ELSEVIER

Diamond and Related Materials 5 (1996) 658–663

DIAMOND AND RELATED MATERIALS

Young's modulus of diamond-coated fibres and wires

E.D. Nicholson [a], T.W. Baker [b], S.A. Redman [a], E. Kalaugher [c], K.N. Rosser [a], N.M. Everitt [c], M.N.R. Ashfold [a], P.G. Partridge [d]

[a] *School of Chemistry, University of Bristol, Cantock's Close, Bristol BS8 1TS, UK*
[b] *Department of Physics, University of Bristol, Cantock's Close, Bristol BS8 1TS, UK*
[c] *Department of Aerospace Engineering, University of Bristol, Cantock's Close, Bristol BS8 1TS, UK*
[d] *Interface Analysis Centre, University of Bristol, Cantock's Close, Bristol BS8 1TS, UK*

Abstract

Diamond-coated fibres and wires were produced by hot filament chemical vapour deposition (HFCVD) of diamond on a variety of core materials including tungsten (W) and silicon carbide (SiC). Fibres with a diamond volume fraction exceeding 95% have been produced. Three different methods of measuring the fibre Young's modulus (a resonance method, a bend test and a tensile test) are presented, together with recent results. Possible applications for such fibres include reinforcements in metal matrix composites (MMCs).

Keywords: Young's modulus; Diamond; Fibres and wires; Metal matrix composites

1. Introduction

Diamond-coated fibres and wires have become a subject of some interest over the past few years due to an ever increasing number of possible applications. Diamond's impressive mechanical properties (high hardness, wear resistance and Young's modulus) and extreme thermal conductivity, chemical inertness and electrical insulating properties suggest a whole range of uses. Those presently under consideration include incorporation into grinding/polishing wheels, reinforcements for composites and thermal conductors.

In general, two approaches to the production of diamond-coated fibres and wires have been considered. Firstly, the properties (e.g. thermal and mechanical) of existing fibres can be improved by a diamond coating. Composites incorporating these fibres can be produced with different properties by varying the morphology, composition and thickness of the diamond coating and varying the volume fraction of the reinforcement. A Ti-alloy composite containing diamond-coated fibres has been produced [1]. To produce a composite, the diamond/SiC fibres were individually coated with Ti-alloy matrix material using physical vapour deposition, after which they were consolidated by hot vacuum pressing at 900 °C.

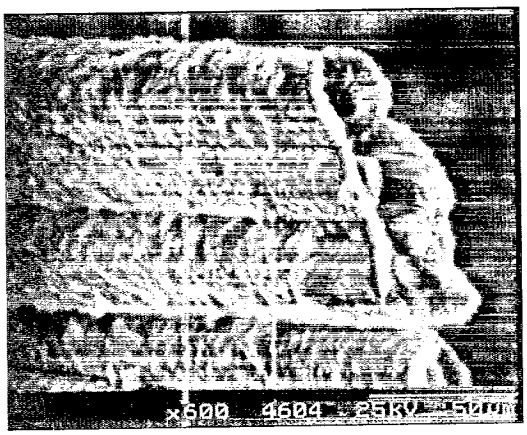
In the second approach, the diamond coating is the matrix, and the core the reinforcement, thus producing a fibre-reinforced diamond matrix [2]. This should substantially increase the fracture toughness of diamond, which is intrinsically low (approximately 5 MPa $m^{1/2}$). Fig. 1(a) shows the consolidation of alumina-based (Nextel) fibres by diamond coating. These fibres, originally part of a tow, were thinned out to produce almost a monolayer prior to coating. A two-dimensional titanium mesh has also been coated (Fig. 1(b)). By using a finer mesh and/or longer deposition times, it may be possible to fill the mesh to produce a reinforced composite sheet.

In this paper, the interest is in new lighter, stronger, stiffer materials for the aerospace industry, particularly metal matrix composites (MMCs) reinforced with continuous unidirectional fibres. Replacing existing reinforcements (such as SiC and W) with diamond-coated fibres or wires would significantly increase the stiffness-to-weight ratio of the composite. Candidate matrix materials include aluminium, magnesium, copper and titanium.

