A NEW METHODOLOGY FOR COMPOSITES MANUFACTURING EVALUATION

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Abstract. *The most used manufacturing processes for laminated polymeric matrix composites are hand lay-up, vacuum bagging and compression molding. Each of one these processes has its own particular characterisitics. Therefore, there is a need for developing a new methodology for comparison purposes. To be able to identify the population size, a set of five ASTM D 3039/3039M tensile specimens were prepared for each manufacturing process. Once the tests are completed, the methodology on design of experiments proposed by Montgomery (2001) was applied. Twenty-five specimens for each manufacturing process were prepared. All specimens were made of eigth layers of plain weave woven fabric E-glass with 50% of epoxy resin volume fraction. For each group of specimens the axial Young's modulus, the ultimate stress, the failure mode were evaluated. Besides, a microscopic analysis was performed to be able to identify the voids formation rate. On the top of all these experimental data an statistical analysis was also performed. Once the macromechanical and the micromechanical analysis were completed a new admensional parameter was proposed. The Generalized Yoke ratio alloweds the comparison of different composites manufacturing process considering the ratio of defects generated during each process. It seems not only to be a very realiable but also a helpful tool for composite manufacturing evaluation.*

Keywords: Composites Manufacturing, Micro and Macro-mechanics, Textile composites, Laminated Composites.

1. Introduction

According to Gutowski (1997), the most important manufacturing process of composites applied at aerospace industry is the hand lay-up of prepregs and autoclave cure. Although the manufacturing processes have been undergoing constant technical changes, the hand lay-up still persists as the method by which more than half of all advanced composite aerospace structure is made. The reason for this large use is due to its extreme flexibility which allows the manufacturing of a large variety of shapes. Besides, hand lay-up does not require a large investment. In this type of manufacturing two distinct phases can be established; the first one consists on fibers impregnation and stacking, and the second one is the cure procedure. According to Jones (1999), the cure can be done by autoclave, in the presence of vacuum (vacuum bagging), under compression (pressing), or to air. Moreover, the composite performance is highly influenced by the cure itself. Bader (2002) mentions that there are many ways of evaluate the composite performance. However, the easiest way is based on stiffness and/or strength. He called the attention that this performance is directed related to the manufacturing process used to produce the composites itself. In his study, he evaluated five different processes, namely, autoclaving, resin transfer molding (RTM), resin film infusion (RFI), pultrusion and compression molded sheet. Moreover, the process selection must be based not only on the materials used but also on component geometry, size and required mechanical properties. In some applications the economical factor must also be considered. Based on this last consideration Bader (2002) concludes that the non-prepreg and non-autoclave processes present the best cost/effectiveness ratio. The work done by Bader (2002) is a comprehensive study on composites manufacturing but he does not considered problems generated during the manufacturing process itself.

Researcher's focus their attention, in general, on mechanical properties estimation without considers the manufacturing processes. Once the mechanical properties are estimated some samples are tested and based on some statistical modeling the effective properties are obtained. Therefore, the manufacturing process analysis is somehow neglected. To improve the composite overall performance, it is needed not only consider the mechanical properties estimation models but also the manufacturing processes with its advantages and limitations. Daniel and Abot (2000) link the two areas by taking into account the problem of void formation on laminated composites. According to them, the voids can be formed either by entrapment of air mechanically or by nucleation from vapors or gases. The mechanical entrapment could due to entrained gas bubbles from resin mixing operations, bridging from large particles, voids from wandering tows, fuzz balls, or broken fibers, or air pockets and wrinkles created during the lay-up. As mentioned by Mallick (1988), the void formation causes the stiffness and strength reduction. He goes further trying to correlate the void formation with two key factors, i.e. time and temperature, during the resin cure. One of his conclusions is that temperature and time are inversely proportional to each other. Moreover, the void formation has to be linked to other factors than temperature and time, e.g. humidity. Huang et al. (2000) point out another problem that could arise during the manufacturing process. It is the induced stress/strain that could lead to a warpage. This phenomenon, very common in thick composites, is mostly associated to the chemical stiffening and volumetric shrinkage of resin during the cure. Therefore, they developed a semi-numerical model to predict the induced stress/strain and avoid the warpage.

The purpose of this paper is to make a statistical study on hand lay-up manufacturing process with cure to air, under compression and assisted by vacuum. Besides, a new methodology to estimate the rate of defects generated during each manufacturing process is proposed.

