PROCESS-INDUCED STRESSES AND THEIR INFLUENCE UPON SOME MECHANICAL PROPERTIES OF CARBON / EPOXY LAMINATES. PART 2: STUDY BY ACOUSTIC EMISSION

Kheir-Eddine TARSHA KURDI[1] and Philippe A. OLIVIER[2]

1: Science and Engineering of Materials Department – Faculty of mechanical engineering
University of Aleppo – Aleppo – Syria
2: Laboratoire de Genie Mecanique de Toulouse, Dept. GMP, IUT Paul Sabatier, 133 avenue de Rangueil,
F 31077 Toulouse CEDEX 4, France — e-mail : philippe.olivier@gmp.iut-tlse3.fr

ABSTRACT

This paper syntheses a part of our on-going work upon residual curing stresses. It focuses on the effects of residual curing stresses upon some basic mechanical properties of $[0^0{}_{16}]$, $[90^0{}_{16}]$, $[\pm45^0{}_4]_s$ and $[0^0{}_2/90^0{}_2]_s$ laminates. By modifying the curing condition, three series of UD, angle-ply and cross-ply laminates were obtained with different levels of residual stress. The manufacturing procedure is detailed in a twin paper (iccm11 #B107). All the mechanical tests carried out here are simple tensile tests performed according to European standards. All the samples test were equipped with a four probes acoustic emission device. A preliminary bibliographic study is used as a basis to make the relation between the magnitude of signals emitted by laminates when loaded and the damaging processes [1,2]. Depending on its magnitude each acoustic signal is assigned to different damage mechanisms [1,2]. By this way, The effect of micro and macro process-induced stresses upon the parameters of the mechanical behaviour of each kind of laminate is then assessed.

1. INTRODUCTION

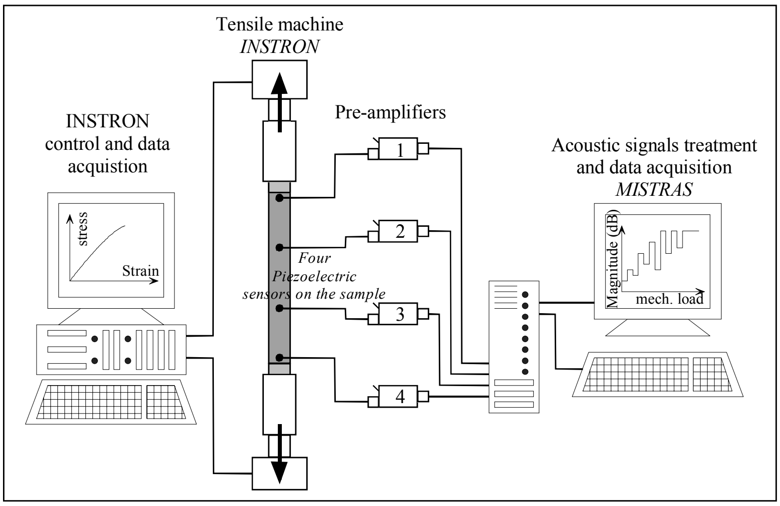
Note to readers: some information given in paper#B107 is used in the present paper.

As mentioned in a previous work [3], comparing the whole literature about residual process-induced stresses with the number of public papers which offer an analysis of the influence of these stresses upon the laminates' mechanical characteristics, clearly shows that only a few authors have dedicated their research work to this specific point. From 1979, Kim and Hahn [5] have studied the first-ply failure taking the residual curing stresses into consideration. The method that they have developed has since then extensively been re-used for the experimental determination of residual stresses' level in cross-ply laminates [6-7]. More recently, some authors have reached the conclusion that the linear behaviour of laminated composites does not seem to be affected by residual curing stresses [8] and have shown that these stresses however affect the non-linear part of laminates' behaviour and plasticity parameters. From a micromechanical point of view, the presence of residual curing stresses at fibre/matrix interface has been both modelled [9] and also integrated into the analysis of single fibre pull-out tests [10-11]. Still from a micromechanical point of view, Papeitis and Galiotis have show how residual curing stresses modify fibre/matrix interfacial failure scheme at room temperature [12]. Among all this literature, our work has consisted in analysing a few aspects of the mechanical behaviour (tensile moduli and ultimate strengths) of various laminates undergoing different levels of residual curing stresses. To this end, mechanical tests were performed according to European standards on simple stacking sequences laminates. The various events occurring during tensile tests such as matrix cracking, interlaminar delamination, fibres breakage, etc., were recorded owing to an acoustic emission device.

