Diamagnetic susceptibility of carbon fibre reinforced epoxy resin composites

One of the important problems in the area of composite materials is the understanding of the overall properties of a particular composite in terms of the properties of individual phases and their morphology. Recently we have initiated at our institution extensive studies of physical properties of various types of composites. In this paper we report our investigations on the magnetic behaviour of epoxy matrix composites containing carbon fibres. Specifically, this study was originated for the purpose of exploring possibilities for the characterization of fibre alignment using non-destructive physical methods. It is well known that graphite crystals are very anisotropic in their physical properties. High strength carbon fibres, having a high degree of preferred orientation of the layer ribbons parallel to the fibre axis, utilize the high Young's modulus parallel to the basal plane in comparison with that perpendicular to the basal plane [1, 2]. There is also a considerable diamagnetic anisotropy [2] between parallel and perpendicular directions with respect to the basal plane. This fact stimulated us to explore this anisotropy effect for possible characterization of fibre alignment in an epoxy matrix. The results and their significance are briefly described below.

The epoxy–carbon composites used in this study were prepared in the laboratories of Celanese Research Corporation. The samples for magnetic susceptibility measurements (approximately of the size 13.6 mm × 0.4 mm × 0.3 mm) were cut from the corresponding composite sheets so that the angle θ between the long axis of symmetry of the sample and the fibres were 0°, 30°, 45°, 60°, and 90°. The estimated error in these angles is about ±2° at most.

Two types of epoxy–carbon composites were investigated. Both were produced from the same type of epoxy base (X-506, Fiberite Co.). One composite contained unidirectionally imbedded carbon fibres (65 vol%) of Celion® GY-50. The other material contained 64 vol% carbon fibres of the Celion® GY-70. Both fibres are commercial products of Celanese research Company, having Young's moduli of 48.5 and 74 million p.s.i. (334 and 510 GPa), respectively.

The magnetic susceptibility (χ) measurements were made at 298 K using the Faraday balance described elsewhere [3]. The sample having particular θ-value was placed in a special quartz bucket which kept the long axis of the sample perpendicular to the magnetic field direction. The values of χ obtained with different fields were the same, indicating that there were no ferromagnetic impurities in the composite samples. The data of χ reported in this paper were obtained using the value $HdH/dz \approx 8 \times 10^6$ Oe^2 cm^{-1}.

The purpose of this investigation was two-fold. First, we wanted to measure the diamagnetic susceptibility $\chi(\theta)$ of carbon epoxy composites as a function of the angle θ between the axis of the fibres and the direction of magnetic field. Second, we were interested to see how close the experimental results agree with simple theoretical predictions of the susceptibility behaviour as a function of θ calculated from the limiting values, i.e., $\chi_{\parallel} = \chi(0°)$ and $\chi_{\perp} = \chi(90°)$.

Simple theoretical considerations show that the magnetic susceptibility of carbon fibres whose axes form an angle θ with the direction of magnetic field is given by

$$\chi(\theta) = \chi_{\parallel} \cos^2\theta + \chi_{\perp} \sin^2\theta. \quad (1)$$

Fig. 1 summarizes the experimental results and their comparison with Equation 1. The closed circular points represent the experimental data at 298 K for samples prepared from the carbon–epoxy composite containing GY-50 fibres. The closed triangular points give the susceptibility values associated with GY-70 fibres. The dashed curves are the theoretical predictions according to Equation 1 using the corresponding measured values of $\chi_{\parallel}$ and $\chi_{\perp}$. It is obvious from Fig. 1 that Equation 1 describes the θ dependence of the diamagnetic susceptibility of epoxy–carbon composites fairly well, although for both types of composites the experimental points lie below the theoretical curve. This deviation does not result from the diamagnetic anisotropy of the epoxy material. In fact, our measurements on epoxy samples without fibres clearly show that this material is magnetically isotropic. Specifically, the magnetic susceptibility of epoxy used to prepare the above composites is about $(-0.53 \pm 0.03) \times 10^{-6}$ cm^3 g^{-1}. This value is independent of the