2. The proposed methodology for composites manufacturing evaluation

The main idea behind to the proposed methodology is to evaluate different composites manufacturing processes. Jones (1999) stated that is virtually impossible to obtain a defect-free composite. Therefore, a good parameter for comparison is the level of defects generated during each manufacturing process. The composite designer must have in mind that variations on stiffness and strength can be due to the choice of the manufacturing process. As mentioned by Kassapoglou (1999), the use of the same technology with slight differences can lead into complete dissimilar mechanical properties, e.g. a typical example is a composite wing box made by hand lay-up and varying the co curing process.

The Yoke ratio, a non-dimensional coefficient, was proposed by Avila et all (2001) as a ratio between the actual and the designed stiffness. Later on, Morais et all. (2002) extended the concept and the Generalized Yoke Ratio (GYR) was developed. The GYR mathematical representation is given by:

$$\upsilon = \left\| \frac{\partial \kappa\left(E_x^{\mathrm{Macro}}\right)}{\partial x} \frac{\partial x}{\partial \kappa\left(E_x^{\mathrm{Micro}}\right)} \hat{i}, \frac{\partial \kappa\left(E_y^{\mathrm{Macro}}\right)}{\partial y} \frac{\partial y}{\partial \kappa\left(E_y^{\mathrm{Micro}}\right)} \hat{j}, \frac{\partial \kappa\left(E_z^{\mathrm{Macro}}\right)}{\partial z} \frac{\partial z}{\partial \kappa\left(E_z^{\mathrm{Micro}}\right)} \hat{k} \right\| \tag{1}$$

where $\kappa(E_x^{macro})$ is the vector which contains the Young's modulus variation, evaluated at macroscopic level, at x direction. The subscripts x y and z denote the three mutually orthogonal directions. The superscript micro and macro are indication of micro and macro-mechanics analysis. Moreover, the symbol $\| \;\|$ denotes the L-2 norm as defined by Golub and Van Loan (1996).

As the GYR definition involves two scales of analysis, a macro-mechanical and a micro-mechanical, the proposed methodology must reflect both. The macro-mechanical analysis is based on the uniaxial tensile test described by the ASTM D 3039/3039M-00 (2000) standard, while the micro-mechanical analysis involves not only the porosity determination via optical microscopy but also the micromechanics modeling.

The interesting point about the GYR is that it represents the rate of defects or voids generated during the manufacturing process. Notice that the micro-mechanical model is considered as a defect-free model, in other words, the micro-mechanical predictions will lead the upper bound stiffness values as the unit cell is idealized. Moreover, an effective quality control methodology for composites can be developed by applying the GYR methodology associated to the statistical analysis.

2.1 Macro-mechanical approach associated to a statistical analysis

This part of this study involves the manufacturing of three sets of laminates. For the first group the hand lay-up with cure to air is employed, while for the second group the cure is vacuum assisted, and finally, for the third group the cure is performed under compression. The composite reinforcement is done by a plain weave woven fabric E-glass WR200 AF-0003) manufactured by TEXIGLASS, while the matrix is an epoxy resin (XR1553) and hardener (HY1246) made-up by VANTICO. The laminate has 8 layers and a 50 % fiber volume fraction. During the compression procedure a pressure equivalent to one atmosphere is applied, while for the vacuum bagging operations a pressure close to 30 mmHg is used. For both cases the pressure is kept constant up to 24 hours.

A variation of the design of experiments methodology proposed by Montgomery (2001) was applied to find the sample size. First a pilot sample of 10 specimens, for each manufacturing process, is prepared. Then, the tensile tests are performed and the Young's modulus, ultimate stress and failure modes are obtained. For the Young's modulus and the ultimate stress the mean value (μ), standard deviation (σ) and the maximum error (E') are computed. The procedure suggested by Montgomery (2001) takes in consideration the sample size of various groups of specimens, with different mean values. For this case, we have:

$$\Phi^2 = \frac{n \cdot \sum_{i=1}^{k} \tau_i^2}{k \cdot \sigma^2} \tag{2}$$

where

$$\tau = \bar{x}_i - \bar{x} \tag{3}$$

k is the number of processes compared, and $\bar{x}_i$, $\bar{x}$ are the mean value for each sample and for all populations together, respectively. Finally, the estimate variance is defined by σ^2. Once the Φ parameter is obtained and the degrees-of-freedom are known, it is possible to fit the sample size (n) using the operating characteristic curves (OC curves).

Once the sample size (n) is defined for all three groups of specimens, they were tested following the ASTM D 3039/3039M-00 (2000) and the following parameters were evaluated: Young's modulus, ultimate strength and maximum load. These mechanical properties were used as key factors for comparison among the three manufacturing processes. A variance analysis must be applied to knowing if these three processes are similar or not. According to Montgomery (2001), the most convenient for statistical analysis considering a normal distribution is the ANOVA parametric model, while when a normal distribution is not applicable non-parametric models, e.g. Kruskal-Wallis, is more adequate. These two methodologies for variance analysis were applied at this paper.

