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Chemical vapour deposition diamond coating on tungsten carbide dental cutting tools

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Abstract
Diamond coatings on Co cemented tungsten carbide (WC-Co) hard metal tools are widely used for cutting non-ferrous metals. It is difficult to deposit diamond onto cutting tools, which generally have a complex geometry, using a single step growth process. This paper focuses on the deposition of polycrystalline diamond films onto dental tools, which possess 3D complex or cylindrical shape, employing a novel single step chemical vapour deposition (CVD) growth process. The diamond deposition is carried out in a hot filament chemical vapour deposition (HFCVD) reactor with a modified filament arrangement. The filament is mounted vertically with the drill held concentrically in between the filament coils, as opposed to the commonly used horizontal arrangement. This is a simple and inexpensive filament arrangement. In addition, the problems associated with adhesion of diamond films on WC-Co substrates are amplified in dental tools due to the very sharp edges and unpredictable cutting forces. The presence of Co, used as a binder in hard metals, generally causes poor adhesion. The amount of metallic Co on the surface can be reduced using a two step pre-treatment employing Murakami etching followed by an acid treatment. Diamond films are examined in terms of their growth rate, morphology, adhesion and cutting efficiency. We found that in the diamond coated dental tool the wear rate was reduced by a factor of three as compared to the uncoated tool.

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

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1. Introduction

Deposition of diamond films onto tungsten carbide is attractive since it can lead to potential improvements in the life and performance of cutting tools including rotary tools and inserts due to the excellent physical and chemical properties of the coatings. Chemical vapour deposition (CVD) diamond coating has the potential to considerably prolong the lifetime of WC-Co dental cutting tools when applied to the machining of highly abrasive non-ferrous metallic alloys, borosilicate glass, human teeth and ceramic materials. However, deposition of adherent high quality diamond films onto substrates such as cemented carbides, stainless steel and various metal alloys has proved to be problematic due to the thermal expansion mismatch and presence of a cobalt binder, which provides additional toughness to the tool but causes poor adhesion and low nucleation density [1–7]. There are a number of potential surface treatments which can be used to overcome these problems including chemical etching, ion implantation, interlayer coating and bias treatment [8]. Various approaches have been used to suppress the influence of Co and to improve adhesion between the diamond coating and the tungsten carbide substrate. For example, chemical treatment using Murakami agent and acid etching has been used successfully for removal of the Co binder from the substrate surface [9]. Gabler *et al* [10] reported that uniform diamond coating was achieved by using a parallel filament position. The coatings were uniform due to the temperature and gas phase composition being more or less constant along the tool, which was positioned vertically outside the filament. Trava-Airoldi *et al* [11] applied a different approach, which used a stepper motor for rotating the cylindrical substrate between a set of two filaments. Therefore, it is evident that it is difficult to deposit CVD diamond onto sharp cutting edges of dental tools, which have a complex geometry, using a single step growth process. In this paper we developed and optimized a new filament arrangement, which has been developed to deposit uniformly CVD diamond on the cutting edges of dental tools.

2. Experimental details

2.1. Substrate preparation

Tungsten carbide (WC-Co) dental burs with a fine WC grain size (1 μm), with a 20–30 mm length and 1.0–1.5 mm diameter were used for CVD diamond deposition. Prior to pre-treatment the cutting tool was ultrasonically cleaned in acetone for 10 min in order to remove loose residues from the surface. The following two step chemical pre-treatment procedure was used. A first etching step, using Murakami's reagent ([10 g $k_3Fe(CN)_6$] + 10 g KOH + 100 ml water), was carried out for 10 min in an ultrasonic bath to etch the WC substrate; this was followed by a rinse with distilled water. The second etching step was performed using an acid solution of hydrogen peroxide (3 ml (96 wt%) H_2SO_4 + 88 ml (30% w/v) H_2O_2), for 10 s, to remove Co from the surface. The substrates were then washed again with distilled water in an ultrasonic bath [12]. Etched surfaces of the substrates were characterized by scanning electron spectroscopy (SEM) and energy dispersive spectroscopy (EDS).

