CHARACTERIZATION OF PREPREG TACK TO AID AUTOMATED MATERIAL PLACEMENT

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ABSTRACT

The lay-up performance during automated material placement (AMP) can be significantly affected by the quality of the adhesion (tack) between the prepreg material and the tool, between adjacent plies, and between the material and rollers on the deposition head. Typically, material temperatures for best processing conditions are determined by trial and error. A new tack test has been developed that can measure the effects of not just material temperature, but also feed rate, on material tack. This characterization can guide machine processing conditions.

To aid optimizing AMP process parameters, tack on different surfaces was characterized experimentally for a commercial uni-directional carbon fiber/epoxy prepreg tape. A peel test fixture recently developed at the University of Nottingham was used to measure tack between a prepreg and a steel substrate. Modifications of the test methodology enabled the measurement of prepreg-prepreg and prepreg-fluorinated ethylene propylene (FEP) tack.

For different surface pairings, tack was measured at a range of temperatures and feed rates. At a given temperature, maximum prepreg-prepreg tack is significantly higher than the corresponding maximum prepreg-steel tack. Both occur at approximately the same feed rate. Prepreg-prepreg tack was also measured as a function of the out-time of the material. The same peak tack values could be achieved with aged material but at slower feed rates. This correlated with the relaxation data observed from rheology measurements. Maximum prepreg-FEP tack is significantly smaller than the corresponding maximum prepreg-steel tack and was observed at feed rates one order of magnitude slower than for prepreg-steel tack.

Employing time-temperature superposition based on rheological data from the prepreg resins the tack force at a given temperature and feed rate can be shifted to a reference temperature by multiplying the feed rate by a shift factor. Shifting of tack data also allows prediction of maximum tack as a function of the feed rate and processing temperature.

This methodology enables optimization of AMP process parameters to achieve maximum production rates and product quality. Additionally, this method is a reliable way to quantitatively measure tack of prepregs and can be used as a standard by both prepreg suppliers and prepreg end users.

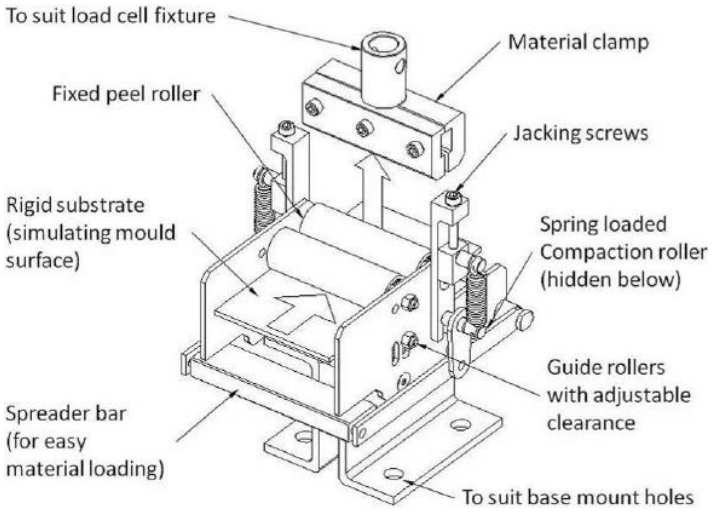
1. INTRODUCTION

In the manufacture of composite components employing automated material placement (AMP) processes, the lay-up performance can be significantly affected by the quality of adhesion (tack) between the prepreg material and the tool, between adjacent prepreg layers, and between the prepreg and rollers on the deposition head. To aid optimizing AMP process parameters, this study aims at characterizing tack on different surfaces experimentally for a commercially available aerospace grade uni-directional carbon fibre/epoxy prepreg tape. A test fixture, recently designed by Crossley [1] for measurement of tack between a prepreg specimen and a rigid tool surface, was adapted to enable measurement of prepreg-prepreg and fluorinated ethylene propylene (FEP)-prepreg tack.

2. EXPERIMENTATION

2.1 Testing Apparatus

The test fixture used in this study comprises an aluminium frame holding two pairs of aluminium rollers (Fig. 1, top). For one pair of rollers, the clearance is fixed during the tests (guide rollers). In the second pair of rollers, the top roller is fixed (peel roller), while springs apply a vertical force on the bottom roller (compaction roller), pushing it against the peel roller. The force can be controlled by tightening or loosening jacking screws which elongate or relax the springs. The fixture is mounted on the base of a universal testing machine. A material clamp, which holds one end of the prepreg specimen, is attached to the cross-head and load cell of the testing machine.



