0)₂ and cured per ply thickness for good quality laminates was 0.137mm (5.4mil). Only one panel was fabricated for the four material types and out-life conditions. Solid laminate panel size was 76.2 cm x 162.5cm. All prepreps were thawed, vacuum bagged and placed under vacuum. The IM7/8552-1 fiber placed panel was manufactured at ATK Iuka. The custom built fiber placement machine is a 7 axis fiber placement machine with diameter capability up to 20ft. The machine has three delivery heads: 32 tows at 0.125 inches wide, 32 tows at 0.128 inches wide, or

24 tows at 0.25 inches wide. All laminates (hand lay-up and fiber placed, “fresh” and OL laminates) underwent a debulk every 4th ply. After the OL panels were completely consolidated, the panels remained in their bag and stored at room temperature with continuous vacuum at room temperature until cured. The autoclave panels (IM7/8552-1 and IM7/977-3) defined as out-life (OL) were stored outside of the freezer a total of 45 days. The out of autoclave (OOA) panels (T40-800b/5320 and IM7/MTM45-1) were exposed to OL conditions for 35 days. It must be noted that the OL conditions were stringent and *not* recommended by the prepreg suppliers. The intent was to out-life each prepreg stack approximately 50% *past* the material supplier recommended outlife.

Laminates were cured according to prepreg supplier recommendations and can be viewed at the Cytec, Hexcel or Advanced Composite Group (ACG) websites.¹⁻⁴ Heating rates were reduced to <1 °C/min in order to mimic a typical rate for large composite structures. After visual inspection, each panel underwent flash thermography and then if areas of concern were discovered another form of Non-Destructive Inspection (NDI) was employed such as through transmission ultrasound (TTU). All test coupons were dried until day-to-day specimen mass change was less than 0.01 % for 48 hr while measuring a witness coupon. Final weights and dimensions were recorded after the specimens reached their required drying levels. They were desiccated to reduce moisture absorption before testing.

COMPOSITE TESTING

Thermal Analysis:

Dynamic mechanical analysis (DMA) was used to evaluate the T_g of each composite material and identify any knock-down associated with prepreg aging. The DMA experiments were performed on a Dynamic Mechanical Analysis (DMA/Model No 2980, TA Instruments), following American Society for Testing and Materials (ASTM) standard D 7028-07. DMA samples 2.5cm (long) by 0.50cm (wide) were cut from each fabricated panel. The samples were examined by optical microscopy for cutting damage, then dried at 71°C until the change in weight was <0.01% for two consecutive days. Additional samples were moisturized at 83 °C +/- 2.5 °C and 85% +/-5% humidity. The samples were considered saturated when the change in weight was <0.01% for 24 hours.

DMA experimental conditions specified in ASTM D7028-07 included a 5 °C/min ramp from room temperature to 50 °C above T_g. The frequency was set at 1 Hz, per the ASTM specification and the amplitude set at 20µm. The ASTM standard called for utilization of a three point bend fixture, however early tests resulted in significant clamp noise from the fixture. The intent of these DMA experiments was to determine T_g not elastic modulus, a single cantilever fixture was used for these tests.

Prepreg Tack Measurements:

Tack tests of the fresh and OL candidate prepreg materials were performed to determine the level of prepreg tack through its ability to adhere to itself and to a vertical surface. The procedure, which follows Cessna Aircraft Company Specification CPTI003⁵, calls for a corrosion resistant steel plate with a commercial 2D finish and a 2.54 cm diameter roller. The tests were to be performed at 23 °C/70°F ± 10°F and 30-60% relative humidity, using two: 7.62 cm by 2.54 cm

prepreg specimens. The OL specimens were stored in an open plastic bag to expose them to ambient humidity and temperature levels. The tack test procedure involved attaching one piece of prepreg to the steel plate with light pressure using the roller. The backing was removed and the second strip was applied on top of the first one and tacked in a similar manner. Finally, the backing from the second strip was removed and the plate was positioned vertically as shown in Figure 1.

The tack level was determined as follows:

- | | |
|--------------------|--|
| A. Tack level I: | Low tack, prepreg is stiff and dry. |
| B. Tack level II: | Dry but slight drape. |
| C. Tack level III: | Slight tack, sticks to itself but not to a vertical surface. Unable to adhere to the vertical tool surface for 30 minutes. |
| D. Tack level IV: | Good tack, prepreg sticks to itself and vertical tool. Adhered to the vertical tool surface for more than 30 minutes. |
| E. Tack level V: | Sticks to the hands or gloves but no resin transfer. |
| F. Tack level VI: | High tack, wet, and sloppy with resin transfer. |

Nondestructive Evaluation (NDE):

Each panel was inspected with FLIR Amber Engineering 25 mm ThermoVision SC6000 with Thermal Wave Imaging 8.0 Software. Flash thermography temperature change reflects thermal diffusivity difference between samples. Typical instrument settings were: 96 kJ flash power, providing a 5-10 °C, frequency 15.00 Hz, flash duration of 20 sec, and an ~20cm square field of view.

