GEOMETRICAL INVESTIGATION FOR THE ELECTRICAL CHARACTERIZATION OF T700/M21 CFRP COMPOSITE DURING CURING

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Abstract
In the aim to use the electrical impedance-metric response of unidirectional CFRP during curing, a very promising approach could be to find the electric current paths in the three orthotropic planes. In such composite structures, current flows mostly along the fibres in the longitudinal direction and via the percolation network in the transverse one. Geometrical parameters should be measured and correlated with each other both at local (plies and inter-plies) and global (laminate) scales taking into account anisotropy and rheological transitions. The experimental approach developed for the purpose allowed us to link the results obtained to the different curing steps. Furthermore, it was shown that all the studied geometrical parameters could be directly deduced from *Vf* and that different laws linking these parameters to the curing time can be deduced (in both plies and inter-plies and for the three orthotropic planes) to serve a future electrical modelling. The intrusive effect of the embedded electrodes in the changes of the geometrical parameters during curing was also studied. The obtained results highlight that they can affect all of them not only because of their own thickness but also of the local induced hindrance of the resin flow.

1. Introduction

Monitoring the manufacturing steps and implementing quality assurance are becoming increasingly significant factors when considering the whole fabrication of composite materials. This optimisation of production should provide reductions in process and material costs, as well as bringing improvements in the properties of the composite structures obtained. In the aim to use the electrical impedance-metric response of carbon/polymeric laminates during curing, a very promising approach could be to find the electric current paths in the three orthotropic planes of composite material. In CFRP laminates, current flows mostly along the fibres in the longitudinal direction and via the percolation network in the transverse one. The higher the fibre volume ratio, V_f, is, the lower will be the electrical resistance values. In the transverse direction, the percolation network is defined by the fibre contact points, *Nc*, and the distance of percolation Δec . Furthermore, the volume ratios of the matrix, V_m, and the porosity (or voids), V_0, influence the inter-fibre capacitive transverse conduction. In the same way, the established percolation-network through the global thickness, t_{glob}, of the laminate produces non-zero electrical conduction in the thicknesses of each ply, t_p, and inter-plies, t_{ip}, (zones between plies). Those parameters (**Figure 1**) should be measured and correlated with each other both at local (plies and inter-plies) and global (laminate) scales taking into account anisotropy and rheological transitions. Thus one of the challenges of our current work is to settle a specific approach enabling the key

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geometrical parameters (t_p, t_{ip}, V_f, V_m, V_0, Nc, Δec) to be determined so that they can be used as inputs to an electrical impedance model of the composite. This latter should predict the electrical response of an U.D. laminate in the three orthotropic planes during curing that will be developed in future works. The experimental approach developed to define the geometrical parameters enabled to link the results obtained to the different curing steps, and also to highlight potential laws that will be used in a future electrical model during curing. As shown in previous works [1-6], embedding thin, soft copper tapes (used as electrodes) between the plies of a laminate (considered here as a sensor) enable the laminate electrical resistance and capacitance to be simply monitored in real time during a cure cycle. Thus, it is relevant to study the intrusive effect of such electrodes in the changes of the geometrical parameters during curing. The obtained results highlight that they can affect all of them (an example is given for the thicknesses in figure 2) not only because of their own thickness but also of the local induced hindrance of the resin flow. The microstructural analyses showed also higher fibres ratios above the electrodes and higher porosities and resin ratios below the electrode embedded in the upper inter-plies. This leads to conclude that the effect of the electrodes has to be taken into account to model the electrical pathway in the studied CFRP. A second type of electrode is under study to minimize these intrusive effects.

