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EFFECT OF PROCESSING VARIABLES ON LARGE PART INFUSION PROCESS

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SUMMARY: The resin infusion process VARTM has been identified as a cost-effective fabrication technique for complex-shape composite structures manufacturing. Dry textile preforms are resin-impregnated, consolidated, and cured in a single step process that eliminates costly prepreg tape manufacture and ply-by-ply lay-up. The principles of the three infusion processes are discussed along with the advantages of each technique compared with traditional composite fabrication methods such as prepreg tape lay-up and autoclave cure. The large number of processing variables and the complex material behaviour during infiltration and cure make experimental optimization of the infusion processes inefficient. Three-dimensional computer models have been developed which can be used to simulate the resin infusion processes. The model formulation and solution procedures are presented and the material property input data required for solution of the model are discussed. Potential benefits of modelization include reduced manufacturing time, cost and risk.

KEYWORDS: Resin infusion, resin formulation, viscosity, gel time, cure progress

INTRODUCTION

The Vacuum Assisted Resin Transfer Molding is a process in which dry reinforcement is converted to medium to high quality composite parts. The technique is based on the driving force for transferring the resin into the reinforcement with an applied vacuum, which at the same time also compact the stacked reinforcement.

The process starts by preparing the preform for the desired part, this includes cutting different types of reinforcement fiber. The second step in the manufacturing cycle is lay-up and draping of the material into the single sided mold. Here the reinforcement material is stacked and positioned into the mold part, and during this process the structure of the reinforcement is distorted to some degree to make the material layers adapt to the curved surface of the mold. This process is called draping. During the lay-up, the precut material is used but the main part of the reinforcement

material is placed directly into the mold from rolls to minimize the distortion of the material and also save lay-up time.

Once the lay-up is complete, the processing can be carried out. First, a vacuum pump is utilized to expel all air from the preform assembly. This will ensure the bagging film tightly covers the fiber reinforcement. Once the system equilibrates, the vacuum will drive the resin flow through the resin distribution tube and across the high-permeable distribution medium. Then, the resin fills the preform in the transverse, or through thickness direction dropping down from the high-permeable distribution medium. The vacuum source continues until the resin system begins to gel. The part may either cure at room temperature, or can be placed in an oven to assist the curing process. The final step in the VARTM cycle is demolding of the cured part and making it ready for it's function. The VARTM process is currently implemented in many fields, such as boat industry, automobile industry, transportation infrastructure and aircraft industry.

This research describes a new approach, were a room temperature processing and room temperature curing epoxy resin system is used for vacuum infusion molding for aerospace tooling. The effect of varying resin system parameters was investigated in order to minimize the process cycle time, whilst retaining laminate properties like mechanical, consolidation and Tg equivalent to autoclave cured laminates.

EXPERIMENTAL PROCEDURE

Resin System

The resin system used in the present work is a two component epoxy specifically developed to fulfill the requirements of the aerospace tooling applications in advanced vacuum molding (VARTM) process. The glass transition temperature of the epoxy based resin system is about 200C as characterized by Dynamic Mechanical Analysis (DMA). The cure schedule of the epoxy system was accelerated using a triethanolamine free base T1377. This modified system exhibits excellent room temperature curing and toughening characteristics for off-model post curing of tooling laminate following a 12 hours room temperature cure. It eliminates the need to use high temperature masters for generating high temperature composite production tools.

Analysis

The viscosity measurements of the resin mix were done on a digital Brookfield viscosimeter model DV2 PLUS using spindle N 3 with a rotating speed of 20 rpm. Change in resin mix viscosity was measured on 10 different formulations with different level of accelerator. No heat was applied to the resin mix to simulate the room temperature cure of the system.

Differential scanning calorimetry (DSC) was used to measure the heat that is evolved during the course of the resin system chemical reaction. Since epoxy reactions release energy during the cure (*i.e*. they are exothermic) DSC is a conventional tool for monitoring their cure. The uncured resin samples, weighing about 15 mg, were placed in an aluminium pan with a sealed lid and

placed opposite to the empty reference pan in the cell chamber. The DSC was then set for temperature range of 25 to 300 °C with a heating rate of 5 °C/min.

