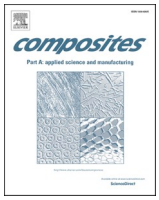
Composites: Part A 188 (2025) 108561

Contents lists available at ScienceDirect

Composites Part A

journal homepage: www.elsevier.com/locate/compositesa



Repeatability and reproducibility of the measurement of prepreg tack following ASTM D8336: Results of a round-robin study

A.M. Joesbury [a], A. Endruweit [a], D. Budelmann [b], S.P. Giannis [c], D.S.A. De Focatiis [a,*], D.B. Call [d], C. Chan [e], G.Y.H. Choong [f], R.A. Clark [g], J.A. Collins [g], D.T. Fishpool [g], J.M. Garber [h], S. Ghose [e], J. Golding [f], S.T. Good [e], B.R. Jones [h], D. Meiners [b], O. Niitsoo [i], C.T. Palmer [j], C.C. Qian [k], A.W. Rivas [i], G. Santacruz Rodriguez [l], R.E. Stillwell [h], J.D.S. Vincent [c], H. Yuan [k]

[a] University of Nottingham, Nottingham, UK
[b] Clausthal University of Technology, Stade, Germany
[c] National Physical Laboratory, Teddington, UK
[d] Toray Composite Materials America, Seattle, WA, USA
[e] The Boeing Company, Seattle, WA, USA
[f] Vestas Technology UK, Isle of Wight, UK
[g] National Composites Centre, Bristol, UK
[h] Spirit Aerosystems, Wichita, KS, USA
[i] Hexcel Corporation, Dublin, CA, USA
[j] Northrop Grumman, Clearfield, UT, USA
[k] University of Warwick, Coventry, UK
[l] Airbus Operations, Getafe, Spain

ARTICLE INFO

Keywords:
A. Prepreg
B. Adhesion
E. Automated fibre placement

ABSTRACT

This study reports the findings from a round robin test exercise investigating ASTM D8336, a method for the measurement of tack of prepregs applicable to automated material placement technologies. Twelve participants tested two prepreg materials in a core study of three temperature and deposition/peel rate combinations, and five participants conducted an extended study based on a test matrix of four temperatures and four rates. In the core study, the repeatability coefficient of variation of obtained tack values ranged from 6.06 % to 15.08 %, while the reproducibility coefficient of variation ranged from 12.37 % to 32.37 %. The probable dominant causes for the observed variability are the limited accuracy of control and measurement of specimen temperature, of compaction force, and of humidity. Using time–temperature superposition, tack mastercurves were constructed from the extended study, identifying values of peak tack, the rate at peak tack and the width of the tack mastercurve, as well as the 95 % confidence and prediction bands. Across all materials and participants, 93.3 % of the repeats from the core study lay within the 95 % prediction bands, supporting the view that a mastercurve provides a greater degree of useful information at a comparable effort for data acquisition.

1. Introduction

Large composite components with low levels of geometrical complexity, in particular for aerospace applications, are frequently manufactured employing processes such as Automated Tape Laying (ATL) or Automated Fibre Placement (AFP). In these processes, computer-controlled machines lay up unidirectional continuous prepreg, typically from carbon fibre and partially cured (B-staged) thermoset resin, at defined orientations to form a laminate. The laminate is then cured in an autoclave at high pressure and at elevated temperature. This process chain allows structural components to be manufactured at the high quality required for aerospace applications, i.e. at high fibre volume fraction (~60 %) and low void content (<2 %) [1].

A potential issue during ATL or AFP processes is wrinkling or bridging of prepreg layers in the presence of local compressive or tensile forces, which would result in the formation of defects in the laminate

* Corresponding author.
E-mail address: davide.defocatiis@nottingham.ac.uk (D.S.A. De Focatiis).

https://doi.org/10.1016/j.compositesa.2024.108561
Received 3 October 2024; Accepted 25 October 2024
Available online 1 November 2024

[2]. This effect is related to insufficient levels of adhesion (tack) between prepreg and tool surface and between the surfaces of prepreg layers. Another potential issue is the build-up of resin on application rollers, which is caused by too much adhesion between prepreg and the surface of the application roller and may cause interruptions of the lay-up process. Hence, the experimental characterisation of prepreg tack on different surfaces and at different parameters can help to predict the behaviour of a prepreg during processing and to optimise the process for maximum deposition efficiency and quality of outcome. Process optimisation can either rely on the direct assessment of experimental results to identify tack maxima as a function of different material and process/test parameters [3–6] or, on the utilization of tack data for AFP process models [7,8]. In any case, reproducible and thus comparable measurement results are mandatory for both basic research and industrial implementation alike.

A comprehensive review of methods for characterisation of tack was given by Budelmann et al. [9]. One of these methods, proposed by Crossley et al. [10], employs a continuous application-and-peel test to imitate the conditions in ATP/AFP processes. In this test, which relies on utilizing a specific test fixture on a universal testing machine, the prepreg is bonded to and immediately peeled from a substrate in a single continuous motion reflecting the lack of separation between bonding and peeling stages in industrial processes. Based on Crossley's original work and subsequent studies [11–14], a tack testing standard, ASTM D8336-21, was developed [15] with the aim of obtaining consistent tack data of industrial relevance.

A round-robin exercise was carried out between 2021 and 2023 involving 12 participants, in which two prepreg materials were tested in a range of conditions according to ASTM D8336-21. The objective of the round robin was to evaluate the repeatability and reproducibility of the test method. This article summarises the results of the round-robin study and further investigates the underlying causes of variability in the test results using previously obtained insights into the tack phenomenon. A series of recommendations are made on how to conduct tack tests so as to reduce the variability, and on how to select test conditions in order to maximise the information obtained from the testing.

2. Test method

2.1. Working principle

The method defined in ASTM D8336-21 can be used to quantify tack between one B-staged prepreg layer and a second B-staged prepreg layer which is bonded to a rigid substrate (ply-ply tack), or between a B-staged prepreg specimen and a rigid metal substrate which represents the surface of a tool used for component manufacture (ply-tool tack). As ply-tool tack forces are generally at a significantly lower level than ply-ply tack forces [12], trends within these data are difficult to identify clearly. Although information relating to ply-tool tack (behaviour of the first ply down) is valuable in a manufacturing scenario, the following detailed discussion will focus on experiments on ply-ply tack.

To prepare a specimen for a ply-ply tack test, a prepreg layer is placed on another prepreg layer bonded to a stiff steel substrate without application of any compaction force. The top surface of the top prepreg layer is covered with a protective film to prevent it from sticking to the peel roller during the test. The interface between the two prepreg layers is partially covered with a protective film, which divides the test into two phases: One where the prepreg layers are separated and one where adhesion can form between the layers. The lay-up is then fed through a test fixture, which is mounted on the base of a Universal Testing Machine, as shown in Fig. 1.

