Advancement of Out Of Autclave (OOA) Technology at Tencate Advanced Composites, USA

Henry Villareal, Scott Unger, Frank W. Lee

Tencate Advanced Composites, USA

18410 Butterfield Blvd.

Morgan Hill, CA 95037

1. ABSTRACT

Out of autoclave (OOA), under vacuum pressure only cured processing of composites has been one of the most important fields of interest in the composite industry in recent years. A great deal of time and resources has been invested by many academic institutions, government agencies and the composite industry to further the OOA technology. The advantages and disadvantages of this technology have been chronicled in many technical publications and presentations. Tencate Advanced Composites, USA (TCAC) has been very active in promoting and supporting the OOA technology for over 20 years. Over 5,000 airplanes used in the general aviation market were built with TCAC's BT250E-1 epoxy system and numerous UAV's were manufactured using both BT250E-1 and TC250 epoxy systems with OOA processes. A specially formulated OOA high temperature cyanate ester system (TC420) with over 500°F temperature resistance is being used in manufacturing large heat shields in space vehicle applications. As the demand for higher performance OOA systems continues to grow and the expectation to match or even exceed the properties of autoclave cured systems amplifies, more technical improvements in this area have been investigated and developed within TCAC and also through continuous collaboration with the composite industry. This paper is a chronicle of a stage by stage advancement of TCAC's new and exciting epoxy OOA materials and process technologies. These new prepreg systems deliver much lower void contents after cure, are easier to inspect under NDI Pulse Echo method and provide better overall hot-wet properties after moisture saturation and better impact resistance.

2. INTRODUCTION

The use of composite materials in aero structures has steadily grown over the past few decades however the recent trend, largely driven by fuel efficiency and carbon emission reductions, is driving design engineers from more traditional metal structures to those made from composites. Carbon and fiberglass composite structures used in commercial aerospace are growing rapidly and the advent of the 787 and A350 aircraft has resulted in a paradigm shift in the volume of carbon composite material usage in critical aero structures to > 50% by weight. Naturally, as the volume of usage of composites increases to high levels, manufacturers have to look toward low cost processes, lean techniques and automation to reduce costs and increase the pounds of structure per hour that can be reduced. Manufacturers around the globe are investigating many variants of Out of Autoclave (OOA) processes with a focus on cost reduction. OOA can be related to many processes such as RTM, VARTM, In situ thermoplastic composite consolidation, thermoforming of thermoplastic composites, et cetera; however the focus of this paper will be upon OOA processing capabilities of TenCate Advanced Composites' next generation thermosetting prepreg systems.

TenCate Advanced Composites (Formerly Bryte Technologies) has its roots in OOA prepreg materials as Bryte was founded based upon a proprietary OOA centric prepregging process. Through that process and its formulating knowledge TenCate has been supplying production ready OOA systems into aerospace applications for over 20 years. These OOA prepreg systems are capable of producing extremely low void (< 2%), nondestructively inspectable composite structures using high volume, production ready oven/vacuum bag processing methods. To date greater than 5,000 FAA certified aircraft and hundreds of production unmanned aerial vehicles (UAV's) and high value, high reliability space vehicles have been built utilizing TenCate's OOA prepreg technology. This OOA legacy is now fueling TenCate's next generation of OOA prepreg systems which are improved mechanical, environmental capabilities, while not hampering the low void, NDI inspectable attributes that TenCate's OOA materials have traditionally offered for the company's customers.

TenCate's new, advanced OOA prepreg systems are focused on increasing resin system performance for demanding aerospace environments. To accomplish this, TenCate has engineered these next generation thermosetting resins to exhibit extremely high hot wet property retention, the key design metric for all polymer based composites, increase toughness and increased temperature resistance. The culmination of these efforts has resulted in the creation of 2 new epoxy products which are available as prepreg with all common fiber reinforcements in unidirectional tape, woven and multi-axial formats.

System	Description	Cure Temperature
TC275	Epoxy Prepreg	275°F - 350°F
TC350-1	High Toughness Epoxy Prepreg	275°F cure with 350°F post cure

These resin systems will enable engineers to push the design envelope for OOA prepreg materials to the same levels as those seen for traditional autoclave cured prepreg systems and set the stage for manufacturing efficiencies that will drive recurring costs down, while minimizing scrap.

3. EXPERIMENTATION

Various experiments were conducted to evaluate Tencate's latest OOA systems and technology. Unidirectional tape and fabric systems of TC275 OOA prepreg system are covered in this paper in details, and some interesting results of TC350-1 which is a high impact resistant system is discussed as well.

3.1 Thermal Analysis

Thermal analysis of resin and laminates were done with the following methods: rheology , glass transition temperature, degree of cure and gel time.

3.1.1 Rheology

The rheology data was collected using Texas Instrument AR2000 ex. For Dynamic test a 1.0 mm gap was used with 2.0% strain at 1 Hz. frequency. For Isothermal test a 0.6 mm gap was used with a shear rate of 1.92 1/s.

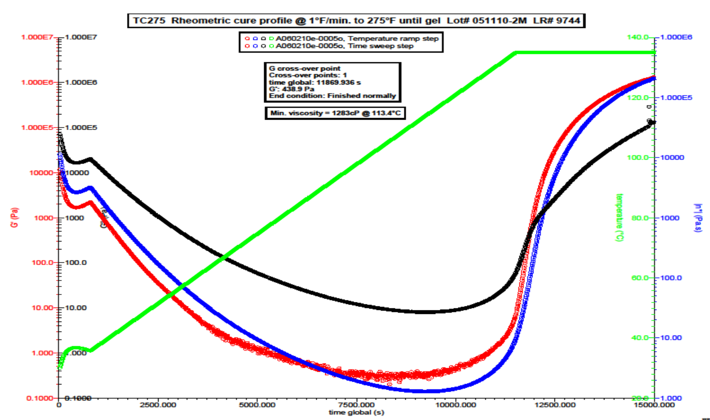


Figure 3.1 Dynamic curve of TC275

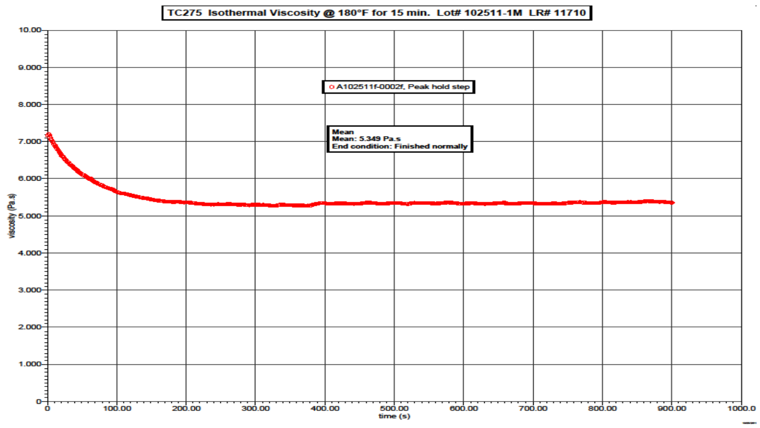


Figure 3.2 Isothermal curve of TC275

3.1.2 Glass Transition Temperature

The glass transition temperature data was tested with Perkin Elmer Dynamic Mechanical Analyzer 7e set – up with a static force of 110 mN, dynamic force of 100 mN at a frequency of 1.0 Hz. Glass Transition Temperature were measured on both dry and wet samples after conditioned at a humidity chamber.

