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RELATIONSHIPS BETWEEN PROCESS PARAMETERS AND MECHANICAL PROPERTIES OF LAMINATED PLATES MADE BY L.R.I

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ABSTRACT: Liquid Resin Infusion (LRI) process is a composite manufacturing process where resin infusion occurs under vacuum. This process includes parameters that could cause variation in the intrinsic properties of composites. The aim of this study is to evaluate relationships between process parameters and mechanical properties of quadriaxial carbon Non-Crimp-Fabric (NCF) and epoxy RTM6 composites, using an experimental plan and statistical analysis. Results showed effects of composite plate sides on Ultimate Tensile Strength (UTS), Ultimate Compressive Strength (UCS), fibers volume fraction (V_f), void content (V_p) and structural thickness (h). UTS, UCS and V_f of composite plates on resin injection side are higher than those of laminated plates on the vacuum side, while the opposite effect is found for V_p and h. In addition of number of High Porous Media (HPM) used and vacuum level achieved during resin infusion process, the morphological properties of infused laminates (V_f, V_p, h) strongly depend on process temperatures. The effects of process parameters on Interlaminar Shear Strength (ILSS) and glass transition temperature (T_g) were also investigated. ILSS is at its higher value when laminates were manufactured using the higher values of number of NCF layers and number of HPM layers, analyzed in unique manner and by the interaction between these two parameters. T_g decreases with increasing of the number of NCF layers and it is the opposite effect with curing temperature. The microscopic analysis was used to explain statistical results found on V_p and its impact on mechanical properties of infused laminates. Higher mould temperatures and / or higher curing temperatures have an adverse effect on quality of infused composite structures.

KEYWORDS : Liquid Resin Infusion, quadri-axial carbon reinforcement, process parameters, experimental plan, properties of laminated Non-Crimp-Fabric and RTM 6, statistical analysis, microscopic analysis.

INTRODUCTION

Liquid Resin Infusion (LRI) is a Vacuum Infusion Process (VIP). Resin infuses through the thickness of preform reinforcement under vacuum. LRI is a low cost composite manufacturing process. It does not require additional pressure to compact the perform and inject the resin. The resin curing occurs outside autoclave. This process uses a vacuum bag as upper mould and High Porous Media (HPM) for distribution. The advantage of LRI is relatively low cost tooling process to manufacture large composite structures for high performance applications. HPM has a higher permeability than perform reinforcement. It allows resin to flow on the surface followed by through thickness perform flowing. The resin flow results from vacuum and capillary effects. The flow speed of resin is affected first by the vacuum level, which is the pressure gradient achieved in the vacuum cavity. This resin speed is also involved by the number of HPM layers and the number of fabric reinforcement layers used. In addition to the preform *reinforcement texture* on which depend the mechanical properties of composite structures, the *process configuration* and the *process temperature* create a variation of these properties. In previous studies, the process parameters were identified using a literature review and an experimental plan [1].

The aim of this study is to know relationship that exists between process parameters and properties of carbon Non-Crimp-Fabric (NCF) - epoxy RTM6 laminates manufactured by LRI. The effects of number of carbon NCF layers, number of HPM layers, mould temperature, injection temperature, curing temperature, and the composite plate sides during their manufacturing, will be analyzed. To achieve this objective, an experimental plan allowed to manufacture laminates under different conditions, according to values of process parameters. This work is carried out to understand and to determine the process parameters that have significant influence on the variation of Ultimate Tensile Strength (UTS), Ultimate Compressive Strength (UCS) and Interlaminar Shear Strength (ILSS) of laminates. The relationships between process parameters and fibers volume fraction and void content of laminates will be analyzed statistically. The quantification of the parameter effects on the quality of composites will be done for better control of manufacturing and for minimizing the variation in composite properties.

EXPERIMENTAL WORK

Materials

Two complementary fabrics of four different unidirectional layers with the same properties (areal weight: 1088 g/m^2), the quadriaxial carbon NCF, Tenax IMS 60 E13 24K 830 Tex, constitute the preform reinforcements. The choice of these two different stack-fabrics was made for the laying-up, to respect mirror symmetry, according to the orientation of reinforcements inside composites. They are commercial grade and supplied by SAERTEX. These fabrics are designed primarily for applications in the better toughness and ultimate strength.

The resin is a premixed epoxy system, a monocomponent thermoset resin HexFlow® RTM 6. RTM 6 is a resin for service temperatures from -60°C up to 180°C (-76°F up to 356°F).