One end of the top prepreg layer is held by a material clamp which is attached to the crosshead and load cell of the testing machine. During a test, the crosshead moves vertically at constant speed, which translates into a horizontal movement of the specimen lay-up through the fixture. In the fixture, the top prepreg layer is pressed against the bottom prepreg layer (bonded to the substrate) at a force, F_c, applied to the compaction roller through the adjustable springs. Thus, a prepreg-prepreg bond is established at a set application rate. Simultaneously, the top prepreg layer is peeled from the bottom layer at a peel rate which

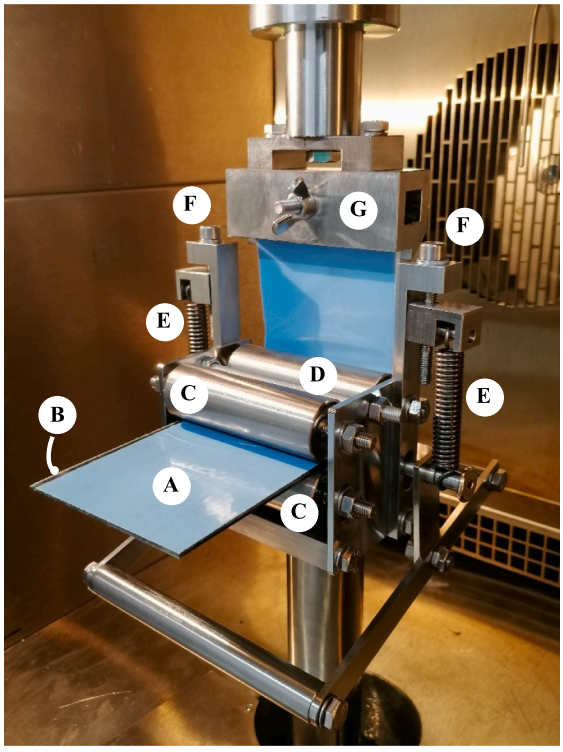


Fig. 1. Tack test fixture used in this study mounted on Universal Testing Machine and enclosed by environmental chamber. Details of the fixture design are summarised in ASTM D8336-21 [15].

is identical to the application rate.

Before a test can commence, a calibration curve is acquired to establish how many turns of the jacking screws loading the springs correspond to a given compaction force. For the calibration, the jacking screws are loosened. One end of a stiff L-shaped steel bracket is attached to the load cell of the test machine using the material clamp. The other end of the bracket is positioned on the loosely suspended compaction roller. Both jacking screws are tightened incrementally (by the same number of turns) such that the compaction roller applies a force to the load cell through the bracket, and the corresponding force readings are recorded. Using a calibration curve shown in Fig. 2, the load pressing the prepreg to the substrate can be adjusted (zero turns corresponds to the substrate with the prepreg lay-up just being in contact with the compaction roller and the peel roller).

During a tack test, force data is recorded as a function of the crosshead displacement. The example in Fig. 3 shows that two distinct phases can be identified in the recorded data: the force in the first phase is related to dissipative effects, such as friction in the system, when the specimen is pulled through the fixture without adhesion forming between the prepreg layers (Fig. 4(a)); the force in the second phase includes the peel force required to overcome adhesion as well as the aforementioned effects (Fig. 4(b)).

The peel force is determined from the difference between the average forces of the two phases. Here, the ranges of values considered for averaging in both phases relate to displacements from 10 mm to 35 mm (phase 1) and from 62.5 mm to 87.5 mm (phase 2), as suggested in ASTM 8336. In these ranges, force values are approximately constant and unaffected by irregularities at the start of the test and at the transition between the phases. Measuring the peel force during a test gives an indication of the strength of adhesion between the two prepreg layers at the given test conditions. The test fixture can be used at a range of crosshead displacement rates, corresponding to deposition and peel rates, as well as in an environmental chamber, allowing tack to be measured at different temperatures. If tack is to be tested at temperatures higher than ambient temperature, the target temperature is set on the control unit of the environmental chamber. After the chamber and the test fixture have attained the target temperature, the specimen lay-up is loaded. It is recommended to use a non-contact method (such as an infra-red thermometer) to measure the specimen surface temperature. The test can commence when the surface temperature is within $\pm 1\,^{\circ}\mathrm{C}$ of the target temperature.

2.2. Previous work

Following the approach outlined by Crossley et al. [11], which is also described in the Appendix of ASTM D8336-21 [15], it is possible to employ a matrix of tack tests at different temperatures and rates to construct a tack mastercurve for a given prepreg at a selected reference temperature using the process of time–temperature superposition (TTS). This requires knowledge of the relationship between time (or rate) and temperature for the prepreg, which is typically obtained from linear viscoelastic rheology on the neat resin used in the prepreg. The application of TTS to tack means that a tack force $F_t(T, r)$, measured at temperature T and rate r, is equal to a tack force $F_t(T_0, ra_T)$ at a reference temperature T_0 and a shifted rate $r_s = ra_T$, where a_T is the shift factor between T and T_0. This implies, for example, that conducting experiments at a low temperature and applying a shift factor to the feed rate allows to extend the range of rates to higher values not easily obtainable in experiments on a Universal Testing Machine. The shift factor-temperature relationship can be successfully modelled through the Williams-Landel-Ferry equation

$$\log a_T = \frac{-C_1 (T - T_0)}{C_2 + (T - T_0)} \tag{1}$$

where a_T is the shift factor at temperature T relative to the selected reference temperature T_0, and the constants, C_1 and C_2, depend on the resin properties.

In a previous study [12], tack was measured for a UD prepreg tape at different feed rates and temperatures to explore the viscoelastic response of the B-staged resin. Tack mastercurves were produced by shifting measured data to a reference temperature applying TTS (Fig. 5). The dependence of tack on the shifted feed rate was successfully approximated by 3-parameter Gaussian curves of the type

$$F_t(T_0, r_s) = F_{tmax} \exp\left(- \left(\frac{\log(r_s) - \log(r_{smax})}{w} \right)^2 \right) \tag{2}$$

The maximum tack value, F_{tmax}, the corresponding rate, r_{smax}, and the width of the curve, w, derived from fitted Gaussian curves, can be used as quantitative descriptors of tack behaviour. The choice of a Gaussian curve to fit the data is empirical, but has shown itself to apply well to many different prepreg systems. The shape of the curves is determined by increasing cohesion within the resin with increasing feed rates

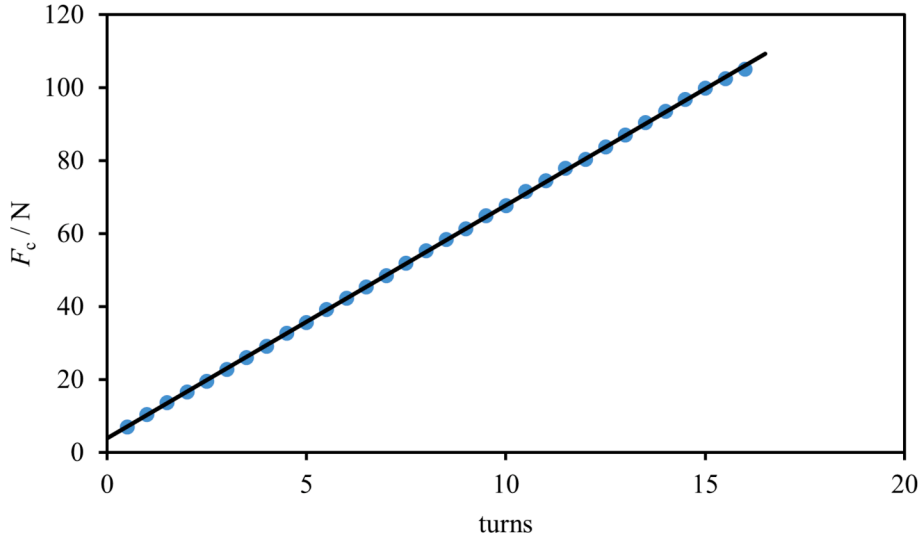


Fig. 2. Compaction force, F_c, as a function of the number of turns of the jacking screws (for the fixture shown in Fig. 1). The intercept is greater than zero due to the initial tension, which must be overcome in order for the spring coils to start to open. If springs with a different spring constant are used, the calibration curve will be different.

