



HAL
open science

ASSESSMENT OF THE EFFECTS OF VOIDS ON SOME PHYSICAL, MECHANICAL AND DAMAGE MECHANICS PROPERTIES OF CARBON/POLYMERIC COMPOSITES

Ph Olivier, Philippe Marguerès, Benoit Mascaro, K. E. Tarsha Kurdi, Francis
Collombet

► **To cite this version:**

Ph Olivier, Philippe Marguerès, Benoit Mascaro, K. E. Tarsha Kurdi, Francis Collombet. ASSESSMENT OF THE EFFECTS OF VOIDS ON SOME PHYSICAL, MECHANICAL AND DAMAGE MECHANICS PROPERTIES OF CARBON/POLYMERIC COMPOSITES. 12th European Conference On Composite Materials, Aug 2006, Biarritz, France. hal-04496787

HAL Id: hal-04496787

<https://hal.science/hal-04496787>

Submitted on 8 Mar 2024

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

ASSESSMENT OF THE EFFECTS OF VOIDS ON SOME PHYSICAL, MECHANICAL AND DAMAGE MECHANICS PROPERTIES OF CARBON/POLYMERIC COMPOSITES

Ph. Olivier¹, Ph. Marguerès¹, B. Mascaro², K.E. Tarsha-Kurdi³ and F. Collombet¹

1 Laboratoire de Génie Mécanique de Toulouse, Equipe PRO²COM,
Dépt. GMP, IUT Paul Sabatier, 133C av. de Rangueil, BP 67701, F 31077 Toulouse CEDEX 4
2 Laboratoire PHASE, Université Paul Sabatier, UFR PCA, Bât 3R1B2, 118 rte. De Narbonne, F 31062
Toulouse CEDEX 4
3 Faculty of Mechanical Engineering, University of Aleppo, SY Aleppo
philippe.olivier@iut-tlse3.fr

ABSTRACT

The work synthesized in this paper focuses on the effects of voids upon some “conventional” mechanical characteristics of composites laminates (i.e. moduli, strengths) as well as on physical properties (i.e. C.T.E., moisture absorption). The reader will also be provided with a first analysis of the effects of voids upon mode I delamination (i.e. critical strain energy release rate). Lastly, owing to ultrasonic (US) absorption measurements, a criterion of void acceptability is tentatively settled. The underlying idea is to demonstrate that interrelating US absorption with void content and laminates mechanical behaviour should enable quality control costs — associated to testing of test pieces cured with structurally important parts — to be reduced by avoiding performing some of the mechanical and chemical tests.

1. INTRODUCTION and AIMS

Voids in composite materials have been extensively studied for the twenty past years. More accurately, concerning voids in composites manufactured from autoclave-cured prepregs the various studied topics are:

- Explaining the origins of voids [1,2];
- Modelling laminates compaction during cure cycle [3,5];
- Modelling voids growth and development [6,7];
- Analysing the effects of voids upon mechanical properties [7,9];
- Modelling the mechanical behaviour of porous laminates [10,12];
- Correlation ultrasonic absorption with void content [13,16].

In spite of a wealth of public literature, some aspects of voids effects still need to be highlighted today. Only a few authors have focussed their work upon the relation between voids and physical properties of laminates. For instance, B.D. Harper et Al. [17] have devoted a part of their research work to analysing moisture diffusion in porous laminates (see also H.S. Choi’s work [18]). Unlike conventional mechanical properties (tensile / compressive / bending moduli, strengths), it seems that none attention has been paid to specific mechanical properties such as fracture toughness (R), critical strain energy release rates (G_{IC} , G_{IIC} , etc.), yet. For this reason and given that the exact mode of failure of composite is influenced by the load-transfer mechanism provided by the composite microstructure (i.e.: voids may act as local flaws), it has been decided to initiate a first analysis of the relations between G_{IC} and the void content of unidirectional laminates. The

preliminary bibliographic study carried out before starting this work has also revealed that the thermomechanical behaviour of laminates undergoing various void contents was still a topic to inspect. Lastly, the absorption of ultrasonic (US) signals in porous laminates has been studied with a twofold purpose. The first purpose was not only to provide the reader with a relation between of ultrasonic absorption and void content — as made in references [13, 16] — but also to take into account the frequency of the ultrasonic signal. The second purpose was to correlate US signals with fracture mechanics properties (e.g. interlaminar shear strength) in order to settle a criterion of void acceptability based on a reference characteristic (mode II critical strain energy release rate). The short-term underlying idea linked to US absorption measurement and correlation to mechanical characteristics is to demonstrate that this kind of measurements can be introduced in a quality assurance procedure. In other words, the aim is to reduce mechanical and chemical quality control costs on test pieces cured with the structural components.

