

Evaluation of Sandwich Structure Bonding In Out-of-Autoclave Processing*

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ABSTRACT

The out-of-autoclave-vacuum-bag-only (OOA-VBO) process is low in capital expenditures compared to the traditional autoclave, however, the material challenges for OOA-VBO workable material systems are high. Presently there are few such aerospace grade prepreg materials available commercially. In this study, we evaluated processing and properties of honeycomb sandwich structure (HC/SS) panels fabricated by co-curing composite face sheet with adhesives by the OOA-VBO process in an oven. The prepreg materials were IM7/MTM 45-1 and T40-800B/5320. Adhesives studied were AF-555M, XMTA-241/PM15, FM-309-1M and FM-300K. Aluminum H/C cores with and without perforations were included. It was found that adhesives in IM7/MTM 45-1/AF-555M, T40-800B/5320/FM 309-1M and T40-800B/5320/FM-300K panels all foamed but yielded high flatwise tensile (FWT) strength values above 8,275 kPa (1,200 psi). IM7/MTM 45-1/XMTA-241/PM15 did not foam, yet yielded a low FWT strength. SEM photomicrographs revealed that the origin of this low strength was poor adhesion in the interfaces between the adhesive and face sheet composite due to poor wetting associated with the high initial viscosity of the XMTA-241/PM15 adhesive.

1. INTRODUCTION

High performance composite materials are conventionally processed in autoclaves which require expensive capital investment for the equipment. Equipment cost increases exponentially with the size of the autoclave. It is also costly to operate an autoclave to manufacture composite structural subcomponents. In addition, the main constraint on the ability to fabricate large composite structural subcomponents is the size of the available autoclave. As an example, NASA's Constellation program Ares V cargo launch vehicle calls for large composite dry structural applications for sizes up to 10 meters (30 feet) in diameter. There are only a few autoclaves in the US which can accommodate such large structures.

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Out-of-autoclave (OOA) processing has emerged as an alternate processing technique to autoclave processing since the early 1990s. Many OOA processing techniques have since been developed which include automated-tape-placement (ATP), filament-winding, vacuum-assisted-resin-transfer-molding (VARTM) and resin-transfer-mold (RTM) [1-5]. In particular, the OOA-VBO (out-of-autoclave-vacuum-bag-only) process in an oven requires a low capital investment, and provides airframe manufacturers with greater design flexibility for the production of large, highly-loaded structural members with complex geometries. Resin impregnated fiber reinforced prepregs are first laid-up and bagged in a flexible plastic bag in an oven. Unlike autoclave processing, OOA-VBO affords only a maximum pressure of 101 kPa (14.7 psi) from the vacuum applied to the bag for composite consolidation. Because of the limitation in pressure, an OOA-VBO workable material must exhibit low viscosity before curing at elevated temperatures, and volatile (reaction by-products, moisture, etc.) management becomes more critical compared to autoclave processing. Voids created by the entrapment of volatiles are detrimental to the mechanical properties of composite laminates.

Because the challenges of OOA-VBO materials are high, there are only two such aerospace grade prepreg systems available commercially. They are IM7/MTM 45-1 from ACG (Advanced Composites Group, Tulsa, OK) and T40-800B/5320 from Cytec (Anaheim, CA). In this work, we evaluated the processing and properties of these two prepreg systems co-cured as face sheets in honeycomb sandwich structures (HC/SS) with four adhesives, AF-555M from 3M (St Paul, MN) [6], XMTA-241 from ACG [7], FM 309-1M and FM-300K from Cytec [8]. Aluminum honeycomb cores from Alcore Inc. (Edgewood, MD) with and without perforations were used. Thermal and rheological measurements were conducted for both adhesives and matrix resins. Flatwise tensile (FWT) strength was measured at room temperature (RT). Fracture surface failure modes between different prepreg/adhesive combinations were also examined by optical and electron microscopy.

2. MATERIALS[†]

The materials used in this study are presented in Table 1. Structural adhesive Scotch-Weld™ AF-555M is a 177°C (350°F) cured, non-woven carrier supported (matte) film received from 3M (St. Paul, MN). XMTA-241/PM15, an experimental 121°C (250°F) OOA-VBO cured adhesive film, was received from Advanced Composites Group (ACG, Tulsa, OK). A free-standing post cure at 177°C (350°F) is recommended by ACG. FM 309-1M (matte supported) and FM-300K (knit supported) films were supplied by Cytec Engineering Materials (Havre de Grace, MD). AF-555M was studied in both as-received and thermally pre-treated forms, while the other adhesives were investigated only as-received, without pre-treatments.