2.2 Micro-mechanical approach: Computer modeling associated to optical microscopy analysis

The methodology applied to compute the elastic moduli via micro-mechanical models involves not only the recognition of a unit cell but also the use of optical microscopy techniques to measure the fiber volume fraction, the distances between the tow fibers, the packing factor, and the filaments diameter. The micro-mechanical model used is based on the unit cell approach for woven fabric composites as defined in Naik (1994). A public domain micro-mechanical code mmTEXlam developed Challa and Shivakumar (2001) was employed. To measure the unit parameter a series of longitudinal and transversal cuts were performed for each manufacturing group. The optical microscopy samples have 1 cm^2 area. They are located in a cylindrical cup filled with resin and later on polished with diamond paste. Once on optical microscope the image measure software QUANTIMET® was used to get the values. At least 10 measurements in each specimen were performed, and the average values were computed.

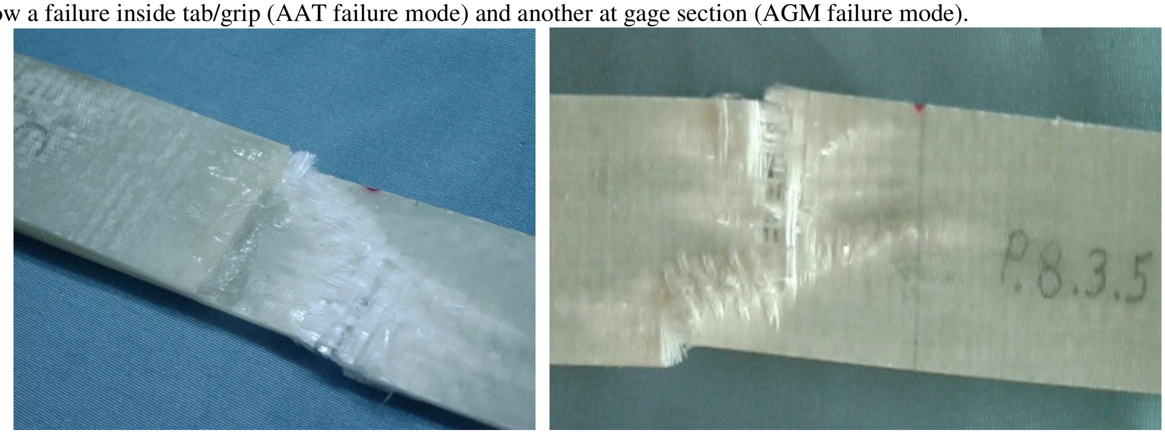
3. Results and analysis

The first question that must be answered is: are these three different types of cure statistically equivalents? The second question is concerned to the way of ranking these processes. The results can be divided into to categories: the first one is the results from the tensile tests and the statistical analysis, while into the second group of results we do have the optical microscopy data, the numerical predictions obtained from the micro-mechanical model and finally the GYR computation. It is important to notice that all three type of cure were studied at same time. The same methodology applied to one group of specimens was applied to the others.

3.1 Tensile tests results and the statistical analysis

From each plate laminated five specimens were prepared following the ASTM D 3039/3039 M standard. For sample size definition the 30 specimens were prepared, 10 for each manufacturing process, and the tested. To avoid any underestimation on sample size population, the mechanical parameters selected must take into consideration stiffness and strength. Therefore, the Young's modulus, the ultimate strength and the maximum load were the mechanical properties chosen. Following Montgomery (2001) the probability of type I error, often called the level of significance of the test, (α) and the probability of type II error (β) are fixed equal to 0.01 and 0.05, respectively. The sample size calculation was performed considering each one of these mechanical properties. By adopting this strategy, we guarantee a statistical consistency for stiffness and strength. By using the operating characteristic curves from Montgomery (2001), it is possible to compute a sample size population (n) equals to 23 with a probability (1-β) of 95%. To be on a safe side a sample size of 25 specimens was selected. By assuming this value we can into consideration any problems during the tensile tests.

The tensile test results will be presented at the following order: ultimate strength, and Young's modulus. The idea is to show the variation of each mechanical property as a function of the manufacturing process, and later on compare the results. Before summarize the results a failure analysis into the specimens must be performed according to the ASTM D 3039/3039M standard. All specimens with failure inside tab/grip should not be considered. Figures 1a and 1b show a failure inside tab/grip (AAT failure mode) and another at gage section (AGM failure mode).