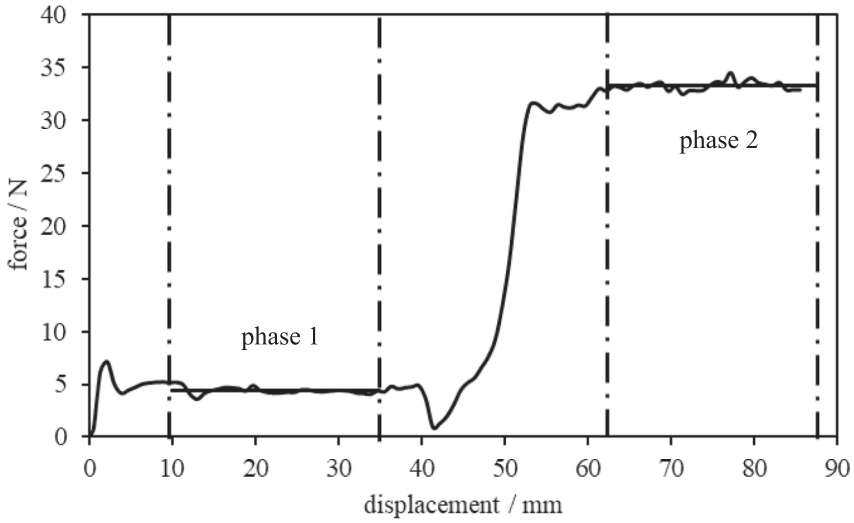


Fig. 3. Example of a typical force–displacement data recorded during a tack test on an aerospace-grade prepreg. The peel force as a measure for tack is calculated from the difference between the average values of the indicated force levels during phase 1 and phase 2.

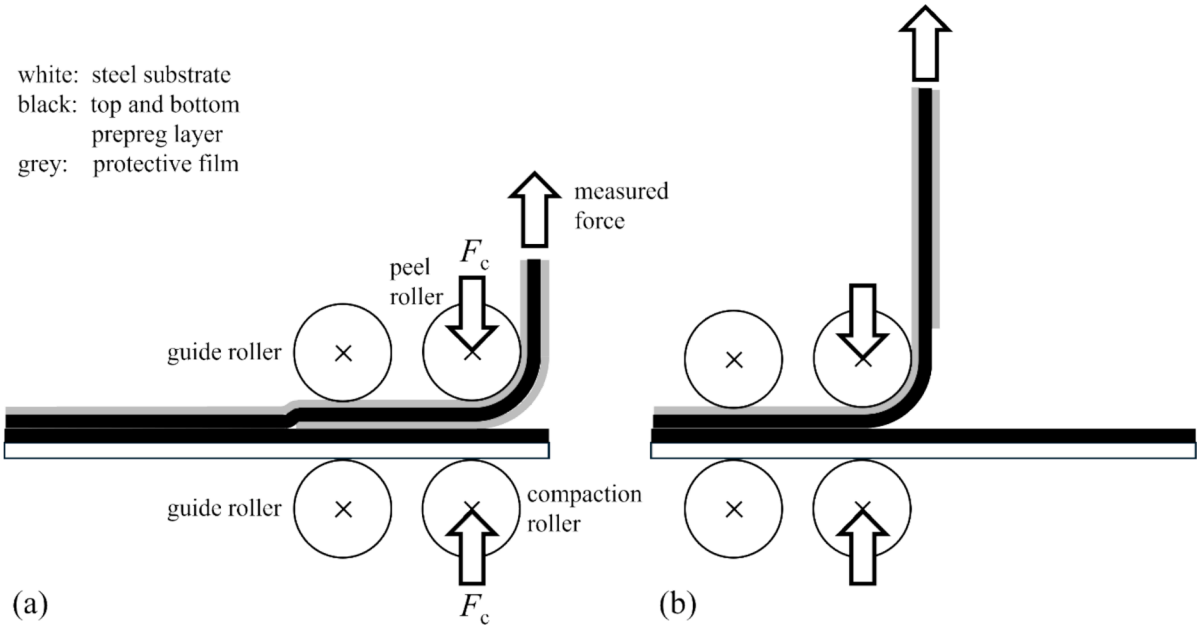


Fig. 4. Schematic of the tack test. (a) Phase 1: prepreg layers are separated by protective film; no adhesion forming. (b) Phase 2: contact between prepreg layers; adhesion can form.

(which dominates at lower rates) and decreasing adhesion between resin and substrate (dominating at higher rates). Both effects depend on the timescale of molecular interactions at the interface between the prepreg layers relative to the timescale of the compaction/separation process.

Different behaviour may be observed for different material pairings at the contact interface (e.g. ply-ply vs ply-tool), as different molecular interactions may occur. The amount of resin at the interface determines the effective contact area [14] and can also have an effect on measured tack.

For specimens stored at room temperature (out of the freezer), maximum prepreg-prepreg tack and the feed rate at maximum tack decrease with increasing out-time [13]. On the other hand, maximum prepreg-prepreg tack and the feed rate at the maximum increase for specimens conditioned at increased levels of relative humidity. In both cases, the molecular mobility in the resin changes: it decreases with increasing out-time, as the degree of cure increases, and it increases with increasing moisture uptake due to plasticization of the resin.

In addition, prepreg-prepreg tack increases with increasing compaction force applied during a test and converges to a limit value. If a compliant peel roller is used, the convergence is faster than for a stiff peel roller, as the true contact area and the duration of compression of the prepreg surfaces are increased. It was also found that measured prepreg-prepreg tack depends on the inter-ply angle, but this effect is not yet fully understood.

3. Round-robin exercise

Two unidirectional (UD) aerospace-grade carbon fibre/epoxy

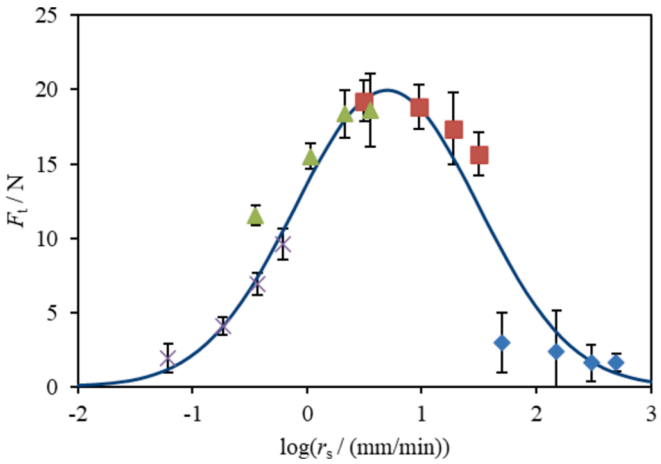


Fig. 5. Example prepreg data from previous work [12] showing the measured tack force, F_t, as a function of the shifted rate, r_s, at a reference temperature, T_0 = 20 °C. Error bars indicate standard deviations within each experiment. Different markers indicate data acquired at different test temperatures, T. A Gaussian fit curve described by Eq. (2) is also shown. Coefficient of determination, R^2 = 0.901.

prepregs, referred to as Materials A and B in the following, were provided for this study, one from Hexcel Corporation and one from Toray Composite Materials America. Both materials were 180 °C cure toughened epoxy resin prepregs. The properties were unknown to the participants carrying out the experiments for characterisation. The prepreg was cut to the dimensions specified in the standard (215 mm × 75 mm for the peeled layer and 140 mm × 80 mm for the layer bonded to the substrate by double-sided adhesive tape) and supplied in kit form to the participants. To prevent the degree of cure of the resin systems from changing, the prepreg was shipped on dry ice and stored in freezers. Before being tested, the specimens were allowed to thaw to room temperature.