Process Parameters

Research and experimental approach have been done to identify the key process parameters which have a greater impact on the performance and the quality of infused composite structures [1, 2]. These parameters were classified and grouped into three sets.

The *texture reinforcement* concerns the preform reinforcement. It refers to the *number of carbon NCF layers*. It would has influence on the quality and mechanical properties of laminates, both on the structure thickness, during the manufacturing process through flow of resin and during resin curing. Laminate plates were manufactured and tested with 2 or 4 carbon NCF layers. The final laminate thicknesses were respectively approximately 2 mm and 4 mm.

The second set is the *process configuration*. It constitutes *vacuum level* achieved in the vacuum cavity during the resin injection process, *number of HPM layers* used, which represents a characteristic parameter for the flow of resin during the structure manufacturing by LRI, and laminate *plate sides* on the mould during infusion process, "*vacuum side or injection side*". The vacuum level represents the absolute pressure in the vacuum cavity. It cannot be imposed and varies according to the assembly. The "vacuum level" was found as a crucial parameter for obtaining a better quality of composite materials [3-5]. Values of vacuum level shown on experimental plan (Table 1) are those obtained during the manufacture of laminates according to each configuration. Furthermore, research prove that the use of HPM or the number of its layers in the configuration could influence the properties of the composite in a unique way, or by interaction with the number of layers of fiber reinforcement [6, 7]. The recent identification of this parameter as an impact factor and its integration into analysis would enable better control of its effects on the properties variation. To manufacture infused composite structures, 1 or 2 HPM with 4×10^{-10} m^2 of permeability were used. Besides, the location of the structure relative to resin inlet is also a factor of variability of composites properties. Indeed, the velocity field of resin flow in the mould depends on where is the structural preform compared to the number of inlet and outlet resin. The variation of the field might cause the formation of type, shape or size of voids in the composites [4, 8, 9]. It has an influence on the integrity of composite structure. There could be effects on the fibers volume fraction and void content as well as on its mechanical properties including in tensile, in compressive and in interlaminar shear strengths. The plate side of laminate, "vacuum side or injection side" is used in this study to quantify influence of the position of specimens during manufacturing on mechanical properties of the structure.

The temperatures used during manufacturing process could generate also variability of morphological and mechanical properties of final composite structures, both by their influence on the viscosity of the resin and its flow velocity through the perform and during the resin curing. The *process temperatures* are the 3rd set. It concerns *mould temperature, injection temperature and curing temperature.* These temperatures vary between two values: the lower and the higher values. The use of different values of temperatures helps to discern their influence.

Experimental plan

Eight process parameters were found for designing and for carrying out an experimental plan. The table used (Table 1) to manufacture infused composites with different process conditions is type of Taguchi $L_{16}(2^{15})$ table. It is an orthogonal table which allows interaction between parameters. This table includes 8 columns of factors which are process parameters found before and classified in the table as follows: *number of NCF carbon* (N_{NCF})*, injection temperature* (T_I)*, interaction between number of NCF and HPM layers* ($Int_{NCF,HPM}$)*, mould temperature* (T_M)*, number of HPM layers* (N_{HPM})*, curing temperature* (T_C)*, plate sides* : *vacuum side or injection side* (P_S) *and vacuum level* (V_L)*.* There are sixteen principal lines and two verification lines which represent these different process conditions. Each factor has two levels of variation except *vacuum level* which the values shown is those obtained during the infusion process. This plan has allowed to manufacture infused carbon NCF - epoxy RTM 6 lanimates under several experimental conditions.

Manufacturing of Infused Laminates

Manufacturing of infused composite structures is made in the same manner as that described in the reference [1]. For each experiment, liquid resin is systematically heated in a resin pot and degassed under vacuum during 10 min. This operation occurs before resin injection in injection tube followed by its infusion into the preform at injection temperature (60°C or 80°C) in the vacuum cavity.

Infusion process continues until resin exit port. The same vacuum level is maintained till end of manufacturing. Manufacturing of infused composites finished by resin curing at curing cycle followed by cooling at ambient temperature (around 20°C). Carbon NCF – epoxy RTM6 infused laminate plates of 400 x 150 mm were manufactured according the process conditions of Table 1.

