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Tackification of textile preforms for resin transfer molding

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Abstract: Tackified textile fiber preforms are used widely in resin transfer molding (RTM) to produce aerospace-grade composite parts. In the present study, a new tackifier was developed to improve RTM laminate performance. The influence of tackifier concentration on spring back, thermal properties and mechanical performance was studied. It has showed that the new tackifier was compatible with the matrix resin and improved the textile handling ability; the ILSS was slightly increased without decreasing of thermal properties, modulus and flexural strength.

Key words: tackifier; resin transfer molding (RTM); performing

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1 Introduction

In recent years liquid composite molding processes such as resin transfer molding (RTM) and structural reaction injection molding (SRIM) have become popular and effective fabrication techniques for many commercial automotive and aerospace applications. In these processes, liquid thermoset resin is combined with continuous fiber reinforcement to yield a composite part. The molding cycle consists of four steps: loading the dry fiber reinforcement into a preheated cavity, resin injection, curing and demolding. One of the bottlenecks in these processes is efficient loading of the fibers into the mold. Usually the individual layers of the reinforcement must be cut and shaped to conform to the various curvatures of the tool surfaces, which can be very time consuming. In order to handle the fiber reinforcement as a single unit, individual layers are bonded by binders or tackifiers. These are normally thermoplastic polymers or thermoset resins that are solid at room temperature, and melt easily on heating. Upon cooling, the resin resolidifies bonding the fibers together. This technique is especially suitable for large parts with complicated geometry, and for consolidation of bulky textile type preforms with 3D textiles^[1]. The use of binder/tackifiers helps in obtaining net-shape preforms which are critical to the fabrication of high performance parts. Wrinkles in the reinforcing fabric often when oversized preforms are compressed into the molding tool, resulting in structural failure of the composite.

Some of the common methods of binder/tackifier application utilize veils, solvent spray and powder. Veils can be placed between adjacent plies of "broad goods" followed by fusing the ply stacks with heat and pressure to form a preform^[2]. Alternatively, solvent spraying of dissolved binder/tackifier onto each broad

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good can also be used, but is not very feasible due to health hazard and an additional requirement for solvent removal. Another approach involves applying the binder/tackifier in form of powder. The resinous powder can be distributed on the fiber mats either with a sifter type apparatus as in this study, by electrostatic spraying^[3], or by extrusion through a slotted die to obtain a flat sheet^[4]. The resin sheet is then incorporated between layers of fiber mats.

In high performance composites, a reactive tackifier is often used to achieve effective consolidation of fiber preforms. Another reason for using the tackifier is to obtain sufficient tack so as to reduce slippage between the layers, and to minimize spring back. The latter occurs when the elastic stresses stored in the fibers during deformation are greater than the adhesion forces due to tack^[5]. The reactive tackifier can be either an uncatalyzed or a catalyzed thermoset resin which is generally a partially reacted matrix resin. It is desired that the reactive tackifier dissolves in, and reacts with the resin injected into the mold cavity during mold filling.

2 Experimental

2.1 Materials

The textile reinforcement for the analysis of the tackifier was T300-40B carbon fiber satin 1/4 weave fabric (style G803) provided by Hexcel composites. With an areal density of 285 g/m², Epoxy resin 3266 from BIAM was employed as the matrix. ES-T321 is an epoxy based uncatalyzed dry solid tackifier manufactured by BIAM.

2.2 Tackification process

The 10% (mass fraction) solution of tackifier in acetone was sprayed onto the fabric. Only one side of the fabric was sprayed, and then it was placed in a convection oven at 70°C for ten minutes to evaporate the acetone. The spraying and drying cycles were repeated until the fabric was loaded with a desired tackifier concentration. A total of four tackified panels were manufactured, and the tackifier concentration of panels was 2%, 6%, 8%, 10% and 13% (mass fraction), respectively.

The powder tackifier was applied manually onto the fabrics using a sifter with 400 μ m average mesh opening. By using an oven that was heated to approximately 90°C, the tackifier was melted onto one side of the fabric. The cycle were repeated until the fabric was loaded with a desired tackifier concentration.

2.3 RTM laminate process

Symmetric 0°-laminates were fabricated using 8 plies of the carbon fiber satin weave fabric and the 3266 RTM resin. The tackifier fabrics were pressed for 30 min at 80°C and 100 kPa in a laboratory press to obtain preforms for simplified mold assembly. The assembled mold was heated to 45°C in a press while vacuum was applied to the cavity. Resin injection was executed at this reservoir and resin injection lines were heated to 45°C. The mold was purged with 3266 RTM resin that was collected in a resin trap. After injection was completed, the outlet valve was closed and pressure was raised to 1 MPa. Cure was performed by heating to 125°C and holding for four hours. The laminates were demolded after cool-down to 60°C. The cured laminates were trimmed and cut into specimens suitable for analysis.

