# STUDY OF CURING CYCLE INFLUENCE ON MECHANICAL PROPERTIES FOR COMPOSITES MANUFACTURED BY RESIN INFUSION PROCESS

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## 1 Introduction

The design of structural parts in composites brings several advantages against conventional materials like steel, wood and concrete specially associated to the well know higher ratio of mechanical properties per density, the excellent weather and corrosion resistance, durability and reduced maintenance costs.
The essence of composites is the composition of different materials to obtain a third one with general advantages against their source materials.
Becomes from this fact also one main concern over the reinforced polymers, the variability of properties from the mixture and consolidation step, what means that manufacturing process can impact significantly over material resistance and consequently that production requires special cares over effectiveness and reproducibility of manufacturing process.
The opening of the composites market demands higher productivity, reliability and cost effectiveness of its processes for assurance of competitiveness.
This work seeks to draw attention and discuss the impact of the productive process on the parts manufactured in reinforced composites in terms of mechanical properties decrease.
The evaluation of process influence on the final properties of the laminate was investigated on epoxy resin infused panels of fiber glass fabric.
This set of material was selected according to its market scale on structural composites parts.
The condition variation can be understood as the variation inherent to a process that can affect two different parts or either the same part in different positions.
Structural optimized designed parts presupposes known material properties, is expected that actual product reflects the behavior of mathematical models and prototypes what can frequently not be true if some variation is not reduced before production startup. Is usual to find overestimated safety factors to hide the effect of these variation what increases significantly the product cost, reducing consequently the interest on designing in composites materials.

## 2 Sources of variation

Several factors can impact negatively on the final properties of the components originated on the many phases of the process.
Reinforced polymeric composite usually requires processes of preparation of the layers and cores, resin catalization, fiber impregnation, curing and finishing.
Critical steps to guarantee the synergic consolidation of matrix and reinforcement are comprehensively expected as major impact sources on actual composites resistance.
Several techniques can be employed to minimize the risks of having such a big variations on the production, as FMEA, standardized methods as described per ISO 9001, Six Sigma, Gauge R&R (Repeatability and Reproducibility), MSA (Measurement Systems Analysis). Kaoru Ishikawa proposed in 1943 e perfected on the following years the cause and effect diagram that classifies the sources of defects from the 6M's: machine, method material, man power, Measurement and mother nature (environment).

### 2.1 Curing

Curing is the step of consolidation of the monolithic structure and consists on one very important step where the parts are under different conditions, with high temperature variations, changes of viscosity a density, transition of physical state, residual stress appearance and setting of defects from previous steps, like voids, dry spots and others.
Is convenient to set Tg (glass transition temperature) requirements for the laminate, to ensure thermal and mechanical stability.

Beyond the variation among different parts, for big structures like airplanes, boats, wind blades, tubes is possible to observe different properties in the same piece, what can definitely lead to premature localized failure, the Fig. 1 is extracted from the first measurement of common convective post cure oven validation and exemplifies the 30% variation found in several points of the equipment.

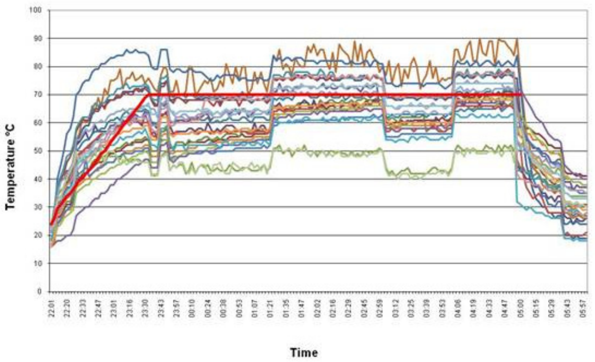


**Fig. 1 – Temperature measuring on convective oven**

## 3 Method

For the mechanical properties comparison of a laminate, the manufacturing process was defined as VARTM (vacuum assisted resin transfer molding), with curing parameters variation and mechanical properties evaluated through ISO standardized methods and normalized for standard fiber fraction.

### 3.1 Panel infusion

The VARTM method ensures process reproducibility and is based on a closed process with two steel molds, sealant tapes and hydraulic system for vacuum and resin transference. The used system scheme is presented on Fig. 2.