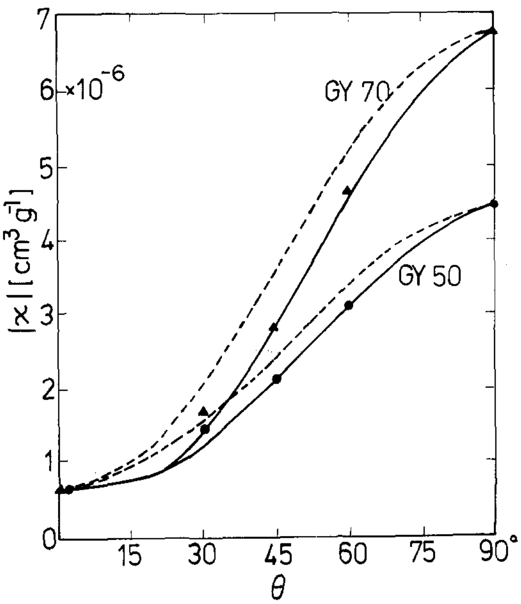

Figure 1 Diamagnetic susceptibility of carbon–epoxy composites at 298 K as a function of the angle between the axis of the fibres and the magnetic field.

direction of the applied magnetic field. Instead, the discrepancies can be traced to imperfect alignment of carbon fibres in the epoxy matrix.

The derivation of Equation 1 assumes perfect fibre alignment, thereby guaranteeing coincidence of the principal axes for the susceptibility tensor with the symmetry axis of the fibre bundle. Any fibre misalignment destroys the coincidence. However, if we assume these two axis systems remain coplanar, then Equation 1 is still valid provided we make the replacement

$$\theta \rightarrow \theta + \delta$$

where δ is the angle of rotation needed to restore coincidence. For $\delta \ll 1$ we obtain a modified equation

$$\chi(\theta) \approx \chi_0 \cos^2\theta + \chi_{90} \sin^2\theta + \delta \sin 2\theta \Delta\chi \quad (2)$$

with $\Delta\chi \equiv \chi_{90} - \chi_0$. χ_0 and χ_{90} are the measured longitudinal and transverse susceptibilities, respectively, and differ from $\chi_{\parallel}$, $\chi_{\perp}$ by the relations

$$\chi_{\parallel} \approx \chi_0 - \delta^2\Delta\chi; \quad \chi_{\perp} \approx \chi_{90} + \delta^2\Delta\chi \quad (3)$$

The solid curves in Fig. 1 are the predictions of Equation 2 with δ chosen to fit the measured susceptibility at $\theta = 45°$. The corresponding δ-values for the GY-50 and GY-70 composites are $-6.28°$ and $-8.12°$, respectively. The agreement between theory and experiment is now much improved, though unexplained discrepancies still persist for $\theta = 30°$.

The microscopic interpretation of δ depends upon the particular fibre geometry. For example, suppose all fibres lie in the same plane but make random angles with the symmetry axis of the bundle. If the i^{th} fibre makes an angle ϕ_i with respect to the bundle axis, the susceptibility tensor for this fibre in the symmetry axis system is

$$\chi_i = \begin{bmatrix} \cos\phi_i & 0 & \sin\phi_i \\ 0 & 1 & 0 \\ -\sin\phi_i & 0 & \cos\phi_i \end{bmatrix} \begin{bmatrix} \chi_{\parallel}^{f} & 0 & 0 \\ 0 & \chi_{\perp}^{f} & 0 \\ 0 & 0 & \chi_{\perp}^{f} \end{bmatrix} \begin{bmatrix} \cos\phi_i & 0 & -\sin\phi_i \\ 0 & 1 & 0 \\ \sin\phi_i & 0 & \cos\phi_i \end{bmatrix}$$