Table 1**:** Experimental plan

Process conditions		Process parameters							
N°	Exp.	Number of NCF layers	Injection temperature (°C)	Interaction N.C.F - HPM layers	Mould temperature (°C)	Number of HPM layers	Curing temperature (°C)	Plate side	Vacuum level (mbar)
1	1V	2	60	1	120	2	160	Vacuum	1.1
2	1I	2	60	1	120	2	160	Injection	1.1
3	2V	2	60	2	100	1	180	Vacuum	1.6
4	2I	2	60	2	100	1	180	Injection	1.6
5	3V	4	80	1	120	1	180	Vacuum	1
6	3I	4	80	1	120	1	180	Injection	1
7	4V	4	80	2	100	2	160	Vacuum	1.4
8	4I	4	80	2	100	2	160	Injection	1.4
9	5V	2	80	1	100	2	180	Vacuum	1.3
10	5I	2	80	1	100	2	180	Injection	1.3
11	6V	2	80	2	120	1	160	Vacuum	1.1
12	6I	2	80	2	120	1	160	Injection	1.1
13	7V	4	60	1	100	1	160	Vacuum	1.4
14	7I	4	60	1	100	1	160	Injection	1.4
15	8V	4	60	2	120	2	180	Vacuum	2.4
16	8I	4	60	2	120	2	180	Injection	2.4
17	9V	4	70	2	110	2	170	Vacuum	1.7
18	9I	4	70	2	110	2	170	Injection	1.7

Testing of Properties

Specimens were cut from each laminate plate manufactured and tested mechanically and morphologically. To determine UTS, UCS, fibers volume fraction (V_f) and void content (V_p) of carbon NCF – epoxy RTM 6 infused composites, three specimens were cut for each property. Four specimens were characterized on DMA to obtain their T_g and six to obtain ILSS. Thickness measurements of specimens in tension, compression, V_f and V_p allowed to do thicknesses analysis of laminate plates according to experimental plan. Table 2 shows the standards of characterization used, the averages mechanical and morphological properties responses and deviations of each experiment.

