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Factors affecting the fracture strength and Young's modulus of CVD diamond-coated fibres

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Abstract

An improved non-contact strain measurement technique was used during tensile testing to measure the fracture strength, failure strain and Young's modulus of diamond-coated fibres produced by hot-filament chemical vapour deposition (HFCVD).

Diamond coated tungsten fibres/wires were produced under a range of deposition conditions. Fibre fracture strengths were in the range 207–1189 MPa, corresponding to diamond strengths of 257–1658 MPa. Fibre strength was strongly inversely related to coating thickness. Examination of the fracture surfaces indicated that two different modes of failure had been undergone.

The fibre Young's modulus was in the range 486–814 GPa. Calculation of the corresponding diamond coating modulus gave values of 559–1054 GPa. © 1997 Elsevier Science S.A.

Keywords: Diamond; Fibres; Stress; Young's modulus

1. Introduction

Metal or polymer matrix diamond–fibre reinforced composites provide an opportunity for the excellent mechanical properties of diamond to be exploited, by decreasing the problems associated with diamond's low fracture toughness [1]. It has previously been found that fibres with thicker diamond coatings are less strong [2] but stiffer [3]. This study investigates the effect of growth conditions on fibre properties with a view to producing fibres with an optimum combination of fracture strength and modulus.

2. Production of diamond-coated fibres and wires

Diamond coating was carried out using a prototype HFCVD fibre deposition reactor [4]. A vertically held coiled tantalum filament was used, surrounded by up to 14, 125 μm diameter tungsten wires, at a substrate–filament distance of 5–6 mm. The wires were abraded with 1–3 μm diamond powder to aid nucleation. Deposition conditions were as follows – chamber pressure 20 Torr, filament temperature 2150 °C, 0.5–1% methane (CH_4) in hydrogen (H_2) and gas flow rate 200 sccm. Coated fibres approximately 100–120 mm long were produced.

3. Experimental details

Tensile testing was carried out using a Hounsfield H5000M single column tensile tester, to provide data on both the stiffness and the fracture stress of the fibres. The non-contact strain measurement system is described elsewhere [5]. However, the system was improved in two ways for this study. A higher magnification system was used, giving an improved resolution of 1.8 μm. The force and extension data were interpolated together using the fracture times, and the gradient of this force–extension curve was used to calculate the Young's modulus of the diamond-coated fibre, rather than the values at the end of the test. Approximately 10 fibres from each growth run were tensile tested, although modulus data was not available for all fibres from each batch.

The coating strength was calculated from the fibre strength by assuming that the stresses in the core and coating are in proportion to their modulus and volume fraction [2]. This assumption is valid if the strain in the coating is equal to that in the core, i.e. if the core and coating remain attached during the test, and no coating cracking occurs. For those fibres for which data was available, the diamond strength was calculated using the measured diamond coating modulus. The diamond

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coating modulus was obtained from the fibre modulus using the rule of mixtures.

4. Results and discussion

4.1. Coating morphology

Coatings between 12 and 51 μm thick were deposited, with the grain size increasing approximately linearly with the thickness. The coating thickness was found to be reproducible between runs of the same conditions [6]. The coatings exhibited mixed morphology – both cubo-octahedral and cubic crystals were seen, with both the ⟨111⟩ and ⟨110⟩ directions perpendicular to the surface. Raman spectroscopy indicated that the lower methane % films were of better quality.

4.2. Strength

The strength of the tungsten cores before coating was found to be 2600 MPa. This value was not decreased by abrasion with diamond powder. However, for wires which had been under deposition conditions in the CVD chamber but which had not completely coated with diamond (possibly due to warping of the filament away from them), the strength was found to have decreased to 600–1300 MPa. This may be due to hydrogen embrittlement and/or the formation of a tungsten carbide layer at the surface. Interface studies on diamond-coated tungsten fibres using Auger electron spectroscopy found a tungsten carbide layer about 2.5 μm thick at the tungsten–diamond interface [7]. The thickness of this carbide layer was not affected by the deposition time.

Diamond-coated fibre strengths in the range 207–1189 MPa were found, corresponding to diamond coating strengths of 257–1658 MPa. Using a three-way ANOVA, the coating thickness was found to have a more significant effect than the methane % on both diamond and fibre strength.

The fibre and diamond strengths decreased with coating thickness (Fig. 1), and consequently with grain size. The larger grains associated with thicker films may decrease strength by acting as strength-limiting flaws, according to the Griffith crack criterion. Pores of the order of the grain size have been seen in CVD diamond, especially in films grown with a high growth rate [8,9]. A similar strength-coating thickness trend was found for diamond coatings on BP SiC fibres [10].

