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Diamond coated dental bur machining of natural and synthetic dental materials

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Diamond coatings are attractive for cutting processes due to their high hardness, low friction coefficient, excellent wear resistance and chemical inertness. The application of diamond coatings on cemented tungsten carbide (WC-Co) burs has been the subject of much attention in recent years in order to improve cutting performance and tool life. WC-Co burs containing 6% Co and 94% WC with an average grain size 1–3 micron were used in this study. In order to improve the adhesion between diamond and the bur it is necessary to etch away the surface Co to prepare it for subsequent diamond growth. Hot filament chemical vapour deposition (H.F.C.V.D.) with a modified vertical filament arrangement has been employed for the deposition of diamond films. Diamond film quality and purity has been characterised using scanning electron microscopy (S.E.M.) and micro-Raman spectroscopy. The performance of diamond coated WC-Co burs, uncoated WC-Co burs, and diamond embedded (sintered) burs have been compared by drilling a series of holes into various materials such as human teeth, and model tooth materials such as borosilicate glass and acrylic. Flank wear has been used to assess the wear rates of the burs when machining natural and synthetic dental materials such as those described above.

1. Introduction

Owing to their excellent physical and chemical properties, chemical vapour deposited (C.V.D.) diamond films have attracted considerable interest in recent years for cutting applications, including rotary tools and inserts. However, deposition of adherent high quality diamond films onto substrates such as cemented carbides, stainless steel and various metal alloys containing transition elements has proved to be problematic. In general, the adhesion of the diamond films to the substrates is poor and the nucleation density is very low [1–6]. The influence of different metallic substrates on the diamond deposition process has been examined [7–11]. The physical and chemical nature of the substrate was found to have a crucial impact on diamond nucleation and its subsequent growth. The interaction between substrate material and carbon species in the gas phase was found to be particularly important and leads to either carbide formation or carbon dissolution. Carbides are formed in the presence of carbon-containing gases on metals such as molybdenum, tungsten, niobium, hafnium, tantalum, and titanium. The carbide layer formed allows diamond to form on it since the minimum carbon surface concentration required for diamond nucleation cannot be reached on pure metals. As the carbide layer increases in thickness, the carbon transport rate to the substrate decreases until a critical level is reached where diamond is formed [7].

Substrates made from metals of the first transition group such as iron, cobalt, and nickel, are characterized by high dissolution and diffusion rates of carbon into those substrates (Table I) [12]. Owing to the absence of a stable carbide layer, the incubation time required to form diamond is higher and depends on substrate thickness. In addition, these metals catalyze the formation of graphitic phases, which is reflected in the graphite-diamond ratio during the deposition process, yielding a low diamond content or an amorphous carbon layer at the interface between the metal and the diamond coating. The catalytic effect is related to the activity of the electrons in the incomplete 3d-shell of these transition metals [8]. The importance of this mechanism in relation to diamond deposition decreases from iron to nickel, corresponding to a gradual filling of the 3d-orbital [12]. This effect occurs whenever the metal atoms come into contact with the carbon species, which can take place on the substrate or in the gas phase [13]. Clearly, the presence of these transition metals can be harmful to diamond deposition even at relatively low concentrations.

TABLE I Solubility and diffusion rates of carbon atoms in different metals at 900 °C

	α-Fe	γ-Fe	Co	Ni
Solubility of carbon (wt.%)	1.3	1.3	0.1	0.2
Carbon diffusion rate (cm^2/s)	2.35×10^{-6}	1.75×10^{-8}	2.46×10^{-8}	1.4×10^{-8}

The present work is concerned with diamond deposition on tungsten carbide cemented with 6 wt.% cobalt. WC-Co substrates are suitable for diamond deposition, but their adhesion strengths to diamond films are relatively poor [14]. The poor adhesion is related to the cobalt binder that is present to increase the toughness of the tool. Much effort has been directed at increasing the adhesion strength of diamond films to WC-Co substrates, including decarburizing the surface prior to deposition [15], seeding WC-Co with diamond powder and annealing prior to deposition [10], removing cobalt atoms at the surface using cobalt etching agents [14, 16–18], and depositing an interlayer as a diffusion barrier [19]. If these deficiencies can be overcome then CVD diamond coatings have the potential to prolong the working life of WC-Co dental burs when applied to the machining of highly abrasive non-ferrous alloys, borosilicate glass, ceramic materials such as porcelain, natural human teeth, or various dental acrylic materials. The presence of cobalt (Co) provides additional tool toughness but it has adverse effect on diamond film adhesion.