2. MATERIAL, MANUFACTURING CONDITIONS and VOID CONTENT

2.1- Material

The main material used for this work is the prepreg HexPly™ UD M21/35%/268/T700GC manufactured by Hexcel Composites France. This prepreg is made of a unidirectional tape (average ply thickness: 260 μm) of T700GC carbon fibres pre-impregnated with the polymeric M21 resin. The initial resin content by weight (measured on prepreg supplied to our Lab.) is $W_r^0 = 37.4\%$.

Results from a previous study [20] were also re-analysed here (ultrasonic testing). The material used in this former work was the Hexcel Composites T2H-EH25-34% carbon/epoxy prepreg.

2.2- Manufacturing conditions

From a thermal point of view, all the laminates were cured according temperature path recommended by the manufacturer of the prepreg for monolithic parts < 15 mm thick [19]. Concerning autoclave pressure route, it has been shown more than ten years ago [20] that adjusting the autoclave pressure (applied at cure cycle beginning) enables to obtain laminates undergoing various void contents. Consequently, this procedure was applied here with the following autoclave pressure values: 0; 0.2; 0.5 and 0.7 MPa. In addition, for each pressure value, a set of laminates (denoted by NPC for “non-pre-compacted”) was cured without performing any vacuum pre-compaction during lay-up, while another set (denoted by PC) was submitted to a 20 min. ply-by-ply pre-compaction under vacuum (-0.085 MPa).

2.3- Void contents after curing ($V_v(\%)$)

The void contents were determined on coupons cut out from $[0^\circ_8]$ unidirectional laminates according to both resin dissolution and image analysis. The obtained results are plotted in figure 1 versus autoclave pressure. Concerning void contents determined by resin digestion, each point is the average of five measurements. Each one of the void contents determined by image analysis has been obtained from twenty micrographic pictures taken on samples cut-out from $[0^\circ_8]$ laminates. Ten images were perpendicular to fibres and 10 parallel to fibres. They were analysed using ImageJ software with specific plug-in. It can be noticed in

Figure 1 that the effects of the 20 min. ply-by-ply pre-compaction under vacuum are quite negligible with this kind of prepreg, while they were considerable with previous generations (epoxy matrix) [20]. From now and until the end of this paper, the values of void contents $V_v(\%)$ used are those determined by image analysis.

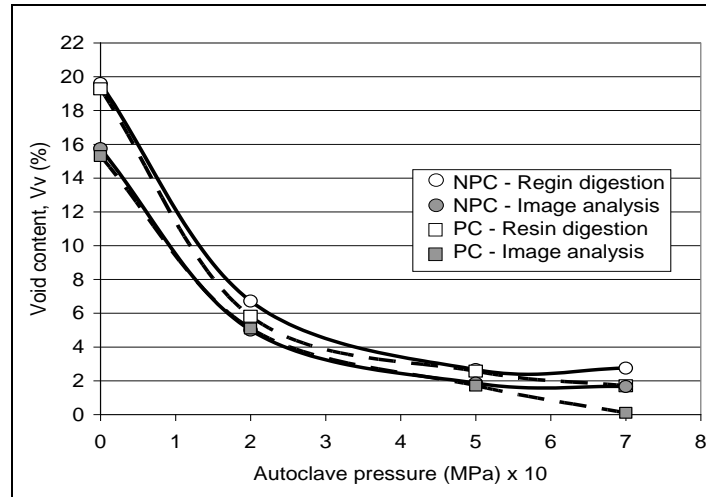


Figure 1: Changes in $[0^\circ_8]$ laminates void content as functions of autoclave pressure and vacuum pre-compaction. Void contents determined either by resin digestion or image analysis.