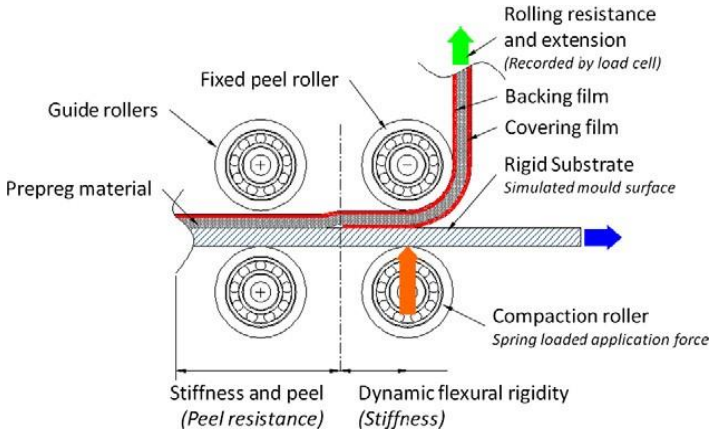


Figure 1: Features of the tack test fixture (top) and detailed view of specimen in fixture (bottom), both adapted from Crossley [1].

For measurement of tack between a prepreg and a rigid substrate, rectangular prepreg specimens with a length of 215 mm and a width of 75 mm were laid up on rectangular substrates made from stainless steel [1]. While prepreg is supplied with a protective film attached to one face (the inner face when on a roll, here referred to as "paper face"), the unprotected face (here referred to as "no paper face") was covered with an additional layer of film to avoid sticking of specimens to the test fixture (Fig. 1, bottom). On the face of the prepreg specimen to be in contact with the substrate, the covering film was removed on a length of 80 mm. By adjusting the calibrated jacking screws, a compression force of 100 N was applied across a specimen width of 75 mm for all tests documented here. It should be noted that additional experiments indicate that the measured tack force converges with increasing compression force and at a target compression force of 100 N, the tack force is not very sensitive to uncertainties in compression force

During the tack test, the cross-head moves upwards at constant (adjustable) speed, which translates into a horizontal movement of specimen and substrate through the fixture at constant feed rate. While specimen and substrate are pulled through the fixture, the first pair of rollers provides guidance. In the second pair of rollers, the compaction roller presses the prepreg specimen against the rigid substrate. At the same time, the cross-head peels the prepreg from the substrate around the fixed top roller. Hence, the prepreg specimen is applied to and peeled from the substrate in a single continuous motion. The contact time is inversely proportional to the feed rate.

The tensile force at the load cell was recorded as a function of the cross-head displacement. Two different phases can be identified during the test (Fig. 2). In the first phase, the part of the prepreg specimen covered on both faces with protective film is compressed between compaction and peel roller. The prepreg surface is not in contact with the substrate. Hence, only the force required to bend the specimen around the peel roller is measured at the load cell during this phase. In the second phase, the part of the prepreg specimen where the protective film was removed, and the surface is in contact with the substrate, is compressed between compaction and peel roller. In this phase, the force to overcome the peel resistance is measured in addition to the bending force.

The average tack force at the given specimen width is calculated from the average measured force for both phases of the experiment according to

$$\text{tack force} = \text{force (phase 2)} - \text{force (phase 1)} + \text{bending force} \quad (1)$$

where the "bending force" refers to the combined effect of bending and friction in the fixture when one layer of protective film is bent round the peel roller, which was quantified separately. It is to be noted that this calculation procedure implies a simplification since it assumes independent bending of all material layers and does not consider the influence of adhesion between prepreg and protective film on the bending stiffness. However, since the material thickness is small, the induced error can be assumed to be small.

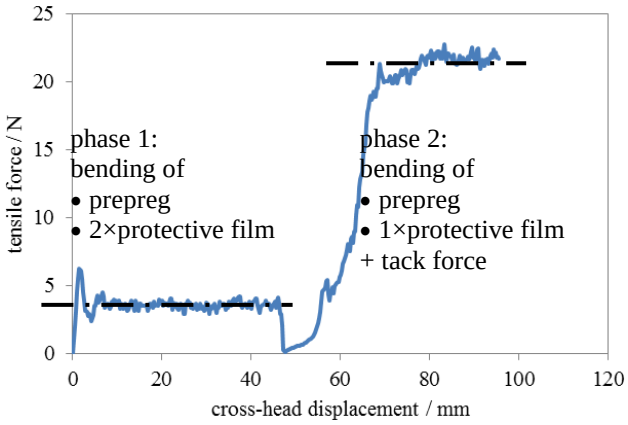


Figure 2: Raw data for prepreg-prepreg tack ("no paper face" on "paper face"), acquired at a temperature of 40 °C and a feed rate of 150 mm/min.