IM7/MTM 45-1 (ACG) [9] and T40-800B/5320 (Cytec) [10] represent two state-of-the-art aerospace grade OOA-VBO carbon fiber reinforced prepreg systems available commercially. Both materials can be cured at 121°C (250°F) under vacuum pressure only, followed by post curing (in-situ or free-standing) at 177°C (350°F).

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

Aluminum honeycomb (H/C) cores (phosphoric acid anodized (PAA) treated), 39.5 kg/m² (8.1 lb/ft²), 0.48 cm (3/16”) cell size and 2.54 cm (1”) height (Al-5052-PAA-8.1-3/16-1.000), were received from Alcore Inc. (Edgewood, MD). Cores with and without perforations were included in this study.

Table 1. Adhesive and prepreg materials used in this study

Adhesive	Adhesive Supplier	Adhesive Weight, kg/m ² (lb/ft ²)	Face sheet Prepreg	Prepreg Supplier	H/C Type
AF-555M	3M	0.39 (0.08)	IM7/MTM 45-1	ACG	Perforated/Non-perforated
XMTA-241/PM15	ACG	0.29 (0.06)	IM7/MTM 45-1	ACG	Non-perforated
FM 309-1M	Cytec	0.39 (0.08)	T40-800B/5320	Cytec	Perforated
FM-300K	Cytec	0.39 (0.08)	T40-800B/5320	Cytec	Perforated

3. EXPERIMENTAL

3.1 Preparation of H/C Core Sheets

The as-received Al H/C cores were cut into 20 cm by 20 cm (8” x 8”) or 30 cm by 30 cm (12” x 12”) sheets. These sheets were cleaned with distilled water and rinsed with acetone, then dried in a forced air circulation oven at 70°C (160°F) for 2 hours. Pre-treated sheets of cores were sealed individually in plastic bags and stored at RT before use.

3.2 Thermal Pre-treatment of AF-555M Films

Sheets of AF-555M adhesive film were sandwiched between porous release cloths and laid on a tool plate inside an oven. The oven was heated at 1°C (2°F) per minute from room temperature (RT) to 110°C (230°F) and held for 2 hours, then cooled down. The pre-treated films were stored in sealed plastic bags in a 4°C (40°F) refrigerator before use.

3.3 Measurement of Viscosity

Viscosities of adhesives (as-received and pre-treated) and matrix resins (MTM 45-1 and 5320) were measured. Dynamic rheological measurements were conducted using a parallel plate fixture on a rheometer (ARES, TA Instruments, Inc) [11]. The lower plate was oscillated at a fixed strain of 2% and a fixed angular frequency of 10 rad/sec while the upper plate was attached to a transducer which recorded the resultant torques. The adhesive or matrix resin films were trimmed to disks of 2.54 cm in diameter. For the materials studied, four to six disks were stacked up for a specimen of 1.2 to 1.5 mm in thickness and then loaded between the parallel plates. The oven was heated according to the vendor recommended temperature cycle for a given specimen. Storage (G') and loss (G'') moduli were obtained as a function of time (t). These moduli were

then converted to the complex viscosity $\eta^*(t)$. The specimen's softening point, minimum viscosity, gelation temperature, and the processing window were identified from the measurements.

3.4 Fabrication of Honeycomb Sandwich Structure (HC/SS) Panel

The quasi-isotropic 16-ply composite face sheet, 20 cm by 20 cm (8" by 8"), [-45/90/45/0]_{2s}, was assembled by stacking up 16 plies of uni-directional prepreg plies. Adhesive films were also trimmed to the same size. Both adhesive and pre-stacked face sheets were individually sealed in a plastic bag and stored in a freezer at -20°C (-10°F) before use.

A pre-stacked 16-ply prepreg face sheet was laid on a steel tool plate covered with Kapton[®] film and release cloth, followed by a pre-trimmed adhesive film of the same size. A 2.54 cm (1") thick H/C core sheet was then laid down on top of the adhesive film, followed by another layer of adhesive film and 16-ply pre-stacked face sheet. Finally, a layer of release film and a steel caul plate were placed on top to complete the assembly. The assembly was dammed on four sides and bagged according to common practices of vacuum bag curing.

The composite prepreg face sheet and the adhesive film were co-cured in an oven according to the cure cycle provided by the supplier for a given prepreg/adhesive combination.

