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DEMONSTRATING THE NEED OF TAKING INTO ACCOUNT THE COMPATIBILITY TACKIFIER/RESIN, AND PROCESSING CONDITIONS TO CONTROL MECHANICAL PROPERTIES OF RTM CFRP

Florentin BERTHET Ecole des Mines d'Albi Carmaux

Pierre DEVOS Ecole des Mines d'Albi Carmaux

Thierry ANSART Centre d'Essais Aéronautique de Toulouse

Abstract

By using the Taguchi method of experimental design, it has been possible to demonstrate the influence of some processing factors on hot interlaminar shear strength and on hot open hole compressive strength of carbon/epoxy composite materials processed by resin transfer molding.

It was demonstrated that mold temperature and injection pressure have an effect on the final quality of the composite.

Moreover, the tackifier/resin compatibility is essential to guarantee conservation of the mechanical properties with temperature.

Introduction

Use of advanced composite materials in defense and aeronautics increased dramatically in the last 30 years because these sectors could bear extra costs of production provided there was a reduction in the component weight. The challenge which now has to be faced by composites materials producers is to reduce their costs. For this reason the Resin Transfer Molding Process (R.T.M.) nowadays provides new interest for the aeronautics industry.

Numerous studies have been performed to simulate flow of resin through preforms with applications in mold design. A new requirement has arisen: the identification of the relationship between process parameters and mechanical properties of the molded component.

Therefore a study had been conducted experimentally to fill a part of the gap. Mechanical properties such as open hole compressive strength (OHCS) and interlaminar shear strength (ILSS) have been evaluated.

Background

Taguchi method

Experimental design is well adapted to evaluate influence of processing conditions on mechanical properties of RTM CFRP because the potential parameters are numerous. Experimental design make it

possible to simultaneously minimize the number of trials necessary and maximize the quality of results.

A test on assumed equality of variance between samples and in samples leads to a partition of factors in three categories: very significant (VS), significant (S), not significant (NS). The greater the variance between two levels, the more significant the concerned factor.

An effect is associated with a factor i and a level j . It represents what should theoretically be added to the average value if the component has been molded at level j of the factor i . That is to say the effect E_{ij} of having molded at level j instead of at average level. This effect is defined as

$$E_{ij} = \text{average at level } j - \text{general average} \quad (1)$$

More details can be found in M.Pillet's book (6).

The main difficulty in this kind of problem is to establish a relationship between what happens microscopically and macroscopically. It is all the more complicated because the involved mechanisms are multiple. Moreover the expected effects are small at room temperature, thus one has had the idea of working at higher temperatures to facilitate the differentiation of materials as functions of their production conditions.

RTM

RTM is a process in which a dry fibrous preform is impregnated by a thermoset to produce composite. In a prior stage called preforming the fibre layup is made to the shape of the component to produce a preform. The role of the tackifier is to keep the shape of the component and to make it stiff enough to be handled prior to injection.

It is now widely accepted, that in RTM resin flow is governed by Darcy's law:

$$\vec{v} = - \frac{[K]}{\mu \varepsilon} \text{grad}(p) \quad (2)$$

v : front velocity in porous media, μ : viscosity, ε : porosity (volume left free by fibers divided by total volume), p : applied pressure K : permeability tensor.

In RTM porous media for impregnation are heterogeneous. In a component there are two levels of heterogeneity, microscopic (fiber level) and macroscopic

(fabric level). This implies that there are two types of flow (around and in bundle) and permeability.

Available studies of the influence of process parameters on mechanical properties are rare. Nevertheless Hayward and Harris(1) Dockum and Shell (2), Steenkamer(3), Young(4) and Michaeli(5) have done some work in this area. But their conclusions are contradictory.

Description of equipment and process

Our equipment consisted of

A square steel mold allowing fabrication of plates 400*400mm in different thicknesses. With this mold we can do peripheral injection (as indicated on figure 1) or directional injection.

An heated 20T hydraulic press

An RTM machine (pressurized pot) allowing injection up to 4 bars

A vacuum pump

A data recorder (pressures, temperatures, vacuum)

The typical resin transfer molding cycle is as follows

- 1) cut out of reinforcements
- 2) assembly of reinforcements
- 3) preforming 5 min at 120°C under 2 bars (cohesion of reinforcements is achieved by a tackifier that is a thermoplastic or a re activable uncatalyzed thermoset).
- 4) mold closure, vacuum application, resin degassing, and injection until mold is filled
- 5) holding pressure until no more material enters the mold
- 6) cure 75 min at 160°C
- 5) mold opening
- 6) post cure in autoclave following a programmed cycle (120 min at 180°C)

Description of experiment

parameters

Parameters chosen for this study are:

mold temperature, injection pressure, holding pressure, fiber bundle size, tackifier level, injection type and interaction between mold temperature and injection pressure. These parameters are varied between two values according to L8 Taguchi table (Table 1).

