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RTM CARBON COMPOSITES: INFLUENCE OF PROCESS PARAMETERS ON COMPRESSION STRENGTH AFTER IMPACT

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SUMMARY: By using the Taguchi method of experimental design, it has been possible to demonstrate the influence of some processing factors on compression strength after impact of carbon/epoxy composite materials processed by resin transfer molding. The parameters studied were injection and holding pressure, mold temperature, fiber bundle size, tackifier level, injection type and the interaction between injection pressure and mold temperature. Statistical analysis of results leads to the conclusion that temperature and pressure conditions modify the interface properties. Moreover, fiber bundle size, pressure, temperature and most likely tackifier level affect the post impact compressive behavior of the materials. Nevertheless, RTM can be considered as tolerant to process conditions variations.

KEYWORDS: resin transfer molding, experimental design, processing, quality, compression strength after impact

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 aerospace manufacturers is to reduce their costs. For this reason, the Resin Transfer Molding Process (R.T.M.) currently 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.

Compression strength after impact is one of the most significant mechanical properties for aerospace requirements. Resin transfer molding has been identified as a process through which it is possible to improve impact resistance.

Therefore a study had been conducted experimentally to fill a part of the gap. Compression strength after impact (C.S.A.I.) has been evaluated.

BACKGROUND

Taguchi Method

Experimental design using the Taguchi method is well adapted to evaluate influence of processing conditions on mechanical properties of RTM CFRP because the potential parameters are numerous. Experimental design makes it possible to simultaneously minimize the number of tests necessary and maximize the quality of results.

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

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 Eij of having been molded at level j instead of at average level. This effect is defined as Eqn 1.

$$\text{Eij=average at level j-general average} \tag{1}$$

More details can be found in M.Pillet's book (1).

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.

RTM

RTM is a process in which a dry fibrous preform is impregnated by a thermoset to produce a 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:Eqn 2.

$$\vec{v} = -\frac{[K]}{\mu\varepsilon}\,\mathrm{grad}(p) \tag{2}$$

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 bundles and in bundles) and permeability.

Available studies of the influence of process parameters on mechanical properties are rare. Nevertheless Hayward and Harris [2] Dockum and Shell [3], Steenkamer [4], Young [5] and Michaeli [6] have done some work in this area. But their conclusions are contradictory.

Impact Testing

Impact test procedure

The impact tests were performed using a drop weight tester. The specimens were clamped at all corners with a rectangular window (75x125 mm), in agreement with Airbus Industrie test method AITM 1-0010. The drop weight was released at a predetermined height to impact the center of the specimen with a given energy. Restrike of the drop weight was prevented by capturing it after first impact.

Non destructive control

Damage areas were measured with C-SCAN.

Compression after impact test procedure

Specimens (150x100 mm) were subjected to a compressive load using an antibuckling C.A.I. test fixture. The compression tests were conducted at a constant cross head speed of 1mm/mn.

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 (using the whole feeder as indicated on Fig. 1) or directional injection (by filling the side feeders).
- An heated 20T hydraulic press
- An RTM machine (pressurized pot) allowing injection up to 4 bars
- A vaccum pump
- A data recorder (pressures, temperatures, vaccum)

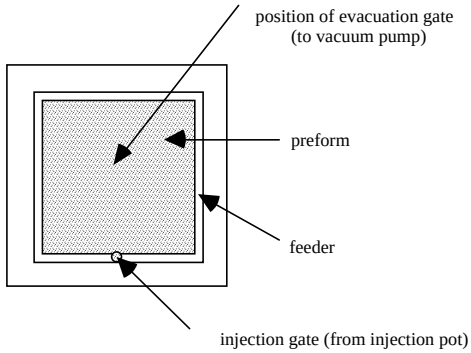


Fig. 1: Top view of half mold with preform

The typical resin transfer molding cycle is as follows

1) cutting 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-actifiable uncatalysed 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
7) mold opening
8) 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).

Table 1: L8 Taguchi table applied to RTM

specimen no.	mold temp. (°C)	injection pressure (bars)	Temp.-pressure Interaction	holding pressure (bars)	tackifier level (g/m^2/ face)	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

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

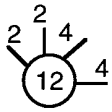
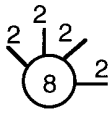
What is specific to RTM is the fiber/matrix interface formation with defect creation during injection caused by unknown mechanisms. From this one assumes that processing influences fiber/matrix bonding and thus the mechanical properties of the composite.

