Analysis Of Vacuum Failures During Curing Of Cfrp Composites.

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ABSTRACT: Carbon fiber reinforced Composite structures posses' high strength and stiffness to weight ratios, also exhibits good fatigue resistance, and are less prone to deterioration caused by corrosion and cracking. Fabrication of these composite structures is highly process dependent. During curing process, the physical, mechanical and chemical properties are imparted to the components, and are generally cured in autoclaves under controlled temperature, pressure and vacuum. Any variation in these parameters leads to change in properties of a composite structure, hence there is a need to analyse the effects of vacuum failures at during curing on mechanical and Ultrasonic Characteristics. Efforts are also made to analyze the root causes for the vacuum failures during curing process. Laminates made of Hexcel 913 carbon/ epoxy prepeg are prepared and cured in the autoclave by varying the vacuum levels at different salient points of cure process during curing. The specimens were prepared as per the DIN 29971 standards to evaluate the DT and NDT characteristics. The Experimental results shows that ILSS will not vary much if the vacuum fails after (Tg) Glass Transition point, hence the vacuum can be maintained up to the Tg point and thereby it can be slowly reduced to zero. This shows that there is a scope to optimize the manufacturers recommended cure cycle so as to reduce the curing cost and time. This research work helps the designers in developing the tools and optimum cure cycles for carbon epoxy composite structures.

Index Terms: Autoclaves, Composites, Curing, Epoxy, Glass Transition, ILSS (interlaminar shear strength), Vacuum.

1. INTRODUCTION

From the invention of epoxy resins by Pierre Castan in 1938 to now a days, epoxy resins attract the interest in Fiber Reinforced polymers due to their excellent balance between various properties like adhesion, electrical insulation, humidity resistance and mechanical properties . Hence these resins are widely used as a matrix for the fiber reinforced polymer structures, which cross links the fibers during curing due to polymerization. Voids in composite laminates have been extensively studied for many years, some authors have focused their work on the influence of voids upon physical and ultrasonic characteristics.[1] The presence of voids has detrimental effects particularly on the matrix dominated mechanical properties [2], The reasons for the voids formation is complex due to the variable factors in layup and processing in autoclaves also it may be due to the environmental factors like moisture absorption. [3] The resin flow models are derived by the Alfred C.Loos and G.S.Springer [4] are used to design the cure cycles for the epoxy matrix composites. But the equations developed are for the flat plate composites constructed from Hercules AS/3501-6 graphite epoxy prepeg tape. The causes for the vacuum failures or the effects of the vacuum at different stages of cure cycle are not focused.

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Curing optimization involves the best setting parameters related to material properties, tooling properties , part geometry etc.[5] , Although few works focused on the cure simulation of epoxy, limited work is carried on the effects of vacuum during curing of hexcel 913 carbon fiber/ epoxy structures. Few researchers [6-11] have studied the effect of vacuum and pressure on voids and also the relation between the voids and the ultrasonic characteristics but they have not focused on the duration of the vacuum in the cure cycle.

2. ROOT CAUSE ANALYSIS OF VACUUM FAILURES.

A root cause analysis has carried out considering from the Layup stage to the curing stage of 10 complex structural parts. It is observed that the vacuum bags fail during curing process as shown in fig.1 due to a) Improper bagging techniques. b) improper placement of valves during bagging c) Leakage in vacuum hoses & valves d) Resin block in tool vacuum lines e) malfunctioning of vacuum controllers / solenoid valves

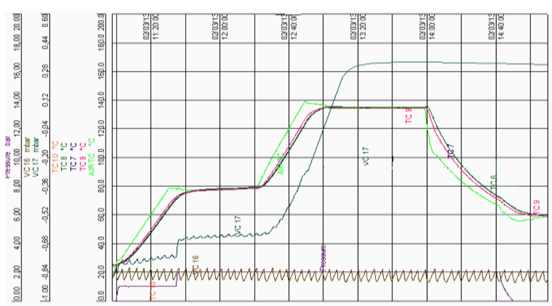


Fig.1 Shows the vacuum failure during curing where the vacuum failed at elevated temperature (135 deg.C)

2.1 Improper Bagging Techniques

The bagging film or a reusable elastic bladder is located over the material being cured and sealed against the tool surface as shown in the fig.2 which is called as the restraints or the dams to prevent the lateral motion and to minimize the resin flow parallel to the tool plate and through the edges.

