JOURNAL OF MATERIALS SCIENCE 23 (1988) 830–834

Strain-controlled torsional test method for screening the performance of composite materials at cryogenic temperatures

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A system has been developed for rapid measurement of the torsional properties of organic-matrix composite materials at temperatures from 4 to 295 K. It offers ease of construction, simplicity of specimen design, small specimen size, rapid specimen turnaround, and low consumption of cryogens. In addition to providing quantitative data on the modulus of rupture and of rigidity, the strain-control feature facilitates analysis of the stress–displacement curve in the region where damage is occurring, providing useful information on the influence of various parameters on the failure mode. The system was optimized for rapid screening of the influence of component variables on the performance of electrical insulators required to function in the cryogenic irradiation environment of superconducting magnets in magnetic fusion energy systems. However, it is also useful in studying the influence of cryogenic environment on unreinforced polymers and on metals and alloys.

1. Introduction

Experimental screening of the influence of a large number of variables on mechanical performance is sometimes necessary when developing materials for demanding applications. For example, in developing organic-matrix composite insulators for use in the superconducting magnets of magnetic fusion energy systems, it is important that we maximize the resistance to degradation by neutron and gamma irradiation at 4 K. As little is known of the performance of composite material under these conditions, we must define the significant parameters that are involved and use the information to develop a data base that will permit optimization of the components from which the insulators are made. This will require screening of the influence of resin type, chemistry, and cure state as well as fibre type, finish, and chemistry under this very difficult environmental condition. Unfortunately, the cost of obtaining the large number of well-characterized specimens required for such a study and of performing the required testing programme would be prohibitive when normal approaches are used. The author has therefore undertaken development of a more efficient procedure for obtaining the required data. The specimen problem was addressed by developing procedures for rapid, in-house production of very highly characterized, 3.2 mm (0.125 in.) diameter specimens as either uniaxially reinforced composites or as neat resin specimens [1]. These specimens need only be cut to length before testing, thereby reducing cost and eliminating surface defects. The specimen configuration eliminates edge effects and permits many specimens to be simultaneously exposed to the adverse environment.

Procedures were developed for testing the specimens in conventional flexure or short-beam shear [2]. However, the non-symmetrical and changing stress state results in highly qualitative data that are difficult to interpret. The author therefore developed several novel test methods that could be used with the rod-shaped specimens to produce more quantitative data. A simple method for determining the longitudinal mode I fracture energy, G_{Ic}, at temperatures to 4 K has been described elsewhere [1]. A torsional test method is described in this paper.

2. System design and operation

The system is designed to apply strain-controlled torque loads of up to 3.53 Nm (500 in. oz) at temperatures from 295 to 4 K. This is sufficient to test composite materials, unreinforced resins, and nonferrous alloys in the form of 3.2 mm (0.125 in.) diameter rods. Materials such as stainless steel may be tested as rods of smaller diameter.

The system is shown in its room-temperature configuration in Fig. 1a and in its cryogenic configuration in Fig. 1b. The torsional load is applied by a 186 W (0.25 h.p.) reversible, variable speed d.c. motor through a speed-reduction gear box. Output torque is delivered through a 90° mitre gear box to a 3.53 Nm (500 in. oz) reaction torque sensor. The sensor is connected to a 6.35 mm (0.250 in.) diameter stainless steel rod that transmits the torque to the specimen. Polytetrafluoroethylene (PTFE) bearings spaced about 28 cm (11 in.) along the rod ensure centring of the rod within a 748 mm (19 in.) long, 12.7 mm (0.5 in.) o.d., 0.79 mm (0.03125 in.) thick wall, stainless steel reaction tube. Roller bearings above and below the load cell minimize friction.

