Experimental Study on Residual Stresses in CFRP Composite Laminates

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Abstract: The residual stresses are the internal stresses between the fiber composite constituents in the absence of externally applied loads and body forces. These residual stresses play a vital role to contribute part shrinkage and warpages, also in determining the life and dimensional stability of a precision composite structure. Residual stresses arise due to inhomogeneous deformation of resin and fibre, as the resins have a very high thermal coefficient of thermal expansion, while the carbon fibres have almost zero coefficient along the length and minimum coefficient of thermal expansion along the transverse direction. The processes involving high temperatures often lead to residual stresses as different regions cool at different rates causing inhomogeneous deformation in a composite part. These stresses affect the structural integrity of the part and lead to crack when external service loads are applied. Efforts are made in this work to analyze and evaluate the presence of residual stresses in Hexcel 913 resin CFRP laminates. DT and NDT tests are carried out as per the ASTM standards to evaluate the influence of cooling rate on residual stresses in carbon/epoxy laminates. It is noticed that the cooling rate plays a significant role in inducing the residual stresses. The experimental results show that the mechanical properties tend to deteriorate with the increase in cooling rate above 5 deg.C/min. Both the theoretical and experimental values are found within a good agreement. This data helps the manufacturers and designers to design the optimum cure cycles for Hexcel 913 resin CFRP composite parts to achieve better quality and to avoid residual stresses.

Keywords: Composites, Cooling rates, curing, Residual Stresses shrinkages.

Nomenclature:

CFRP = Carbon Fiber Reinforced Polymer.
DSC = Differential Scanning Calorimeter.
DT = Destructive testing.
NDT = Non Destructive Testing.

I. INTRODUCTION

The residual stresses are the process induced stresses which are frozen in a molded part. It can arise in almost every step of the manufacturing process [1-4]. It can be either flow induced or thermal induced. Residual stresses arise due to inhomogeneous deformation of resin and fibre, as the resins have a very high thermal coefficient of thermal expansion, while the carbon fibres have almost zero coefficients along the length and minimum coefficient of thermal expansion along the transverse direction [5], the generation of residual stresses in the matrix surrounding the fibres is most easily understood in terms of the chemical shrinkage associated with the transformation of the resin from a liquid to a solid during the curing process. The shrinkage behaviour of a curing polymer as explained in the literature [6,7,8,9] is as shown in the Fig.1

In the stage a-b: The resin volume increases when heated from the room temperature To to the polymerization (cure) temperature Tc, due to thermal expansion before any chemical shrinkage occurs.

In the second stage b-c: around the cure temperature Tc, the resin volume starts to decrease due to cross-linking reactions resulting in significant chemical shrinkage, overtaking the thermal expansion. In the case of exothermic chemical reactions, an additional temperature peak may be observed, which is represented by point e.

In the third stage c-d: The volume decreases due to thermal contraction of the cured polymer during cooling to room temperature T with a CTE that is proportional to the degree of conversion. The slope between c-d is lower than for a-b, since the CTE in the glassy state (obtained after cure) is much lower than in the liquid state. In trajectory b-c during the polymerization reaction two points must be noted: the gel-point and the vitrification point. Appreciable residual stresses is induced in the matrix during cooling stage of the curing process as the resin (lamine) cools down to the room temperature after being cured at high temperatures. During this process the solidified resin tries to shrink, but it is resisted by the embedded stiff fibers which have very low coefficients of thermal expansion than the resin.
Residual stress may occur during curing process in autoclaves due to variations in density and mechanical properties experienced due to change in thermal properties as the material solidifies from the mold wall to the center, changing pressure, temperature, molecular and fiber orientation. Also these residual stresses are induced due to variation in the cooling rate from the mould tool to its center. Consequently parts with non uniform thickness or poorly cooled areas are prone to unbalanced cooling and thus to residual thermal stresses [10]. For complex parts the thermal induced residual stress distribution is further complicated by non uniform wall thickness, mold cooling and mold constraints to free contraction. The mechanical interaction consists of the mismatch in CTE between the tool and the composite. Often the CTE of the tooling is higher than that of the composite. This mismatch can result in significant residual stresses or a through-the-thickness stress distribution in the composite part, depending on the interface conditions between the tool and the composite [11-15]. Hence non uniform thickness of the tool, tool geometry, and non uniform part thickness contributes to non uniform cooling rates that lead the way to residual stresses. Previous research work on the residual stresses are generic and are not specific to a particular resin system, but the cure temperature and process parameters tends to change from one resin to another. Hence there is a scope to analyze and evaluate the residual stress in Hexcel 913 carbon epoxy material due to variation in cooling rate during autoclave curing process. This study will be used to design autoclave cure cycles to minimize /eliminate the residual stresses and to accomplish better quality.

II. EXPERIMENTAL WORK

The laminates are fabricated using Hexcel V913/G801 carbon epoxy prepreg material that has 40% of the resin content. Four test laminates of size 200 x 200 mm are fabricated as per the standard lay-up sequence and are cured in an autoclave under controlled vacuum, pressure (2 bar) & temperature (135 deg. C) at different cooling rates as shown in the Table 1.

TABLE 1. Details of the laminates cured at different cooling rates.

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Lamine</th>
<th>Cooling rate in °C/min</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>L1</td>
<td>0.6</td>
</tr>
<tr>
<td>2</td>
<td>L2</td>
<td>1.2</td>
</tr>
<tr>
<td>3</td>
<td>L3</td>
<td>2.4</td>
</tr>
<tr>
<td>4</td>
<td>L4</td>
<td>4.2</td>
</tr>
</tbody>
</table>

The cured laminates are tested for internal defects like voids, delaminations using ultrasonic A-scan, results are obtained and found the ultrasonic gain or the DB level are within the acceptable levels as shown in the Table 2.