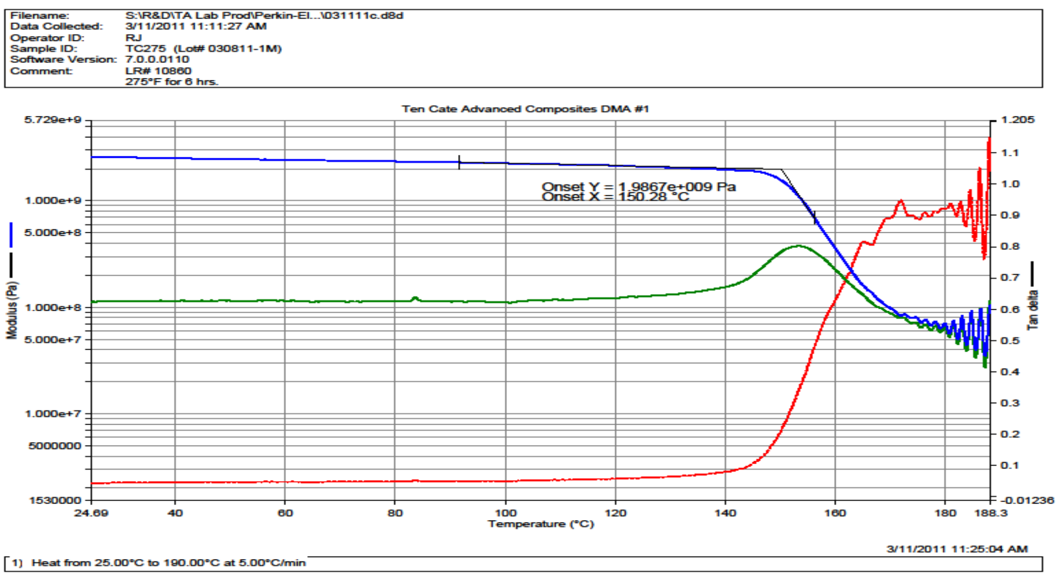


Figure 3.3 Neat Resin Glass Transition Temperature of TC 275 after cured at 275F for 6 hours (DMA)

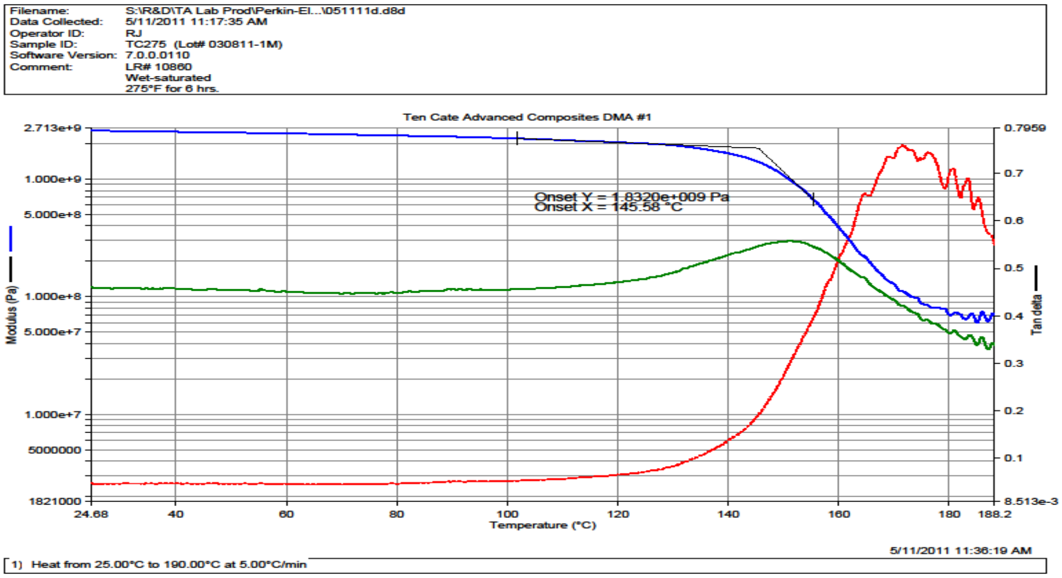


Figure 3.4 Neat Resin wet Tg of TC 275 after moisture saturation (DMA)

3.1.3 Degree of Cure

Degree of cure data was verified using Perkin Elmer Diamond DSC and TA Instrument DSCQ20. A heat rate of 10°C/min was used. Multiple cure profiles were performed to determine the optimum cure of the systems.

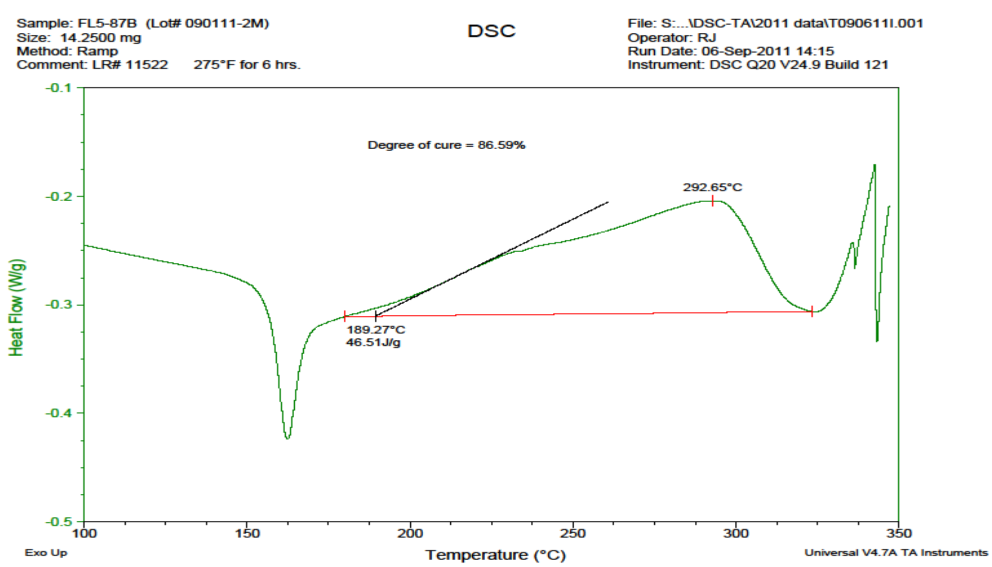


Figure 3.5 Degree of cure curve of TC275 cured at 275F for 6 hours

3.1.3 Gel Time

Gel time was determined using Omega CN76000 set at specified plate temperature.

3.2 Voids and Porosity Analysis

Void content of the laminates was done using three methods: Ultrasonic Non Destructive Inspection (NDI), microscopy and fiber volume by acid digestion.

3.2.1 Ultrasonic NDI

Ultrasonic non- destructive (NDI) was used for the C-scanning of composite laminates. It provides images (amplitude, thickness and B-scan) and amplitude histograms for classifying laminate quality. NDT Automation ultrasonic immersion scanner was used and UTwin software for the data analysis. All laminates were inspected after cure using pulse echo 5 mHz transducer.

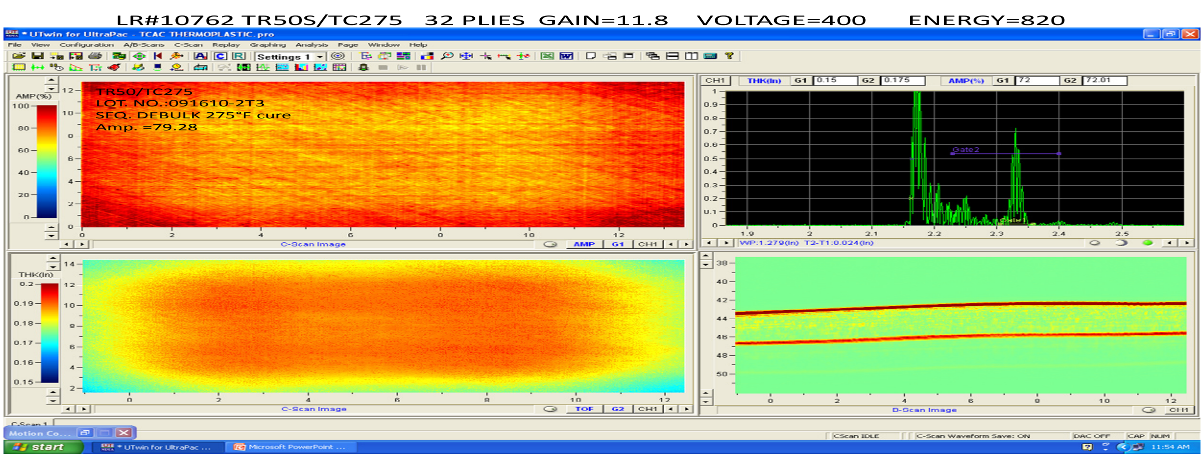


Figure 3.6 32 ply quasi isotropic laminate made with TR50/TC275 uni-tape shows the amplitude and a clear view of the back wall