A total of 12 laboratories participated in this exercise (Table 1). For the purpose of this study, test fixtures complying with ASTM D8336-21 were manufactured at the University of Nottingham. These were provided on loan to participants who did not already have ASTM-compliant fixtures on site. Four of the participating laboratories had prior experience with the test method. Test data submitted by all participants was anonymised for this report.

The following test series were carried out for each of the two materials:

- Core test series: Five repeats at each of the three combinations of temperature and rate listed in Table 2 (11 participants for each material, as both material suppliers named in Table 1 supplied one material each and only tested their own material). These test conditions were selected based on preliminary experiments used to identify combinations of temperature and rate appropriate to these materials with respect to tack.
- Extended test series: A matrix of four different rates, i.e. 50 mm/min, 150 mm/min, 300 mm/min and 500 mm/min, and four different temperatures. i.e. 20 °C, 30 °C, 40 °C and 50 °C (5 participants for each material). This 4 × 4 matrix of test conditions was intended to allow the viscoelastic response of the prepregs to be explored.

Table 1
Participants of the round-robin exercise.

Participant	Sector
Airbus Operations, Getafe, Spain	Aerospace
The Boeing Company, Seattle, WA, USA*	Aerospace
Northrop Grumman, Clearfield, UT, USA	Aerospace
Spirit Aerosystems, Wichita, KS, USA	Aerospace
Vestas Technology, Isle of Wight, UK*	Wind energy
Hexcel Corporation, Dublin, CA, USA	Material supplier
Toray Composite Materials America, WA, USA*	Material supplier
Clausthal University of Technology, Stade, Germany	Research
National Composites Centre, Bristol, UK	Research
University of Nottingham, UK*	Research
National Physical Laboratory, Teddington, UK	Research
University of Warwick, UK	Research

* These participants had experience of tack testing using the standard test method prior to the round robin

Table 2
Combinations of temperature, *T*, and application-and-peel rate (which corresponds to the crosshead speed), *r*, used in the test series with five repeats at three different conditions.

Material	Test Condition 1		Test Condition 2		Test Condition 3	
	T/°C	*r*/(mm/min)	*T*/°C	*r*/(mm/min)	*T*/°C	*r*/(mm/min)
A	30	150	40	50	50	300
B	30	50	40	150	50	500

For all tests in this study, a stiff peel roller was used. The compaction force was set to 100 N (at a specimen width of 75 mm) based on a previous study which indicated that tack was relatively insensitive to the compaction force at this level [12]. This level of compaction force is expected to produce a pressure in the same order as that of typical AFP processes. In the ply-ply tack tests carried out here, both prepreg plies were aligned, such that the fibre direction was parallel with the long axis of the substrate.

The participants were asked to follow the instructions summarised in ASTM D8336-21 when carrying out the tests. To help with data collection and evaluation, Excel workbooks were prepared for reporting the data specified in the standard (one sheet for each individual test). All participants were asked to populate these workbooks with the data they acquired.

In preliminary tests on the prepregs studied here, it was observed that some of the backing paper, which is attached to the prepreg for protection and also prevents it from sticking to the peel roller of the test fixture, was relatively thick compared to the prepreg thickness. The paper buckled or wrinkled when the specimen was bent around the peel roller, an effect which had not been observed in previous experiments with different backing papers. To avoid any effect on the test results (see Appendix A for an example), it was suggested to break the bond between the backing paper and the prepreg and re-apply the backing paper immediately prior to tack testing. Covering the prepreg surface with the backing paper without it being bonded allows sliding and avoids buckling. Removing the backing paper is not an option as this would allow the prepreg to adhere to the peel roller during a test.

4. Results

4.1. Core tests

The submitted data obtained in the 5 × 3 core test series are summarised in Table 3. A full listing of all data acquired by the participants can be found in the ASTM Research Report D30-2001 [16].

4.2. Extended tests

To create the mastercurves in this study (as indicated in Section 2.2), the prepreg manufacturers provided the necessary shift factor information for the purpose of evaluation of this aspect of the test method. The shift factors were obtained from rheometry of prepreg constituent neat resin as described in ASTM D8336-21, Appendix X2 [15]. The parameters in Eq. (1) provided by the manufacturers were C_1 = 14.9; C_2 = 107.5 °C at T_0 = 25 °C for material A, and C_1 = 6.7; C_2 = 51.4 °C at T_0 = 30 °C for material B. Making use of the corresponding shift factors, the

Table 3
Summary of tack results for Materials A and B (at the conditions detailed in Table 2) obtained in the core tests. Average values and standard deviations of the tack force, F_t, from five repeats are given for each of the eleven test series.

F_t/N					
Test Conditions A1	Test Conditions A2	Test Conditions A3	Test Conditions B1	Test Conditions B2	Test Conditions B3
25.4 ± 3.1	28.6 ± 2.2	23.6 ± 3.5	21.9 ± 3.3*	10.9 ± 2.2*	28.5 ± 2.7*
22.5 ± 1.9	31.9 ± 1.6	27.7 ± 4.8	29.1 ± 1.6	11.1 ± 0.4	10.2 ± 1.0
13.3 ± 1.8*	30.2 ± 3.5*	34.4 ± 2.1*	22.4 ± 2.7	12.4 ± 1.4	13.0 ± 1.2
26.5 ± 1.3	31.9 ± 2.3	29.6 ± 0.8	33.6 ± 3.2	8.9 ± 2.0	8.9 ± 0.8
27.5 ± 3.5	27.3 ± 1.4	24.6 ± 2.3	6.0 ± 1.6*	20.9 ± 2.1*	16.8 ± 1.5*
12.1 ± 1.3*	19.7 ± 2.6*	24.9 ± 1.8*	34.1 ± 4.5	7.6 ± 1.2	8.5 ± 0.9
26.5 ± 1.7	30.3 ± 1.8	27.5 ± 2.5	30.3 ± 0.7	11.1 ± 0.4	8.9 ± 2.2
18.0 ± 1.0	29.3 ± 1.3	23.3 ± 1.1	27.1 ± 2.3	5.9 ± 1.9	11.4 ± 1.6
19.0 ± 1.6	23.9 ± 2.0	23.4 ± 2.0	35.5 ± 1.3	4.7 ± 0.7	3.5 ± 0.4+
30.4 ± 1.1	32.9 ± 1.9	25.3 ± 1.2	31.7 ± 1.4	8.0 ± 0.5	8.8 ± 1.9
26.3 ± 1.3	24.1 ± 0.4	25.0 ± 1.9	28.8 ± 1.6	7.7 ± 1.7	5.4 ± 0.6

* Data series with potentially inconsistent data based on the descriptors defined in ASTM E691.
+ Data series with three repeats only.

test rates from the 4 × 4 test matrices of submitted test data were shifted to a reference temperature of T_0 = 20 °C in order to construct tack mastercurves. The results from all five participants doing the extended test series are shown in Fig. 6 for the two materials. A region of peak tack can be observed in all datasets, falling at higher shifted rates due to adhesive failure, and at lower shifted rates due to cohesive failure in the prepreg-prepreg bond.