Various approaches have been used to suppress the influence of Co and to improve adhesion [20]. A significant factor for the adhesion of C.V.D. diamond to WC-Co substrates is the mechanical interlocking that occurs at the coating-carbide interface. Therefore, it is essential to pre-treat substrates both to reduce the surface Co concentration and create a proper interface roughness [21]. Chemical treatment using Murakami reagent and acid etching has been used successfully for removing the Co binder from the substrate surface and this has resulted in an adherent diamond film [22, 23].

In this paper we report the results of our investigation of diamond film deposited on WC-Co dental burs using a H.F.C.V.D. system and subsequent machining results on human teeth, borosilicate glass, and acrylic material. Even though considerable work has been done on C.V.D. diamond deposition very little work has been done in applying the process to cylindrically shaped substrates such as dental burs. Even less has been reported on the performance and characterisation of C.V.D. diamond coated dental burs.

2. Experimental

2.1. Substrate preparation

Two sets of laboratory tungsten carbide (WC-Co) dental burs (AT23 LR) 20–30 mm in length and 1.0–1.5 mm in diameter with fine WC grain sizes (1 μm) [supplied by Metrodent Ltd, UK.], were used for C.V.D. diamond deposition process. Prior to pre-treatment the cutting tools were 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 step etching, using Murakami's reagent ([10 g $K_3Fe(CN)_6$] + 10 g KOH + 100 ml water) was carried out for 10 min in ultrasonic bath to etch the WC substrate, followed by a rinse with distilled water. The second step etching 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 [24]. The etched surfaces of the substrates were characterised by scanning electron microscopy and energy dispersive spectroscopy (E.D.S.).

2.2. C.V.D. diamond deposition

Diamond films were deposited onto the cutting edge of the burs at 5 mm distances from a tantalum wire filament, which measured 0.5 millimetres in diameter and had approximately 10 to 12 cm in length as the hot zone. The coiled filament was held vertically within the vacuum deposition chamber, as opposed to the commonly used horizontal filament position employed in H.F.C.V.D. systems [25]. To ensure a uniform coating the dental burs were positioned centrally and coaxially within the coils of the filament [26]. The gas phase was a mixture of methane and hydrogen [CH_4/H_2] containing 1% CH_4 with an excess of H_2, the volume flow rate for hydrogen was 200 standard cm^3/min, while the volume flow rate for methane was 2 standard cm^3/min. Prior to C.V.D. diamond deposition, the tantalum filament was carburised for 30 min with 3% CH_4 with excess hydrogen. The deposition time and pressure in the vacuum chamber were 15 h and 20 Torr (2660 Pa), respectively. Depositions on the substrate were carried out over a temperature range of 800–1000 °C. The filament temperature was measured using two-colour optical pyrometer and found to be between 1800–2100 °C depending upon the filament position. Diamond films were characterised by SEM and energy–dipersive spectroscopy (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.

2.3. Dental bur machining: Drilling experiments

In order to examine the cutting performance of the diamond coated dental burs materials such as borosilicate glass, acrylic teeth, and natural human teeth were drilled. The drilling unit (Fig. 1) was specifically constructed with a water-cooling system so that maximum spindle speeds of 250,000 revolutions per minute (r.p.m), feed rates of between 5–20 μm per revolution, and cutting speeds in the range 100 to 200 m/min for drilling with dental burs could be achieved.

After the dental burs were coated and examined for adhesion they were used to machine a number of dental materials. The coated burs were compared with uncoated burs to distinguish them in terms of their

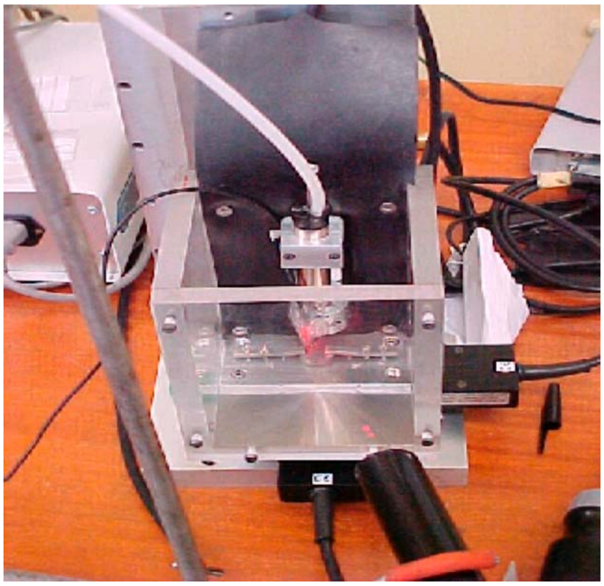


Figure 1 Dental bur drilling machine.

drilling behaviour. The drilling unit shown in Fig. 1 is constructed using three principal axes each controlled using a d.c motor connected to a Motionmaster^{TM} controller. A laser light source is focused onto the rotating spindle in order to measure the speed of the dental bur during drilling. Post machining analysis was performed using a scanning electron microscope to detect wear on the flanks of the cutting edges.