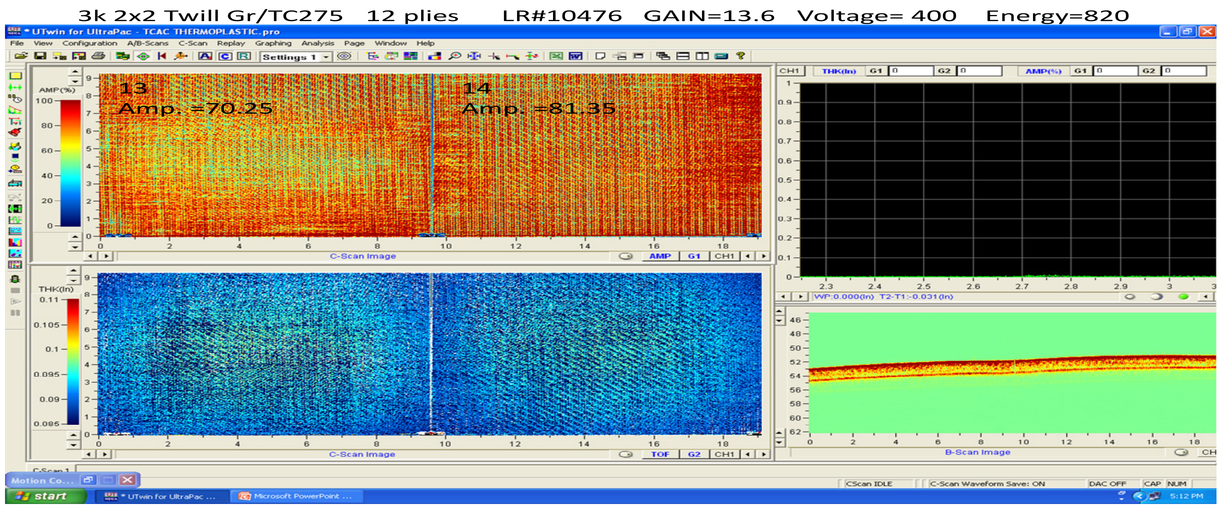


Figure 3.7 12 ply laminate made with 2x2 twill/TC275 fabric shows the amplitude and a clear view of the back wall

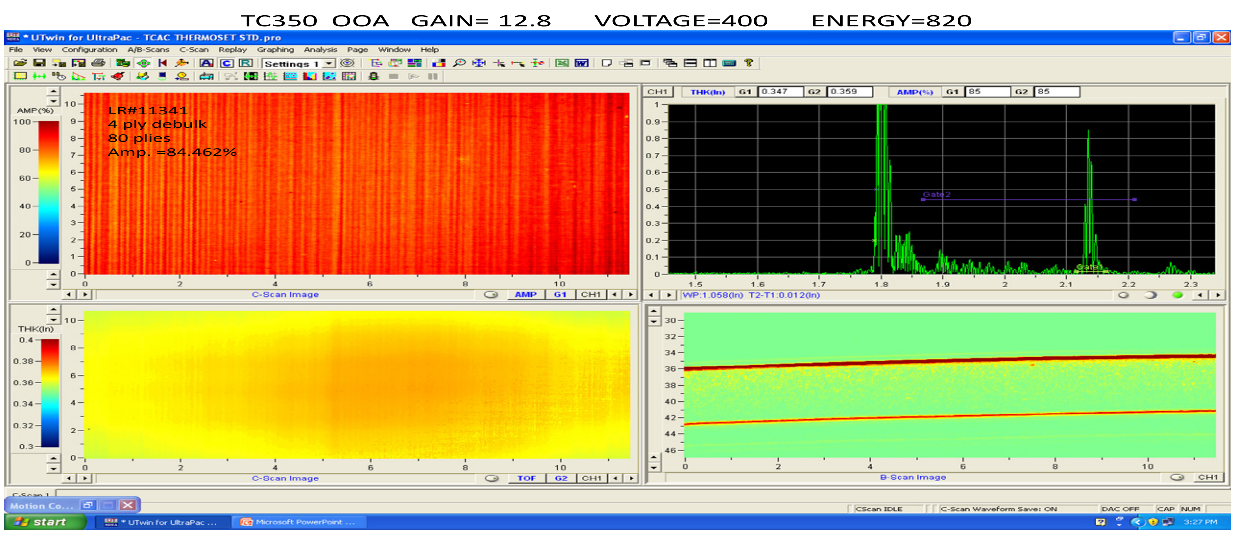


Figure 3.8 80 ply laminate made by IM7/TC350-1 tape shows the amplitude and a clear view of the back wall

3.2.2 Microscopy

All cross section samples for microscope inspection were first potted and cured. They were then polished using Ecomet 4000 variable speed grinder- polisher by Buehler with polishing medium polycrystalline diamond suspension solution with 9 to 0.05 microns particles. Pictures were taken at different magnifications using an Olympus BHT digital camera. The percent of void in each sample was evaluated using the Image J software analyzer.

3.2.3 Fiber Volume by acid digestion.

Acid digestion was performed with the use of Mars Machine by CEM Corporation. Samples were placed inside a plastic vessel with 40 ml. of concentric nitric acid and run through a pre-determined cycle.

3.3 Debulking and Sample Fabrication

The debulking procedure for OOA laminates was one of the important factors to achieve high laminate quality. The prepregs were debulked every 2 plies to 4 plies to minimize the trapped air within the system before curing. The layup and cure of each product were done per recommended cure cycles unless otherwise specified. All data was generated using vacuum bag process only and laid up using LU-1.5 reduced caul plate method (Fig. 3.1) for TC275 and LU-1 method (Fig. 3.2) for TC350-1. Autoclave cured laminates, which were used for comparison, were made with the same debulking procedures.

3.4 Resin Preparation, Mixing and Coating

Proper degassing procedure was used during the preparation of neat resins to minimize the entrapped air in the resins. Vacuum level was maintained at least 28 in. of Hg or higher during resin mixing to obtain optimum degassing of the resins at the mixing temperature range.

3.5 Prepregging /Laminating

Prepregging was performed using a refined and specialized film and calendaring methodology which produces prepregs optimized for use in out of autoclave processes.

3.5.1 Prepreg Physical Testing

The appearance and the quality of the prepregs were validated by via the resin flow, resin content and volatile tests,

3.5.2 Laminate Fabrication

All laminates fabricated for each mechanical test was based on the ASTM test method as listed.

3.5.3 Debulk, Lay-up and Cure

An excellent debulking procedure was necessary to minimize entrapped air between plies. Pulled vacuum was at least at 27 in. Hg. TC275 system was debulked every 4 plies for 5-10 min. each (Figure 3.9) until the needed plies for the sample was achieved while TC350-1 was debulked every 4 plies for 15 min. until all the needed plies were laid up for the samples . For TC 275 and TC350-1 woven fabric systems, both systems were debulked every 2 plies for 5 – 10 min.

