

RE.PUBLIC@POLIMI

Research Publications at Politecnico di Milano

Post-Print

This is the accepted version of:

L. Di Landro, A. Montalto, P. Bettini, S. Guerra, F. Montagnoli, M. Rigamonti Detection of Voids in Carbon/Epoxy Laminates and Their Influence on Mechanical Properties Polymers and Polymer Composites, Vol. 25, N. 5, 2017, p. 371-380 doi:10.1177/096739111702500506

The final publication is available at https://doi.org/10.1177/096739111702500506

Access to the published version may require subscription.

When citing this work, cite the original published paper.

Detection of Voids in Carbon/Epoxy Laminates and Their Influence on Mechanical Properties

Luca Di Landro^{a*}, Aurelio Montalto^b, Paolo Bettini^a, Stefania Guerra^c, Fabrizio Montagnoli^d, Marco Rigamonti^c

ABSTRACT

Defects, such as voids and delaminations, may significantly reduce the mechanical performances of components made of composite laminates. Distributed voids and porosity are generated during composite processing and are influenced by prepregs characteristics as well as by curing cycle parameters. On the basis of rheological and thermal analyses, as well as observations of laminates produced by different processing conditions, curing pressure appears the most influent factor affecting the void content. This work compares different methods for void analysis and quantitative evaluation (ultrasonic scan, micro-computed tomography, acid digestion, SEM image analysis) evidencing their applicative limitations. Carbon/epoxy laminates were produced in autoclave or oven by vacuum bag technique, using different processing conditions, so that void content ranging from 0 % to 7 % volume was obtained. Effects of porosity over laminates mechanical performances are analyzed. The results of tensile and compressive tests are discussed, considering the effect that different curing cycles have over void content as well as over fiber/resin fraction. Interlaminar strength, as measured by short beam shear tests, which is a matrix-dominated property, exhibits a reduction of failure strength up to 25% in laminates with the highest void content, compared to laminates with no porosity.

KEYWORDS

Composite laminates; void content; autoclave processing; image analysis.

1 INTRODUCTION

The aerospace industry commonly employs high performance composite parts produced by lamination and autoclave curing of prepreg materials, which are made of reinforcing fibers, in the form of woven fabric or unidirectional tape, impregnated with uncured thermoset resin. The lamination and curing of prepreg plies are critical, because any improper procedure at these stages implies the creation of permanent defects, which reduce the final mechanical-performances. Trapped voids and delaminations represent the most common defects due to unsuitable production procedures. Air bubbles, absorbed moisture and solvents can volatilize and expand during heating stage, causing the formation of voids. All defects present at resin consolidation remain trapped inside laminate; therefore, the removal of imperfections should be assured before resin gelation [1].

Fabric prepregging process often requires the employment of solvents such as methyl ethyl ketone, acetone, dichloromethane or xylenes to control resin viscosity in the impregnation stage. Although most of solvent is removed later in the process, a residual amount invariably remains in the final prepreg, notwithstanding the application of heating stages. The content of residual solvent can be in some case well above 1 % wt in

^a Dipartimento di Scienze e Tecnologie Aerospaziali, Politecnico di Milano, 20156 Milano, Italy,

^b Dipartimento di Meccanica, Politecnico di Milano, 20156 Milano, Italy,

^cTechnologies Lab Non-metallic Materials Science, AgustaWestland, 21017 Cascina Costa VA, Italy

^d NDI Unit, AgustaWestland, 21017 Cascina Costa VA, Italy

^{*} Corresponding author: luca.dilandro@polimi.it

commercial prepregs [2]. Even though the solvent content is low in terms of weight, it is however well enough to produce extensive voids in the final products, in case of improper lamination procedure. During production of laminates, if the vapor pressure of solvent is greater than curing pressure the evaporation of volatile matter causes the growth of voids inside the matrix [3]. On the other hand, in some instances, for example in case of sandwich panels production, high autoclave curing pressure is not advisable and significant void content is likely to remain in the laminate. It is predictable that the development of porosity is faster when the resin reaches a low viscosity value during heating; at the same time, most of generated voids are expected to be removed thanks to the vacuum bag action and to the excess resin flow before gelling. An appropriate choice of cure cycle ensures an optimal reduction of voids so to obtain a laminate with acceptable residual porosity. Laminate thickness and plies distribution is also expected to have influence on the remaining void content.

Voids have detrimental effects over the mechanical behavior of composite laminates since they generally decrease overall static and fatigue strength. Porosity mainly affects matrix and interface dominated mechanical properties, such as interlaminar shear strength, and must be usually limited to very low values [3-7]. On the other hand, situations where the presence of porosity may positively affect specific mechanical response of composite laminates are reported [8].