Void Analysis:

The void volume and fiber content of each flat laminate panel was calculated following ASTM 3171-76 with six samples tested per material. The matrix material was digested in hot sulfuric acid and the remaining carbon fibers were filtered through a fine mesh screen. The fibers were flushed with water followed by an acetone rinse. The acetone was evaporated overnight in a fume hood and the fibers were then dried in an oven at 100 °C prior to weighing.

Short Beam Shear:

Short beam shear (SBS) tests were conducted on samples sectioned from the solid laminate composites. Specimens were sectioned with a diamond wheel from each composite material ensuring a smooth finish as indicated by photomicrographs. The samples were thoroughly dried and then tested at room temperature. A minimum of six samples were tested for each composite using ASTM D2344 *Standard Test Method for Short-Beam Strength of Polymer Matrix Composite Materials and Their Laminates* as a guide. A displacement rate of 0.127cm/sec (0.05 inches/second) was used for all tests. Figure 2 depicts the standard three-point bend setup. Testing was terminated after a load drop of 30%. All samples were photographed along both edges to document the type and degree of cracking.

Fracture Toughness - Double Cantilever Beam (DCB):

Mode 1 interlaminar fracture toughness delamination resistance curves of four quasi-isotropic fresh and OL composites laminate materials were compared in accordance with ASTM D5528-01. Experimental tests were conducted on six double cantilever beam (DCB) coupon specimens for each composite. Modified Beam Theory (MBT) data reduction method was utilized to generate delamination resistance curves. Specimens were dried as previously described and then tested in ambient conditions. DCB specimens consisted of new and OL materials, three autoclave: IM7-977-3, IM7/8552-1, IM7/8552-1 fiber placed (FP), and one out-of-autoclave (OOA) IM7/MTM45-1. All tests were conducted at ambient conditions. The fractured surfaces of all materials were examined with a scanning electron microscope (SEM).

The DCB coupon specimens were machined from a 32-ply quasi-isotropic composite laminate flat panel with 0 degree plies at the mid-section as shown in Figure 3. The DCB specimens were 14 cm long, 2.5 cm wide and 0.46 cm thick. Panels were fabricated with a pre-cured insert: 2.5 cm wide, and 0.0254 mm thick non-adhesive Airtech A4000 solid release red film inserted along one edge.

All tests were conducted in a 223 kN load machine with a 115 kg load cell. The load machine was controlled with a MTS Test Star II system. A Vic Snap, video camera system was integrated into the control system to measure and record crack growth within the specimen. A voltage regulator was also integrated into the control system to introduce a marker into the data stream at select points during crack growth. Each coupon specimen was weighed, measured and visually inspected for any anomalies. The sides of each specimen were polished to visually enhance the film insert. Top and bottom surfaces were cleaned for the application of adhesive and metal tabs. After drying to a constant mass loss, all specimens were placed in a desiccator to maintain low moisture content until tested. Specimens were removed from the desiccator and installed in the load machine with pins inserted through the attached metal tabs. Loads and displacements were subsequently balanced. All tests were conducted in displacement control, and displacements were applied at a rate of 1.27 mm per minute. Upon crack growth of 3 to 5 mm, loading was stopped and displacement was returned to zero. The location of the crack tip was marked. Displacement was reapplied and crack growth was notated every mm - six consecutive times. This was repeated 3 times for each 5 mm crack growth for a total of 18 notations.

Compression and Open Hole Compression:

This test effort was designed to evaluate dry ultimate compressive strength at ambient conditions for fresh and OL conditions of the following fiber/resin systems: IM7/8552-1, IM7/8552-1 FP (fiber placed), IM7/977-1, and MTM 45-1. Fresh T40-800b/5320 was available for compression; however, out-life T40-800b/5320 was unavailable due to poor consolidation resulting in a poor quality laminate. All test coupons were dried until day-to-day specimen mass change was less than 0.01 % for 48 hr while measuring a witness coupon. All specimens were machined as follows: Compression samples were machined per ASTM D6641. Nominal dimensions were 13.97 cm long and 1.27 cm wide with thicknesses that varied according to material. They were tested using a combined loading (CL) fixture.

Open-hole compression (OHC) specimens were machined per ASTM D6484. Nominal dimensions were 30.48 cm long, 3.81 cm wide, and 0.391-0.495 cm thick, with 0.635 cm hole

diameter. All tests were performed on an MTS universal servo-hydraulic frame, using a platen-to-platen loading configuration that incorporated an environmental chamber.

