

Laboratory-scale Screening of Mechanical Properties of Resins and Composites: Relevance to Composites for Aerospace Applications

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ABSTRACT

In the early development of resins which may be candidate matrix materials *for continuous fibre composites, there exists a need to evaluate their mechanical properties in a way that has relevance to the requirements of aerospace applications. Consequently, a procedure for screening the properties of a resin sample has been established for this purpose where approximately 50 g of resin are required to obtain objective measurements of fracture toughness, fracture strength, yield strength and tensile modulus. These tests are subsequently complemented by a mechanical characterisation of the performance of composites. This screening procedure for composites measures unidirectional compressive strength, transverse strength in flexure (both with and without hot/wet conditioning), short-beam shear strength and inter-laminar fracture toughness.*

Results from these screening procedures suggest that data for resins and composites may be linked and also reflect performance in aerospace hldustry tests such as compression after impact.

1 INTRODUCTION

The aerospace industry has **established its** own approach for evaluating a new continuous fibre reinforced composite.¹ As a minimum requirement,

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some aspect of damage tolerance (e.g. compression after impact) would be measured, as well as some aspect of compressive strength (e.g. open-hole compression, often conducted at an elevated temperature after conditioning in hot water). Other mechanical property tests might also be required, such as, for example, interlaminar fracture toughness, shear strength and tensile modulus/strength. Performance of a new composite in these tests would be set against a clearly defined reference. Successful acceptance against this requirement would then open another route for further evaluation. Naturally, such screening and selection is a healthy sign that a critical appraisal is conducted of materials for aerospace engineering applications.

To prepare candidate materials for industrial screening it is likely that at least 3 kg of composite in pre-preg form must be available. In turn, this would demand a mass of 1.5 kg of polymer to be available for conversion into pre-preg, assuming a carbon fibre system with 60% by volume of reinforcement. These figures reflect likely loss of materials through various causes. When the polymer is obtained from a development or commercial polymerisation process, this quantity of material is often readily available, but when the polymer has never been previously synthesised or its chemistry is new or uncharted the preparation of 1500 g of material represents a major task. There is therefore a large incentive to be able to screen very small quantities of polymer and pronounce on its potential in a composite system. In a technical sense, the mechanical property evaluation by the aerospace industry is describing aspects of stiffness, strength and toughness of a composite in some practically relevant manner. Therefore, small-scale mechanical screening approaches are required to provide a similar service.

The purpose of this paper is twofold. First, we describe a procedure requiring small resin samples which allows polymers to be screened for likely performance as composite matrices. Second, a separate procedure is described, also with a small material requirement, which is designed to predict the performance of continuous fibre reinforced composites in the larger-scale tests which are important to the aerospace industry. The resin screening procedure aims to evaluate 50g of polymer and pronounce on stiffness, toughness and strength, and the composite screening procedure aims to characterise up to 1 m^2 of pre-preg (after subsequent consolidation into appropriate laminates). Two procedures have been considered necessary even though the results from a resin screening are aimed at evaluating performance of the resin as a composite matrix. This is because resin screening cannot evaluate the consequence of impregnating a fibre with the polymer, nor can it articulate on the choice of system. Therefore, the composite screening is considered to be a necessary and natural sequel to resin screening.

Finally, to develop an objective measure of mechanical properties, great emphasis will be placed on fundamental properties in both types of screening procedure. A 'fundamental' property is interpreted as a property that is independent of test geometry, as far as is possible. To this end, the next section will involve some discussion of candidate tests and will include an account of some tests that did not prove successful for composite screening purposes. The final section of the paper will describe some preliminary results in the applications of these screening procedures and accommodate a limited link with the more usual aerospace methods.

It should, of course, be emphasised that the motivation for the development of these screening procedures is not the replacement of existing aerospace screening tests. Instead, it is merely to replace the onerous, costly and time-consuming task of preparing large quantities of new polymer compositions and new fibre/matrix combinations ahead of a visible commercial incentive.