TABLE 2. Details of Ultrasonic ‘A’ Scan values of the tested laminates.

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Lamine</th>
<th>Ultrasonic gain or DB level</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>L1</td>
<td>32</td>
<td>satisfactory</td>
</tr>
<tr>
<td>2</td>
<td>L2</td>
<td>31</td>
<td>satisfactory</td>
</tr>
<tr>
<td>3</td>
<td>L3</td>
<td>30</td>
<td>satisfactory</td>
</tr>
<tr>
<td>4</td>
<td>L4</td>
<td>29</td>
<td>satisfactory</td>
</tr>
</tbody>
</table>

The DSC (Differential Scanning Calorimeter) tests are carried out to check the onset and the peak temperatures, and also to evaluate the degree of cure. DSC curves are as shown in the Fig 2. The DSC curves signifies the amount of exothermic reaction that occurred during curing process and the amount of heat ‘dh’ retained in the laminates indicates complete curing process.

![DSC curves for the four laminates obtained from the DSC test.](image-url)
A. ILSS CALCULATIONS:
The specimens of size 10 mm X 20 mm are prepared for testing ILSS (Inter Laminar Shear Strength) as per the ASTM D2344 standards.

The ILSS (Inter Laminar Shear Strength) signifies the compaction strength or the bonding strength between the fibers and the matrix (resin) in the composite structure; generally it is calculated using the formula,

$$\text{ILSS} = \frac{3}{8} \left( \frac{F}{b \times t} \right) \text{ MPa},$$

Where $F$ - Peak Load, Newton’s, $b$ - Width of the specimen, mm & $t$ - Thickness of the specimen, mm

B. TENSILE STRENGTH CALCULATIONS:
Tensile strength = $\left\{ \frac{F}{(b \times h)} \right\}$ MPa, where $F$ – Force in N., $b$ - width of the specimen in mm, $h$- Thickness of the specimen in mm.

C. FLEXURAL STRENGTH CALCULATIONS:
Flexural strength = $\frac{3}{2} \left( \frac{F \times L}{b \times h^2} \right)$ MPa. Where $F$- Force in N., $L$- span length in mm., $b$- specimen width in mm, $h$- Specimen thickness in mm.

The ILSS, tensile strength and flexural strength values of the laminates cured with different cooling rates in the autoclave are as shown in the Table 3

III. RESULTS AND DISCUSSIONS
The ILSS, ultimate tensile strength and flexural strength values are tabulated in the Table.3 the interlaminar shear strength values are within the acceptable limits for the laminates cured with the cooling rate from 0.6 to 4.27 deg.C/min, and there after the ILSS drops gradually, similarly in the case of tensile & flexural strength values.

The behavior of tensile strength with the cooling rate is plotted in the Fig.4, which depicts that the tensile strength improves as the cooling rate is increased up to 2.4 deg.C/min, as the cooling rate exceeds beyond 2.5 deg.C/min the tensile strength decreases gradually, due to the presence of residual stresses.

<table>
<thead>
<tr>
<th>SI NO</th>
<th>LAMINATE</th>
<th>COOLING RATE (deg C/min)</th>
<th>ILSS, MPa</th>
<th>UT. TENSILE STRENGTH (MPa)</th>
<th>FLEXURAL STRENGTH MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>L1</td>
<td>0.6</td>
<td>74.83</td>
<td>99.73</td>
<td>1082.617</td>
</tr>
<tr>
<td>2</td>
<td>L2</td>
<td>1.2</td>
<td>75.48</td>
<td>100.42</td>
<td>1008.873</td>
</tr>
<tr>
<td>3</td>
<td>L3</td>
<td>2.4</td>
<td>75.57</td>
<td>100.76</td>
<td>886.6118</td>
</tr>
<tr>
<td>4</td>
<td>L4</td>
<td>4.27</td>
<td>75.23</td>
<td>100.35</td>
<td>1113.969</td>
</tr>
</tbody>
</table>

Table 3. Details of the ILSS, Tensile and Flexural Strength values.

The Degree of Cure(D.O.C), Glass transition temperature and the Average residual stresses are tabulated as shown in the Table 4.
The interlaminar shear strength (ILSS) values were found increasing up to the cooling rate of 2.4 deg.C/min, as the residual stresses are increasing the material tends to lose its stiffness and the specific modulus decreases, in turn reducing the inter laminar shear stresses.

Similarly the theoretical ILSS values are calculated as shown in the Fig.8, where the linear equation is fitted to find the mathematical relationship between the ILSS values to the cooling rate, i.e the ILSS is -0.2869 times the cooling rate. This depicts a continuous down trend in the ILSS values as the cooling rate increases beyond 4 deg.C/min, the theoretical values of residual stresses are calculated and plotted as shown in the Fig. 7.

The theoretical relationship between the residual stresses and the cooling rate is represented by the slope of a linear equation. i.e residual stresses are -0.0591 times the cooling rate. However both theoretical and experimental approach residual stresses are in a good agreement with each other.

IV. CONCLUSIONS.

Experimental results in this study show that the higher cooling rates significantly affect the part strength due to the presence of residual stresses. It is also noticed that the Inter laminar shear stresses are optimum at cooling rates between 2 - 2.5deg.C/min. However these stresses are within the acceptable limits from 0.6 deg.C/min to 5 deg.C/min and hence the cooling rates can be recommended within this range for curing 913 carbon epoxy prepeg materials.

Flexural and Tensile strength decreases at the higher cooling rates due to the presence of residual stresses. This study shows that the residual stresses are sensitive and a serious problem in processing fiber composite parts in autoclaves. Hence the designers can make use of this work in modeling and to optimize the cure parameters especially the cooling rate to eliminate residual stresses from the cured components.

V. REFERENCES

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