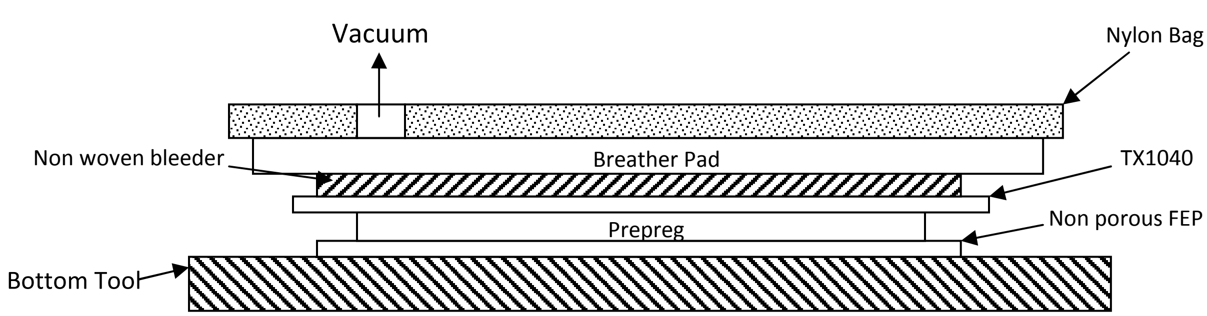


Figure 3.9

An additional ply of porous Teflon coated glass(TX1040) and 1 ply of non woven bleeder were used to help the removal of entrapped air and it was replaced after being used for 2 -3 times of debulking

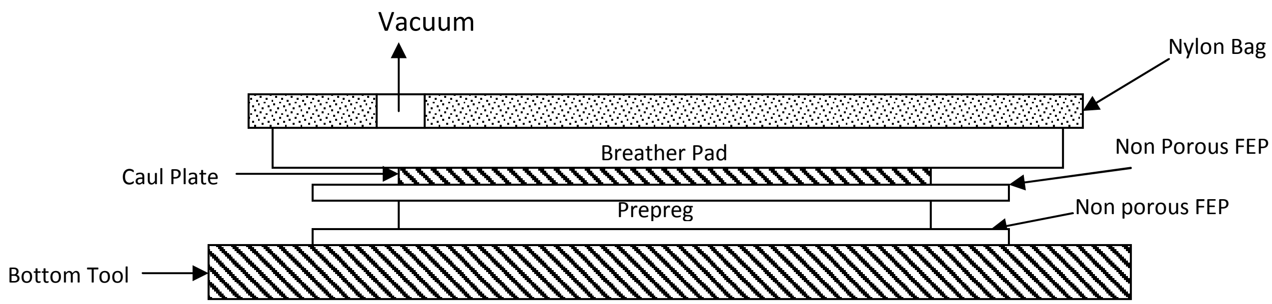
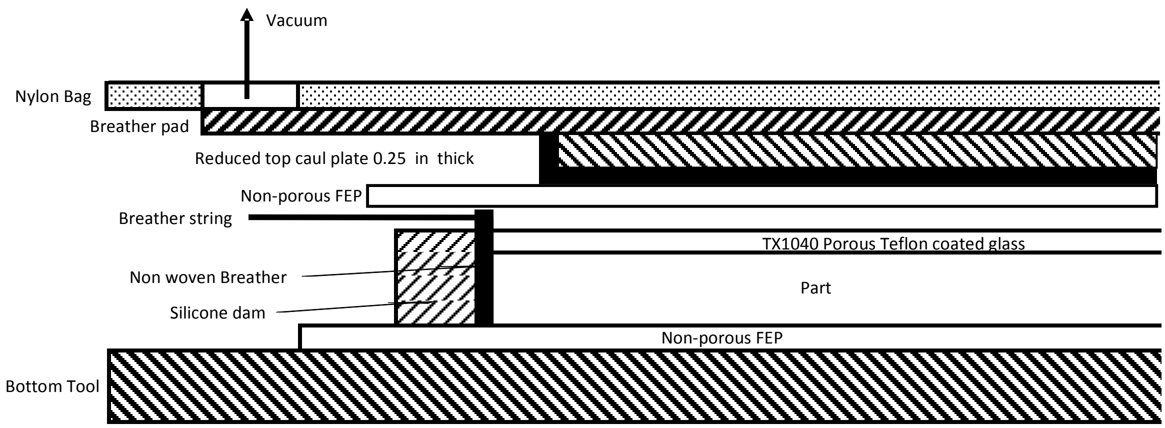


Figure 3.10

After the debulking step, TC275 system was processed using the following bagging technique (Figure 3.11) to make all the samples for mechanical testing.

Thermocouple wires were used to monitor and record the temperature and a vacuum sensor was used to monitor the vacuum during the curing process.



Note: The breather string must be in the edge of the part, must not lay on the top of the panel and must extend out past the seal to touch the breather pad material as shown below

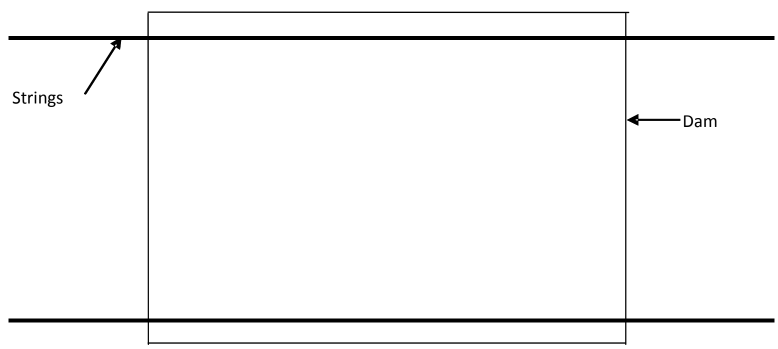


Figure 3.11 (LU-1.5 method)

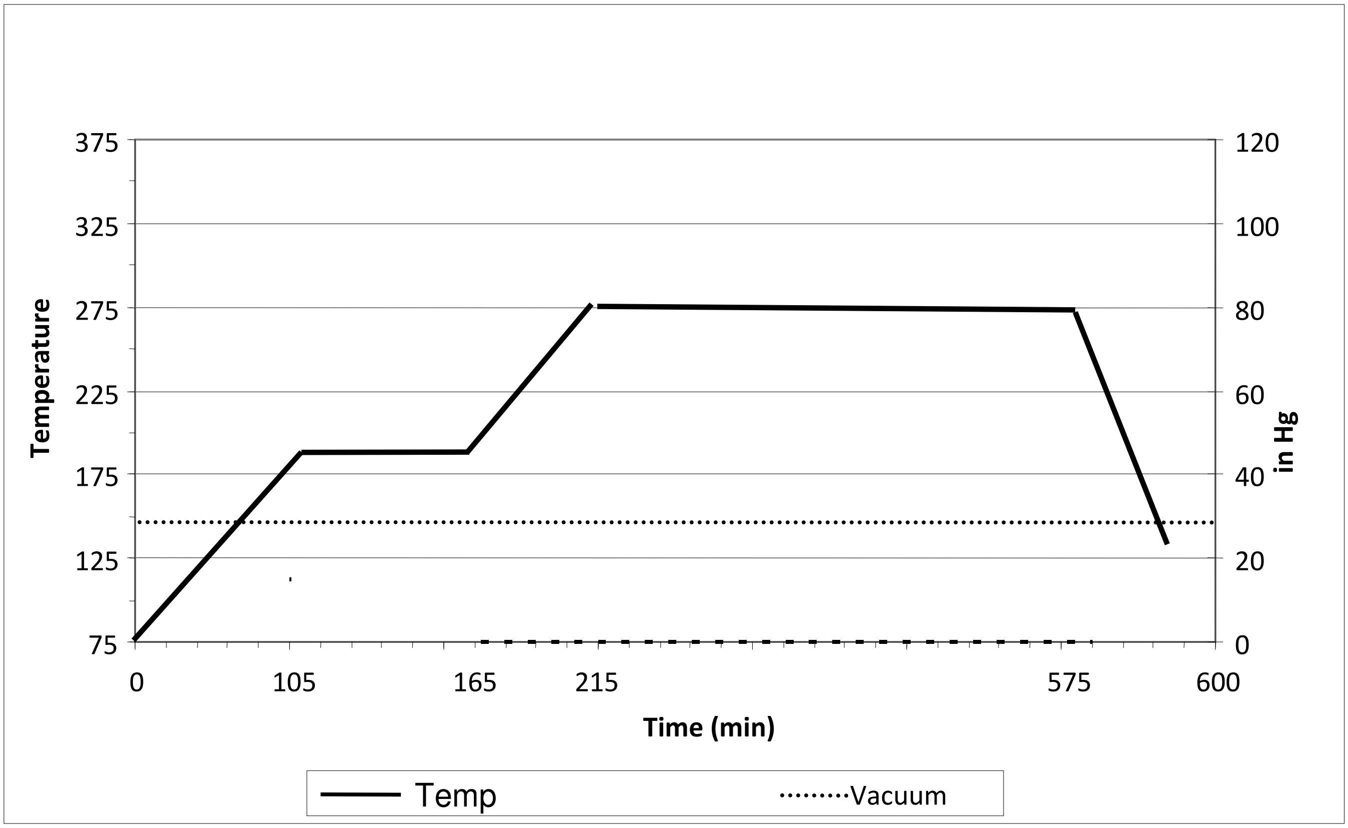
TC350-1 was laid up with the regular LU-1 bleed schedule which was similar to LU-1.5 bleed RCP with the exclusion of TX1040 and the top caul plate used was the same size of the actual uncured laminate. The vacuum bag integrity was checked prior to to starting cure cycle and leak rate shall not exceed 5 in. Hg in 5 minutes.