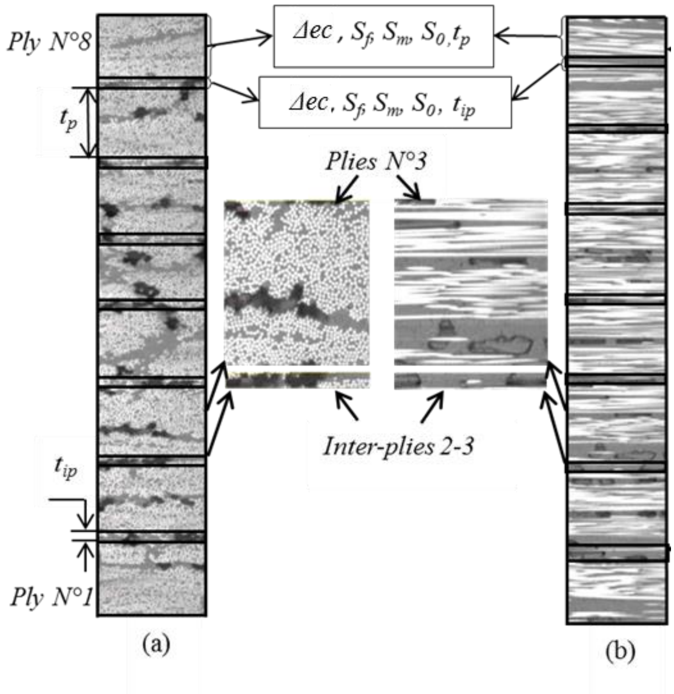

Figure 1. Reconstruction of the thickness in (T, Z) (a) and (L, Z) (b) planes and the across-section electrical equivalent model

2. Material, instrumentations and cure cycle

The specimens used are unidirectional composites made from carbon prepregs/epoxy T700/M21. The thickness chosen for this study is 2 mm (corresponding to 8 plies of prepregs). The plates produced are square (100 mm x 100 mm). Electrodes are inserted between the plies 1-2 and 7-8 perpendicular to the fibre direction. Two types of electrodes (6 mm wide) were used. The first one consists in a 50-microns-thick copper tape and the second one in a 100-microns-thick copper fabric. Once instrumented and draped the material underwent two different cooking cycles. Studied specimens were oven and autoclave cured with the same cure cycle (isothermal: 120 min at 180 °C; and slopes of heating and cooling: 2°C/min).

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3. Experimental approach

Image analyses were performed on « virgin » and instrumented samples to characterize the influence of the electrodes on the changes in of the geometric parameters for the planes (*L*, *Z*) and (*T*, *Z*) (Figure 2). Given the very large number of images to be analysed, we decided to implement automatic image analysis procedures particularly for the determination of surface fractions (fibre, matrix and porosity) and the number of inter-fibres contacts [5].

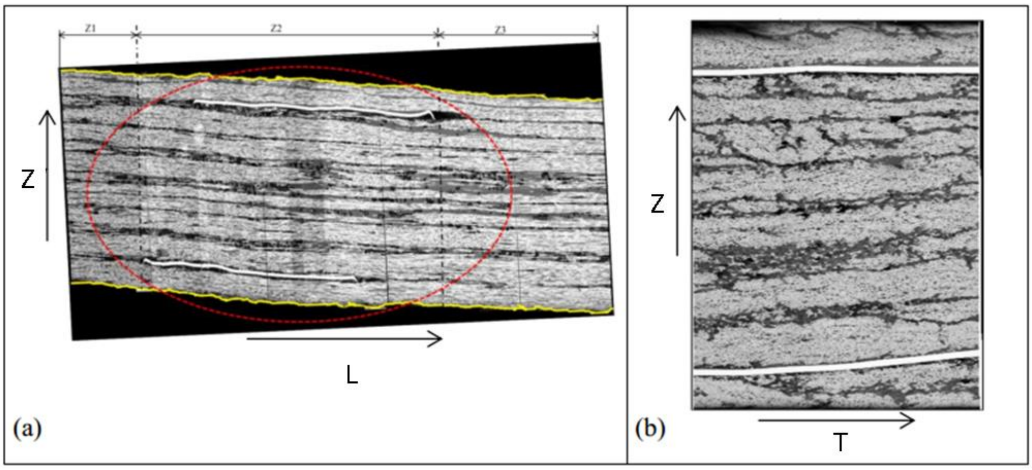

Figure 2. Micrograph of the global thickness of T700/M21 composite material containing electrodes between plies 1-2 and 7-8 (a) changes in the global thickness considering the presence of the embedded electrodes in the heart of T700/M21 composite material (b)

3. Results on geometrical parameters

Table 1 shows the results obtained for the surface ratios of virgin samples using both manual and automatic approaches. Results are in good agreement but the automatic approach exhibits the results correlate well but the automatic approach allows for smaller standard deviations. Results obtained in plane (*T*,*Z*) are in good correlation with volume ratios obtained using chemical dissolution. We therefore hypothesize that the fibre volume fraction, *Vf*, is considered equal to the surface fraction *Sf* obtained in (*T*, *Z*). **Figure 3** shows the averaged changes in surface ratios in plies and inter-plies (for both planes (*L*,*Z*) et (*T*,*Z*)) for uninstrumented and instrumented specimens which were oven and autoclave cured. The obtained results for S_f are always higher (inversely for S_0 and S_m) for autoclave curing regardless the type of instrumentation (pressure effect) and are better (inversely for S_0 and S_m) using the copper fabric electrode. This can be explained by the fact that the fabric enables to drain the flow of resin.

