Letters

Statistics of torsion measurements on carbon fibre composites

Frequently, when making measurements of the mechanical properties of carbon fibre composites, insufficient samples are available to give a reasonable estimate of the spread of the results. This note concerns the statistics associated with the measurement of shear modulus, G, shear strength, τ, shear strain at failure, γ (where γ equals the radius of the specimen x the angle of twist per unit length), and fibre volume loading V_f, of a batch of approximately 20 unidirectional carbon fibre resin specimens in the form of square cross-section rods 150 mm long. All were apparently identical and were made over a period of one week by the same person from high strength surface treated carbon fibre and a standard resin mix (100 parts by weight Epikote 828, 80 pbw methyl nadic anhydride hardener, and 1 pbw benzyl-dimethylamine accelerator) curved for $2\frac{1}{2}$h at 120° C. The nominal loading was 60 vol%. The centre 100 mm of each specimen was turned down to a diameter of 6.2 mm. For comparison purposes, ten pure resin specimens were made from a similar mix and cured in the same way.

The shear modulus, shear strength and shear strain at failure were measured for either material, as described in [1], at a shear strain of 0.5 rad min^{-1} and temperature of 23° C. When composite specimens failed there was a marked dip in the torque rotation characteristic, while resin ones were judged to have failed when the torque remained constant with rotation. It was easier to measure the failure strain in the former case than in the latter. For both types of material it was checked that the shear strength recorded at failure was a maximum. A microscopic examination of polished cross-sections of composite specimens showed that they were void-free. The fibre content was determined by digesting the resin in sulphuric acid and hydrogen peroxide.

The results of measurements of G, τ, γ and V_f for composite and resin specimens, together with standard deviations and coefficients of variation (C. V.), are listed in Table I. The information for the composite material was also plotted in histogram form and a normal distribution curve fitted using the data in the table. The fit, measured by the χ^2 test, was very good, the results for G, τ, and γ all being significant at the 25% probability level and those for V_f at the 10% level.

None of the variables (G, τ, γ) showed any correlation with fibre volume loading over the actual range studied here (56 to 62 vol%). This was confirmed for G by assuming that the dependence of G on V_f was a given by [2], and correcting all modulus measurements to 60 vol%. The mean modulus was then 5.2 GN m^{-2}, the standard deviation 0.3 GN m^{-2} and the coefficient of variation 5.7%. The only difference between the two sets of results is a slightly greater standard deviation for the corrected ones. Thus the scatter in G is certainly not due to small changes in fibre volume loading.

It should be noted that Heaton [2] used a finite difference technique to solve the equilibrium equations for a square array of circular fibres, and gave the ratio G_c/G_m as a function of G_f/G_m, where c, m, and f refer to the composite, matrix and fibre respectively, for a series of volume loadings cor-

TABLE I

Property	No. of specimens	Mean	Standard deviation	C.V. (%)
Carbon fibre composites				
Shear modulus (GN m^{-2})	20	5.2	0.28	5.5
Shear modulus corrected 60 vol%	20	5.2	0.3	5.7
Shear strength (MN m^{-2})	18	68.7	5.45	7.9
Shear strain at failure (deg mm mm^{-1})	18	1.7	0.22	13.0
Volume loading (%)	18	59.8	1.7	2.8
Resin				
Shear modulus (GN m^{-2})	10	1.8	0.05	2.8
Shear strength (MN m^{-2})	10	55.1	2.2	4.0
Shear at strain failure (deg mm mm^{-1})	10	3.25	0.22	6.8

