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Carbon fibres reinforced composites. Electrical impedance analysis: a gateway to smartness

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Carbon fibres reinforced composites. Electrical impedance analysis: a gateway to smartness

For more than 7 years now, our research work is focussed upon the electrical behaviour of carbon fibre reinforced composites. Effectively carbon/polymeric matrix laminates can be considered as an electrically conductive network (i.e. carbon fibres) embedded in an insulating medium (i.e. the polymeric matrix). Consequently, from an electrical point of view laminated composites have been modelled owing to combinations of electrical resistances (i.e. fibres themselves and the contact points between the fibres) and capacitances (i.e. polymeric matrix). During their manufacturing, composite material such as thermoset matrix composites for instance, undergo various changes in their physical (including electrical and mechanical) properties and in their geometrical characteristics. In fact, in the case of manufacturing processes such as oven curing (vacuum bag), autoclave curing or heating plate press, beside the chemorheological changes, the laminated parts are the place of a compaction phenomenon inducing changes in fibre volume fraction and thicknesses. The idea developed in this paper is that since the carbon/polymeric matrix laminate can be considered as an electrical network, it can then be viewed and used as a sensor for following the material state during the manufacturing process enabling thus defects to be detected and this without any additional sensing material or device.

Keywords: electrical impedancemetry, cure monitoring, CFRP, functionalization, SHM

Subject classification codes: include these here if the journal requires them

Introduction

In this section, the reader will be first provided (first sub-section) with a brief state of art about the way composite laminates can become smart materials with the aim to following their state during their manufacturing process. The second, third and fourth subsections will remind the results that were obtained upon the measurement of composites electrical impedance during autoclave or oven-curing of carbon/epoxy laminates made from unidirectional prepreg tapes.

A brief state of art: smart composites with embedded sensors for cure monitoring

The topic of cure monitoring was extensively studied since the mid 70's. Composites cure monitoring of thermoset matrix composites was performed according various aims and sensors such as:

- In-situ determination of thermoset matrix degree of cure;
- In-situ measurement of resin viscosity, gelation and vitrification;
- In-situ measurement of the in-plane and/or out-of-plane strains undergone by a laminated part while cured;
- In-situ measurement of the changes in laminate thickness during cure;
- In-situ determination of heat flux;
- In-situ determination of resin flow front in LCM/RTM (liquid composite moulding/resin transfer moulding) processes and measurement of permeability;
- In-situ measurement of thermoset resin pressure during cure.

Whatever the aim of cure monitoring was, a better control of curing conditions to achieve high quality parts (geometrical specifications, initial damages such as voids or resin starvation...) remained one of the main underlying idea as well as improving the knowledge level of the various phenomenon occurring during the manufacturing of a laminated part.

Several experimental methods and sensors were developed, improved and used to address in-situ monitoring. The data provided by the various sensors used in these

methods were frequently real-time recorded and then used to set-up control systems of the manufacturing devices (autoclave temperature and pressure, RTM pressure injection gates ...).

One of the first attempts (1976) to set-up an experimental method to monitor the changes in a composite material during its curing in a heated platen press was performed on a glass and on graphite / epoxy composites owing to a dielectric device [1]. A year later, D.J. Crabtree [2] used “ion graphing” to follow the composite during its curing. That method consisted in aluminium coated polyimide electrodes placed on both sides of laminates to monitor the changes in the D.C. resistivity of the resin throughout its curing. These changes (i.e. in dielectric permittivity ϵ' and dielectric loss factor ϵ'') were related to those occurring in viscosity (or with other physical parameters of interest) with the use of an appropriate modelling effort. The aim was to determine the right time to apply pressure on the laminated part. In 1979, W.W. Houghton et Al. [3] used simultaneously the “ion graphing” method and acoustic emission to real-time determine the extent of cure and to assess the effects of the cooling rate on a laminate.

Among the very first works devoted to use the information given by in-situ sensors (placed on or embedded in laminates) in order to set up a closed-loop control of hot-press or autoclave, one can notice the research made by D. Day [4] with dielectric sensors and the advances settled in the mid-eighties by D.E. Kranbuehl [5] owing electromagnetic sensors with the same purpose. Dielectric property measurements are sensitive to ionic mobility and dipolar relaxation; the obtained electrical properties are DC and AC conductivity, dielectric permittivity and dielectric loss.