2. ACOUSTIC EMISSION DEVICE AND BIBLIOGRAPHICAL RESULTS

2.1 Acoustic emission equipment

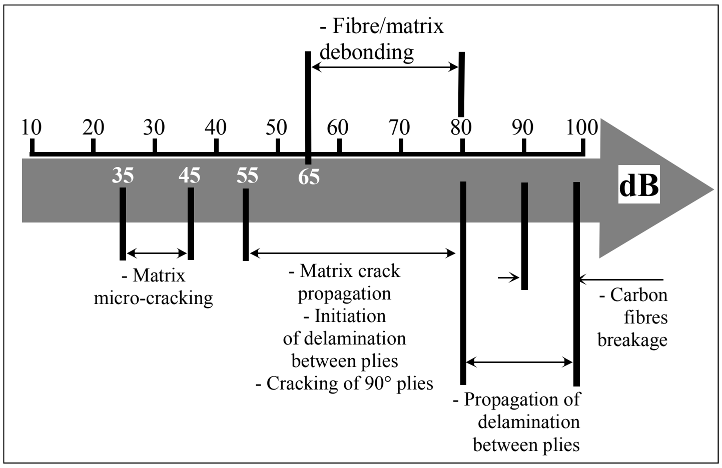
Tensile tests have been performed on an 8561 INSTRON according to conditions described in European standards EN 2561 and EN 2597. An exception has been made about the displacement speed of the INSTRON moving crosshead. A 0.5 mm/min speed was used instead of a 2 mm/min speed recommended in the standards. Lowering the crosshead displacement speed enables to make a more accurate distinction between the acoustic events. The acoustic emission device is schematised in Fig. 1. The tensile test samples were all equipped with four piezoelectric sensors. Two of them are located near the sample end-tabs in order to eliminate noise signals coming from end-tabs and INSTRON jaws.



"**Fig. 1.** Acoustic emission device used during tensile tests."

2.2 Acoustic emission and damaging process: background

Acoustic emission has been used for several year to assess the different events occurring during the mechanical testing of composites [1,2]. All damaging mechanisms for which the propagation ends to cause the fracture of a composite part, generate sounds, noises, elastic waves or acoustic emission. The results obtained by M Benzeggagh [1] are concerning glass/epoxy woven laminates tested in bending. Those of O. Siron [2] on carbon/epoxy are synthesised in Fig. 2. A special attention must be paid to O. Siron results because they were obtained during tensile tests performed on carbon/epoxy laminates autoclave cured. This corresponds to the kind of material and mechanical tests performed in this work. In this work on acoustic emission the magnitude values given in Fig. 2 will be taken as reference for correlating acoustic signals and damaging process.



"**Fig. 2.** Synthesis of refs. [1,2] results: correlation between the magnitude of acoustic signals and kind of damages."

3. MANUFACTURING CONDITIONS AND PHYSICAL CHARACTERISATION

The material used for this study as well as the manufacturing conditions are detailed in iccm11 paper #B107. As explained in this twin paper, the cure cycles were designed to produce different levels of residual curing stresses while all the physical properties of the laminates were imperatively kept constant. In other words, this means that the temperature route of the cure cycles was modified as shown in Fig. 1 of paper #B107, while respecting the following constrained functions:

- matrix degree of cure α measured by DSC: $97\% \leq \alpha \leq 99\%$
- matrix glass transition temperature T_g measured by DSC: $150°C \leq T_g \leq 154°C$
- fibre volume fraction V_f% maximum allowed change for laminates having identical stacking sequences: $\Delta V_f\,\% = \pm\, 0,2\%$
- void content (volume fraction) V_v%: $V_v = 0\%$

As a summary, it can be said that this physical characterisation campaign clearly shows the equivalence between the three cure cycles, since laminates' matrix degree of cure α remains constant and T_g values lie between the allowed limits. Furthermore, if one considers laminates which have the same stacking sequence, curing these laminates at 160, 180 or 200°C does not induce any changes in V_f%. Consequently, none of the changes that could be detected in their mechanical behaviour will be due to a modification of their physical characteristics (please report to iccm11 paper #B107: Tables 1, 2 and 3).