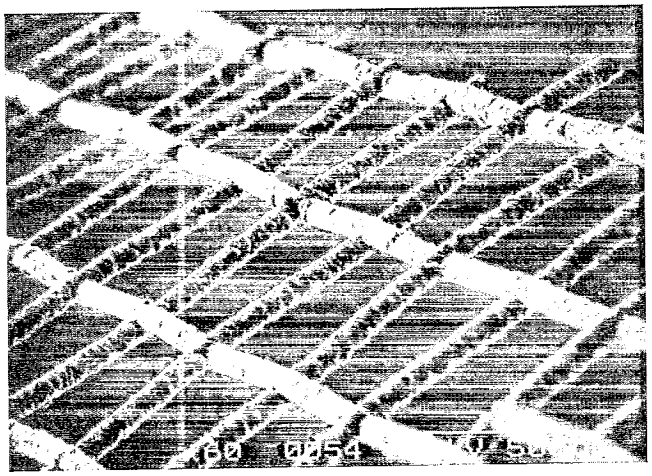
2. Production of diamond-coated fibres and wires

Diamond-coated fibres have been produced by both microwave plasma and hot filament chemical vapour deposition (HFCVD) on a variety of substrates including SiC [2–5], W [4,5], copper [5] and carbon [5–7].

SSDI 0925-9635(95)00361-4



(a)



(b)

Fig. 1. (a) Nextel (alumina-based) fibres (diameter, 10 μm) which have been joined together along their length by the growing diamond film. (b) A 20 μm diameter two-dimensional tungsten mesh (wire separation, approximately 0.5 mm) coated with diamond 30–40 μm thick. At the crossover points of the mesh the W wire is embedded in diamond forming a ridged fibre array.

The substrate fibres and wires in this investigation were diamond coated by HFCVD (reactor made by Thomas Swan and Co., Cambridge, UK). The sample wires (14 per run) surround a vertically held tantalum filament, at a distance of 5–6 mm from the filament. Samples with a working length of 100–120 mm are produced. The substrate temperature is determined by the filament temperature, which is measured using an optical pyrometer. Depending on the conditions, the deposition rate can vary from 0.5 to 1 μm h^{-1}. To date, deposition has been carried out for up to 105 h, producing fibres with over 95% volume fraction of diamond (Fig. 2). The deposition conditions for the samples tested by resonance are as follows: pressure, 20 Torr; 0.75 CH_4 in H_2; filament temperature, 2150–2180 °C. Deposition was carried out for 103 h. Those tested by the tensile test differed in the methane concentration used (0.5%) and the deposition time (72 h).

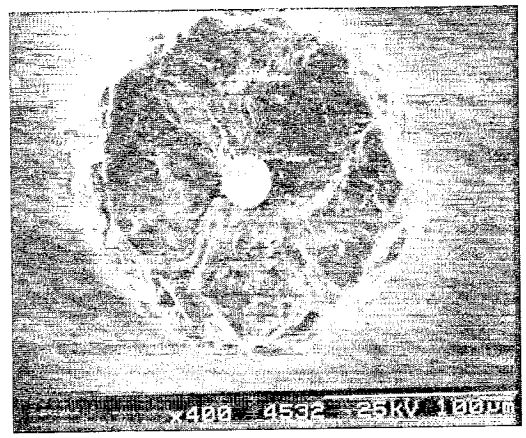


Fig. 2. A 25 μm diameter W wire coated with approximately 65 μm of diamond, producing a fibre with a volume fraction of diamond of about 97%.

3. Young's modulus measurement

Three different approaches to measuring the Young's modulus have been considered: resonance methods, bend testing and tensile testing. The basis of resonance methods [8,9] is the measurement of the natural or resonant frequency of vibration of a sample which, together with a knowledge of the sample density and dimensions, yields the Young's modulus. The most accurate resonance methods impose the minimum constraint on the sample. Resonance techniques applied to fibres have concentrated on methods where the fibre is clamped rigidly at one end and excited into vibration. Such techniques introduce large length errors. A more accurate method involves supporting the sample at nodes for fundamental flexural vibration (0.224 of the length from each end). The sample is excited into resonance and the resonant frequency detected. The resonant frequency relates directly to the Young's modulus.

Bend testing methods have been used widely to measure wire and fibre stiffness [10,11]. The basis of bend testing is the measurement of the deflection, or change in radius of curvature, of a beam by application of a known load. This information, together with the sample dimensions, is used to calculate the modulus. The load can be applied centrally whilst the sample is supported at both ends (three-point bend), or at one end of the sample with the other rigidly held (two-point bend).

The most widely used technique for measuring the modulus of stiff fibres is the tensile test. This involves measuring the load–elongation behaviour of samples of known cross-sectional area [11,12]. In general, difficulties have been experienced in gripping the sample successfully and measuring the small strains experienced by stiff fibres.

