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Chemical vapour deposition of diamond coatings onto molybdenum dental tools

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Abstract

The growth of polycrystalline diamond films onto molybdenum rods and dental burrs by using a new hot filament chemical vapour deposition (CVD) system has been investigated. Negative dc bias voltage relative to the filament was applied to the molybdenum substrate prior to deposition. This led to much improved film adhesion and increased nucleation density. There was a factor of four improvement in the adhesive force from 20 to 80 N when a bias voltage of −300 V was employed to the substrate.

The CVD coated molybdenum dental burr was found to give much improved performance and lifetime compared to the conventional sintered diamond burr. The CVD diamond burr showed no signs of deterioration even after 1000 operations whereas the conventional sintered diamond burrs were ineffective after between 30 and 60 operations. This represents a 30-fold improvement when CVD is applied. CVD diamond growth onto dental burrs has the potential for replacing exciting technology by achieving better performance and lifetime in a cost-effective manner.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Molybdenum is used as a base material for fabricating conventional diamond dental burrs. These burrs are made by embedding synthetic diamond particles into the working surfaces using a binder matrix material containing nickel ions, which can contaminate the oral tissues [1, 2]. The adhesion of the diamond particles depends on the properties of the binder matrix, and under high speeds and extreme abrasive conditions these particles come off easily during application on the patient or during use in the dental laboratory. In addition, the performance and lifetime of these tools is highly variable due to the inhomogeneity of particle size and binder properties.

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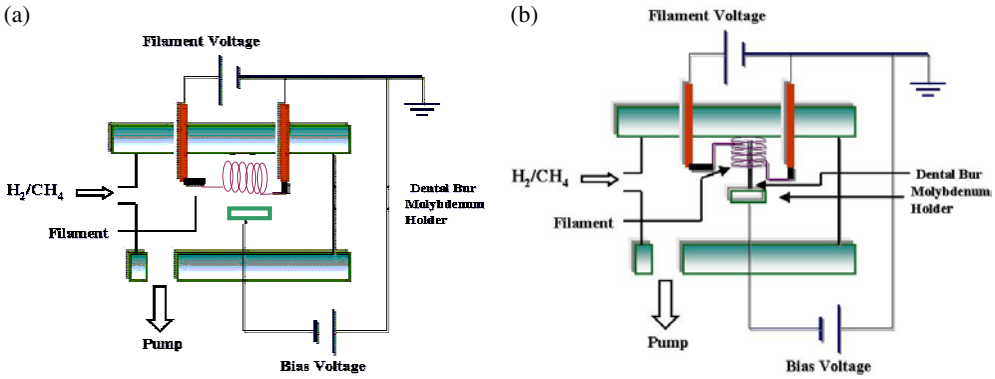

Figure 1. (a) Schematic of the HFCVD system (conventional filament arrangement). (b) Schematic of the HFCVD system (new modified filament arrangement).

This technology dates back to the 1950s and is difficult to automate and the burrs produced have a relatively short lifetime. Therefore, it is highly desirable to have the capacity to deposit adherent diamond films onto the molybdenum surfaces on a large scale economically using a suitable surface treatment process. It is also advantageous to have the capacity to re-coat used dental burrs and restore their original performance. Chemical vapour deposition (CVD) is an established and promising technology that has the potential to deposit diamond coatings onto dental burrs which are adherent, reproducible and economical. The technology is well established for many applications and has the capacity to re-coat used dental burrs.

In this paper, we have investigated the use of CVD technology for coating molybdenum substrates with polycrystalline diamond which may be optimized and scaled to produce diamond coated molybdenum dental burrs economically. A new modified hot filament CVD system has been used in this study to grow diamond films onto molybdenum rods and dental burrs.

The properties of the polycrystalline diamond deposited dental burrs have been investigated using scanning electron microscopy and Raman spectroscopy.