Since that time several experimental methods were tested always aiming to monitor the

changes in a composite material during its cure. A lot of work was performed using dielectric or micro-dielectric devices which data, when associated to temperature information, can yield the extent of reaction [6–12]. Some authors focussed their work on ultrasonic methods [13-16] and this since 1952 (it was on unreinforced thermoset resin [13] at that time). Since the degree of cure (α) of thermoset matrices can be associated with their viscosity (η), the ultrasonic velocities and sound attenuation can be linked to the cure state. This latter affects the absorption of the ultrasonic signals and the scattering inside the composite. A few others researchers used pressure sensors [17-18], thickness gauges and thermocouples [17]. Since the eighties, some research teams have proposed to use of infrared transmitting optical fibres embedded in a composite laminate to monitor the cure cycle [19] or optical fibre based matrix refractive index measurement [20].

Manufacturing reliability, reproducibility and implementation of quality assurance are more and more significant factors when considering the whole manufacturing process of composite parts. Since the beginning of the nineties, a well-documented literature shows that huge efforts were performed with the aim to improve past technologies and to build new technological solutions to real-time cure monitoring. The measuring principles studied were:

- Dielectric devices [21-32] and in some cases developed for RTM process [21, 25, 30, 32];
- Ultrasonic methods [33-42], in RTM process [42];
- Measurements of composite electrical resistivity [43-49], in RTM process [47] to detect flow front location and in RTM process with SMARTweave [49].

- Heat of reaction (heat flux) monitoring [50-51];
- Fluorescence spectroscopy based on fibre optic sensors [52-54];
- In-situ FTIR spectroscopy [55-58];
- Pressure sensors [59-63];
- Optical fibres, intensity-based sensors (transmission spectroscopy, evanescent wave spectroscopy, refractive index measurement, microbending signal attenuation) [62-73];
- Optical fibres with Extrinsic Fabry-Perrot Interferometer (EFPI) sensors [51, 65, 74-79];
- Optical fibres with Bragg grating (FBG) [74, 81-94], in filament winding [95];
- Optical fibres with hybrid sensors (FGG + EPFI) [96-98];
- Mixed technologies (FBG+SMARTweave) [99], (ultrasound+optical fibres) [100].

A few words about curing high performances thermoset matrix composites

This paper synthesizes the results that were obtained upon carbon fibres / epoxy matrix composites laminates. The material used in this study is an unidirectional prepreg tape: Hexcel Composites T700/M21. During their curing thermoset matrices under some structural changes [101]. Uncured thermoset matrices usually exhibit a low viscosity. From their initial liquid state, they go through two phase changes: *i* gelation (the whole matrix is a gel) and *ii* vitrification (the whole matrix becomes a vitrified solid near the end of its curing). While the degree of cure (α) of the epoxy matrix progressively

increases from low values up to values close to 100% at end of cure, the resin viscosity (μ) decreases under the effect of molecular chains linearization and then growth quickly under the effect of matrix gelation. As shown in previous sub-section they are several ways to follow (from both theoretical and experimental points of view) those phase changes. In our on-going work using optimized (in frequency) electrical impedance measurements shows that the material exhibits its smart behaviour as it becomes this way a sensor of its own physical state. Therefore, this means that provided the material is correctly interrogated it behaves as a sensor of phase changes [102] as illustrated in Figure 1.