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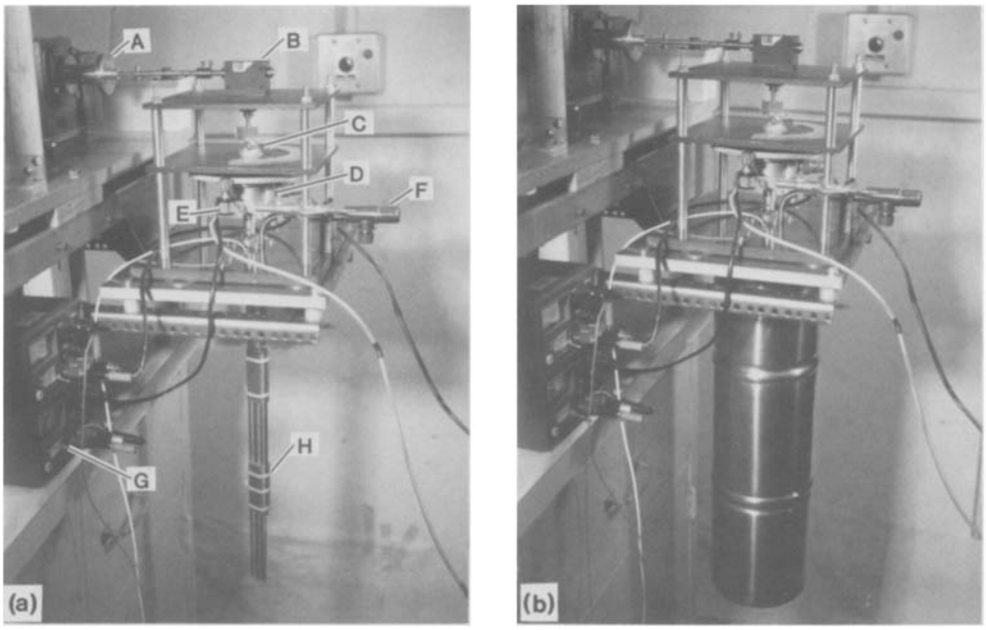

Figure 1 (a) Overall view of the torsional test facility. A, drive motor and reduction gear box; B, 90° mitre gear box; C, angular scale with indicator; D, torsion load cell; E, potentiometer; F, helium transfer line; G, power supplies; H, reaction tube, helium level indicator, and helium transfer line. (b) View of the test facility with 5 litre helium dewar installed.

Angular displacement is determined by a geared ten-turn precision potentiometer driven by a large spur gear fastened to the load cell. The ratio between the driven and driving gears of 4.8 to 1.0 provides a total available angular displacement of about 720 deg. An excitation of 30 V d.c. provides a good balance between sensitivity and noise. An indicator attached to the drive shaft allows the angular rotation to be calibrated directly from an angular scale. The potentiometer is supported by a clamp fastened to one of the vertical support rods of the system, allowing it to be rotated away from the driving gear, and permitting the output of the potentiometer to be zeroed regardless of the position of the driving gear.

The torque sensor has an output of 5 $\mathrm{mV\,V^{-1}}$ at rated capacity and is excited by 20 V d.c. It is calibrated by resistors simulating a known torque value. It may also be directly calibrated by dead-weight loading.

Specimens are typically 88.9 mm (3.5 in.) long, although shorter specimens may be used. They are held in 9.5 mm (0.375 in.) diameter 6061-T6 aluminium endcaps by a series of eight set screws arranged radially in two banks around each endcap as illustrated on Fig. 2. The endcaps are centre drilled to a diameter closely matching that of the specimens, and to a depth of 34.9 mm (1.375 in.), establishing a 25.4 mm (1.0 in.) gauge length between the first bank of retaining screws.

This gripping arrangement allows the endcaps to be reused, confines the failure zone to the gauge length, and allows the undamaged portions of the specimen to be recovered for further testing. For example, the undamaged 31.75 mm (1.25 in.) ends of an 88.9 mm (3.5 in.) long specimen may be subsequently tested in short-beam shear and for fracture strength, G_{Ic} [1].