3.5 Measurement of Flatwise Tensile (FWT) Strength

Each co-cured HC/SS panel was cut to yield three 5 cm by 5 cm (2" by 2") FWT test specimens. Face sheets of each specimen were lightly cleaned and roughened by sand blasting using a 220 grit size and 275 kPa (40 psi) nozzle pressure (Econoline Inc, Grand Haven, MI), cleaned by a damp cloth and dried in an oven at 75°C (160°F) for 1 hour. The pre-treated specimens were then bonded between two 5 cm (2") aluminum blocks using Hysol 9395 two-part adhesive. The adhesive was allowed to cure at RT overnight, followed by oven curing at 75°C (170°F) for 1 hr. FWT strength measurements were conducted on an MTS test frame according to ASTM C297 specifications [12].

3.6 Optical Micrographic Examination

Fracture surfaces of the failed FWT specimens were examined with a Canon EOS D60 camera using macro photo lens MP-E, 1-5x, and characterized according to ASTM D-5573 specifications [13]. Fracture surface morphology was also investigated by scanning electron microscope (Hitachi S-3700N SEM).

4. RESULTS And DISCUSSION

4.1 Adhesive Bonding Characteristics of As-received vs. Pre-treated AF-555M

As-received AF-555M adhesive exhibited a minimum viscosity of 290 Poise (P) at 120°C during a temperature ramp of 1°C (2°F)/min from RT. Such a low viscosity rendered superior processability for this adhesive. However, when heated to elevated temperatures the AF-555M foamed under vacuum. The origin of the foaming reaction was unknown, but was dependent upon the heating rate and vacuum level. Thermal pre-treatment will stiffen the material by

partially advancing the degree of cure of the matrix adhesive, and thus inhibit/retard the foaming reaction by depressing the growth rates of air and gaseous volatiles. It is conceivable that severe thermal pre-treatment will effectively increase viscosity of the adhesive to an extent that becomes unbondable in the HC/SS fabrication.

The thermal pre-treatment condition of 110°C for 2 hrs was found to yield an adhesive which exhibited a minimum viscosity of approximately 2,200 P at 120°C. The foaming reaction was also significantly suppressed when heated in an oven under vacuum. A four panel test matrix was designed which included two material variables, i.e. as-received vs. pre-treated adhesive and cores with and without perforations. IM7/MTM 45-1 16-ply prepreg, quasi-isotropic face sheets were co-cured with the adhesives in each panel. Results were tabulated in Table 2 and plotted in Figure 1.

Table 2. AF-555M bond strengths and fracture failure modes of HC/SS panels

Panel ID	Adhesive Condition	H/C Type *	Face sheet Per Ply Thickness, cm (in)	FWT Strength **, kPa (psi)	Fracture Mode ^, %
TH-011110	As-received	Perforated	0.0132 (0.0052)	8,667 ± 538 (1,257 ± 78)	95 Co/5 Ft
TH-082609	As-received	Non-perforated	0.0127 (0.0050)	6,343 ± 1,110 (920 ± 161)	50 Co/50 Ad
TH-010510	110°C/2-hr Pre-treated	Perforated	0.0130 (0.0051)	9,536 ± 1,041 (1,383 ± 151)	95 Co/5 Ft
TH-080409	110°C/2-hr Pre-treated	Non-perforated	0.0130 (0.0051)	8,708 ± 1,200 (1,263 ± 174)	95 Co/5 Ft

*H/C core: Al-5052-PEP-8.1-3/16-1.000 from Alcore, Inc

** Average of 3 specimens tested

^ Qualitative assessment of the fracture surfaces: Co - cohesive failure, Ad - adhesive failure, Ft - fiber tear

It is evident from Figure 1 that the pre-treated AF-555M adhesive yielded higher FWT strengths in the HC/SS bonds in both types of Al HC core. These strength values compared favorably to the 7,585 kPa (1,100 psi) reported by the supplier (3M) in similar Al-composite HC/SS bonds [2].

As-received adhesives with perforated cores yielded a decent value of 8,667 ± 538 kPa (1,257 ± 78 psi). However, a much lower strength value of 6,343 ± 1,110 kPa (920 ± 161 psi) was measured for the non-perforated cores when bonded with the same adhesive.