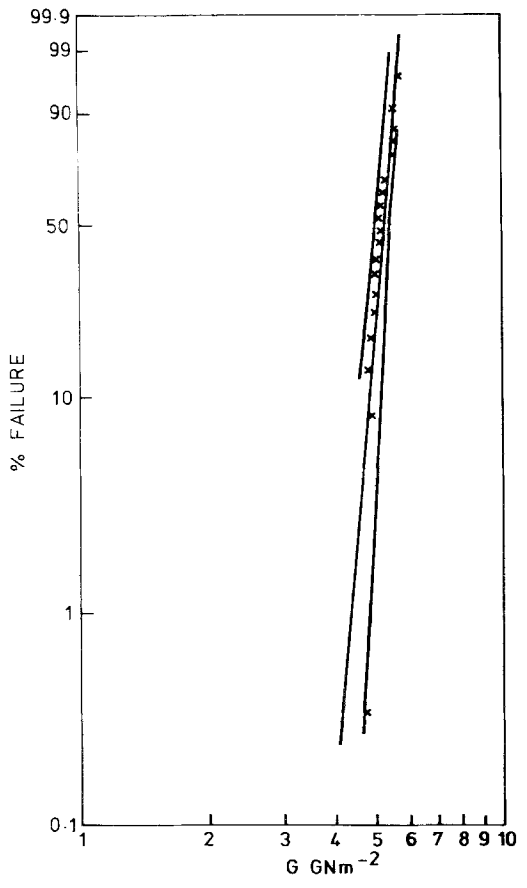

Figure 1 Percentage failure versus G.

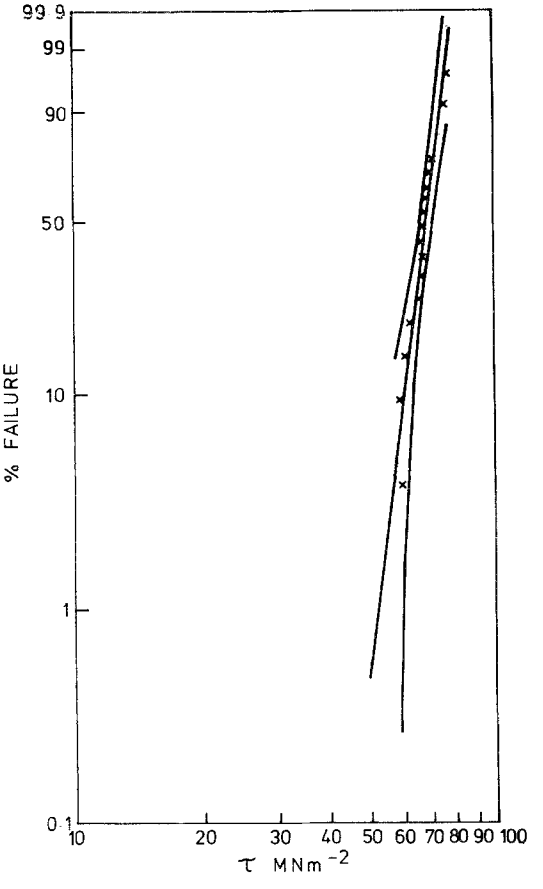

Figure 2 Percentage failure versus τ.

responding to different filament spacings. The simplest way of correcting modulus measurements to 60 vol% fibre is to choose a value of G_f to give agreement between theoretical and experimental moduli at 60 vol% and to obtain the complete curve relating G_c and V_f for this choice from [2]. The characteristic is approximately linear between 56 and 62 vol%, and if ΔG_c and ΔV_f are the changes in modulus and volume fibre percentage over this range the following relationship holds, $\Delta G_c = 15\,\Delta V_f$, and can be used to correct composite moduli to 60 vol%.

The coefficients of variation for G and τ are similar to those obtained for the tensile and interlaminar shear properties of composites (see [3]), with the variation in modulus again being less than that in strength. As both τ and γ are measured at break it might be expected that the coefficients of variation should be similar. This is not so because of the small slope of the torque rotation characteristic as failure is approached. Quite large changes in shear strain in this region will cause only small variations in shear stress.