For the first set of experiments, the tack between a uni-directional carbon fibre/epoxy prepreg tape at a width of 75 mm and steel substrates was measured as described above. Both faces of the prepreg were tested.

The existing procedure was then adapted for prepreg-prepreg tack testing by bonding one prepreg layer onto a steel substrate ("no paper face" in contact with substrate) using double-sided adhesive tape. For the tack tests, the bond between the prepreg and the substrate needs to be stronger than the tack between the prepreg layers. Adhesive tape wash chosen because it did not affect the properties of the resin in the prepreg, and was found easier to handle than liquid adhesives. Tack was measured for the surface pairings "no paper face" on "paper face" and "paper face" on "paper face" similar to prepreg-steel.

During the tape placement process, prepreg tapes are applied and compacted using a roller coated with fluorinated ethylene propylene (FEP). To measure tack between prepreg and FEP, a 75 μm thick FEP film, cut to a length of 215 mm and a width of 75 mm, was attached to the load cell. The film was peeled from a prepreg layer bonded onto a steel substrate (i.e. FEP in contact with "paper face"), and tack force measured.

For each configuration, tack was measured at different temperatures and different feed rates. For temperature control, the test fixture was enclosed in an environmental chamber. An example for typical results is shown in Fig. 3.

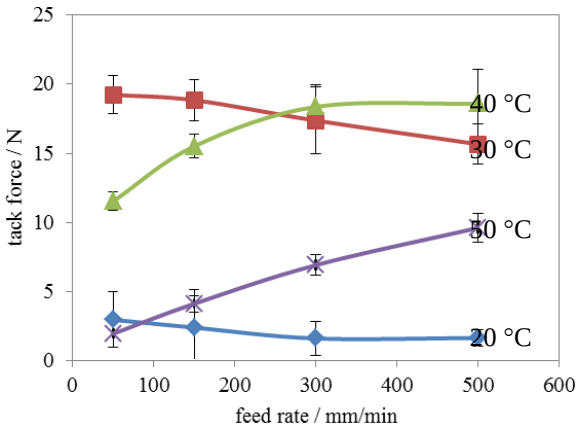


Figure 3: Prepreg-prepreg tack data ("paper face" on "paper face"), acquired at four different temperatures and four different feed rates.

2.2 Time-temperature Superposition

Typically, the behaviour of a polymer at one temperature can be related to that at another temperature through a shift in the time scales involved. This principle of time-temperature superposition (TTS) is frequently applied to rheological data to produce a rheological master curve, effectively extending the frequency range beyond the measurable range.

To determine the shift factors for TTS, a reference temperature T_0 is selected, and the storage and loss moduli, G' and G'', measured at other temperatures T are shifted horizontally (by multiplying the frequency, ω) until optimal overlap is achieved with data at the reference temperature. This means that the modulus G (i.e. G' or G'') at temperature T and frequency ω is equal to the modulus G at the reference temperature and shifted frequency ωa_T, i.e.

$$G_T(\omega) = G_{T_0}(\omega a_T) \quad . \tag{2}$$

The dependence of the shift factor on the temperature is commonly described by the Williams, Landel and Ferry equation (WLF):

$$\log_{10} a_T = \frac{-C_1 (T - T_0)}{C_2 + (T - T_0)} \quad . \tag{3}$$

The WLF equation can be used to determine a shift factor to or from the reference temperature to or from any chosen temperature.

Crossley et al. [2] demonstrated that the principle of TTS can be applied to tack measurements of prepregs, using shift factors obtained from complementary rheological measurements. This implies that the tack force, F, measured at temperature T and feed rate r, is equal to the tack force at the reference temperature and shifted feed rate ra_T:

$$F_T(r) = F_{T_0}(r\, a_T) \tag{4}$$

This process was employed for shifting tack data obtained in this study. It allows tack data at high feed rates, which are experimentally not achievable due to limitations on the cross-head

speed on the testing machine, to be obtained through shifting of data acquired at low temperatures.