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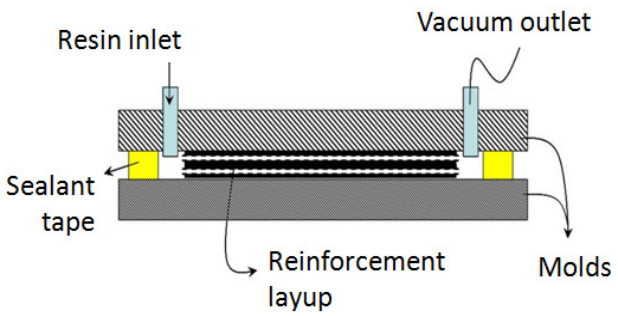


**Fig. 2 – VARTM Scheme**

Panels were manufactured with bidiagonal glass stitched fabrics (non crimp) 1000g/m$^2$ areal weight and appropriate low viscosity epoxy resin for the infusion process.

Final construction is a [(+45/-45), (+45/-45)/ (+45/-45), (+45/-45)] where non symmetric construction were preferred to ensure alignment of the layers referenced on the polyester stitching.

### 3.2 Curing

Based on the product technical data-sheet two curing cycles were defined, representing low and high curing conditions, first is based on 5 hours at 60ºC cycle and second on 9 hours at 80ºC.

### 3.3 Characterization

For laminate characterization glass transition temperature is measured, mechanical properties evaluated and normalization process performed, for statistical comparison characteristic value is calculated and results ploted on comparative charts.

#### 3.3.1 Glass transition temperature (Tg)

As thermal transition implies several physical property changes on the material and considering that as high is the polymerization degree, higher will be the Tg value, the laminate cure can be evaluated through Tg measurement. One of the affected properties is specific heat that can be indirectly determined on differential scanning calorimetry (DSC) analysis.

For standardization of DSC analysis main three interest points can be measured: onset, midpoint and end Tg, first is the temperature where specific heat curve starts to change, last one is the temperature where transition is nearly finished and middle is calculated based on both onset and end values, for the purpose of this study onset value will be reported.

For these sense DSC analysis is usually performed in two heating curves, first for actual curing evaluation and second for potential curing.

Picture 2 is an example of DSC analysis performed on an glass fiber reinforced epoxy laminate.

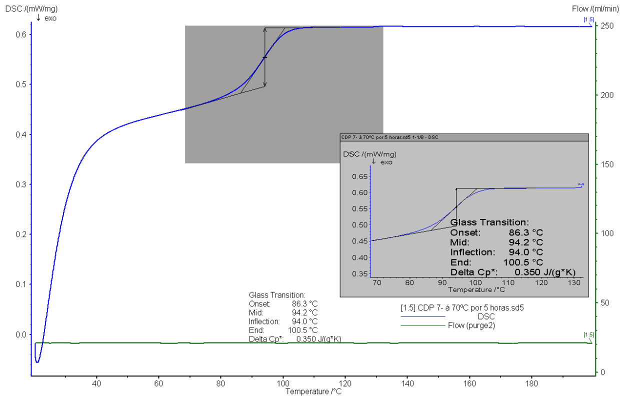


**Fig. 3 – DSC analysis**

## 3.4 Mechanical tests

For curing evaluation impact several methods could be adopted according to loading parameters defined for application. Some traditional physical tests are tensile, compressive, shear strength, fatigue, impact and flexural among many other possibilities. For each characteristic several standardized methods can be applied as the most famous ones: ISO, ASTM, and DIN.

For this study ISO 527-4 type 3 that coupon is presented on Fig. 4 were determined for tensile test, ISO 14126 with ITRII fixture presented on Fig. 5 used for compressive test and ISO 178 method for flexural properties determination.