The flank wear of the burs was estimated by S.E.M. analysis at a selected time interval of between 1 and 3 min. Prior to S.E.M. analysis diamond coated burs were ultrasonically washed with 6 M H_2SO_4 solution to remove any unwanted machining material. For comparison, conventional P.C.D. (polycrystalline diamond) sintered burs with different geometry were also tested on the same substrate materials.

2.4. Dental bur machining: Machining experiments

To examine the machining characteristics of coated and uncoated dental burs, a specially constructed clamp was developed to locate over the tooth to prepare it for the location of a crown. Fig. 2 shows the basic construction of the clamping device and air turbine driven dental bur located on top of a tooth.

The clamping device is located onto the tooth to be machined and allows the tooth to be machined by incorporating a wire driven driving mechanism that attaches itself onto the clamp so that the dental bur can rotate at the appropriate cutting speed. The driving mechanism is attached to the air operated hand piece that provides the power to drive the mechanism, clamp, and dental bur (Fig. 2). The bur was rotated at 20,000–30,000 r.p.m., with a feed rate of between 0.2–0.5 mm/revolution without a water spray that is used primarily to remove unwanted tooth material during clinical practice and not to cool the bur itself. The uncoated and coated dental burs were also compared with commercial sintered diamond burs machining acrylic material. This was used to simulate the machining of dentine. Borosilicate glass was used to simulate the machining of enamel. Sulphuric acid was used to remove machining detritus from the surface of the dental burs, and the flank wear of the dental burs was estimated using magnified images of the worn bur produced using a scanning electron microscope.

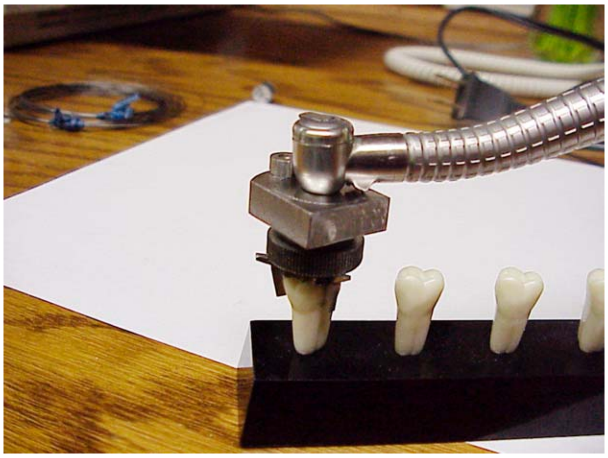


Figure 2 Air operated spindle unit attached to the clamping device and driving unit attached firmly to the tooth.

3. Results and discussion

3.1. Substrate preparation and diamond deposition

The crystallinity of as-grown films was analysed using a scanning electron microscope (S.E.M.). In addition, Raman spectroscopy (Kaiser holoprobe conventional Raman spectrometer) was used to monitor the carbon-phase purity of the deposited films. The chemical composition of the WC-Co surface was analysed using E.D.S. (Oxford pentafet). Fig. 3 shows the surface of the WC-Co dental bur substrate prior to chemical etching. The effects of etching the WC-Co substrate surfaces are shown in Fig. 4. Murakami's solution has chemically attacked the WC-Co substrate. No cobalt peaks could be detected in the E.D.S. spectrum. In addition to this effect, acid etching produced a roughened surface.