For composite mechanical properties coefficients of variation are approximately double the corresponding values for pure resin specimens. Thus the introduction of reinforcing fibres, though improving modulus and strength and reducing shear strain at failure, lead to greater scatter in results possibly because of imperfect fibre alignment, interface effects such as poorly bonded areas, and changes in the resin properties when this is cured between fibres rather than in bulk. The constraint due to fibres could lead to steric effects in the resin, changes in concentration of the accelerator due to adsorption on fibre surfaces, and a changed exotherm. Shear failure is better defined experimentally for a composite than for a resin specimen, yet the coefficient of variation is twice as large in the former case than in the latter. This

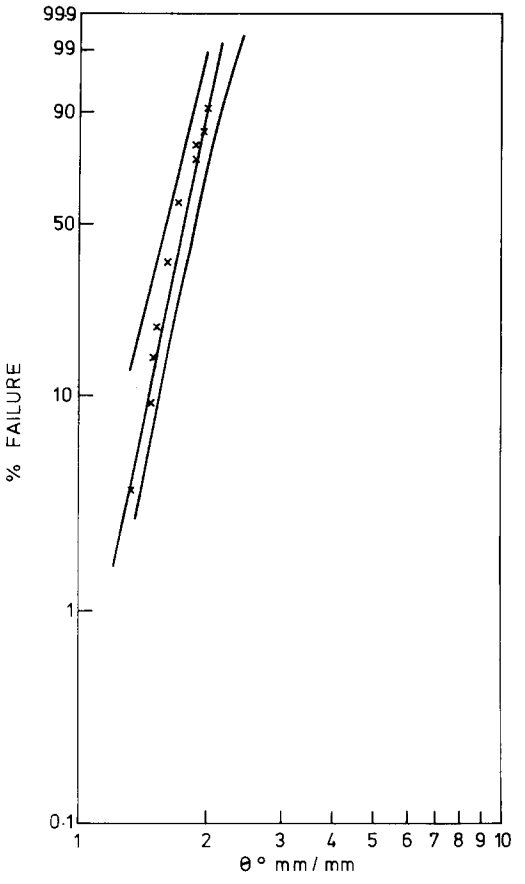

Figure 3 Percentage failure versus θ.

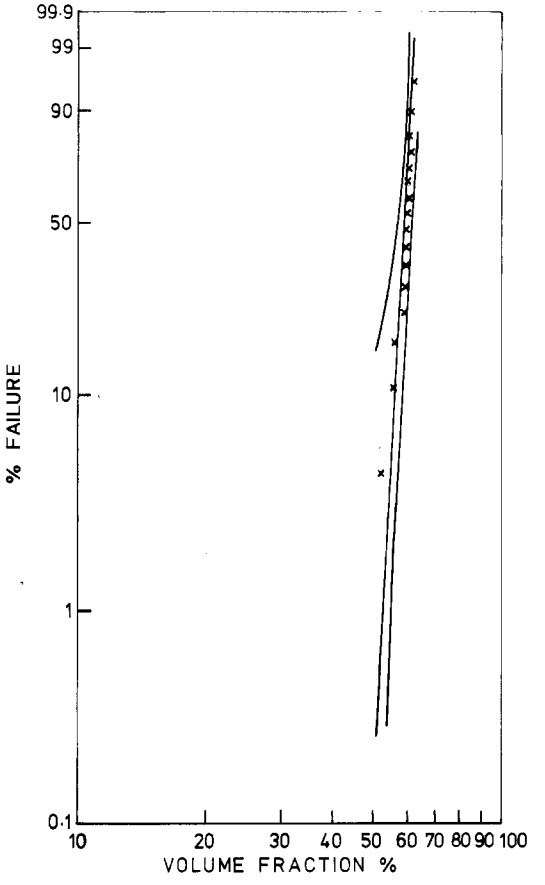

Figure 4 Percentage failure versus volume fraction.

indicates that the greater spread in failure strain results for the resin is not due to imprecision in defining failure, but reflects the basic behaviour of the material.