The evaluation of void content in composite parts is usually required by aerospace production procedures, especially for structural components, to guarantee mechanical performance and safety standards. Void contents as low as 1%, or 2.5 to 5 % volume in certain circumstances, are usually considered acceptable for structural components [9]. It should be considered, moreover, that the presence of porosity can affect also physical properties other than mechanical behavior; for example, moisture diffusion rate in the consolidated laminates can increase, thus affecting components aging when exposed to wet environment [10].

It is worth noting that such voids have origin within the prepreg resin and are thus expected to be distributed more or less evenly in the whole produced laminates, as long as a homogeneous distribution of temperature and pressure is assured during curing. This situation may be somewhat different from lamination defects typical of other materials or procedures, e.g. glass fiber mat and hand lay-up, which arise from low material homogeneity and/or limited expertise of laminating personnel: in such situations, localized voids of variable dimensions or delaminations are likely.

The detection of porosity in an intrinsically inhomogeneous material as a fiber composite laminate is not straightforward. Different techniques can be employed to estimate the void content of composite parts such as ultrasonic analysis, thermography, micro-tomography, microscopy observation and acid digestion [4, 5, 11, 12]. Daniel [13] used image analysis to assign a correlation between the ultrasonic attenuation and porosity; the "real" values assumed as reference derived from image analysis with optical microscopy. Liu et al. showed that cure pressure as well as its application time influence porosity content in laminates. In particular, voids can be significantly reduced choosing a proper time for pressure application. The amount of void was assessed by ultrasonic scanning and density measurements [14]. Optical microscopy and image analysis were employed to characterize pore size, shape and location in the laminates. Kite et al. [1] and Zhu et al. [15] also employed image analysis, through optical microscopy, to obtain statistical information about amount, shape, size, and orientation of voids in carbon/epoxy and glass/epoxy systems. In their study, Kite et al. [1] showed that the estimation of void content might differ more than 1% between image analysis and acid digestion in preimpregnated fabrics. Moreover, the difference of results increases in unidirectional laminate due to oblong, cigar-form voids. Kastner et al. [9] used X-rays micro CT scanning for the measurement of void in composite laminates. They showed that a reliable evaluation is obtained provided proper threshold values are selected in the analysis; the threshold choice is however recognized as a critical issue and that some calibration is necessary for and effective measurement. Moreover, high resolution results require long scanning times and complex volume reconstruction procedures. Somewhat different estimated void contents according to the measure/calculation method adopted is thus to be expected.

All these techniques can reach consistent results, but usually show intrinsic limitations such as shape/geometrical restrictions or the need of destructive procedures; besides, some methods provide relative or indirect measures of porosity. A further, relevant drawback of these techniques is that data from different

analyses are not directly comparable. The two-dimensional nature of optical microscopy and the number of observed micrographs affect the accuracy of image-analysis; a large set of images is usually necessary for a reliable statistical evaluation, making this technique quite expensive and time consuming, provided automated techniques are not employed [16]. Acid digestion and density based measurements are non-local analytical technique, based on bulk volume/density measurements providing well averaged estimations of void content. However, accurate values of fibers and plain resin density are required; such data are often difficult to be determined for laminates produced from prepregs and small errors in their measurement remarkably influence the estimation of void content.

In this research, a number of carbon composite laminates with different thickness and different void contents were produced. A first thermal/rheological analysis of employed prepregs allowed to select proper processing conditions to reach variable void fractions. From this analysis, indications about adequate processing parameters for actual laminates production could be drawn. Actual void content in representative laminate specimens was measured by different methods, i.e. thickness and density measurements, acid digestion, ultrasonic C-scan, computed X-rays micro-tomography (micro CT); image analysis of laminate sections observed at SEM was also used, which produced reliable results with convenient advantages in terms of testing procedure and image interpretation compared to optical microscopy. The results of all techniques were then compared.

The void content of all produced laminates was thus measured by SEM/image analysis before their mechanical characterization. It is worthy to note that, in addition to void amount, a variation in the processing procedure may affect other laminates properties, such as glass transition temperature and fiber/resin content which also have marked influence on overall mechanical response. Therefore a straightforward relationship between mechanical performances and void content or distribution cannot be drawn directly. All obtained laminates were tested to determine the tensile, interlaminar, flexural and compressive response. The influence of void content, together with different processing conditions, over mechanical properties is thus discussed.

2 EXPERIMENTAL

2.1 Materials

Carbon fabric/epoxy prepreg (G0803 – HexPly 6376) was employed for the laminates production. Fabric style is five harness satin, nominal weight 285 g/m², thickness 0.29 mm, 3000 filament/tow,7.2 tows/cm warp and weft. Impregnation resin was HexPly 6376 epoxy, formulated for aerospace use. Tensile strength 105 MPa, tensile modulus 3,60 GPa for the cured resin are reported. A small amount of dichloromethane (0-2% wt) can be present in prepreg as residual solvent of the impregnation process [17].