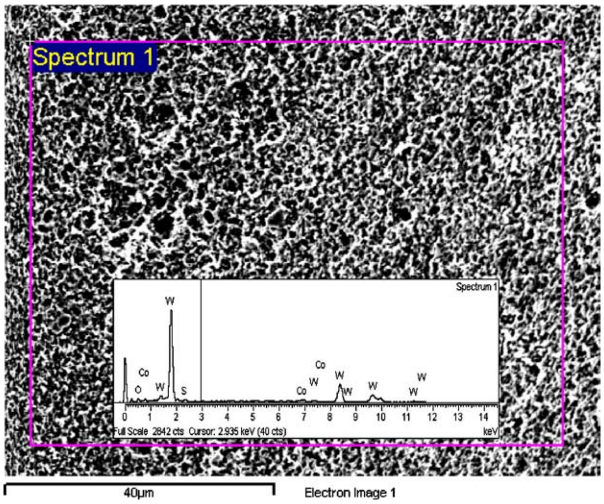


Figure 3 WC-Co dental bur substrate treated with Murakami's solution and before acid etching. The X-ray spectrum shows the appearance of a cobalt peak present in the matrix of the dental bur.

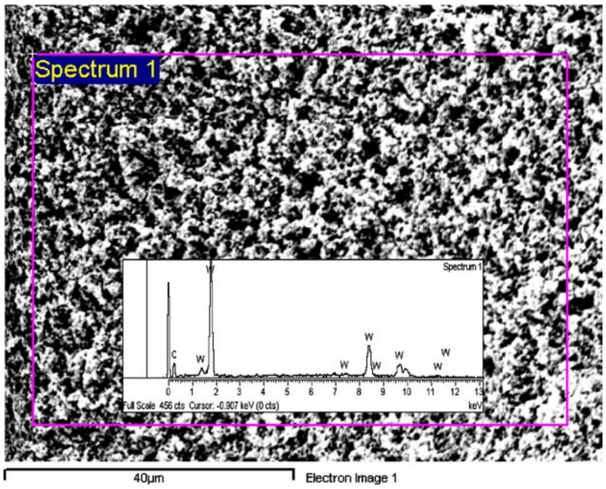


Figure 4 WC-Co dental bur substrate treated with Murakami's solution and after acid etching. The X-ray spectrum shows an absence of the cobalt peak.

To ensure that the diamond deposition process was working correctly, information from previous experimental research suggests that the diamond deposition rate on molybdenum wire coated under identical deposition conditions is about 1 μm/h, thus after 5.5 h deposition time, a diamond layer of 5–6 μm thickness is obtained. In order to assess the effectiveness of the deposition process used in these experiments, a molybdenum wire was coated with diamond and the thickness was found to be 5.5 μm. On the surface of WC-Co dental burs, the deposition rate was probably much higher since the surface was much rougher at both the cutting edge and on the tip of the burs.

Adherent diamonds consisting of mainly (111) faceted diamond crystals were deposited on WC-Co dental burs as shown in Fig. 5. The modified filament arrangement gave uniform and dense diamond coating even though the substrate is non-planar with a complex geometry. The design of the filament and substrate in the reactor offers the possibility of uniformly coating even larger diameter cylindrical substrates.

The morphology of the surface of the dental bur is extremely rough making the bur desirable for dental machining applications. Raman analysis was performed in order to evaluate the quality and stress imparted in C.V.D. diamond films. The Raman spectrum shown in Fig. 6 shows a single peak at 1335 cm^{-1} at the tip, middle and at the end of the dental bur indicating that diamond is deposited at each of these points. The diamond peak on the Raman spectrum is shifted to a higher wave number of magnitude 1335 cm^{-1} than that normally experienced in an unstressed coating where the natural diamond peak occurs at 1332 cm^{-1}. This indicates that the stress is compressive. The results of Raman analysis on WC-Co substrates at several different locations on the tool have shown that the 1335 cm^{-1} peak consistently indicates that there are compressive stresses in the coating and that this is uniformly distributed.

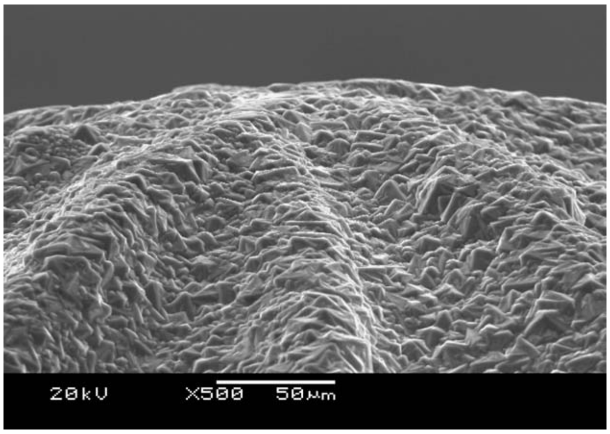


Figure 5 (111) faceted CVD diamond coated dental bur.

