

## MECHANICAL PROPERTIES OF DIAMOND FIBRES

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### ABSTRACT

Tungsten and silicon carbide fibres have been coated with diamond using the HFCVD technique. The diamond volume fraction varied between 26% and 73%. Resonance in bending tests gave a Young's modulus of 880 GPa for the diamond coating. Tensile testing indicated that the diamond fracture strength was between 600 MPa and 2000 MPa, depending on the coating thickness, and thus the grain size, of the diamond. The strain to failure of the diamond coating in bending was approximately 0.15% for 25  $\mu\text{m}$  thick films.

### INTRODUCTION

Polycrystalline diamond film deposited on thin cores makes an attractive material for high modulus fibres if the stiffness of the single crystal can be reproduced. The specific stiffness of diamond compares well with that of high modulus carbon fibre. In addition, the fibre is electrically insulating if deposited on a non-conducting core, which could be an advantage for stealth applications. The commercial potential of the stiff fibre will depend on its fracture strength and strain to failure compared to other existing fibre systems such as carbon fibres and silicon carbide or other ceramic fibres.

Diamond fibres have been grown on silicon carbide (SiC) and tungsten (W) cores using hot filament CVD. The following paper reports the modulus, tensile fracture strength, and strain to failure (from bend testing) of diamond coatings of such fibres.

### EXPERIMENTAL DETAILS AND THEORY

#### Fibre growth

The diamond coating for this work was carried out in specially designed growth reactor, constructed in collaboration with Thomas Swan<sup>1</sup>. It is based on conventional hot filament CVD reactors, but instead of planar substrates long metallic or ceramic wires are arranged around a vertical central filament so that they are parallel to the long axis of the filament.

The source gases used were methane and hydrogen in the ratio 0.75:100. Total gas flow rate was 200 std  $\text{cm}^3 \text{min}^{-1}$ , and the chamber pressure was 20 Torr. Diamond growth rates were 0.5-1  $\mu\text{m h}^{-1}$ .

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The filament was made from tantalum wire and the filament to substrate distance was 5-6 mm. As a quality control tool, the temperature of the filament was read using a two colour pyrometer, imaging 2-3 coils (of 0.5 mm wire diameter) in the reading area, and the current adjusted to keep the temperature readings within the range 2050°C-2150°C. (These should not be taken as an absolute values of temperature since the emissivity of the filament was not checked, nor allowance made for any deviation from black body behaviour). The single wire cores did not give a large enough image area to use this technique, but from the diamond morphology and previous experiments, the substrate temperature was estimated to be approximately 900°C.

Two substrate materials were selected. The metallic substrate used was 125 µm and 25 µm diameter W wire. The ceramic substrate used was 100 µm diameter SiC fibres, grown on a W core, supplied by DRA Sigma. All substrate materials were abraded before growth by dragging the fibres through 1-3 µm diamond grit. Coating thickness varied between 10 µm and 46 µm, giving 26 - 73% volume fraction of diamond.

## **Modulus testing**

Elastic moduli in bending were measured for one batch of fibres using resonance equipment based on a system developed by Cranfield Research Institute [1]. The fibres were rigidly clamped at one end and the fibre excited by a frequency ramp, 10 Hz to 210 Hz. An image of the fibre was focussed on a split-diode detector. When the fibre vibrated, the difference signal contained a component at the frequency of resonance. The resonance of the clamped fibre was detected using a Hewlett Packard 35660A spectrum analyser (which also supplied the frequency ramp). In order to eliminate the effects of end corrections, 5 measurements were made on each individual fibre, shortening the fibre by 5-10 mm each time. The mean fibre diameter was determined by measurement with a micrometer at 5 different points along the fibre. Usually the first harmonic,  $f_1$ , was easier to measure than the fundamental frequency,  $f_0$ .