The flow progress of the resin system was investigated on a home testing system simulating the VARTM process of a well-determined carbon fibre layers sequence layed up on a flat glass plate. Fig. 1 is a schematic configuration of the VARTM procedure used in our experimental procedure.

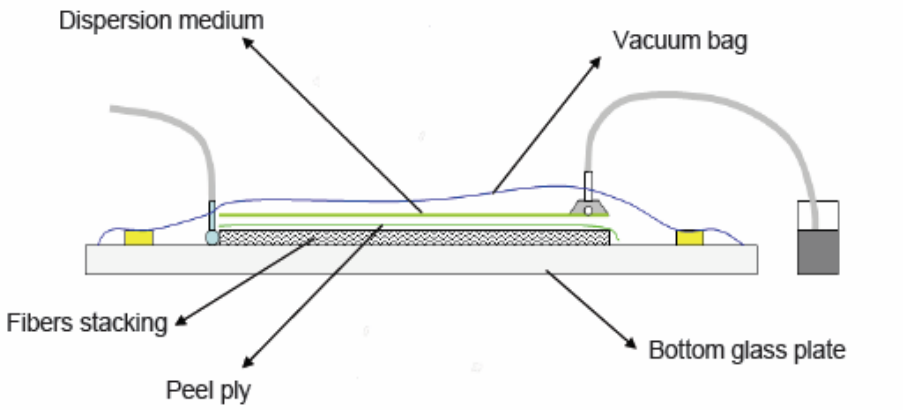

Fig. 1 Schematic presentation of VARTM experimental set-up.

EXPERIMENTAL RESULTS

For the purpose of resin system measurement, 10 formulations were mixed in the proportion listed in Table 1.

Table 1 Resin formulation for viscosity measurements

	RESIN MIX (Components content)			
Sample	**Resin**	**Hardener**	**Accelerator**	**Total**
1	0.625	0.356	0.019	100.0%
2	0.625	0.350	0.028	100.0%
3	0.625	0.344	0.031	100.0%
4	0.613	0.368	0.018	100.0%
5	0.610	0.366	0.024	100.0%
6	0.606	0.364	0.030	100.0%
7	0.588	0.412	0.000	100.0%
8	0.572	0.428	0.000	100.0%
9	0.556	0.444	0.000	100.0%
10	0.500	0.500	0.000	100.0%

The proposed resin formulations are based upon chemical nature of the resin and modification by substituting a small amount of hardener by accelerator. Results are illustrated in Fig. 2 and as we can see, the non-accelerated resin systems take a minimum of 5 hours to pass from liquid to gel state. In other side, accelerated resin system gel occur in less than 3 hours. Unlike high temperature resin systems, no viscosity decrease was observed before growing up to gel point. This can be interpreted as an initial data to take in account when starting such resin system cure process. Since the gel state of thermoset resins is different from system to system, it is also dependant from the chemical nature of the system and being dependant on the degree of cure, useful life and purity. Therefore, in the case of VARTM working process, the resin system rheology is useful especially during infusion period since the flow properties are directly related to the resin system viscosity. After the gel stage, a better knowledge of the cure progress as function of the crosslinking density and chain mobility is very helpful in terms of physical and mechanical properties of the moulded structure.

Defining the gel time as a limit state for our VARTM process we consider the formulation 2 in Table 1 as an optimum choice regarding to the process time and moulded tooling surface area. This resin formulation was then used for the rest of the testing program.