2 DISCUSSION OF SCREENING TESTS

2.1 Resin screening

It is assumed that about 50 g of polymer are available for resin screening. This can be converted, via a compression moulding process, into a plaque of thickness 3mm. Usually this provides a sheet of approximate areal dimensions $150 \text{ mm} \times 100 \text{ mm}$. The aim of resin screening is to obtain some objective measurements of toughness, strength and stiffness. In the discussion of these tests, the selection of plaque dimensions should become apparent.

Toughness measurement can be achieved through the application of fracture mechanics techniques.² In particular, the measurement of fracture strength, K_{bc} , and fracture toughness, G_{bc} , should ensure a geometryindependent approach to toughness determination. Certain technical and practical criteria must be satisfied, however:

(i) Test specimens should be relatively small and several specimens (at least five) should be tested, to approximate a statistically significant value. We have therefore adopted a single-edge-notched, three-pointbend geometry. Common specimen and test dimensions are length $=$ 80 mm, depth $(W) = 10$ mm, thickness $(B) = 3$ mm, and span = 50 mm. The notch is machined into the specimen depth and has a tip radius as small as possible, and never greater than 30 μ m.

(ii) Size criteria need to be applied to validate application of the linear elastic fracture mechanics approach and geometry independence in the test.³ In particular, two size criteria must be satisfied

$$
B > 2.5 \left(\frac{K_{\text{lc}}}{\sigma_y}\right)^2 \tag{1}
$$

and

$$
W > 5\left(\frac{K_{\rm lc}}{\sigma_{\rm y}}\right)^2\tag{2}
$$

where $\sigma_{\rm v}$ is the yield stress measured under the same conditions of temperature and loading rate as the K_{1c} measurement.

Equation (1) suggests that a plaque thickness of 3 mm (i.e. $B = 3$ mm) is a compromise between meeting this size criterion and achieving adequate length and width dimensions in the plaque to accommodate these and other resin screening tests. The toughness of candidate polymers can vary widely. At the outset of this work, we were unclear as to the range of fracture toughness values that we might encounter. Consequently, a set of test conditions were selected that would maximise the successful achievement of the size criteria just described. As high rates of deformation and low temperatures both have the effect of raising the yield stress, test conditions of 1 m/s and -65° C were selected. The 1 m/s test speed is achieved with an instrumented falling weight impact apparatus 4 and the low temperature is achieved by immersion before testing in a low-temperature bath. The test speed of 1 m/s is selected to avoid dynamic effects associated with higherspeed impact tests and therefore to ease the interpretation of the forcedeflection signal which is monitored during impact.⁵

At the completion of the work reported in this paper we were aware of the likely fracture toughness magnitudes that could be encountered, particularly with epoxy type resins. This experience subsequently led us to conduct the fracture toughness measurements at 23°C and 1 mm/min on a screw-driven universal testing machine. In addition, we found it desirable to ensure that a natural crack was present in the notched bend specimens by inserting a new razor blade into the notch before conducting a fracture mechanics test. All fracture data presented in this paper, however, relate to the conditions previously mentioned, namely 1 m/s and -65° C. Naturally, it should not be assumed that toughness data at the two sets of test conditions should have the same values.

Stiffness assessment in our resin screening procedure aims to determine a number of parameters. A stiffness/temperature function is obtained, to determine the glass-rubber transition temperature, T_e , and rate of change of

stiffness with temperature using standard dynamic mechanical analysis techniques. The T_e can be related to service performance in terms of a limiting temperature of application. 6 Another measured stiffness parameter is an accurate 23°C small strain tensile modulus, which can be calculated from a three-point bending stiffness at a test speed of 5mm/min on a Universal testing machine. In this test the beam span-to-depth ratio (100:3) is selected so that shear contribution to the measured displacement is always less than 1% .

Strength is assessed in terms of a yield stress at 23°C. The relevance of yield strength has already been discussed in terms of the fracture mechanics size criteria (eqns (1) and (2)). In addition, by specifying the failure criterion (yielding in this case) a consistent measure of strength can be achieved.