2.2. Diamond deposition

Experiments were carried out in a modified hot filament chemical vapour deposition (HFCVD) system, which is shown schematically in figure 1. The system was built with a water cooled stainless steel chamber (20 cm in diameter, 30 cm in length), which is connected to a rotary pump. MkS mass flow controllers accurately controlled the amount of gas flowing

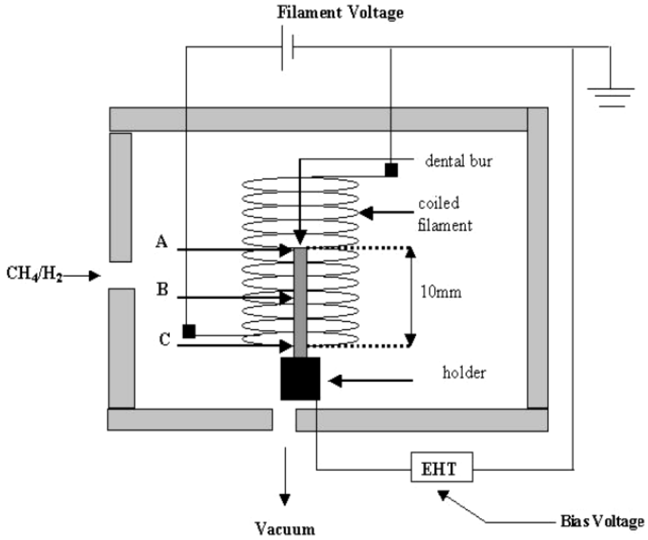

Figure 1. A schematic diagram of the CVD diamond deposition.

into the chamber. The gas sources used were 1% methane in excess hydrogen; the hydrogen flow rate was 100 sccm while the methane flow rate was 1 sccm. The deposition time and pressure in the vacuum chamber were 15.0 h and 26.6 mbar respectively. The filament temperature was measured using an optical pyrometer and values between 1700 and 2100 °C were obtained. The substrate temperature was measured to be between 800 and 1000 °C using a K-type thermocouple in direct thermal contact with the dental bur.

The system allowed an independent DC bias to be applied between the substrate and the filament. Tantalum wire, 0.5 mm in diameter, 12 cm in length (uncoiled), was used as the hot filament. The filament is mounted vertically with the dental bur held in between the filament coils as opposed to the horizontal position in the conventional HFCVD systems. The new vertical filament arrangement used in modified HFCVD systems improves the thermal distribution [13]. The dental bur was vertically mounted on a diamond coated molybdenum substrate holder and positioned centrally and coaxially within 5 mm distance of the coiled filament [14]. Before deposition the filament was pre-carburized for 30 min in 3% methane with excess hydrogen to enhance the formation of a tantalum carbide layer on the filament surface in order to reduce the tantalum evaporation during diamond deposition [15]. Diamond films were characterized by SEM and EDS. Micro-Raman spectroscopy measurements were performed in back-scattering geometry at room temperature by using a Dilor XY triple spectrometer equipped with a liquid nitrogen cooled charge coupled device detector and an adapted Olympus microscope.

3. Results and discussion

The surface morphology of predominantly (111) faceted octahedral shape diamond films was obtained. The film thickness was measured to be 15–17 μm thick after diamond deposition for 15 h. Figure 2 shows the SEM micrograph of a CVD diamond coated dental bur at the cutting edge. The film is homogeneous with uniform diamond crystal sizes. Typically the crystal sizes are in the range 8–10 μm. As expected the surface morphology is rough making the dental burs extremely desirable for abrasive applications. Figures 3 and 4 show an SEM