3. VOIDS and PHYSICAL CHARACTERISTICS

3.1- C.T.E.

The coefficients of thermal expansion (CTE) of $[0^\circ_8]$ porous laminates were determined using a Perkin-Elmer TMA7 according three different directions (see Figure 2). The obtained results show that the average value of the longitudinal CTE α_x (// to fibres) decreases by less than 10% when the void content ($V_v(\%)$) increases up to 20%. For identical changes in $V_v(\%)$ (i.e. from 1.7 up to 19.6%), α_y and α_z decrease respectively by -20% and -30%. Despite of these changes in CTE on a large range of $V_v(\%)$ values, it seems important to note that up to a void content of about 5%, all the CTE values remain constant.

3.2- Moisture absorption

Sets of $50 \times 50 \times 2$ mm samples with $V_v(\%)$ ranging from 1.7 up to 19.6% were cut of from $[0^\circ_8]$ laminates. Within 30 minutes after their curing they were dried at 60°C in an oven for 144 hours (until their weight remain steady) and then placed in a clear room at 22°C and a $50\% \pm 5\%$ RH (relative humidity) for 80 days. These samples were weighted at various times during their stay in the clean room (as shown in figure 3).

4. MECHANICAL BEHAVIOUR DEPENDING ON VOID CONTENT AND ULTRASONIC ABSORPTION

4.1- Ultrasonic testing: absorption depending on void content

The analysis of ultrasonic signals absorption were performed on $[0^\circ_8]$ laminates, immersed

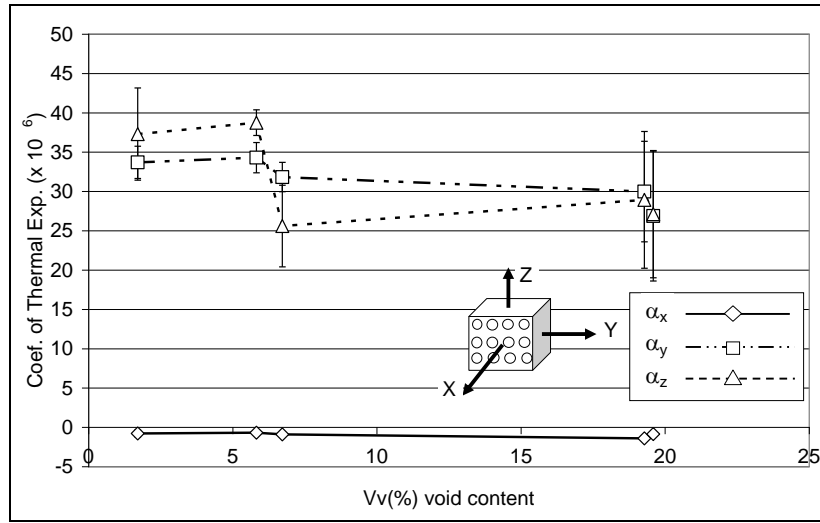


Figure 2: Changes in the three CTE of $[0^8]$ laminates as a function of void content ($V_v(\%)$).

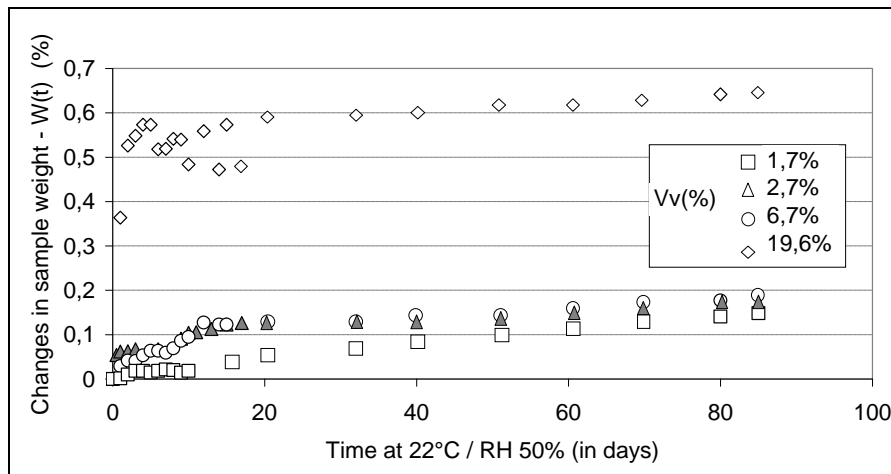


Figure 3: Changes in samples weight (%) during their stay in clean room depending on their void content.