TC 275 cure cycle used to make all the mechanical test samples was listed as the following:

a. Pull vacuum (minimum 28 in. Hg)
b. Heat at 1°F/min to 180± 5°F
c. Hold at 180°F± 5°F for 1 hour
d. Heat at 2°F/min to 275± 5°F
e. Hold at 275°F± 5°F for 6 hours

f. Cool at 5 to 7°F/min to <140°F
g. Removed parts from oven below 120°F

Figure 3.12 TC275 Cure Cycle



3.5.4 Machining, Drying and Conditioning

All cured laminates were C-scanned by Ultrasonic NDI scanner to assess the quality. All scanning results were evaluated and at times analyzed by cross section method.

All mechanical test specimens were machined according to specifications based on the ASTM methods. Before mechanical testing, all specimens were dried at 212°F for 1 hour. After drying, specimens were kept at room temperature environment of 70±10 °F or in a desiccator until the tests.

For environmental conditioning, all specimens were put inside a humidity chamber at 145°F and 85% humidity after being dried at 212° F for one hour, unless specified. The moisture saturation was achieved when the traveler samples were observed to have change less than 0.05% for two consecutive 7 day periods.

3.5.5 ***Mechanical Testing Methods***

ASTM test methods were used for all the testing of laminate samples. An average of 5 specimens for each test was done. Specimens were tested at different environment conditions and defined as:

RTD = 70±5°F, room temperature dry
ETD = 180±5°F dry
ETW = 180±5°F, wet (saturated)
CTD = -65±5°F, dry

Type and ASTM Method Used

Tests	ASTM Method
Tensile Strength & Modulus	ASTM 3039
Compression Strength& Modulus	ASTM D 695
Compression Combined Loading Test	ASTM D 6641
Short Beam Shear	ASTM D2344
Flexural Strength & Modulus	ASTM D 790
In Plane- Shear Strength & Modulus	ASTM D 3518
Open Hole Tensile Strength	ASTM D 5766
Open Hole Compression Strength	ASTM D 6484
Compression After Impact test	ASTM D 7136/7137

4. Discussion and Results

A major source of voids in composite laminates, especially processed with out of autoclave method (OOA), is entrapped air (1). Voids in the resultant laminates will lead to reductions in strength, high variability in design allowable data and will create major issues with non-destructive inspection techniques should the void levels go beyond 2%. It is critical to design prepreg systems and lay-up methods to remove all the entrapped air effectively and efficiently. A prepreg system , which provides pathway for more rapid removal of air, is desired (2). An excellent debulking procedure during lay-up is essential to minimize trapped air between plies. During the cure process, air must be removed from all locations in the part through breathable edge dams prior to the gelation of the resin (3). Those above mentioned criteria are as important as the formulation of a good OOA prepreg system which includes resin and fiber.

4.1 TC 275

TC275 prepreg system was developed to meet all the criteria in OOA process and also to meet the demanding environmental conditioned requirements which are to exhibit very high hot wet property retention. The first study of the system was to develop a good cure profile. The system was cured at 4, 5 and 6 hours at 275°F and as the data presented in Table 4.1. These data were very comparable to each other. From Table 4.2, the 4 hr. cured laminate samples were observed to pick up slightly higher moisture content compared to both 5 and 6 hour cures at 275°F. This might indicate that the 4 hour cure sample at this cure temperature and time did not offer the most optimal cure level. After careful evaluation of these data we observed that this system can be cured at this time span, but the 6 hour cure seemed to be offer slightly better hot/wet mechanical numbers. The system was also cured in a temperature range at 275°F (6 hours), 300°F (3 hours) and 350°F (2 hours). The resultant laminates were tested. The mechanical properties and glass transition temperatures (Tg) were listed in Table 4.3 and Table 4.4 for tape and fabric respectively. The higher cure temperatures gave slightly higher glass transition temperatures. The mechanical properties, however, were very close to each other. The higher cure temperature laminates absorbed a higher amount of moisture upon saturation. This was due to the higher free volume expansion of the matrix at higher temperature cures. It was observed, based on the very similar dry, hot and hot/wet mechanical properties, that this system could be processed at 275°F to 350°F with the specific cure times. It is a matter of choice for the users based on their own preferences, capabilities and tool requirements.

TC275 was found to have very low moisture absorption after saturation. The moisture saturation percentage was under 0.4% for laminates and <1.2% for neat resin cured after 6 hours at 275°F. We conditioned all laminate samples at 145°F and 85% humidity and the neat resin was conditioned at 160°F and 85% humidity. The key to have a very good hot/wet resistant resin system or prepreg system is to have low moisture uptake after moisture exposure. The typical epoxy resin system picks up about 3-4% by weight of moisture at saturation. The Tg value retention of this system after moisture saturation was about 90%.

The mechanical properties of laminates made from TR50S/TC275 tape and 2x2 twill Gr/TC275 woven prepregs were cured at 275°F for 6 hours (Table 4.5 and Table 4.6) The data showed again very high hot/wet retention values, between mid-80 to upper 90% retention. Large thick and thin parts were made via OOA process with TC275. These parts are shown in Figure 4.1. The NDI C-scan of those panels showed very clear back wall transmission and high amplitude. The polished cross sections of these panels as shown in Figure 4.2 were usually below 0.5%. It is a moderately impact resistant system, based on the CAI number.

Mechanical Tests	4 hr. Cure		5 hr. Cure		6 hr. Cure	
	RT	HWT	RT	HWT	RT	HWT
Compression Strength(ksi)	200.0	194.0	191.1	204.1	207.7	194.2
Compression Modulus(msi)	17.5	15.3	18	17.5	18.5	16.6
Short Beam Strength(ksi)	12.4	8.6	12.9	9.2	12.9	8.6
Flexural Strength(ksi)	240.3	169.5	244.8	187.9	240.6	197.5
Flexural Modulus(msi)	12	11	12	11	13	13
Tensile Strength(ksi)	306.2	283.0	325.3	274.7	310.9	303.7
Tensile Modulus(msi)	18.8	11.5	19.6	10.7	17.6	18.9
TG by DMA – (°C)	147.5 (dry)	130.9 (wet)	146.1 (dry)	132.0 (wet)	144.9 (dry)	128.9 (wet)

Table 4.1 Material Property Summary 275°F Cure (All Raw Data)

Cure Profile	% Moisture
4 hr. Cure @ 275°F	0.4218
5 hr. Cure @ 275°F	0.3865
6 hr. Cure @ 275°F	0.3850
3 hr. Cure @ 300°F	0.4693
2 hr. Cure @ 350°F	0.4929