Prepreg was stored at -18 °C and was kept at room temperature for at least 4 hours before its employment for tests and laminates production.

2.2 Rheological and thermal analysis of prepreg

Rheological tests were applied to prepreg material to follow the evolution of resin viscosity with time so to estimate the gel-time of epoxy matrix. Torsional dynamic-mechanical tests (DMTA) were carried out with a Rheometrics RDA II rheometer at 1 Hz frequency; the specimens consisted of rectangular beam laminates [(0,90)]₅ with dimensions 40 mm x 9 mm obtained by overlaying of 5 plies of prepreg. Tests were conducted with 1% strain. It should be noted that, given the non-homogeneous nature of prepreg, the results reflect the viscosity variation of the matrix; the measured complex viscosity was thus assumed as a conventional viscosity. On this basis, a reference gel-time was arbitrarily defined when the measured viscosity reached three times the minimum value during cross-linking. It can be observed that such value approximately corresponds

to the point of maximum viscosity increase rate. This choice allowed a consistent comparison of gel-times measured in different cycles.

Two different time-temperature cycles schemes were adopted, i.e. isotherms at a pre-selected temperature between 155 °C and 180 °C up to system consolidation or heating ramps at 3°C/min up to a pre-selected temperature, followed by isotherm. This last procedure reproduces thermal cycles suggested for the actual laminates production [18].

DSC analysis (TA Instruments DSC 2010) was carried out on prepreg material to follow the curing evolution during the curing cycle prescribed by the producer, which involves a heating ramp of 3 °C/min, an isotherm at 175 °C for 2 hours, a cooling ramp at 3 °C/min. DSC analyses were also carried out on produced laminates to evaluate the residual reaction heat.

2.3 Production of laminates

Flat, square laminates were produced by overlaying 6, 12 or 18 prepreg plies of 500mm x 500 mm; a stacking sequence of $[(0,90) / (+45,-45) / (0,90) / (+45,-45) / (0,90)]_n$ with n=1, 2 or 3 was adopted. Vacuum bagging and autoclave curing completed the laminates production; thermocouples were inserted in the panel to monitor actual temperature during curing. Four different cure cycles produced panels with different porosity levels. The autoclave temperature was programmed to provide a heating ramp of 3°C/min up to 175°C, followed by an isotherm for 120 minutes, as suggested by producer technical datasheets [18]. Different pressure levels were set in each cycle.

Cycle A - Partial vacuum inside bag (- 0.5 bar rel. pressure) for the whole cycle and no external pressure.

Cycle B - Full vacuum inside bag (-0.9 bar rel. pressure) for the whole cycle and no external pressure.

Cycle C - Full vacuum inside bag (-0.9 bar rel. pressure) until temperature reaches 60 °C, then atm pressure inside bag; autoclave pressure 3 bar (pressure ramp 0.25 bar/min).

Cycle D - Full vacuum inside bag (-0.9 bar rel. pressure) until temperature reaches 175 °C, then atm pressure inside bag; autoclave pressure 7 MPa (pressure ramp 0.25 bar/min).

Strips, 50 mm wide, were cut from panel edges and discarded. The thickness of each cured laminate was then measured in 12 different locations to estimate thickness uniformity and average value. Thickness differences well below 4% the average value were always recorded (below 2% in most cases). As expected, a marked effect of cycle pressure over average thickness was however observed due to the different void amount and resin flow.

Ultrasonic testing (UT) was employed to evidence possible delaminations or markedly un-even distribution of voids in the panels. Through transmission, C-scan tests were carried out by a PANAMETRICS 9100 instrument on composite plates. Scan parameters (gain, ref. level) were adjusted according to specimen thickness and overall attenuation. All laminates were apparently homogeneous, with no evidence of delaminations; only the thickest (18 layers) plates produced with A and B cycles (no autoclave pressure applied) showed a somewhat un-even distribution of voids evidenced by different local attenuation response.

2.4 Void analysis

The laminates were cut by a diamond saw to produce the coupons for the following mechanical tests and void analyses. In void analyses, with the aim to compare different void measurement methods, the same specimens or portions of the same specimens were analyzed in sequence by SEM, then by micro-CT scan, and eventually by density/acid digestion. Void measurements by SEM were then repeated on the panels employed for mechanical measurements.