We discount the effects of cure and post cure because these two steps are not characteristics of RTM.

What is specific to RTM is the fiber/matrix interface formation with defect creation during injection caused by unknown mechanisms. From this one assumes that processing influences fiber/matrix bonding and thus the mechanical properties of the composite.

materials

Four fabrics were used, the fabric style was the same, but fabrics are woven from fiber bundles of different size (3K or 6K), and contained two possible levels of tackifier (5g/face/m² or 15g/face/m²). When different fiber bundle size are used, different thickness had to be used to achieve comparable fiber volume fraction (K_{vf}) (Table 2).

The injected resin is a special RTM single component epoxy resin .

Presentation of data and results

Results are summarized in table 3. For each of the processing conditions, five samples are cut from the plate. Average and standard deviation of evaluated properties are put into this table.

Interpretation of datas

ILSS at 120°C

Fiber bundle size, injection pressure, interaction, mold temperature and tackifier level (in increasing order of importance) have a significant (S) effect on ILSS at 120°C. Inversely, injection type and holding pressure have no significant effect on ILSS at 120°C (Table 4).

Our results, confirm conclusions given by Kittelson and al.(7), on importance of resin/tackifier compatibility. The new aspect is the effect of this incompatibility on mechanical properties. Presence of tackifier in the resin reduces the glass transition temperature significantly by 30°C. This loss is all the more important if the tackifier level is greater. At higher temperatures, materials with lower glass transition temperature, have reduced mechanical properties.

ILSS at 120°C is lower for an injection pressure of 2bars and a 100°C mold temperature as can be seen in figure 2. We assume that temperature and pressure conditions influence partition of resin flow between micro and macroscopic levels. A hot preform will make it easier for the resin to flow closer to fibers because of the associated viscosity reduction (equation 2), thus flowing microscopically. Inversely, for a low mold temperature and higher pressure (2 bars), flow will be preferentially macroscopic, in macrospace left free between two plies.

This accounts for some weakness of this resin rich area in shear.

OHCS at 120°C

In increasing order of importance, interaction, mold temperature, injection pressure and tackifier level influence significantly open hole compressive strength at 120°C (table 5).

Open hole compression at 120°C is weaker for high injection pressure at 140°C mold temperature as can be seen on figure 3. High temperature will favor microscopic flow and higher pressure disrupts fibers at both micro and macro scale. Compression is known to be sensitive to fiber misalignment (buckling).

conclusion

Experimental design made it possible to demonstrate the effect of some processing factors on mechanical properties of RTM CFRP, especially for hot ILSS and hot OHCS. We established the (limited) effect of temperature and pressure conditions during injection on final quality of composites. Moreover, the problem of resin/tackifier compatibility has to be considered to guarantee the conservation of mechanical properties with heat. Quantitatively, loss of mechanical properties is more significant at higher tackifier levels. The micro/macro competition explains why OHCS and ILSS are differently

influenced by processing conditions, and why studies on the subject disagree.

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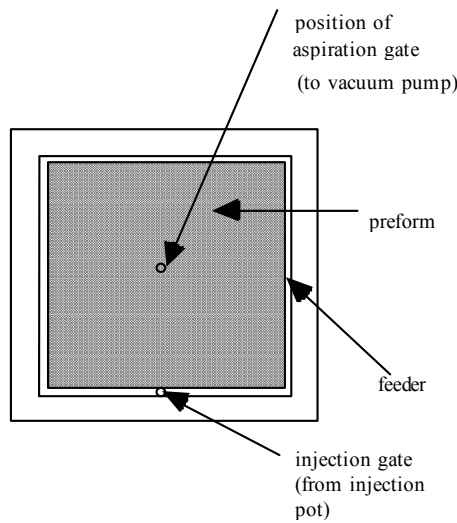


Figure 1: Top view of half mold with preform

N°	mold temperature (°C)	injection pressure (bars)	Interaction temperature - pressure	holding pressure (bars)	tackifier level (g/face/m ²)	injection Type	fiber bundle size (filaments)
1	100	1	1	2	5	directional	3000
2	100	1	1	3	15	peripheral	6000
3	100	2	2	2	5	peripheral	6000

4	100	2	2	3	15	directional	3000
5	140	1	2	2	15	directional	6000
6	140	1	2	3	5	peripheral	3000
7	140	2	1	2	15	peripheral	3000
8	140	2	1	3	5	directional	6000

Table 1: L8 Taguchi table applied to RTM problem



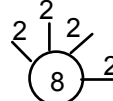
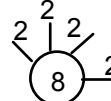
trial	3K	6K
ILSS	 Kvf=55% thickness 2 mm	 Kvf=50% thickness 2 mm
Open hole compression	 Kvf=59% 3mm [0/45/90/-45] _S	 Kvf=59% 4,5mm