Focus upon electrical measurement methods

They are several ways to follow (from both theoretical and experimental points of view) those phase changes. A large number of references have been given in subsection 1.1. Nevertheless, when focussing upon electrical measurement methods for cure monitoring it appears that some authors measured electrical resistance [103-104], dielectric constants [105-107] or more recently electrical impedance [108-111]. One of the advantage of electrical impedance (Z) -using its modulus $|Z|$ and argument θ measurements- (eq.1, 2 and 3) lays in the fact that both resistance (carbon fibres and contacts between adjacent carbon fibres) and capacitance (thin zone of insulating matrix) are scanned (see Figure 3). Consequently, any phenomenon affecting the reinforcement fibres network (i.e. resistance changes) or the thermoset matrix (i.e. capacitance changes) will be detected.

$$Z = \frac{R}{1+j.2.\pi.f.R.C} \quad (1)$$

$$|Z| = \frac{R}{\sqrt{1+(2.\pi.f.R.C)^2}} \quad (2)$$

$$\theta = -\arctan (2. \pi. f. R. C) \quad (3)$$

where f is the current frequency (Hz), R material resistance (Ω), C its capacitance (F) and j imaginary number.

Impedancemetry for monitoring carbon/epoxy laminates behaviour during their curing

In our current work electrical impedance (Z) measurements were developed because of their extremely high sensitivity to any change in the composite material during its curing. Effectively, as shown in our previous papers [109-111] unidirectional composite laminates behave as an electrical resistance in L direction (according carbon fibres axis) and as the association of a capacitance and a resistance in the two other directions (T for transverse and Z for through-thickness). This is illustrated in Figure 2 where a schematic representation of T700/M21 carbon/epoxy laminates microstructure is given. Prior to curing very light flexible electrodes (i.e. thin Kapton® copper coated strips or copper mesh strips) are placed between the laminate prepreg plies. The layup of prepreg plies is then put under a vacuum bag and autoclave or oven-cured according to the cure cycle shown in Figure 2.

The through-thickness (in Z direction –see Figure 2) absolute value of impedance $|Z_z|$ and its argument θ_z are real-time recorded as a function of current frequency f (Hz) (see eq. 1, 2 and 3) showing thus the capability to detect both the gelation and the vitrification of the epoxy matrix during cure (Figure 3) [111]. The maximum changes in the impedance are obtained for a frequency at 10 kHz. The electrical behaviour is mostly capacitive (frequency dependent) until the middle of the isothermal curing dwell. Beyond this point, the behaviour becomes frequency independent thus mainly resistive. The resistive response corresponds to the lower frequency measurement

(10 Hz). This frequency gives the higher values of the impedance modulus $|Z_z|$ all cure cycle long and a null value of the impedance argument θ_z beyond the point corresponding to the middle of the isothermal dwell.

In order to highlight the effect of the electrical bench, the theoretical changes in the impedance modulus $|Z_z|$ and argument θ_z of an equivalent RC circuit were compared to those of the electrical measurements obtained on samples at room temperature after curing. The theoretical response exhibits a cut-off frequency at 200 kHz. The experimental measurements show not only a divergence between the impedance modulus beyond 10 kHz, but also unexpected changes in the impedance argument θ (a decrease followed by an increase) highlighting the effect of the parasitic access elements beyond 10 kHz. To get the best highlighting of both capacitive and resistive behaviours of the composites during their curing, the optimal frequencies chosen for the study are respectively 10 kHz and 10 Hz.

Manufacturing defects in composite laminated parts

A large number of papers have been published about the manufacturing defects in laminated composite materials and especially upon high performances composites. Manufacturing defects can be classified into three families: *i* defects arising from laminate layup and cure, *ii* defects arising from the composite part machining and assembling phases and lastly *iii* geometry defects [112]. It has been decided to focus our study upon the first family of defects. Those defects can become very detrimental for the integrity of laminated parts and for their mechanical characteristics (moduli, ultimate strengths) [113] (see Figure 4). Manufacturing defects such as voids (usually generated by entrapped air or absorbed water) (Figure 4) and resin rich areas are stemming from curing issues. For instance, one of those issues can be a lack of pressure

allowing voids to grow inside the laminated part. Here the combination of electrical impedance and the composite seen as a sensor of its own state is used to detect some faults during a cure cycle which can give birth to large amounts of defects. This is why this measurement loop will be used (next section) to detect a manufacturing issue.