2.4.1 SEM and image analysis

The image analysis of composite laminates for void evaluation is generally carried out using optical microscope micrographs at 50 -100 magnification; automated stage movement is usually preferred to scan a large sample area. However, the discrimination between fibers, resin and voids from optical micrographs is often problematic and somewhat subjective; sensibly flat, accurately ground and polished surfaces are to be examined and laminate specimens usually need to be prepared with mounting resin before observation, following a costly and time-consuming procedure. Moreover, small voids with characteristic dimensions of few micrometers can result of difficult detection [1, 16]. Micrographs at the same or higher magnification can be gathered at SEM with remarkably better clarity and capacity to distinguish the different constituents and possible artefacts deriving from specimen preparation [19]. The images of laminates were obtained by a SEM Hitachi Table-Top TM 3000. Sections of specimens were polished using a sequence of abrasive papers (P300, P600, P1200 and diamond paste of 6 μ m), as suggested by Clements [20]. Polished specimens were washed into an ultrasonic bath, dried and observed at SEM. Evaluations of void contents by image analysis were done at 50 X on sets of 12 micrographs taken at different locations for each laminate.

High-contrast images were collected and analyzed by a dedicated software (Image Pro Plus[©]) to identify and evaluate void sections, as well as void dimension distribution. After a contrast enhancement, each pore was numbered for subsequent voids count and analysis. An example of the adopted procedure is presented in fig. 1, which shows the elaboration steps from the original micrograph to the final voids count.

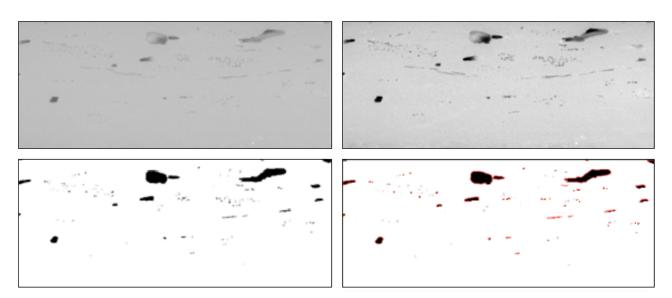


Figure 1: Elaboration steps of a digitalized SEM image for voids counting and analysis.

2.4.2 X-rays micro-computed tomographic analysis

Specimens 20 mm x 7 mm x thickness were analyzed in a micro CT scanner (Varian BIR Actis 130/150, resolution $5 \mu m$), threshold density values for the segmentation and resin/fiber/void phases identification were arbitrarily selected; this may introduce some uncertainty in the void content estimated on the basis of reconstructed volume images; however, this is expected to be limited considering the marked density difference between voids and composite constituents [6, 9]. No analysis of voids shape was performed.

2.4.3 Density and acid digestion measurements

Small specimens were weighted (0.5-1 g) and their density was measured according to ASTM D792. The specimen were digested in sulfuric acid/hydrogen peroxide and the recovered fibers were washed and weighted. The fiber and void amounts were calculated on the basis of composite, matrix, fiber weight and density according to ASTM D3171.

2.5 Mechanical tests

The laminates were mechanically tested to estimate the effects of different curing cycles and void content over tensile, compressive and inter-laminar properties.

2.5.1 Short Beam Shear (SBS) tests

SBS tests were carried out according to ASTM D2344 standard. Although the test is intended to characterize unidirectional fiber composites, considering that all tested laminates had equal fibers, matrix and lamination sequence the test was adopted for comparison purposes [4]. A MTS 858 loading machine with 10 kN load cell was employed. The testing procedure requires short-beam specimens with high thickness/length ratio, therefore only 18 layers laminates were employed; at least 5 specimens out of each laminate type were tested at 1 mm/min crosshead speed; specimen dimensions were 35 mm length x 13 mm width; laminate thickness ranged between 4.9 and 5.9 mm according to laminate type.

2.5.2 Tensile tests

Tensile tests were performed according to ASTM D3039 standard. Due to gripping problems with thick specimens, which showed failure in correspondence of grips, only 6 plies laminates were tested at 2mm/min crosshead speed. Rectangular specimens 250 mm x 25 mm, with thickness ranging between 1.60 and 1.97 mm were used. An Instron 5982 load testing machine with 100 kN load cell and 50 mm gage length extensometer was employed.

2.5.3 Compression tests

Compression tests were performed according to ISO 14126 standard (method 2). Rectangular specimens 12.5 mm x 110 mm were cut from 6 layers laminates and end-tabbed with the same type of material. The same Instron dynamometer/load cell above indicated was used at 1 mm/min crosshead speed.

3 RESULTS AND DISCUSSION

DMTA tests of prepregs during different curing cycles allow to estimate gel times and compare curing evolution. In fig. 2, the variation of prepreg conventional viscosity, measured by DMTA as complex viscosity, during ramp + isotherm curing cycles is reported for 155 °C to 175 °C maximum temperature. With these cycles, minimum viscosity levels are maintained up to times exceeding 50 min. In order to minimize voids, compaction pressure should be applied before resin gelation and viscosity increase [21]. As a matter of facts, with cycles C and D, maximum pressure is reached in somewhat shorter times. The gel times estimated in all tested cycles (isotherms and ramp + isotherms) are plotted in fig. 3. It can be observed that gel times decrease with increasing maximum temperature; in cycles with 180 °C and 185° C maximum temperature, no further marked reduction of gel times was recorded; this suggests that in actual productions, gelation occurs when the temperature reaches about 175 °C.