RESULTS AND DISCUSSION

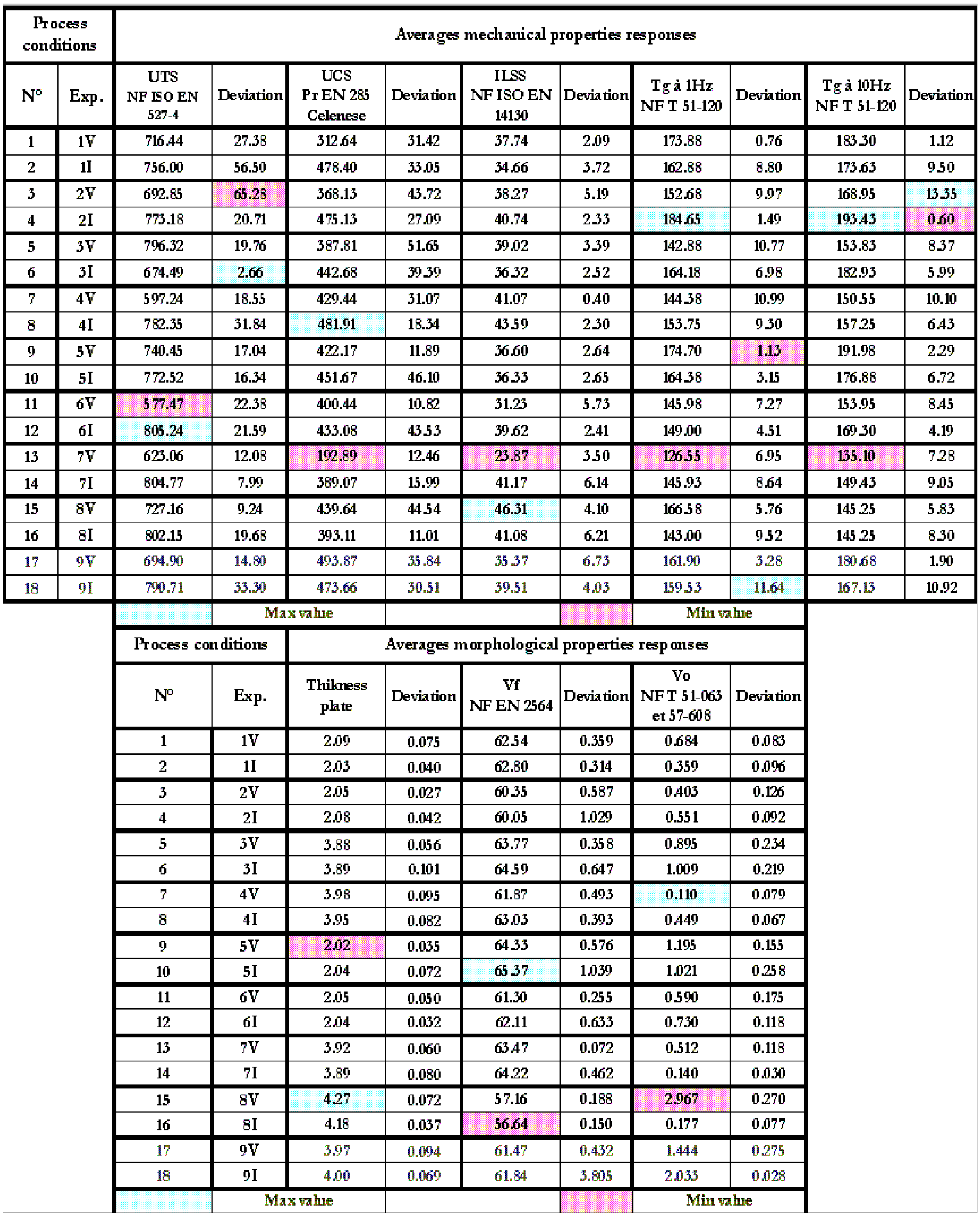
Statistical Analysis

Statistical analysis consists of determine the coefficients of model response by multilinear regression and probability of nullity of these coefficients. The general model can be written as a linear expression of response variable Y function of normalized parameters (eqn 1).

$$\boxed{Y = C + a_{NCF} N_{NCF} + a_{T_I} T_I + a_{Int} Int_{NCF,HPM} + a_{T_M} T_M + a_{HMP} N_{HPM} + a_{T_C} T_C + a_{P_S} P_S + a_{V_L} V_L}$$

The parameter is considered to have significant (S) effects on the result if the probability of nullity of its coefficient is less or equal to 5% ($\alpha \leq 5\%$). When in the analysis of variance, probability is less than 1% ($\alpha < 1\%$), parameter is considered to be very significant (VS) on result. It has low significance (LS) effect when α is between 5% and 10% and not significant (NS) effect in the other cases.

Table 2**:** Outline of average results



Process conditions		Averages mechanical properties responses									
N°	Exp.	UTS NF ISO EN 527-4	Deviation	UCS Pr EN 285 Celenese	Deviation	ILSS NF ISO EN 14130	Deviation	Tg à 1Hz NF T 51-120	Deviation	Tg à 10Hz NF T 51-120	Deviation
1	1V	716.44	27.38	312.64	31.42	37.74	2.09	173.88	0.76	183.30	1.12
2	1I	756.00	56.50	478.40	33.05	34.66	3.72	162.88	8.80	173.63	9.50
3	2V	692.85	65.28	368.13	43.72	38.27	5.19	152.68	9.97	168.95	13.35
4	2I	773.18	20.71	475.13	27.09	40.74	2.33	184.65	1.49	193.43	0.60
5	3V	796.32	19.76	387.81	51.65	39.02	3.39	142.88	10.77	153.83	8.37
6	3I	674.49	2.66	442.68	39.39	36.32	2.52	164.18	6.98	182.93	5.99
7	4V	597.24	18.55	429.44	31.07	41.07	0.40	144.38	10.99	150.55	10.10
8	4I	782.35	31.84	481.91	18.34	43.59	2.30	153.75	9.30	157.25	6.43
9	5V	740.45	17.04	422.17	11.89	36.60	2.64	174.70	1.13	191.98	2.29
10	5I	772.52	16.34	451.67	46.10	36.33	2.65	164.38	3.15	176.88	6.72
11	6V	577.47	22.38	400.44	10.82	31.23	5.73	145.98	7.27	153.95	8.45
12	6I	805.24	21.59	433.08	43.53	39.62	2.41	149.00	4.51	169.30	4.19
13	7V	623.06	12.08	192.89	12.46	23.87	3.50	126.55	6.95	135.10	7.28
14	7I	804.77	7.99	389.07	15.99	41.17	6.14	145.93	8.64	149.43	9.05
15	8V	727.16	9.24	439.64	44.54	46.31	4.10	166.58	5.76	145.25	5.83
16	8I	802.15	19.68	393.11	11.01	41.08	6.21	143.00	9.52	145.25	8.30
17	9V	694.90	14.80	493.87	35.84	35.37	6.73	161.90	3.28	180.68	1.90
18	9I	790.71	33.30	473.66	30.51	39.51	4.03	159.53	11.64	167.13	10.92
			Max value					Min value			