Table 4.2 Moisture Absorption Summary of TR50S/TC275 Unidirectional Tape

***Humidity Chamber Setting @ 85%RH and 145 F**

Mechanical Tests		275° Cure (LR# 10210)		300° Cure (LR# 10229)		350° (LR# 10246)	
		0°	90°	0°	90°	0°	90°
TS (ksi)	**Room Temp.**	344.1	8.0	318.6	9.4	329.8	8.6
	180°F(dry)	382.9	6.62	340.1	6.1	337.9	6.8
	14 Days HW	344.4	6.9	344.5	6.5	350.7	6.2
	21 Days	345.3		332.8		342.6	
	30 Days	323.7		317.4		335.2	
	Saturated	336.1		303.5		331.9	
	-65°F	340.7		314.2		348.3	
TM (Msi)	**Room Temp.**	19.4	1.1	17.6	1.1	19.5	1.1
	180°F(dry)	21.3	1.0	20.8	1.0	21.0	0.9
	14 Days HW	21.0	1.0	20.4	0.8	21.4	0.9
	21 Days	20.6		21.0		20.5	
	30 Days	20.8		21.2		21.0	
	Saturated	20.9		21.8		20.6	
	-65°F	18.8		17.3		19.3	
CS (ksi)	**Room Temp.**	216.5		210.0		187.0	
	180°F(dry)	205.1		207.1		195..5	
	14 Days HW	202.7		249.2		187.2	
	21 Days	214.6		212.6		213.9	
	30 Days	213.3		233.8		205.2	
	Saturated	196.7		192.3		192.4	
	-65°F	219.8		244.8		213.3	
CM (Msi)	**Room Temp.**	19.2	1.4	20	1.6	18.4	1.4
	180°F(dry)	19.5	1.2	21.2	1.2	17.6	1.1
	14 Days HW	17.9	1.0	17.7	1.0	17.0	0.9
	21 Days	17.3	0.9	18.2	1.0	16.6	0.9
	30 Days	17.7	1.2	17.1	1.2	17.5	1.4
	Saturated	18.3		16.7		14.8	
	-65°F	18.8	1.6	19.3	1.4	18.5	1.3
FS (ksi)	**Room Temp.**	243.7		276.3		235.8	
	180°F(dry)	201.9		218.6		195.2	
	14 Days HW	197.1		191.3		190.2	
	21 Days	207.9		196.4		188.1	
	30 Days	177.0		172.7		170.0	
	Saturated	205.8		192.2		195.6	
	-65°F	279.1		299.9		301.6	
FM (Msi)	**Room Temp.**	12.5		14.2		12.5	
	180°F(dry)	12.3		13.4		11.7	
	14 Days HW	12.9		12.5		12.0	
	21 Days	12.3		11.9		11.5	
	30 Days	11.6		11.6		11.7	
	Saturated	12.8		11.8		12.0	
	-65°F	12.2		12.7		12.9	
SBS* (ksi)	**Room Temp.**	12.9		12.5		13.3	
	180°F(dry)	9.5		9.5		9.9	
	14 Days HW	8.3		8.8		8.8	
	21 Days	8.3		8.3		8.6	
	30 Days	8.2		8.10		8.5	
	Saturated	8.5		8.4		8.5	
	-65°F	17.2		16.6		16.0	
CS 6641 (ksi) 90/0/90	**Room Temp.**	232.6		224.3		236.0	
	180°F(dry)	225.7		220.3		216.2	
	14 Days HW	198.5		209.0		213.7	
	21 Days	218.3		212.4		199.1	
	30 Days	187.9		208.9		201.9	
	Saturated	205.8		204.5		202.8	
	-65°F	271.6		269.6		278	
Tg by DMA	**Room Temp.**	144.8°C		148.1°C		169.6°C	
	14 Days	132.2°C		133.3°C		150.1°C	
	21 Days	130.6°C		134.8°C		149.9°C	
	30 Days	129.3°C		137.3°C		146.2°C	
	Saturated	128.9°C		136.9°C		149.8°C	

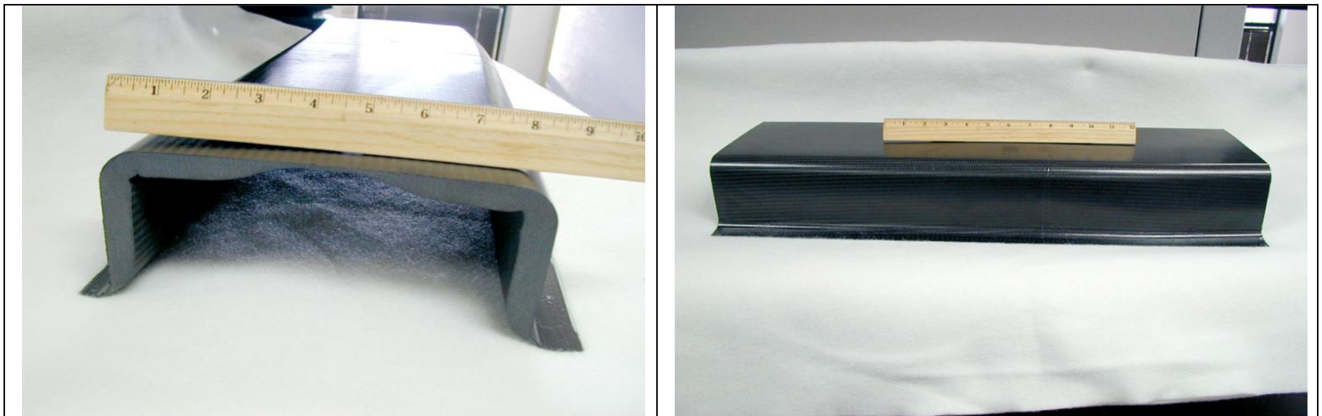
Table 4.3 Testing results of TR50S, 15K/TC275 Unidirectional Tape Normalized to 60% FV

***SBS data not normalized**

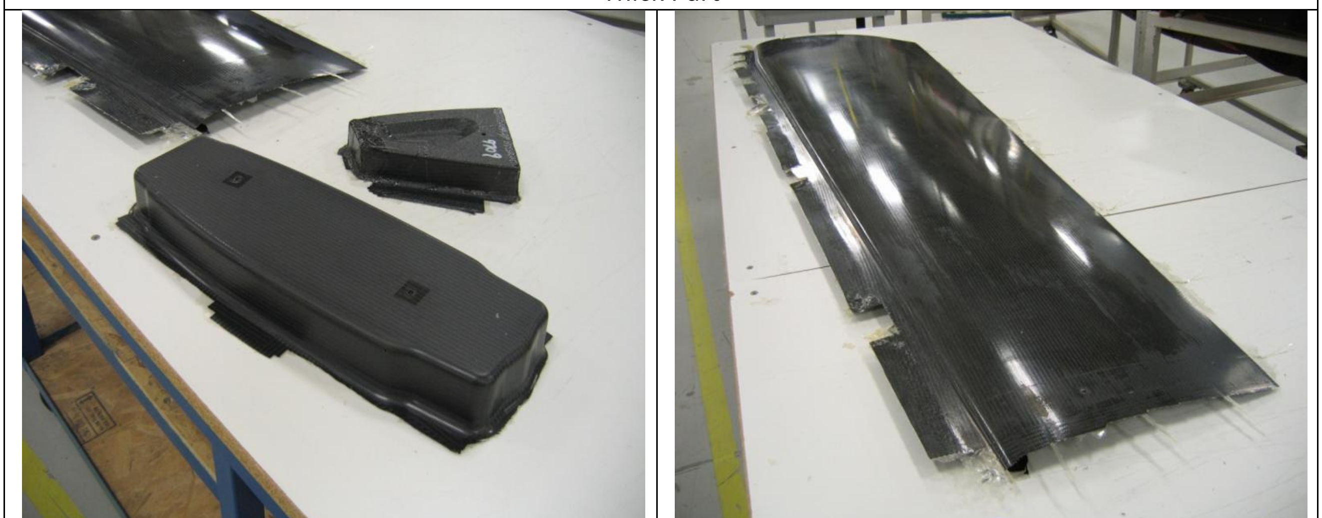
Mechanical Tests		275° Cure (LR# 10210)		300° Cure (LR# 10229)		350° (LR# 10246)	
		0°	90°	0°	90°	0°	90°
TS (ksi)	Room Temp.	146.1	137.0	141.4	140.9	146.0	133.9
	180°F(dry)	151.7	142.3	143.2	143.7	142.8	137.9
	21 Days HW	152.9	144.8	139.0	139.8	140.8	143.3
	Saturated	145.5	149.7	145.0	141.5	134.4	141.8
	-65°F	147.0	143.1	135.6	139.3	127.9	126.6
TM (Msi)	Room Temp.	10.0	10.8	10.3	10.1	10.5	11.0
	180°F(dry)	10.8	10.0	10.8	11.0	10.7	10.7
	21 Days HW	10.8	10.6	10.7	10.9	11.1	11.0
	Saturated	10.8	11.1	10.8	11.0	10.8	11.1
	-65°F	9.2	9.4	9.6	8.8	9.5	9.3
CS (ksi)	Room Temp.	114.1	118.6	114.5	117.9	114.2	119.3
	180°F(dry)	105.8	108.0	107.0	108.4	104.6	113.1
	21 Days HW	101.9	100.4	101.6	104.2	106.1	104.2
	Saturated	98.5	98.8	103.4	99.3	103.4	97.1
	-65°F	122.5	126.5	125.0	125.7	126.3	127.1
CM (Msi)	Room Temp.	9.6	9.5	9.3	9.4	9.5	9.6
	180°F(dry)	8.9	8.9	9.0	9.2	8.9	9.1
	21 Days HW	8.8	9.0	9.0	8.9	8.6	8.9
	Saturated	8.6	9.2	8.1	8.8	8.8	9.1
	-65°F	9.8	9.5	9.2	9.1	9.66	9.8
FS (ksi)	Room Temp.	150.8		148.9		154.4	
	180°F(dry)	125.5		123.9		130.1	
	21 Days HW	115.6		118.1		114.8	
	Saturated	115.0		116.7		117.5	
	-65°F	169.8		176.1		176.4	
FM (Msi)	Room Temp.	7.7		7.8		7.9	
	180°F(dry)	7.4		7.4		7.4	
	21 Days HW	7.4		7.4		7.5	
	Saturated	7.2		7.3		7.3	
	-65°F	7.7		7.9		7.9	
SBS* (ksi)	Room Temp.	10.5		10.0		10.5	
	180°F(dry)	8.1		7.7		8.3	
	21 Days HW	7.1		7.4		6.9	
	Saturated	7.4		7.2		7.2	
	-65°F	12.7		12.4		12.4	
CS 6641 (ksi)	Room Temp.	100.0	99.4	102.3	96.6	107.8	107.1
	180°F(dry)	87.6	84.4	89.3	89.6	86.4	86.8
	21 Days HW	79.4	84.3	85.0	81.6	82.4	84.0
	Saturated	71.9	87.1	78.3	83.9	80.0	84.0
90/0/90	-65°F	134.9	126.2	140.3	134.7	126.5	130.8
Tg by DMA	Room Temp.	145.3°C		152.8°C		164.0°C	
	21 Days	131.8°C		1391°C		150.7°C	
	Saturated	134.7°C		136.4°C		149.1°C	

