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THE PRODUCTION OF CVD DIAMOND-COATED WIRES AND FREE-STANDING DIAMOND TUBES

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Abstract

Using the hot filament method, diamond films have been deposited uniformly onto the outside surfaces of thin metallic wires of varying composition. Two methods have been used to fabricate diamond tubes: (a) removal of the metal core using a chemical reagent and (b) creating the tube *in situ* by CVD onto a coiled wire.

1. INTRODUCTION

The ability to coat metallic wires with diamond to form diamond fibres with very high elastic modulus may have important applications for advanced composite materials [1)]. Metal matrix composites (MMCs) based on Al, Mg or Ti and diamond fibres, may offer the advantages of being lightweight and strong combined with the stiffness and rigidity required for aerospace applications. Successful fabrication of diamond-coated wires has recently been reported [2-4)], although very little data on their mechanical properties are yet available. In this paper we discuss some of the important considerations when fabricating diamond-coated wires, and also demonstrate an innovative method of producing free-standing diamond tubes.

2. EXPERIMENTAL

2.1 Hot filament growth conditions

To diamond-coat wires, it is usual to suspend the filament and the wires vertically, with each wire being kept straight and under tension by using a small weight. The wires can be positioned in two geometries:

(a) Inside the coils of the filament [2,3)]. This allows wire diameters up to a few mm to be coated, but is difficult to control and has poor run-to-run reproducibility.

(b) Parallel to but outside the filament [4)]. This has the advantage of being controllable and reproducible as well as providing the ability to coat many wires simultaneously. However this geometry is limited to coating wires of diameter <200 μm otherwise shadowing effects occur and the coating can become non-uniform.

We use a commercially available hot filament reactor (Thomas Swan & Co Ltd, Cambridge, U.K.) which is specifically designed for coating up to 20 wires simultaneously of length 15 cm. The wires are spring-loaded to keep them straight and taut during deposition, and are positioned a distance of 6 mm from the filament. We use a 1% methane in hydrogen gas mixture, at a constant pressure of 20 Torr (2700 Pa), with a Ta filament temperature of 2000 C, and a substrate temperature of around 900 C, giving a diamond deposition rate of about 0.5-1 μm h^{-1}.

3. RESULTS

3.1 Diamond Coating of Wires

1) Copper.

Since Cu does not form a carbide, diamond coatings do not adhere and readily exfoliate. It is, however, possible to grow thick coatings of diamond on thin (<50 μm) Cu wires but not thin coatings on thick Cu wires. [3)]

2) Titanium.

Ti forms a carbide layer, and since there is a significant solubility for carbon in Ti, the carbide layer continues to grow throughout the diamond deposition [5)]. If the deposition time is too long the increasingly thick TiC layer eventually causes cracks in the diamond coating. [3)]

3) Tungsten.

W also forms a carbide layer during the initial nucleation stages of CVD but, compared to Ti, carbon diffusivity is much lower. Therefore after an initial induction period the WC layer thickness remains constant at <3 μm independent of deposition time [6)] (Fig.1). Thus thick diamond films can be readily deposited onto W wires of various diameters [3)] (Fig.2).

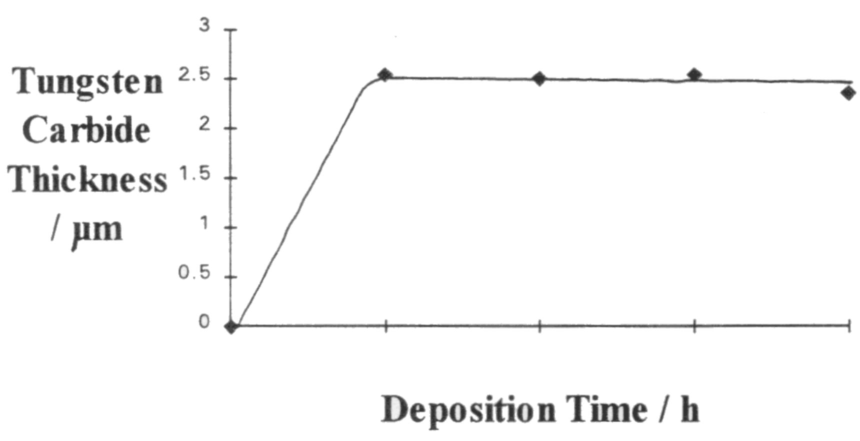


Fig.1. Thickness of interfacial tungsten carbide layers measured by scanning Auger analysis on W wires at different diamond deposition times.