Detection of a manufacturing issue: vacuum bag leak

During their curing (in an oven or in an autoclave) composite laminated parts are surrounded by various bagging products such as release films (avoiding bonding between part and mould), peel plies (giving laminates a rough surface facilitating further bonding), bleeding plies (for limiting and absorbing resin excess flow), breathing clothes (for uniformising pressure and vacuum distribution onto laminated part surface). Lastly the laminated part and its bagging product are placed under a vacuum bag (nylon film). With the purpose of demonstrating that carbon / epoxy laminates can become smart materials provided they are connected to the right measurement loop, it has been decided to simulate a leak occurring in the vacuum bag during curing. To this end different laminates were cured under identical temperature, vacuum and pressure paths (Figure 5-a). Two cure cycles were performed without creating any manufacturing issues while 6 other cure cycles were performed with manufacturing issues (i.e. vacuum bag leaks). Thus, during the curing leaks were made in the vacuum bags at three different times chosen in consideration of epoxy matrix state: *i* near minimum viscosity, *ii* near gelation and *iii* near vitrification (Figure 5-b).

The measurement loop is similar to the one shown in Figure 2 while the current frequency was set at 10 Hz because the purpose here was not to detect phase changes. In order to show how leak detection is reproducible each cure cycle was done two times. Obtained results are gathered in Figure 6. Vacuum bag leaks occurring near minimum

viscosity or near gelation give birth to consequent changes in $|Z_z|$. In addition, when a leak occurs in these early stages of a cure cycle, $|Z_z|$ value remain always much higher than the ones exhibited by the laminates cured without any manufacturing issue (i.e. reference cure cycles in Figure 6). The case of vacuum leaks occurring near epoxy matrix vitrification requires a finer analysis. Effectively at that stage the matrix is under a gel state (synonymous of a very high viscosity) and begins becoming a solid. Thus, pressure effects on the laminate consecutively to leak apparition are lower.

Nevertheless, as it can be seen in Figure 6 the leaks are detected but the $|Z_z|$ value remains at the same level as the ones recorded during reference cure cycles. Lastly, performing each cure cycle two times enables to show that our measurements are very reproducible.

Concluding remarks

Among all the available techniques, electrical impedancemetry measurements using the material (T700/M21 CFRP here) as a sensor seems to be a very promising method to ensure following the material state and enabling defects to be detected during curing. Indeed, any changes in the resistive or capacitive behaviour of the material can be linked to changes in the percolation network (fibres network) or in the rheological parameters of the matrix due to thermal and pressure effects. For the purpose, we performed a frequency sweep and used both impedance modulus and argument to define the optimum frequencies where electrical parameters exhibit the higher sensitivity to be determined, taking into account effects of the electrical bench. Thus, oven and autoclave cure monitoring and fault detection were possible enabling, in the future, process control adjustment or to sort bad parts out of production. This could lead to effective cost reduction in post-production parts control. Considering CFRP

structures in service, additive functions could be added such as structural health monitoring, heating function to defrost structures or for active non destructive evaluation (NDE) using IR methods for example.