3. RESULTS AND DISCUSSION

Thermal Analysis

The Tg of autoclave and OOA composites are shown in Table 1. There were two comparative data sets that were performed for the DMA measured Tg: a comparison of the reported Tg's and as-measure dry Tg's; and second, the as-measured dry Tg compared to wet Tg or Tg knockdown. The dry Tg's correspond well with vendor data with the exception of IM7/MTM45-1. The MTM45-1 composite dry Tg was 20-30 °C higher than Tg's reported by Advanced Composite Group (ACG). IM7/8552-1 had the least wet Tg drop of 26 °C. The dry-wet Tg knockdown for most of the composites was typically 35-40 °C. IM7/MTM45-1 wet Tg knockdown was 46 °C. While fresh MTM45-1 wet Tg appeared to be lower than the other composites, the OL wet Tg value was unchanged from its dry Tg.

Tack

Heavy lift launch vehicles will inevitably require large composite dry structures that will test the out-time limits for commercial preregs. The recommended out-life of each material, obtained from the respective vendor datasheet, is as follows: IM7/977-3 (30 days), IM7/8552-1 (21+ days), IM7/MTM45-1 (21 days), and T40-800B/5320 (21 days). Communications with both Cytec and Hexcel indicated that OL for their autoclave prepreg IM7/977-3 and IM7/8552-1, would endure longer OL times than reported in the datasheets. Tests to evaluate OL of both autoclave and vacuum bag only (VBO) cured composites were an essential aspect of this study. Therefore, an extension of the autoclave and OOA preregs out-time was set at approximately 50% past their reported out-times. Tack tests were an accurate test of prepreg OL. As described earlier, application of roller pressure to the prepreg over a steel plate aided in the determination of OL. The tack test results are summarized in the Table 2. IM7/977-3 retained tack significantly better than the other three composites.

In general, the autoclave processed composites retained tack for a much longer time than the OOA preregs. The IM7/977-3 retained tack and some drape through 60 days out-time at room temperature. The tack and drape of IM7/8552-1 dropped off quicker than IM7/977-3. Both OOA processable materials tended to lose tack and drape at a comparable rate. After 21 days, the OOA prepreg tack was rigid or stiff. At that time, the prepreg would not stick to itself or the steel substrate. Cytec reported that this batch of T40-800b/5320 prepreg experienced off-nominal conditions during the prepregging process. These conditions contributed to the rapid disappearance of tack for 5320.

NDE

Flash thermography provided qualitative differences for the fresh and OL panels. All of the panels presented no indication of localized void or delamination, just a nominal variation in thermal diffusivity as given by the varied cooling rates between the panels. A compilation of the

thermography results is shown below in Figure 4. The Y-axis in the inset chart is defined as TSR or thermographic signal reconstruction. TSR is a non-dimensional unit or product of the way that Thermal Wave Imaging processes the data. TSR is related to the temperature change and reflects thermal diffusivity difference between samples. While there are several factors that relate to an accurate comparison of thermography results, greater panel porosity (lower density) leads to lower panel thermal diffusivity and therefore, panel surface remains warmer longer after the heat pulse from the flash hood. The T40-800b/5320 OL panel showed very poor consolidation as noted in Figure 4 by its increased residual heat over that of the other panels. Due to the extremely poor consolidation, T40-800b/5320 OL panel was not evaluated further.

Void Analysis

Void volumes for the autoclave processed panels (IM7/8552-1 and IM7/977-3) were not significantly different in both the fresh (F) and (OL) panels. As shown in Table 3, the comparison of IM7/8552-1 hand layup and fiber-placed (FP) were also not significantly different. The processing and resin flow characteristics for IM7/977-3 resulted in higher carbon fiber volumes. Large differences in the IM7/MTM45-1 fresh and OL panels were evident by high void volumes (~8%) for the OL life panel. All four fresh composite systems processed nominally with low void volumes (<2%). The prepreg OL of IM7/8552-1 was tested for hand-laid and fiber-placed panels. The differences between these 8552-1 panels were not significant. However, it is noteworthy that the fiber-placed panel was as good as or slightly better than the hand-laid panel.

Short Beam Shear (SBS)

Figure 5 shows the room temperature, average short beam shear strengths and their standard deviations as a function of fresh and OL material. For the IM7/977-3, IM7/8552 and IM7/8552 (fiber-placed) there was little change in strength for the OL compared to the fresh material. Visual inspection suggested that the IM7/MTM45-1 had slightly lower shear strengths. The other three materials have SBS strengths of approximately 70 MPa (10000 psi). The strength for the IM7/MTM45-1 is 82% of this level. The reasons for reduction in SBS are clear by examining the microstructure. Figure 6 shows the untested fresh and the OL IM7/MTM45-1. The cross-sectional edge of the OL IM7/MTM45-1 exhibits extensive voids. Fluorescence metallography of IM7/MTM45-1 (outlife) in Figure 7 illustrates voids are prevalent in the brightened or whitened areas are prevalent within the tows. Close examination of the OL IM7/MTM45-1 has a large amount of resin-rich areas, voids, and trans-ply cracks compared to the fresh material.