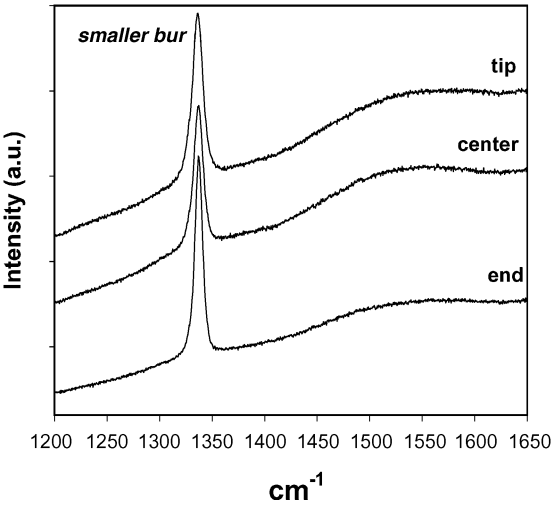
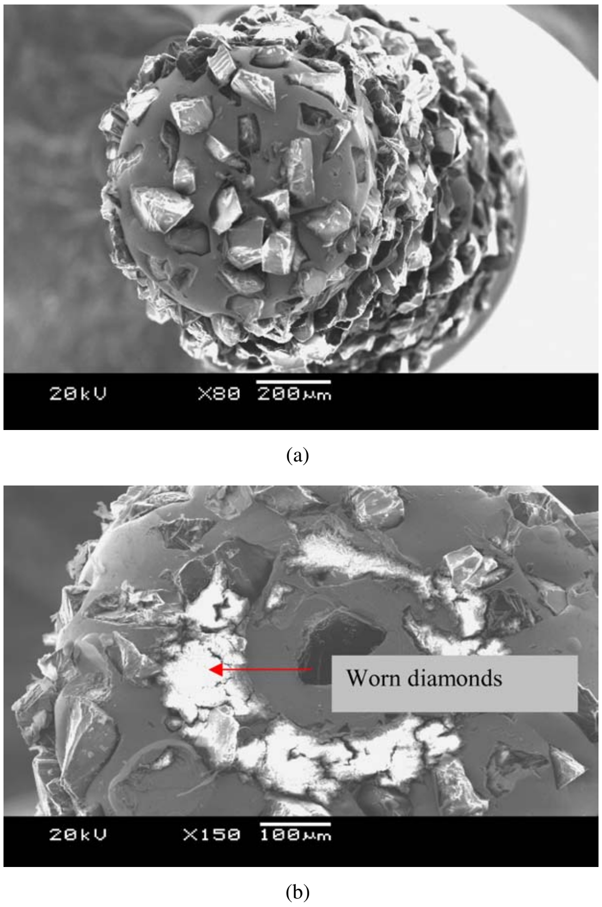


Figure 6 Raman spectrum of the diamond film deposited onto the surface of a dental bur.



(a)

(b)

Figure 7 (a) Inhomogeneous surface of PCD diamond sintered bur. (b) PCD diamond sintered bur after testing with glass.