4. PROCESS-INDUCED STRESSES

In this part of our work, $[0°_{16}]$, $[90°_{16}]$, $[0°_2/90°_2]_s$ and $[\pm45°_4]_s$ laminates will be submitted to tensile tests. In order to make an analysis of the influence of residual stresses upon their tensile Table 4 of paper #B107 are not described as a function of matrix degree of cure α since, as previously explained in the "manufacturing conditions" paragraph, the whole study has been performed at a constant value of α.

4.1 Residual stresses on a micromechanical level

It is important to mention that the aim of these calculations on a micromechanical level have never been the set-up of one or other refined modelling. The idea here is simply to get information about the distribution and the level of residual stresses in order to analyse the effects of the stresses upon mechanical behaviour of UD laminates. To this end, the computation were made according to refs. [13,14] modelling, the results are presented in Fig. 3. The input data are given in Table 1 for the fibres. The fibre properties are given according to Fig. 3 coordinates. The epoxy matrix properties used for that computation have determined according to inverse-methods from tests performed on UD specimens [15] (Table 2). As shown by Fig. 3, the preponderant stresses in the micromechanical elementary representative volume are the σ_{rr} and $\sigma_{\theta\theta}$ compressive stresses in the fibre and the σ_{rr} compressive stress in the matrix. Their levels increase when increasing curing dwell temperature. Since the preponderant residual stresses are compressive, a better fibre / matrix adhesion could be expected. In other words this means that the ultimate tensile strengths $\sigma_{xx}^{ult.}$ and $\sigma_{yy}^{ult.}$ of UD laminates should theoretically increase when increasing the level of residual stresses in UD laminates.

"**Table 1.** Properties used for the computation of residual stresses on a micromechanical level."

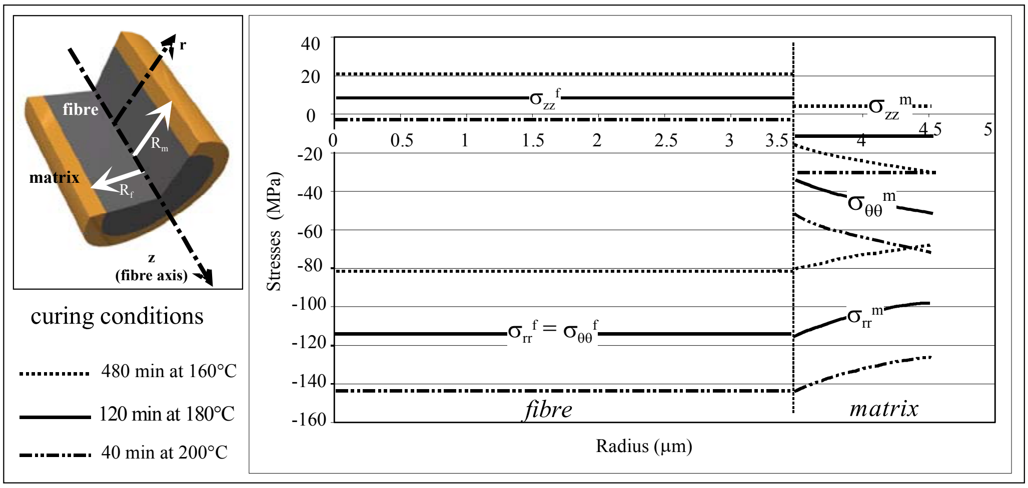
Carbon Fibre (according to ref. [3])									
radius R_f (µm)	E_z (MPa)	E_r (MPa)	G_{zr} (MPa)	ν_{zr}	ν_{rz}	ν_{rr}	α_z (x 10^{-6}) °C^{-1}	α_r (x 10^{-6}) °C^{-1}	V_f%
3.5	230000	14400	50000	0.32	0.02	0.4	-0.1	19.7	60

"**Table 2.** Properties used for the computation of residual stresses on a micromechanical level."