in water, using two 5 MHz transducers (\varnothing 13 mm). One transducer for emission and the other for signal reception. These tests were first carried out on laminates manufactured from HexPly™ UD M21/35%/268/T700GC prepreg, but also on porous laminates manufactured from Hexcel Composites T2H-EH25-34% carbon/epoxy prepreg [20]. Absorption was expressed as a function of frequency (f) within the spectral bandwidth of the US emitting transducer. The absorption coefficient $\alpha(f)$ (in dB/mm) is given by (equation (1)):

$$\alpha(f) = \frac{1}{e} \times 10 \times \log \frac{(T \times |S_o(f)|)^2}{|S_M(f)|^2} \quad (1)$$

With: $S_0(f) = FFT(s_0(t))$; $S_M(f) = FFT(s_M(t))$; $s_0(t), s_M(t)$, signals transmitted in water and throughout material respectively (in dB); e , laminate thickness (in mm); T , coefficient of transmission at interfaces (function of acoustic impedances Z).

The absorption coefficient's slope S_α (in $\text{dB}\cdot\text{mm}^{-1}\cdot\text{MHz}^{-1}$) is the slope measured by fitting the plot of global absorption $\alpha(f)$ (equation (1)) as a function of the frequency band (f) of the transmitted spectrum (see ref. [14]) depending on $V_v(\%)$. The obtained results are gathered in Table 1.

4.2- Tensile and shear properties of porous laminates and S_α

Several mechanical tests have been performed according to E.N. standard on coupons cut out from $[0^\circ_8]$ laminates made from HexPly™ UD M21/35%/268/T700GC prepreg. The changes in longitudinal and transverse tensile moduli (E_l, E_t) and ultimate strengths ($\sigma_{l,u}$; $\sigma_{t,u}$) and those in ILSS have thus been determined as a function of void content. Table 1 summarises the obtained results. They are compared to those obtained more than ten years ago [20] on laminates manufactured from T2H-EH25-34% carbon/epoxy prepreg. Table 1 also shows the values of the slope of absorption coefficient S_α measured on porous laminates.

$V_v(\%)$	$S_\alpha(\text{dB}/\text{mm}/\text{MHz})$	$\sigma_{l,u}(\text{MPa})$	$E_l(\text{MPa})$	$\sigma_{t,u}(\text{MPa})$	$E_t(\text{MPa})$	ILSS (MPa)
T2H-EH25-34%						
0	----	2007	143165	66	9205	98♦
0.3	0.2665	1761	132650	61	8728	90
0.7	0.369	1654	127544	55	8423	89
3.9	0.8159	1603	128084	53	8314	79
6.8	1.2874	1589	131428	49	8160	78
10.3	2.0295	1543	126668	41	7934	69
M21/35%/268/T700GC						
0	----	2282	142000	65	8400	105♦
1.66	0.4673	2039	135923	76	10183	99
1.71	0.4545	1970	134925	67	7520	93
1.86	0.4851	1864	128216	57	7557	92
5	2.1525	1885	124988	46	6340	71
5.09	2.4228	1754	124522	48	6481	70

Table 1: Influence of void content upon tensile characteristics and ILSS of $[0^\circ_8]$ in correlation with $V_v(\%)$ and S_α . ♦: reference values taken for void-free laminates.

4.3- Analysis of changes in ILSS as functions of S_α and $V_v(\%)$

The changes in the interlaminar shear strength of M21/35%/268/T700GC and T2H-EH25-34% $[0^\circ_8]$ laminates have been plotted as functions of S_α and $V_v(\%)$ in figure 4. The experimental points have been fitted using power laws (i.e.: $y = a \cdot x^n$) and this for both materials. It is important to mention that ILSS values measured on void-free laminates are considered as references ones (see ♦ in table 1) and are not used for the fitting. These

regressions enable to define the critical values or critical thresholds of s_{α}^c and v_v^c (%). s_{α}^c and v_v^c (%) are respectively the slope of the absorption coefficient and the void content values from which the mechanical characteristics start to decrease, provided that they remain steady for lower S_{α} and V_v (%) values. s_{α}^c and v_v^c (%) are spotted in figure 4.