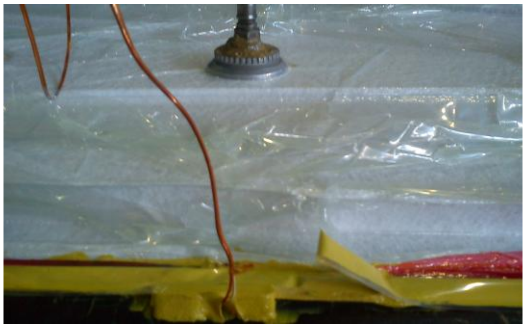


Fig.2 Vacuum bagging with thermocouple and vacuum valve.

Vacuum is applied between the bagging material and the material being cured such that the plies of material are compressed through the thickness against the mold. In some instances, an interim debulk step to enhance the quality of the finished product through improved consolidation. After vacuum bagging the vacuum leak rate from the bag is to be ensured before loading the part in to the autoclave for curing. Generally the allowable leak rate is only 50 milli bar for 5 mins. If the bagging is improper the leak rate will be more, generally the vacuum leak will be at thermocouple connection loops as shown in the fig.3 where the thermocouple is pulled out due to turbulence of air in the autoclave as the sealant becomes soft after heating. Hence it is recommended to provide the inbuilt thermocouples along with the tools and proper sealing to be ensured at the thermocouples provided for the part end.

Fig.3 Thermocouple pulled out during curing that leads to vacuum failure.

2.2 Improper placement of Vacuum valves

If the vacuum valves are not placed properly on the breather then the vacuum flow will be blocked as a result white patches or the voids in the cured part as shown in the fig.4.

Fig.4 Improper placement of valve, where the vacuum flow is blocked.

Even when the valves are placed on the part with insufficient layers of breather over the part or using a thin breather the resin enters the vacuum valves through the breather as shown in the fig.5 Due to this the volatile gases will be retained in between the layers and leads to defects like voids and improper distribution of resin, Hence the valves are to be placed on the ends with proper layers of breather to avoid direct contact of the resin to the valve.

Fig.5 Resin collection in the vacuum valve due to the use of thin breather, where the vacuum flow is blocked.

2.3 Leakage in Vacuum Hoses & valves

Vacuum hoses and the valves that are fitted with the connectors or the male plugs as shown in the fig 6. are properly tightened and checked before connecting it to the part to ensure the leakages. If the hoses and the valves are not fitted properly, it may read maximum vacuum initially, but at some point after application of pressure it fails abruptly causing part rejections due to defects. If part geometry is more complex to withstand the vacuum then it is more difficult to achieve uniform consolidation and to avoid wrinkling.

Fig.6 Vacuum hose fitted with the connector.

2.4 Resin Block in tool inbuilt Vacuum Lines

At present latest NMG metallic tools with nickel coating have inbuilt vacuum lines for suction and measurement which avoids the use of vacuum valves and also saving in bagging time as shown in the fig 7. But when an external pressure is applied on the prepreg stack that causes the excess resin to flow into bleeder. Sometimes due to improper bagging the resin flows in to the inbuilt vacuum lines and blocks the lines that causes defects in the cured part, hence the vacuum lines needs to be ensured for proper vacuum flow after every curing.

Fig.7 NMG metallic tools with inbuilt vacuum lines form suction and measurement.

2.5 Malfunctioning of vacuum controllers/ solenoid valves

Vacuum controllers fitted with the autoclaves are to be calibrated periodically to ensure the safe working condition as shown in the fig. 8.

Fig.8 Calibrated autoclave Vacuum controllers.

Even the solenoid valves are ensured to work in auto mode for continuous supply of vacuum during the curing process in autoclaves.