(a) (b)

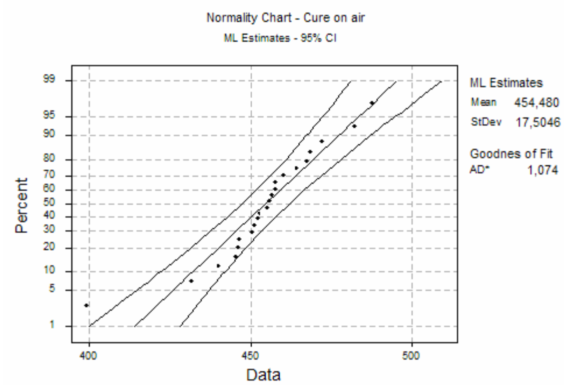
Figure 1: Failure mode on laminated composite with cure on air.

A summary of the ultimate strength results is presented in Table 1. From this table, it is possible to conclude that some similarities among the manufacturing processes can be observed. This is due to the facts that the standard deviation value for cure on air and cure under pressure are located inside the 95 % confidence interval for cure vacuum assisted. Beside the mean and median values are close, which is evidence of a symmetric distribution.

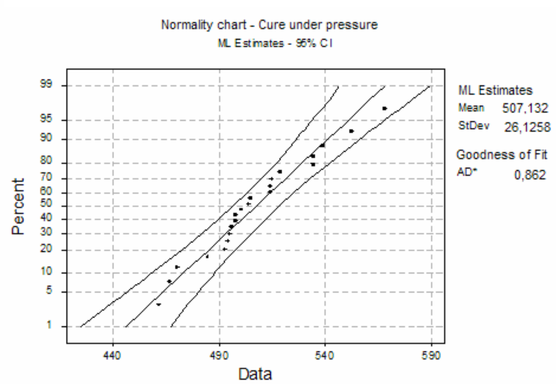
Table 1: Ultimate strength as a function of cure process

Ultimate Strength [MPa]	Hand lay-up lamination with cure		
	On air	Under pressure	Vacuum assisted
Mean value	454,48	507,13	465,87
Std. deviation	17,92	26,74	23,11
Median	455,40	502,04	465,75
Variance	321,00	715,06	534,26
95% interval of confidence for mean	446,54-462,42	495,28-518,99	454,73-477,01
95% interval of confidence for std. deviation	13,78-25,60	20,57-38,21	17,47-34,18
95% interval of confidence for median	450,21-460,35	495,07-515,03	457,14-480,81

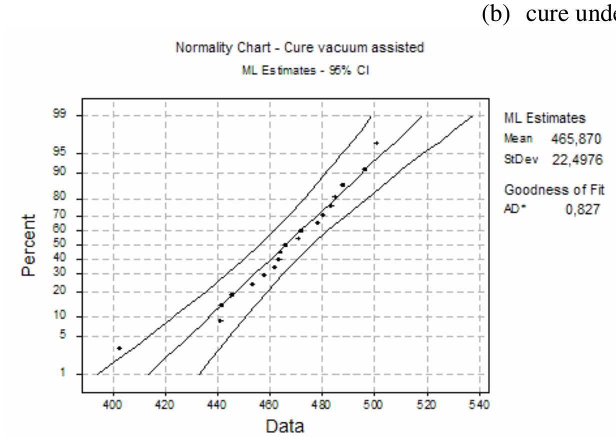
The next step is to check how close the data from the tensile tests are to a normal distribution. To perform such test the normality charts must be applied. Figures 2a through 2c show the normality charts for cure process. The only cure process which does not a clear indication of a normal distribution, according to Myers and Well (2003) for a normal distribution the AD* parameter has to be less than 1, is the cure on air. However, as the AD* value for this case is close to the unit, we can assume a normal distribution for this case without loss of generality.



(a) cure on air



(b) cure under pressure



(c) cure vacuum assisted

Figure 2: Normality charts considering the ultimate strength

Before perform a variance analysis a check on variance resemblance must be considered. Following Montgomery (2001), the most adequate check on variance equivalence for a group with high evidence on normal distribution is the Bartlett's test. Moreover, when the normal distribution can not be assumed Montgomery (2001) suggests the Levene's test. Figure 3 shows the Bartlett's and Levene's testes for the ultimate strength data. The three groups of data will have the same variance if and only if their level of significance (α) is smaller than the P-value computed by the test. In our case α is made equal to 0,05 (5%) and the P-value computed by Bartlett's test is 0,200. Therefore, it can be assumed that all three cure processes have the same variance. As a consequence, the ANOVA variance analysis can be applied.