To obtain the constants in Eq. (3), oscillatory rheometry was carried out on the neat resin used to produce the prepreg. Storage modulus and loss modulus were determined at different temperatures using an ARES Rheometer (TA Instruments). The tests were carried out using a frequency sweep, at 0.5 % strain, and 25 mm diameter parallel plates. In order to determine the shift factors for TTS, a reference temperature T_0 was selected (20 ºC), and the moduli were measured at this temperature and other temperatures. Moduli shift to achieve optimal overlap with the reference temperature data and fit of the WLF equation were carried out using the rheometer software.

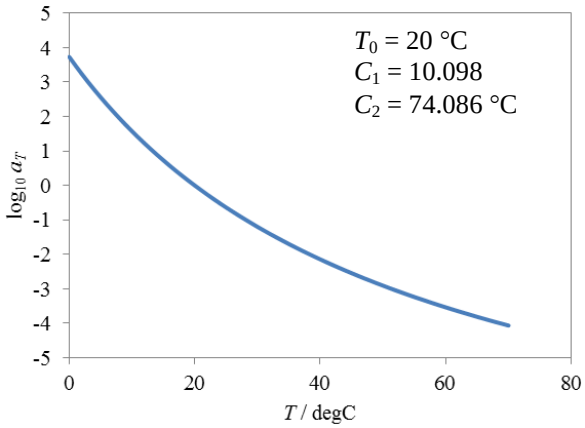


Figure 4: Shift factors for resin with heat history as a function of temperature, *T*, obtained from the WLF equation.

The prepreg supplier provided two sets of neat resin: the first one was the resin film used for prepregging and the second one was the resin film after it had been exposed to the heat and duration of the prepreg cycle. Although data was generated for resin samples with and without heat histories, for generating the tack master curves, the data from the sample with heat history was used. Results for the shift factor and the constants for Eq. (3) are given in Fig. 4.

3. RESULTS

Tack was measured for the surface pairings described in Section 2. For comparison of results, all data were shifted to the reference temperature T_0 = 20 °C. The resulting tack master curves (plotted in Figs. 5 to 7) typically have a bell shape, although this is less obvious if the absolute tack values are low. Here, the shifted data in the top diagram in Fig. 6 correspond to the un-shifted original data shown as an example in Fig. 3. In addition, the maximum observed tack values for each surface pairing and the shifted feed rate at which the maximum occurs at T_0 are listed in Table 1.

The data indicates that the tack force for the “paper face” in contact with the steel substrate tends to be higher than for the “no paper face” in contact with steel (by 59 % at maximum tack). Prepreg-prepreg tack tends to be lower for “paper face” on “paper face” than for “no paper face” on “paper face” (by 16 % at maximum tack). The maximum prepreg-prepreg tack is significantly

higher than the maximum prepreg-steel tack (by factors between 2.2 and 4.2). The shifted feed rate at maximum tack is 14 % smaller than for prepreg-steel tack.