3. EXPERIMENTAL SETUP

Five Test Laminates of size 200 x 200 made of carbon BD V913/40%/G801 . The prepegs are cut along the fiber wrap direction for the symmetric balanced layup sequence . It is cured as per the manufacturer recommended cure cycle as shown in the fig. 9. Vacuum levels are varied at each stage.

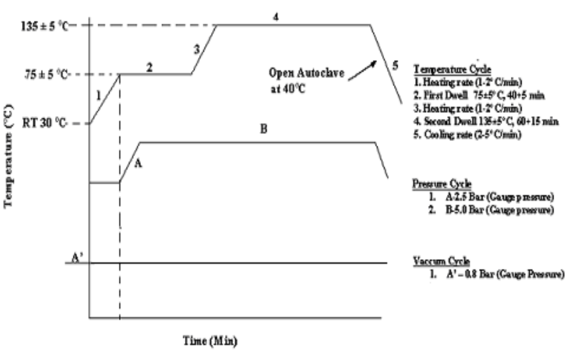


Fig.9 Manufacturers recommended two step cure cycle where the vacuum is applied throughout the cure process.

Vacuum is removed from the bags at the various salient points for each specimen during curing.

Specimen no. 1 considered as the reference panel which is cured with full vacuum throughout the cure cycle.

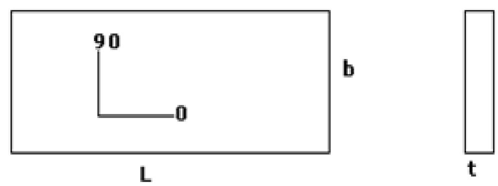
Specimen no.2 cured without vacuum throughout the cure cycle.

Specimen no.3 with full vacuum up to the start of first dwell (75 deg C)and there after the vacuum is vented to zero until the end of the cure cycle.

Specimen no.4 with full vacuum up to the end of the first dwell and thereafter the vacuum is vented out for the rest of the cure cycle.

Specimen no.5 with full vacuum up to the start of second dwell (125 deg C) and there after the vacuum is vented out.

After curing the laminates were tested for visual and NDT tests like A-Scan and the Ultrasonic C-Scan to check for the voids , internal cracks, FODs, etc. and then the specimens were cut in dimensions of 20x10x2mm as per the DIN 29971 standards to test the ILSS values.



Length L= 20 mm, width b= 10 mm, thickness t= 2 mm.

ILSS CALCULATIONS :

ILSS - Inter Laminar Shear Strength

$= \frac{3}{4} \{ F / (b \times t) \}$ mpa

Where F = Peak Load, Newton's,

b = Width of the specimen, mm &

t = Thickness of the specimen, mm

4. RESULTS & DISCUSSIONS:

All the specimens were tested for compaction using the A Scan & C-Scan and found the attenuation level was high for the specimen cured without vacuum through out the cure cycle, and also it was observed white patches which indicate improper resin flow and compaction due to insufficient vacuum during gellation. As shown in **the table 1&2.**

Table 1. Ultrasonic 'A' Scan Values of the test Laminates.

Specimen no.	Description	Attenuation in DB (gain)	Remarks
1	With full vacuum	31	
2	**Without vacuum**	**NO TRANSMISSION**	**No compaction**
3	Fail @ start of 1st dwell	36	
4	Fail @ end of 1st dwell	35	
5	Fail @ start of 2nd dwell	36	

Table 2. Ultrasonic 'C' Scan Values of the test Laminates.

Specimen no.	description	Attenuation DB (gain)	Remarks
1	**With full vacuum**	**24**	
2	**Without vacuum**	**34**	**high**
3	**Vacuum Vent at start of Ist dwell**	**24**	
4	**Vacuum Vent at end of Ist dwell**	**24**	
5	**Vacuum Vent at start of IInd dwell**	**24**	

The fig 10- 13 shows the profile view of the Ultrasonic 'C' Scan plots of the test laminates.