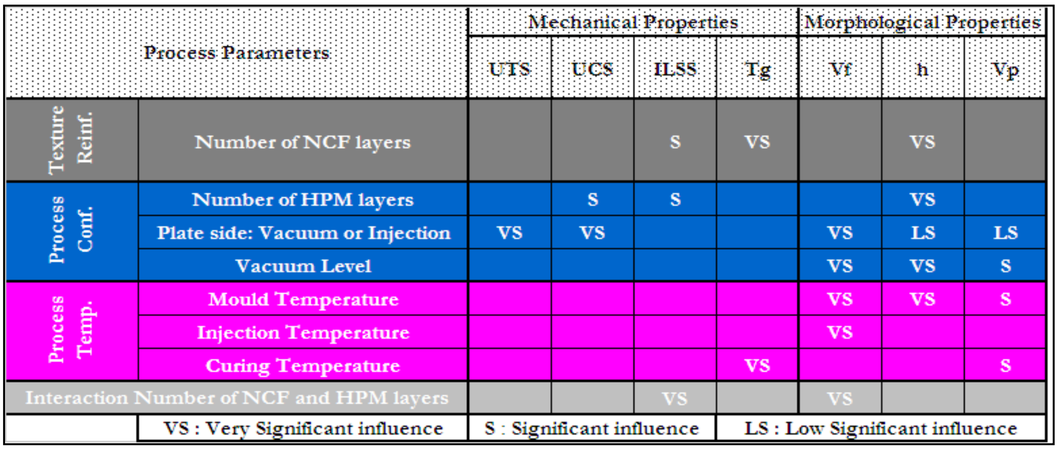
Process conditions		Averages morphological properties responses					
N°	Exp.	Thikness plate	Deviation	Vf NF EN 2564	Deviation	Vo NF T 51-063 et 57-608	Deviation
1	1V	2.09	0.075	62.54	0.359	0.684	0.083
2	1I	2.03	0.040	62.80	0.314	0.359	0.096
3	2V	2.05	0.027	60.35	0.587	0.403	0.126
4	2I	2.08	0.042	60.05	1.029	0.551	0.092
5	3V	3.88	0.056	63.77	0.358	0.895	0.234
6	3I	3.89	0.101	64.59	0.647	1.009	0.219
7	4V	3.98	0.095	61.87	0.493	0.110	0.079
8	4I	3.95	0.082	63.03	0.393	0.449	0.067
9	5V	2.02	0.035	64.33	0.576	1.195	0.155
10	5I	2.04	0.072	65.37	1.039	1.021	0.258
11	6V	2.05	0.050	61.30	0.255	0.590	0.175
12	6I	2.04	0.032	62.11	0.633	0.730	0.118
13	7V	3.92	0.060	63.47	0.072	0.512	0.118
14	7I	3.89	0.080	64.22	0.462	0.140	0.030
15	8V	4.27	0.072	57.16	0.188	2.967	0.270
16	8I	4.18	0.037	56.64	0.150	0.177	0.077
17	9V	3.97	0.094	61.47	0.432	1.444	0.275
18	9I	4.00	0.069	61.84	3.805	2.033	0.028
	Max value					Min value	

The general model is reduced to parameters whose effects are statistically significant. It is a model with only significant parameters (VS, S, or LS). Table 3 below summarizes the statistical analysis results of characterisations found. It specifies for each process parameter, significance effect on a desired property.

Effects of Process Parameters on Property Responses

The results of statistical analysis allowed to identify the best models associated with each of properties of table 3. Equations 2 to 8 illustrate linear models for each property analyzed.