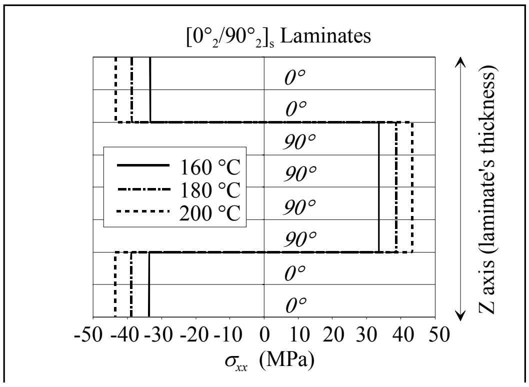
Epoxy resin (according to ref. [3])						
radius R_m (µm)	E_m (MPa)	G_m (MPa)	ν_{mr}	α_m (T° < Tg) (x 10^{-6}) °C^{-1}	α_m (T° > T_g) (x 10^{-6}) °C^{-1}	V_m%
5.5	4500	1800	0.4	70	162	40

4.2 Residual stresses in $[0°_2/90°_2]_s$ and $[\pm45°_4]_s$ laminates

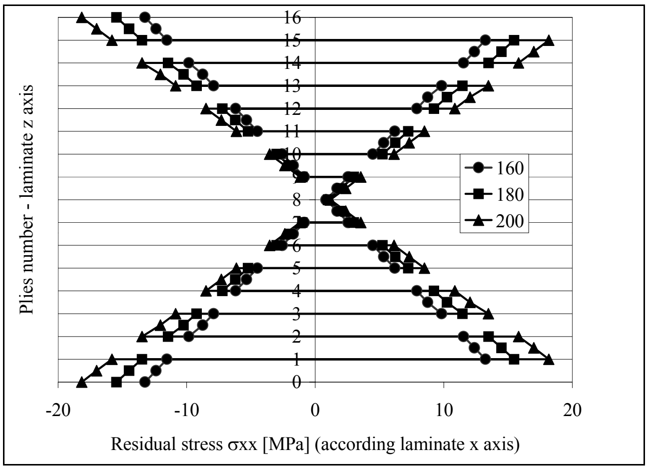
Residual stresses in $[0°_2/90°_2]_s$ and $[\pm45°_4]_s$ laminates were computed using the date summarised in Table 4 of paper #B107 associated to a specific modelling which takes into accounts the effects of laminate / tool-plate interaction [3]. The results for $[0°_2/90°_2]_s$ laminates depending on the curing condition are reported in Fig. 4 while those relative to $[\pm45°_4]_s$ laminates have been plotted in Fig. 4. As it can be seen in Fig.4, increasing curing dwell temperature from 160°C up to 200°C results in an increase by 28% in σ_{xx}. σ_{xx} are the residual stresses in the $[0°_2/90°_2]_s$ laminates x axis (either in 0° or in 90° plies). Concerning $[\pm45°_4]_s$ laminates, the changes in curing conditions have only a small effect on the level of residual curing stresses (Fig. 5). Nevertheless, it will be seen that those small differences in residual stress levels undergone by $[\pm45°_4]_s$ laminates will have a great influence upon the mechanical behaviour of these laminates when submitted to a tensile test.



"**Fig. 3.** Micromechanical elementary representative volume and distribution of residual stresses according z, r and θ."



"**Fig. 4.** Theoretical distribution of residual stresses throughout the thickness of a $[0°2/90°2]_s$ laminate depending on curing conditions."



"**Fig. 5.** Residual stresses theoretical distribution through $[\pm 45°_4]_s$ laminates thickness. Residual stresses are plotted depending on the curing dwell temperature (160, 180 or 200°C)."