2. Experimental details

Deposition of the diamond films was performed using a custom-built HFCVD system, (figure 1) previously described in detail [3, 4]. Diamond films were grown onto rod molybdenum substrates as well as complex shaped molybdenum (Mo) dental burrs. The Mo rod was vertically placed on a substrate holder. The tantalum filament was mounted vertically with the rod held centrally and concentrically in between the filament coil, as opposed to the commonly used horizontal arrangement [5]. The total gas flow rate was maintained at 100 sccm. The substrate temperature was varied between 800 and 1000 °C with a K-type thermocouple and the pressure was maintained constant at 26.6×10^2 Pa (N m^{-1}). The important CVD process parameters are summarized in table 1.

Negative biasing was achieved by applying a negative voltage (0–300 V) relative to the filament to the substrates using a DC power supply. The biasing stage was done for 30 min prior to the deposition stage. Subsequently for the growth stage the bias voltage was switched off.

The morphology of the as-grown diamond films was ascertained by the use of a Jeol scanning electron microscope (SEM, model JSM 5600 LV). The quality of the polycrystalline

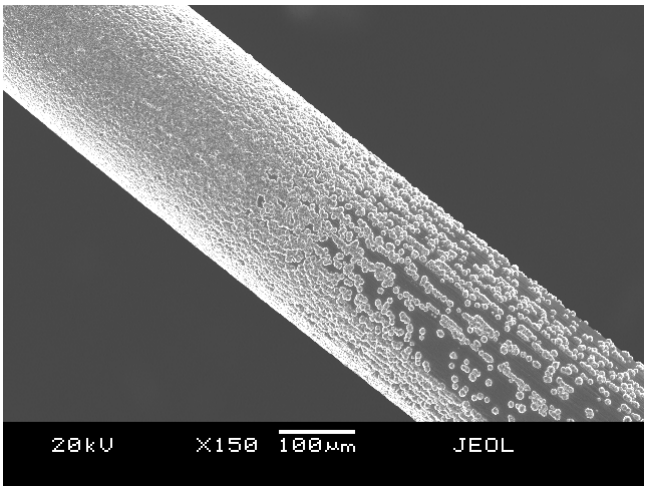

Figure 2. SEM of Mo rod deposited for 5 h using 1% methane in excess hydrogen.

Table 1. CVD process parameters used to deposit diamond films.

Process parameters	Values
Tantalum filament	0.5 mm diameter
Deposition time	5 h
Gas mixture	1% CH_4 in excess H_2
Pressure	26.6 mbar
Substrate temperature	800–1000 °C
Filament temperature	1700–2100 °C
Substrate	Molybdenum
Distance: substrate/filament	4–5 mm
Pre-treatment	1 μm diamond powder
Supply voltage	13 V
Current	13 A

diamond films was monitored by using Raman spectroscopy (Kaiser holoprobe) with a 532 nm Nd:YAG laser as the excitation source. X-ray diffraction (XRD) from a Phillips PW170 diffractometer was used for further confirmation of film quality.

3. Results and discussion

Initial experiments were carried on molybdenum wires and rods in order to optimize the CVD process for the deposition of polycrystalline diamond onto dental burrs. Figure 2 shows a SEM micrograph of diamond films deposited onto a molybdenum rod using a vertical filament arrangement in a hot filament CVD system. The substrates (molybdenum rods and dental burrs) were placed concentrically at the centre of the tantalum filament. This region of the filament gave the best thermal distribution and plasma intensity was at its maximum. The conventional filament arrangement commonly used and the new modified arrangements are illustrated in figures 1(a) and (b).