4. Experimental details

4.1. The resonance method

The technique of supporting a sample at nodes for a particular mode of vibration has previously been applied to larger samples [8,9]. Its applicability to smaller samples is under test here. The Young's modulus can be determined from either the flexural or longitudinal vibrations; however, it is easier to excite the flexural vibration and so this was employed. For a homogeneous, isotropic cylindrical rod, the Young's modulus and the fundamental frequency of the flexural vibration are related by

$$E = 1.28868 \frac{\rho l^4}{d^2} f_{res}^2 \quad (1)$$

where d, l and ρ are the fibre diameter, length and density respectively and f_{res} is the resonant frequency.

The experimental apparatus is shown in Fig. 3(a). The sample mount consists of knife edges (the sample supports), which are mounted on the edges of micrometer callipers. The distance between them is set to that required for resonance for a particular sample length. Sample excitation is achieved using an audio speaker, located directly beneath the centre of the sample. The frequency of excitation is tuned using a variable frequency function generator which is connected to the speaker via an audio amplifier. Resonance detection for specimens with a fairly smooth reflecting surface is carried out by interferometry. Alternatively, for specimens with a rough surface, such as many of the diamond-coated fibres and wires produced in this investigation, resonance is detected using position detection. This works using the same apparatus but omits the need for the reference arm of the interferometer.

The resonant frequency was measured as a function of length. Each sample was tested at 8–10 different lengths. The frequency range for the fibre samples tested was 100–300 Hz. Calibration of the equipment was carried out using Pyrex (borosilicate glass) rods of know properties. The sample details and moduli measured are given in Table 1.

4.2. The bend test

The two-point bend test was chosen over the three-point bend test as it has a wider applicability to the samples under investigation. The deflection under load is greater and therefore it is easier to measure. Much shorter samples can be used, allowing very small diameter fibres (10 μm), which cannot be coated in great lengths, to be tested. Also, it should be possible to test fibre cores, which tend to sag at longer lengths.

The proposed experimental apparatus is shown in Fig. 3(b). The fibre is clamped in position as shown. A low load compression load cell (0–200 g) is attached to a non-rotating micrometer at one end and a displacement arm at the other. On the end of the displacement arm is a horizontally mounted rod of small diameter. The length of the sample is determined by the position of another small diameter rod placed under, and just touching, the fibre, and the position at which the load is applied. Displacement of the fibre is produced by the micrometer. The load cell simultaneously measures the load. As the displacement is large (10–40 mm) in comparison with the total compression distance of the load cell (approximately 10 μm), the assumption that the fibre displacement is equal to the micrometer displacement will produce only a small error. This technique is presently under construction.

4.3. The tensile test

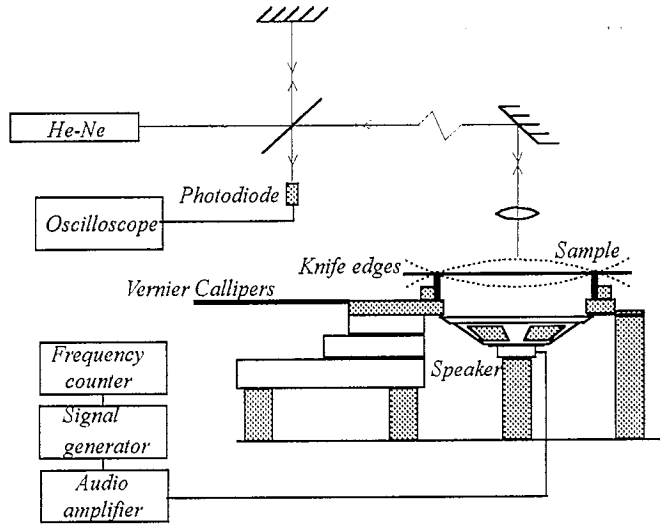
The tensile test can measure both the stiffness and the fracture stress of fibres. Due to the very small strain to failure of diamond-coated fibres, traditional methods of correcting for the compliance of the load cell are inadequate. Therefore a direct method of measuring the fibre strain is used [13].