Naturally, a range of materials need to be accommodated by the screening procedure. At a thickness of 3mm, many polymer matrix materials will not exhibit yielding in tension at 23°C. Consequently, a uniaxial compression test has been adopted. Square-sided specimens (side A and thickness B) are compressed on a Universal testing machine at 5mm/min. Frictional effects between specimen and machine can be minimised by application of an appropriate grease, but there will probably remain a difference between true stress, σ_T , and applied stress, σ_A . These stresses are related by $⁷$ </sup>

$$
\sigma_{A} = \sigma_{T} \left(1 + \frac{\mu A}{4B} \right) \tag{3}
$$

where μ is a coefficient of friction.

Tests on several specimens with varying values of the side dimension, A, allow the true yield strength to be determined from a plot of apparent yield stress vs *A/4B;* usually four specimens are tested.

In conclusion, resin screening of a 50-g plaque of polymer will provide the following information:

2.2 Composite screening

The 1 m^2 of pre-preg is converted into two consolidated laminates of layup $[0]_{24}$ with an aluminium crack starter (usual dimensions 150 mm \times 100 mm. fibres aligned in the long dimension) and $[0]_8$ (usual dimensions 150 mm \times **150 ram).**

Both laminates are ultrasonically C-scanned to ensure adequate consolidation and both are subjected to a quality control testing procedure to ensure good impregnation of the fibres with polymer and as a general confirmation of adequate plaque quality. To this end a short-beam shear strength test is conducted on the $[0]_{24}$ laminate and a transverse flexural strength is conducted on the $[0]_8$ laminate. Three further mechanical property screening tests are then conducted on these two plaques:

(i) Inter-laminar fracture toughness is measured at 23° C on two or three double cantilever beam (DCB) specimens cut from the $[0]_{24}$ laminate incorporating aluminium crack starters. The general procedure for conducting this test is described elsewhere. 8 In our procedure, a crack is propagated from the aluminium crack starter as usual, but crack length during the test is monitored with the aid of a crack length detector foil affixed to the DCB specimen.⁹

Force, deflection and crack length are then monitored with a micro-processor (Hewlett-Packard 9816) programmed to conduct a full 'area' analysis of the data, to determine the opening mode fracture toughness, G_{1e} .

(ii) Compressive strength is measured on specimens cut from the $[0]_8$ laminate.¹⁰ Coupon specimens (dimensions $80 \text{ mm} \times 13 \text{ mm}$) are machined from the unidirectional laminate and end tabs are affixed to both ends of the specimen to achieve an eight-ply central section of length 4.7 mm. Some of these specimens are conditioned for 14 days in water at 82° C and some specimens are tested at 23° C without conditioning in water. All specimens are supported in an antibuckling jig designed to improve stability in the test. Although most specimens are observed to fail in the central eight-ply gauge section, occasionally clamp/end-tab failures occur. Whenever this occurs these values are ignored.

The exposure of the specimens to a hot and wet environment for a prolonged period and subsequent compression in the direction of fibre alignment will examine the influence of moisture uptake by the matrix. If moisture is absorbed by the matrix, its stiffness will be reduced and, in turn, the unidrectional compressive strength will be reduced. This has direct relevance to service applications when high compressive strength is a requirement on a structural component.

The ratio of compressive strength unconditioned at 23° C to that from the hot/wet test will therefore be a critical parameter and should be as close to unity as possible.

(iii) Hot/wet conditioning followed by compression does not critically evaluate the fibre-matrix interface. A comparison of the unconditioned transverse flexural strength at 23° C with that determined for hot/wet conditioned specimens, however, will give a direct indication of the sensitivity of the fibre-matrix interface to the hot/wet environment. Transverse flexure applies a bending stress perpendicular to the fibre direction and hence this loading mode encourages fracture at any weakness between fibre and matrix. Consequently, hot/wet conditioning (section (ii) above) will expose any possible interface inadequacy.

The ratio of transverse strength unconditioned to hot/wet conditioned should therefore be as close to unity as possible. Naturally, there will be few materials where this ratio remains at unity.