Gaussian curves defined by Eq. (2) were fitted to each dataset, obtaining values for the peak tack, F_{tmax}, the shifted rate at peak tack, r_{smax}, and the width of the bell curve, w. In practical application, this

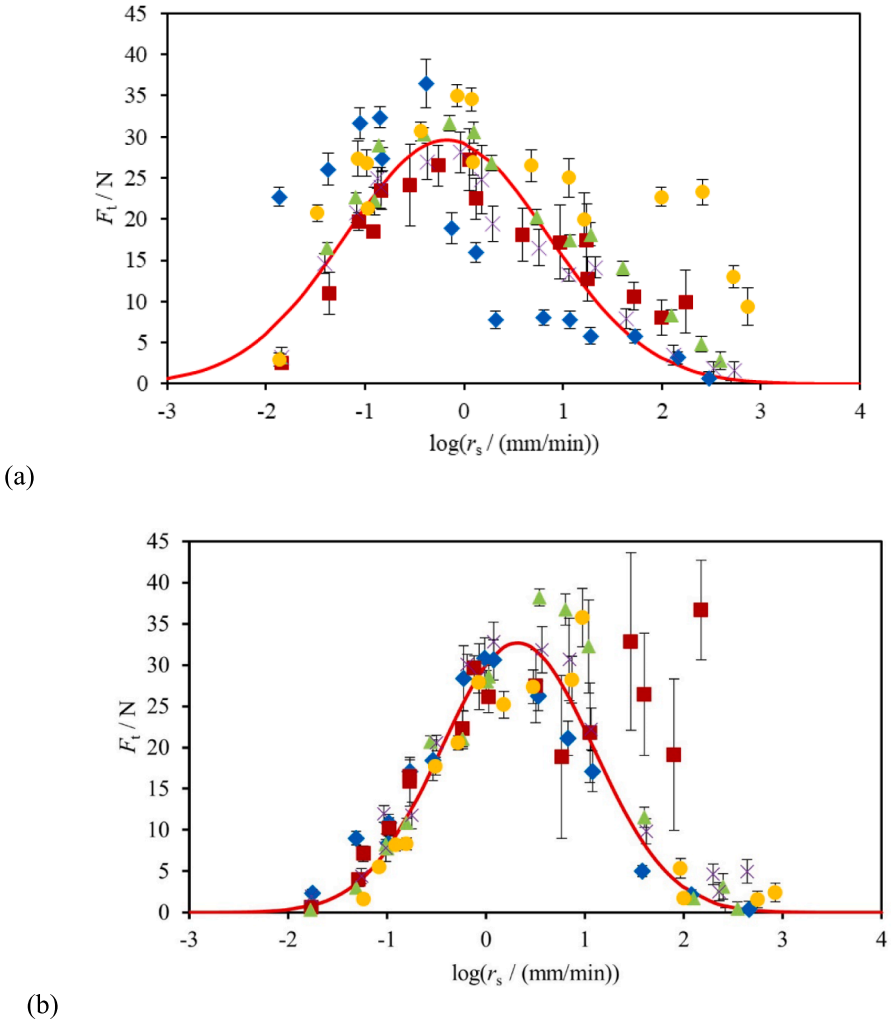


Fig. 6. All data obtained in the 4 × 4 test matrix shifted to T_0 = 20 °C. Error bars indicate standard deviations within each experiment. Gaussian curves described by Eq. (2) were fitted to combined data and are also indicated. (a) Material A; F_{tmax} = 29.6 N; r_{smax} = 0.68 mm/min; w = 1.45; coefficient of determination, R^2 = 0.700. (b) Material B; F_{tmax} = 32.6 N; r_{smax} = 2.09 mm/min; w = 1.09; R^2 = 0.919. Different markers indicate data obtained by different participants.

fitting process allows the feed rate at maximum tack at a given temperature to be estimated. For each fit, the error between the measured and shifted data and the Gaussian curve was weighted by the inverse of the variance recorded for each data point, to better reflect the uncertainty arising from each individual measurement. A summary of the parameters describing the mastercurves obtained by each participant is shown in Table 4. Mastercurve functions were also fitted to the combined datasets from all 5 participants as shown in Fig. 6.

Table 5 shows averages and standard deviations of the parameters determined in Table 4, indicating the level of agreement between the different laboratories.

5. Discussion

5.1. Repeatability and reproducibility statistics

Following the guidelines detailed in ASTM E691 on how to run a round-robin study and how to evaluate the data [17], 12 data sets out of 66 from the core test series were identified as potentially inconsistent (see Table 3), because the values for the between-participant consistency or for the single participant consistency approached or exceeded critical values. Further details on the process of inconsistent data identification are given in Appendix B. As it was not possible to repeat the corresponding tests, these data sets were not considered in the subsequent analysis. Similarly, one data set with less than five repeats was also not considered. For both materials and for all test conditions, a minimum of 8 data sets were considered in each analysis.

The Repeatability Coefficient of Variation indicates the variability in test data for the same material, obtained using the same test method and the same experimental set-up by the same operator within one test series. Values of $s_r/\bar{x}$ range from 6.06 % to 15.08 %. On the other hand, the Reproducibility Coefficient of Variation indicates the variability in test data for the same material, obtained using the same test method, but in a different laboratory by a different operator on a different day, i.e. between different test series. Values of $s_R/\bar{x}$ range from 12.37 % to 32.37 %. Table 6 lists values of the Repeatability Coefficient of Variation, $s_r/\bar{x}$, and the Reproducibility Coefficient of Variation $s_R/\bar{x}$ for all the data at all test conditions. Detailed definitions of these quantities [17] are summarised for reference in Appendix B.

The values of the Repeatability Coefficient of Variation are consistent with previous observations for a single laboratory (on an aerospace-grade prepreg different from the ones tested here), where the coefficient of variation had been determined to be 11 % (5 repeat tests) [15].

5.2. Potential causes of variability

Potential sources of scatter in the data related to the measurement of tack, which may affect both the within-lab repeatability and between-lab reproducibility, are as follows:

Table 4
Parameters of fitted Gaussians representing tack mastercurves for all participants' data for both tested materials at a reference temperature $T_0 = 20$ °C: Maximum tack, F_{tmax}, feed rate at maximum tack, r_{smax}, and a measure for width of the curves, w. Coefficients of determination, R^2, are also given.

Material	F_{tmax}/N	r_{smax}/(mm/min)	w	R^2
A	28.9	0.07	1.75	0.924
A	29.9	1.07	1.33	0.895
A	32.9	0.87	1.49	0.885
A	27.7	0.66	1.52	0.933
A	34.3	3.43	2.20	0.748
B	29.4	1.33	1.15	0.958
B	34.4	3.12	1.30	0.924
B	36.2	2.52	1.12	0.962
B	34.1	2.23	1.22	0.970
B	31.0	2.36	1.05	0.953

Table 5
Averages of fitted Gaussian parameters for both tested materials at a reference temperature $T_0 = 20$ °C: Maximum tack, F_{tmax}, feed rate at maximum tack, r_{smax}, and a measure for width of the curves, w. The arithmetic mean and standard deviation were used for F_{tmax} and w, and the geometric mean and standard deviation for r_{smax}. Coefficients of determination, R^2, refer to the fits to combined data in Fig. 6.