The fibres are mounted on test cards using epoxy resin producing a gauge length of approximately 70 mm. A 14 μm diameter marker fibre is attached at each end of the fibre, using cyanoacrylate (Fig. 3(c)). The card is gripped in a Hounsfield H5000M tensile tester, with a 500 N load cell. After the sides of the test cards are cut, the lower grip of the tensile tester is moved downwards at 0.5 mm min^{-1} until the fibre breaks. The motion of each marker fibre shadow is tracked using a CCD linear array camera. The relative motion of the two markers during loading gives the fibre extension, without any contribution from the machine compliance or slip of the fibre through the epoxy resin. The original length of the fibre can be calculated from the initial marker positions. The gathering of the load and extension data is currently not synchronized, and so the stiffness is calculated from the fracture stress and strain to failure (Eq. (2)) rather than the slope of the stress–strain curve

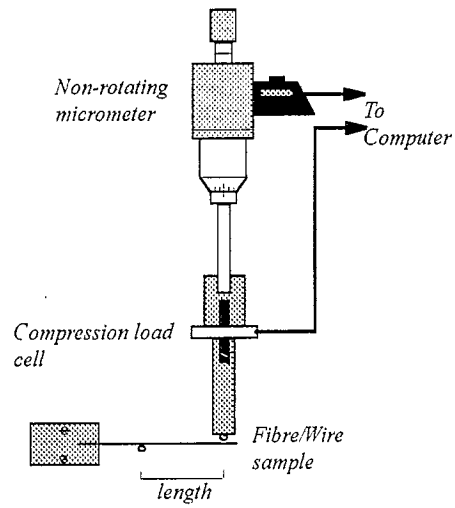
$$E = \frac{Fl}{A\delta l} \quad (2)$$

where E is Young's modulus, F is the load at fracture, l is the original length and δl is the extension at fracture.

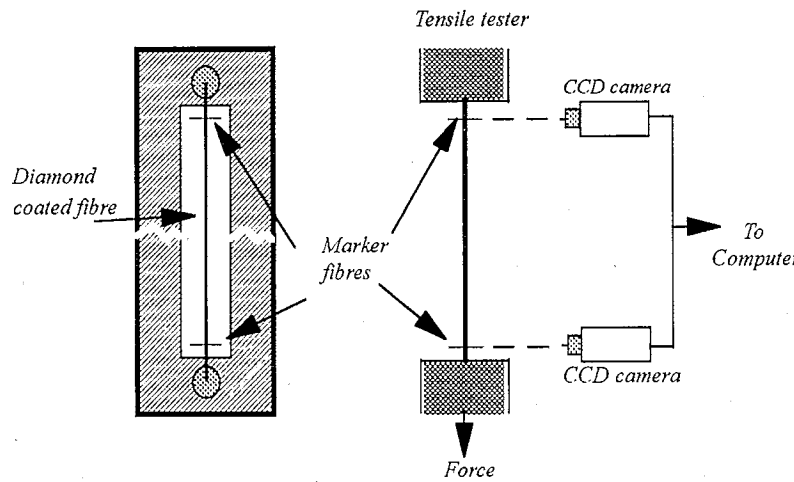
Tensile testing produces modulus values for the composite fibre. To obtain separate core and coating moduli, it is assumed that the rule of mixtures applies. Details of the samples tested and the values measured are given in Table 2.



(a)



(b)



(c)

Fig. 3. (a) The resonance test apparatus. (b) The bend test. (c) The tensile test.

Table 1
Sample details and modulus measured by the resonance test

Sample type	Average diameter (μm)	Film thickness (μm)	Volume fraction diamond (%)	Young's modulus (GPa)
Pyrex standard	3500	—	—	61.4 61.0[a]
W (50 μm diameter)/diamond				
1	217.0	83.6	94.7	898
2	219.5	84.7	94.8	851
3	226.0	88.0	95.1	900
4	211.0	80.5	94.4	848

[a] Data Book value (*PYREX® Glass Data Book*, Jobling, Sunderland, 1964, p. 2).