Materials

Four fabrics were used, the fabric style was the same (balanced fabric), but fabrics are woven from fiber bundles of different sizes (3K 400g/m^2 or 6K 600g/m^2), and contained two possible levels of tackifier (5g/m^2/face or 15g/m^2/face). When different fiber bundle size are used, different layups had to be used to achieve comparable bending stiffness (Table 2).

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

Table 2: Layups used for different fiber bundle size

	3K	6K
C.S.A.I.	 Kvf=59% 4,5mm [0/45/90/-45/0/45]$_S$	 Kvf=59% 4,5mm [0/45/90/-45]$_S$

PRESENTATION OF DATA AND RESULTS

For each of the processing conditions, one plate is produced and in each plate six rectangular samples were cut (150 (in O° direction)x100mm). Specimens were impacted at 5J(x2), 10J, 15J, 25J, 35J.

Table 3: Mass and energy impact conditions

Impact energy (J)	Drop weight (kg)
5	2,55
10	2,55
15	2,55
25	6,69
35	6,69

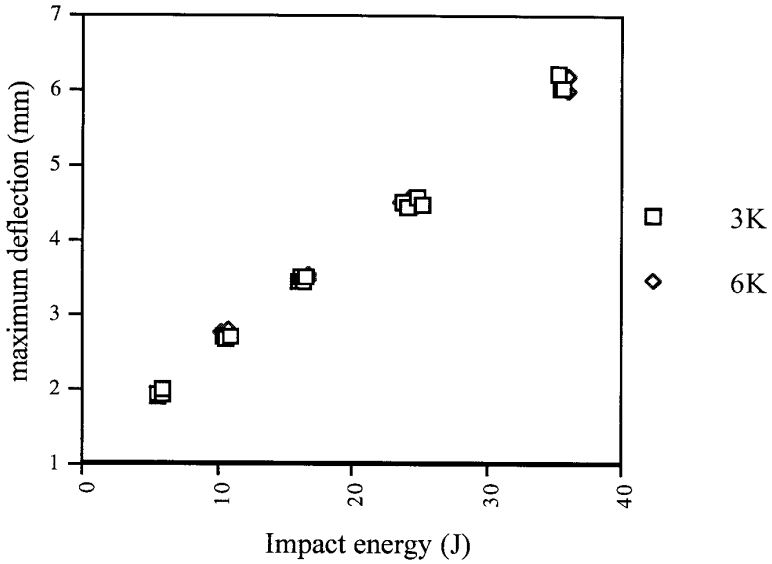


Fig.2: Maximum deflection at the centre of RTM plates during impact testing

As shown in Fig.2 different fiber bundle sizes do not lead to different maximum deflections. It proves that working at the same bending stiffness [8] is justifiable and that the change in properties is a consequence of processing and not a consequence of layup.