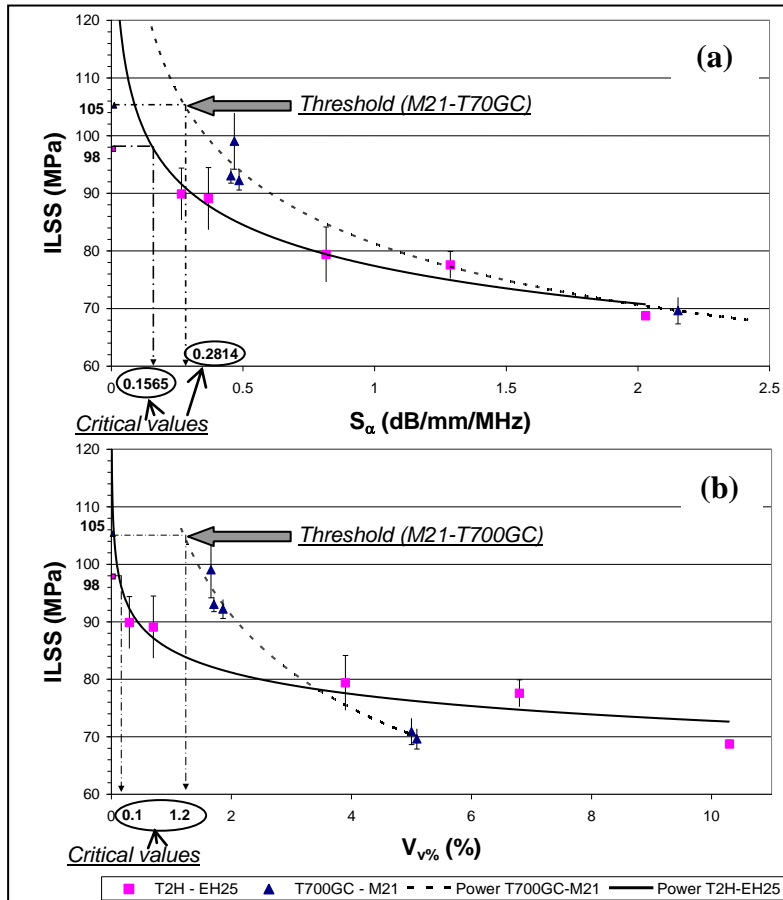


Figure 4: (a): ILSS of porous [0°8] laminates as a function of the slope of absorption coefficient S_{α} and (b): ILSS of porous [0°8] laminates as a function of void content V_v (%).

Similarly s_{α}^c and v_v^c (%) critical threshold values have been calculated for tensile characteristics using equally power laws to fit the experimental data given in table 1. Table 2 gives, for each mechanical property, a comparison of the void contents at the critical threshold determined according to two different ways. These void contents were determined on the one hand from mechanical tests experimental data (as shown in figure 4) and on the other hand from ultrasonic absorption tests via S_{α} . At that point, it should be mentioned that several authors have found that the relation between the absorption coefficient $\alpha(f)$ (in dB/mm) and the void content V_v (%) can be described owing to a linear regression [13,14, 21-22]. In our case, for both materials studied here, it has been found that the logarithms of the void contents $\text{Log}_{10}[V_v]$ and of the slope of the absorption coefficient $\text{Log}_{10}[S_{\alpha}]$ can be linked using a linear relation. Consequently, a power law has been used to correlate S_{α} and V_v (%): $V_v\% = 3.91 \times (S_{\alpha})^{1.77}$ for T2H-EH25-

34% and $V_v\% = 2.90 \times (S_\alpha)^{0.67}$ for M21/35%/268/T700GC. Table 2 data show a good agreement between the two ways of determining critical v_v^c (%) values and therefore the interest of using power laws.