Fracture Toughness - Double Cantilever Beam (DCB)

The room temperature dry (RTD) delamination resistance curves of three OL composites are shown in Figure 8. Each composite curve was an average of six specimen curves. Similar data trends were observed for the fresh DCB composites. Test data showed a high arching resistance curve for IM7/MTM45-1 OL specimens. Tests also showed that the three autoclave composites have similar resistance curves with small initial increases in slope, which transition to horizontal lines over the length of the specimens. Fractured surfaces of all materials were examined using scanning electron microscope (SEM) and there were significant differences in the autoclave and out-of-autoclave composites (IM7/MTM45-1). Comparison of the fracture surfaces of IM7/977-3

and IM7/MTM45-1 is shown in Figure 9. The IM7/977-3 specimen exhibited significant interfacial bonding of epoxy (977-3) to fiber (IM7). The IM7/MTM45-1 specimen had bare fibers and lower interfacial bonding. The residual morphology of MTM45-1 toughening agents was apparent and contributed significantly to the high fracture toughness values shown in Figure 8. However, this fracture toughness method (ASTM D5288) is only valid for single phase resin systems. It was apparent from the photomicrographs in Figure 9B that the toughening agent had undergone phase-separation. It should be noted that the initiation fracture toughness value was still comparable with other systems despite this being a two phase resin system.

Fracture toughness testing of T40-800b/5320 is underway and cannot be compared with the other three composites. Comparison of the delamination curves for hand-laid and fiber-placed IM7/8552-1 indicated that the fiber-placed IM7/8552-1 had slightly higher fracture toughness. At this time, there is no explanation for these slight differences as their cure cycles were identical.

Compression and Open Hole Compression (OHC)

A summary of both open-hole and compression results are shown in Table 4. IM7/977-3 had the highest compression strength and IM7/MTM45-1 was the weakest. The OOA strength fell about mid-way within the range of autoclave composite results. There was only a slight, but not statistically significant, decrease in strength associated with fiber placement. Only the IM7/MTM45-1 OL material had significant drop in compression strength. The greater than 50% reduction in average strength is consistent with an increased void fraction and would make this fiber/resin system unusable in most applications when significantly past its OL.

The composites processed from fresh prepregs produced OHC values that had less scatter in compressive strength suggesting that the results were dominated by the intentional flaw. Also the fresh, out-of-autoclave (OOA) samples had similar strengths to the autoclave composites. The hand-laid and fiber-placed IM7/8552-1 out-life composite OHC strengths were very similar. As was seen with the standard compression test results, out-life OOA composite OHC results were significantly lower. As mentioned previously, the T40-800b/5320 out-life OHC and compression samples were not available due to the poor quality of the out-life panel.

4. CONCLUSIONS

The dry-wet T_g knockdown for most composites was typical for intermediate modulus carbon fiber/epoxy 180 °C composites. It was surprising there was no T_g knockdown for the IM7/MTM45-1 OL panel. The void content for the OL IM7/MTM45-1 panel was quite large compared to the autoclave composites. With higher void volumes, the moisture saturated OL MTM45-1 panel should have plasticized and had a lower T_g. Flash thermography was primarily useful at a gross level of panel quality comparison. Panel quality differences correlated well with void volumes from acid digestion destructive testing.

The loss of tack life for the OOA prepreg has a significant impact on the manufacturing of good quality complex shaped composite structures. Both IM7/977-3 and IM7/8552-1 exhibit sufficient tack after 45 days to maintain drape in applications where tooling has a shaped surface. IM7/977-3 maintains enough tack after 60 days of ambient exposure that it sticks to itself with sufficient strength on a vertical surface. IM7/8552-1 does not maintain sufficient tack after 60 days. The OOA prepregs IM7/MTM45-1 and T40-800b/5320 lost enough tack after 10 days that the ability

to maintain position on a vertical tool surface is significantly reduced to a point that plies adhere to each other but not to the tool surface.

Short beam shear strengths of all fresh composite systems were nominal compared to unidirectional SBS values from each vendor. The loss of tack of the OL IM7/MTM45-1 affected cure process resin flow. For this OOA composite, trapped air was not removed resulting in a high void volume. Cure cycle modifications may have improved the quality of the IM7/MTM45-1 OL panel. In order to maintain relevance to curing large composite structures, the cure cycles for the fresh and OL composites were maintained the same.

The autoclave composites, IM7/977-3 and IM7/8552-1, resistance crack growth was similar. The crack propagation resistance curves for both the fresh and OL composites were also similar; therefore, only the OL crack propagation responses are shown in Figure 8. Only slight differences were noted for the hand-laid and fiber-placed IM7/8552-1 composites. The fresh and OL fiber-placed 8552-1 panels had slightly higher crack resistance than the hand laid panels. However, MTM45-1 is a highly toughened resin system. It has a soluble thermoplastic added to the thermosetting resin. During a representative cure cycle, the thermoplastic can separate out to form a second phase. Phase separation near the fiber-resin interface is also aided by the local nucleation forces resulting in a phase inversion. Apparently, the slow heating rate during cure enabled phase separation. During this process, the thermoset precipitates in a thermoplastic matrix. Remnants of the phase separation process are observed in the Figure 9b. These phase separated pockets are not voids or some kind of foam structure. The second phase acts to either blunt the crack or divert it, making the crack path more tortuous and giving rise to a typical rising R-curve behavior.