For a collection of N such fibres we obtain

$$\chi = \sum_{i=1}^{N} \chi_i = N \begin{bmatrix} \chi_{\parallel}^{f} + \Delta\chi^{f}\langle\sin^2\phi\rangle & 0 & \Delta\chi^{f}\langle\frac{1}{2}\sin 2\phi\rangle \\ 0 & \chi_{\perp}^{f} & 0 \\ \Delta\chi^{f}\langle\frac{1}{2}\sin 2\phi\rangle & 0 & \chi_{\perp}^{f} - \Delta\chi^{f}\langle\sin^2\phi\rangle \end{bmatrix} \quad (5)$$

where $\Delta\chi^{f} \equiv \chi_{\perp}^{f} - \chi_{\parallel}^{f}$ and $\langle\ldots\rangle$ denotes an average over N fibres. It is a straightforward matter to show that the tensor susceptibility of Equation 5 is diagonalized by a rotation through

$$\delta = \arctan\left(\frac{\langle\sin^2\phi\rangle - K}{\langle\frac{1}{2}\sin 2\phi\rangle}\right) \quad (6)$$

where

$$2K = 1 - \sqrt{[1 - 4(\langle\sin^2\phi\rangle - \langle\sin^2\phi\rangle^2 - \langle\tfrac{1}{2}\sin 2\phi\rangle^2)]} \quad (7)$$

For small deviations ϕ we find

$$\delta \approx \langle\phi\rangle, \quad (8)$$

showing that, for the assumed fibre geometry, δ measures the average deviation angle of the fibres from the symmetry axis of the bundle*. Other, more sophisticated fibre geometries would lead to more refined interpretations for δ, but we want to emphasize that the existence of such a δ leading to Equation 2 is independent of the microscopic interpretation we impose upon it.

It should be remarked that recently Scott and Fischbach [4] have discussed an interesting correlation between the total (tensor trace) magnetic susceptibility, χ_T, and the Young's modulus, E, Specifically, they find that there exists a straight line relationship between χ_T and E such that high values of E correspond to large values of χ_T. The quantity $\chi_T = 2|\chi_\perp| + |\chi_\parallel|$ equals $9.3 \times 10^{-6}\,cm^3\,g^{-1}$ for the composite containing GY-50 and $14.1 \times 10^{-6}\,cm^3\,g^{-1}$ for that containing GY-70. Since GY-70 has Young's modulus larger than GY-50, the above correlation is confirmed. However, quantitatively our data do not agree with the correlation line (Fig. 1 [4]) proposed by Scott and Fischbach. The reasons for this discrepancy are not clear at the present time.

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*It might be argued that $\langle\phi\rangle = 0$ defines the symmetry axis of the bundle, in which case higher order terms in the expression for δ must be retained.

The precracking of fracture toughness specimens of ceramics by a wedge-indention technique

The lack of agreement in the values of fracture toughness obtained by different workers for similar grades of alumina and other ceramic materials is partly due to the absence of a reliable method for precracking the test specimens [1, 2]. A similar problem existed with the determination of fracture toughness values for hardmetals but has recently been solved by the development of a wedge-indentation technique for precracking [3]. Successful use has now been made of wedge indentation to precrack ceramic specimens using alumina and silicon carbide as representative materials.

An indentation technique for precracking ceramics is attractive because it would be simple for small industrial laboratories to use. Pyramid indentation techniques for precracking have been used but they are not widely accepted because of uncertainties about the effect of residual stresses and the geometry of the cracks associated with the indentation. Attempts have been made to remove the stresses by annealing [4] or by grinding [4] away the surface containing the indentation; but annealing could affect the structure and integrity of the material at the crack tip, whilst the small crack size makes it difficult to remove the deformed material completely without removing a substantial part of the crack.

The problems associated with precracking were overcome for hardmetals by using a wedge-shaped indenter (Fig. 1) to produce planar cracks at the midspan position of 4-point bend test specimens. The cracks were much larger than those formed by pyramid indentation and only a small amount of the crack was lost when the plastically deformed zone around the indentation was removed by grinding away the surface with a diamond wheel. It was found that annealing was not a satisfactory way of removing residual stresses associated with the indentation since it produced effects that gave