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Diamond and Related Materials 12 (2003) 1766–1770

DIAMOND AND RELATED MATERIALS
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Stiffness measurements of diamond fibre reinforced plastic composites

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Abstract

Diamond fibre reinforced poly(methylmethacrylate) matrix composite test pieces have been fabricated and their Young's modulus values measured as a function of number of fibres incorporated. The fibres were 50 μm tungsten cores coated with 50 μm of CVD diamond, giving a total fibre diameter of 150 μm with 89% diamond by volume. The composites were made with fibres situated (i) close to one edge, or (ii) close to two opposite edges of the test pieces, in order to maximise the reinforcing effect of the fibres. Plastic matrix composites (PMC) containing two layers of 100 fibres (equivalent to a diamond fibre volume fraction of only 3.5%), exhibit a three-fold increase in the Young's modulus value over that for a corresponding sample of the pure polymer. This is a significantly greater increase than is achieved by inclusion of comparable fibres made from materials such as C fibre, SiC or for the uncoated W wire cores.

Keywords: Diamond fibres; Plastic matrix composites; Young's modulus; Reinforcement

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1. Introduction

The ability to deposit thin films of polycrystalline diamond on different substrates has enabled scientists and engineers to exploit some of its superlative properties in a variety of electronic devices and mechanical applications [1]. However, until recently, one property of diamond that has rarely been utilised is its extreme stiffness. One potential route to exploit the very high Young's modulus of diamond is in the form of thin wires or fibres, in which the chemically vapour deposited (CVD) diamond coatings are grown uniformly onto the surface of a metal or ceramic core wire [2]. Such 'diamond fibres' have core diameters ranging from 10 to 200 μm, with the thickness of the diamond coating ranging from 10 to 100 μm, giving them Young's modulus values that are significantly higher than those of conventional reinforcing fibres, such as SiC or C-fibres [3,4].

To exploit this exceptional stiffness, it is necessary to embed many fibres in a matrix material to produce a fibre-reinforced composite. Previously, such diamond fibres have been used to make stiff, lightweight metal matrix composites (MMCs), using Ti as the matrix material [5–7]. However, in order to obtain uniform fibre distribution within the Ti matrix, it was necessary to sputter coat each fibre with a thin uniform layer of Ti, which added extra time and expense to the manufacturing process. Furthermore, it was necessary to add substantial numbers of diamond fibres to the metal matrix in order to obtain appreciable benefit, and this high fibre volume fraction made Ti MMCs prohibitively expensive. One way to reduce the cost of diamond fibre reinforced composites is to reduce the volume fraction of fibres required. This can be achieved in two ways. First, by using a matrix material such as plastic, which has a much lower Young's modulus than diamond. Incorporation of even a small number of fibres will then make a substantial difference to the overall composite mechanical properties. Such a stiff, strong, but extremely lightweight, and relatively inexpensive plastic matrix composite (PMC) might find numerous uses as a structural material in, say, aerospace applications, where high performance for minimum weight is paramount. If continuous fibres are used aligned in the same direction, then the properties of the composite become anistropic. Different values for the composite Young's modulus will be obtained perpendicular to the fibre direction to those parallel to it.

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doi:10.1016/S0925-9635(03)00272-3

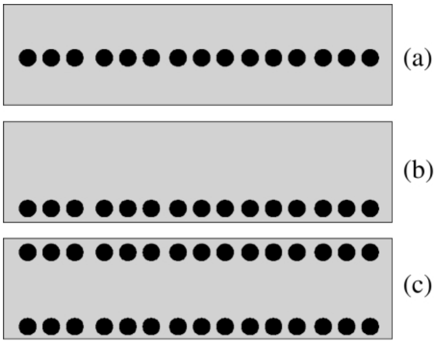

Fig. 1. Possible positions for the layers of fibres within the matrix test pieces. (a) in the centre, as used in previous studies [4], (b) one layer at one edge and (c) two layers close to opposite edges.

A second way to reduce the costs of fibre reinforcement is to simply use less fibres, but to position them in ways that maximise their effect. For example, it has been shown that placing fibres close to the edges of a composite, rather than evenly throughout the bulk, leads to a much greater stiffness [8], see Fig. 1.

Such PMCs were first demonstrated in our previous paper [9], using diamond fibres of 200 μm diameter (100 μm W wire with 50 μm diamond coating, giving a diamond volume fraction per fibre of 75%). These were embedded within a matrix made from poly(methylmethacrylate) also known as PMMA or perspex. We found that adding only 1% volume fraction of diamond fibres increased the stiffness of the composite by many times and that the stiffness was proportional to the number of fibres used. However, having the fibres situated in one layer in the centre of the test piece is not ideal, since the centre-line experiences minimum bending stresses and the fibres are not providing full reinforcement.