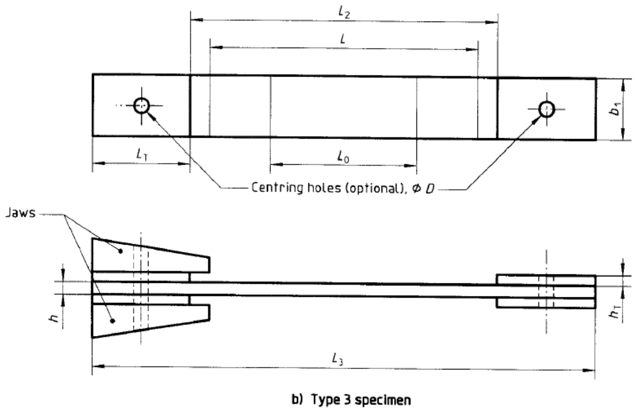


**Fig. 4 – Coupon for ISO 527-4 Type 3**

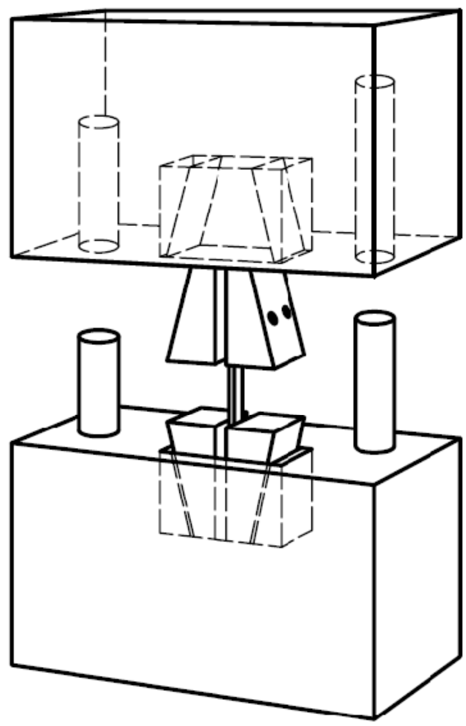

**Fig. 5 – ITRII Fixture for ISSO 14126**

## 3.5 Normalization

For results comparison a target fiber weight fraction (FWF) of 67.5% is set and the actual values of stress and modulus can be adjusted using equation *1*, for FWF determination ASTM D2584 is performed and volume fraction ($V_F$) is calculated using equation *2*.

$$X_{adjusted} = X_{measured} \frac{FVF_{standard}}{FVF_{measured}} \quad (1)$$

$$V_F = \frac{\rho_M \cdot W_F}{\rho_F \cdot W_M + \rho_M \cdot W_F} \quad (2)$$

Where, V is volumetric fraction, ρ the density W the weight fraction and the subscript index F the fiber and M the matrix

The equation *2* can only be applied as resin is degassed before infusion process; otherwise voids content should be included on the equation.

It is. important to mention that the void content affects severely laminate properties and in this sense was eliminated from this study although it is an important source of variation for composites processing.

### 3.6 Characteristic values

For statistical relevance evaluation, a 95% confidence interval was adopted and the upper and lower characteristic values calculated based on the results dispersion per the equations *3* and *4*.

$$x^* = x - \sigma \cdot z\left(1 + \frac{1}{\sqrt{n}}\right) \quad (3)$$

$$x^* = x + \sigma \cdot z\left(1 + \frac{1}{\sqrt{n}}\right) \quad (4)$$

Where:

$x^*$ = calculated characteristic value
$x$ = average
σ = standard deviation
z = 1.645 (for 95% confidence interval);
n = number of samples

### 4 Results

Results are ploted on comparison charts of stress and modulus for lower and higher curing degrees on the charts of Figs. 6 to 11

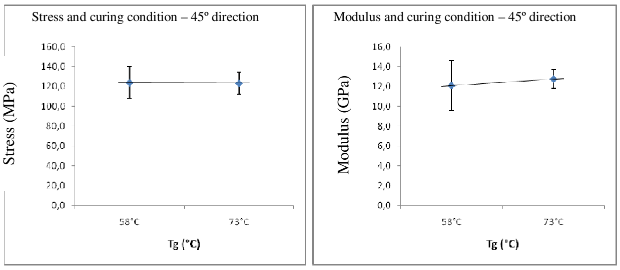


**Fig. 6 - Tensile properties – 45º direction**

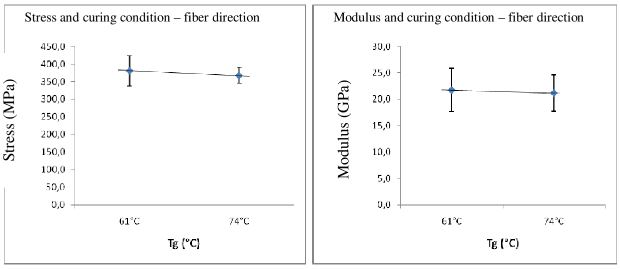


**Fig. 7 - Tensile properties – fiber direction**

No differences is observed among tensile test results both for stress and modulus, the degree of curing is not an impacting variable for the evaluated range variation.

