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Cylindrical CVD diamond as a high-performance small abrading device

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Abstract

Columnar grain structure studies have been carried out on a small-curvature radius cylinder substrate for the development of small abrading devices. An enhanced hot filament-assisted technique was used. The substrate holder has a movable mechanism magnetically coupled to a d.c. motor placed outside the reactor chamber. Free-standing diamond films as thick as 1 mm were obtained on molybdenum wire substrates. The substrates varied from 500 up to 1200 μm diameter and were as long as 50 mm. A relationship of the curvature radius of the substrate surface with the growth rate and the spread of the column volume has been observed. A preferential diamond (111) surface morphology has been obtained, which is strongly dependent on diamond growth parameters, including the substrate position and its angular velocity. Grain size up to around 0.15 mm was obtained. In this work we also report an interesting application involving the characteristics of the (111) diamond surface as a small abrading device. Some results of cylindrical diamond burrs and diamond drills for non-ferrous wear are shown. © 1998 Elsevier Science S.A. All rights reserved.

Keywords: Odontological burr; CVD diamond; Grain size; Abrading

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1. Introduction

CVD diamond, with its singular properties and the simple synthesis processes, became an interesting area of investigation. The challenge in order to explain the basic of the growth mechanism has been hard work for many researchers. However, the spreading of application areas using CVD diamond with innovative characteristics shows the main effort in terms of studies and investment, and how different kinds of new devices can be created for industrial processes [1,2]. High-quality diamond coating on pre-shaped parts and synthesis of free-standing shapes of diamond are now a reality. Polycrystalline diamond films have been deposited on a variety of substrates, including metals, refractory metals, semiconductors and ceramics [2]. Most applications of CVD diamond are categorized as advantageous substitution of traditional products. The novelty products are the ones only possible due to the advent of CVD diamond technology. Applications of CVD diamond are far beyond the traditional technology. CVD diamond-coated cutting tools, abrading devices, heat sinks, optical coatings, substrates for multichip module technology (MCM) and electron field emitters, is only a small list of items in a profitable market [1]. This will guarantee an important economical impact of this emerging technology.

More specifically, columnar growth studies became an attractive area of investigation in which the surface polycrystalline morphology is used for abrading applications [3]. The diamond burr for odontological and related use, for instance, is a new device in the emerging technology, where a conical shape of a (111) morphology was widely studied and a scaling-up for industrial productions was also calculated [4].

CVD diamond production cost is under US$ 10.00 per carat [5], which is still considered to be high, but is already quite competitive in many areas. Cost reduction is previewed prospectively. As a consequence, the economical scale-up of CVD diamond is considered a viable processing alternative. Even though the production cost of the CVD diamond burr is much higher than conventional ones, its performance is much superior and the prices need to be evaluated by a cost/benefit ratio. The CVD diamond burrs clearly show a large advantage. For studies of spherical and conical shape, a conventional hot filament-assisted reactor (HFCVD) was used; the growth parameters and substrate shape and holder have been described elsewhere [3]. This work presents the investigation of a new tip shape: cylindrical. For a cylindrical shape some improvements have been accomplished. A new concept of substrate

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holder and new parameters of diamond growth have been investigated. Temperature, grain size, gas concentration, thickness, stress, and diamond quality have been studied as a function of the substrate diameter and length.

2. Experimental procedures

A typical diagram of the apparatus for HFCVD is presented in Fig. 1. The reactor is a water-cooled vertical wall of a stainless steel tube, 60 mm in diameter and 250 mm in length, and with easily changeable end flanges. Four flanges are placed close to the reaction zone. Two of these flanges were used as windows in order to allow the observation of the filament and substrate position. The other two were used as the holder for the substrate end and for a magnetic coupling feedthrough.