Material	F_{tmax}/N	r_{smax}/(mm/min)	w	R^2
A	30.7 ± 2.8	0.68 ± 4.13	1.66 ± 0.34	0.700
B	33.0 ± 2.8	2.23 ± 1.37	1.17 ± 0.10	0.919

Table 6
Round-robin statistics. Here, T and r indicate the target values of the test temperature and the application-and-peel rate. s_r, s_R and $\bar{x}$ are the repeatability standard deviation, the reproducibility standard deviation and the average of averages of the tack force, F_t.

Material	T/°C	r/(mm/min)	$\bar{x}$/N	Coefficients of variation/%	
				Within-lab repeatability	Between-lab reproducibility
				$s_r/\bar{x}$	$s_R/\bar{x}$
A	30	150	24.7	8.15	17.96
A	40	50	28.9	6.06	12.63
A	50	300	25.5	9.87	12.37
B	30	50	30.3	7.93	15.13
B	40	150	8.6	15.08	32.37
B	50	500	9.4	14.57	27.13

- Previous experiments have shown that, for some prepregs, the amount of resin and its distribution can vary between surfaces [12]. This can result in different tack being measured for different surface pairings. For this study, instructions provided to participants stated clearly which surface pairings to test. Hence, the probability of inconsistencies in results because of testing of incorrect surface pairings seems low.
- Contamination of the specimen surfaces or condensation on the surfaces (which may occur if specimens at freezer temperature are exposed to humidity levels typical to ambient conditions) can have an effect on measured tack. All operators carrying out the test series were experienced with prepreg lay-up, hence the probability for unsuitable treatment is low.
- The application/peel rate, i.e., the crosshead speed, which determines the viscoelastic response of the prepreg, can be set on universal testing machines, typically with high accuracy. However, at the beginning of a test and/or at the transition between the two phases of the test (see Fig. 3), there may be slack in the top layer. As the layer is straightened, the effective peel rate may vary momentarily (at constant crosshead speed). This may result in peaks occurring in the recorded force. These peaks were excluded from the range of force values where averaging is applied. Hence, the probability of measured tack being affected by inaccurate effective rates is low.
- The true temperature at the interface between the prepreg layers can have a significant effect on measured tack, again because of the viscoelasticity of the prepreg. An uncertainty in temperature of ±1 °C is considered acceptable. Most temperature readings reported by the participants were within ±1 °C of the target temperature, and the rest were generally within ±2 °C. However, depending on the method for controlling and/or monitoring the temperature, the measured temperature at the interface may differ from the true temperature by a few °C. It is recommended in ASTM D8336-21 to measure the temperature on the visible specimen surface using a non-contact thermometer once the specimen lay-up is loaded in the test fixture and the environmental chamber is heated. Here, some participants measured the temperature on the specimen surface

using an infrared thermometer or a thermocouple, some measured the temperature underneath the specimen, and some measured the temperature in the environmental chamber. Potential inconsistencies in temperature control/monitoring have a high probability to affect the measured tack. For the data acquired here, the maximum change in tack force with temperature is approximately 3 N/°C at any feed rate.

- The effect of out-time, i.e. prolonged exposure to ambient temperatures, on the properties of prepregs (specifically, the degree of cure) is well known [13]. Specimen kits were shipped on dry ice as is industry standard to minimise this effect. All participants were asked to store the specimens in a freezer once received and only take the specimens out for testing. The probability that specimens could have been exposed to ambient temperature for a significant amount of time (e.g. during shipping) is low.
- While the humidity could not be controlled during the tack tests, all participants were asked to record the relative humidity (*RH*). The values of *RH* reported by different participants vary between approximately 5 % and approximately 60 %. Here, some participants measured *RH* in the environmental chamber containing the test fixture and the specimens, while others measured it in the laboratory outside the environmental chamber. Through plasticization of the resin with increasing moisture content [12], varying levels of *RH* may have a significant effect on tack measured at a given temperature and feed rate. In a previous study, prepreg-prepreg tack was measured for specimens conditioned at different *RH* at a range of temperatures and feed rates, the measured data were shifted to a reference temperature, and Gaussian curves were fitted to the shifted data. It was found that an increase in *RH* from 33 % to 59 % resulted in an increase in maximum tack of 25 %, while the feed rate at maximum tack increases by as much as a factor of two.
- The effect of compaction force on tack has previously been found to be limited [12], especially for larger compaction forces such as 100 N (across a 75 mm specimen width) as used here, where tack levels converge. However, if the compaction force is lower than the target, the formation of adhesion between the prepreg layers may be affected, and the measured tack may be significantly lower than expected for the target value of the compaction force [12]. Prepreg-prepreg tack measured at a single temperature and feed rate was found to decrease by 33 % for a reduction in compaction force from 100 N to 50 N and to increase by 16 % for an increase in compaction force from 100 N to 150 N. This means that applying the calibration procedure and/or the adjustment of the compaction force incorrectly could have a significant effect on results, particularly if the true compaction force is too low. For the compaction springs in the test fixtures used by most participants in this study, the spring constant is 3.22 N/mm. This implies that thickness variations between specimens in a test series or between test series (less than 1 mm) will only have a small effect on the true compaction force. On the other hand, it is to be considered that both prepreg plies should be laid up on the substrate without any compaction force applied, such that compaction of the lay-up is only applied through the spring-loaded roller. If a compaction force is incorrectly applied before the lay-up is placed in the test fixture, this may influence the tack behaviour.
- The angle between the fibres in the prepreg layers can have some effect on results in ply-ply tests. In a previous study, tack measured at an angle of 45° between the plies was approximately 20 % higher than tack for plies aligned in parallel [12]. In this round-robin study, all prepreg specimens were pre-cut such that the probability for significant misalignment between the plies was very low. Any effect on measured tack is unlikely.

A general issue with experimental material characterisation is the familiarisation of operators with setting up and running the tests [17]. As the test method used in this study was developed relatively recently, only 4 participants had previous experience with the method (see Table 1). It is thought that the number of data sets identified as potentially inconsistent and the scatter in the remaining data sets both could be reduced through better familiarisation of all participants with conducting the experiments.

A revised version of the standard was published in 2024 [18], in which the results of the round-robin were included as part of a precision statement. To improve the repeatability and reproducibility of tack tests, and to minimise the effect of the factors identified above, the more significant amendments made to the standard were:

- The terminology was clarified to make the instructions easier to follow.
- The calibration procedure for the compaction springs was revised to minimise variability due to uncertain compaction force.
- Specific instructions to break bond between prepreg and backing paper were added to avoid any effect of paper buckling on the test results.
- More detailed instructions on reporting of the temperature measurement method were added to provide greater traceability.

It should be noted that even after reducing the variability of the test method, there will still be some variability in the test data arising from specimen variability.

5.3. Comparison of core and extended test results

The availability of 4 × 4 test matrices (extended test series) in addition to 5 repeat tests at 3 conditions (core test series) for 5 participants allows an evaluation and comparison of the level of information that can be gained by carrying out repeat tests at the same condition and by constructing a corresponding tack mastercurve. In order to compare results from the two test series, further statistical analyses are carried out on the 4 × 4 test matrices to obtain the 95 % confidence interval and 95 % prediction bands. For each participant who carried out both core and extended tests, and for each material, the number of tack measurements from the repeats of the core tests that lie within the 95 % prediction band derived from the extended test series was determined. One graphical example of this exercise is shown in Fig. 7.