Optical photographs of the fracture surfaces for these four panels are shown in Figure 2. A pair of photographs representing the core and the face sheet sides of the fracture surfaces is included for each panel. Fracture surfaces for the panels bonded with pre-treated AF-555M adhesives exhibited near-zero voids, regardless of the core type used. The adhered fillets on the core side were evenly distributed around the cell walls, and the hexagonal shapes of the fractured adhesive resin adhered to the composite substrate on the face sheet side were clearly defined. The surface fracture was clearly dominated by the cohesive failure mode, resulting in higher FWT strengths.

Fracture surfaces for the panels bonded with as-received AF-555M adhesive contained voids. Voids were particularly evident and abundant in the fractured adhesive resin on the face sheet side. Because of the superior fluidity of as-received AF-555M adhesive, the effect of vacuum was evident in that the composite substrate was visible. For both panels, the hexagonal shape of the fractured adhesive resin on the face sheet was distorted because resin thicknesses were not

uniform across the residue. In addition, the adhesive fillets were not evenly adhered to the core walls and the surface fracture was characterized by the adhesive failure mode resulting in lower FWT strength values.

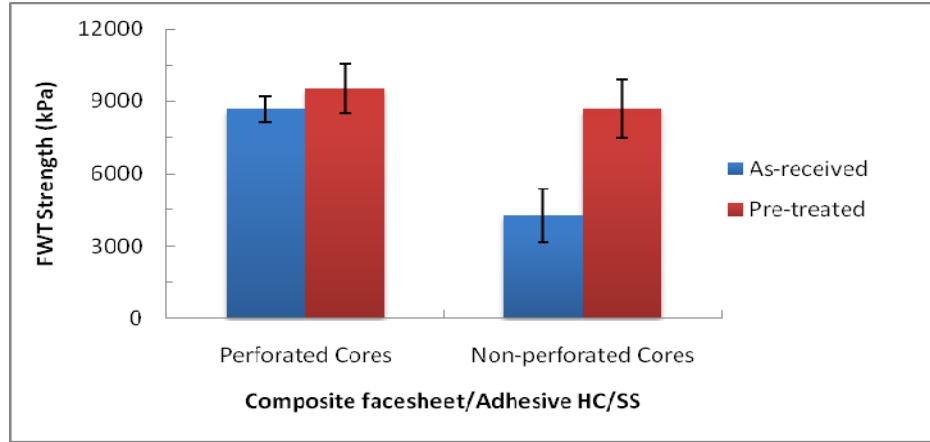


Figure 1. Effects of adhesive treatment and core type on the FWT strength of AF-555M adhesive.