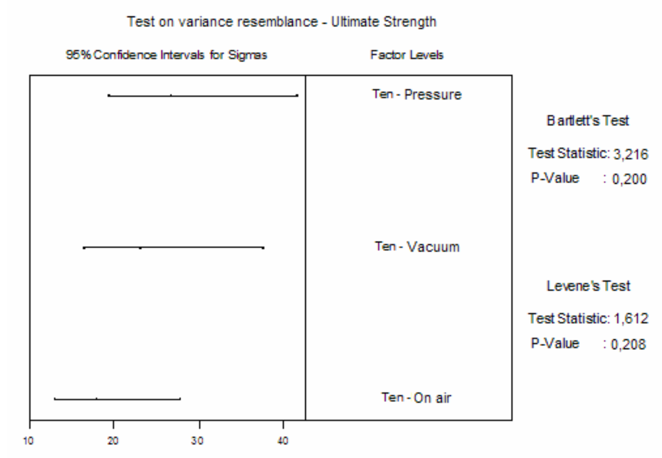


Figure 3: Bartlett's test for ultimate strength

The last stage is to use the variance analysis, in this case ANOVA, to check if the hypothesis of equivalence of the cure processes is true of false. In figure 4 is shown an output data from MINITAB considering the ANOVA analysis. As it can be seen the P value is less than the level of significance (α). Therefore, the hypothesis of resemblance among the cure processes must be rejected. However, there is an overlap region between the cure on air and cure vacuum assisted. They can be seemed as similar processes but not equivalents. We can conclude that considering strength the three cure processes can be considered different, in other words, each cure process will result in composites with unlike strength.

```
Analysis of Variance for Anova Ultimate Strength
Source      DF        SS        MS        F        P
C9           2     33455     16727    31,99    0,000
Error       60     31374       523
Total       62     64829
                                   Individual 95% CIs For Mean
                                   Based on Pooled StDev
Level        N      Mean     StDev   --------+---------+---------+--------
Ten - pr    22    507,13     26,74                             (----*---)
Ten - va    19    465,87     23,11         (----*----)
Ten -air    22    454,48     17,92   (----*----)
                                     --------+---------+---------+--------
Pooled StDev =     22,87                    460       480       500
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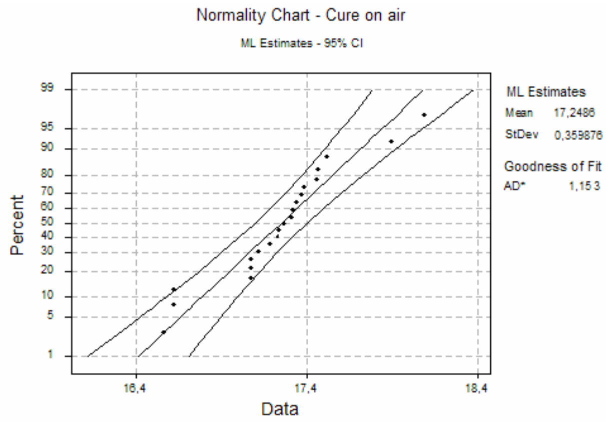
Figure 4: ANOVA analysis for the ultimate strength parameter

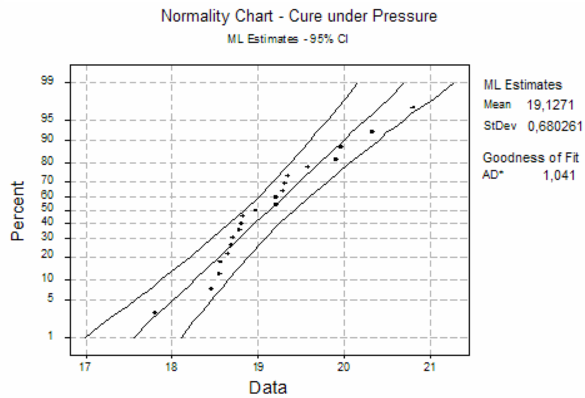
The same statistical analysis must be performed considering the stiffness. The stiffness results, represented by the Young's modulus, are summarized on Table 2. An analysis on Table 2 shows that although the results are not very dispersed they don't seem to be equivalent to each other. The normality charts shown in Figures 5a through 5c indicate that they can not be considered as a normal distribution for the cure vacuum assisted and for the cure on air. The only type of cure which could have a normal distribution is the one under pressure. Considering that all three cure processes may not follow a normal distribution, the best test to check if they have the same variance is the Levene's test. By observing figure 6, it is possible to conclude