It is clearly evident that, at the centre of the filament, the diamond crystals are formed uniformly, evenly distributed and with a high crystallite density. However, at the filament

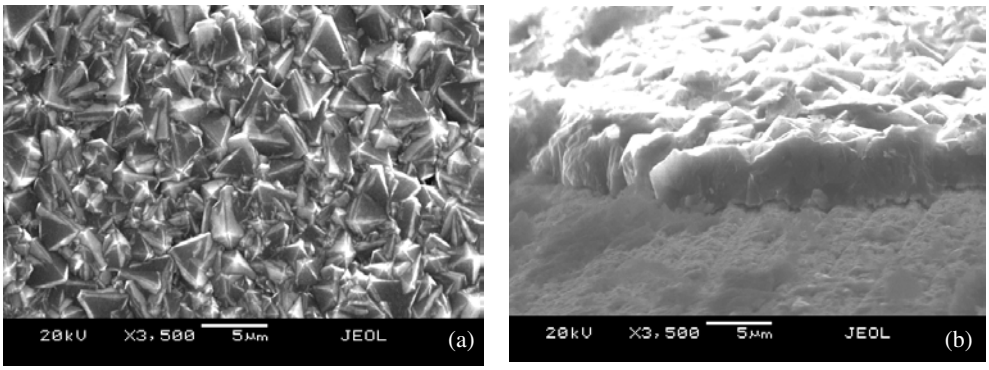

Figure 3. (a) Close-up SEM image of a diamond film deposited on a Mo rod. (b) SEM image of a diamond film cross section deposited onto a Mo rod.

end, the diamond crystallite density is much lower, due to the lower temperature at the end of the filament compared to that at the centre of the filament. Sein *et al* [6] investigated the temperature variation in WC–Co rods held concentrically within the core of the filament and found temperature variations of several hundred degrees centigrade. The difference in temperature from the centre to the end of the filament is expected to have a considerable effect on the morphology of the film, hence explaining the non-uniform diamond film growth on the molybdenum rod. Trava-Airoldi [7] also found temperature variations of about 50 °C from the centre of the wire to the ends using a conventional arrangement where the substrate was held parallel to the filament. This distribution was due to the heat conduction through the substrate and the heating distribution from the hot filament. The differences between the temperature variations of Sein *et al* compared to Trava-Airoldi *et al* can be explained in terms of filament and substrate positioning. Therefore the positioning of the rod substrates within the filament arrangement is a critical requirement in order to obtain diamond films of optimum quality and adhesion.

The crystallite sizes of the films were studied using SEM and figures 3(a) and (b) show close-up views of polycrystalline diamond films on the molybdenum rod. Typically the average crystallite size is between 2 and 3 μm, with a film thickness of approximately 4 μm. The film consisted mainly of (111) faceted diamond crystals. The films were grown for 5 h in the hot filament CVD reactor onto substrates after abrasion with 1 μm diamond powder and washed in an ultrasonic bath. In order to confirm that the films deposited were indeed diamond Raman analysis was carried out (figure 4). Typically a peak at 1335 cm^{-1} was evident with a broad band centred at approximately 1500–1600 cm^{-1} due to disordered diamond phases. Sein *et al* investigated the difference in stress between the centre of the rod compared to the edges. A single sharp peak was observed with a shift of 2 cm^{-1} towards higher wavenumbers.

For the extreme conditions of high speed (up to 50 000 rpm) and abrasive forces encountered during dental burr operation it is essential that the diamond films deposited on the molybdenum dental burrs adhere strongly to the substrate. In order to enhance adhesion and thus prevent delamination of the diamond film from the substrate negative biasing of the substrate was employed. Figure 5 shows the effect of negative substrate biasing on the adhesion of diamond on molybdenum substrates. It is clearly evident that, as the bias voltage is increased, the adhesion also increases. This may be due to the creation of additional surface sites due to ion bombardment of the substrate during the biasing stage, which gives enhanced diamond nucleation densities. As the bias voltage is increased from 0 to −300 V the adhesive

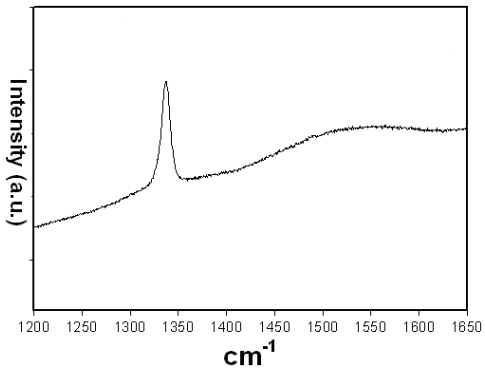

Figure 4. Raman spectra of diamond films with a bias voltage on the molybdenum rod.