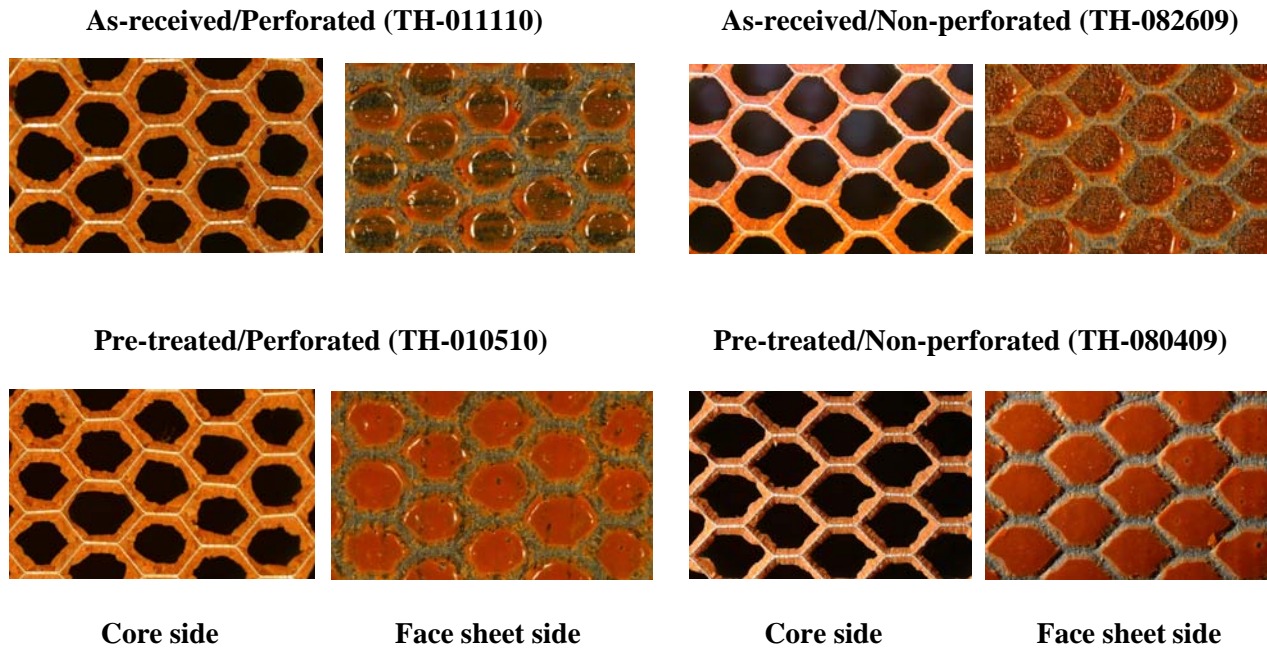


Figure 2. Optical photographs for the fracture surfaces of four HC/SS panels bonded with AF-555M adhesive

4.2 Adhesive Bonding Characteristics of XMTA-241/PM15

As-received XMTA-241/PM15 adhesive exhibited a minimum viscosity of 7,100 P at 80°C during a temperature ramp of 1°C (2°F)/min from RT. This viscosity is three orders of magnitude higher than the AF-555M adhesive, and resulted in inferior surface wetting during HC/SS fabrication. This adhesive did not foam under vacuum at elevated temperatures.

Two pre-stacked 16-ply quasi-isotropic [-45/90/45/0]_{2s} IM7/MTM 45-1 face sheets were co-cured with the adhesive according to the cure cycle provided by the supplier (ACG). Adhesive test results are tabulated in Table 3. The measured FWT strength value was 4,730 ± 724 kPa (686 ± 105 psi), which is about 75% of the strength value obtained for the AF-555M adhesive bonded as-received on the same non-perforated cores (6,343 ± 1,110 kPa (920 ± 161 psi) in Table 2). The fracture surfaces were dominated by the adhesive failure mode, an indication of poor interfacial adhesion.

Table 3. Bond strength and fracture failure mode of XMTA-241/PM15 adhesive*

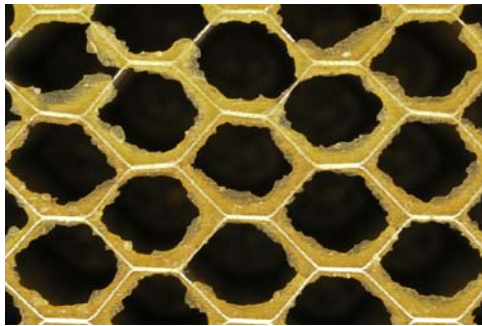
Panel ID	Adhesive	Prepreg	Face sheet Per Ply Thickness (cm/in)	FWT Strength [^] , kPa (psi)	Fracture Mode ^{^^} , %
TH-110509	XMTA-241/PM15	IM7/MTM 45-1	0.0127/0.0050	4,730 ± 724 (686 ± 105)	5 Co/95 Ad

*Al-5052-PEP-8.1-3/16-1.000 non-perforated cores and as-received adhesive were used

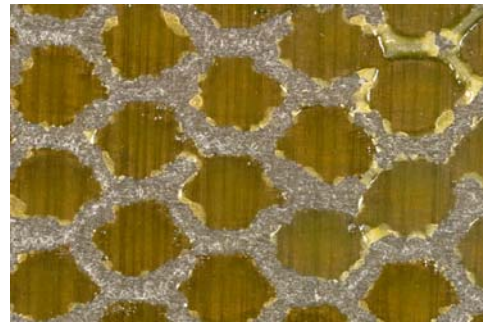
[^]Average of 3 tested specimens

^{^^}Qualitative assessment of the fracture surfaces: Co - cohesive failure, Ad - adhesive failure

As-received/Non-perforated (TH-110509)



Core side



Face sheet side

Figure 3. Optical photographs of the fracture surfaces of XMTA-241/PM15 HC/SS panel.

Optical photographs of the fracture surfaces of the core and the face sheet sides are shown in Figure 3. The adhesive fillets were poorly and unevenly adhered to the core walls. The prevailing adhesive failure mode was evident from both sides of the fracture surfaces, resulting in a low FWT strength value. The hexagonal shapes of the fractured adhesive resin on the face sheet were distorted. The residual resin layer was thin so the composite substrates were visible through the

adhesive. No voids were observed in either adhesive fillets on the cores or the resin on the face sheet side of the sandwich panel.

