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DIAMOND AND RELATED MATERIALS

CVD diamond-coated fibres

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Abstract

Diamond-coated fibres have been fabricated using hot filament chemical vapour deposition (CVD) and tested for mechanical stiffness. The fibres coated include small (<20 μm diameter) commercially available samples such as Nicalon, Tyranno, Nextel, Altex and Hi-Nicalon, as well as thicker (100–150 μm diameter) SiC-based fibres produced by Textron and B.P. Mechanical bend tests show that the fibres were significantly stiffened by the addition of a CVD diamond layer of a few micrometres thickness, giving a Young's modulus value close to that for natural diamond.

Keywords: CVD diamond; Insulating fibres; Composites

1. Introduction

In the last few years CVD diamond technology has made a number of major advances, particularly for deposition onto flat substrates [1]. However, the technology of diamond-coating metal wires and non-metallic fibres is still in its infancy, with only a few reports appearing to date [2–7]. Most such reports concern the fabrication of diamond-coated metal wires made from materials such as Cu, Ti and particularly W. Hollow tubes of diamond have also been manufactured, both by removing the central metal core from a diamond-coated wire using an etchant, e.g. a concentrated acid [2,3], and by fabricating the tube in situ by CVD onto a coiled wire [8]. One of the suggested applications for diamond-coated wires with high specific strength is as reinforcing fibres in advanced composite materials. Diamond-coated wires will be electrically conducting owing to their metal cores, and so any composite made from them will also be conducting. For most applications (aircraft components, panels, gearboxes, etc.) this will not be a problem, but for some applications a non-conducting material is required. Instances might include situations where high electrical or magnetic fields are present, or where it would be desirable for the material to be transparent to electromagnetic radiation (e.g. 'stealth' applications). Such can be achieved by depositing diamond, uniformly, onto fibres with high electrical resistivity. This paper demonstrates the feasibility of producing such diamond-coated non-metallic fibres and provides a few preliminary modulus measurements.

2. Experimental

We have divided the insulating fibres that are commercially available into three main categories, depending upon composition and thickness:

(i) Thick SiC-based fibres. These fibres are currently used in the aerospace industry as reinforcing agents in metal matrix composites (MMCs) [9]. They are typically composed of a very thin core (either carbon fibre or W wire) coated in SiC by a CVD process to a total fibre diameter of about 100–150 μm. The presence of a small conducting core within the SiC fibre means that it cannot be considered to be completely insulating; however the relatively large thickness and stiffness of the fibre make for ease of handling and subsequent diamond CVD.

(ii) Silica fibres. These can be composed of either glass or quartz and can be solid or hollow. Their thickness is upwards of 10 μm.

(iii) Special ceramic fibres. These are novel fibres that have been developed recently for a variety of specialist applications. They are often composed of combinations of Si, C and O and, depending upon the composition, can be considered to be SiC with O added, or silica with C added. Other elements such as B or Ti are also incorporated as and when required in order to improve selected properties, such as electrical resistance, which can be varied from almost completely insulating to

semiconducting. The specific compositions of these fibres are given in Table 1. Other alumina-based or boron-based fibres are available but are yet to be studied.

Diamond-coating these fibres was performed in a standard hot filament reactor using typical CVD conditions (1% methane in hydrogen, 200 sccm gas flow rate, Ta filament temperature of 2000 °C, 20 Torr pressure). The filament was positioned vertically and the fibres were hung parallel to the filament at a distance of about 6 mm [2,3]. The temperature of the fibres during deposition was estimated to be about 900 °C. Handling and coating the stiffer SiC fibres usually posed no problems; however the thinner and more flexible fibres sometimes moved away from the filament during deposition as a result of convection currents within the reactor. This was cured by attaching a small weight to the bottom of the fibre, making the fibre hang straight and taut next to the filament.

To increase initial diamond nucleation rates, the thicker SiC and glass fibres were manually abraded with 1–3 μm diamond powder prior to deposition; however this procedure proved too damaging for the thinner fibres. Instead, these were pulled through a slurry containing diamond powder and methanol, and then cleaned. This is believed to distribute diamond seed crystals evenly along their length. The deposition rate was typically 0.5–1 μm h^{-1}.

3. Results

The results for diamond-coating the various fibres are detailed in Table 1.

(1) The type (i) SiC-based fibres were all very amenable to diamond CVD, with thick continuous films being grown uniformly over lengths of several centimetres (see Fig. 1). A two-point bend test was performed by measuring the deflection produced when known weights were hung from the centre of the fibre. This gave values for the Young's modulus for the composite fibre (Textron + 22 μm of diamond) of 850 ± 100 GPa), compared with 360 ± 20 GPa for the uncoated fibre, showing that the diamond coating has stiffened the fibre considerably. Deconvolution of the effect upon the modulus of the SiC core allows us to calculate the effective modulus for the CVD diamond, which gives a value of 1100 ± 200 GPa (cf. 1050 GPa for natural diamond).