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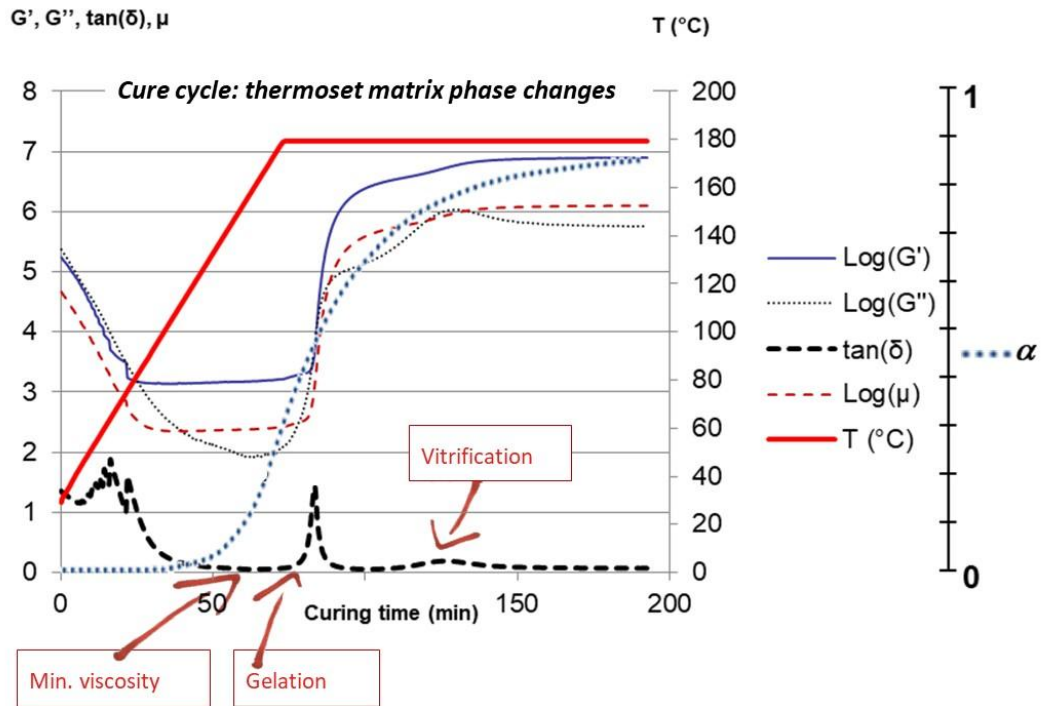


Figure 1. M21 epoxy matrix phase changes during curing followed by rheology and differential scanning calorimetry. Measured parameters: storage (G') and loss (G'') shear moduli, their ratio $\text{Tan}(\delta)$, viscosity (μ) and degree of cure (α).

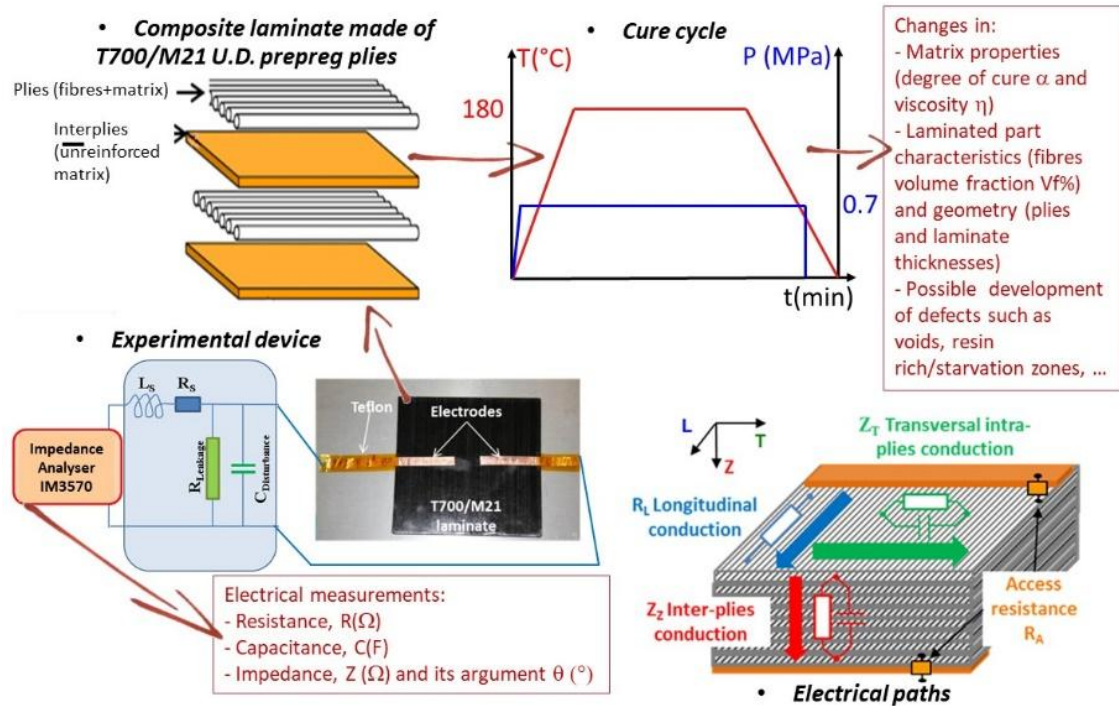


Figure 2. Electrical measurement loop, laminated composites and their electrodes, a typical cure cycle and the equivalent electrical circuits, schematic representation of electrical paths through an unidirectional composite laminate.