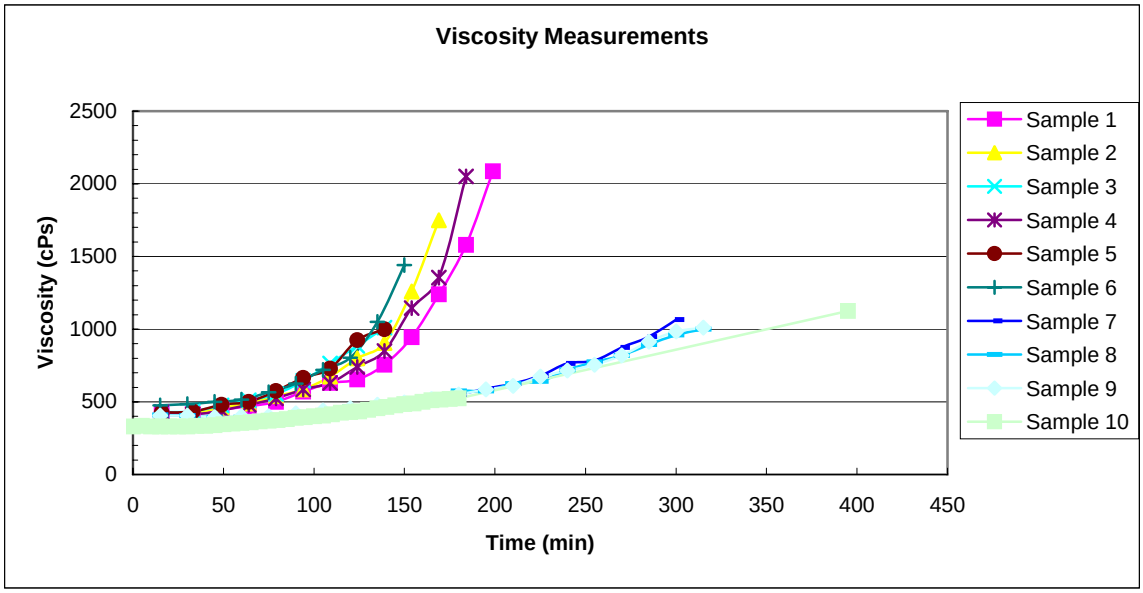

Fig. 2 Viscosity measurements for resin system formulations.

The curing behavior of the resin system formulation 2 was examined under nitrogen by DSC at a rate 5 C/min to determine the optimum curing conditions. Typical DSC thermogram is shown in Fig. 3.

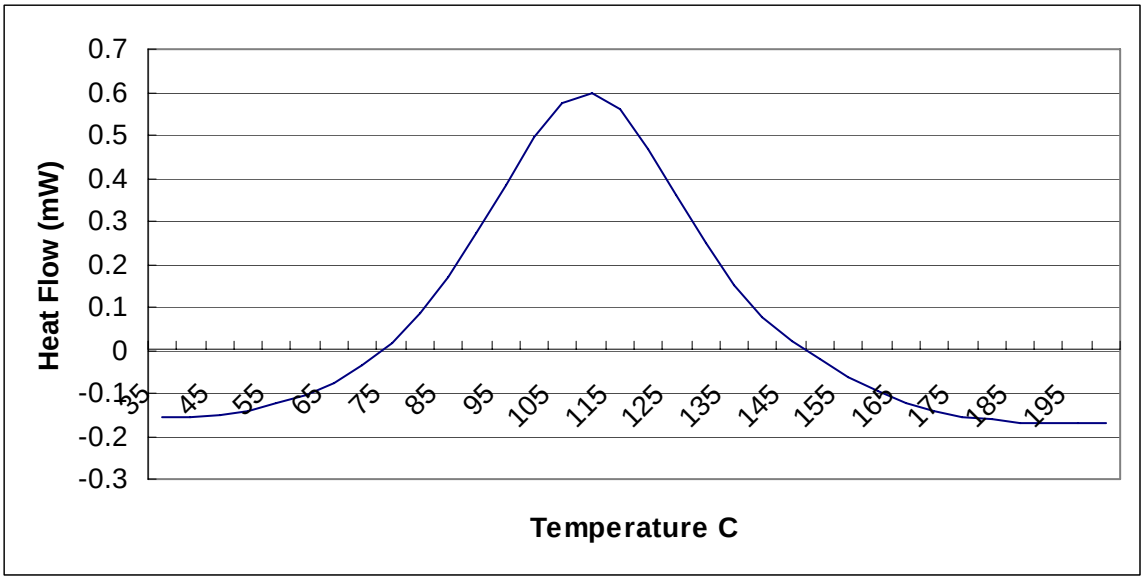

Fig. 3 DSC thermogram of resin system formulation 2
at a heating rate of 5 C/min.