2.4 Tackifier-preforming analysis

Rheology of the tackifier resins and the RTM resins was investigated using Brookfield DV-I. The experiments were executed with a heating rate of 2°C/min. The preforming performance of the different tackifiers was characterized by measuring spring back dimension of preforms using a U-Shape Bending test.

Eight plies of the tackified fabrics were fixed in a U-Shape Bending tool under a pre-specified temperature and pressure. After cooling, the preform was taken out of the tool and spring back dimension was measured. A Philips XL-30 scanning electron microscope (SEM) was used to study the distribution and location of the tackifier on the carbon fabrics.

2.5 RTM laminate analysis

Dynamic Mechanical Testing and Dynamic mechanical analysis (DMA) experiments were conducted on the RTM laminates using a TA Instruments DMA Q800. The experiments were carried out at a heating rate of 5°C/min to 300°C, a frequency of 1Hz and amplitude of 50 μ m. Glass transition temperature was reported as the peak in loss modulus. Flexural properties were determined by 3-point bending test according to ASTM D790-93. Interlaminar shear strength (ILSS) tests were also performed in a three point bending apparatus according to ASTM D2344-84.

3 Results and discussion

3.1 Tackifier-preforming Analysis

The cure and rheological behaviors of the ES-T1 tackifier and the 3266 resin were measured using a Brookfield DV-I Viscosity Instrument respectively. The results are summarized as follows.

From the scanning viscosity at a heating rate of 2°C/min (see, Fig.1), the viscosity data shows a gel point at 125°C for 3266 resin. The decrease of viscosity in the early heating period indicates that the temperature effect dominates. The rapid increase of viscosity after the early period means that the chemical reaction starts and the resin reacts to the gel point. ES-T321 shows no gel point, but has a much higher viscosity than 3266 resin. The impregnation of the preform was carried out at 45°C where the tackifier maintained solid like characteristics resulting in improved resistance against the opposed resin flow.

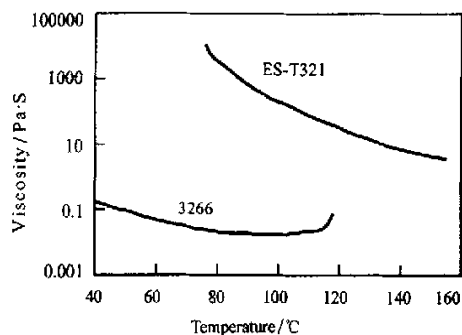


Fig.1 The viscosities of tackifier and RTM resins (heating rate: 2°C/min)

In Fig.2, the surface of the tackifier treated fabrics is shown. As found in previous research, the spray tackifier shown in Fig.2(a) was more uniformly distributed than the powder tackifier shown in Fig. 2(b). This is primarily attributed to the finer, more homogeneous spray application.

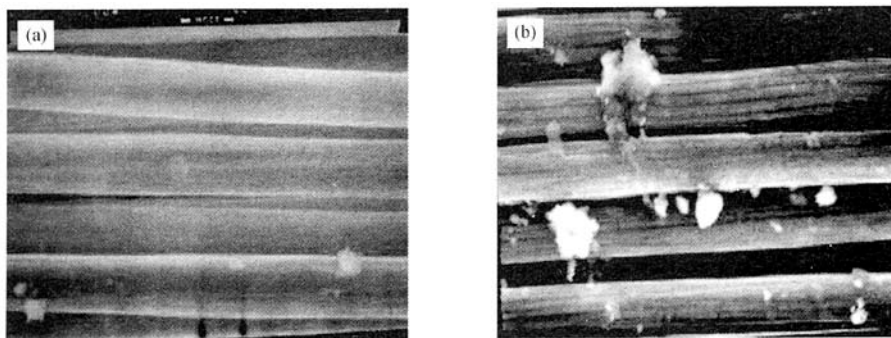


Fig.2 Tackified woven fabric surfaces containing
(a) — coverage by spray tackifier; (b) — coverage by powder tackifier

The effect of both tackifier and tackifier concentration on preform spring back is shown in Fig.3. Springback is an important characteristic in analyzing tackifier performance since it provides information about how well a tackifier is capable of retaining the desired preform shape. Spring back was found to decrease with increasing tackifier contents as measured for the tackifier. Interestingly, when using the same concentration, the spray tackifier prevented spring back more effectively than the powder tackifier. Since the solvent coating causes the filaments to become stiff, the decrease in spring back can be attributed to the increase in the elastic modulus of the fibers from a more uniform coating of the tackifier on the filament. It should be mentioned, however, that tack or interply adhesion was greater in the case of the powder technique compared with the tackifier applied by the solvent method. When powder tackified, most of the tackifier remains on the fabric weave surface. That is why the spring back control in this case is not as good as in solvent method. These observations indicate that both interlayer and intralayer area coverage of the tackifier are important for the control of interply adhesion and fiber preform spring back.