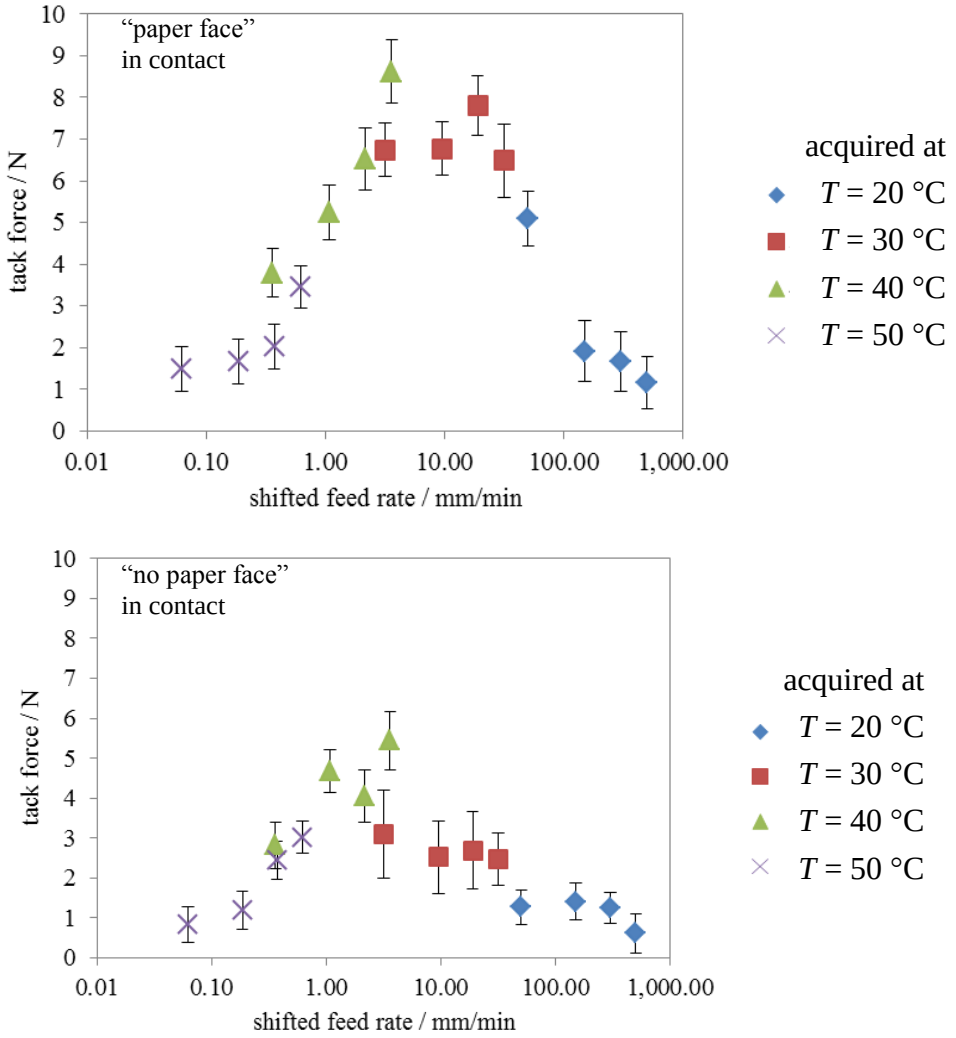


Figure 5: Measured tack force for prepreg tape on steel as a function of feed rate at various temperatures, shifted to the reference temperature T_0 = 20 °C; average values and standard deviations are indicated.

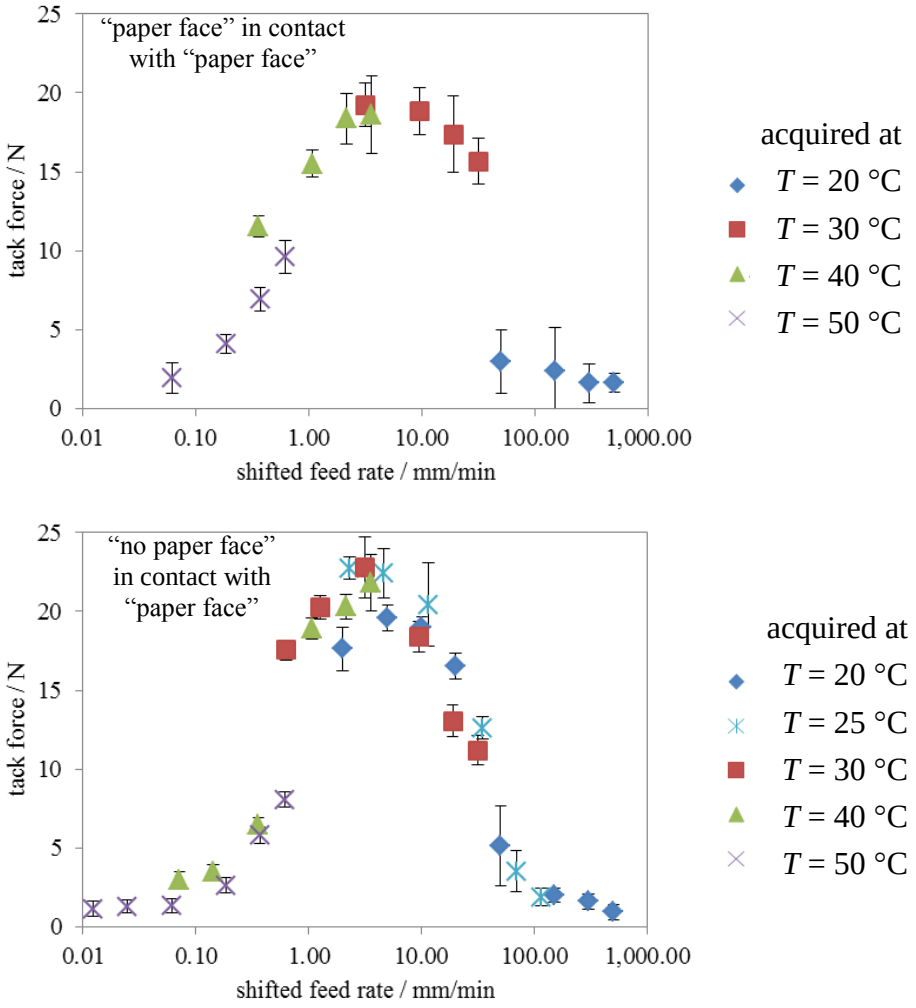


Figure 6: Measured tack force for prepreg tape on prepreg tape as a function of feed rate at various temperatures, shifted to the reference temperature T_0 = 20 °C; average values and standard deviations are indicated.