5. MECHANICAL CHARACTERISATION AND ACOUSTIC EMISSION

5.1 Mechanical behaviour of [0°$_{16}$] laminates

Mechanical tests have been performed on samples cut out from [0°$_{16}$] laminates cured according to each of the three cure cycles. Fig. 6 shows the typical behaviour of [0°$_{16}$] samples during a tensile test depending on their curing temperature. The left side of Fig. 6 is a plot of the strain versus stress curves, while the right side exhibits the acoustic emission results. The acoustic emission diagrams show both the magnitude of the acoustic signals and the energy released by the sample during the tensile test. Magnitude and energy are plotted versus the tensile load applied to the laminated sample. If it remains difficult to notice any difference in the elastic behaviour (i.e. longitudinal tensile moduli E_{xx}), it can be seen that the longitudinal ultimate tensile strength (denoted by $\sigma_{xx}^{ult.}$) depends on the curing dwell temperature. To make mechanical behaviour more explicit, a synthesis table is included in Fig.6. This table gives moduli E_{xx} and $\sigma_{xx}^{ult.}$ average values depending on curing dwell temperature and duration. It shows that the average $\sigma_{xx}^{ult.}$ of [0°$_{16}$] laminates cured 20 min at 200°C is about 15% higher than the average $\sigma_{xx}^{ult.}$ of [0°$_{16}$] laminates cured 480 min at 160°C. The changes recorded in E_{xx} between the two cure cycles (160 and 200°C) are lower than 6%. One must bear in mind that modulus measurement uncertainty which is only about 0.46%.

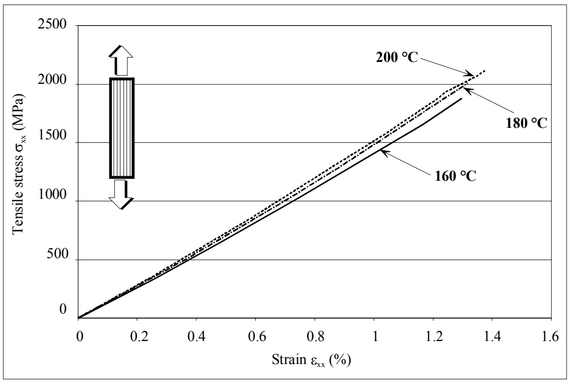
Concerning acoustic emission, only the results relative to laminates cured 480 min at 160°C and 40 min at 200°C are reported on the right side of Fig. 6. From the very beginning of the tensile tests the magnitude of acoustic signals increases quickly to reach the level of 70dB. Whatever the cure cycle is, 70dB signals are recorded for a tensile stress of about 180 MPa. The first signals for which the magnitude reaches 90dB appear for a 250 MPa tensile stress when samples are cured at versus 550 MPa when samples are cured at 200°C. As shown in Fig. 2, 70dB signals can be associated to fibre/matrix debonding and 90 dB (or higher) signals to carbon fibres breakage.

5.2 Mechanical behaviour of [90°$_{16}$] laminates

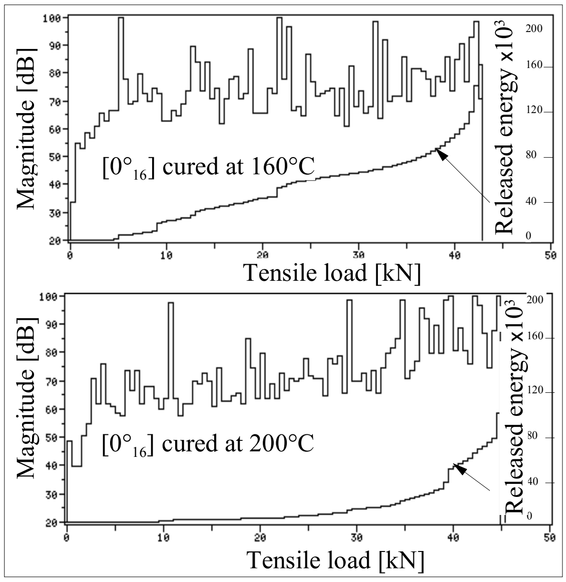
As it has been made for [0°$_{16}$] laminates, the main parameters of the tensile behaviour of [90°$_{16}$] are synthesised in the table included in Fig. 7. Finally, acoustic emission results are reported in Fig. 7 right side. The transverse tensile modulus E_{yy} do not seems to depend on curing temperature. The average changes in E_{yy} between the laminates cured at 160°C and those cured at 200°C do not exceed 2%. The transverse ultimate tensile strength $\sigma_{yy}^{ult.}$ is more sensitive to different cure cycles. A 14% increase in $\sigma_{yy}^{ult.}$ is recorded between laminates cured for 480 min at 160 and those cured for 40 min at 200°C.