Table 3: Summary of statistical results



Process Parameters		Mechanical Properties				Morphological Properties		
		UTS	UCS	ILSS	Tg	Vf	h	Vp
Texture Reinf.	Number of NCF layers			S	VS		VS	
Process Conf.	Number of HPM layers		S	S			VS	
	Plate side: Vacuum or Injection	VS	VS			VS	LS	LS
	Vacuum Level					VS	VS	S
Process Temp.	Mould Temperature					VS	VS	S
	Injection Temperature					VS		
	Curing Temperature				VS			S
Interaction Number of NCF and HPM layers				VS		VS		
	VS : Very Significant influence	S : Significant influence		LS : Low Significant influence				

Statistical Results of Mechanical Properties Analysis

$$UTS = 735.93 + 36.07\,P_S \qquad (2) \qquad UCS = 416.47 + 17.54\,N_{HPM} + 26.66\,P_S \qquad (3)$$

$$ILSS = 38.38 + 1.20\,N_{NCF} + 2.15\,Int_{NCF,HPM} + 1.31\,N_{HPM} \qquad (4)$$

$$T_g(1Hz) = 155.77 - 9.45N_{NCF} + 7.21T_C \text{ et } T_g(10Hz) = 166.72 - 9.71N_{NCF} + 7.66T_C \qquad (5)$$

Statistical Results of Morphological Properties Analysis

$$V_f = 61.28 - 1.18\,Int_{NCF,HPM} + 0.59T_I - 0.77T_M + 0.25\,P_S - 2.01V_L \qquad (6)$$

$$V_P = 0.96 + 0.18T_M + 0.20T_C - 0.16\,P_S + 0.34V_L \qquad (7)$$

$$h = 3.08 + 0.94\,N_{NCF} + 0.03T_M + 0.02\,N_{HPM} - 0.01\,P_S + 0.15V_L \qquad (8)$$

Effects of Texture Reinforcement

The number of NCF layers increases ILSS and structural thickness, and decreases Tg (eqn 4, 5 and 8). The variability of properties occurs when this influence parameter goes from 2 to 4 NCF layers during manufacturing process. Thus, ILSS is better when using 4 NCF and Tg when using 2 NCF. The composite structures are thicker with 4 NCF. Equation 8 predicts a thickness of 4.02 mm for 4 NCF with codification (+1) and 2.14 mm for 2 NCF with codification (-1) in analysis.

Effects of Process Configuration

Process configuration has significant influence on mechanical and morphological properties. In fact, the use of 2 layers of HPM instead of 1 creates an increase of UCS, ILSS and the thickness of the structure. The plate sides of structure is found to be have a VS effect on UTS, UCS, V_f, and a LS effect on V_p and thickness variation (table 3). It decreases V_p and thickness, and increases others properties listed before. This creates variation of 9.80% of UTS and 12.80% of UCS (eqn 2 and 3) of average of plan of experiments. The prediction of analysis is to obtain UTS about equal to 700 MPa for a composite vacuum side which has a codification (-1), and about 772 MPa for a composite injection side with a codification (+1).

In other cases, when good vacuum level is obtained during manufacturing process (less than 1.5 mbar), the morphological properties are better. A good vacuum level produces an increase of 6.55% of average of V_f in the plan of experiments. Laminated plates on the injection side have V_p less than those on the vacuum. In addition of this, a good vacuum level achieved allows to obtain better quality of composite structures, referring to their void content less than 1% and their fibers volume fraction more than 62%.

The planar microscopy analysis through the thickness section views for composites manufactured explain statistical results. Three void locations were defined [10]: "matrix voids", void inside areas rich in matrix and not comprising any fibers, "preform voids" where void is inside areas rich in fibers, the area is primarily composed of reinforcing preform (i.e. intra - tow voids) and finally, "transition voids", voids are always positioned adjacent to the preform but not inside fiber tows. Fig. 1 shows that voids formed on the injection side plates are barely visible. They are perform voids and are small sizes ($\phi \approx 4\ \mu m$). For vacuum side plates, formation of voids is usually doing in resin pockets (areas rich in matrix) and some in transition location. These voids are large sizes ($\phi > 100\ \mu m$) and in irregular shapes.