Mech. Propy.	T2H-EH25-34%			M21/35%/268/T700GC		
	S_α^c (dB/mm/MHz)	V_v^c (%)	V_v^c (%) Power law	S_α^c (dB/mm/MHz)	V_v^c (%)	V_v^c (%) Power law
E_l	0.0000	0.00	0.00	0.0945	0.62	0.59
E_t	0.0589	0.02	0.03	0.4455	1.71	1.68
$\sigma_{l,u}$	0.0175	0.00	0.00	0.0196	0.21	0.21
$\sigma_{t,u}$	0.1611	0.12	0.15	0.5062	1.85	1.83
ILSS	0.1565	0.13	0.15	0.2814	1.21	1.24

Table 2: Void content V_v (%) and S_α at critical thresholds for various mechanical properties compared to V_v (%) values computed from results of ultrasonic experiments (S_α values) using a power law.

4.2- Mode I delamination: voids effects upon G_{IC}

Series of $[0^\circ_{16}]$ laminates were manufactured in order to produce mode I delamination specimens. The tests were performed on $0^\circ/0^\circ$ interfaces according to E.N. standard. The critical strain energy release rate G_{IC} was computed according to Berry's method. Figure 5 shows how the voids can modify G_{IC} value. Unlike in porous unreinforced epoxies [23], as expected the voids in composites do not induce any toughening mechanism.

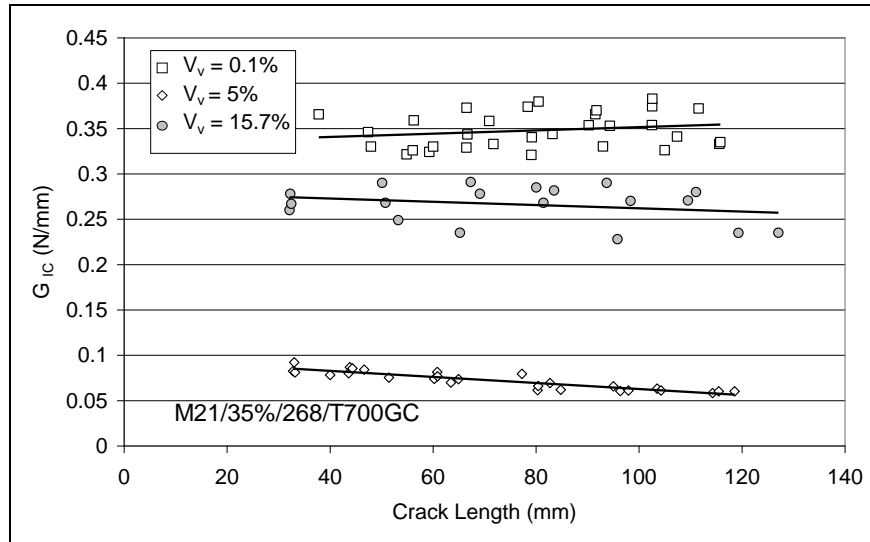


Figure 5: Changes in G_{IC} as a function of void content for a $0^\circ/0^\circ$ interface.

5 CONCLUDING REMARKS

The changes in various physical and mechanical properties as a function of void content have been determined. An interrelation between US absorption and V_v (%) and laminates mechanical behaviour has been settled. This should contribute to reduce the quality control costs by avoiding performing some of the mechanical and chemical tests.

ACKNOWLEDGEMENTS

We gratefully acknowledge Airbus France (ESWT contract #WD042292) for its financial support on voids in composites, Hexcel Composites France for material supply.