Table 1: Measured maximum tack values for different material combinations (average values and standard deviations are given) and shifted feed rates at which the maxima occurred (at T_0 = 20 °C).

material combination	max. tack force / N	feed rate / mm/min (at max.)
prepreg – steel ("paper face")	8.6 ± 0.8	3.6
prepreg – steel ("no paper face")	5.4 ± 0.7	3.6
prepreg – prepreg ("paper face" – "paper face")	19.2 ± 1.4	3.1
prepreg – prepreg ("no paper face" – "paper face")	22.8 ± 2.0	3.1
FEP – prepreg ("paper face")	1.6 ± 0.5	0.6

FEP-prepreg tack is generally very low. Hence, the coefficient of variation, i.e. the ratio of standard deviation to average value, is high (Fig. 7), although the standard deviation is in the same order of magnitude as for prepreg-steel and prepreg-prepreg tests (Table 1). The maximum FEP-prepreg tack is significantly smaller than the maximum prepreg-steel tack (by factors between 0.3 and 0.2). The maximum in tack force between the FEP film and the prepreg occurs at significantly lower shifted feed rates than for prepreg-steel and prepreg-prepreg tack (by factors of approx. 0.2).

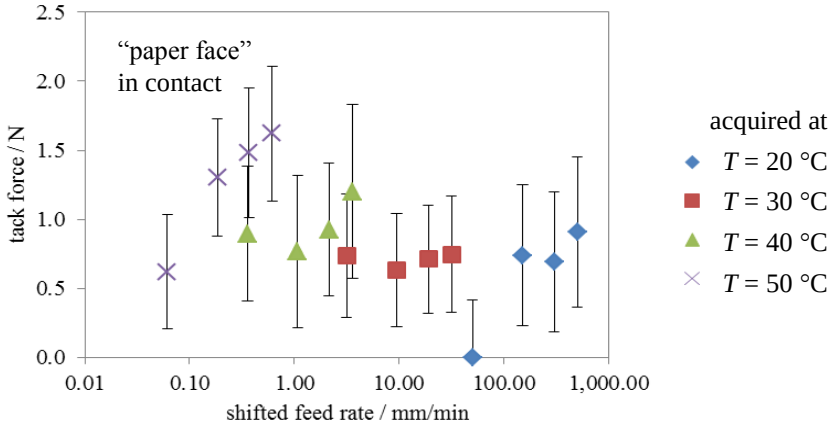


Figure 7: Measured tack force for FEP on prepreg tape as a function of feed rate at various temperatures, shifted to the reference temperature T_0 = 20 °C; average values and standard deviations are indicated.

In addition, the effect of the inter-ply angle on measured prepreg-prepreg tack was studied for the pairing "no paper face" on "paper face", at T = 30 °C and a feed rate of 50 mm/min. The orientation of the ply bonded onto the substrate was incremented by 15°, starting from 0°. The measured tack increased continuously with increasing inter-ply angle (Fig. 8). However, for ply angles greater than 60°, adhesion between the bottom prepreg layer and the substrate failed, while there was still adhesion between both prepreg layers. For these cases, the actual tack force was higher than the recorded value. Ignoring the values at ply angles of 75° and 90°, tack was found to increase by approximately 33 % from 0° to 60° (Table 2).

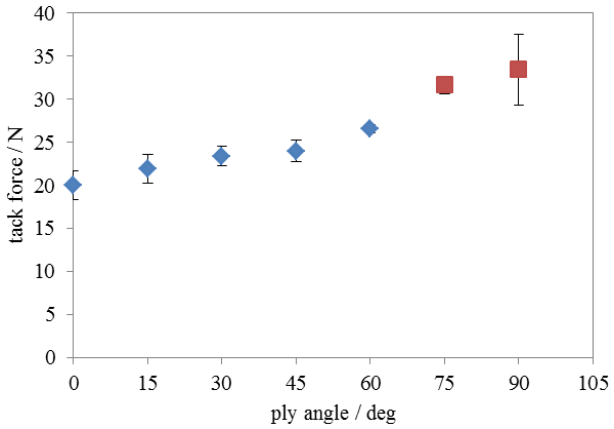


Figure 8: Effect of ply angle on measured prepreg-prepreg tack

Table 2: Increase in prepreg-prepreg tack with increasing ply angle.