The origin of the weak FWT strength for this adhesive was determined by the 3-D SEM photomicrographs shown in Figure 4. Also included for comparison is a photograph of the fracture surface from the pre-treated AF-555M adhesive which yielded a high FWT strength of $8,708 \pm 1,200$ kPa ($1,263 \pm 174$ psi). The adhesive failure resulting in low FWT strength for the XMTA-241/PM15 adhesive was due to poor adhesion in the interfaces between the adhesive and the composite face sheet.

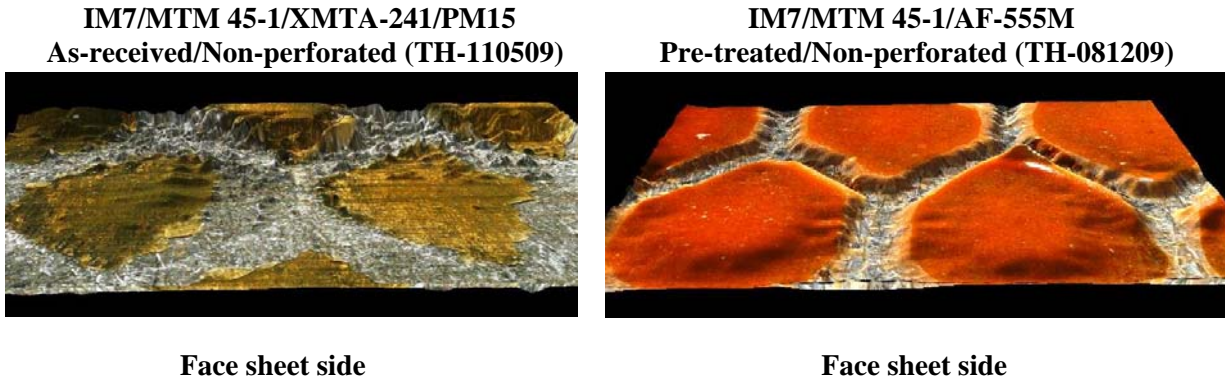


Figure 4. 3-D SEM photomicrographs of the fracture surfaces of the composite face sheet sides of two HC/SS panels

4.3 Adhesive Bonding Characteristics of FM 309-1M and FM-300K

As-received FM 309-1M adhesive exhibited a stable minimum viscosity of 4,200 P at 75° - 135°C during a temperature ramp of 1°C (2°F)/min from RT. This viscosity is approximately one order of magnitude higher than the as-received, and approximately two times that of the pre-treated AF-555M adhesive. Foaming was noted for the FM-300K adhesive but not for the FM 309-1M adhesive under vacuum at elevated temperatures.

Table 4. Bond strengths and fracture failure modes of FM 309-1M and FM-300K adhesives*

Panel ID	Adhesive	Prepreg	Face sheet Per Ply Thickness (cm/in)	FWT Strength [^] , kPa (psi)	Fracture Mode ^{^^} %
TH-021010	FM 309-1M	T40-800B/5320	0.0122/0.0048	10,467 ± 407 (1,518 ± 59)	90 Co/5 Ad/5 Ft
TH-021210	FM-300K	T40-800B/5320	0.0046/0.0117	8,618 ± 407 (1,250 ± 19)	50 Co/45 Ad/5 Ft

* Al-5052-PEP-8.1-3/16-1.000 perforated cores and as-received adhesives were used

[^] Average of 3 tested specimens

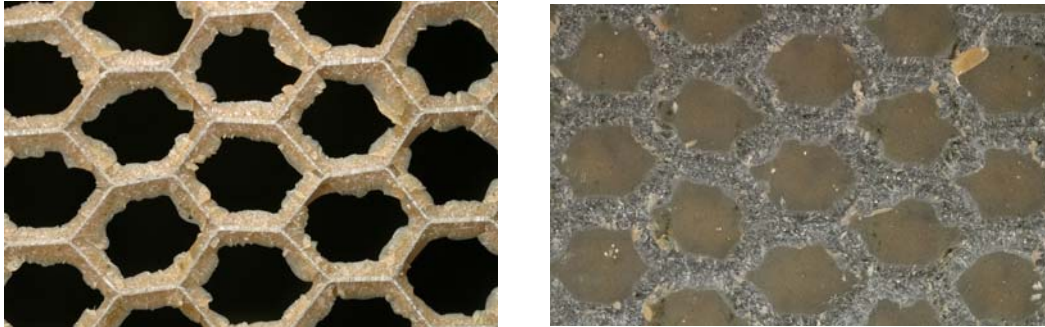
^{^^} Qualitative assessment of the fracture surfaces: Co - cohesive failure, Ad - adhesive failure, Ft - fiber tear