Whatever the cure cycle is, all the acoustic signals remain below 60dB. As expected this shows that [90°$_{16}$] samples failure occurs without any fibre breakage and in addition without any fibre/matrix debonding if it is hypothesised that 65dB corresponds to this phenomenon (according to Fig.2 results [1-2]). As a summary, the transverse ultimate tensile strength $\sigma_{yy}^{ult.}$ appears to be higher for laminates cured at 200°C than for those cured at 160°C while the residual stresses (from a micromechanical point of view) are lower in laminates cured at 160°C than in those cured at 200°C. An explanation could be found in ref. [9].

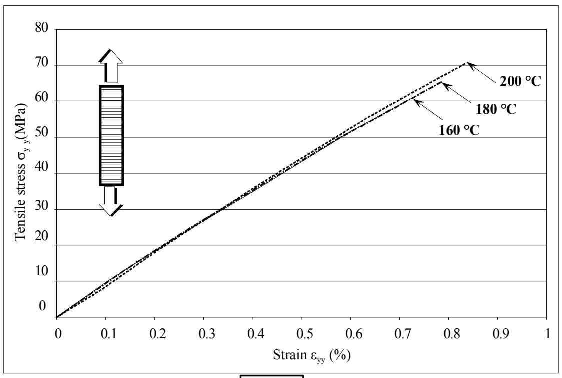
Effectively, as shown in ref. [9], when the critical normal stress on fibre/matrix interface is small, the ultimate tensile strength of [90°$_n$] laminates increase together with the level of residual curing stresses. This is what it can be seen with our results on [90°$_{16}$] laminates which confirm T. Ishikawa's conclusion on off-axis strength of UD laminates [9]. However, the problem to solve on this particular point, is that acoustic emission diagrams (Fig. 7) do not exhibit any signal equal to or higher than 65dB the magnitude at which fibre/matrix debonding occurs !



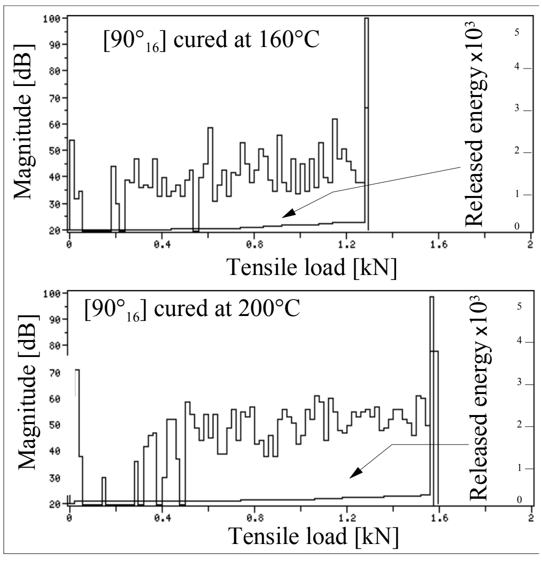
Curing dwell	E_{xx} (MPa)	$\sigma_{xx}^{ult.}$ (MPa)
160°C/480min	136350	1775
180°C/120min	142340	2005
200°C/40min	145110	2080



"**Fig. 6**. Results of tensile tests and acoustic emission on [$0°_{16}$] samples. The table gives moduli and ultimate strengths average values depending on curing dwell temperature and duration."



Curing dwell	E_{yy} (MPa)	$\sigma_{yy}^{ult.}$ (MPa)
160°C/480min	9170	60
180°C/120min	9200	65
200°C/40min	9370	70



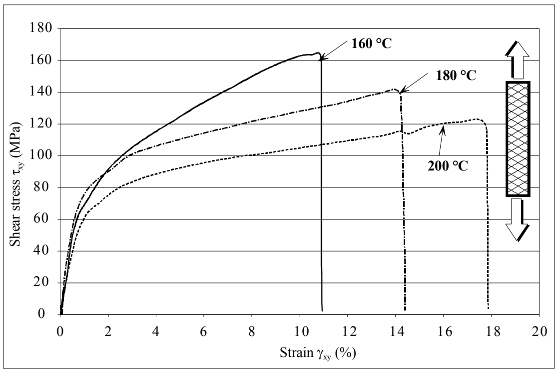
"**Fig. 7**. Results of tensile tests and acoustic emission on [$90°_{16}$] samples. The table gives moduli and ultimate strengths average values depending on curing dwell temperature and duration."