The OHC and compression values for both the fresh and OL composites were very similar. However, the OL OOA composites were obviously taken much too far past their vendor specified out-life. The minimal difference in compression strengths for the fiber placed and the hand-laid IM7/8552-1 bodes well for the future use of fiber placed composites.

In summary, the autoclave composites (IM7/977-3 and IM7/8552-1) had significant tack life, well past their manufacturers recommended tack life times. It was impressive that IM7/977-3 maintains tack up to 60 days. Moreover, it is assuring that the autoclave systems preserve their mechanical performance after 45 days in out-life conditions. While the out of autoclave composites have comparable mechanical and thermal performance after processing fresh prepreg, the OOA prepregs thermal and mechanical performance was significantly reduced after curing the OL prepreg. Recent advances in OOA technologies are rapid and many. There will likely be several advances in both mechanical and thermal properties. For applications requiring long OL prepregs, a focus on improving tack and prepreg processability will be necessary in order to produce high quality composites with mechanical properties equivalent to or greater than autoclave composites.

REFERENCES

1. Cytec IM7/977-3: <http://www.cytec.com/engineered-materials/products/Datasheets/CYCOM%20977-3.pdf>
2. Hexcel IM7/8552-1: http://www.hexcel.com/NR/rdonlyres/B99A007A-C050-4439-9E59-828F539B03A4/0/HexPly_8552_eu.pdf

3. Advanced Composite Group IM7/MTM45-1: http://www.advanced-composites.co.uk/PSG_Electronic_Files/Aerospace_PSG_Files/PDFs/PDS1205_MTM45-1_Issue4.pdf
4. Cytec T40-800b/5320: <http://www.cytec.com/engineered-materials/products/Datasheets/CYCOM%205320Rev1-3.pdf>
5. Cessna Aircraft Company Specification CPTI003