Close alignment of the endcaps is required to achieve free rotation within the reaction tube. The relatively long specimen aids in this alignment. Additionally, the endcap alignment may be fine tuned by adjusting the screws in the set nearest the gauge length. As shown by Fig. 2, the inner diameter of the endcaps has been enlarged in this region to allow this operation to slightly tilt the endcaps relative to the specimen.

The drive shaft mates with a slot in the upper endcap; the bottom endcap is pinned to the stainless steel reaction tube. The bottom 114 mm (4.5 in.) of the reaction tube is internally polished to reduce friction. The lower endcap protrudes slightly from the reaction tube to facilitate specimen insertion and removal. Compliance of the system is determined from a stress against angular rotation plot produced with a solid aluminium rod inserted into the specimen position.

When inserting the specimen, the drive shaft is rotated to a predetermined angular position that

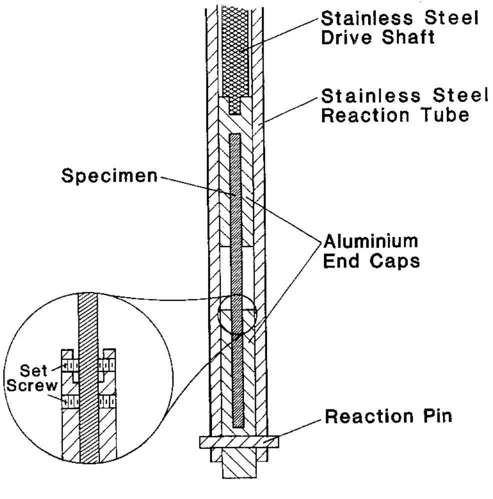

Figure 2 Sketch of specimen installation in the reaction tube and details of the specimen gripping system. The specimen is held by eight set screws arranged radially around the endcaps in two banks of four screws each. Countersinking in the vicinity of the first bank of screws permits correcting the alignment of specimens that are not perfectly straight.

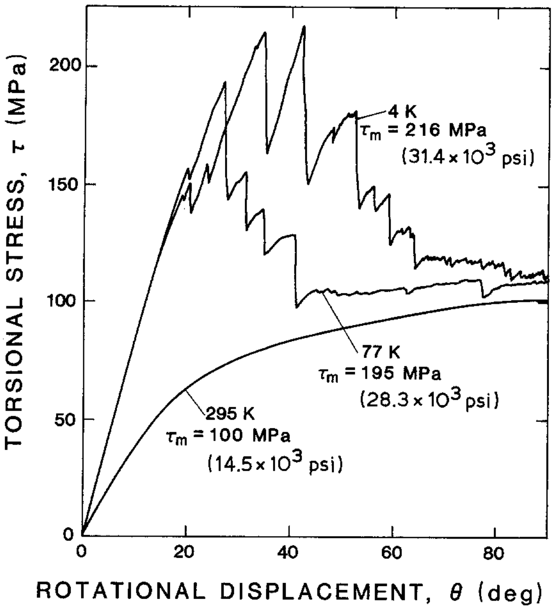

Figure 3 Typical torsional stress–displacement curves obtained at 295, 77, and 4 K with epoxy matrix composite specimens uniaxially reinforced with 48 vol % type E glass. The G-11CR designation identifies the resin formulation as duplicating that of the commercial product. Angular displacement rate $0.3° \sec^{-1}$.

allows the blade of the shaft to mate with the slot in the upper endcap while simultaneously allowing the retaining pin to be inserted into the lower endcap. Proper alignment between the upper and lower endcaps is established by assembly in a fixture.

The system incorporates a helium-level indicator to ensure that only enough liquid helium is added to cover the specimen when working at 4 K. Precooling with liquid nitrogen and careful liquid helium transfer reduces helium consumption to approximately 2 litre per test. The cycle time for successive tests with specimens already assembled into the endcaps is of the order of 20 to 30 min.