Table 2: Layup used for different fiber bundle size

N°	hot ILSS (MPa)	standard deviation	hot open hole compression (MPa)	standard deviation
1	43,54	1,16	261,25	4,81
2	38,92	1,97	226,8	6,11
3	42,9	1,47	254,2	6,21
4	33,94	1,31	226,2	18,82
5	38,125	1,62	226,8	4,35
6	44,62	1,58	255,4	6,34
7	37,98	1,6	198,2	2,48
8	45,02	1,72	236,8	7,83

Table 3: Results of experimentation (averages and std dev. are calculated on 5 specimens)

	Effect (MPa)	F _{exp}	F _{thco95}	F _{thco99}	
mold temperature	0,8	8,63	4,17	7,56	VS
injection pressure	-0,67	5,98	4,17	7,56	S
holding pressure	-0,006	0	4,17	7,56	NS
tackifier level	-3,39	152,78	4,17	7,56	VS
injection type	-0,47	2,99	4,17	7,56	NS
fiber bundle size	0,61	4,95	4,17	7,56	S
interaction P-T	0,73	7,17	4,17	7,56	S

Table 4: variance analysis table for ILSS at 120°C

	Effect (MPa)	F _{exp}	F _{theo95}	F _{theo99}	
Mold temperature	-6,4	18,26	4,17	7,56	VS
injection pressure	-6,86	20,91	4,17	7,56	VS
holding pressure	0,59	0,15	4,17	7,56	NS
Tackifier level	-16,2	116,86	4,17	7,56	VS
injection Type	2,06	1,88	4,17	7,56	NS
Fiber bundle size	0,44	0,087	4,17	7,56	NS
interaction P-T	-4,94	10,87	4,17	7,56	VS

Table 5: Variance analysis table for open hole compression at 120°C

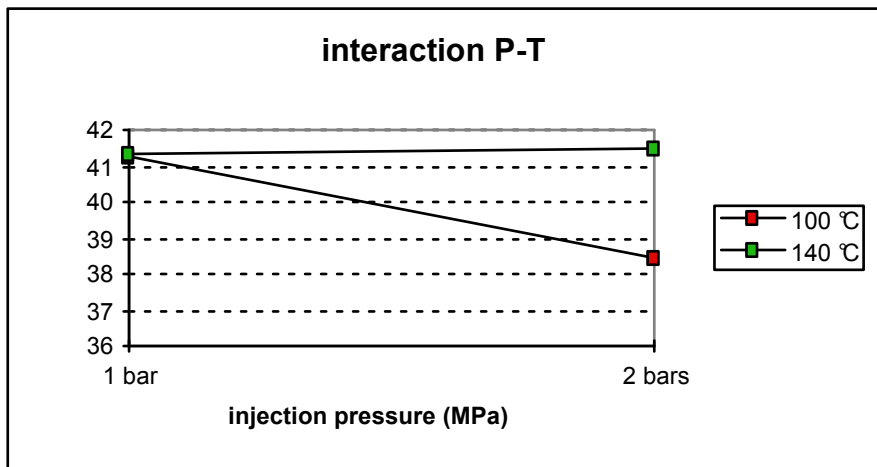


Figure 2: Effects graph of temperature-pressure interaction for ILSS at 120°C

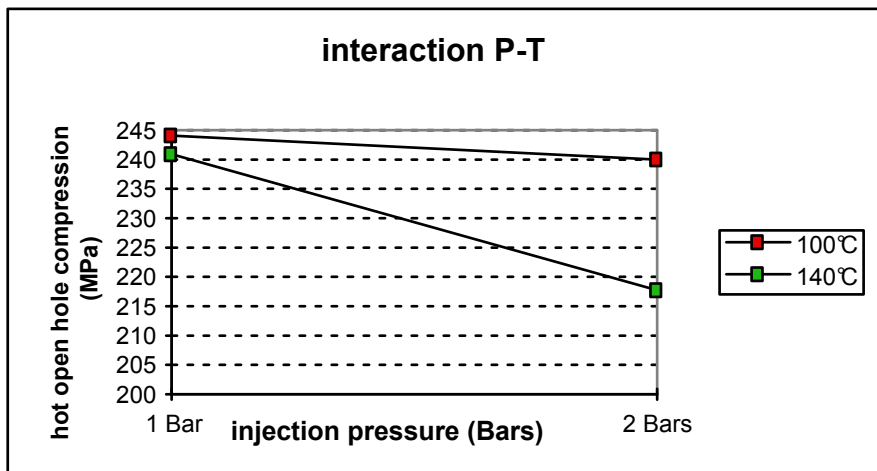


Figure3: Effects graph of temperature-pressure interaction for open hole compression at 120°C

Keywords: Resin Transfer Molding, Experimental design, processing, quality