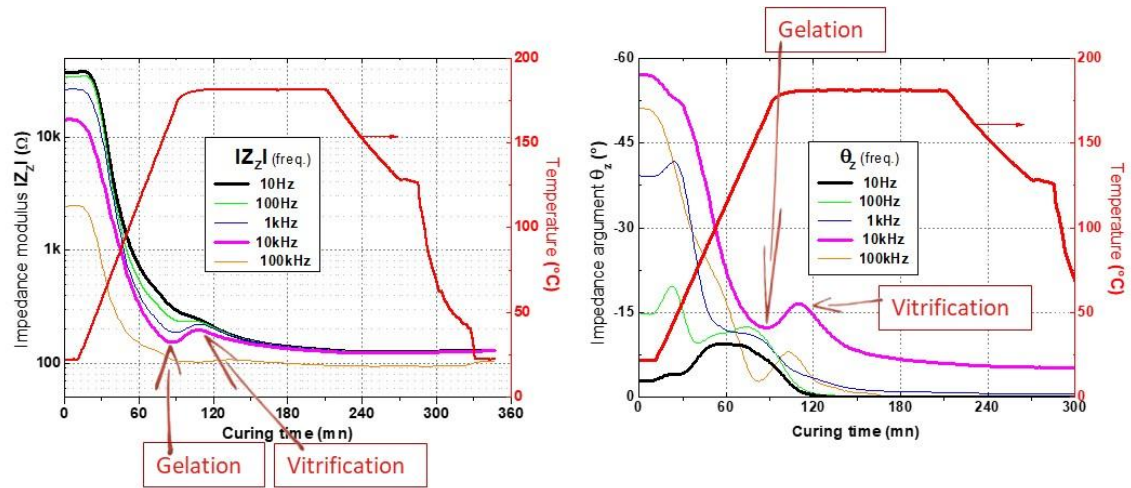


Figure 3. Impedance absolute value $|Z|$ (left) and impedance argument θ_z (right) recorded during the autoclave-curing of a $[0^\circ_8]$ laminate as a function of current frequency.

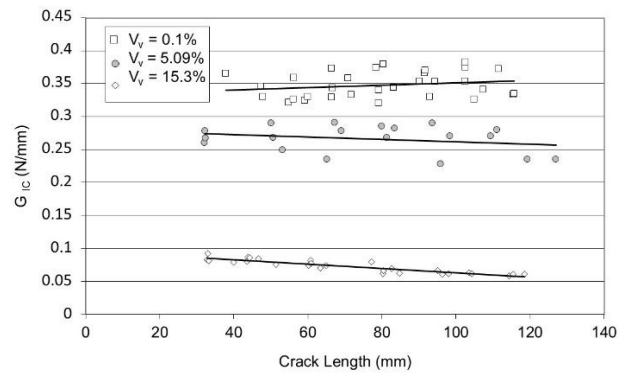
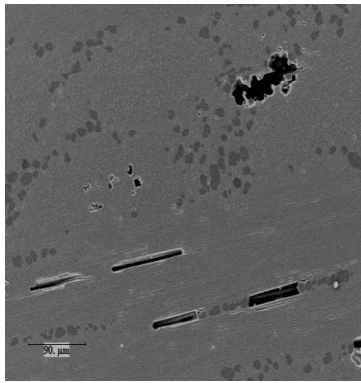
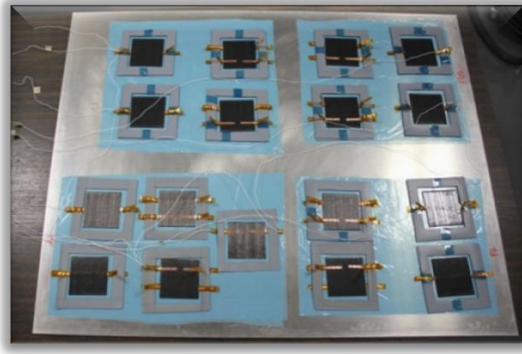
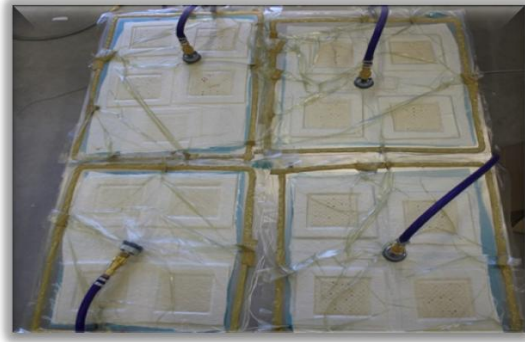