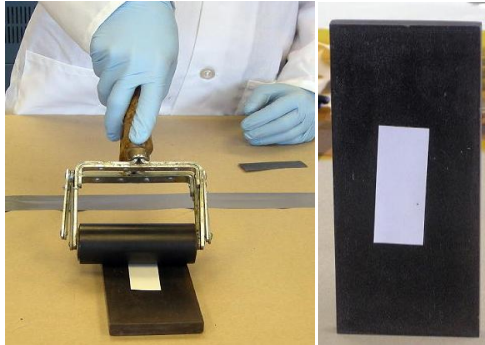


Figure 1. Tack test roller and vertical steel plate tests

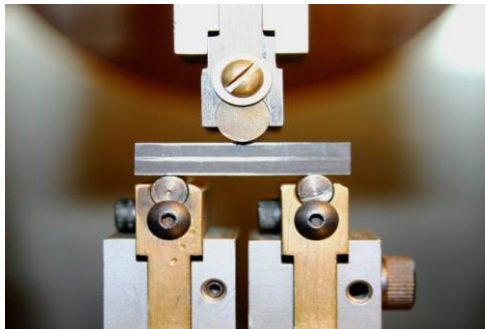


Figure 2. Short beam shear test fixture and sample

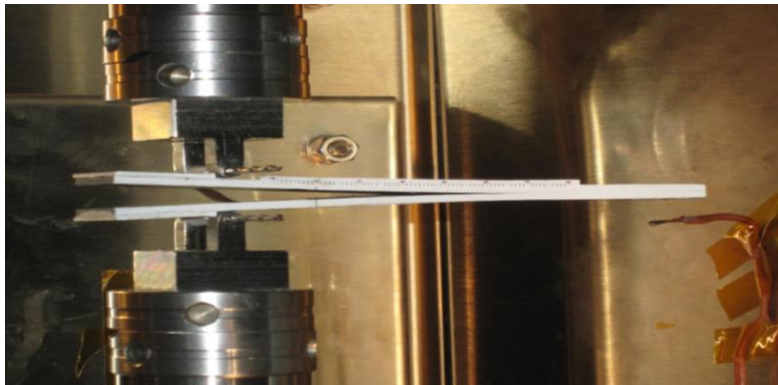


Figure 3. Double cantilever beam fixture and specimen

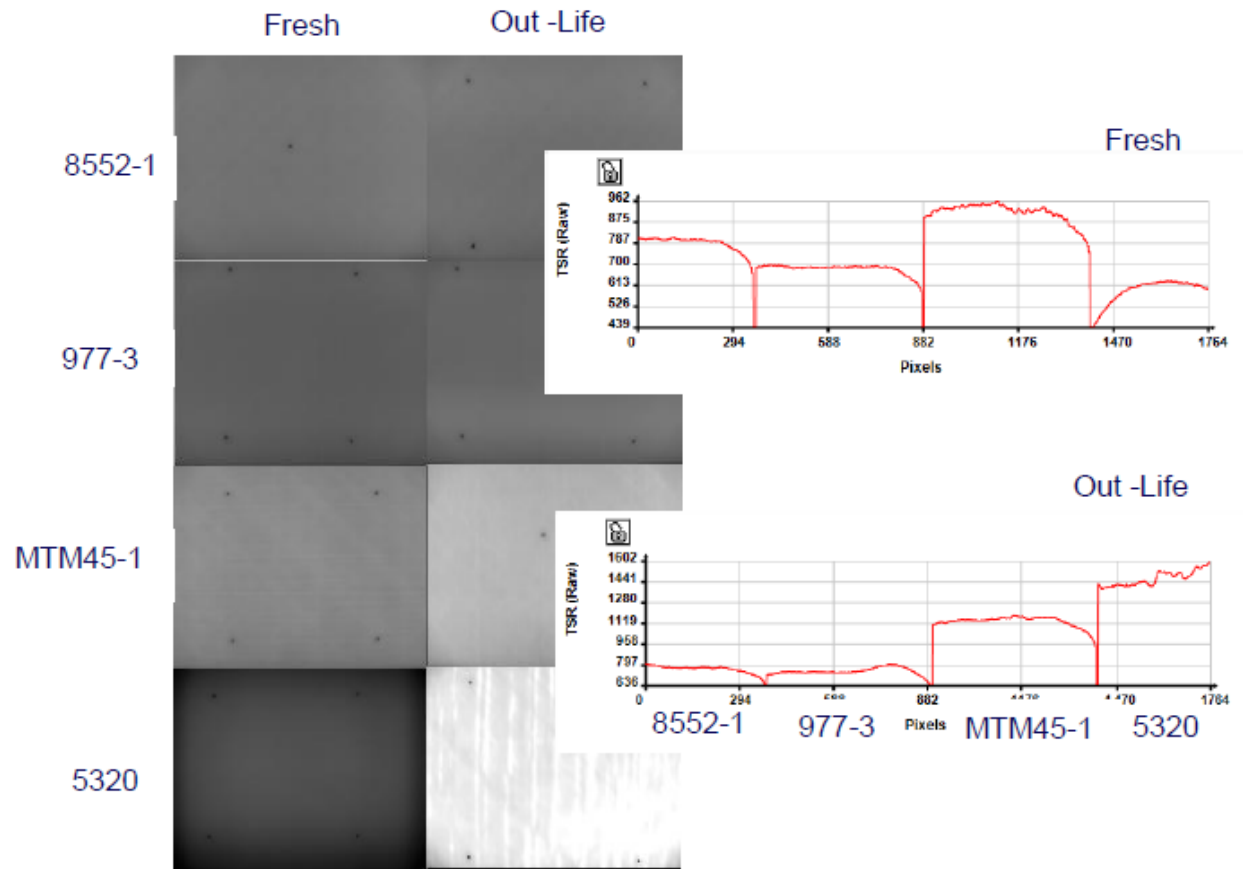


Figure 4. Flash Thermography comparison of autoclave and OOA composites