The magnetic coupling feedthrough was used for substrate rotation. A polished molybdenum rod with a diameter of 0.55–1.2 mm and of 70 mm in length was used as a substrate without any kind of other surface treatment. A set of two straight and parallel tungsten filaments of 0.14 mm diameter and separated by a distance of 3 mm was used. This set of filaments allowed the best thermal distribution close to the rotating substrate. The distance between substrate and the set of filaments were varied up to 10 mm. The temperature of the hot filaments and the temperature of the substrate were maintained at about 2277 and 777°C, respectively. These temperatures were monitored by an optical pyrometer and by a thermocouple, respectively. The gas components were 2 vol.% methane, and hydrogen at a purity better than 99.99%. The total flow rate of the gas mixture of 100 sccm and a typical 50-Torr pressure inside the reactor was kept constant for all experiments.

A roughly homogeneous film of cylindrical geometry was obtained after each growth run. In order to obtain a diamond tube, some of these films were submitted to acid attack of HNO_3/HCl (3:1), for 2 h at 70°C, to remove the molybdenum rod. For diamond tube cutting, a pulsed copper vapor laser with 15 W power and 10 kHz repetition rate was used. Each burr tip was brazed on stainless steel rods to obtain the final burr. The burrs were tested by drilling 2-mm thick glass plates.

3. Results and discussions

First of all, we carried out some experiments in order to find the best conditions for growing diamond films on a cylindrical surface. The experimental investigations were carried out by growing free-standing films on the tube and measuring growth rate, film quality, thickness, grain size and total stress as a function of the tube length. A straight molybdenum rod of 0.5–1.2 mm diameter and 70 mm long in rotation movement was used. The angular speed investigated from 0.2 to 10 rpm did not influence the diamond film properties. The temperature distribution on the substrate surface, in the range of 50 mm, was measured for a fixed distance between the substrate and the set of filaments (around 3 mm), for molybdenum rods of 500 μm, and 0.9 and 1.2 mm, as shown in Fig. 2.

Fig. 2 shows the substrate temperature change from the end to the center, which is more accentuated for molybdenum wire with smaller diameter. This distribution is due to the heat conduction through the substrate and the heating distribution from the hot filament. Also, the gas temperature close to the cooled reactor wall is lower than in the center. The substrate temperature does not change much in the range of 30 mm close do the center.

For the remaining of this work, only the 0.9-mm diameter molybdenum rod substrates were considered. This diameter is more convenient for our application studies. A Raman analysis was performed in order to

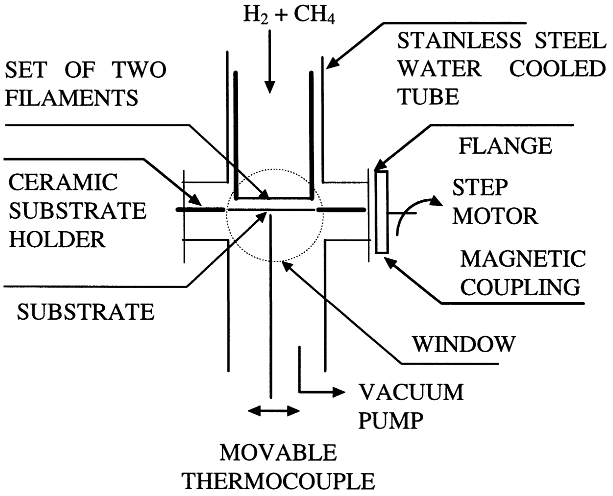

Fig. 1. Schematic diagram of the HFCVD for diamond deposition on a cylindrical substrate.

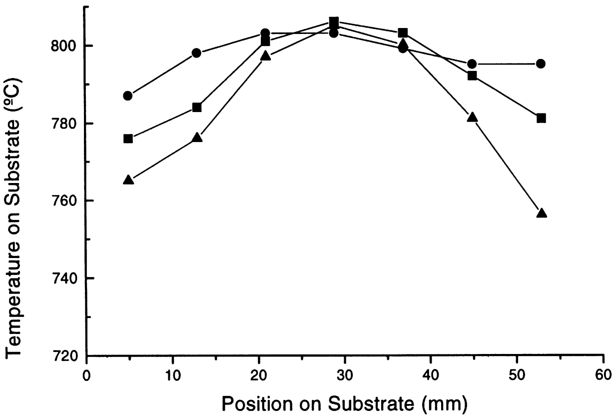

Fig. 2. Temperature distribution as function of substrate length for (●) 1.2 mm, (■) 0.9 mm and (▲) 0.5 mm diameter.