that for the Young's modulus the three cure processes do not have the same variance as the P value is higher than the level of significance used (5%). As a consequence of these results the Kruskal-Wallis technique must be used. Figure 7 shows an output for the Kruskal-Wallis analysis. The three cure processes must be considered different as they have a P-value smaller than the level of significance.

Table 2: Stiffness versus cure process

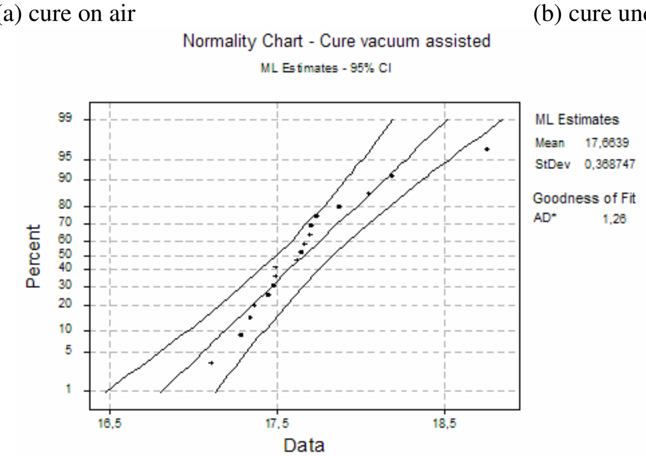
Young's modulus [GPa]	Hand lay-up lamination with cure		
	On air	Under pressure	Vacuum assisted
Mean value	17,25	19,13	17,66
Std. deviation	0,37	0,70	0,38
Median	17,26	18,97	17,64
Variance	0,14	0,49	0,14
95% interval of confidence for mean	17,08-17,42	18,81-19,44	17,48-17,85
95% interval of confidence for std. deviation	0,28-0,53	0,53-1,01	0,28-0,57
95% interval of confidence for median	17,10-17,38	18,70-19,32	17,46-17,72



(a) cure on air



(b) cure under pressure



(c) cure vacuum assisted

Figure 5: Normality charts considering the stiffness parameter

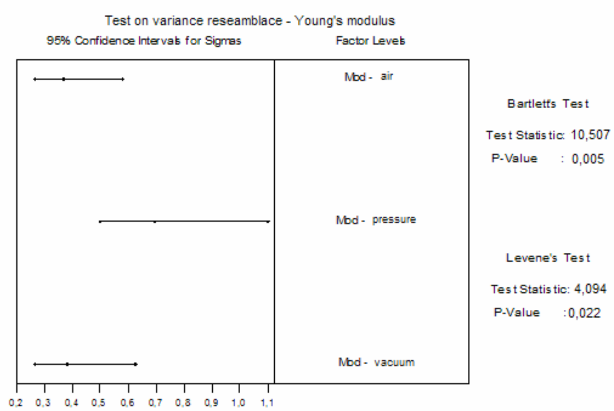


Figure 6: Levene's test for Young's modulus

Kruskal-Wallis Test: Stiffness

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Kruskal-Wallis Test on Modulus

C9           N    Median    Ave Rank        Z
Mod - air   21     17,26        14,4    -5,25
Mod - pr    21     18,97        49,4     6,16
Mod - vá    18     17,63        27,2    -0,95
Overall     60                  30,5

H = 43,19  DF = 2  P = 0,000
H = 43,20  DF = 2  P = 0,000 (adjusted for ties)
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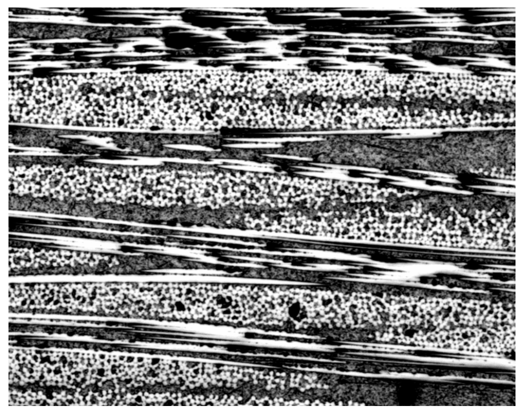
Figure 7: Kruskal-Wallis analysis

From all data shown before, it is possible to conclude that the three cure processes will produce composites with stiffness and strength statistically different, even though they have the same volume fraction and the same fiber orientation and stacking sequence. Therefore, the cure process has direct influence on the composite's mechanical properties. Finally, a last question must be answered: Is it possible to rank the three cure processes? To get this answer a micro-mechanical analysis must be performed and associated to the macro-mechanical analysis.

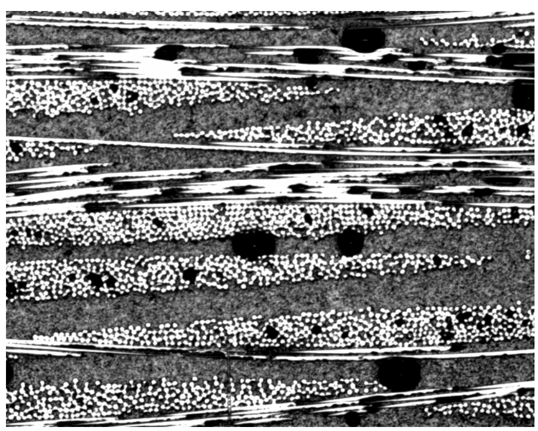
3.2 Micro-mechanical model and optical microscopy analysis