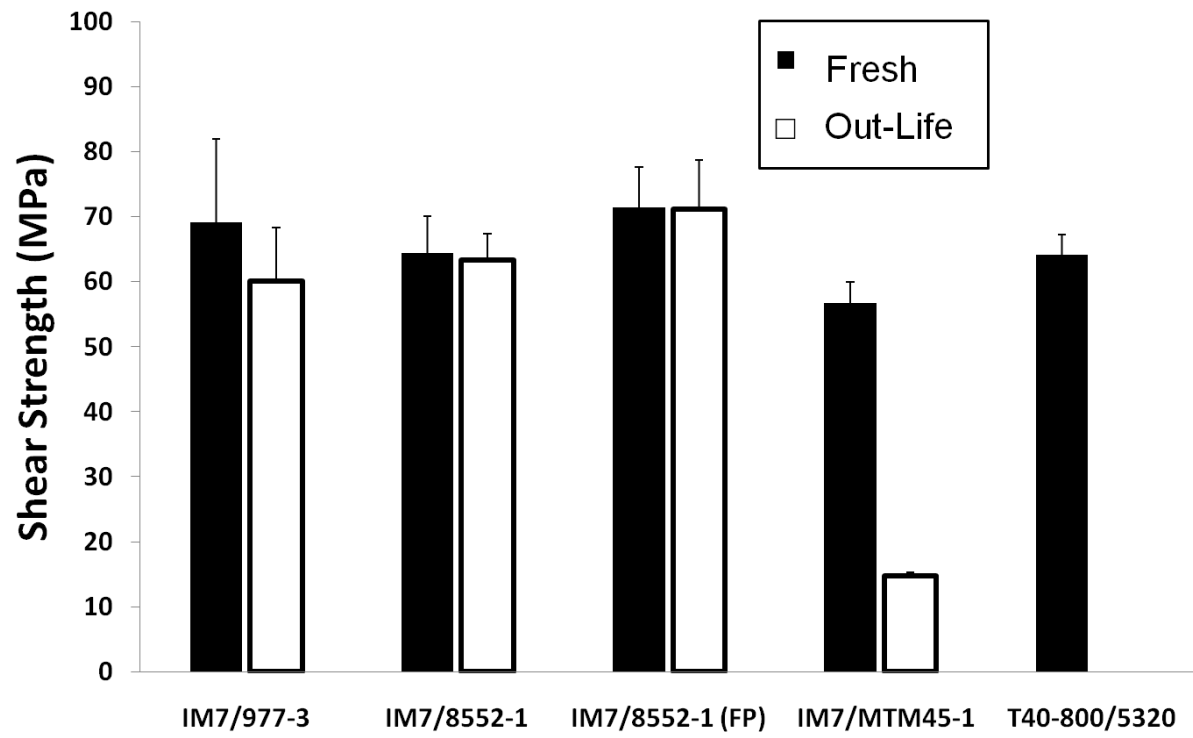


Figure 5. Short Beam Shear (SBS) Comparison

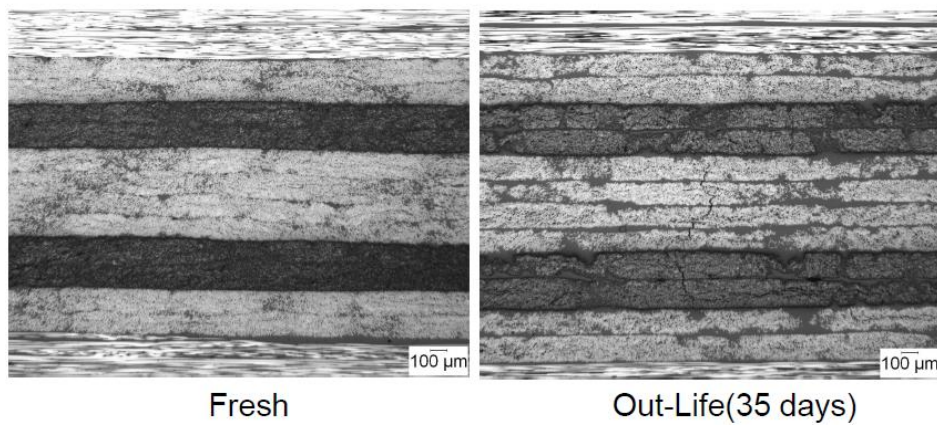


Figure 6. IM7/MTM45-1 fresh and out-life short beam shear specimens

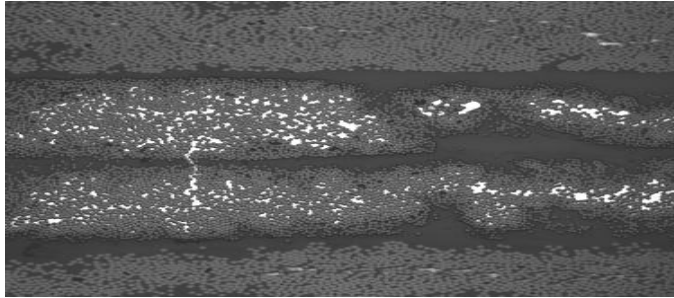


Figure 7. Void content of 35 day outlife IM7/MTM45-1 cross-section (100x)

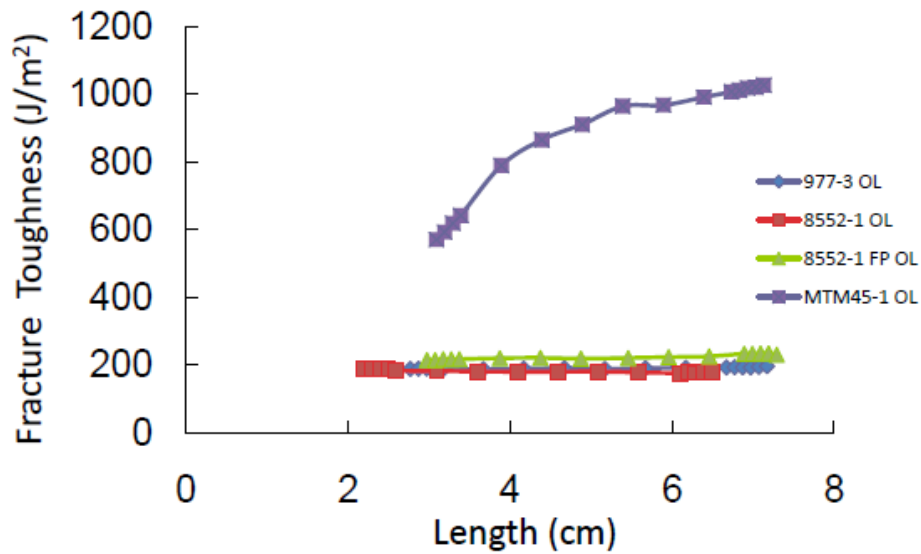
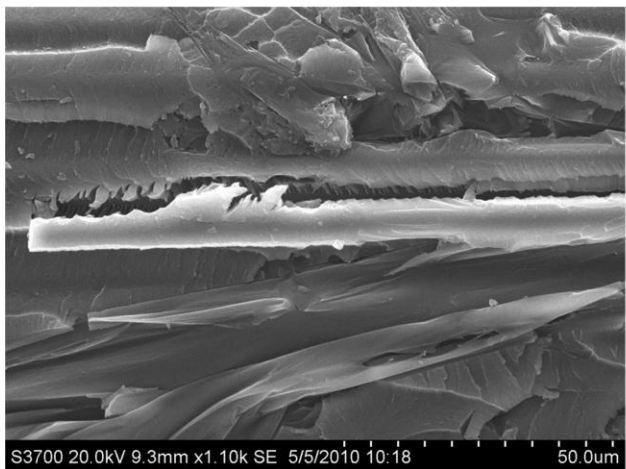