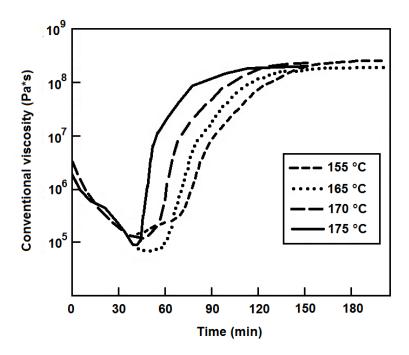


Figure 2: Evolution of prepreg viscosity (conventional) during ramp + isotherm curing cycles

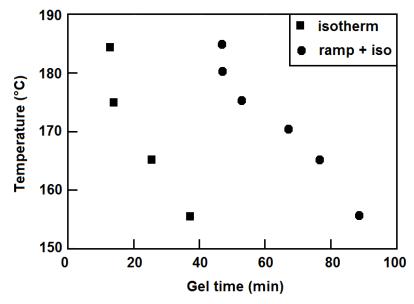


Figure 3: Gel-time estimated by DMTA tests in isotherm conditions and in ramp + isotherm cycles.

This is also confirmed by DSC analysis reproducing a ramp + isotherm curing cycle (fig. 4). It can be observed that the exotherm peak approximately corresponds to the reaching of the maximum temperature (175 °C). During the following isotherm, the evolved heat flow progressively reduces until the reaction is nearly complete.

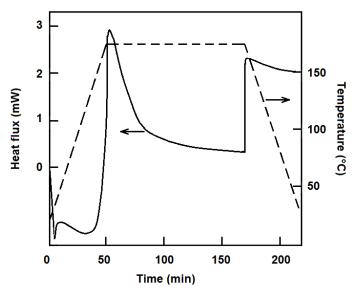


Figure 4: DSC analysis of a ramp + isotherm cycle

The evaluation of residual heat in produced laminates confirmed that curing was equally complete at the end of all cycles; only on heating at higher temperatures, an additional small exotherm peaking at about $270~^{\circ}$ C was presented.

The measurement of laminates thickness obtained with definite number of plies (Fig. 5) gives a first indication of void presence in different laminates. It can be observed that on increasing applied pressure, relevant thickness reduction results for all lamination sequences. However, considering that resin flow is consistently influenced by number of plies, resin temperature and autoclave pressure, no reliable void estimation can be obtained from such data only.

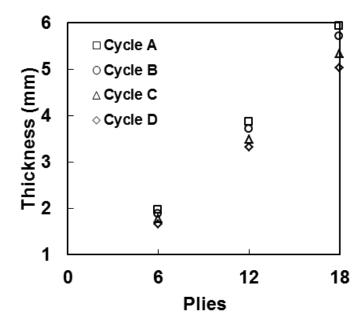


Figure 5: Measured thickness of laminates produced with different curing cycles.

3.1 Image analysis

The estimation of void content from 2D images obtained at the microscope is subjected to intrinsic errors due to the discrete nature of observations. For high volume fractions of homogeneously distributed voids and/or

extremely large scanned areas, a correspondence between areal and volume fractions is commonly accepted. When few voids with undetermined geometry are present, discrepancies between 2D measurements and actual volume cannot be avoided; such discrepancies are dependent on a number of geometrical as well as statistical variables [22]. However, under the assumption of void contents below 10 % volume and for voids dimensions smaller than 0.1 mm, i.e. consistent with analyzed specimens, a statistical analysis shows that an observation area of about 10^{-5} - 10^{-4} m² could be sufficient to get volume fraction estimates within 20 % error [22].

For a better comparison of different methods for void content estimation, the designated techniques were applied to selected specimens (20 mm x 7 mm x 1.7/2 mm) with different void amount, which were cut at specific locations of plates cured according to cycles A, B, C. These were analyzed in sequence through image analysis, micro-computed tomography and acid digestion.

The results of the different void measurements are reported in figs. 6 and 7. The three bars with different gray levels in each column in fig. 6 refer to the same specimen and give an indication of the different void detection capability of the different techniques. On the other hand, the different heights of equally coloured bars within the same cycle are determined by detection accuracy of the method, but also by possible actual difference in the void content of the specific specimens. An estimation of absolute accuracy of each method cannot be derived, since no absolute reference is available. By arbitrarily assuming as reference volume of voids that resulting from conventional acid digestion (fig. 7), differences in measured voids are within 1.2% and 0.5% for microtomography in case of high void content (7-8%) and low void content (3-4%) respectively; for SEM image analysis, differences are within 2.4% and 1.2% in case of high and low void content respectively.