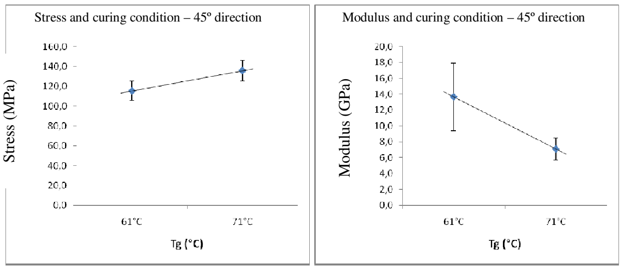


**Fig. 8 - Compressive properties – 45º direction**

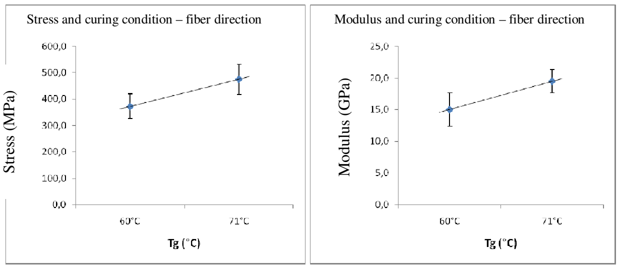


**Fig. 9 - Compressive properties – fiber direction**

For compressive properties significant influence is detected for both stress and modulus properties. For fiber direction roundly 15 to 30% improvement in stress is achieved while Tg increases from 60ºC to 70ºC and there is an increase of 30% on elastic property for fiber direction and 50% decrease for 45º while Tg increases near 10ºC.

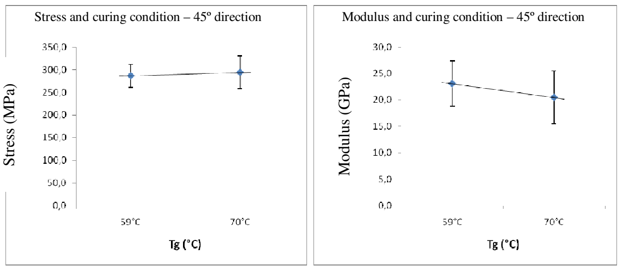


**Fig. 10 - Flexural properties – 45º direction**

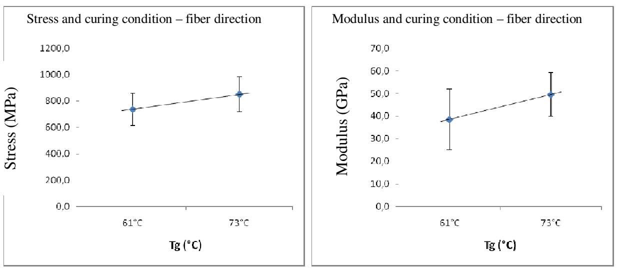


**Fig 11 – Flexural properties – fiber direction**

Although some trend can be perceived on flexural properties, the results are not statistically conclusive on the evaluated condition.

## 5 Conclusion

The most significant evaluated impact of curing condition was observed on compressive properties.

An interesting observed point is the higher results dispersion of less cured coupons, probably because of higher instability of the cross links, less homogeneous microstructure of the reinforced polymer or higher dispersion of amorphous zones.

General compressive properties improved in correlation with curing degree, but for non fiber direction where elastic modulus decreased with higher Tg, although rupture stress increased. A possible explanation for this unexpected fact is that for predominant shear load the lower cure act as an efficient load distributor until catastrophic failure occurs.

Is a fact that process variables affect the properties of fiber reinforced polymers and the knowledge of these influences can be a key tool for the success of an effective component design.

For a more comprehensive observation of cure influence different curing conditions should be evaluated and even more load modes adopted, as fatigue tests, impact, mode I, short beam and many others.

Interesting results could be also obtained if different environment were investigated, as for example salty atmosphere, alkaline ambient, high or sub ambient temperatures and others.

## References

ASTM D2584 - 08 Standard Test Method for Ignition Loss of Cured Reinforced Resins, ASTM, 2008

ISO 14126:1999 - Fibre-reinforced plastic composites -- Determination of compressive properties in the in-plane direction, ISO, 1999

ISO 178 :2010 - Plastics -- Determination of flexural properties, ISO, 2010.

ISO 527-4:1997 - Plastics -- Determination of tensile properties -- Part 4: Test conditions for isotropic and orthotropic fibre-reinforced plastic composites, ISO 1997

ISO 9001:2008 - Quality management systems – Requirements, ISO, 2008

MOMENTIVE – “EPIKOTE™ Resin MGS® RIMR 135 and EPIKURE™ Curing Agent MGS® RIMH 134–RIMH 137” – Technical Data Sheet, 2006

SAERTEX – BIDIAGONAL-GLASS-FABRIC 1000g/m2 – Technical Data-Sheet, 2004