Table 1. Comparison of the surface ratios obtained by manual and automatic approaches of non-instrumented T700/M21 composite material

	(*L*,*T*) plane			**(*L*,*Z*) plane**			**(*T*,*Z*) plane**			**Global values**		
	S_f(%)	S_m(%)	S_0(%)	S_f (%)	S_m(%)	S_0(%)	S_f (%)	S_m(%)	S_0(%)	V_f (%)	V_m (%)	V_0(%)
Manual approach	55.02 ±11.08	39.79 ±4.83	5.1 ±3.47	46.75 ±1.33	45.93 ±1.11	7.3 ±1.91	51.60 ±1.55	40.91 ±2.12	7.50 ±1.14	-	-	-
Automatic approach	57.35 ±5.41	36.23 ±4.64	6.6 ±1.76	46.49 ±1.06	46.65 ±0.88	6.8 ±0.88	52.35 ±0.65	40.42 ±0.66	7.23 ±0.47	-	-	-
Chemical dissolution	-	-	-	-	-	-	-	-	-	51.75 ±3.33	39.06 ±7.97	9.19 ±5.2

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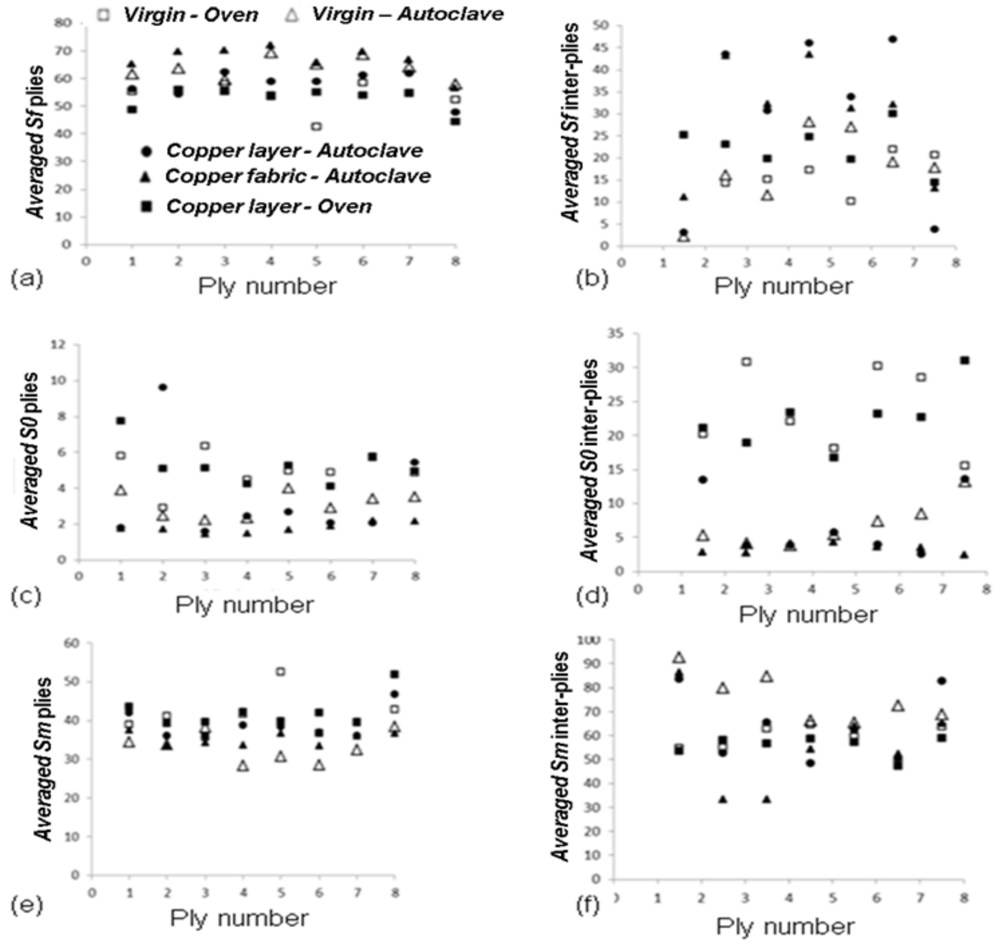

Figure 3. Changes in surface fractions of fibres (a-b); porosity (c-d) and matrix (e-f) following the thickness of the laminate; without instrumentation (virgin) and with instrumentation (copper tape or fabric) for both oven and autoclave curing

The averaged values of *Δec* (**Table 2**) in the plane (*L*,*Z*) were deduced from those of *Nc* and V_f obtained in (*T*,*Z*) within each ply and inter-ply. Results are lower for autoclave curing regardless the instrumentation (pressure effect) and also lower using copper fabric electrodes (better resin flow).