REFERENCES

- 1- Kardos, J.L., Dudukovic, M.P, Dave, R., “Void Growth and transport during Processing of Thermosetting-Matrix Composites”, *Advances in polymer Science*, **80** (1986) 101-123.
- 2- Kardos, J.L., Dave, R., Dudukovic, M.P, “Voids in composites”, *Proceedings of ASME Manufacturing International’88, Edited by Gutowski T.G., Atlanta 17-20 April, IV* (1988) 41-48.
- 3- Gutowski, T.G., Cai, Z. et Al., “Consolidation experiments for laminate composites”, *J. of Comp. Mat.*, **21(7)** (1987) 650-669.
- 4- Gutowski, T.G., Cai, Z. et Al., “The Consolidation of composites”, *Proceedings of ASME Manufacturing International’88, Edited by Gutowski T.G., Atlanta Georgia 17-20 April, IV* (1988) 13-25.
- 5- Kim, J.W., Kim, H.G., Lee, D.G., “Compaction of thick carbon/phenolic composites with autoclave method”, *Composite Struct.*, **66** (2004) 467-477.
- 6- Kardos, J.L., Dudukovic, “Void formation and transport during laminate processing: an initial model framework”. *Composite materials: quality assurance and processing, ASTM STP 797, C.E. Browning Editor*, (1983) 96-109.
- 7- Patel, N., Lee, L.J., “Modeling of void formation and removal in liquid composite molding. Part I: Wettability analysis”. *Polymer Comp.*, **17(1)** (1999) 96-103.
- 8- Hancox, N.L., “The effects of flaws and voids on the shear properties of CFRP”, *J. of Mat. Science*, **12** (1977) 884-893.
- 9- Judd, N.C.W, Wright, W.W., “Void and their effects on the mechanical properties of composites - An appraisal”, *SAMPE Journal*, **February 14(1)** (1978) 10-14.
- 10- Farouk, A., Langrana, N.A., Weng, G.J., “Modulus prediction of a cross-ply fiber reinforced fabric composite with voids,” *J. Comp. Mat.*, **1** (1967) 294-309.
- 11- Park, J.Y, Zureick, A.H., “Effects of filler and void content on mechanical properties of pultruded composite materials under shear loading”, *Polymer Comp.*, **26** (2005) 181-192.
- 12- Qui, Y.P., Weng G.J., “Plastic Potential and Yield Function of Porous Materials with Aligned and Randomly Oriented Spheroidal Voids”, *International J. of Plasticity*, **9** (1993) 271-290.
- 13- Costa, M.L., De Almeida, S.F.M., et Al., “The influence of porosity on the interlaminar shear strength of carbon/epoxy and carbon/bismaleimide fabric laminates”, *Composites Science & Tech.*, **61(14)** (2001) 2101-2108.
- 14- Jeong, H., Hsu, D.K., “Experimental analysis of porosity-induced ultrasonic absorption and velocity change in carbon composites”, *Ultrasonics*, **33(3)** (1995) 195-203.
- 15- Stone, D.E.W., Clark, B., “Non destructive determination of void content in carbon fibre-reinforced plastic by measurement of ultrasonic absorption”, *R.A.E. Technical Report*, No 74612 (1974).
- 16- Nair, S.M., Hsu, D.K., Rose, J.H., “Porosity estimation using the frequency dependence of the ultrasonic absorption”, *J. of Non-Destructive Evaluation*, **8(1)** (1989) 13-26.
- 17- Harper, B.D, Staab, G.H., Chen, R.S., “A note upon the effects of voids upon the hygral and the mechanical properties of AS4/3502 graphite epoxy”, *J. of Comp. Mat.*, **21** (1987) 280-289.
- 18- Choi, H.S., Ahn, K.J., et Al., “Hygroscopic aspects of epoxy/carbon fiber composite laminates in aircraft environments”, *Comp. Part A*, **32** (2001) 709-720.
- 19- Technical data sheet of HexPly M21: www.hexcelcomposites.com
- 20- Olivier, P., Cottu, J.P. and Ferret, B., “Effects of cure cycle pressure and voids on some mechanical properties of carbon/epoxy laminates”, *Composites*, **26** (1995) 509-515.
- 21- Mouritz, A.P., “Ultrasonic and interlaminar properties of highly porous composites”, *J. of Comp. Mat.*, **34(3)** (2000) 218-239.
- 22- Liu, L., Zhang, B.M., et Al., “Effects of cure cycles on void content and mechanical properties of composite laminates”, *Comp. Struct.*, **73** (2006) 303-309.
- 23- Kim, N.H., Kim, H.S., “Micro-void toughening and its mechanism”, *J. of Applied Polymer Science*, **98** (2005) 1290-1295.