Table 4.4 Testing result of G30-500 3k, 2x2 Twill GR/TC275 Fabric Normalized to 60% FV

***SBS data not normalized**



Thick Part



Thin Parts

Figure 4.1 Parts made by TC275 OOA System

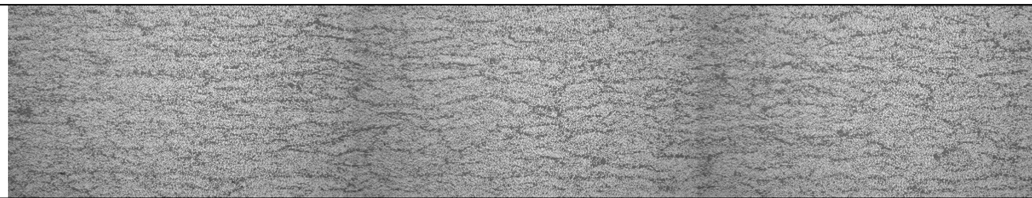
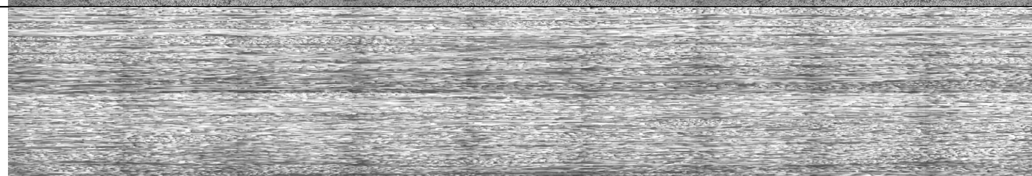
TR50S/TC275 0° LR# 10041 Void = 0.063%	
TR50S/TC275 90° LR# 9881 Void = 0.198%	
G30-500 3k, 2x2 Twill GR/TC275 90° LR# 9867 Void = 0.345%	
G30-500 3k, 2x2 Twill GR/TC275 90° LR# 9867 Void = 0.057%	

Figure 4.2 Microscopic Cross-Section of TC275 OOA System

***10x 5 magnifications**

Mechanical Tests	RTD	ETD	ETW	CTD
0° Tensile Strength (ksi)	344.1	382.9	336.1	340.7
0° Tensile Modulus (msi)	19.5	21.3	20.9	18.8
90° Tensile Strength (ksi)	7.99	6.6	6.9*	NA
90° Tensile Modulus (msi)	1.1	0.96	1.0*	NA
0° Compression Strength (ksi)	216.5	205.1	196.7	219.8
0° Compression Modulus (msi)	19.2	19.5	18.3	18.8
0° Flex Strength (ksi)	243.7	201.9	205.8	279.1
0° Flex Modulus (msi)	12.5	12.2	12.8	12.2
CS 6641(ksi)	232.6	225.7	205.8	271.6
Short Beam Shear (ksi)	12.9	9.5	8.5	17.2
Open Hole Tensile (ksi)	67.6	70.7		
Open Hole Compression(msi)	39.6	35.1		
In Plane Shear Strength (ksi)	11.9	11.0		
In Plane Shear Modulus(msi)	0.447	0.475		
CAI Strength (ksi) 1150 in-lb	35.7			
CAI Strength (ksi) 1500 in-lb	21.1			

Table 4.5 Testing result of TR50S/TC275 Unidirectional Tape

***14 days in the humidity chamber (not saturated)**

Mechanical Tests	RTD	ETD	ETW	CTD
0° Tensile Strength (ksi)	146.1	151.7	145.5	147.0
0° Tensile Modulus (msi)	10.1	10.8	10.8	9.2
90° Tensile Strength (ksi)	137.0	142.3	149.7	143.1
90° Tensile Modulus (msi)	10.8	10.8	11.0	9.4
0° Compression Strength (ksi)	114.1	105.8	98.5	122.5
0° Compression Modulus (msi)	9.6	8.9	8.6	9.8
90° Compression Strength (ksi)	118.6	108.0	98.8	126.5
90° Compression Modulus (msi)	9.5	8.9	9.2	9.5
0° Flex Strength (ksi)	150.8	125.5	115.0	170.0
0° Flex Modulus (msi)	7.7	7.4	7.2	7.7
Short Beam Shear (ksi)	10.5	8.1	7.4	12.7
CAI Strength (ksi) 1500 in-lb	34.2			

Table 4.6 Testing results of G30-500 2x2 Twill Gr/ TC275 Fabric

4.2 TC 350-1

The TC350-1 technology evolved from one of our standard toughened 350°F cured epoxy prepreg systems, TC 350. It was intended to develop a higher toughness epoxy system which could be cured with an out of autoclave (OOA) process. Due to some tooling constraints for some potential customers we have also developed and established a lower temperature cure profile which can be cured initially at 275F and then free standing post cured at 350°F for 2 hours. The data for this study was presented in Table 4.7. Based on the data established up to this point, it is recommended that TC 350-1 could initially be cured at 275°F for 3 hours via OOA process and then free standing post cured at 350°F for 2 hours.

As it has been well documented, the debulking step is very important to make good panels. Great efforts were done to investigate this step and maximize the outcome of the laminates. Table 4.8 shows that different attempts were used to investigate this important step. We have tried 2 ply, 4 ply and RT and warm debulking within 10-15 minutes and the resultant laminates were observed to be all very good and similar. It was observed that this system is very user friendly, in handling and shop life.