The equation for the fundamental bend frequency of a coated core can be re-arranged to give [1]:

$$L = \frac{1}{\sqrt{f_0}} \cdot \left[ \frac{A^4 [E_1 r_1^4 + E_2 (r_2^4 - r_1^4)]}{16 \pi^2 [r_1^2 d_1 + (r_2^2 - r_1^2) d_2]} \right]^{1/4} - L_0 \quad (1)$$

where  $f_0$  is the fundamental frequency, A is a constant, which is 1.8752 for the system used,  $E_1$  is the modulus of the fibre core,  $E_2$  is the axial modulus of the diamond coating,  $r_2$  is the radius of the coated fibre, L is the length of fibre,  $L_0$  is the end correction to the length,  $d_1$  is the density of the fibre core and  $d_2$  is the density of the diamond.

The first harmonic,  $f_1$ , was assumed to have a frequency exactly 6.25 times higher than  $f_0$ , and linear plots of L versus  $1/\sqrt{f_1}$  were constructed.

## **Tensile testing**

Tensile testing of the diamond fibres was carried out using a Hounsfield H5000M universal testing machine. Single fibres were fixed to a card with a central slot 72 mm long, and once positioned in the machine, the sides of the mount were cut so that only the fibre was load bearing. Tensile loading was carried out at a constant displacement rate of 0.5 mm/min, and the load and time recorded 5 times a second. The result was discarded if fracture occurred

outside the fibre gauge length. Diamond coating thickness was calculated from measuring the fibre diameter under an optical microscope.

Assuming that the core and coating remain united and intact, the strain in the fibre and coating is the same and the ratio of stress in core and coating is the same as the ratio of Young's moduli. The relation of the stress in each component and the overall fibre stress (overall load per unit area) can be estimated using a rule of mixtures. Thus, assuming that the diamond coating/core interface remains intact, the stress in the diamond coating can be calculated:-

$$\sigma_{\text{diamond}} = \frac{\sigma_{\text{fibre}}}{V_{\text{diamond}} + (1 - V_{\text{diamond}}) \frac{E_{\text{core}}}{E_{\text{diamond}}}} \quad (2)$$

where  $\sigma$  is the stress,  $E$  is the Young's modulus and  $V$  is the volume fraction of the particular phase.  $V_{\text{core}} = 1 - V_{\text{diamond}}$ . Assuming that the coating fractures before the core, the diamond fracture stress can be calculated from the maximum fibre stress.

### **Bend radius testing**

The SiC fibre core material and one other batch of fibres were tested for minimum bend radii using equipment based on that produced for the optical fibre industry [2]. A short length of fibre was bent into a wide arc between two parallel plates. One of the plates was moved towards the other at a speed of 1 mm/sec, and the separation of the plates at fracture of the fibre was recorded. The process was repeated with at least 5 fibres of each type.

However, for the length of fibre available, the bend radius of the diamond coated fibres proved to be too large for the equipment described above. Therefore the bend radius of these fibres was tested by the more simple technique of carefully bending single fibres round solid objects of progressively smaller radius of curvature until fracture occurred. An approximate bend radius was calculated by using the mean of the last successful radius and the radius around which failure occurred. Again at least 5 fibres were tested.

If a fibre is bent in the shape of a uniform circular arc, then :-

$$\frac{\sigma}{y} = \frac{E}{R} \quad (3)$$

where  $\sigma$  is the stress at a distance  $y$  from the neutral axis,  $E$  is the Young's modulus and  $R$  is the radius of curvature. Assuming that failure occurs at the surface of the fibre in the diamond coating, the elastic strain to failure of the diamond is given by the ratio  $r/R$  where  $r$  is the fibre radius and  $R$  is the minimum radius of curvature.

However if the curvature of the fibre is not a circular arc, then a correction must be applied. In the case of a fibre bent between two jaws, it has been shown that

$$\sigma = 1.198E \frac{d}{D - d} \quad (4)$$

where  $d$  is the fibre diameter and  $D$  is the jaw separation [2].

## RESULTS

### Elastic Modulus

The elastic modulus was tested using the resonance method on 3 diamond fibres from the same growth run, with 30  $\mu\text{m}$  of diamond on a 95 $\mu\text{m}$  diameter SiC core. The results were analysed assuming a modulus of 370 GPa for the DRA Sigma fibres. This gave  $877 \pm 84$  GPa (mean  $\pm$  SD) for the modulus of the diamond coating.