3.2 RTM laminate analysis

Laminates were fabricated by RTM to investigate the influence of ES-T321 tackifier on RTM laminate performance. Four laminates containing different tackifiers were made with a fiber volume content of 60%. fabric that was sprayed with ES-T321 tackifier had a total loading of 4%, 6%, 8%, and 13% (mass fraction), respectively.

3.3 Tackifier washout resistance

Epoxy based tackifier resins are soluble in epoxy based RTM resin systems. Hence, it is possible that the tackifier dissolves into the RTM matrix before the mold-filling step is completed. As a result, the tackifier can be washed out or migrate through the laminate due to the resin flow. Kittelson *et al.* found that at a rather high injection temperature, the tackifier had low viscosities and were therefore more prone to wash-out^[6]. In previous research by Seferis *et al.*, it was suggested that when injection was conducted at temperatures below the softening of the tackifier, migration could be effectively reduced^[7]. This was obtained by reducing the injection mold temperature and by selecting a higher molecular weight for the tackifier.

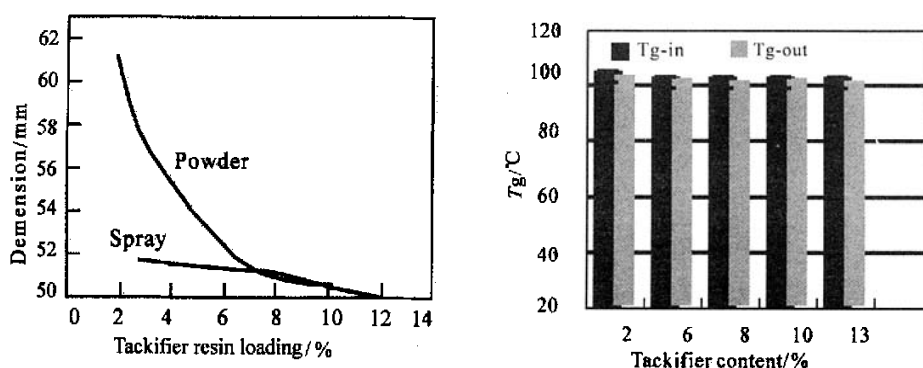


Fig.3 Spring back effect and glass transition temperatures of laminates containing different tackifiers

The method used to quantifiably measure tackifier migration involved DMA. The glass transition temperatures (T_g) defined as the peak in the loss modulus curve, were measured along the resin flow path (down the length of the laminate) for each specimen. The results are shown in Fig.3. DMA analysis showed that the glass transition temperature for all materials were $(103 \pm 2)^\circ\text{C}$ at all measured locations. However, the extent of the variation was within the error of DMA measurements. Hence, these results suggested that the uncatalyzed tackifier resins reacted with the bulk matrix and a significant tackifier migration did not occur.

3.4 Laminate properties

The flexural moduli were calculated using the linear section between 25% and 75% of the strain to failure value which results are depicted in Fig. 4. The results show that essentially no decrease in modulus occurred when only small amount of tackifier were utilized. These results confirmed that matrix epoxy network was not affected significantly by the existence of the tackifier, and that the tackifier resin took part in the reaction of 3266 RTM resin without leading to plasticization of the matrix.

The observed flexural strength values followed the same trend as the determined modules. Interlaminar shear strength test(ILSS) were also performed on the laminates and the results are summarized in Fig. 4. The results show that the interlaminar shear strength was slightly increased with increased tackifier load.

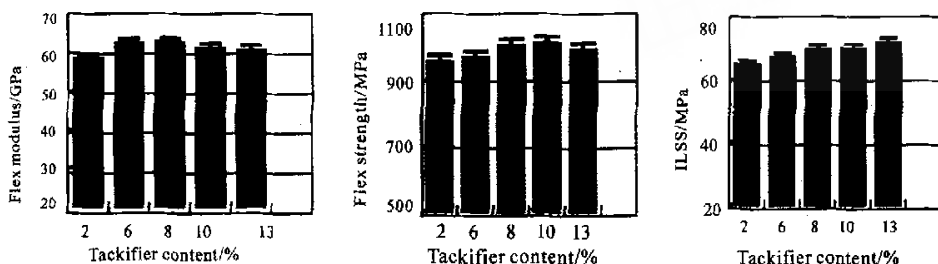


Fig.4 Flexural modulus, strength and ILSS of laminates containing different tackifiers

4 Conclusions

RTM laminates containing different tackifier concentrations were manufactured. The influence of tackifier concentration on springback, thermal properties and mechanical performance was studied. It was shown that the powder tackifier were less uniformly distributed on the textile surface than the spray tackifiers, leading to a lower preform integrity as shown by springback testing. Resin transfer molded laminates were fabricated with spray tackified preform. Dynamic mechanical analysis showed that the glass transition temperature was not greatly affected by the presence of tackifier at any of the concentrations tested. Furthermore, it was found that the tackifier was not washed out during injection and remained in the interlayer where they were initially applied. The spray tackified materials showed that the tackifier slightly increases the ILSS, and no decrease in modulus and flexural strength of the composite materials was observed.

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