ply angle	0°	15°	30°	45°	60°	75°	90°
increase in tack	-	10 %	17 %	20 %	33 %	58 %	67 %

All results for prepreg-steel, prepreg-prepreg and FEP-prepreg tack show identical trends (as in the example in Fig. 3): At low temperatures, maximum tack occurs at low feed rates. With increasing temperature, the maximum in tack moves to higher feed rates. Application of TTS results in bell curves (Figs. 5 to 7). Tack data can be related to the appearance of the surfaces in contact, which gives some qualitative indication of the failure mode in peel.

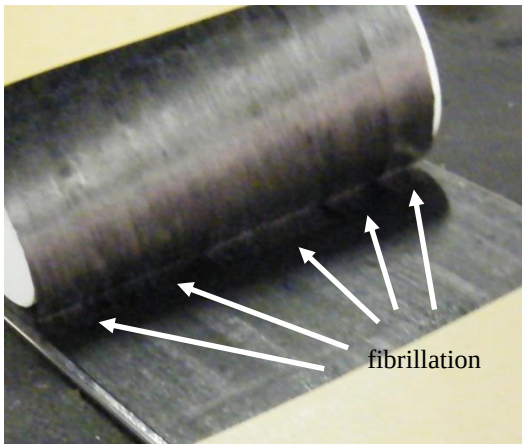
During AMP, typically two failure modes are observed. The first one is cohesive failure that occurs within the resin layer leaving heavy resin deposition on the substrate plates. The second type of failure is interfacial failure that appears to occur at the surface through apparent lack of complete contact. There may be a third type of failure resulting from the resin-fiber interaction. As the resin stiffness decreases with the application of heat, good contact between plies is maintained. However, further elevation of temperature leads to the resin losing its cohesive stiffness resulting in good contact but failure within the resin itself. A peak in tack is observed at a viscoelastic stiffness where sufficient contact is possible and the resin/prepreg has sufficient cohesive strength. Visual observations of resin threads forming between surfaces are consistent with resin softening at higher temperatures.

As described in Section 2, the prepreg is applied to and peeled from the substrate in a single continuous motion. The contact time, which is inversely proportional to the feed rate, dictates the cohesion time for molecular bonds to form. The contact time also controls the peel time, which influences the strain rate. At any given temperature, superposition of two competing effects determines tack and results in the shape of the bell curves in Figs. 5 to 7:

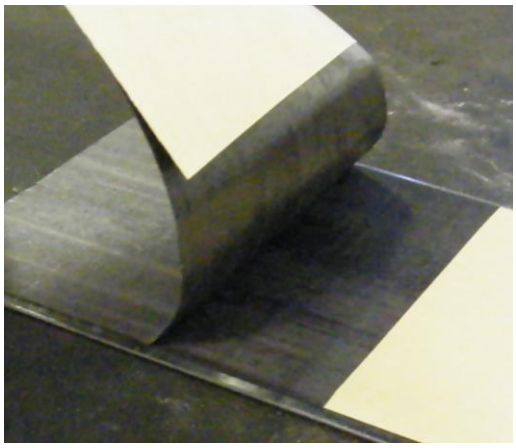
- Decreasing feed rate corresponds to increasing peel time (decreasing strain rate). The further to the left of the maxima in the bell curves for tack as a function of the feed rate, the more time for adhesion between layers to form, but also the more time for loss of cohesion within the resin. Hence, tack is dominated by cohesive failure in the resin, and long drawn-out resin threads may be observed (Fig. 9, left).

- Increasing feed rate corresponds to decreasing cohesion time. The further to the right of the maxima in the bell curves for tack as a function of the feed rate, the more elastic the behaviour of the viscoeleastic resin, and the less time available for adhesion to develop between layers. Hence, tack is dominated by adhesive failure, and little or no resin threads are formed between the surfaces (Fig. 9, right).

The photographs in Fig. 9, which relate to the tack data in Fig. 6, are two examples for the appearance of the surfaces in contact near peak tack (left image) and to the right of peak tack (right image).