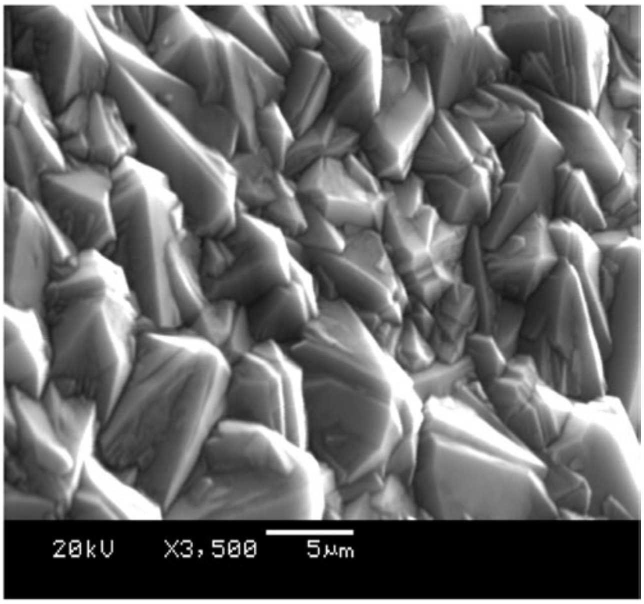

Figure 2. The CVD diamond coated cutting edge of dental bur.

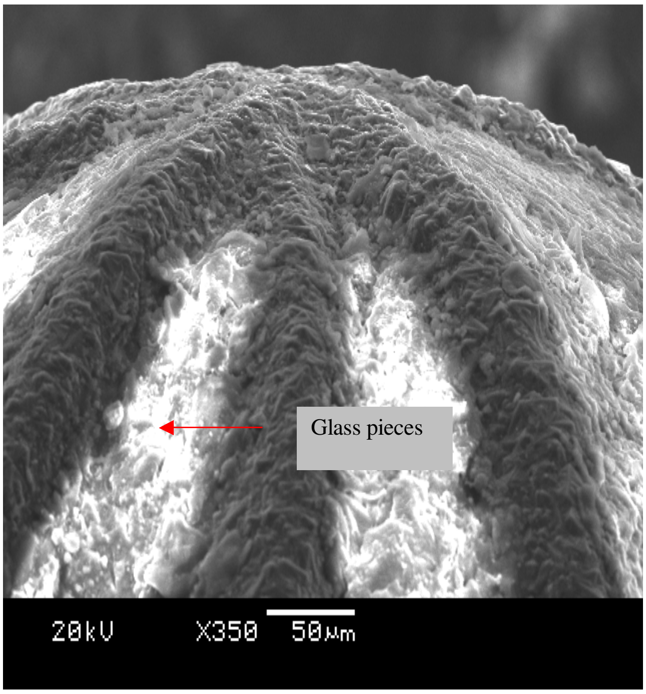

Figure 3. A close view of the bur after testing with borosilicate glass.

image of a CVD diamond coated dental bur after machining tests on borosilicate glass and acrylic/porcelain teeth respectively for 5 min at a cutting speed of 3600 rpm. After machining, it is clearly indicated that the diamond films are still intact on the pre-treated WC substrate, the

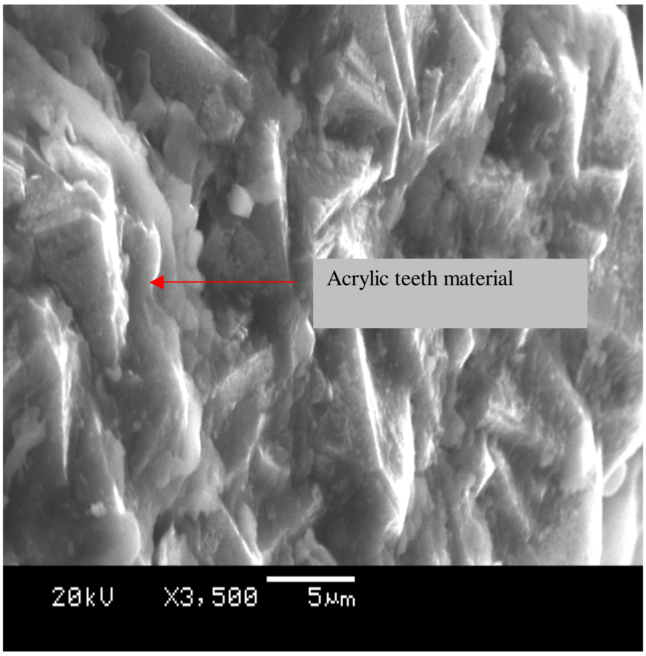

Figure 4. A close view of the bur after testing with acrylic teeth.