These adhesives were co-cured with T40-800B/5320 prepreg during the fabrication process of HC/SS panels. The same bagging and curing procedures were followed as with the sandwich

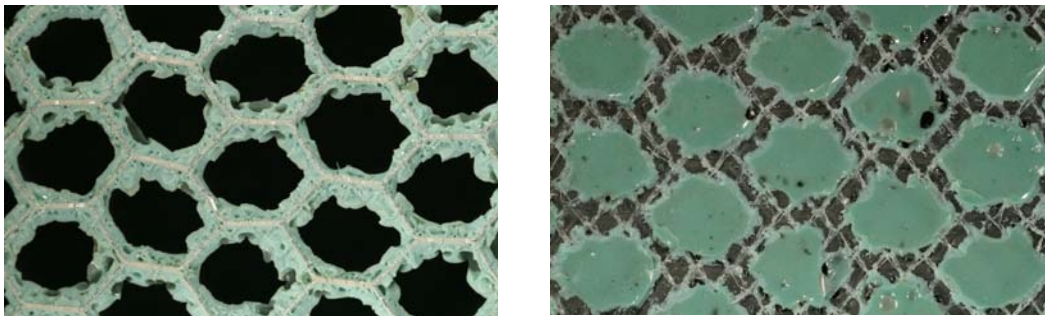
panels described above. The T40-800B/5320 cure cycle provided by the supplier (Cytec) was used. Results were tabulated and are shown in Table 4.

FM 309-1M adhesive was recommended by Cytec as the adhesive for HC/SS panel fabrication in OOA-VBO processing. This adhesive yielded the highest strength of $10,467 \pm 407$ kPa ($1,518 \pm 59$ psi) among all adhesives evaluated in this work. The FM 300K adhesive was designed by Cytec as an autoclave adhesive. Surprisingly, a high strength value of $8,618 \pm 407$ kPa ($1,250 \pm 19$ psi) was obtained for this adhesive from OOA processing.

As-received/Perforated FM 309-1M (TH-021010)



As-received/Perforated FM 300K (TJ-021210)



Core side

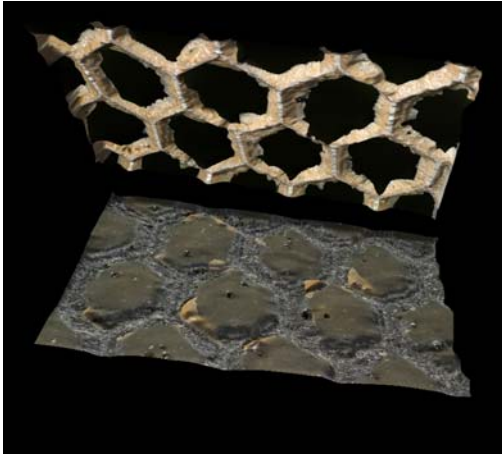
Face sheet side

Figure 5. Optical photographs of the fracture surfaces of FM 309-1M and FM-300K adhesives

Optical photographs of the fracture surfaces are shown in Figure 5. For the FM 309-1M adhesive, fillets were evenly adhered to the core walls and the hexagonal shapes of the fractured adhesive resin on the composite face sheet side were mostly intact with a few air bubbles entrapped inside. Pieces of adhesive that had broken away from the fillets on the cores were also evident on the composite face sheet side in the photograph.

For the FM-300K material, adhesive fillets were voidy and poorly distributed on the core sides. Voids in the fillets were due to foaming in the adhesive. The hexagonal shapes of the fractured adhesive resin on the composite face sheet side were mostly misshapen, with few air bubbles entrapped inside. Naked knit carrier fabric was left and visible on the composite face sheet side fracture surface. Some adhesive failure between the adhesive and the composite substrate was evident as well. Despite foaming, high FWT strength values were measured for this adhesive (Table 4).

**T40-800B/5320
/FM 309-1M**



**T40-800B/5320
/FM 300K**

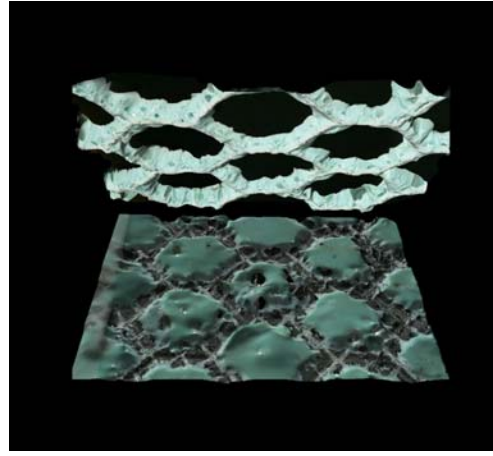


Figure 6. 3-D SEM microphotographs of the fracture surfaces on the composite face sheet sides.