Out of 150 core tests considered in this comparison, a total of 140 (corresponding to 93.3 %) lie within the 95 % prediction bands. The outliers are present only in Material B, and 5 of them are from the same set of 5 repeats at the same condition. This clustering suggests that the deviation is caused by inaccuracy of the particular mastercurve rather than the core tests. It is also a reflection of the empirical nature of the mastercurve function, which may be more or less applicable for different material systems. Nevertheless, the fact that 93.3 % of the core tests lie within the 95 % prediction bands provides reassurance that the process of carrying out 4 × 4 test matrices to construct mastercurves can be expected to provide at least a similar level of information as five repeats at three conditions, with the additional benefit of providing this level of information across the rate and temperature spectrum. It is also to be considered that tests were repeated five times at three conditions (rate and temperature pairs) in the core tests, whereas a single test was carried out at 16 distinct conditions in the extended test series. Thus, both test series require similar levels of operator effort, but the evidence suggests that more useful information may be obtained by carrying out a 4 × 4 test matrix and constructing a tack mastercurve if TTS information is available.

6. Conclusions

A 12-participant round robin test exercise was carried out to evaluate the repeatability and reproducibility of ASTM D8336-21, a method for the measurement of tack of prepregs. A core study involved two materials tested at three conditions of temperature and rate in ply-ply configuration with five repeats. An extended study was also conducted

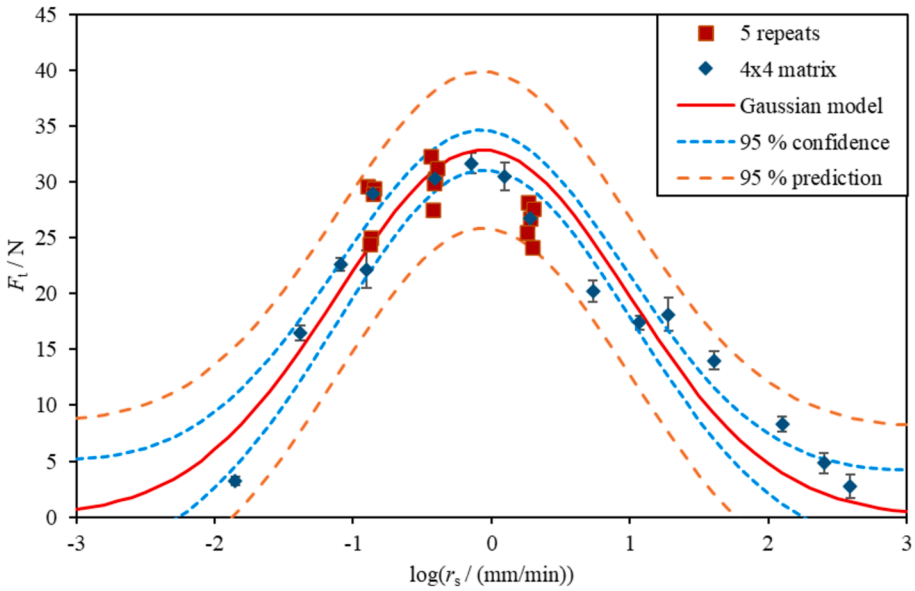


Fig. 7. Example of experimental data for Material A (core tests and extended tests) with 95 % confidence bands and 95 % prediction bands. For this example, all repeats lie within the prediction bands, although one point is very close to the lower band.

by five participants on a 4 × 4 test matrix of temperature and rate with a single test at each condition to construct a mastercurve for the ply-ply configuration.

The core study found that the repeatability coefficient of variation, indicating the variability in test data for the same material, ranged from 6.06 % to 15.08 %, consistent with prior observations at a single laboratory. The reproducibility coefficient of variation, indicating the operator and laboratory variation, ranged from 12.37 % to 32.37 %. A qualitative analysis of the factors affecting variability which arise from the test method was carried out. The three probable dominant causes for the variation are: (1) the effect of test temperature, and particularly the manner in which the specimen temperature is measured; (2) the effect of compaction force, and particularly the issues surrounding the application of the correct compaction using the spring-loaded roller; (3) the effect of humidity, an aspect that could not be controlled during the round robin study. Note that 13 out of 66 data sets had to be excluded from the analysis, either due to a lack of consistency based on ASTM E691′s recommendations on evaluating results from a round robin study, or because the number of repeat tests deviated from the requirements. Prior experience with the test method would have likely increased the number of data sets that could be used in the analysis.

The extended study enabled the construction of tack mastercurves using time–temperature superposition. From these, the peak tack, the rate at peak tack and the width of the tack mastercurve could be obtained, providing useful data in the process of maximising tack for specific automated manufacturing processes. The data was also used to determine 95 % confidence intervals and 95 % prediction bands. A comparison of the data from the core study and the data from the extended study for each material and participant showed that 93.3 % of the measurements from the core study lay between the 95 % prediction bands obtained from the mastercurve. Since the two studies involve 15 tests for each material for the core and 16 tests for the extended, the level of operator effort and material required is similar. It is therefore recommended that, where time–temperature shifting information is available, a test matrix of rates and temperatures is carried out to construct a mastercurve, as this will provide the user with a greater insight into the prepreg tack behaviour than an equivalent number of repeats.

CRediT authorship contribution statement

A.M. Joesbury: Data curation, Investigation, Project administration, Writing - original draft, Writing - review & editing. **A. Endruweit:** Conceptualization, Investigation, Formal analysis, Methodology, Visualization, Writing - original draft, Writing - review & editing. **D. Budelmann:** Investigation, Writing - review & editing. **S.P. Giannis:** Investigation, Writing - review & editing. **D.S.A. De Focatiis:** Conceptualization, Investigation, Formal analysis, Methodology, Project administration, Visualization, Writing - original draft, Writing - review & editing. **D.B. Call:** Conceptualization, Investigation, Methodology. **C. Chan:** Investigation. **G.Y.H. Choong:** Investigation. **R.A. Clark:** Investigation. **J.A. Collins:** Investigation. **D.T. Fishpool:** Investigation. **J.M. Garber:** Investigation. **S. Ghose:** Conceptualization, Investigation, Methodology. **J. Golding:** Investigation. **S.T. Good:** Investigation. **B.R. Jones:** Investigation. **D. Meiners:** Investigation. **O. Niitsoo:** Investigation. **C.T. Palmer:** Investigation. **C.C. Qian:** Investigation. **A.W. Rivas:** Investigation. **G. Santacruz Rodriguez:** Investigation. **R.E. Stillwell:** Investigation. **J.D.S. Vincent:** Investigation. **H. Yuan:** Investigation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

This work was supported by the Engineering and Physical Sciences Research Council, as part of the EPSRC Future Composites Manufacturing Research Hub Grant Number EP/P006701/1, and of the EPSRC Impact Acceleration Account Grant number EP/X525765/1.

D. Budelmann and D. Meiners gratefully acknowledge financial support by the German Research Foundation (Deutsche Forschungsgemeinschaft – DFG) as part of project number 458900231.

S.P. Giannis and J.D. Vincent gratefully acknowledge financial support by the UK's Department for Science Innovation and Technology through the National Measurement System Programme as part of project number ID45.

C.C. Qian and H. Yuan would like to acknowledge financial support by the China Scholarship Council (CSC), and technical support by Paul

Hadlum at the University of Warwick.

Appendix A

An example of the effect of stiff backing paper on the recorded force–displacement data is shown in Fig. A.1. Both curves were recorded for the same material, at the same test conditions. The peaks in the data for prepreg with attached backing paper are a result of varying bending stiffness due to buckling/wrinkling of the paper. Note that the minima in the blue curve coincide with the orange curve, indicating that they are a reflection of the true tack behaviour.