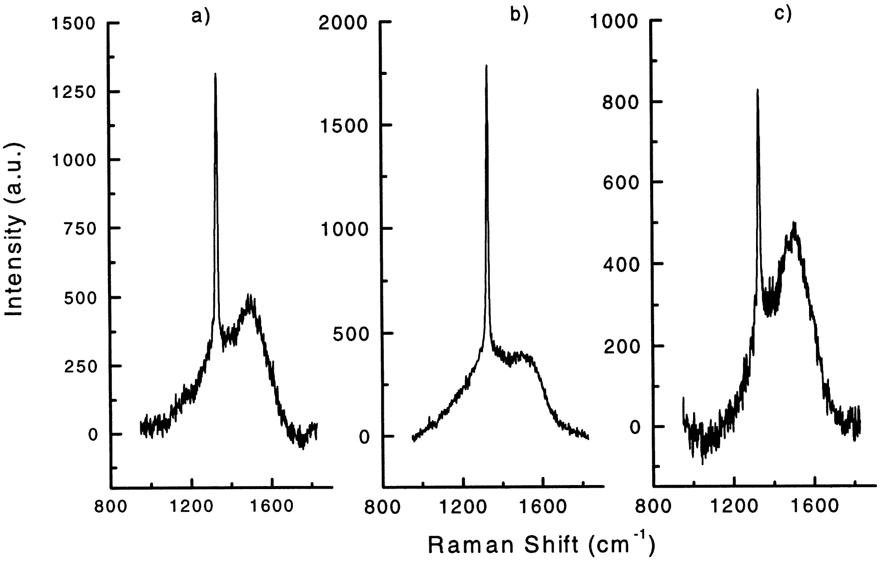

Fig. 3. Raman spectrum from a tube of free-standing diamond film at the two ends (a and c) and at the centre (b).

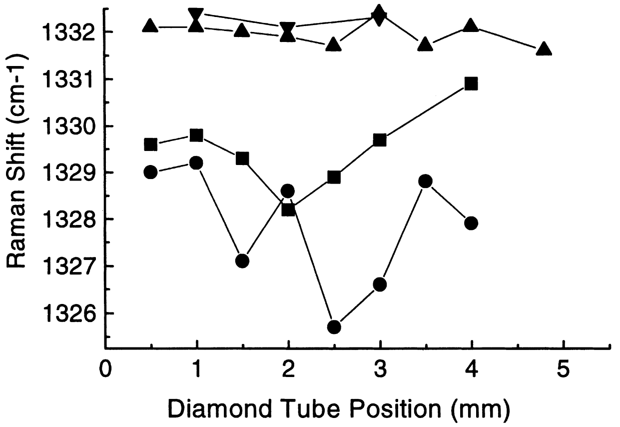

Fig. 4. Raman shift for diamond film as a function of its length for (■) T1, (●) T2, (▲) T3 and (▼) T4.

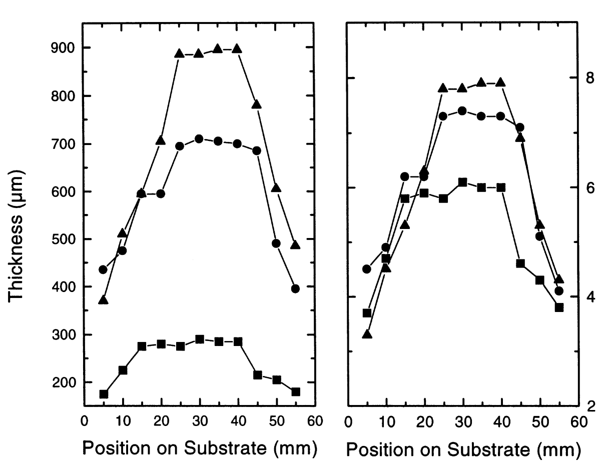

Fig. 5. The dependence of the thickness and the growth rate with the diamond film length for (■) 47, (●) 94 and (▲) 113 h of diamond growth.

evaluate the quality and total stress of the CVD-diamond cylinder film. Fig. 3 shows the Raman spectra with a clear peak centered at 1330 cm^{-1}, and a wide peak centered in 1550 cm^{-1}. The spectrum of the center position shows a better film quality than in both ends. Several samples were analyzed and the results are quite similar.