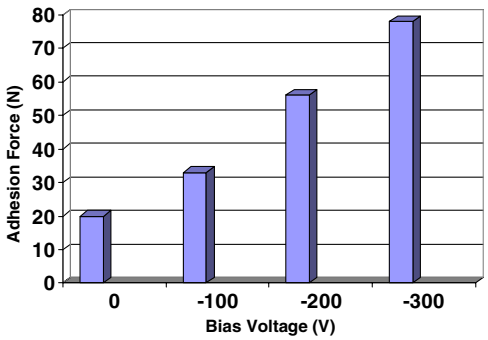

Figure 5. Effects of negative bias voltage on the adhesive force.

force increases from 20 N by a factor of 4 to about 80 N. It has been suggested that, as the bias voltage increases, there is increased ion bombardment of the negatively biased substrate by positively charged ions in the plasma generated by the hot filament. These results are in agreement with Wang *et al* [8] who also found that negative biasing gave much improved film to substrate adhesion.

Figure 6 shows a SEM micrograph of polycrystalline diamond deposited onto a molybdenum dental burr. It is evident that the average crystallite sizes were of the order of 2–3 μm, similar to the molybdenum rods as expected with a preferred crystal orientation of (111). The real life performances of conventional sintered and coated dental burrs were compared by using them in dental laboratory operation at 30 000 rpm on borosilicate glass. The CVD coated burr gave a much superior performance, with practically no deterioration in performance even after 1000 operations. However, the conventional burrs were inconsistent, failing after 30–60 operations due to the manufacturing variations arising from inhomogeneous crystal sizes and the binder material.

4. Conclusions

Adherent diamond films were successfully deposited using a new modified hot filament CVD onto molybdenum rods and dental burrs. The application of a negative substrate bias voltage to the substrate gave an improvement in adhesion of about 400% from 20 to 80 N adhesive force. In addition, CVD diamond burrs remained effective even after 1000 operations whereas the

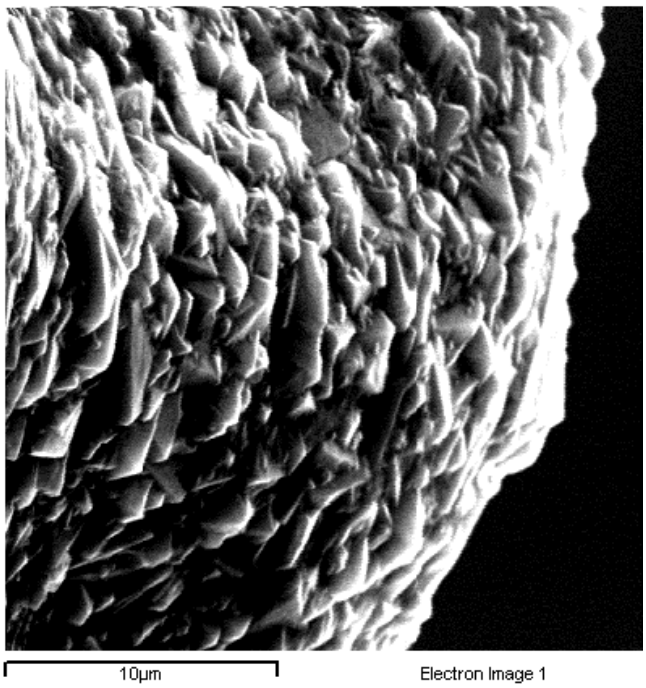

Figure 6. SEM of a molybdenum dental burr.

conventional sintered diamond burr distorted after about 30–60 operations in the laboratory. CVD technology is potentially a viable technology and a much improved way of producing diamond dental burrs compared to conventional sintered dental burrs.

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