3-D SEM photomicrographs of the fracture surfaces are compared in Figure 6. In each photograph the core side (top) and the composite face sheet side (bottom) were matched as mirror images. For the FM 309-1M adhesive, well adhered and void-free fillets on the core side were evident, while well defined, nearly void-free fractured adhesive residues on the composite face sheet side were seen. For the FM 300K adhesive, voids were present on both core and composite face sheet sides. Naked knit fabric carrier with adhesive pulled out from the composite substrate was also evident on the surface.

5. SUMMARY

Rheological and mechanical properties of the four adhesives evaluated in this work are summarized in Table 5 and plotted in Figure 7. HC/SS panels made from as-received adhesives were used in comparison. Adhesives with low minimum viscosities (AF-555M and FM-300K) tended to foam. The foaming, however, had marginal effect on strength values, presumably due to adequate surface wetting. Adhesives with a high minimum viscosity did not foam, and resulted in quite different FWT strengths and fracture failure modes between the FM 309-1M and XMTA-241/PM15 adhesives. It appears that a moderate minimum viscosity between 2,000 to 4,000 P is preferred as evident in the pre-treated AF-555M (TH-010510 in Table 2) and the FM 309-1M (TH-021010 in Table 4) adhesive results. In this viscosity range, the adhesive exhibited adequate fluidity for surface wetting under vacuum pressure, and the foaming reactions were sufficiently retarded/inhibited, resulting in high FWT strength values and the desired failure mode.

Table 5. Summary of adhesive properties*

Panel ID	Adhesive	Prepreg	Minimum Visc.^ (P)	Adhesive Foaming	FWT Strength, kPa (psi)	Fracture Mode^^, %
TH-021010	FM 309-1M	T40-800B/5320	4,200	No	10,467 ± 407 (1,518 ± 59)	90 Co/5 Ad/ 5 Ft
TH-011110	AF-555M	IM7/MTM 45-1	310	Yes	8,667 ± 538 (1,257 ± 78)	95 Co/5 Ft
TJ-021210	FM-300K	T40-800B/5320	1,000	Yes	8,618 ± 407 (1,250 ± 19)	50 Co/45 Ad/ 5 Ft
TH-110509	XMTA-241/PM15	IM7/MTM 45-1	7,100	No	4,730 ± 724 (686 ± 105)	5 Co/95 Ad

*Fresh adhesives were used

^Measured by following the respective cure cycle of the face sheet prepreg composites

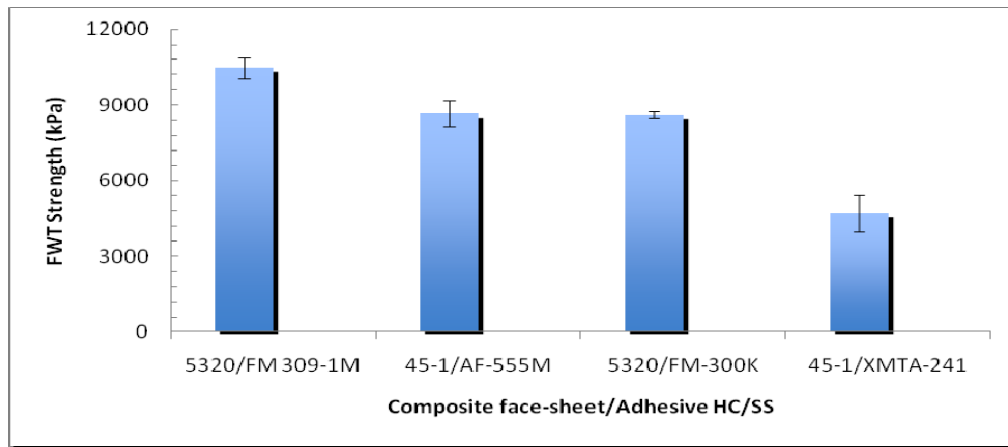


Figure 7. FWT strength of HC/SS panels with four different adhesives.

6. ACKNOWLEDGEMENT

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