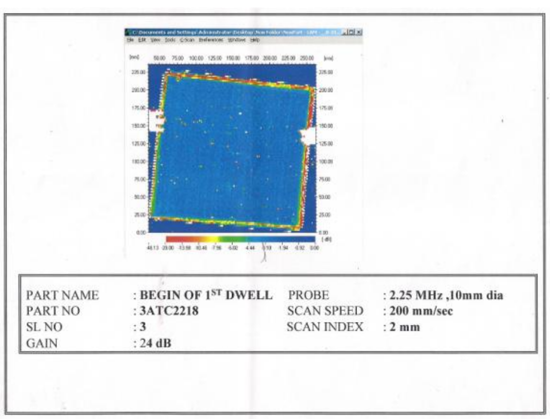


Fig.10 Ultrasonic 'C' Scan view of the specimen cured after vacuum vent out at the begin of first dwell (75 deg.C)

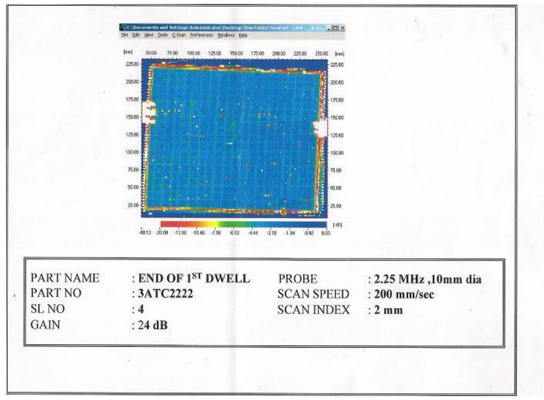


Fig.11 Ultrasonic 'C' Scan view of the specimen cured after vacuum vent out at the End of first dwell (75 deg.C)

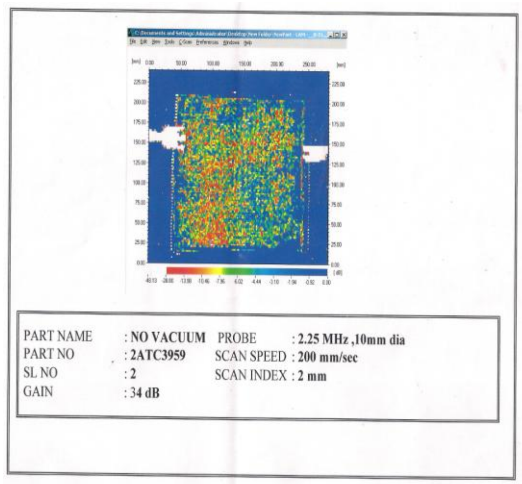


Fig.12 Ultrasonic 'C' Scan view of the specimen cured without vacuum, the yellow spots indicate the existence of Voids in the laminate.

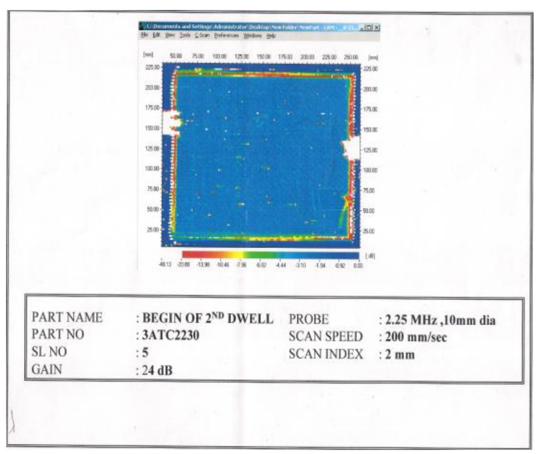


Fig.13 Ultrasonic 'C' Scan view of the specimen cured after vacuum vent out at the begin of second dwell (135 deg.C)

It is also found that the specimens cured with vacuum is vent out at the begin of the first dwell and at the end of the first dwell results in attenuation levels as same as the Master or the reference specimen cured with full vacuum. This indicates that the vacuum plays a vital role during the first ramp and the gel point where the resin changes its phase from semi solid to the gel state during which it emits the volatile gases because of the chemical reaction .There after even in the absence of vacuum it will not have any effect for the strength or on the quality of the part because the resin viscosity increases and the cross linking slowly diminishes that leads to the solidification stage.