Figure 8. Fracture toughness of delamination resistance of out-life samples

(A) IM7/977-3



(B) IM7/MTM 45-1

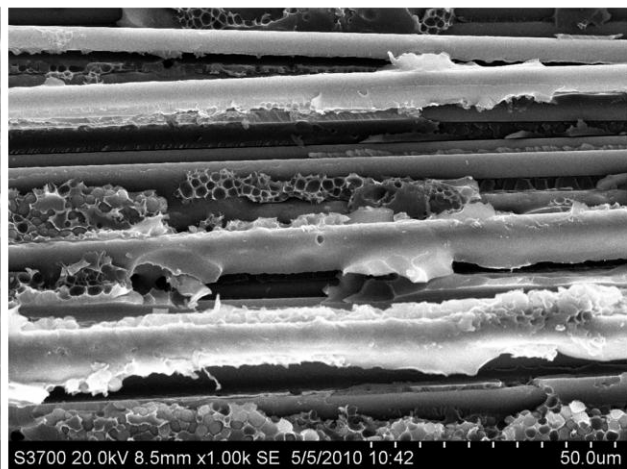


Figure 9. Comparison of fresh IM7/977-3 and IM7/MTM45-1 DCB fracture surfaces

Table 1. Glass transition temperatures fully dried composites

Sample	Reported T _g (°C)	Dry T _g (°C)	Wet T _g (°C)	Knockdown (°C)
IM7/977-3 New	201	204	167	37
IM7/977-3 OL	--- ^a	204	167	37
IM7/8552-1 New	212	210	184	26
IM7/8552-1 OL	---	216	184	32
IM7/8552-1 New-FP	212	212	177	35
IM7/8552-1 OL-FP	----	216	175	41
IM7/MTM 45-1 New	180	215	169	46
IM7/MTM 45-1 OL	----	200	200	0
T40-800B/5320	204	207	167	40

Table 2. Tack life characterization

Autoclave Materials	Out-Life/Tack Level				
	0 days	15 days	30 days	45 days	60 days
IM7/977-3	V	IV	IV	IV	III
IM7/8552-1	IV	IV	II	II	I
OA Materials	Out-Life/Tack Level				
	0 days	10 days	21 days	35 days	
IM7/MTM45-1	IV	II/III	II	I	
T40-800b/5320	IV	II	I	I	

Table 3. Composite void analysis from acid digestion

Sample	Void Content		Fiber Volume	
	% Void	Std. Deviation	% Fiber Vol.	Std. Deviation
977-3 F	1.2	0.8	63	4.6
977-3 OL	1.5	0.6	63	1.6
8552-1 F	0.9	1.2	59	3.0
8552-1 OL	0.6	0.8	59	2.0
8552-1 F-FP	0.5	1.1	59	2.6
8552-1 OL-FP*	0.1	0.6	58	0.7
MTM45-1 F	0.1	0.1	58	0.4
MTM45-1 OL	8.3	0.6	53	1.4
5320 F	0.1	0.2	59	0.2

Table 4. Open Hole Compression and Compression - Fresh and Outlife Composites

Sample	OHC – Fresh (MPa)/STD	OHC – Out-life (MPa)/STD	Compression Fresh (MPa)/STD	Compression Outlife (MPa)/STD
IM7/977-3	348/16	349/5	439/19	403/26
IM7/8552-1	335/11	356/9	381/24	391/24
IM7/8552-1 (FP*)	354/16	356/13	352/45	379/30
IM7/MTM45-1	342/25	170/7	365/18	164/12
T40-800b/5320	374/28	----- ^a	409/17	----- ^a

^a Sample not available