In this paper we present the results of further work in this area, with particular regard to the position of the fibres within the composite test pieces. The diamond fibres used are much smaller than in the previous study, having a 50 μm tungsten core coated with approximately 50 μm of diamond, giving a total fibre diameter of 150 μm, with a diamond volume fraction per fibre of 89%. But the main difference is that instead of placing the fibres in the centre of the test piece (as shown in Fig. 1a), we now position the fibres in either one layer close to one edge (Fig. 1b) or two layers close to opposite edges (Fig. 1c), where the reinforcement should have optimal effect. Again we note that although PMMA is not generally used as a structural plastic, it was chosen because (i) it is easy to make in the laboratory using cheap easy-to-obtain reagents, (ii) it is transparent, allowing the position of the fibres within the PMC and any cracks, bubbles or defects to be visible and (iii) it is reasonably rigid, but has a Young's modulus (approx. 5 GPa) significantly less than that of the diamond fibres (approx. 800 GPa [4,7]).

2. Method

2.1. Diamond fibre fabrication

The diamond fibres were deposited in a hot filament CVD reactor that was specially adapted for fibre substrates. The choice of substrate core material is limited to those materials which can survive the aggressive deposition conditions and which can be extruded in the form of wires. Past experience [2] has shown that despite its unwanted high density and cost, tungsten is one of the very few materials which are suitable for use as wire cores. Forty tungsten wires of length 10 cm and diameter 50 μm were loaded into the CVD reactor and clamped in a cage at a set distance of 5 mm from two 2-mm thick tungsten hot filaments. The process used 1% CH_4 in H_2 with a total flow of 200 sccm at a pressure of 20 Torr, and the filament temperature was 2200 K (as measured by a two-colour optical pyrometer). These conditions deposited diamond uniformly around the circumference of the W cores at a rate of approximately 0.7 μm h^{-1}. Thus, for a 50 μm coating, we required 70 h (3 days) continuous growth. Examples of the diamond fibres can be seen in Fig. 2.

2.2. Polymer matrix composite fabrication

PMCs were made by embedding these fibres into a block of PMMA. Blocks of PMMA were cast in a brass mould (100×20×5 mm) using a standard bulk polymerisation process. The monomer reagent, methyl(methacrylate) (Aldrich) was stirred with 0.5% by weight of a benzoyl peroxide initiator (Aldrich) at 90–100 °C for 10–20 min, until the solution became thick and syrupy. In this state, the solution was allowed to cool and remained liquid until subsequently baked. The cool, syrupy solution was then poured into the mould to a depth of approximately 0.5 mm, and then placed into an oven in air at 40 °C for 10 h. This caused the PMMA to set into a (soft) solid. The setting process

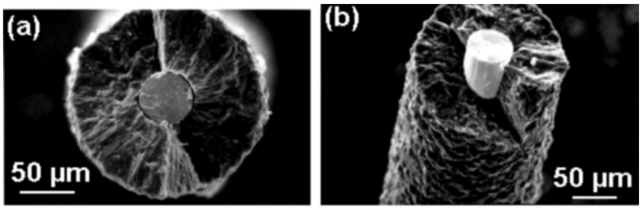

Fig. 2. SEM photos of representative diamond fibres, showing the 50 μm W core surrounded by, in this case, ∼60 μm of diamond, giving a total fibre diameter of 170 μm. (a) a cross-section of a fibre and (b) a view of the cleaved end of one fibre.

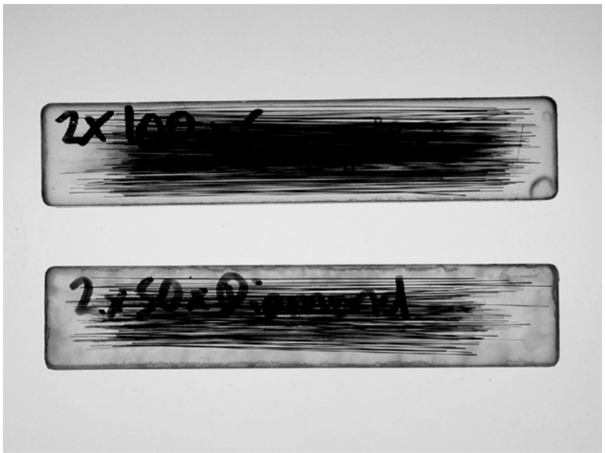

Fig. 3. Photograph of two PMCs. The top one has two layers of 100 carbon fibres each the lower one has two layers of 50 diamond fibres.

also caused the volume to shrink by approximately 15%. A number of diamond fibres were then manually placed lengthways into the mould using needle-point tweezers, ensuring that the fibres were all aligned parallel to each other and evenly spaced out. More liquid polymer was then poured over the fibres to encapsulate them and the mould was again baked at 40 °C for 10 h. In order to test the effect of different geometries of fibres upon mechanical properties, two different fibre arrangements were produced, with one or two layers of fibres, as shown in Fig. 1b,c. The fibre layers were ~1 mm from the edge of the sample. After the PMC was complete, it was given a final oven bake at 80–90 °C for 1 h to complete the polymerisation process and to harden the perspex. The resulting cast PMC was machined to create rectangular samples of dimensions 100×20×4 mm. PMCs were produced containing varying numbers of diamond fibres. For comparison, PMCs were also produced using commercially available (Textron) SiC fibres, pure tungsten wires (identical to those used as the cores in the diamond fibres) and C-fibres. An example of two of the PMCs can be seen in Fig. 3.