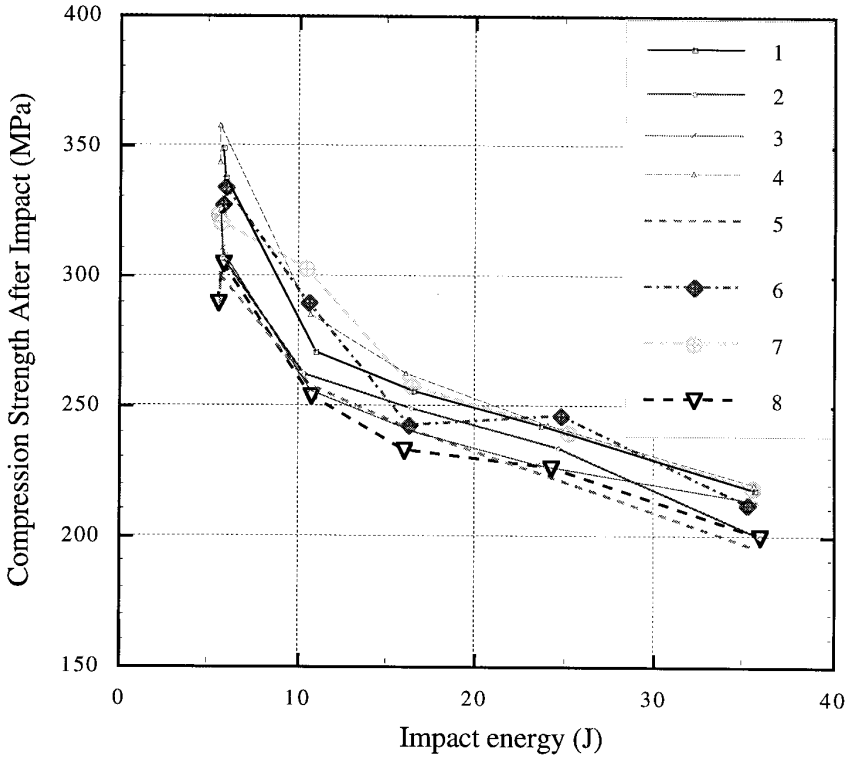


Fig.3: Compression strength after impact in resin transfer molding

The plates produced with different materials can be easily differentiated on Fig.3 by bundle size: 1,4,6,7 small (3K), 2,3,5,8 large (6K). But as presented in Fig.3 the data are not easy to interpret and some more work is required .

INTERPRETATION OF DATA

Damage Surface at 5J Impact

For an impact event damage initiation first appears at the fiber/matrix interphase at low levels of impact energy. There is an energy threshold at which microcracks appear at the fiber/matrix interface. Then cracks propagate in the matrix and at the interface between two plies. Thus the damage surface (measured by C.SCAN), for a low impact energy, will reflect the level of fiber and matrix bonding. Results of variance analysis in agreement with Taguchi method appear in Table 4.

The calculated value Fexp is more important than the tabulated value Ftheo95 for mold temperature and Ftheo99 for fiber bundle size. Mold temperature and fiber bundle size have a significant effect, and very significant respectively on damage area at 5J. For example, a mold temperature increase from 100°C to 140°C is associated with a reduction of the damaged area of 40 mm^2 that is to say a variation of twice the effect (19.9 mm^2).

Table 4: variance analysis table for damage surface at low impact energy

	Effect (mm^2)	**Fexp**	**Ftheo95 nu1=1,nu2=8**	**Ftheo99 nu1=1,nu2=8**	**sign.**
mold temperature	-19,9	6,21	5,32	11,26	S
injection pressure	6,63	0,69	5,32	11,26	NS
holding pressure	-0,38	0	5,32	11,26	NS
tackifier level	-11,63	2,13	5,32	11,26	NS
injection type	-1,88	0,06	5,32	11,26	NS
fiber bundle size	46,38	33,8	5,32	11,26	VS
P-T interaction	13,63	2,92	5,32	11,26	NS

Increasing mold temperature favours fiber/matrix bonding (damage area reduction with increasing temperature as indicated by the negative sign of mold temperature effect).

Influence of fiber bundle size is due to a structure effect (although laminates have bending stiffness as similar as possible) and also a difference of impregnation induced by fiber mismatch. The bigger the fiber bundle the more difficult to impregnate the bundle and the more important the damage area.

Kittelson and al. [7] have shown influence of tackifier compatibility with the resin. At higher temperatures tackifier level influences compression strength. In our study this parameter had an effect, but was not statistically significant. It is likely that tackifier (level) is also important (if temperature has to be considered).

Compression Strength after Impact

A least square fit of experimental data points leads to an equation of CAIS as a function of impact energy summarized in Table 5. Data are fitted to a power law : Eqn 3.

$$C.S.A.I. = K.(E_i)^n \tag{3}$$

K: coefficient, n:exponent, E_i: incident energy

Analysis of coefficient and exponent by the taguchi method is shown in Table 6 and in Table 7. Pooling method was used to artificially increase the degree of freedom (D.O.F. to 3) of the residual so as to draw conclusions more easily.

Fiber bundle size, mold temperature and holding pressure have a (very) significant effect on coefficient (Table 6).

Holding pressure has a positive effect on coefficient of C.S.A.I.. For example, increasing the holding pressure from 2 to 3 bars is associated with a variation of twice the effect (+8.5 MPa) that is to say an increase of coefficient of 17 MPa.