As a general trend, it can be observed that micro CT scanning and acid digestion produce similar average results. As a matter of facts, in both methods information comes from the whole specimen volume. Somewhat lower values are found by SEM image analysis, which analyses plane sections cut at random positions. However, in the range of low void contents, which is of interest for practical situations, comparable results are confirmed. When voids exceed 5-6% volume, which implies that more than 10-12 % resin is missing in the laminate, higher discrepancies are observed with the different methods. In such situations, voids orientation effects are expected, which may lead to estimation errors, different for the different methods.

It is worth noting that since the surface polishing and the focusing is definitely less critical in SEM analysis than in optical microscopy observations, the specimens preparation becomes considerably faster and less cumbersome and SEM images of sufficient area can be easily gathered few minutes. As a result, also in terms of investigation time and cost, SEM-image analysis can be considered very convenient compared to optical microscopy-image analysis but also compared to acid digestion and microCT scanning, which require very long specimens processing or scanning times respectively.

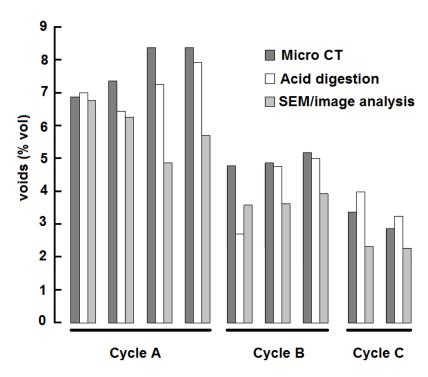


Figure 6: Voids measured in laminates produced with different curing cycles.

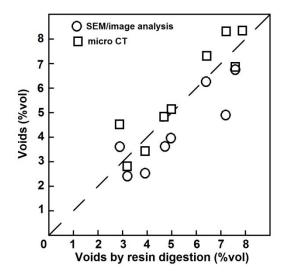


Figure 7: Comparison among three analysis techniques.

After the comparison of different techniques, the void content of all produced laminates was measured by SEM /image analysis by observations of strips cut from all panels. The average void content estimated from 12 micrographs for each panel are reported in Table 1. As a general trend, it can be observed that laminate thickness doesn't seem to be of clear importance for the void content. It is also observed that, although a medium autoclave pressure level (3 bar) is about sufficient to limit void to a fairly low level, higher pressure (7 bar) effectively reduces void to negligible values.

	6 plies	12 plies	18 plies
	void content	void content	void content
	(% vol)	(% vol)	(% vol)
Cycle A	7.2	6.6	6.6 (*)
Cycle B	5	4.4	4.6 (*)
Cycle C	2.2	1.4	1.7
Cycle D	< 0.1	< 0.1	< 0.1

^(*) not fully homogeneous, as indicated by UT

Table 1: Void contents measured by SEM/image analysis in differently cured laminates.

SEM/image analysis allows the counting and the dimension estimation of individual voids. Fig. 8 reports, as example, the dimension distributions recorded in specimens obtained by cure cycle B. It was observed that only with cycle A and B (no autoclave pressure applied) some voids reached diameters in the order of few hundreds of micron. In no case larger voids, possibly indicating presence of extended dry areas or delaminations, were detected.

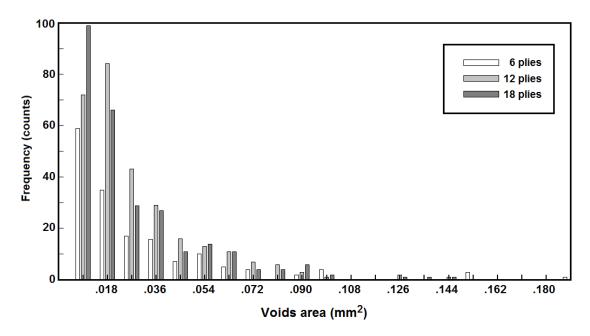


Figure 8 – Voids dimensions measured by image analysis in laminate sections produced with cure cycle B

3.2 Mechanical tests

It is well recognized that the presence of voids in the laminates influences considerably the mechanical response. On the other hand, a comparison of mechanical behavior of laminates obtained by different processing conditions should take into proper account also other effects induced by the manufacturing procedure. Given the same temperature cycles adopted, no appreciable variation of curing advancement was expected; this was however checked and confirmed by DSC analysis of cured laminates. Conversely, since pressure cycles affect resin flow, somewhat different fiber contents were expected, as also indicated by measured thickness values (fig. 5). As a consequence, fiber–dominated properties should be compared with reference to fiber content/number of fabric plies, instead of overall specimen thickness.