2.3. Young's modulus testing

The PMCs were tested to destruction using a screw-driven materials test machine. The samples were subjected to a standard four-point loading test, as illustrated in Fig. 4, and the central deflection, d (in mm), was measured as a function of the applied load, p (in N). For such a system, the Young's modulus, E (in Pa), is given by [10]:

$$E=\frac{G}{4w}\left(\frac{L}{h}\right)^3 \tag{1}$$

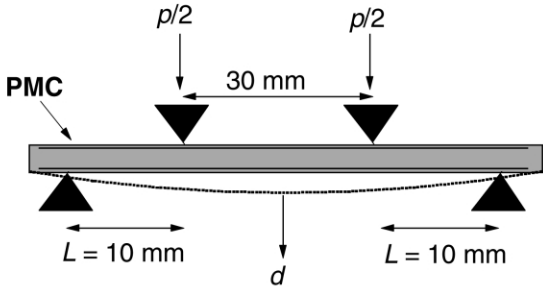

Fig. 4. A schematic diagram of the set-up for four-point bend testing.

where L is the distance between the upper and lower loading points (10 mm), w and h are the width and height of the PMC sample, respectively, and G is the gradient of the straight line graph of d against p, in units of N m^{-1}. Care was taken to measure the dimensions of each individual test piece accurately, especially h, since E has a cubic dependence upon this parameter. The 4-point bend apparatus was calibrated using test pieces with known Young's modulus values made from pure PMMA (E=3–5 GPa) and pure aluminium (E=70 GPa), having the same dimensions as the PMCs. An example of the data from a four-point bend test is given in Fig. 5.

3. Results

Fig. 6 shows the results of the Young's modulus of various PMCs as a function of number of fibres. For the composites with only one layer of fibres, the values of E start out consistent with literature values for pure PMMA (approx. 5 GPa), and increase monotonically with the number of embedded fibres, up to a value of

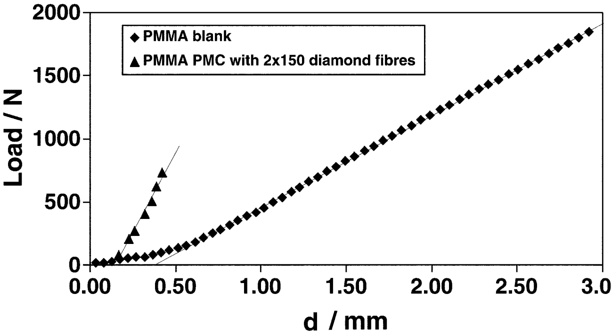

Fig. 5. Load vs. deflection curve from two four-point bend tests of a pure PMMA test-piece (◆) and a PMMA PMC test piece containing two layers of 150 diamond fibres (▲). There is an initial non-linear part in the curves due to the samples being not completely flat, and also to slack being taken up in the apparatus. Thereafter the graphs are linear and the line of best fit to this linear region is used to obtain the gradient, G, which is required in Eq. (1). The steeper gradient of the PMC curve highlights the increases stiffness of the PMC. Note that the PMC fractured after a load of 750 N, showing that increased stiffness gives rise to increased brittleness as well.

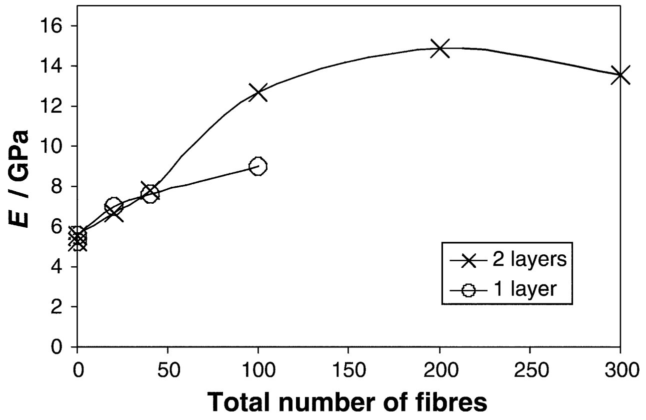

Fig. 6. A plot of the Young's modulus, *E*, of the PMCs against total number of embedded fibres, for composites with one layer of diamond fibres (as depicted in Fig. 1b) and two layers of fibres (as depicted in Fig. 1c). Due to the time involved in making the fibres and composites, only one sample of each type was made and tested. But an estimate of the experimental error was made by making two composites under identical conditions and measuring their moduli. The two measurements agreed to within ~10%, and so this gives an approximate measure of the variation expected in the other plotted points.