An alternative way of presenting the data of interest to designers, is by the use of a Weibull plot [4]. The Weibull relationship is $S = \exp(N/N_0 m)$, where S is the percentage of specimens having a property greater than N. N_0 is a constant, the characteristic life, and m is the Weibull slope. The Weibull slope, m, and median value of the variable with 95% and 5% confidence levels, are listed for the composite in Table II and the results shown graphically in Figs. 1 to 4. The large values of m for fibre volume fraction and shear modulus indicate the low scatter associated with these parameters. Median values are similar to mean ones.

It is also of interest to consider the maximum percentage errors calculated on the basis of those in individual measurements of diameter, torque etc. Individual percentage errors are listed in Table III and the resulting percentage errors in shear modulus and strength, and shear strain at failure for either resin or composite specimens, and the fibre volume fraction of a composite, in Table IV. Comparing these with the coefficients of variation for the various resin and composite properties indicates that the spread in resin modulus and fibre volume loading may be entirely due to experimental technique, but that otherwise the variation is largely a

TABLE II

Property	Weibull slope m	Median value
Carbon fibre composites		
Shear modulus (GN m^{-2})	21.6	5.25 ± 0.25
Shear strength (MN m^{-2})	15	69 ± 2
Shear strain at failure (deg mm mm^{-1})	9.5	$1.75^{+.07}_{-.15}$
Volume loading (%)	35.4	59 ± 1

TABLE III

Variable	Percentage error
Diameter	0.3
Length	0.5
Torque	1.0
Angular rotation (for modulus)	1.3
Angular rotation to failure	1.0
Weight of fibre or composite	1
Density of fibre or composite	1

TABLE IV

Property	Maximum percentage error
Shear modulus	4.0
Shear strength	1.9
Shear strain at failure	1.8
Volume loading	3.0

material property. In particular, note the small percentage error in the shear strain at failure compared with the large coefficient of variation of the measured results.

Acknowledgement

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Cooling-rate determination in splat-cooling of oxides

The technique of ultra-fast quenching from the melt (splat-cooling) developed by Duwez and co-workers about fifteen years ago has raised much interest in materials science research as shown by the large volume of work carried out in this field and reported in two exhaustive reviews [1, 2]. The very high cooling rates available in the splat-cooling technique (10^5 to $10^{8\circ}$ C sec^{-1}) made possible the preparation of new metastable cristalline and non-cristalline phases, providing a whole range of materials of unknown properties.

The quenching rates attainable by splat-cooling have been determined experimentally by three main methods. The first, due to Predecki *et al.* [3], consists of propelling a molten metallic sample by the "gun" technique on a substrate on which two dissimilar metals have been placed. The splat establishes a contact between the two metals and constitutes the hot junction of a thermocouple, the e.m.f. of which is recorded on an oscilloscope. A second method, due to Matyja *et al.* [4], is based on the study of the microstructure of a rapidly cooled alloy and uses the relationship between secondary dendrite arm spacing and cooling rate. A power relation between these two factors is established at low rates of solidification and may be used, by extrapolation, at higher cooling rates. The third method, proposed by Burden and Jones [5], is based on a method similar to the previous one, but takes into account the $\lambda^n R$ = constant correlation between the interlamellar spacing, λ, of an eutectic structure and its growth rate, R.

These methods have given rise to much controversy [6–9] in which we do not intend to participate here. We shall only consider that the proposed methods give only estimates for the cooling rates rather than precise measurements.

Splat-cooling has been so far almost exclusively applied to metallic systems except for some incursions in the field of oxides by Sarjeant and Roy [10–12]. The method has recently attracted more ceramists [13–17] and it seemed interesting to investigate what cooling rates are achieved during splat-cooling of oxides in comparison with metals and alloys.

The method of Burden and Jones [5] was considered. Experiments were carried out with the system NiO–CaO at the composition of the eutectic point (58% mol NiO). The melting point of mixtures of this composition is about 1720° C [18].

The splat-cooling device used in this study is of