The strength was found to decrease with increasing methane %. The methane % affects the grain size, growth rate, and amount of sp^2 carbon in the film. The higher nucleation density of higher methane % films means that, for the same coating thickness, these films have smaller grain sizes. A smaller grain size might be expected to give higher strength. However, the increased

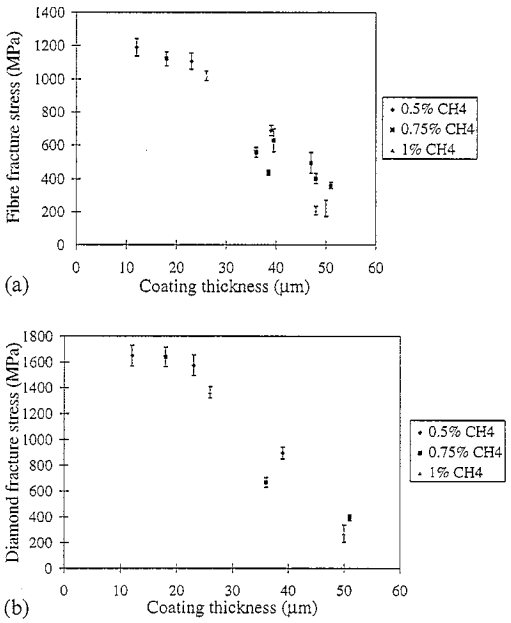

Fig. 1. (a) Decrease in fibre fracture strength with increasing coating thickness (error bars ±1 SE), (b) Decrease in diamond fracture strength with increasing coating thickness (error bars ±1 SE).

growth rate of higher methane % films leads to a greater likelihood of the presence of pores and other defects. The sp^2 carbon will possess weaker bonds than the tetrahedrally-bonded carbon: however, its presence at grain boundaries may blunt cracks and delay crack propagation [11]. It would seem that in this case the presence of more defects and sp^2 carbon were the significant factors.

4.3. Fracture surfaces

Examination of the fibre fracture surfaces indicated two modes of failure – coating and core fracture in the same location (Fig. 2), and coating and core fracture in different regions, accompanied by pull-out of the core (Fig. 3). The absence of a decrease in the slope of a force–extension plot towards the end of the test indicates that core and coating failure occurred almost simultaneously. This supports the validity of the diamond fracture stress calculation. The absence of a decrease in slope before failure was in contrast to previous work on diamond-coated silicon carbide fibres by other authors [10]. In their study, examination after failure showed a number of radial cracks in the coating, indicating coating cracking before failure of the silicon carbide core.

Coating and core fracture in the same location indicates a strong core-coating interface. The crack appears to start in the coating, travel across the interface and enter a small distance into the core before deflection (Fig. 4). The second mode indicates a weak interface. Failure may initiate in either the core or coating,

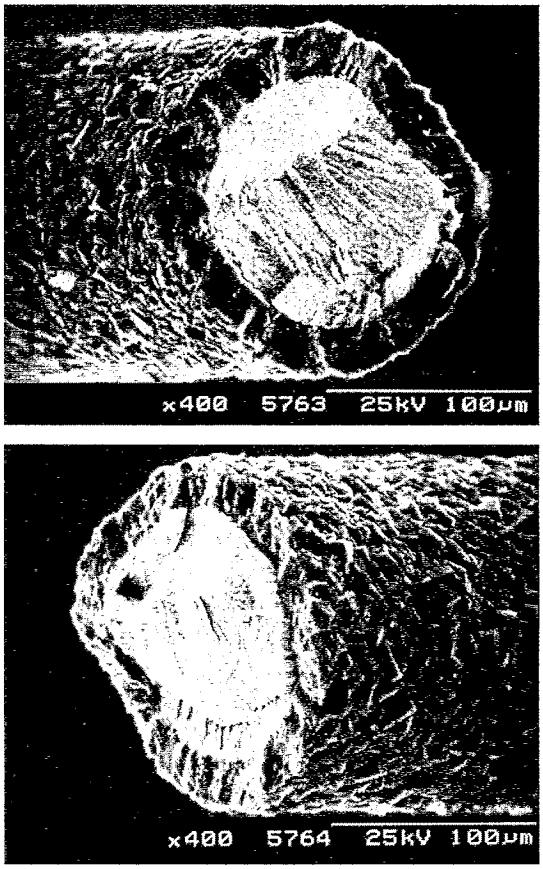

Fig. 2. Fracture surface – fracture without pull-out.