### Tensile strength

The fracture stress of the diamond coating was calculated assuming that the modulus of the diamond was 877 GPa (see above). The core moduli were taken as 370 GPa and 411 GPa for the SiC and W cores respectively. Mean diamond fracture strengths for various fibre batches are shown in Fig. 1. (95% confidence limits =  $\pm 2$  SE)

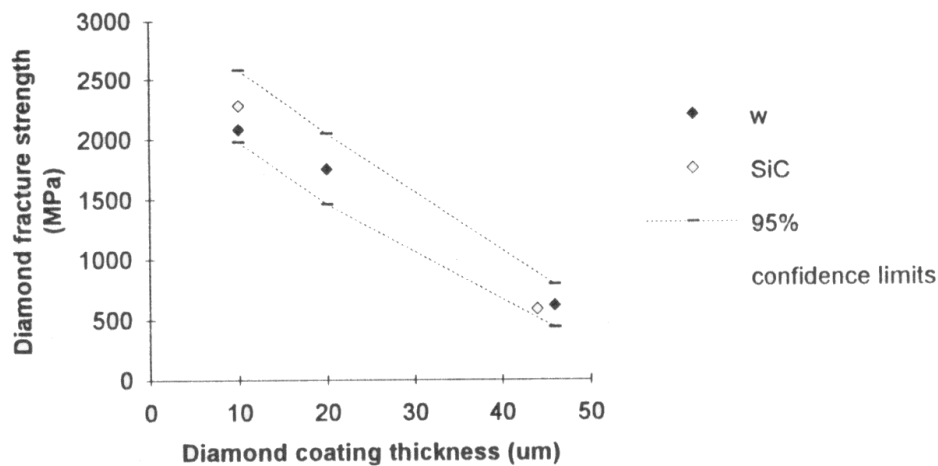


Fig. 1 Decrease in diamond fracture strength with increase in coating thickness

SiC core fibre straight from the spool gave a breaking stress of  $3000 \pm 232$  MPa (mean  $\pm$  SE). SiC fibre which had been given an abrading pre-treatment like those on which diamond was deposited gave a mean strength value of  $2388 \pm 132$  MPa.

### Bend radius

25  $\mu\text{m}$  thick diamond coating failed at about 0.15 % strain whether it was deposited on a 25  $\mu\text{m}$  W core (mean fibre bend radius  $45 \pm 3.3$   $\mu\text{m}$ ) or a 125  $\mu\text{m}$  diameter W core (mean fibre bend radius  $141 \pm 11.9$   $\mu\text{m}$ ). The abraded SiC failed at about 1.4% strain.

## DISCUSSION

### Equivalence of tests

The tensile test procedure gave a slightly lower breaking stress for SiC core fibre straight from the spool than the quoted value of 3750 MPa. It is therefore possible that the test is under-estimating the fracture strength of diamond, due to extra bending stresses introduced when the sample is gripped. A one-tailed t-test comparing the strength of abraded SiC fibre with fibre straight from the spool suggests that the decrease in fracture strength caused by the pre-treatment of the core is significant ( $p < 0.05$ ). However, it will not affect the elastic modulus of the core.

The bend tests carried out at DRA might have been expected to show smaller minimum bend radii than the method of bending fibres round a selection of solid objects, because of the increased possibility of surface damage during the latter tests. However, tests using both methods on abraded SiC fibre, and also on W cores (groups of 5 fibres) showed no statistically significant increase in the minimum bend radii of the fibres when using the simple test.

### Mechanical properties of diamond

The resonance tests produced a value for the bend modulus of diamond that correspond well with both the quoted modulus for single crystal diamond and the range of values found for polycrystalline diamond using vibrating reed tests [3, 4].

The tensile test results suggest a fracture stress for diamond in the range of 600-2000 MPa. These values thus overlap well with the values found by bulge testing technique [5, 6]. There is a substantial decrease in fracture strength with increase in coating thickness ( $r = 0.96$ ,  $p < 0.01$ ), see Fig. 1, which is independent of the fibre core type. It is probable that the decrease in fracture strength is caused by an increase in grain size with increase in coating thickness, see Fig. 2. Such a relation has also been suggested by work on flat substrates [5]. However, the apparent fracture stress of the thin coatings will also tend to be higher because the sampling volume is less.