5.3 Mechanical behaviour of [±45°$_4$]$_s$ laminates
As shown by results reported in the table located in Fig. 8 (left side), shear modulus and ultimate strength are higher for laminates cured at 160°C than for those cured at 200°C. In that case, the mechanical response of the [±45°$_4$]$_s$ samples is dominated by shear effects (τ_{xy} shear stress in principal material coordinates see ref. [13]) and, as shown by the stress versus strain curves in Fig.8, large inelastic strains.

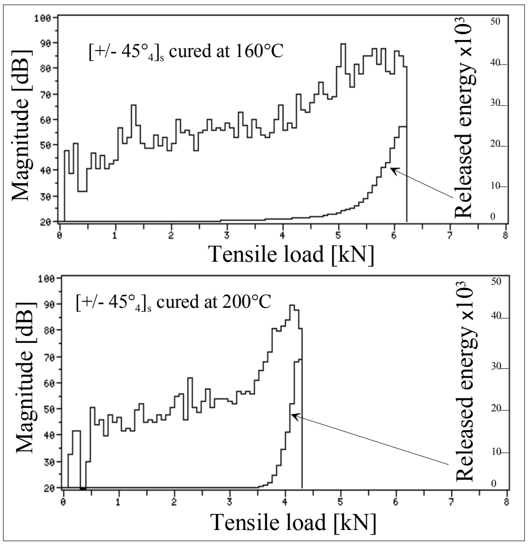
As expected and shown in the right side of Fig. 8 the laminate failure occurs without any fibre breakage, since none of the acoustic signals reach and go beyond 90dB. Let's now assume (as previously made) that for our laminates a 65dB magnitude [1-2] effectively corresponds to fibre/matrix debonding. In that case, for laminates cured at 160°C or at 200°C, the very firsts fibre/matrix debondings occur respectively for a 210 and a 180 MPa mechanical tensile stress (Fig. 8 right side). Consequently, this shows that angle-ply tensile test is the loading case for which the influence of residual curing stresses upon the laminates ultimate strength appears the more clearly. Indeed, increasing the residual stress level at +45°/-45° interfaces by changing the curing conditions results in a weakening of [±45°$_4$]$_s$ laminates, as shown by the experimental results (Fig. 8).

5.4 Mechanical behaviour of [0°$_2$/90°$_2$]$_s$ laminates
Symmetrical cross-ply laminates exhibit a tensile behaviour which clearly depends on the curing conditions (as shown by the stress versus strain curves in the upper left side of Fig. 9) and therefore on residual curing stresses level. Between [0°$_2$/90°$_2$]$_s$ laminates cured for 480 min at 160°C and those cured for 40 min at 200°C, the x axis (0° plies orientation) tensile modulus increases by more than 8%. Similarly, the ultimate tensile strength according to x axis grows by 15%. When referring to Table 2 of paper #B107, it can be noticed that physical characteristics (i.e. T_g and degree of cure α) are almost constant whatever the cure cycle is. This is the consequence of the constrained functions used to design the cures cycles. Moreover, Table 3 of paper #B107 shows that for [0°$_2$/90°$_2$]$_s$ laminates, V_f% undergoes a maximum 0.5% change when changing the curing conditions. Applying the rule of mixtures and classical laminated plates theory enables to show that for [0°$_2$/90°$_2$]$_s$ laminates a theoretical increase by 0.5% in V_f% results in a theoretical 0.75% growth in the apparent tensile modulus E_{xx}. This means that the 8% changes in E_{xx} recorded here during the tensile tests, cannot be exclusively ascribed to the small variations in V_f% generated by the different cure cycles. Eliminating the changes in T_g and α which are extremely low and showing that the small variations in V_f% alone cannot generate the 8% changes in E_{xx}, enables to say that these changes are due to the changes in residual curing stresses (see Fig. 4). The same is true for ultimate stresses. In consequence, when correlating Fig. 4 results and tensile test results it appears that both modulus and ultimate tensile strength increase with increasing residual stress level.