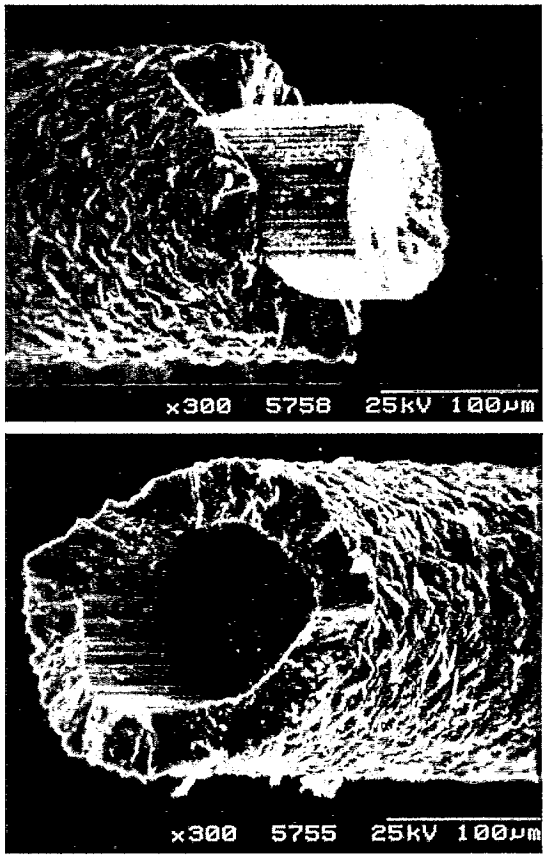

Fig. 3. Fracture surface – core delamination and pull-out.

followed immediately by delamination of the coating and failure of the remaining fibre component. For pull-out mode failure, longitudinal cracks of the order of 300 µm long were seen in some samples (Fig. 5). The pull-out distance was approximately 1–2 times the core diameter. Drory's coated SiC fibres failed by a pull-out mechanism with similar pull-out to core diameter ratios [10]. However, in his fibres cracking of the coating occurred somewhat before failure of the whole fibre. The failure mode of the fibre and its fracture strength did not appear to be related, although the fracture surface of only one fibre from each batch was examined. The fibres exhibiting failure by pull-out might be expected to be stronger, owing to absorption of the fracture energy by deflection of the crack along the interface.

4.4. Young's modulus

For diamond coatings 12–51 µm thick, the fibre Young's modulus was in the range 486–814 GPa. Calculation of the corresponding diamond coating modulus gave values of 559–1054 GPa. For 0.5 and 1% coatings, the diamond modulus increased with coating thickness. For 0.75% coatings, the opposite trend was

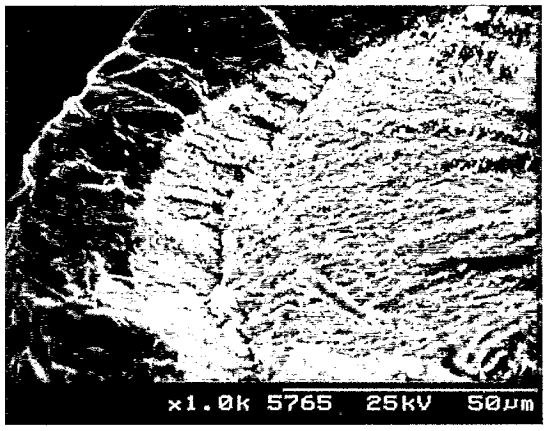

Fig. 4. Close-up of fracture surface without pull-out.

seen (Fig. 6). Using resonance testing to determine the fibre modulus gave the same trend for the 0.75 and 0.5% coatings, but a decrease in stiffness with thickness for the 1% coatings [6].

Factors which might affect the modulus are the film quality – a poorer quality film would be expected to have a lower modulus – and grain size. Although larger grains might be expected to give a higher modulus due to less grain boundary sliding and less disruption of the crystal lattice, pores of the order of the grain size are

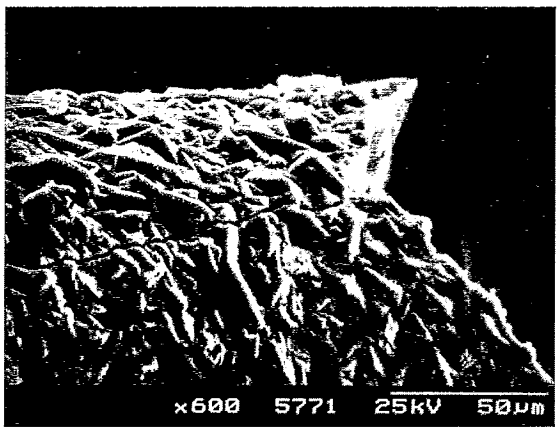

Fig. 5. Longitudinal crack from pull-out fracture surface.