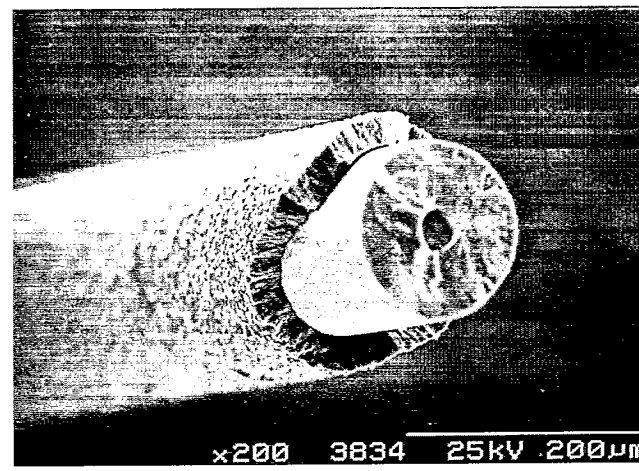

Fig. 1. Electron micrograph of a diamond-coated Textron SiC fibre. The inner core is C-fibre, coated in SiC, with 22 μm of diamond uniformly deposited around the outside.

(2) The type (ii) silica-based fibres all proved to be incompatible with diamond CVD. Although continuous adherent diamond films could be grown on the various silica and quartz fibres, the brittle fibres often broke during deposition or on cooling, probably as a result either of etching or stresses building up owing to the mismatch in thermal expansion coefficients of diamond and the fibre material.

Table 1
Types of insulating fibre and the results of diamond CVD

Name (supplier)	Diameter (μm)	Composition	CVD compatibility
SCS6 (Textron)	145	SiC on 33 μm C	Good
SiC (BP)	100	SiC on 15 μm W	Good
Glass fibre—solid	10–100	SiO_2	Shatters
Glass fibre—hollow	10–100	SiO_2	Shatters
Quartz fibre—solid	10–100	SiO_2	Shatters
Quartz fibre—hollow	10–100	SiO_2	Shatters
Carbon fibre	5–10	Carbon	Etches
Hi-Nicalon (Nippon)	14	Si (63.7%), C (35.8%), O (0.5%)	Good
Tyranno (Ube)	8.5–11	Si (51%), C (28%), O (18%), Ti (3%)	Good
Nextel 480 (3M)	10 × 13	Al_2O_3 (70%), SiO_2 (28%), B_2O_3 (2%)	Good
Altex (Sumitomo)	12 × 17	Al_2O_3 (85%), SiO_2 (15%)	Etches
Nicalon (Nippon)	14	Si (56.5%), C (31.2%), O (12.3%)	Etches
Ti-coated Nicalon	14+2	[Si–C–O] Ti outer layer	Good

(3) Some of the type (iii) ceramic fibres were compatible with diamond CVD. For example, Tyranno and Nextel fibres could be coated easily in continuous diamond layers, in some cases doubling or trebling the thickness of the resulting composite fibre (see Figs. 2 and 3). With such a high volume fraction of diamond, these fibres are extremely stiff, with measured modulus values again close to that of natural diamond. If, during CVD, the fibres were held in close proximity or touching, the diamond coatings on each fibre would fuse together producing a "double-fibre" or multiple-fibre bundle (see Fig. 4). This suggests the possibility of fabricating diamond-coated two-dimensional mattings or cross-weaves, for use in composites.

(4) Some of the fibres, however, were not compatible with diamond CVD, with the aggressive gas mixture etching them away after only a few hours of attempted deposition. This has been reported previously for C fibres [6,10]. One method of protecting such fibres during deposition is to add a thin barrier layer of a suitable material, such as Cu [6]. However, in order for the final diamond-coated fibre to remain insulating, the barrier layer must either be non-conducting itself or become non-conducting during the CVD process. To this end we sputter-coated 2 μm of Ti onto some Nicalon fibres prior to diamond CVD. This thin Ti layer protected the Nicalon from etching away long enough for a continuous diamond coating to form (see Fig. 5), and in the process the Ti layer itself became wholly converted to insulating TiC. We are presently evaluating whether this strategy works for the other etchable fibres.