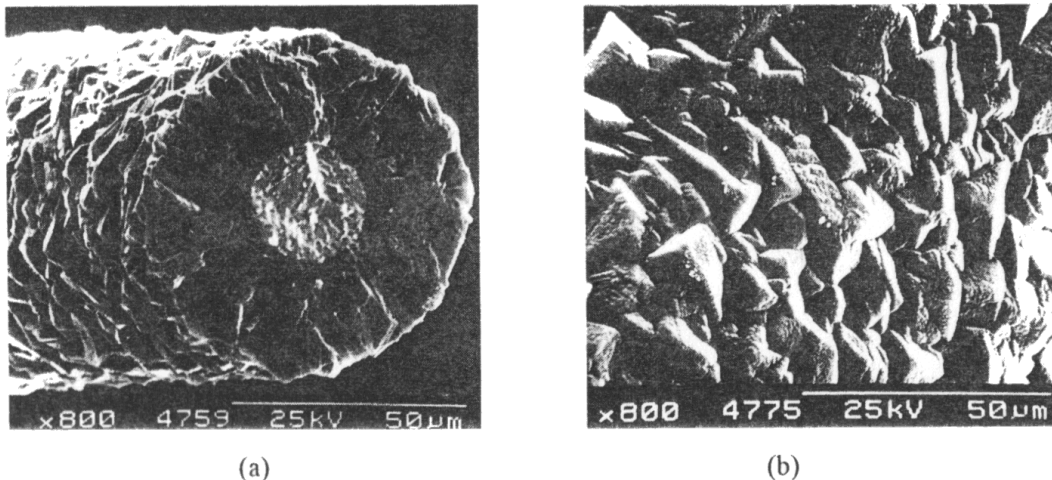


Fig. 2 Diamond coated W cores; (a) 25 µm thick film and (b) 46 µm thick film

The bend tests showed failure of the fibre at 0.15 % strain at the outer surface of 25  $\mu\text{m}$  thick diamond coatings. The stress in the outer surface of the diamond at this strain is approximately 1300 MPa. This fracture strength agrees with the tensile test results within experimental error, although the sampling volume is smaller in the bend test and might therefore be expected to give a higher value. Since the bend strength is very dependent on the surface flaw deposition, the agreement between the two techniques would also suggest that failure is occurring at the diamond outer surface in the tensile test, rather than at the W or SiC / diamond interface.

An average fracture toughness ( $K_{Ic}$ ) for single crystal diamond is 4.2 MPa  $\text{m}^{-1/2}$  [7]. If this value is combined with the fracture stress of 1300 MPa found in the bend test it gives a critical flaw length of 3  $\mu\text{m}$ . The reported values of  $K_{Ic}$  for CVD polycrystalline films are actually slightly higher than that for natural diamond [6, 8]), and this value for critical flaw length is therefore a conservative one. Thus an approximate critical flaw length for 25  $\mu\text{m}$  thick diamond would be 3-4  $\mu\text{m}$ . Using the same strain to failure the critical flaw strength is 1-2  $\mu\text{m}$  for 10  $\mu\text{m}$  thick film, and 16-17  $\mu\text{m}$  for 45  $\mu\text{m}$  thick films. (An approximate stress concentration factor of 1.12 was used for these calculations. The true stress concentrations will be determined by the actual shape of the flaws). These values for critical flaw size are of the order of the grain size of the material, see Fig. 2, and the fracture behaviour is consistent with that of other polycrystalline CVD material.

## CONCLUSIONS

The diamond fibres exhibited a very high modulus of 880 GPa, and a strain to failure in bending of about 0.15%. Critical flaw size is consistent with polycrystalline ceramics produced by CVD methods. The tensile strength, 600 MPa - 2000 MPa was dependent on coating thickness, probably related to grain size and/or morphology changes.

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