The Raman analyses also give information about the total stress [6]. If the diamond Raman peak is shifted to a wavenumber higher than that natural diamond peak (1332 cm^{-1}) the stress is compressive, if it is shifted to the other side the stress is tensile. Each unit of cm^{-1} shift from 1332 cm^{-1} corresponded to 2.47 GPa. Fig. 4 shows the Raman shift along the diamond tube. Four samples were analyzed. T1 had a thickness at the center of around 0.25 mm, and T2 of around 0.70 mm on the molybdenum wire substrate. T3 is sample T2 after dissolving the molybdenum wire. Sample T4 shows a curve obtained from small pieces (5 mm long) from sample T3 after laser cutting and brazing on stainless steel rods.

The diamond film presents tensile stress for T1 and T2 samples. At the center of the diamond film the stress is higher than at the ends. This can be explained because of the larger thickness in the center. Also, the stress is higher for T2, showing that the stress is higher for thicker diamond films on a cylindrical substrate. The stress is lower for sample T3 than T1 and T2. The stress for T4 is quite similar to T3, and both have a Raman shift very close to that of natural diamond. This indicates that stress is relieved after the removal of the molybdenum rod. The thickness and growth rate along the free-standing CVD diamond film for three different growth times are presented in Fig. 5.

The thickness and growth rate clearly increase from the ends to the center of the cylindrical diamond film. Certain-

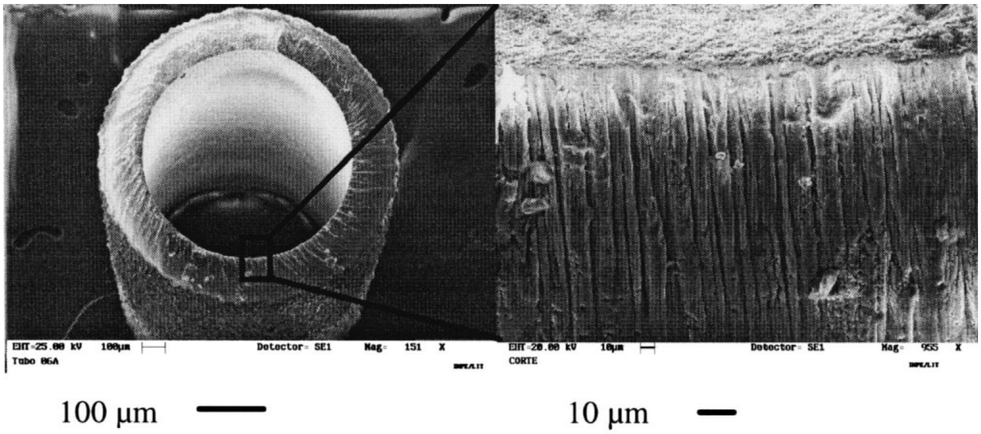

Fig. 6. Morphology of the diamond film and a detail of the columnar growth.

ly this phenomenon occurs during growth due to inhomogeneous temperature distribution along the cylindrical diamond film. Probably there is also some influence of inhomogeneous gas temperature close to the water-cooled walls.

Fig. 6 shows the morphology with preferential (111) structure and a detail of the cross-section of the diamond tube with an apparent columnar structure.

The diamond tubes were laser cut to obtain the small (5 mm) cylindrical burr tip. The columnar structure is very apparent in the laser-cut region. The heat conduction is faster during the laser-cutting procedure in the column growth direction than across the grains.