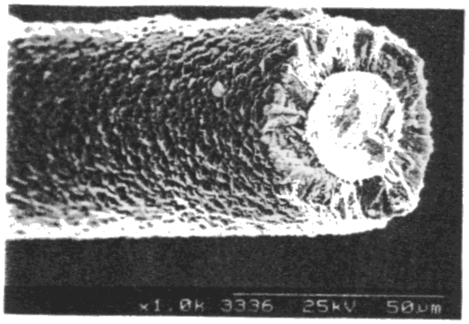


Fig.2. Diamond-coated 25 μm diameter W wire. The wire length was 10 cm, with a diamond thickness of about 17 μm.

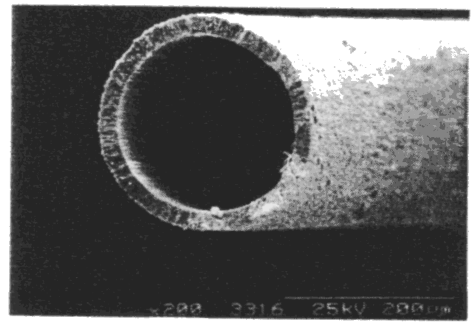


Fig.3. A free-standing diamond tube made by etching out the W core. Inside diameter of the tube is 250 μm, with a 22 μm wall thickness.

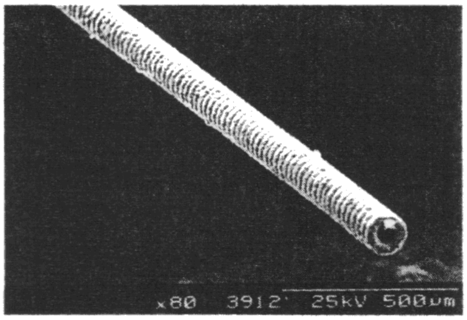
3.2. Production of Free-standing diamond tubes

1) Chemical Etching.

This involves etching out the metal core using a suitable chemical reagent (e.g. conc nitric acid for Cu, fluorinated HCl solution for Ti, and hot (100 C) 30% H_2O_2 solution for W). This method works well on diamond-coated W wires (see Fig.3), but with Ti often leaves a carbide residue [3]. Furthermore it is unclear whether such chemical etching will succeed for long lengths of tube, since the etchants and reaction products may not be able to diffuse distances of several cm through tubes of such small diameter. However, when successful, this method may be useful to produce low density short diamond fibre MMCs. [1]

2) Coils

An alternative method involves winding a W wire (of diameter typically 25 μm) around a former (usually another W wire of diameter 25-100 μm) into a helix. Diamond CVD onto such a helix can result in diamond fibre springs [1], which may be useful for micro-engineering applications. If, however, the coils of the helix are closely spaced, such that their pitch is only a few μm or even touching, then subsequent diamond CVD results in the coatings of neighbouring coils fusing together to form a continuous tube (see Figs.4 and 5). This has the advantage of producing the tube *in situ* - without the need for dangerous acids - thereby allowing tube-lengths of several cm to be achieved readily. The W coil remains embedded inside the tube walls.



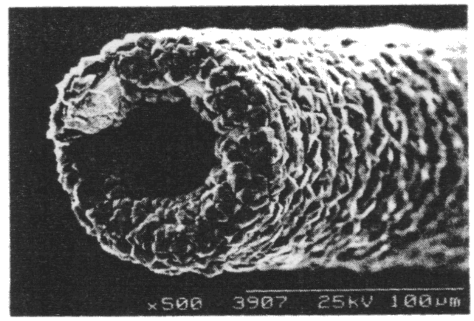


Fig.4. Diamond tube produced by CVD onto a W coil.

Fig.5. Close up of a tube produced by CVD onto a W coil, showing the 'fusing together' of the coils.

4. ACKNOWLEDGEMENTS

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5. REFERENCES

1. P.G. Partridge, P.W. May and M.N.R. Ashfold: to appear in Mater. Sci. Technol., 1994.
2. P.W. May, C.A. Rego, R.G. Thomas, M.N.R. Ashfold, K.N. Rosser, P.G. Partridge and N.M. Everitt:, Proc. 3rd Int. Symp. Diamond Mater., Honolulu, Hawaii, 1993, The Electrochemical Soc. (1993) p955.
3. P.W. May, C.A. Rego, R.M. Thomas, M.N.R. Ashfold, K.N. Rosser, and N.M. Everitt: Diamond and Rel. Mater., **3** (1994) 810.
4. P.W. May, C.A. Rego, R.M. Thomas, M.N.R. Ashfold, K.N. Rosser, P.G. Partridge and N.M. Everitt: J. Mater. Sci. Lett., **13** (1994) 247.
5. S.S. Park and J.Y. Lee: J. Appl. Phys., **69** (1991) 2618.
6. Q.S. Chia, C.M. Younes, P.G. Partridge, G.C. Allen, P.W. May and C.A. Rego: J. Mater. Sci., in the press.