Figure 4. Left: voids developed during the cure cycle inside the plies of a T700/M21 cross-ply laminate. Right: delamination, changes in mode I strain energy release rate G_{IC} on a $(0^\circ/0^\circ)$ interface as a function of laminate void content ($V_v\%$).



a



b

Figure 5. a- instrumented T700/M21 CFRP samples b- curing leak in the vacuum bag at the bottom left

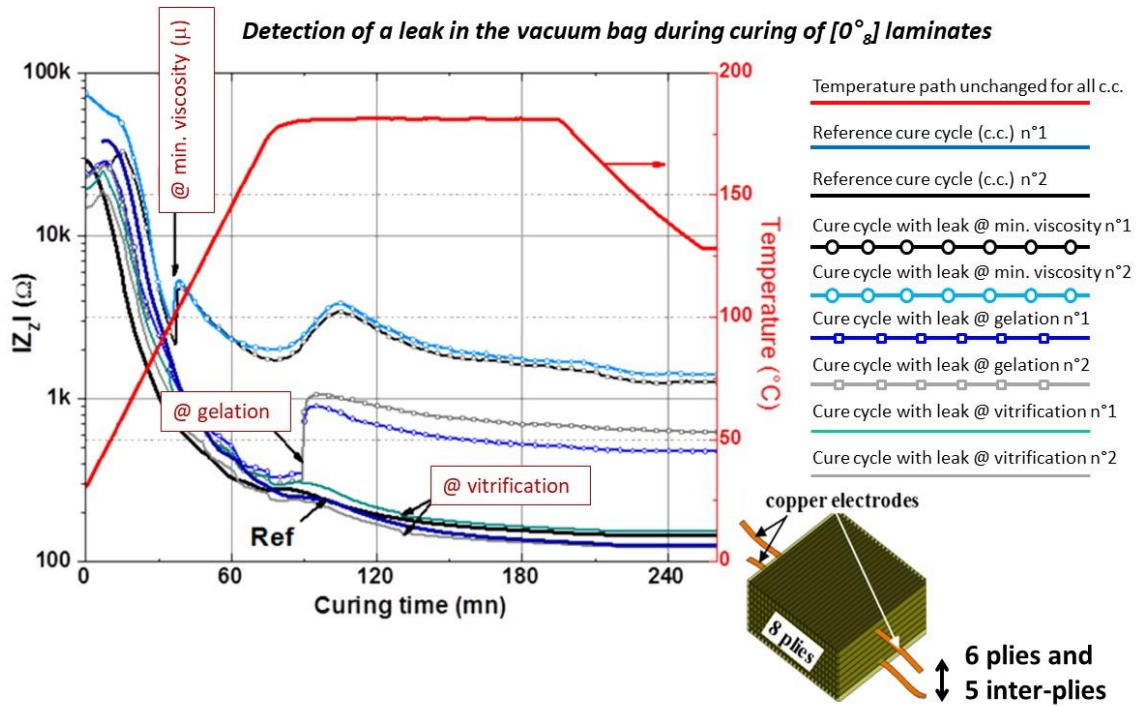


Figure 6. Changes in laminate through-thickness impedance $|Z_z|$ during a cure cycle. Vacuum bag leak detection. Reference cure cycles 1 and 2: no leaks. Other cure cycles leaks were made at specific times: @min. viscosity, @gelation and @vitrification of matrix.