In summary, the composite screening procedure provides the following information:

2.3 Unsuccessful attempts at composite screening tests

During the experimental investigation of possible screening tests for composites we were unsuccessful in the development of a measurement of the impact energy to initiate delamination damage and in the development of a mini compression after impact procedure. Nevertheless, the technical aspects of these investigations were of considerable interest and therefore the outcome of the technical work is now reported.

Instrumented falling weight impact at low input energies was explored to measure the onset of delamination damage. Full details of this work are reported elsewhere.¹¹ In summary, however, the aim was to monitor the force-deflection signal on quasi-isotropic and cross-ply laminates to identify a characteristic feature that could be identified and related to the onset of delamination damage during the impact of a plate-type specimen.

This approach was not successful because delamination damage was shown not to be the first failure event in impact. Instead, tension surface splitting which involved transverse cracking preceded the development of delimitation cracking. The transverse cracking process could be identified on the force-displacement curve during impact, but the delamination cracking process was not apparent.

Compression after impact (CAI) has a special significance in aerospace screening and therefore it seemed logical to explore a small-scale version of this test. It was possible to develop a mini CAI test, but it proved difficult for the mini CAI to resolve important differences in damage tolerance.

The mini CAI specimen was cut from a small plaque of $[-45, 0, +45, 90]_{4s}$ laminate. The specimen had dimensions of 50 mm \times 50 mm and could be impacted with a falling weight impact machine where the specimen was simply supported on a ring of diameter 40 mm with an impactor of diameter 10 mm. Specimens could be impacted with a range of input energies sufficient to cause delamination damage (as detected by ultrasonic C-scanning) but not enough to propagate the delamination damage to the edge of a specimen. In practice, this produced a range of input energies per unit thickness up to about 3 J/mm for these nominally 4.5 mm thick plates.

A compression specimen was then prepared from the impacted specimens by affixing polymethylmethacrylate (PMMA) sheet (3 mm in thickness) to either side of the composite to make a stable block specimen. Araldite 2005

Fig. 1. Mini compression after impact test; 32 ply $[-45, 0, 45, 90]_{45}$ at 0.5 mm/min.

was successfully used for the bonding process. This block specimen was compressed without buckling, and fracture was initiated in the composite in the region of delamination damage. The mini CAI procedure therefore accommodates the requirements of the full damage tolerance test.

Results from these tests are illustrated in Fig. I, where compressive strength is plotted against impact energy per unit thickness of composite. Results relate to two carbon fibre epoxy composites of considerably different damage tolerance, namely IM7/934 and IM7/51010A (both materials supplied by the Fiberite Corporation (USA)). The damage tolerance of these materials in the full CAI test at 6.67 J/mm impact energy per thickness (1500 in-lb/in) was measured at 130 and 225 MPa, respectively (see Fig. 6) below and Section 4). The results in Fig. 1 indicated little difference in CAI strength at the lower regimes of impact energy per thickness. Consequently, larger values of impact energy per unit thickness are required to distinguish the damage tolerance of the materials and thus it was concluded that a mini CAI cannot be successful because the specimen's size restricts the input impact energy.

3 DISCUSSION OF RESULTS

Our resin and composite screening procedures have been applied to a large number of materials over the last 2 years. In discussing results from these tests, two approaches will be used. First, we shall examine the potential value of these procedures through a discussion of the relationships between one property and another. In this context, all the materials will be epoxy-based resins and carbon fibre reinforced epoxy composites. No further details of the samples will be provided, as the purpose of the discussion is to illustrate the philosophy in adopting the screening procedures. Second, we shall illustrate the procedures by specific reference to three materials, where some of the aerospace industry tests will also be included.

The first stage of screening is to conduct measurements on the resin. Quite simply, it is necessary to establish whether measurements of the resin toughness are related to toughness for the composites and whether resin moduli are related to compressive strength for the composites.