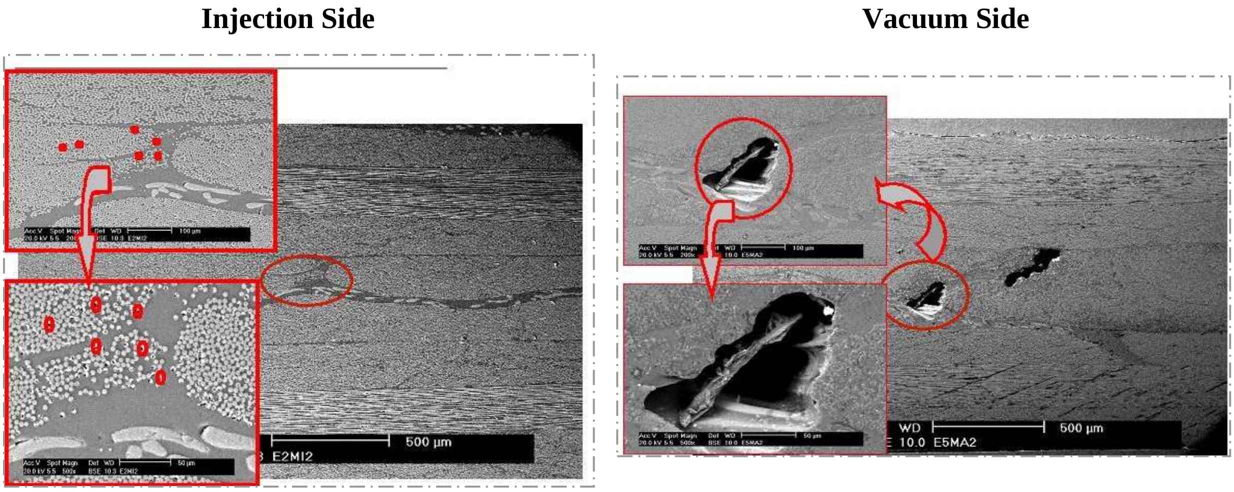

Injection Side **Vacuum Side**

Fig. 1 : Microscopic analysis of void formation inside plate

Results (observations and analysis) [10-13] prove that void formation depends strongly on flow velocity of the resin during the manufacturing process of the composite. This effect generates a significant impact on mechanical strength of composite. Voids located only in the matrix reduce the load bearing cross-section of the composite, while those located in contact with fibers are also detrimental to fiber/matrix adhesion. ILSS of infused laminates is at its higher value when they were manufactured using 4 NCF layers and 2 HPM layers. These remarks also explain the results found in term of reduction of UTS and UCS between injection side and vacuum side plate (about 10% in tension and 13% in compression). These results are comparable for those done by Liu [14]. The void content has adverse effects on ultimate mechanical properties of composites regardless of their location (matrix, preform or transition voids).

Effects of Process Temperatures

Variation of process temperatures during composite manufacturing have VS effect on T_g, V_f and structure thickness, S effect on V_p of infused laminates (table 3). V_f is better when they were manufactured with higher injection temperature (80°C instead to 60°C). But, when manufacturing was made with higher mould and / or higher curing temperature (120°C instead to 100°C and / or 180°C instead to 160°C), the quality of composites is in general diminished (an decrease of V_f and an increase of V_p: eqn 6 and 7).

The microscopic analysis goes with the results found. Infused laminates manufactured using higher curing temperatures have several large size of voids greater than 100 µm. These voids are usually matrix voids and / or transition voids unlike those of laminates made with lower curing temperature (Fig. 2). These last cases have small sizes of voids ($\phi \approx 20$ µm). They are either matrix voids or perform voids ($\phi \approx 4$ µm).