Table 3. ILSS (Interlaminar Shear Strength) values of the test specimens.

Specimen no.	Description	ILSS (Mpa)	Load N	Remarks
1	With full vacuum	67	1993	
2	Without vacuum	44	1596	**Low**
3	Vacuum Vent at start of 1st dwell	66	1991	
4	Vacuum Vent at end of 1st dwell	68	2002	
5	Vacuum Vent at start of 2nd dwell	66	1960	

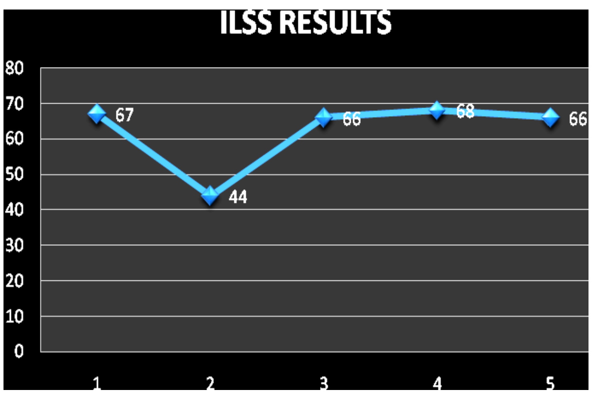


Fig.14. ILSS values curve of the specimens, where the specimen no.2 indicates low strength.

From the table. 3 it is noticed that the Interlaminar Shear strength (ILSS) is very low for the specimen which is cured without vacuum. This signifies that the presence of voids and the compaction of the layers is poor, as a result all the mechanical properties like tensile, flexural and the stiffness will be low. Whereas the other specimens exhibit appreciable strength equal to the reference laminate. Hence the existing cure cycle recommends for the vacuum through out the cure cycle and by this experiment it is evident that the cure cycle can be modified so as to save the power and curing cost. In the recommended cure cycle shown in fig.15 the vacuum can be cut off or vented out at 80 deg.C after the application of pressure that over comes the static vapor pressure of the resin.

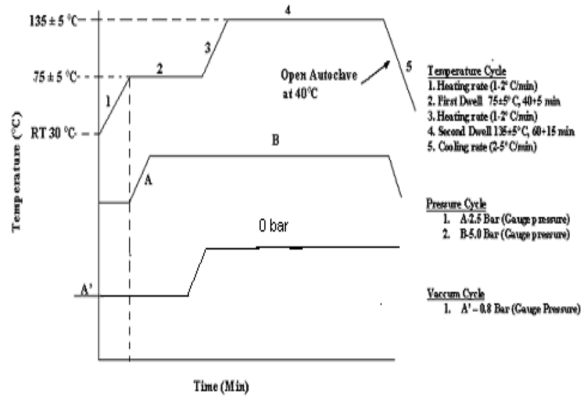


Fig.15. Recommended cure cycle for 913 monolithic parts, where the vacuum is cut off gradually after increasing the external pressure.

5. CONCLUSIONS:

1. Vacuum plays a vital role on the compaction of the composite part in the 913 resin system up to the first dwell temperatures 75-80 deg.c.
2. The specimen cured without vacuum exhibits the presence of voids and low mechanical properties.
3. Vacuum can be vent out at the first dwell gradually as the external air pressure is increased.
4. It is also recommended to ensure the vacuum level (vacuum leak check) before taking up for curing.
5. A vacuum failure during curing also occurs due to leakage in vacuum hoses and Vacuum valves. Hence these are to be checked before using it for curing for every cure cycle.
6. If the Bagging technique and bagging materials including bleeder materials and cauls, etc are not proper then due to vacuum failure at the edge loops (i.e., bridging) causes non-uniformity in material compaction and resin flow affecting the quality of the finished product.
7. If the number of interim debulk cycles and debulk time, or insufficient vacuum debulking cause's thickness and surface finish variability as well as wrinkles in the finished part.
8. Number of vacuum ports, location of vacuum ports, and vacuum integrity during cure cycles; materials are consolidated through the thickness during cure as the resin flows and gels. Vacuum integrity affects the level of compaction

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