Table 5: equations for different process conditions

Plate n°	equation
1	$y=521x^{-0,2478}$ $R^2=0,967$
2	$y=468x^{-0,2301}$ $R^2=0,96$
3	$y=438x^{-0,2082}$ $R^2=0,97$
4	$y=532x^{-0,2503}$ $R^2=0,987$
5	$y=426x^{-0,2106}$ $R^2=0,983$
6	$y=501x^{-0,2379}$ $R^2=0,955$
7	$y=469x^{-0,2092}$ $R^2=0,97$
8	$y=421x^{-0,2052}$ $R^2=0,97$

Table 6: Variance analysis for coefficient y:C.S.A.I. x:E_i

	Effect	sum of squares	DOF	Var.	Fexp	Ftheo95 ν1=1,ν2=3	Ftheo99 ν1=1,ν2=3	sign.
Holding pressure	8,5375	583,1	1	583,1	12,60587	10,1	34,1	S
injection pressure	-2,99	406,4	1	406,4	8,785889	10,1	34,1	NS
Mold Temp.	-17,8075	2537	1	2537	54,84249	10,1	34,1	VS
Fiber bundle size	-33,7625	9119	1	9119	197,1426	10,1	34,1	VS
residual		138,8	3	46,26				

Table 7: variance analysis for exponent

source	Effect	sum of squares	DOF	Var.	Fexp	Ftheo95 ν1=1,ν2=3	Ftheo99 ν1=1,ν2=3	sign.
Holding pressure	0,005963	0,0003	1	3E-04	6,629955	10,1	34,1	NS
injection pressure	-0,00669	0,0004	1	4E-04	8,340294	10,1	34,1	NS
Mold Temp.	-0,00919	0,0007	1	7E-04	15,74159	10,1	34,1	S
Fiber bundle size	-0,01139	0,001	1	0,001	24,18302	10,1	34,1	S
residual		0,0001	3	4E-05				

Finally mold temperature and fiber bundle size have rather a negative effect on compression strength after impact. Higher temperatures and fiber bundle size lead to a reduction of compression strength of the material (Fig.4), and residual property also decreases faster with impact energy . Different temperature and pressure conditions during injection influence the mechanical property of the resin. It was proved that fiber bundle size, temperature and pressure conditions have a statistically significant effect on compression strength after impact.

These process parameters are supposed to act through modifying the resin's capacity to prevent fiber microbuckling and energy release rate (G_{IIc}).

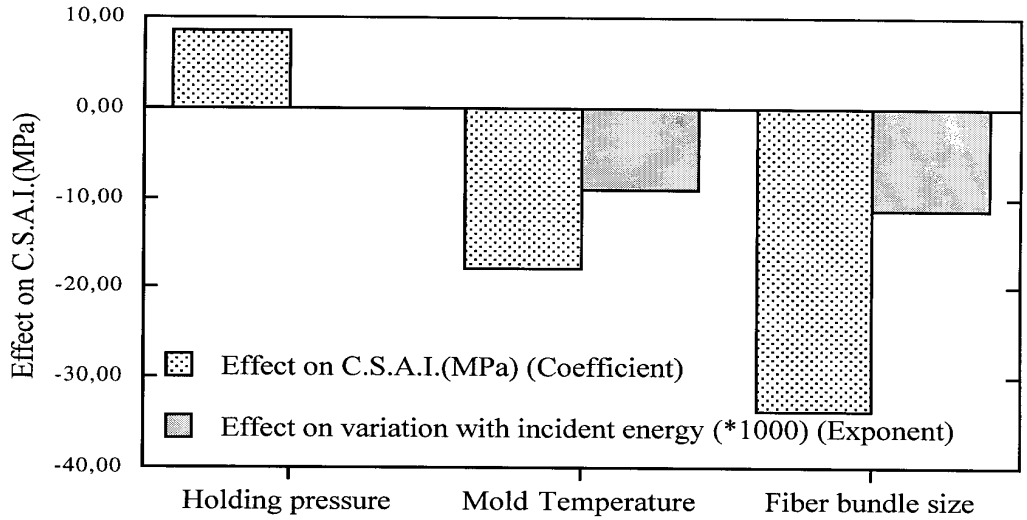


Fig.4: Effects of statistically significant process conditions on after impact compression behavior.

CONCLUSION

Experimental design made it possible to demonstrate the effect of some processing factors on mechanical properties of RTM CFRP. We established the (limited) effect of temperature and pressure conditions during injection on the final quality of composites. The initiation of cracks is favoured by large fiber bundles and low injection temperature. The compression and residual compression resistance are influenced by pressure, temperature and fiber bundle size. The most significant effect found was fiber bundle size. Nevertheless, RTM can be considered as tolerant to process condition variations.

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