All tensile and compression tests were carried out on differently processed laminates with the same number of plies, so the corresponding break load was compared. For a better comparison, void content actually measured by SEM/image analysis on each specimen set was considered.

As expected, interlaminar properties are remarkably affected by voids. In particular, a marked decrease of SBS with increasing void volume was observed (fig. 9); a reduction of SBS of about 25% with 6.6 % void was estimated, which is also consistent with measurements reported for different composites [4].

The tensile strength of fiber composites is a fiber dominated property. As a matter of facts, when compared on the break force basis, laminates with different void content give quite similar results, indicating a limited, yet not negligible, effect of voids. A reduction of tensile failure load limited to about 4% was recorded in laminates with 4-6 % void (fig. 10). The presence of voids with relevant dimensions, comparable to the critical fiber length, can result in localized accumulation of fiber breaks, which can activate laminate failure; in carbon/epoxy systems, critical lengths in the range of fractions of mm, i.e. comparable to many of voids dimensions observed in present laminates (fig. 8), have been measured [23].

The presence of voids appears to affect in a more relevant way the compressive strength; the results of compression tests, again reported in fig. 10 as failure load vs. void content, indicate a reduction of the compression load-carrying capacity of about 10 % when about 4% voids are present. Compressive failure involves fiber buckling, which leads to reduced strength compared to tensile loading situations; fiber microbuckling is particularly effective in fabric reinforced laminates, where fiber misalignment is intrinsically present [24]. As a matter of facts, compressive failure loads less than 50% of tensile loads were found (fig. 9 and 10). A possible explanation for the apparent increase of compressive strength with cycle A laminates is related to the lower resin flow experienced compared to cycle D: a higher resin content within fiber bundles may improve the buckling load irrespective of the presence of small voids in the resin-reach zones. As already mentioned, in the case of cycle A, laminates with higher thickness (about 18% thicker than cycle D laminates) and higher resin content, due to consistently lower resin flow are obtained.

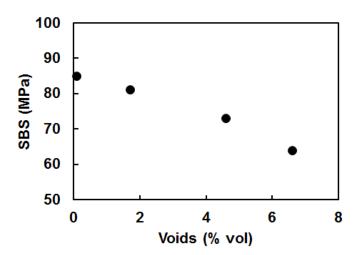


Figure 9 – SBS strength as function of void content

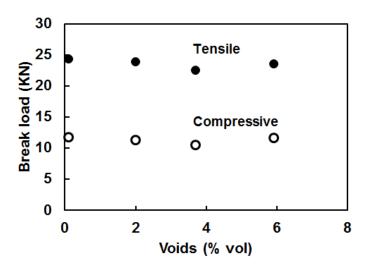


Figure 10 - Tensile and compressive failure load as function of void content

4 CONCLUSIONS

The investigation about the generation and detection of voids in composite laminates, as well as of their influence over final performances of composite laminates rises great interest particularly in case of composites employed in structural applications. Thermal and rheological analysis of the curing prepreg is indicated as a powerful tool to define a relationship between processing parameters and void generation. Different void detection methods are compared and their advantages/disadvantages are discussed on the basis of obtained results. SEM-image analysis appears as a simple, direct and relatively fast technique, compared to other methods, such as optical microscopy-image analysis, CT scan or matrix digestion.

The presence of voids is confirmed as a main issue affecting general laminate mechanical performances, although also other processing dependent and relevant aspects, such as the different fiber/resin content, should be taken into account for a correct comparison of differently processed materials. As a concluding remark, it should also be considered that prepregs with different fabric styles or thread dimension, as for instance with glass fibers, can have a different quantitative mechanical response to the presence of voids.

5 ACKNOWLEDGEMENTS

The authors wish to thank A. Ferrarese and M.R. Pagano for their contribution to the experimental activity. The technical and financial support of Agusta-Westland is gratefully acknowledged. Large part of experimental activity was carried out at Lab. AMALA of Politecnico di Milano.

6 REFERENCES

[1] A.H. Kite, D.K. Hsu, D.J. Barnard, *Determination of porosity content in composites by micrograph image processing*, 34th Annual Review of Progress in Quantitative Nondestructive Evaluation, **975**, 942-949 (2008).

[2] B.S. Hayes, J.C. Seferis, R.R. Edwards, *Self-Adhesive Honeycomb Prepreg Systems for Secondary Structural Applications*, Pol. Composites, **19**, 54-64 (1998).