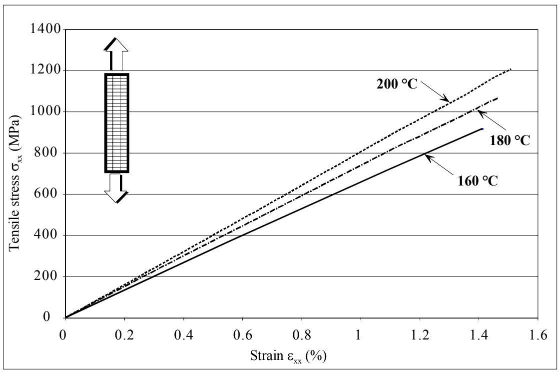
The use of acoustic emission during tensile test on cross-ply laminates enables the first 90° ply failure to be exactly detected. This means that the first ply failure method which is usually used for residual stresses determination in cross-ply laminates [7] can be applied in this study. Effectively, on the one hand we have the acoustic emission activity of [0°$_2$/90°$_2$]$_s$ samples while they are tested and on the other hand Fig. 2 results that establish that the failure of 90° plies is associated to signals starting from 55 up to 80dB. For each one of the tensile test performed on [0°$_2$/90°$_2$]$_s$ laminates, the stress σ_{xx} for the first signal reaching at 55dB has been recorded. The obtained results (average values) are reported in Table 3 together with $\sigma_{yy}^{ult.}$ for [90°$_{16}$] laminates (Table of Fig. 7) and the theoretical values of residual stresses given in Fig. 4.



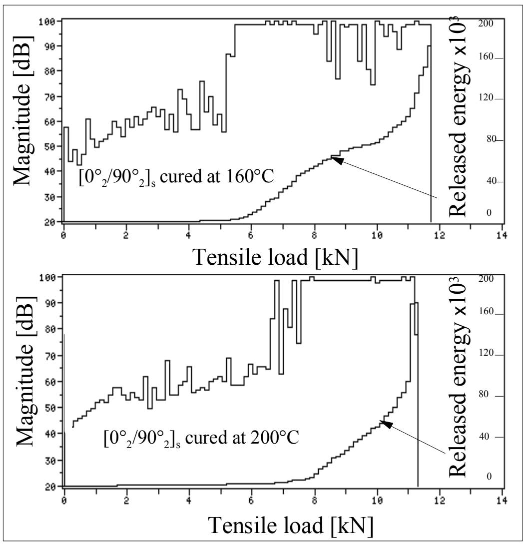
Curing dwell	G_{xy} (MPa)	$\tau_{xy}^{ult.}$ (MPa)
160°C/480min	4920	160
180°C/120min	4810	140
200°C/40min	4770	120



"**Fig. 8**. Results of tensile tests and acoustic emission on $[45°_4]_s$ samples. The table gives moduli and ultimate strengths average values depending on curing dwell temperature and duration."



Curing dwell	E_{yy} (MPa)	$\sigma_{yy}^{ult.}$ (MPa)
160°C/480min	66650	920
180°C/120min	70780	960
200°C/40min	72700	1080



"**Fig. 9** Results of tensile tests and acoustic emission on $[0°_2/90°_2]_s$ samples. The table gives moduli and ultimate strengths average values depending on curing dwell temperature and duration."

"**Table 3.** Application of the first ply failure method to $[0°_2/90°_2]_s$ laminates."

Curing conditions	$\sigma_{yy}^{ult.}$ *(MPa)* *(Table of Fig. 7)*	σ_{xx} *(MPa) for the first signal reaching 55dB average value*	σ_{xx} *residual theoretical (MPa) (Fig. 4)*	σ_{xx} *residual* $(\sigma_{xx}r = \sigma_{yy}^{ult} - \sigma_{xx})$ *(MPa) "first ply failure method"*
480 min at 160°C	60	105	35	45
40 min at 200°C	70	130	48	60

6. CONCLUSION

Without performing any further analysis, this first work enables to show a few elements of the relations between residual curing stresses and some mechanical characteristics of simple stacking sequence laminates. The results synthesised in this paper must obviously be examined in more details. Results concerning UD laminates behaviour must, in particular, be analysed together with the predictions of residual stresses on a micromechanical level.

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