The heat flow curve of the tested formulation shows a complete exothermic reaction and does not exhibit any endotherm. This is an indication that the mix containing the base resin, hardener and accelerator reacts in one time without any lag between hardener and accelerator and it indicates also that no crystallization was present in the used hardener. Note that the hardener was heated at 55 C for 1 hour and cooled at room temperature for one day before the system mix to ensure it's purity and to melt all crystals formed during the storage. The heat of cure was determined by integrating the curve heat flow vs. time and the amount was 460 J/g, which is closer to the supplier's value of 485 J/g. Comparing the present analysis to supplier's non-accelerated scan at the same heating rate as shown in Table 2, the presence of accelerator shifts the reaction on the temperature axis. This means that the reaction starts earlier in the case of accelerated resin, but it takes the same time to achieve a complete cure.

Table 2 Reaction temperature obtained by dynamic scan at 5 C/min

Formulation	**T initial (C)**	**T peak (C)**	**T final (C)**
Non-accelerated	121	156	255
Accelerated	45	110	180

After achieving these two sets of experiments, we proceed to try the VARTM process with the resin system formulation 2. Two kilogram of resin mix was formulated according to Table 1 and we layed up 10 layers (30 x 152 cm) of carbon fiber including NCF and Twill 2 x2 as shown in Table 3.

Table 3 Carbon stacks identification

Layer Number	Type	Identification
1	Carbon	3K - 2x2 Twill
2	Carbon	12K - NCS
3	Carbon	12K - NCS
4	Carbon	12K - NCS
5	Carbon	12K - NCS
6	Carbon	12K - NCS
7	Carbon	12K - NCS
8	Carbon	12K - NCS
9	Carbon	12K - NCS
10	Carbon	3K - 2x2 Twill

The lay-up was done on a glass plate used to have a good visual follow up of the resin flow during the process. The complete system used for the VARTM test is illustrated in Fig. 1. The resin mix was degassed under vacuum for 20 minutes at room temperature. The infusion test was done under vacuum of 29 inches of Hg. The inlet resin tube was clamped and then released to let the resin flow in the spiral tubing, then through the distribution media and into the stacked fiber as preform. Pictures of the flow follow up are shown in Fig. 4.

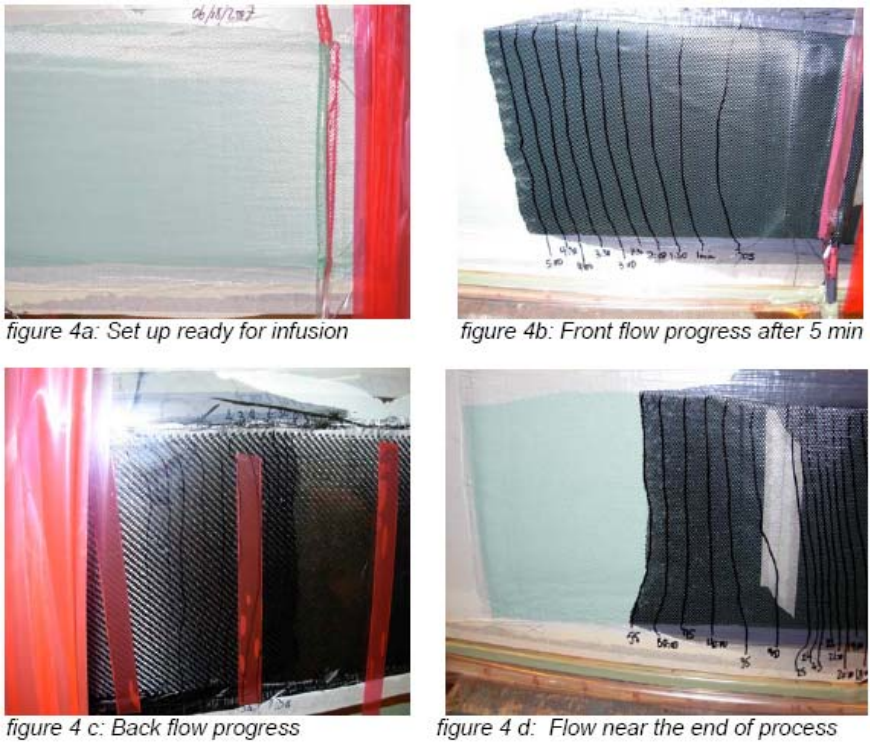

figure 4a: Set up ready for infusion

figure 4b: Front flow progress after 5 min

figure 4 c: Back flow progress

figure 4 d: Flow near the end of process

Fig. 4 Flow marks on the front and back faces of the experimental mold.