Table 2. Percolation distances (µm) according to the thickness of the plies , inter-plies and laminate

	Autoclave			Oven	
Averaged *Δec*	**Copper fabric**	**Copper tape**	**Uninstrumented**	**Copper fabric**	**Copper tape**
Plies	41.24	52.05	31.25	64.30	38.32
Inter-plies	73.40	144.36	76.86	163.8	151.95
Laminate	56.25	95.13	54.05	110.75	95.14

Table 3 presents the averaged values of global and local thicknesses for planes (*L*,*Z*) and (*T*,*Z*) . As expected, thicknesses obtained in autoclave are always lower than those obtained in oven (pressure effect). Instrumented specimens are thicker than virgin ones because of the size of electrodes (50 µm for copper tape and 100 µm for copper fabric). Thicknesses of specimens instrumented with copper fabrics are smaller than those instrumented with copper tapes (no resin flow between copper-tape electrodes).

Figure 4 shows the structural distribution of fibres, porosities and matrix surrounding the electrodes placed in inter-plies 1-2 and 7-8. These micrographs highlight that copper fabric electrodes enable

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resin flow contrary to copper tape electrodes leading not only to better fibre, porosity and matrix ratios but also better plies/electrodes contacts.

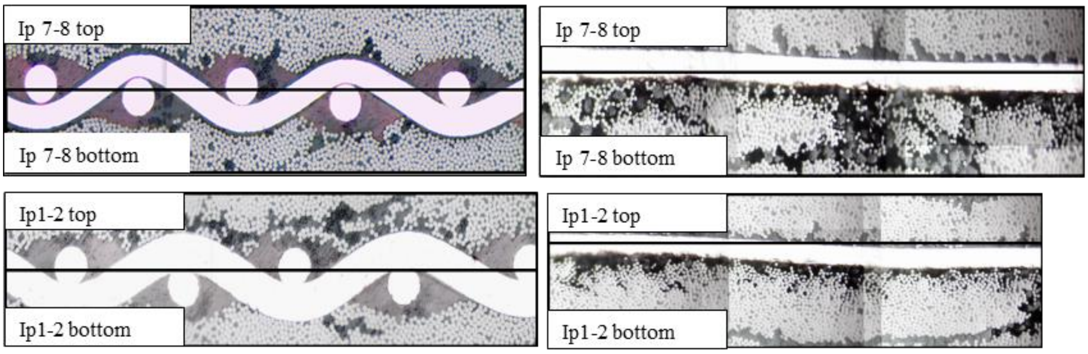

Figure 4. Fibres, porosities and matrix distribution

Table 2. Comparison of thicknesses (mm) in the plane (*L*,*Z*) and (*T*,*Z*)

	$t_{laminate}$		t_{plies}		$t_{inter\text{-}plies}$	
	(*L*,*Z*)	(*T*,*Z*)	(*L*,*Z*)	(*T*,*Z*)	(*L*,*Z*)	(*T*,*Z*)
Virgin - Oven	2.783	-	0.307	-	0.046	-
Virgin - Autoclave	2.282	-	0.260	-	0.029	-
Copper tape - Oven	2.860	2.740	0.307	0.31	0.058	0.043
Copper tape - Autoclave	-	3.357	-	0.2605	-	0.035
Copper fabric - Autoclave	2.410	2.399	0.256	0.252	-	0.054

3. Electrical measurements during curing

The feasibility to monitor the curing of unidirectional CFRP using electrical impedancemetry was highlighted in previous works [1-6]. To monitor the cure cycle, through-the-thickness electrical measurements were undertaken using two-point measurement (minimum value of the impedance modulus about 100Ω, much higher than the parasitic access resistance (R_S~5 Ω)). A frequency sweep of sinusoidal current ranging from 10Hz to 5MHz enables to obtain the spectral evolution of the module ($|\underline{Z}|$) and the phase (θ) of the electrical impedance $\underline{Z}$. The resistance R_{pz} is deduced at low frequency and the capacity C_{pz} at high frequency (**Figure 5**). Obtained results show the same trend of the electrical response for oven and autoclave curing. However R_{pz} remains lower during autoclave curing (pressure effect) and C_{pz} stay smaller during isothermal for autoclave curing. At the end of the curing, autoclave R_{pz} (45.7 Ω) is 2.75 times smaller than oven R_{pz} (126 Ω) and autoclave reaches 7.3 nF against 8,8 nF for oven C_{pz}. We deduce an oven inter-ply resistivity of 8400 Ω.m and 3050 Ω.m in autoclave. C_{pz} should have remained higher during autoclave curing. This could be linked to the effect of feed wires. Indeed feed wires are ten meters long for the autoclave against 3 meters for the oven leading to higher parasitic element for autoclave electrical measurement (330pF for autoclave and 40pF oven). Adding the fact that autoclave R_{pz} is almost three times lower, this explains the underestimated value of C_{pz}. Post-curing electrical measurement with short feed wires exhibit a C_{pz} value reaching 15 nF for autoclave cured specimens. As shown in **Figure 6** electrical measurements exhibit lower resistance values using copper fabric electrodes for autoclave cure monitoring. These results are in good agreement with microstructural observations.