The apparent shear strength in torsion (modulus of rupture) τ, at any given torsional load T, is given by the relationship

$$\tau = 16T/\pi d^3 \qquad (1)$$

where d is the specimen diameter. When T is the torsional load at failure, τ is the apparent ultimate shear strength, $\tau_{\max}$. These values are apparent because they are based on the assumption that the effective radius of the specimen remains unchanged during the fracture process. Although this is in error, the data are valid for comparative purposes.

The elastic shear stiffness (modulus of rigidity), G, is given by the relationship

$$G = \tau L/\theta r \qquad (2)$$

where L is the gauge length, θ is the angular displacement in radians, and r is the specimen radius. Alternatively, the modulus of rigidity may be calculated by the relationship

$$G = 10.18TL/\theta d^4 \qquad (3)$$

which is derived by substituting Equation (1) in Equation (2).

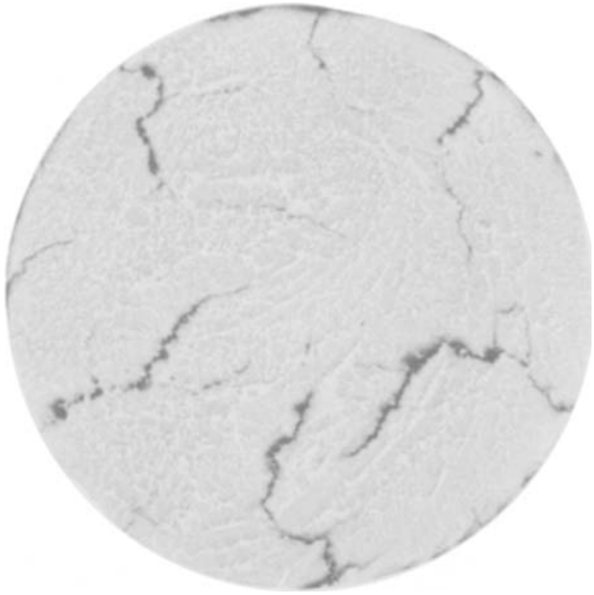

Figure 4 Typical cross-section of a 3.2 mm diameter, glass-fibre-reinforced specimen stressed in torsion at 4 K.

3. Results and discussion

Typical torsional stress–displacement curves produced to 90° rotation at 295 K, 77 K, and at 4 K with uniaxial glass-fibre-reinforced composite specimens are presented on Fig. 3. The specimens contained 48 vol % type E glass reinforcement in a bisphenol A epoxy matrix cured with an aromatic amine. This material is designated G-11CR because it simulates the commercial G-11CR high-pressure industrial laminate that is widely used for cryogenic applications [3].

At 295 K, the material deformed in a viscoelastic manner, without evidence of fracture. However, cooling to 77 K caused transition to a brittle behaviour, with a substantial increase in modulus and strength. Further cooling to 4 K had little effect on the modulus but resulted in a further strength increase. Linearity of the curve up to the first indication of failure indicates negligible plastic deformation at cryogenic temperatures for this resin system.

Because the specimen does not catastrophically fail, the strain-controlled torsional test method provides an opportunity for detailed study of the failure mode beyond the initial fracture event. As may be seen in Fig. 3, the onset of fracture events is indicated by a series of small load drops. This may reflect minor cracking of the thin epoxy film that forms the specimen surface. This is followed by a series of very large load drops, between which the stress is observed to significantly recover. This implies that failure is occurring through development of several major cracks rather than by development and propagation of a single crack. The cross-section of a failed specimen shown in Fig. 4 confirms that this is indeed the case.