approximately 8–9 GPa after 100 diamond fibres have been embedded. The increase is larger for the double layer PMCs, reaching a plateau of ~15 MPa for two layers of 100 fibres. This corresponds to a three-fold increase in the Young's modulus value of the PMC for a diamond fibre volume fraction of only 3.5% (see Table 1). It is not surprising that two layers of fibre reinforcement gives approximately twice the stiffness increase of a single layer. The plateau in the first curve suggests that there is a limit to the reinforcement that can be achieved by diamond fibres placed along the edges in this manner. Addition of further fibres close to the edge beyond this limit has little effect, or may even decrease the modulus. This may be possibly due to the fibres being so closely packed that the polymer matrix is no longer able to effectively surround each fibre. Therefore, to obtain even greater stiffness increases, more fibres would need to be embedded throughout the bulk of the PMC.

To compare these modulus increases with those from other common reinforcing fibres, Table 1 shows the Young's modulus values of equivalent PMCs as a function of fibre volume fraction, fabricated using diamond fibres, C-fibres, Textron SiC fibres and the uncoated 50-μm diameter tungsten cores. Clearly, the other fibres have very little effect upon the modulus at these low volume fractions and only the diamond fibres show significant benefits. Also, it is clear that the layout and position of the fibres within the PMC is important. When fibres are positioned perpendicularly to the bending direction there is only a slight increase in PMC modulus. When 50 fibres were positioned in the centre of the test piece the composite had a lower modulus (7.2 GPa, see Table 1) than for 50 fibres placed in a single layer at one edge (approx. 8 GPa, Fig. 6), and a still lower value than for a composite with two edgewise layers (approx. 9 GPa, Fig. 6). This highlights the extra benefit obtained by positioning the fibres at the edge of the composite, thus optimising their reinforcing effect for the minimum number of fibres.

Another factor which remains to be investigated is the relative thickness of the diamond fibres layers

Table 1

Young's modulus of PMMA PMCs reinforced with various fibres. The volume fractions of fibres within the PMC test pieces were estimated from the equation: $F = \pi r^2 N/wh$, where N is the total number of fibres used in the PMC, r is the radius of the fibre, and w and h are the width and height of the PMC, respectively. The sixth entry in the table is for two layers of diamond fibres arranged crosswise, i.e. perpendicularly to the long axis of the test piece. The final entry is for a one-off sample made using the same procedures outlined in the main text but with a single layer of diamond fibres positioned in the centre of the PMC (as depicted in Fig. 1a)

Fibre	Fibre diameter/ μm	No. of layers× no. of fibres in each layer	Estimated volume fraction of fibres within the PMC	Young's modulus/ GPa
No fibres (pure PMMA)	–	–	0	5.24
Tungsten wire	50	2×20	0.08%	5.63
Textron SiC	100	2×20	0.31%	5.89
Carbon fibre	8	2×1000	~0.10%	5.34
Diamond fibre (+W core)	150	2×20	0.70%	7.80
Diamond fibre (+W core)	150	2×20 (crosswise)	0.70%	5.40
Diamond fibre (+W core)	150	1×20	0.35%	7.64
Diamond fibre (+W core)	150	2×100	3.5%	14.86
Diamond fibre (+W core)	200	1×50 (centre of PMC)	2.6%	7.2

compared to the overall height of the PMC test piece. It is known [11] that the distance between two reinforcing layers and also their distance from the edges of the test piece can both affect the measured Young's modulus values. In the present work these effects have been minimised by ensuring that the test pieces were very similar in size, however, this is an area for future investigation.

4. Conclusions

Diamond fibre reinforcement shows great promise as a method to fabricate strong, stiff, but lightweight structural components. Positioning the fibres at the edges of the composite allows maximum exploitation of the reinforcing properties of the diamond fibres, obtaining maximum increases in stiffness with minimal fibre usage. However, the utility and practicality of such advanced materials will depend upon economic factors, such as manufacturing costs and machinability of the PMCs, as well as the ability to scale up the fabrication process to industrial levels.

Acknowledgments

We wish to thank Mike Ashfold, Jason Riley and other members of the diamond group at Bristol for advice during this project. We also wish to thank Terence Cosgrove of the Bristol polymer group for help with making bulk perspex and Guy Pearn and Ian Milnes from the Mechanical Engineering workshops for help with the testing.

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