"paper face" on "paper face",
T = 40 °C, feed rate 500 mm/min;
resin threads between prepreg layers at contact line (fibrillation);
tack force (18.6 ± 2.4) N



"paper face" on "paper face",
T = 20 °C, feed rate 50 mm/min;
no evidence for resin threads;
tack force (1.9 ± 1.0) N

Figure 9: Surfaces of tested specimens at different test parameters.

In the tests for measurement of FEP-prepreg tack, virtually no tack (< 1 N) was measured at temperatures of 20 °C and 30 °C. At T = 40 °C, a stick-slip effect was observed, and at T = 50 °C, resin was found sticking to the FEP film surface after the tests, forming longitudinal bands (Fig. 10). It is thought that these somewhat randomly spaced bands reflect local maxima and minima in compression force across the tape width and are related to thickness variations of the prepreg tape.

Surface morphology and distribution of resin on the surfaces are also thought to cause the difference in prepreg-steel tack for different surface orientation (Table 1) and in prepreg-prepreg tack for different inter-ply angles.

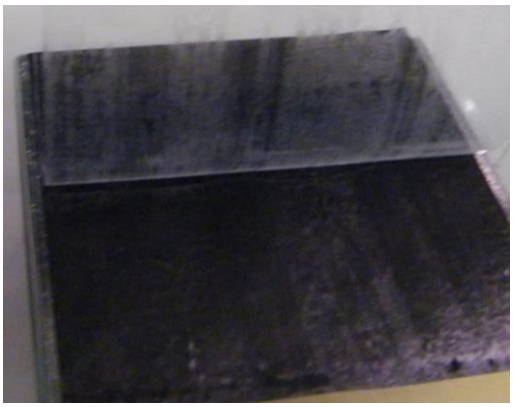


Figure 10: Longitudinal resin bands on FEP film.

Employing TTS, tack data at a reference temperature can be shifted to any arbitrary temperature using a new shift factor obtained in analogy to Eq. (3). The feed rate for the peak tack force can be shifted using the same method. This allows the feed rate required to obtain maximum tack at a given process temperature to be predicted. Feed rates at peak tack for different material combinations are listed in Table 3 for a range of temperatures. It is to be noted that these data are based on the measured maximum tack values. Fitting a bell curve to the measured data to find the maximum would give slightly different results. Hence, shifted data listed in Table 3 indicate orders of magnitude rather than accurate feed rates.

Table 3: Predicted feed rate at peak tack for different material combinations as a function of temperature, *T*.

T / °C	feed rate / mm/min		
	prepreg-steel	prepreg-prepreg	FEP-prepreg
10	0.10	0.08	0.02
20	3.60	3.10	0.60
30	57.18	49.24	9.53
40	504.49	434.42	84.08
50	2929.41	2522.55	488.24
60	12496.53	10760.90	2082.76

4. CONCLUSIONS

For a commercial uni-directional carbon fibre/epoxy prepreg tape, tack on different surfaces was characterized experimentally to aid optimization of AMP process parameters. A recently developed test fixture, which had so far been used for measurement of tack between a prepreg and a steel substrate, was adapted to acquire additional data for prepreg-prepreg and FEP-prepreg tack. For prepreg on steel, prepreg on prepreg, and FEP on prepreg, and different prepreg surface orientations, tack was measured at a range of temperatures and feed rates. While, at low

temperatures, maximum tack occurs at low feed rates, the maximum in tack moves to higher feed rates as the temperature is increased.

In addition, rheological data for the resin in the prepreg were acquired, which allowed shift factors for time-temperature superposition to be determined. Through multiplication of the feed rate with these shift factors, the tack force, measured at a given temperature and feed rate, could be shifted to any arbitrary reference temperature. Comparison of tack data indicates that maximum prepreg-prepreg tack is significantly higher than the corresponding maximum prepreg-steel tack. At a given reference temperature, both occur at approximately the same shifted feed rate. For FEP on prepreg, virtually no tack was measured at low temperatures. The maximum in FEP-prepreg tack, which was recorded at higher temperatures, is significantly smaller than the corresponding maximum in prepreg-steel tack. At the same reference temperature, it was observed at shifted feed rates one order of magnitude smaller than for prepreg-steel tack. Measurement of prepreg-prepreg tack at fixed temperature and feed rate for different inter-ply angles (under 60°) indicated that tack increases continuously with increasing inter-ply angle.

The capability to predict the feed rate for maximum tack at any temperature through shifting of tack data means that this work has considerable potential to contribute to the optimization of AMP process parameters in achieving maximum production rates and product quality.

5. ACKNOWLEDGEMENT

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