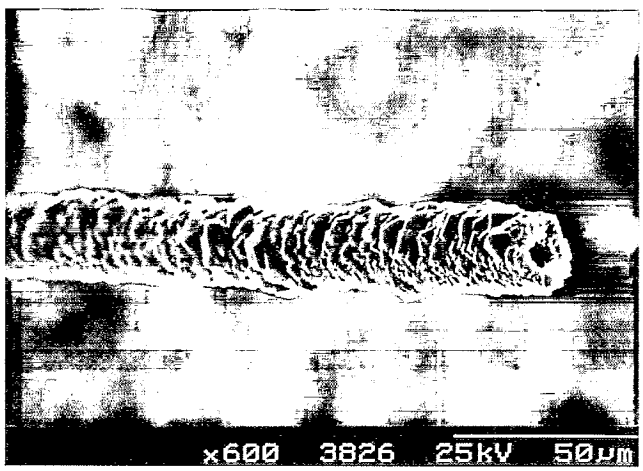

Fig. 2. A diamond-coated Tyranno fibre. The diamond coating has effectively trebled the diameter of the composite fibre.

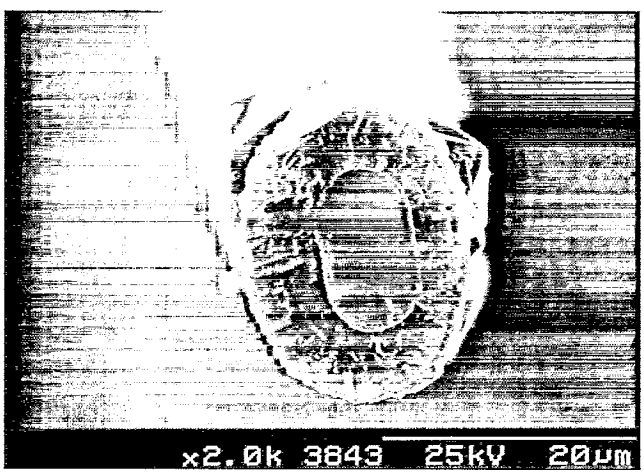

Fig. 3. A diamond-coated oval Nextel fibre.

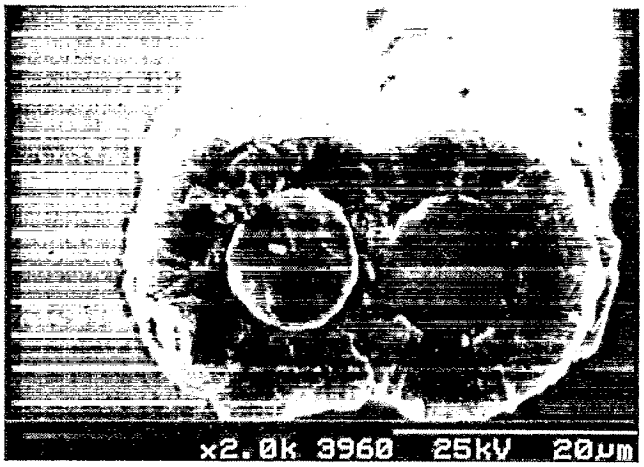

Fig. 4. A "double fibre" formed when the diamond coatings on two adjacent Tyranno fibres fused together. This approach can be extended to produce multiple fused fibre bundles.

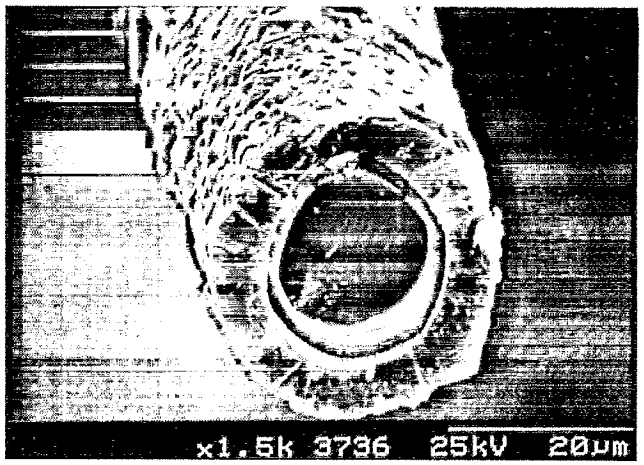

Fig. 5. A Nicalon fibre sputter-coated with a 2 μm protective barrier layer of Ti, then coated with diamond.

4. Conclusions

We have demonstrated that it is possible to deposit diamond onto thin fibres with high electrical resistivity. Such diamond-coated insulating fibres are much stiffer than the uncoated ones, suggesting potential uses as reinforcing agents in advanced composite materials. The economic viability of diamond-fibre technology has yet to be realised, however, and the use of diamond-coated fibres might at first be limited to specialised aerospace

applications. We still require more mechanical test data before the full capabilities of these fibres are known, in particular accurate values for the strength and stiffness properties of diamond-fibre-reinforced polymer and metal composites. Nevertheless, our initial results are encouraging and may provide innovative routes to advanced materials.

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