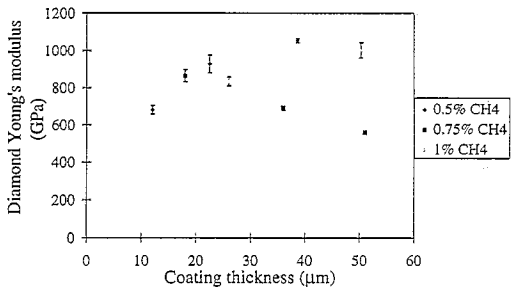

Fig. 6. Variation in diamond modulus with coating thickness (error bars ±1 SE).

likely to be present, which would decrease the modulus. The presence of pores is less likely in slower growing films, i.e. the lower methane % films. The increase in modulus with grain size for 0.5% films can thus be explained by the smaller grain boundary area at the film surface. The decrease in modulus with grain size for 0.75% films can be explained by the effect of pores. However, the quick growing 1% methane films would also be expected to show this trend, as was indeed found using resonance testing.

Using resonance testing to determine the fibre modulus gave higher modulus values for batches grown under the same conditions, probably due to differences in the test conditions [5]. Sources of error for modulus measurement by tensile testing include diameter variations along the length of the fibres, and the fibres not being perfectly straight. Any bending in the fibre prior to extension can have a detrimental effect on the measured modulus. Because a whole batch was tested, a larger variation in modulus for each type of fibre would be expected than from the resonance test, in which only three or four very straight fibres were tested. However, the tensile test is more commonly used as a standard test for fibre properties.

5. Conclusions and future work

The high strength of natural diamond (3.75 GPa) [12] indicates that there is potential for a large improvement in the strength of CVD diamond-coated fibres. Methods of strength improvement that will be investigated include control of core properties, grain size control, to produce small grain sizes for larger coating thickness, thus giving fibres that are both stiff and strong tailoring of the interface properties to produce a preferred failure mode and surface treatment, such as laser smoothing.

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References

[1] P.G. Partridge, P.W. May and M.N.R. Ashfold, *Mater. Sci. Techol., 10* (1994) 177.
[2] N.M. Everitt, R.A. Shatwell and E.M. Kalaugher, MRS Spring Meeting, San Francisco, April 17–21, 1995, *Mater. Res. Soc. Symp. Proc., 383 (1995)*, p. 379. in M.D. Drory, D.B. Bogy, M.S. Donley and J.E. Field (eds).
[3] E.D. Nicholson, J.E. Field, P.G. Partridge and N.M.R. Ashfold, MRS Spring Meeting, San Francisco, April 17–21, 1995, in M.D. Drory, D.B. Bogy, M.S. Donley and J.E. Field (eds). *Mater. Res. Soc. Symp. Proc.*, 383 (1995) p. 101.
[4] Reactor made by Thomas Swan and Co., Cambridge, UK.
[5] E.D. Nicholson, T.W. Baker, S.A. Redman, E. Kalaugher, K.N. Rosser, N.M. Everitt, M.N.R. Ashfold and P.G. Partridge, *Diamond Relat. Mater., 5* (1996) 658.
[6] E.D. Nicholson, J.R. Weeks, M.N.R. Ashfold, to be presented at Diamond 1996, September 8–13, Tours, France.
[7] Q.S. Chia, C.M. Younes, P.G. Partridge, G.C. Allen, P.W. May and C.A. Rego, *J. Mater. Sci., 29* (1994) 6397.
[8] G.D. Barbor, W.A. Yarbrough and K. Gray, in A. Feldman, Y. Tzeng, W.A. Yarbrough, M. Yoshikawa and M. Murakawa (eds.), *Applications of diamond films and related materials, 3rd International Conference*, 1995, p. 551.
[9] D.C. Harris, Development of CVD for IR optical applications, *Statue report and summary of properties, Rep nr NAWCWPNSTP 8210*, 1994 (Naval Air Warfare Centre Weapons Div, China Lake, CA).
[10] M.D. Drory, R.J. McClelland, F.W. Zok and F.E. Heredia, *J. Am. Ceram. Soc., 76* (1993) 1387.
[11] P. Fallon, Ph.D. thesis, 1993.
[12] J.E. Field, in J.E. Field (ed.), *The properties of natural and synthetic diamond*, Academic Press, London, 1992, p. 484.