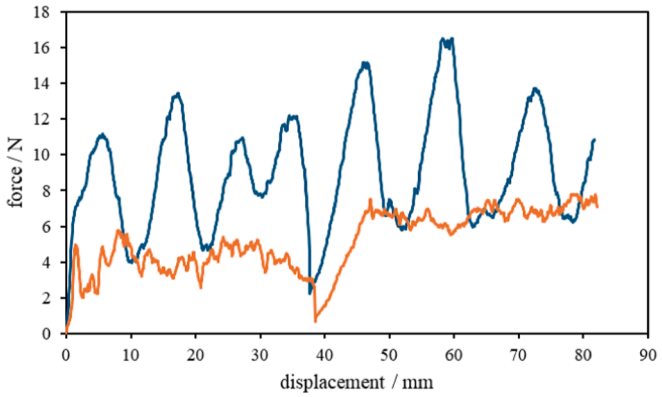


Fig. A.1. Example force-displacement data affected by buckling of stiff backing paper attached to the prepreg (blue line); data for the same prepreg at the same test conditions with the backing paper peeled off and placed back in the same position (orange line).

Appendix B

The statistical evaluation of data (for a given material and test series) followed the method described in ASTM E691 [16]. For the core tests, the number of participants was $p = 11$, the prescribed number of repeats in a test series was $n = 5$.

For each individual participant, the average value of the tack force, F_t, is

$$\overline{x} = \sum_{i=1}^{n} \frac{x_i}{n}, \tag{A.1}$$

where x_i are the n measured values of F_t. The standard deviation of F_t is

$$s = \sqrt{\sum_{i=1}^{n} \frac{(x_i - \overline{x})^2}{n-1}}. \tag{A.2}$$

Considering the data submitted by all p participants, the average of participants' average tack values is

$$\overline{x}' = \sum_{j=1}^{p} \frac{\overline{x}_j}{p}. \tag{A.3}$$

The deviation of each participant's average from the average of averages is

$$d = \overline{x} - \overline{x}'. \tag{A.4}$$

The standard deviation of participants' averages is

$$s_{\overline{x}} = \sqrt{\sum_{j=1}^{p} \frac{d_j^2}{p-1}}. \tag{A.5}$$

The precision of the test method is characterised in terms of the repeatability standard deviation,

$$s_r = \sqrt{\sum_{j=1}^{p} \frac{s_j^2}{p}}, \tag{A.6}$$

and the reproducibility standard deviation

$$s_R = \max\,(s_r, s_R^*), \tag{A.7}$$

where

$$s_R^* = \sqrt{s_{\bar{x}}^2 + s_r^2 \frac{n-1}{n}}. \quad (A.8)$$

The consistency of data submitted by a participant with the data submitted by others is assessed based on the between-participant consistency,

$$h = \frac{d}{s_{\bar{x}}}, \quad (A.9)$$

which indicates how the average of one specific participant's data compares with the average of the other participants, and the single-participant consistency,

$$k = \frac{s}{s_r}, \quad (A.10)$$

which indicates how the variability of one specific participant's data compares with all of the participants combined. For the values of p and n applicable here (i.e. 11 and 5, respectively), critical values of h and k at a significance level of 0.5 % are $h_c = 2.34$ and $k_c = 1.83$. According to ASTM E691, data approaching or exceeding the critical values indicates that the data may be inconsistent. It is also suggested that values of h and/or k differing significantly from those for other data sets could indicate inconsistency. Ideally, these experiments are repeated, and a new statistical evaluation of the updated data is carried out.

Data availability

The data is all available as part of an ASTM report, and will be made available on request.

References

[1] Naresh K, Khan KA, Umer R, Cantwell WJ. The use of X-ray computed tomography for design and process modeling of aerospace composites: A review. Mater Des 2020;190:108553.

[2] Bakhshi N, Hojjati M. An experimental and simulative study on the defects appeared during tow steering in automated fiber placement. Compo Part A - Appl Sci Manuf 2018;113:122–31.

[3] Chakraborty D, Kodagali K, Kattil SR, Miller D, Sockalingam S, Sutton MA. A novel experimental technique to determine the tack development during automated placement of uncured thermoset carbon/epoxy tows. Manuf Lett 2024;40:109–12.

[4] Belhaj M, Dogangeh M, Hojjati M. Experimental investigation of prepreg tackiness in automated fiber placement. Compos Struct 2021;262:113602.

[5] Pan H, Qu W, Yang D, Li J, Ke Y. Analysis and characterization of interlaminar tack for different prepreg materials during automated fiber placement. Polym Compos 2022;43:4737–48.

[6] Szpoganicz E, Demleitner M, Hübner F, Oh Y, Kweon Y, Lee H, et al. Phenolic prepregs for automated composites manufacturing – Correlation of rheological properties and environmental factors with prepreg tack. Compos Sci Technol 2022;218:109188.

[7] Budelmann D, Schmidt C, Steuernagel L, Meiners D. Adhesion-cohesion balance of prepreg tack in thermoset automated fiber placement. Part 2: Ply-ply cohesion through contact formation and autohesion. Compo Part C - Open Access 2023;12:100396.

[8] Wang Y, Mahapatra S, Belnoue JPH, Ivanov DS, Hallet SR. Understanding tack behaviour during prepreg-based composites' processing. Compos Part A - Appl Sci Manuf 2023;164:107284.

[9] Budelmann D, Schmidt C, Meiners D. Prepreg tack: A review of mechanisms, measurement, and manufacturing implication. Polym Compos 2020;41:3440–58.

[10] Crossley RJ, Schubel PJ, Warrior NA. The experimental determination of prepreg tack and dynamic stiffness. Compos Part A - Appl Sci Manuf 2012;43(3):423–34.

[11] Crossley RJ, Schubel PJ, De Focatiis DSA. Time–temperature equivalence in the tack and dynamic stiffness of polymer prepreg and its application to automated composites manufacturing. Compos Part A - Appl Sci Manuf 2013;52:126–33.

[12] Endruweit A, Choong GYH, Ghose S, Johnson BA, Younkin DR, Warrior NA, et al. Characterisation of tack for uni-directional prepreg tape employing a continuous application-and-peel test method. Compos Part A - Appl Sci Manuf 2018;114:295–306.

[13] Smith AW, Endruweit A, Choong GYH, De Focatiis DSA, Hubert P. Adaptation of material deposition parameters to account for out-time effects on prepreg tack. Compos Part A - Appl Sci Manuf 2020;133:105835.

[14] Choong GYH, Endruweit A, De Focatiis DSA. Analysis of contact area in a continuous application-and-peel test method for prepreg tack. Int J Adhes Adhes 2021;107:102849.

[15] ASTM. D8336–21. Standard test method for characterizing tack of prepregs using a continuous application-and-peel procedure. West Conshohocken, PA: ASTM International; 2021.

[16] ASTM RR:D30-2001. Interlaboratory Study to Establish Precision Statements for ASTM D8336 - Test Method for Characterizing Tack of Prepregs Using a Continuous Application-and-Peel Procedure. West Conshohocken, PA: ASTM International, 2024.

[17] ASTM E691. Standard practice for conducting an interlaboratory study to determine the precision of a test method. West Conshohocken, PA: ASTM International; 2011.

[18] ASTM D8336-24. Standard test method for characterizing tack of prepregs using a continuous application-and-peel procedure. West Conshohocken, PA: ASTM International; 2024.