The resin screening data in our early work provided two expressions for the fracture toughness of the resin. The first of these is the direct measurement of G_{IC} , which is made at -65° C and 1 m/s. These conditions were selected to provide geometry-independent data under constrained conditions, i.e. similar to those experienced by the resin when it is constrained by the continuous fibres in a composite. Second, fracture toughness can be estimated at 23°C from the measurements conducted at

low temperatures. The argument involves some assumptions. For example, if the fracture strength is assumed to be temperature independent (as is the case for many resins²), then the K_{1c} measured at -65° C will be numerically equal to the K_{16} at 23°C. The resin screening procedure involves a measurement of tensile modulus, E, at 23^oC. G_{1g} at 23^oC can therefore be calculated as follows:

$$
G_{1c}(23^{\circ}\text{C}) = \left[\frac{K_{1c}^{2}(-65^{\circ}\text{C})}{E(23^{\circ}\text{C})}\right]
$$
(4)

Figure 2 shows a plot of composite interlaminar toughness vs fracture toughness obtained at -65° C. A linear regression analysis of these data results in a correlation coefficient of 0.70. If the G_{1a} at 23^oC was used instead of the low-temperature property for the resin then a correlation coefficient of 0.57 is obtained. It would not be expected that resin toughness and composite toughness would result in a correlation coefficient of unity, because crack propagation through the matrix of the composite can occur by one of several failure mechanisms.¹² Therefore, a correlation coefficient as high as 0-70 implies that the measurement of resin toughness is indicative of toughness in the composite.

A link between resin modulus and composite compressive strength is examined next, and Fig. 3 shows compressive strength obtained on

Fig. 2. Resin G_{1e} vs composite G_{1e} (resin G_{1e} at -65° C, composite G_{1e} at 23^{\circ}C).

Fig. 3. Compressive strength vs resin modulus.

unidirectional laminates plotted against resin modulus at 23:C. Two different measurements are included for the compressive strength of the composites, namely, unconditioned specimens tested at 23~C and hot/wet conditioned specimens tested at 82°C. Again, linear regression analysis suggests that resin modulus is linked to the compressive strength for the composite. A correlation coefficient of 0.54 is obtained for the unconditioned specimens and a correlation coefficient of 0.74 is obtained for the hot/wet case.

It is apparent, therefore, that the measurements of resin modulus and resin toughness provide a useful screen of downstream performance in the application of resins as composite matrices. It must be emphasised that these two properties cannot provide a complete picture.

The achievement of high modulus and high toughness in a resin is traditionally seen as mutually exclusive. Toughness of the resin can also be expressed as a ductility factor,¹³ which is derived from $(K_{1c}/\sigma_v)^2$. Previous work has shown, for a wide range of thermoplastics, that the ductility factor provides a useful expression of toughness. 14

Figure 4 shows the ductility factor for the resins plotted against resin modulus. Linear regression analysis yields a correlation coefficient of 0-40. Although this suggests some likelihood of the exclusion of high toughness with high modulus, it does not rule out the possible achievement of an appropriately designed resin composition. In addition, the provision of these properties from the resin screening can be seen to be helpful.

Fig. 4. Resin ductility factor vs modulus.

It has already been mentioned that resin screening must be complemented by composite screening to examine the fibre-matrix interface. Moreover, when the composites are conditioned in a hot/wet environment it is important to be able to describe the influence on the matrix and interface separately. To this end, a summary view is presented by the data in Fig. 5, which plots fractional reduction in compressive strength for unidirectional laminates against fractional reduction in transverse flexural strength. The fractional reduction terms relate the properties in the unconditioned state to properties after hot/wet conditioning (as described in Section 2.2). Several points emerge from the data in Fig. 5. First, the range of reductions as a result of hot/wet conditioning can be studied for the separate properties of unidirectional compressive strength and transverse flexural strength. Second, a linear regression analysis of these data indicates low correlation (a coefficient of 0-09 was obtained). It has been our contention, in designing tests for composite screening, that the compressive strength test on the unidirectional laminate, with its influence from hot/wet conditioning, relates to moisture uptake by the resin. Hot/wet conditioning for the transverse flexural strength specimens, on the other hand, could relate to interface effects. It would not be expected that these mechanisms are related and the low correlation coefficient supports this view.