A consistent feature of torsional stress–displacement curves of composite specimens at cryogenic temperatures is the eventual development of a region in which the average torsional stress remains fairly constant with additional torsional displacement. This region may be seen beyond 40° rotational displacement at

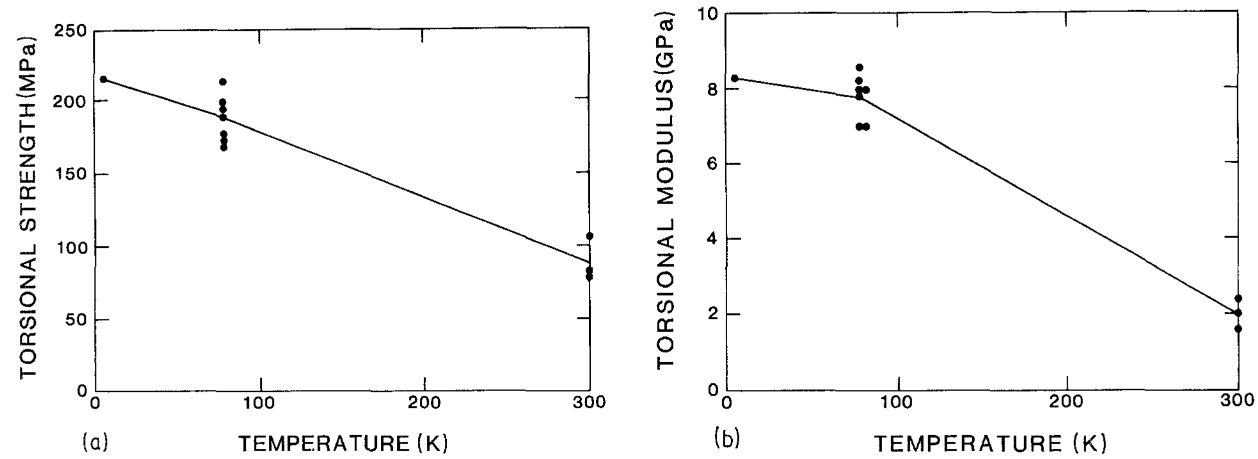

Figure 5 Temperature dependence of the torsional properties of the G-11CR composite specimens. (a) Torsional strength (modulus of rupture), (b) torsional modulus (modulus of rigidity).

77 K and beyond 60° displacement at 4 K on Fig. 3. An explanation for this phenomenon may have been provided by Outwater [4], who has noted that the torque required to drive a diametral crack along a composite rod specimen having uniaxial reinforcement will remain essentially constant as the crack deepens. This suggests that the onset of the constant stress region reflects the coalescence of individual cracks to form a diametral crack, and that the load drops observed within this region reflect the extension of the crack along the specimen gauge length. Cross-sectioning specimens stressed into this region has confirmed the existence of such a diametral crack.

Plots of the temperature dependence of the torsional properties for a series of G-11CR specimens presented on Figs 5a and b show that cooling to cryogenic temperatures increases the strength by about a factor of 2 and increases the modulus by about a factor of 4. These figures also show that the test procedure is capable of producing data having a reasonably low scatter.

Fig. 3 shows that the load drops produced during deformation at 4 K are much more prominent than those produced during deformation at 77 K. Because the test is performed under strain control, the magnitude of each stress drop is proportional to the newly cracked area. This indicates that the total cracked area generated by deformation at 4 K is much greater than that generated at 77 K, suggesting that cooling from 77 to 4 K has decreased the fracture strength of the material.

The work per unit volume done during torsional deformation and fracture is proportional to the area under the stress–displacement curve. As this is a measure of material toughness, it is a parameter of interest in a material screening programme. Fig. 6 illustrates the temperature dependence of this parameter for two composite materials. Taking 90° as a standard rotational displacement, cooling from 295 to 77 K increased the average work absorbed during deformation by about a factor of 2 for the G-11CR material. However, this parameter increased by a