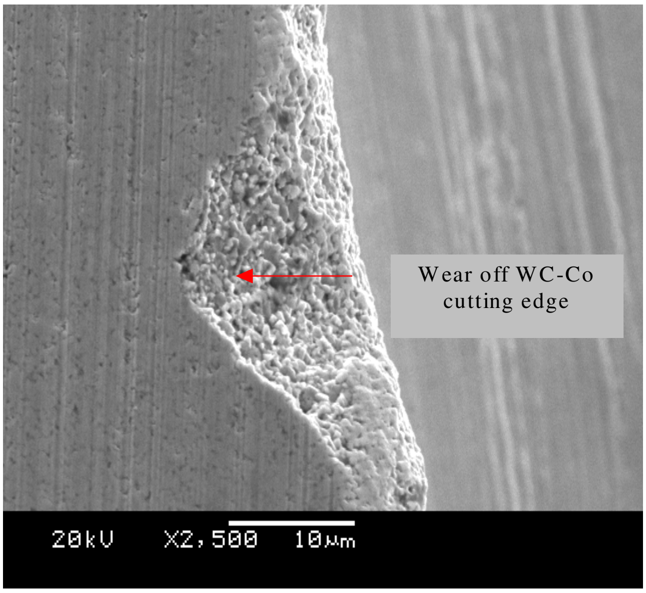

Figure 5. The cutting edge of an uncoated WC-Co dental bur after machining with glass.

diamond coating displayed good adhesion and there is no indication of diffusive wear after the initial tests drilling 500 holes. Figure 5 shows a micrograph of an uncoated WC-Co dental bur tested on the borosilicate glass using the same machining conditions. The uncoated WC-Co bur displayed considerable flank wear along the cutting edge of the bur. The areas of flank

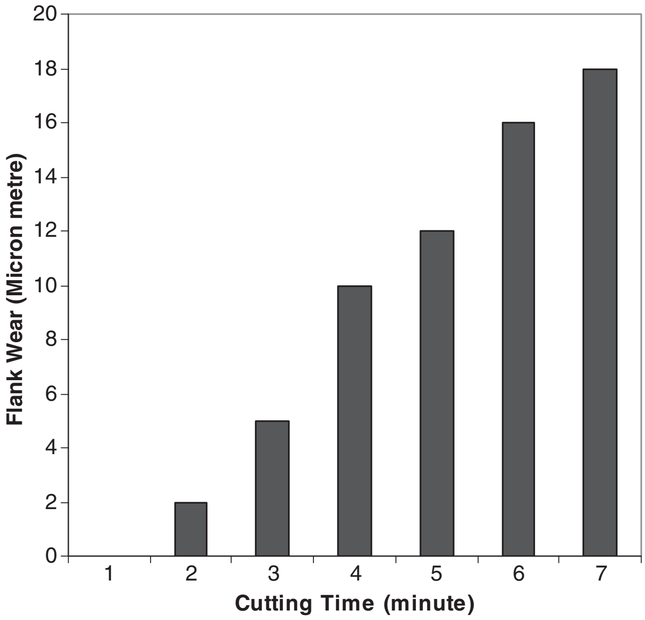

Figure 6. Flank wear (μm) as a function of time (min).

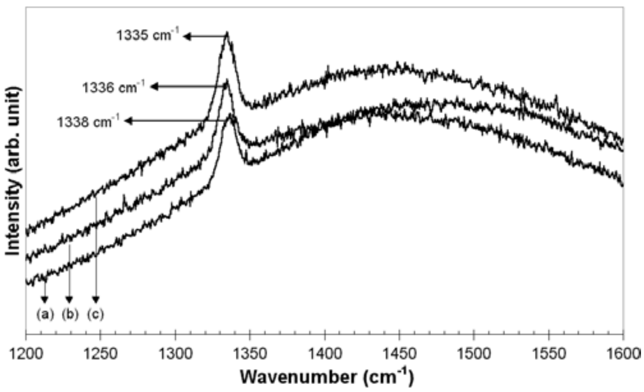

Figure 7. Raman spectra of the diamond films.