Data generated from marking the front and back flow positions are shown in Fig. 5. As we can see from this figure and for the first 20 inches, the front flow exhibits a higher impregnation

speed compare to the back one. This phenomenon is explained by the lag in impregnation caused by preform thickness crossing. After this stage, both the back and front flows show the same behavior with almost zero difference in impregnation speed.

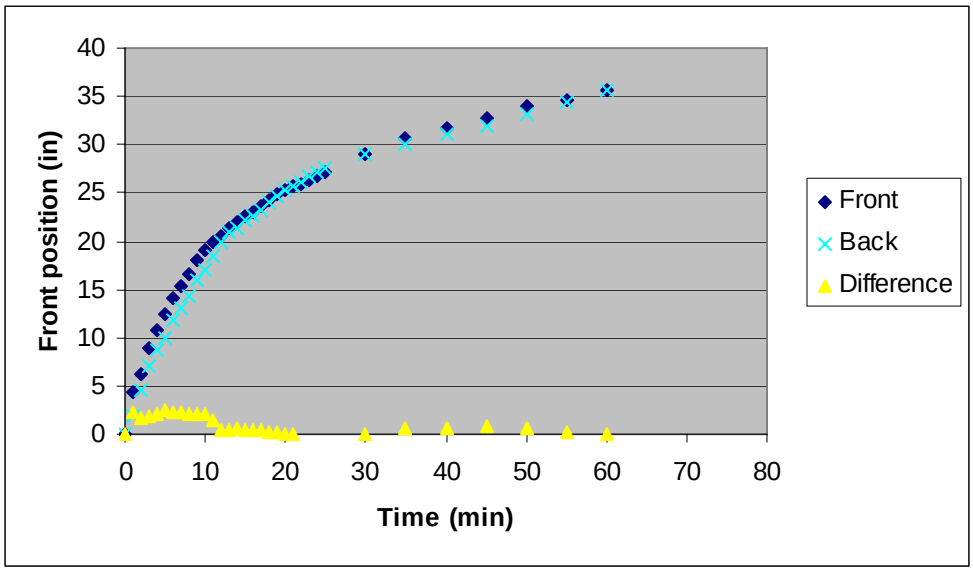

Fig. 5 Font and back flow positions as a function of time.

DISCUSSION

The strategy proposed in the present work is the use of viscosity and cure kinetics of a formulated resin system to understand the VARTM flow and cure process of aerospace tooling manufacture. The accelerated resin system showed a small change for the first 30 to 40 minutes and gelled around 3 hours at room temperature. This is being considered as good input data for the manufacturing process (VARTM) since we found that the rate of the impregnation flow is around 1 inch / min. Therefore, considering the infusion of a big surface tooling, one can place the channels spaced at 30 inches or less to get a non-disturbed or uniform impregnation flow without any potential change in the resin viscosity.

Moreover, the cure kinetics of the accelerated resin indicated an early start of the polymerization without affecting the reaction time. As we observed from viscosity testing, the only difference between the all formulations tested is the time to gel start. Consequently, the VARTM process can be considered as resin system gel dependent for the impregnation time and gel to peak cure for the part success.

CONCLUSION

The present work was based on the study of VARTM process optimization in terms of filling time and cure properties. A well-formulated resin was used for that purpose but with components levels change. We processed with the accelerated system to evaluate the effect of the accelerator presence on the gel time and how the formulated resin will react. The important result found in our study is the compromise between gel time acceleration and cure reaction progress. The use of DSC and viscosity analysis was of a great help in our study and this in term of establishing appropriate cure cycle for our tooling manufacture by VARTM process.