Table 2
Sample details and modulus measured by the tensile test

Sample type	Average diameter (μm)	Film thickness (μm)	Volume fraction diamond (%)	Young's modulus (GPa)
BP Sigma SiC				
1	102	—	—	362
2	98	—	—	357
W (125 μm diameter)/diamond				
1	205.3	40.15	62.9	892
2	204.2	39.6	62.5	810
3	204.2	39.6	62.5	713
4	196.8	35.9	59.7	772
5	206.0	40.5	63.2	789
6	205.4	40.2	63.0	684
7	203.0	39.0	62.1	668

5. Results and discussion

5.1. The resonance method

As the fibres are predominantly diamond (approximately 95%), the core should have little effect on the overall modulus. The fibres were therefore treated as homogeneous. The average value of the fibre modulus (874 ± 45 GPa, for a 95% confidence interval) is more than a factor of two greater than that of SiC fibre (400 GPa). If these fibres were coated with 10 μm of Ti-alloy (modulus 115 GPa) and consolidated into a composite, a composite with 84% diamond would be produced. Using the rule of mixtures, the composite modulus works out at approximately 750 GPa, which is over 3.5 times greater than the modulus of current Ti-alloy/SiC fibre composites (approximately 206 GPa) with about 30% fibre volume fraction.

A problem with fibres with such a high volume fraction of diamond is their lack of toughness. This may be remedied by producing graded fibres, by renucleating several times during a growth run to avoid a large grain size and by producing interfaces which should act as barriers to crack propagation.

For samples with a lower volume fraction of diamond (less than 90%), the core properties would have to be taken into account. Eq. (1) would require modification in this case. Work is presently underway to develop this equation.

5.2. The tensile test

From a batch of ten diamond-coated fibres, two broke at the clamped ends; this is thought to be due to compressive and bending forces produced by imperfect sample mounting. A third fibre gave a very low modulus value, which appeared to be due to cracking of the film during handling. These results were discarded. The average modulus for the remaining fibres (Table 2) was 761 ± 100 GPa (for a 95% confidence interval). Using the rule of mixtures, the average modulus calculated for the diamond coating from this fibre modulus is 973 GPa.

Considerable problems are caused in the handling and mounting of fibres due to their extremely brittle nature. In the future, two batches grown under identical conditions will be tested to give a reasonably large sample of valid results.

6. Conclusions and future work

The total experimental errors for the resonance test and tensile test were calculated to be 10% and 15% respectively. As different samples were used in each test, the results cannot be compared directly. However, the fibres with a higher volume fraction of diamond used in the resonance test were expected to give higher modulus values than those tested by the tensile test. Plans for the future include the testing of every fibre batch using both of these techniques and also bend testing, producing fibre batches with different morphology and film thickness for testing, and developing a more exact analysis for calculating the Young's modulus of fibres with a volume fraction of diamond of less than 90%. In addition, MMCs incorporating these fibres will be produced, and their mechanical properties measured.

Acknowledgements

We thank the EPSRC and Bristol University for financial support.

References

[1] E.D. Nicholson, J.E. Field, P.G. Partridge and M.N.R. Ashfold, *Mater. Res. Soc. Symp. Proc.*, in press.
[2] M.D. Drory and R.J. McClelland, in M. Sakai et al. (eds.), *Advanced Materials (III/B), Trans. Mater. Res. Soc. Jpn.*, Vol. 16B, 1994, pp. 869–871.
[3] M.D. Drory, R.J. McClelland, F.W. Zok and F.E. Heredia, *J. Am. Ceram. Soc., 76* (1993) 1387.
[4] B. Marayama, R.K. Everett, L.S. Cook, A. Morrish, P. Natishan, P. Pehrsson and A.E. Edelstein, in A.R. Busell, F.F. Jamet and A. Massiah (eds.), *5th European Conf. on Complex Materials, Bordeaux, France, 7–10 April, 1992*, 1992, pp. 715–720.
[5] A.A. Morrish, J.W. Glesener, M. Fehrenbacher, P.E. Pehrsson, B. Maruyama and P.M. Natishan, *Diamond Relat. Mater., 3* (1993) 173.
[6] J. Ting and M. Lake, *J. Mater. Res., 9* (3) (1994).
[7] J. Ting and M.L. Lake, *J. Min. Met. Mater. Soc., 46* (1994) 23.
[8] *ASTM Designation C623-71*, ASTM, 1971, p. 229 (reapproved 1985).
[9] S. Spinner and W.E. Tefft, *Am. Soc. Test. Mater. Proc., 61* (1961) 1221.
[10] C.J. Burstone and A.J. Goldberg, *Am. Orthod., 84* (1983) 95.
[11] J.D.H. Hughes, *Br. Ceram. Trans. J., 87* (1988) 181–188.
[12] *ASTM Designation D3379-75*, ASTM, 1975 (reapproved 1989).
[13] J.E. Emrick and H.L. Gegel, *ASD Technical Report 61-168*, (Aeronautical Systems Division, Wright-Patterson Air Force Base, OH, December, 1961).