We compared the OOA cured data vs. autoclave cured data in Table 4.9. The values were similar if not identical. This database showed that this system was capable to be processed via OOA to obtain autoclave cured laminate properties. The polished cross sections of both IM7/TC350-1 tape and AS4 PW/TC 350-1 as observed in Figure 4.3 showed both systems had very minimum voids (<0.4%). Part of the reasons for this excellent data generated in the OOA process and was comparable to the autoclave process of the same system was due to the very low void content after OOA cure. As it is quite clear to very faithful participant of the OOA technology, the debulking step is very important to make good panels. Great efforts were done to investigate this step and maximize the quality of the laminates. Table 4.8 shows that different attempts were used to investigate this important step. We have utilized 2 ply, 4 ply and RT and warm debulking within 10-15 minutes and the resultant laminates were observed to be all very good and similar. It was observed that this system is very robust.

The compression after impact (CAI) value when cured under autoclave environment yield very good values (37 ksi, 1500 in-lb) and the CAI value of TC350-1 processed by OOA is being tested by an accredited third party test lab. The value will be available at the SAMPE presentation. All the environmental (160F and 85% humidity) conditioned data are being generated. Thick laminates of up to 80 plies were made with very clear C-scan back wall due to very minimum voids (Figure 3.8). Large and complex parts are being made both internally and externally.

Cure Temperature	SBS(ksi)	TG(°C)	Degree of Cure (%)
275°F for 3 hrs	17.8	147.6	66.8
275°F for 5 hrs	18.3	156.8	71.6
275°F for 3 hrs then PC to 350°F for 1 hr	19.9	197.2	82.4
275°F for 5 hrs then PC to 350°F for 1 hr	20.1	195.5	85.7
275°F for 3 hrs then PC to 350°F for 2 hrs	20.7	201.6	84.8
275°F for 5 hrs then PC to 350°F for 2 hrs	20.1	209.9	84.6
350°F cure for 2 hours	20.1	208.0	84.7

Table 4.7 OOA Cure Profile Study of TC350-1

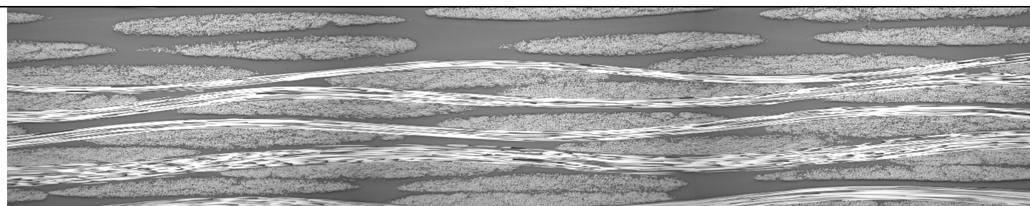
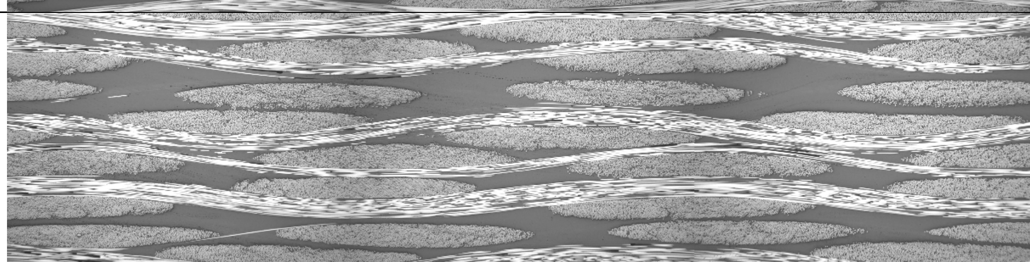
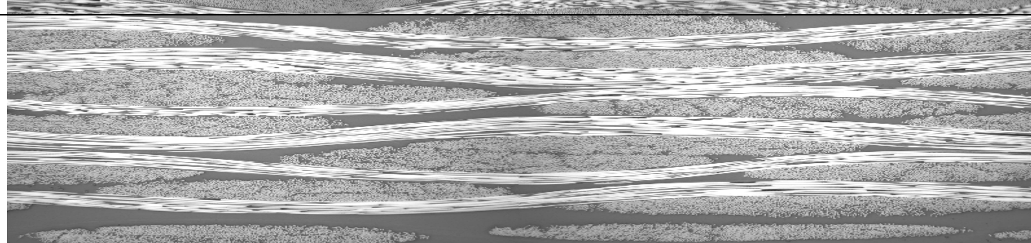
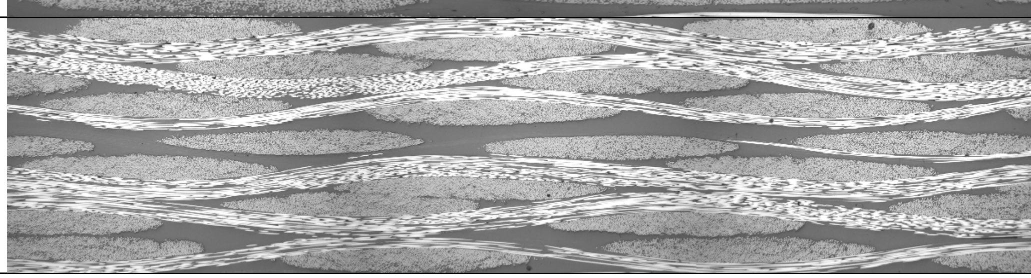
Autoclave 0° Void = 0.468%	
Autoclave 90° Void = 0.296%	
Oven (OOA) 0° Void = 0.339%	
Oven (OOA) 90° Void = 0.120%	

Figure 4.3 Microscopic Cross-Section of AS4C 3k PW, 202gsm/TC350-1 (10x5 magnification)

Debulk Procedure	SBS(ksi)	TG(°C)	CS(ksi)	CM(Msi)
4 ply debulk with FEP (Autoclave Cure)	20.9	208.3	263.28	23.588
4 ply debulk with FEP (Oven Cure)	20.1	209.8	271.68	24.056
4 ply warm debulk with FEP (Oven Cure)	20.0			
2 ply debulk with FEP (Oven Cure)	19.0		266.38	23.272
4 ply debulk with TX1040 (Oven Cure)	20.3			
2 ply debulk with TX1040 (Oven Cure)	21.1			

Table 4.8 Debulk Study of TC350-1

Type of Test	OOA cured Laminate (RTD)	Autoclave cured Laminate (RTD)
0° Tensile Strength (ksi)	395.1	381.8
0° Tensile Modulus (msi)	23.6	23.0
0° Compression Strength (ksi)	271.6	263.3
0° Compression Modulus (msi)	23.3	23.5
0° Flex Strength (ksi)	335.5	NA
0° Flex Modulus (msi)	13.9	NA
Short Beam Shear (ksi)	20.1	20.9
CAI Strength (ksi) 1500 in-lb	TBD	37.4

Table 4.9 Testing results of IM7/TC350-1 Unidirectional Tape

5. Conclusion

Out of autoclave (OOA) materials development over the past 10 years has been very Edisonian in nature and not typically embodied the manufacturing processes with respect to both the materials and the processes used to produce parts by OOA methodologies. Prepreg system development has therefore not typically encompassed the materials and processes required to produce large scale production parts that not only meet design requirements but are also very robust from a manufacturing and non destructive inspection perspective. TenCate is addressing this next generation need with its TC275 and TC350-1 OOA prepreg systems. TC275 offers mechanical property performance similar to autoclave cured structural composite materials but also brings to the design space a product that is very friendly to the OOA manufacturing processes currently employed in the industry, has standard setting hot/wet performance and can be easily inspected with current non-destructive inspection techniques. TC350-1 embodies similar manufacturing and inspection attributes to TC275, but also brings increased toughness and open hole strength properties that have classically only been available in autoclave curable composite materials. Another OOA development that Tencate has been focusing on is a high temperature resistant (Tg>300°C) cyanate based system with excellent structural integrity, toughness and manufacturability for space, launch vehicle and other critical aerospace applications.

6. References

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