- [3] B.S. Hayes, E.N. Gilbert, J.C. Seferis, *Scaling complications of dual temperature cure resin prepreg systems in airplane part manufacture*, Composites Part A, **31**, 717-725 (2000).
- [4] M.L. Costa, S.F.M. D'Almeida, M.C. Rezende, *The influence of porosity on the interlaminar shear strength of carbon/epoxy and carbon/bismaleimide fabric laminates*, Composites Science and Technology, **61**, 2101-2108 (2001).
- [5] S.F.M. D'Almeida, Z.S.N. Neto, *Effects of void content on the strength of composite laminates*, Composites Structures, **28**, 139-148 (1994).
- [6] A. Makeev, Y. Nikishkov, *Fatigue Life Assessment For Composite Structure*, Proceedings of the 26th Symp. of the Int. Committee on Aeronautical Fatigue, Montreal, Canada, 1-3 June 2011, p. 119.
- [7] H. Koushyar, S. Alavi-Soltani, B. Minaie, M. Violette, *Effects of variation in autoclave pressure*, temperature, and vacuum-application time on porosity and mechanical properties of a carbon fiber/epoxy *Composite*, Journal of Composite Materials, **46**, 1985-2004 (2012)
- [8] B. Arthurs, D.J. Bull, V. Arumugam, A.R. Chambers, C. Santulli, *Porosity Effect on Residual Flexural Strength Following Low Energy Impact of Carbon Fibre Composites*, Polymers and Polymer Composites, **23**, 205-212 (2015).
- [9] J. Kastner, B. Plank, D. Salaberger, J. Sekelja, *Defect and Porosity Determination of Fibre Reinforced Polymers by X-ray Computed Tomography*, 2nd Int. Symp. on NDT in Aerospace, Hamburg, 22-26 nov. 2010
- [10] D. Gueribiz, F. Jacquemin, M. Rahmani, S. Fréour, R. Guillén, K. Loucif, *Homogenization of moisture diffusing behaviour of composite materials with impermeable or permeable fibres Application to porous composite materials*, Journal of Composite Materials, **43**, 1391-1408 (2009).
- [11] T. Chu, S.S. Russell, J.L. Walker, *Porosity measurement in laminate composites by thermography and FEA*, Proc. of the 2001 Soc. for Experim. Mech. (SEM) Ann. Conf., Portland, OR, June 2001, p. 571-578.
- [12] E.A. Birt, R.A. Smith, A review of NDE methods for porosity measurement in fibre-reinforced polymer composites, Insight, **46**, 681-686 (2004).
- [13] I.M. Daniel, S.C. Wooh, I. Komsky, *Quantitative porosity characterization of composite materials by means of ultrasonic attenuation measurements*, Journal of Nondestructive Evaluation, **11**, 1-8 (1992).
- [14] L. Liu, B.M. Zhang, D.F. Wang, Z.J. Wu, *Effects of cure cycles on void content and mechanical properties of composite laminates*, Composite Structures, **73**, 303-309 (2006).
- [15] H. Zhu, B. Wu, D. Li, D. Zhang, Y. Chen, *Influence of Voids on the Tensile Performance of Carbon/epoxy Fabric Laminates*, J. Mater. Sci. Technol., **27**, 69-73 (2011)
- [16] C. Santulli, R.G. Gil, A.C. Long, M.J. Clifford, *Void content measurements in commingled E-glass/polypropylene composites using image analysis from optical micrographs*, Science Engineering Composites Materials, **10**, 77-90 (2002).
- [17] Hexcel Hexply® 6376 175 °C Curing Epoxy Matrix Material Safety Data Sheet, 2007

- [18] Hexcel Hexply® 6376 175 °C Curing Epoxy Matrix Technical Data Sheet, 2007. http://www.hexcel.com
- [19] S. Paciornik, S.F.M. D'Almeida, *Measurement of Void Content and Distribution in Composite Materials through Digital Microscopy*, Journal of Composite Materials, **43**, 101-112 (2009)
- [20] L.L. Clements, *Fiber Composite Materials*, in Metals Handbook, vol.9 -Metallography and Microstructures, 9th ed., pp.587-597, ed. American Society for Metals, 1985.
- [21] A. Baker, S. Dutton, D. Kelly, *Composite Materials for Aircraft Structures*, cap. 5, p.127, 2nd ed., AIAA, Reston VA, 2004
- [22] B.F. Dyson, M.S. Loveday, M.G. Gee, *Materials Metrology and Standards for Structural Performance*, cap. 13, p.274, Ed. Springer, 1995
- [23] L. Di Landro, A.T. DiBenedetto, J. Groeger, *The Effect of Fiber-Matrix Stress Transfer on the Strength of Fiber Reinforced Composite Materials*, Polymer Composites, **9**, 209-221 (1988).
- [24] N.A. Fleck, Compressive failure of fiber composites, Adv. Appl. Mech, 33, 43-117 (1997)