wear were further investigated at the cutting edge of the dental bur. Figure 6 shows the area of flank wear plotted against time after tests on borosilicate glass. It is evident that a longer duty cycle of machining could cause a higher rate of flank wear on the cutting edge of the tool. Therefore, the cutting edge of a WC-Co dental bur should have significant thickness of CVD diamond, which will enhance not only the quality of cutting but also prolong the tool life [16]. It was found that in the CVD diamond coated tool the wear rate was reduced by a factor of three as compared to the uncoated dental tool. In the uncoated tool the cutting edges were ineffective after 2.5 min and uncoated tools lasted longer than 7 min with the coating still intact. The cutting speed employed was 3600 revolutions min^{-1} with a feed rate of 12 mm min^{-1} and the material machined was borosilicate glass. The Raman spectrum shown in figure 7 shows that at the tip, centre and end of the cutting tool, single sharp peaks at 1335, 1336 and

1338 cm^{-1} respectively were observed for different positions. The Raman spectrum also gives an indication of the stress in the diamond coating. The results of Raman analysis on the WC-Co substrates at several different locations on the tool have shown indications of compressive stress in the coating [16, 17].

4. Conclusions

An etching treatment of the substrate carried out in order to remove the surface cobalt results in better adhesion between the diamond coating and the substrate. Thicker coating of CVD diamond at the cutting edges is expected to give a tool longer life and better machining quality. The performance and lifetime of the CVD coated dental bur are far superior to those of the uncoated WC-Co bur with the wear rates being reduced by a factor of three with the application of a CVD diamond coating. Further work is required to study the effects of diamond film adhesion and thickness at the cutting edge based on the performance of the tool.

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References

[1] Murakawa M and Takeuchi S 1991 *Surf. Coat. Technol.* **49** 359
[2] Leyendecker T, Lemmer O, Jurgens A, Esser S and Ebberink J 1991 *Surf. Coat. Technol.* **48** 253
[3] Reineck J, Soderbery S, Eckholm P and Westergren K 1993 *Surf. Coat. Technol.* **57** 47
[4] Yaskiki T, Nakamura T, Fujimori N and Nakai T 1992 *Surf. Coat. Technol.* **52** 81
[5] Wang H-Z, Song R-H and Tang S-P 1993 *Diamond Relat. Mater.* **2** 304
[6] Kanda K, Takehana S, Yoshida S, Watanabe R, Takano S, Ando H and Shimakura F 1995 *Surf. Coat. Technol.* **73** 115
[7] Inspector A, Bauer C E and Oles E J 1994 *Surf. Coat. Technol.* **68/69** 359
[8] Cappelli E, Pinazari F, Ascarelli P and Righini G 1996 *Diamond Relat. Mater.* **5** 292–8
[9] Amirhaghi S, Reehal H S, Wood R J K and Wheeler D W 2001 *Surf. Coat. Technol.* **135** 126
[10] Gabler J, Schafer L and Westermann H 2000 *Diamond Relat. Mater.* **9** 921–4
[11] Trava-Airoldi V J, Moro J R, Corat E J, Goulart E C, Silva A P and Leite N F 1998 *Surf. Coat. Technol.* **108/109** 437
[12] Kamiya S, Takahashi H, Polini R and Traversa E 2000 *Diamond Relat. Mater.* **9** 191–4
[13] Sein H, Ahmed W and Rego C 2002 *Diamond Relat. Mater.* **11** 731–5
[14] May P, Rego C, Thomas R, Ashfold M N and Rosser K N 1994 *Diamond Relat. Mater.* **3** 810
[15] Wang W, Liao K, Wang J, Fang L, Ding P, Esteve J, Polo M C and Sanchez G 1999 *Diamond Relat. Mater.* **8** 123
[16] Sein H, Ahmed W, Jackson M, Ali N and Gracio J 2003 *Surf. Coat. Technol.* **163/164** 196
[17] Tang W, Wang Q, Wang S and Lu F 2001 *Diamond Relat. Mater.* **10** 1700