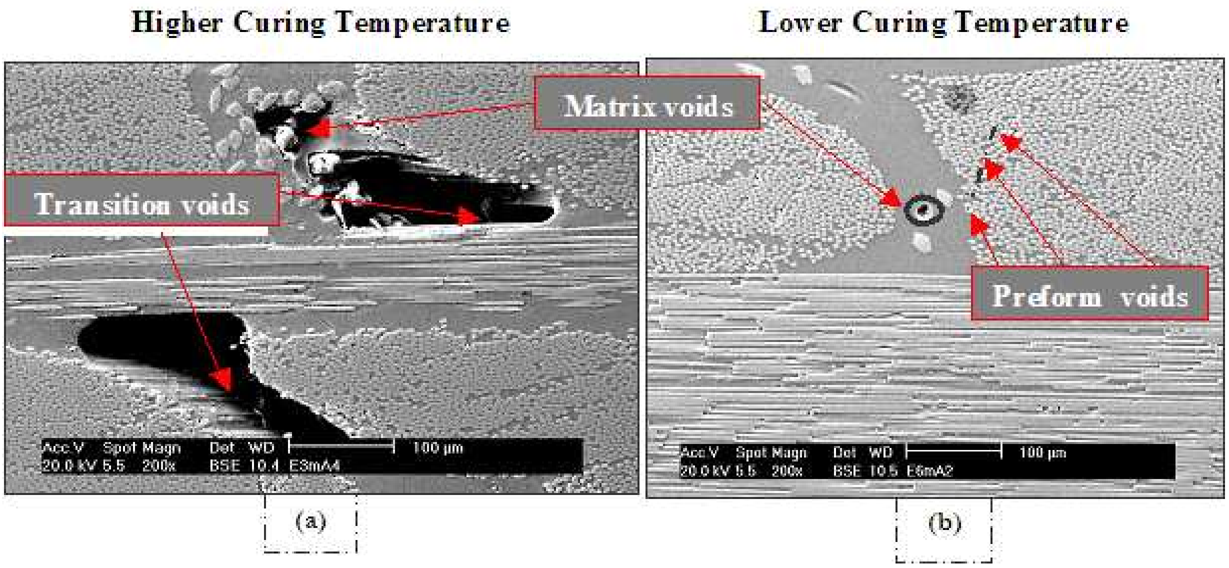


Fig. 2 : Microscopic analysis of effect of curing temperature

In another case, microscopic analysis of effect of mould temperature is made on composites manufactured at lower curing temperatures (160°C). Voids are more visible with higher mould temperatures (120°C) than with lower one ($\phi \approx 30$ µm instead to 2.5 µm, Fig. 3). A conclusion could be an increase of temperature might cause an expansion of voids [15].

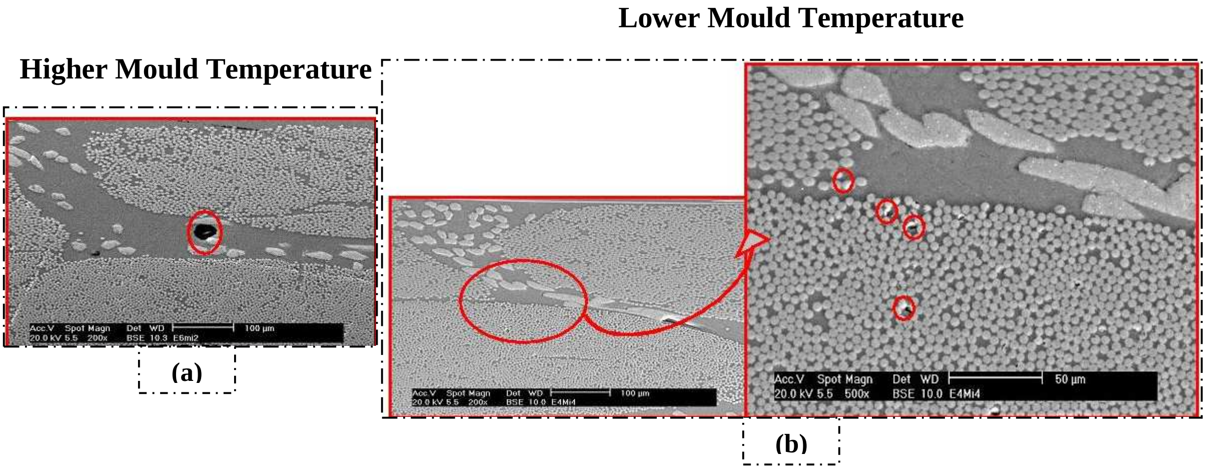


Fig. 3 : Microscopic analysis of effect of mould temperature

CONCLUSION

This study aimed to evaluate influence of process parameters on mechanical properties of carbon NCF and epoxy RTM6 laminates produced by Liquid Resin Infusion (LRI) process. The process parameters were identified. Seven parameters of direct influence and an interaction was found. An experimental plan was carried out to manufacture infused laminates with different process conditions using Taguchi L_{16} table. Statistical analysis of effects of parameters on UTS, UCS, ILSS, glass transition temperature, fibers volume fraction, void content and thickness of composites structures was performed.

This method allowed to write equation of each property's response of model according to effects of influence parameters. Results show influence of number of NCF layers on ILSS, Tg and infused composites thickness. The number of HPM layers, the plate sides (vacuum side or injection side) and vacuum level achieved during manufacturing, have effects on both the variation of morphological and mechanical properties of infused laminates. The microscopic analysis explains and verifies statistical results found. Only injection temperature of resin has an positive influence on the fibers volume fraction when it is in its higher level during composite manufacturing. Regarding the others temperatures, manufacturing with higher mould and / or higher curing temperatures has adverse effects on quality of composite structure and in particular on its void content.

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