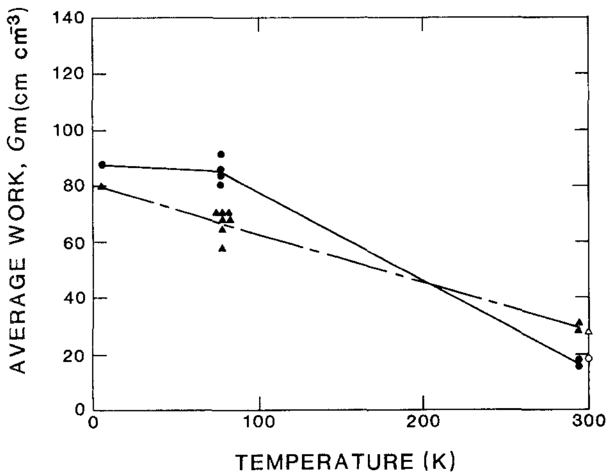

Figure 6 Temperature dependence of the average work required per unit volume to deform composite specimens to a 90° rotational angle. The impregnating epoxy system contains a flexibilizing agent reducing its toughness at room temperature, but increasing it at cryogenic temperatures. Data from unreinforced resin specimens were available only at 295 K, but show that at that temperature no difference exists between the reinforced condition. G-11CR: (▲) composite, (△) resin. Impregnation epoxy: (●) composite, (○) resin.

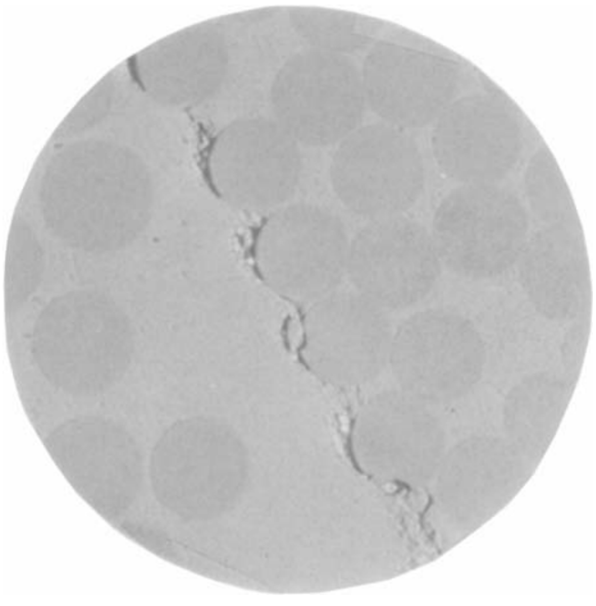

Figure 7 Photomicrograph illustrating the influence of the fibre–matrix interface in controlling the fracture path during torsional deformation of a uniaxial glass-fibre-reinforced specimen at 4 K. Direction of crack propagation is lower right to upper left. Average fibre diameter 12 μm.

factor of 4 to 5 for a composite system made with an epoxy resin formulated for impregnation of superconducting magnets. This impregnation system contains a flexibilizing agent that increases its toughness at cryogenic temperatures, while lowering its toughness at room temperature. Fig. 6 also shows that the average work done during deformation of the unreinforced resins and that of the reinforced resins are the same at room temperature. This indicates that the viscoelastic behaviour of the resins are controlling the room-temperature deformation, with or without the glass reinforcement.

The influence of the fibre–matrix interface in controlling the fracture path during torsional deformation of composite specimens at cryogenic temperatures is illustrated in Fig. 7. The test method should, therefore, be of particular value in studies assessing the influence of various parameters on the integrity of this interface.

4. Conclusion

The strain-controlled, torsional test method can provide an efficient and inexpensive method for screening the influence of component and environmental parameters on the performance of fibre-reinforced composite materials at temperatures from 295 to 4 K. A test facility using inexpensive rod-shaped specimens may be easily constructed. In addition to providing quantitative information on the torsional strength and modulus, analysis of the stress–deformation curve in the damage accumulation region provides valuable information on the failure mechanisms. The torsional test method, therefore, appears superior to the conventional flexure methods for screening composite materials performance.

Acknowledgement

This work was supported by the Office of Fusion Energy, US Department of Energy, Washington, DC.

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Received 5 January
and accepted 4 March 1987