The analysis of data from the resin and composite screening tests has been

Fig. 5. Fractional reduction in compressive strength vs fractional reduction in transverse flexural strength for compression.

based on conclusions from regression analyses. It has been our aim to explore the trends that are implicit in these data but, of course, there are some problems. First, a correlation coefficient of unity would not be expected in any of the presentations of the results because not all of those factors that could influence toughness or strength of a composite were present in a single test. Second, an encouraging correlation coefficient does not establish a relationship based on cause and effect. Whilst acknowledging the absence of conclusive proof in our discussion of results, the encouraging implications from these trends give confidence in the adoption of the screening procedures.

The final stage in this discussion is to examine the application of these screening procedures to some established continuous carbon fibre reinforced epoxy composites. All the materials were prepared by the Fiberite Corporation (USA). The thermosetting resins had grade numbers 934, 974 and 51010A; the composites were pre-pregged by Fiberite and comprise these resins with 60% by volume of Hercules carbon fibre IM7. Three sets of experiments were conducted on these materials:

- **a resin screening analysis (results in Table 1)**
- **a composite screening analysis (results in Table 2)**
- **a full evaluation of compression after impact (see Fig. 6)**

Resin	Т.	E	$\lbrack \sigma_v \rbrack$ comp	$K_{\rm bc}$ G_{13} $(+23^{\circ}C)$ $(+23^{\circ}C)$ $(-65^{\circ}C, 1m/s)$	
	$(^\circ C)$	(GPa)	(MPa)	$(MPa\sqrt{m})$ (kJ/m^2)	
934	205	3.5	170	$1 \cdot 1$	0 ₃
974	130	30 ⁰	137	$1-3$	0.7
51010A	115	7.7	96	$1 - 4$	0.8

TABLE I Results from the Resin Screening Procedure

A number of comments emerge from these results:

- (i) The toughness of the resins follows the order $51010A > 974 > 934$. The toughness of the composites follows the same order.
- (ii) The stiffness of the resins is in the order $934 > 974 > 51010$ A. Hot/wet compressive strength does not quite follow this order, although this property is greatest for 934 and smaller for 974 and 51010A with these showing similar hot/wet strength.
- (iii) The transverse strength in flexure is more susceptible to the conditioning than is the compressive strength. The hot/wet transverse strength in flexure follows the order $51010A > 974 > 934$. This is likely to reflect an interface difference after these materials have been conditioned. This observation may account for the higher composite interlaminar fracture toughness of 51010A than was anticipated from its resin toughness.
- (iv) The compression after impact behaviour displayed in Fig. 6 follows the order (say at 6.67 J/mm (1500 in-lb/in)) 51010A > 974 > 934. This pattern is reflected in the composite fracture toughness and this order would also be qualitatively predicted from the resin screening results.

Naturally, there is a danger in over-interpreting the results from three sets of tests on three materials. However, these trends and the general pattern of

Composite $(60\%~bv)$ volume	Transverse strength in flexure		Short-beam shear strength	Unidirectional compressive strength (MPa)		Inter-laminar fracture toughness
of fibre) $(23^\circ C)$		$(23^{c}C)$ (MPa) Uncond H W cond	(MPa)		23°C uncond 82°C H/W cond	$(kJ \, m^2)$
IM7/934	85	35	96	1180	900	0.3
IM7/974	99	48	93	1 2 7 0	810	06
IM751010A	137	87	73	1000	810	ŀl

TABLE **2** Results from the Composite Screening Procedure

results discussed earlier provide some confidence in the adoption of our screening procedures.

4 CONCLUDING COMMENTS

The purpose in this work has been to establish a testing strategy for the evaluation of composites for aerospace applications, whilst recognising that many of the aerospace industry screening tests for mechanical performance of composites are well established. Two additional tiers of testing have been added, particularly for those occasions when only small quantities of material are available. These tests involve stiffness, toughness and strength measurements of resins and composites. There is some basis for suggesting that these tests reflect mechanical performance in the aerospace industry evaluations.

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