Finally, in order to compare the CVD diamond and conventional burr some wear testing was performed. Fig. 7 shows, after test, scanning electron micrographs of a conventional cylindrical burr and the burr developed in this work. The conventional burr is shown after only 5 min of water-cooled drilling and the CVD diamond burr is shown after 80 min. It was observed that the conventional one lost almost all the diamond grain, while the CVD diamond did not change the morphology, remaining as new.

4. Conclusions

We have investigated a series of growing parameters of free-standing CVD diamond film on cylindrical surface in rotation movement. Free-standing CVD diamond tubes from 0.50 up to 2.5 mm diameter and from 0.15 up to 0.90 mm thick was obtained with good quality. It was possible to obtain grain size for use as a cylindrical odontological burr and other related uses. Using a substrate with a 60-mm length free for diamond growth, a 50-mm long tube of the free-standing film can be used as a diamond burr. Studies of morphology, diamond quality and growth rate along the free-standing cylindrical tube have shown that technically a new device is reached. This also gives us support to get calculations for economical feasibility. It was shown that the total stress decreases after the molybdenum rod is dissolved in a convenient acid. Also, it was verified that the stress remains very low after the diamond tube is brazed on the stainless steel rod. Finally, comparative testing between the CVD diamond and the conventional burr has shown the advantage of the first one, giving us a support to test for industrialization.

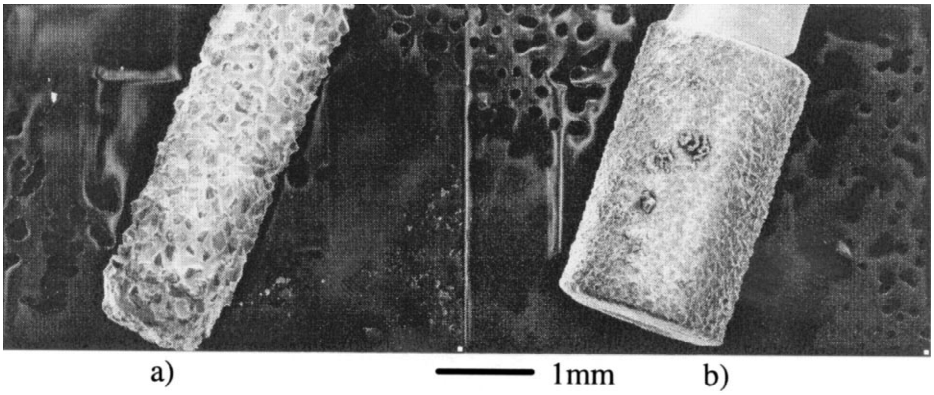

Fig. 7. Comparison between cylindrical conventional burr (a) and CVD diamond burr (b) after drilling test.

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References

[1] J.V. Busch, J.P. Dismukes, A comparative assessment of CVD diamond manufacturing technology and economics, in: K.E. Spear, J.P. Dismuskes (Eds.), Synthetic Diamond: Emerging CVD Science and Technology, John Wiley & Sons, NY, 1994, p. 581.

[2] V.J. Trava-Airoldi, E.J. Corat, V. Baranauskas, Diamond chemical vapour deposition: emerging technology for tooling applications, chapter contribution for the book on advanced ceramics for cutting toll applications, in: J. Low (Ed.), Trans. Tech. Publications, vol. 3, Switzerland, 1997, p. 195.

[3] V.J. Trava-Airoldi, E.J. Corat, A.F.V. Pena, N.F. Leite, V. Baranauskas, M.C. Salvadori, Diamond Rel. Mater. 4 (1995) 1255.

[4] V.J. Trava-Airoldi, E.J. Corat, E. del Bosco, N.F. Leite, Surf. Coat. Technol. 76–77 (1995) 797.

[5] E. Sevillano, J.A. Casey, R. Gat, S. Jin, V.J. Berkman, R.S. Post, D.K. Smith, in: Meeting Program Section of 4th International Symposium on Diamond Materials, The Electrochemical Society Inc. 187th Meeting Program Section, Reno, NV, 1995, p. 137.

[6] G. Heinrich, T. Grogler, S.M. Rosiwal, R.F. Singer, R. Stockel, L. Ley, Diamond Rel. Mat. 5 (1996) 304.