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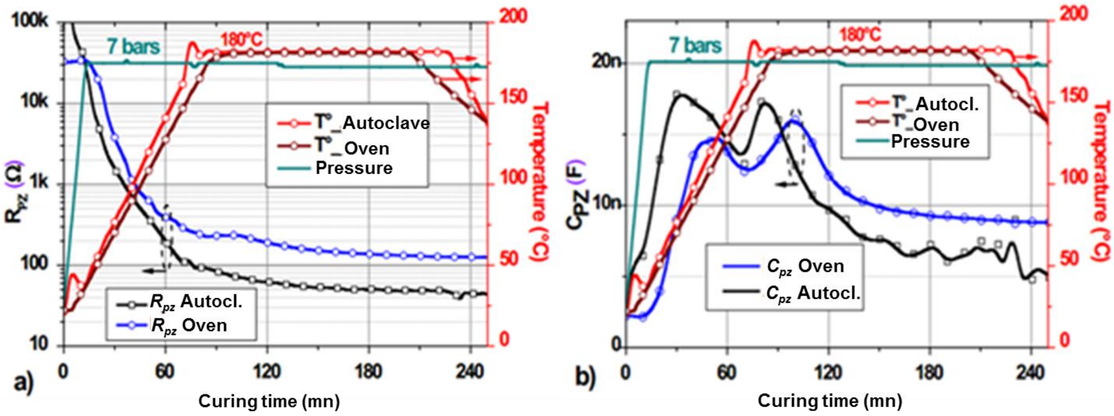

Figure 5. Evolutions of R_{pz} (a) and C_{pz} (b) during autoclave and oven curing

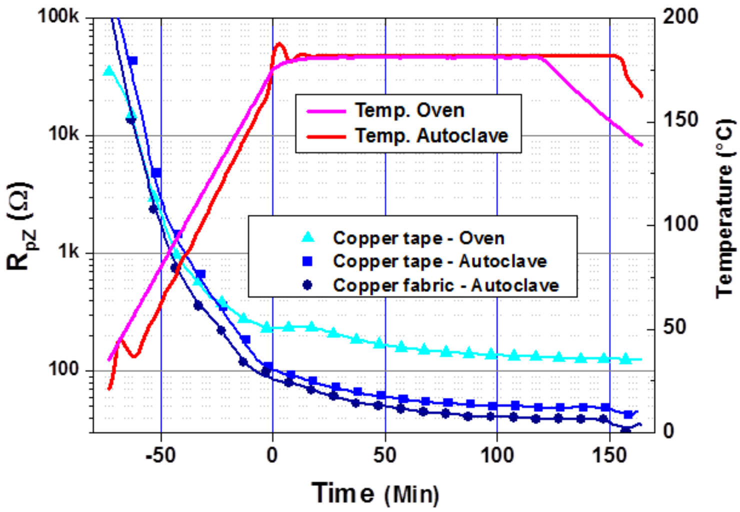

Figure 6. Changes in R_{pz} during autoclave and oven curing using copper -tape and –fabric electrodes

4. Conclusion

In this study microstructural analyses were undertaken to determine the set of geometrical parameters necessary for the understanding of the electrical conduction within unidirectional T700/M21 CFRP taking into account the manufacture process and type of electrode. Two types of electrodes were compared: copper tape and copper fabric. Results demonstrates that pressure (autoclave curing compared to oven curing) improves the quality of inter-fibre contacts and fibre/electrodes contacts: better fibre, porosity and matrix ratios and better percolation parameters. Furthermore the use of copper fabric electrodes allows resin flow contrary to copper tape leading to better geometrical parameters and better plies/electrodes contacts. Electrical measurements undertaken to monitor curing confirm those results. In this context, the copper fabric electrodes are good candidates because they are less intrusive from a point of view of the intrinsic composite structure and enable more accurate electrical measurements.

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