To be able to compute the elastic moduli using mmTEXlam (Challa & Shvikumar, 2001) the volume fraction must be known. During the manufacturing process the exact amount of resin and fibers are known, but the actual values can be only approximated, as the void formation rate is highly dependent on the cure processes. Therefore, a microscopic analysis must be performed for a better estimation. From each test group longitudinal and transverse cuts were carried out and specimens with 1 cm^2 were prepared. The procedure of encapsulating, polishing and attacking the surface follows the one proposed by Sawyer and Grubb (1996). After some previous test, the magnifications of 60 and 800 times were selected. Moreover, twenty image analysis of each sample were completed. By using the QUANTIMET 600 image software we are able to compute not only the fiber volume fraction but also the tow packing factor. Figures 8a-8c show different array formations due to the different cure processes. The black dots are voids formation. Moreover, the percentage of void formation is summarized on Table 3.

(a) Cure on air



(b) Cure under pressure



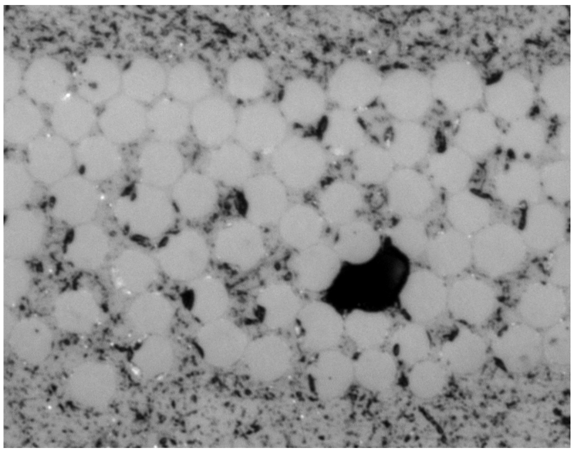
(c) Vacuum assisted cure

Figure 8: Micrographs (60 X) – void formation

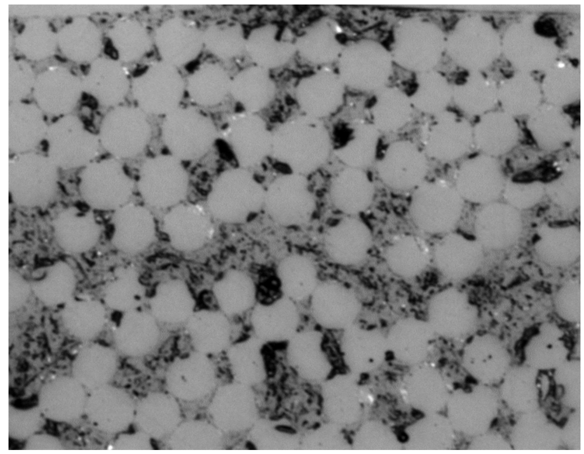
Table 3: Void formation as a function of the cure process

Void [%]	Hand lay-up lamination with cure		
	On air	Under pressure	Vacuum assisted
Mean value	2,14	0,70	1,85
Std. deviation	1,48	0,43	1,55
Median	1,63	0,58	1,47
Variance	2,18	0,18	2,41
95% interval of confidence for mean	1,45-2,84	0,50-0,90	1,13-2,58
95% interval of confidence for std. deviation	1,12-2,16	0,33-0,63	1,18-2,27
95% interval of confidence for median	1,17-2,51	0,36-0,78	1,08-2,29

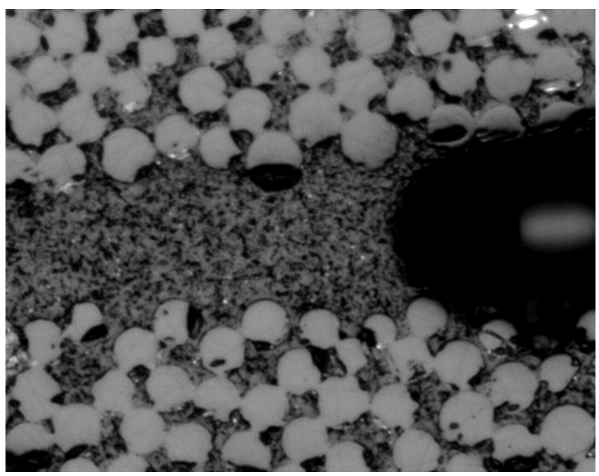
The fiber volume fraction by using image analysis, via QUANTIMET, shows a slight variation from cure on air, under pressure and vacuum assisted, i.e. the volume fractions were 49%, 50% and 50%, respectively. The tow packing factor however, presented a larger variation. For cure on air the packing factor was 62%, while for cure under pressure the result was 64% and for vacuum assisted 61%. It is interesting to notice from figures 9a-9c that tow packing factor is really a function of the cure process, in other words, the resin infiltration and location is influenced by the cure process. The large void observed in figure 9c can be evidence a problem during the vacuum formation. This void can be result of an air bubble trapped during the cure. With all these information, the micro-mechanical analysis can finally be performed. The mmTEXlam previsions for the three processes studied are listed into Table 4.



(a) Cure on air



(b) Cure under pressure



(c) Vacuum assisted cure

Figure 9: Micrographs (800 X) – tow packing

Table 4: mmTEXlam stiffness estimates

Properties	Hand lay-up lamination with cure		
	On air	Under pressure	Vacuum assisted
E_{xx} [GPa]	21,980	23,060	22,380
E_{yy} [GPa]	21,980	23,060	22,380
G_{xy} [GPa]	3,966	4,198	4,027
ν_{xy}	0,126	0,128	0,124

As expected the cure under pressure presented the highest stiffness. This could be due to the small number of voids generated during the manufacturing. The final step is the generalized yoke ratio calculation. As the void formation is represented by an interval, it is better to plot the GYR as a function of those. The results can be seemed at figure 10. The GYR seems to capture the kernel of the problem, in other words, the results are coherent as the void formation increases the GYR decreases. It is important to mention that a defect-free composite must have a GYR equals to one.

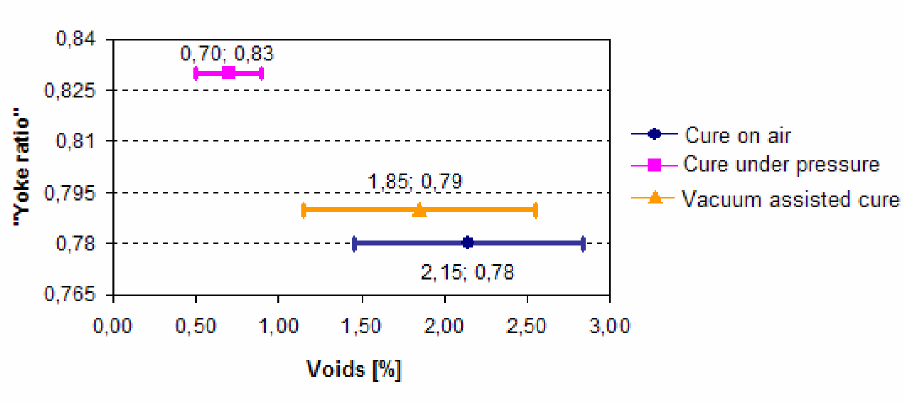


Figure 10: GYR representation as a function of void formation

By analysis figure 10, it is possible to conclude that the cure process which leads best results is the cure under pressure followed by the vacuum assisted cure and the cure on air.

4. Closing Comments

A new methodology for composite manufacturing evaluation was proposed. This new methodology takes into consideration not only the standard macro-mechanical approach but it is also associated to a micro-mechanical approach. Moreover, a statistical study considering variance analysis was also employed during the macro-mechanical tests. By doing this, it is possible to guarantee the data consistency.

It was statistically proven that cure on air, cure under pressure, and vacuum assisted cure are different. Furthermore, the stiffness and strength of a set of composites with same fiber and matrix volume fractions, same fiber orientation, and stacking sequence will have distinct stiffness and strength due to the cure process.

The generalized yoke ratio was able to capture the void formation. As a consequence the three cure processes studied could be ranked. The cure under pressure seems to offer the best results following by the vacuum assisted cure and the cure on air. Finally, the generalized yoke ratio appears to be a promising technique for composites manufacturing evaluation.

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