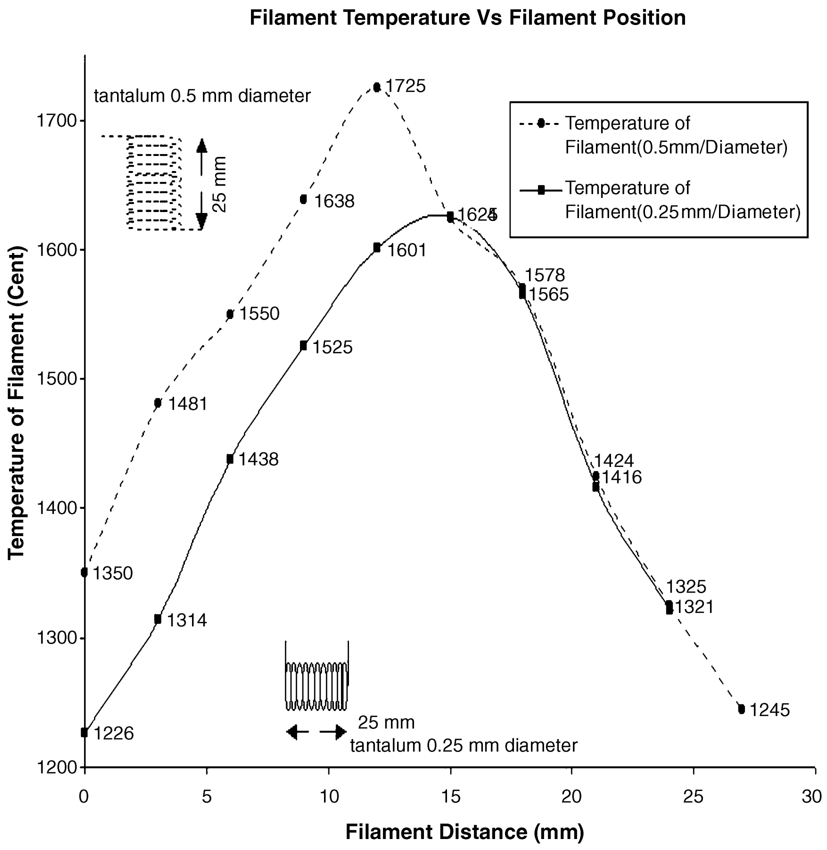


Figure 8 Measurements of in-process filament temperature during the diamond deposition process.

In contrast, Fig. 7(a) is a close up view from SEM micrograph of a conventional PCD sintered bur. The diamond particles are imbedded onto surface with a suitable binder matrix material such as nickel. Typically the surface is inhomogeneous and sizes of particles are range from 50–200 μm causing considerable variation in the cutting performance of the tool (Fig. 7(b)).

The type of filament used during the deposition of diamond to WC-Co substrates has a significant effect on the amount and quantity of diamonds deposited to the substrate material. Fig. 8 shows how the filament coil temperature changes from the end to the centre of the filament. The diameter of tantalum wire used in the experiments was 0.5 mm. It is assumed that the best thermal distribution is obtained at the centre of the filament coil where the highest temperature was measured. The etched dental burs were placed inside the filament at the central point of the filament in order to gain the highest thickness of diamond possible on the bur. Trava-Airoldi *et al.* [27] indicated that substrate temperatures can be different from the end to the centre, and is more accentuated for a molybdenum filament wire with a smaller diameter. This could be due to heat being conducted through the substrate and heat being distributed from the hot filament [27].

An important factor that could affect the final performance of the dental bur is the adhesive toughness of the diamond on the substrates. Endler *et al.* [28] and Kamiya *et al.* [29] have developed a new method for the quantitative evaluation of the adhesive toughness of diamond films onto Co-cemented WC substrates. They found that the adhesive toughness of diamond on WC to be in the range of 20–37 J/m^2. Commercial burs exhibited much higher adhesive toughness than flat substrates due to the large surface roughness and the absence of interfacial voids. This factor needs to be investigated in detail for non-planar dental burs.

3.2. Stress analysis

Raman analysis was performed in order to evaluate diamond phase purity and the level of stress in the diamond film. The Raman spectrum shown in Fig. 6 shows a single peak at 1335 cm^{-1} for the tip, center, and the end of the dental bur. The spectrum provides information about the nature of stress in the diamond coating. The diamond peak is shifted to a higher wave number than that for natural diamond, which peaks at 1332 cm^{-1}. This indicates that a compressive stress exists in the coating. Ager and Drory [30] investigated biaxial stresses in diamond film grown on titanium alloy by Raman spectroscopy and developed a model that describes the relationship between singlet and doublet photon scattering and the biaxial stress as follows:

$$\sigma = -1.08(\nu_s - \nu_o)\,\text{GPa} \quad \text{for singlet phonon} \qquad (1)$$

$$\sigma = -0.384(\nu_d - \nu_o)\,\text{GPa} \quad \text{for doublet phonon} \qquad (2)$$

where, ν_o is 1332 cm^{-1}, ν_s is the observed maximum of the singlet in the spectrum and ν_d is the observed maximum of the doublet in the spectrum. From 1 and

2 we obtain,

$$\sigma = -0.567(\nu_m - \nu_o)\text{GPa} \qquad (3)$$

where the observed peak position, ν_m is assumed to be between the singlet and the doublet, i.e. $\nu_m = \nu_s + \nu_d$.

The stress at the tip, center, and base of the dental bur was found to be 1.7, 2.3, and 3.4 GPa in compression using the conventional CVD deposition technique. The temperature of the coating at these points corresponded to 840, 908, and 952 °C. The modified hot filament CVD process described in this paper gave a uniform compression value of 1.7 GPa throughout the bur.

3.3. Dental bur machining: Drilling and machining experiments

The effectiveness of using HFCVD coated dental burs was measured by comparing uncoated burs, HFCVD dental burs, and sintered diamond burs when drilling and machining extracted human teeth, acrylic material, and borosilicate glass. The latter two materials being used as substitute materials for enamel and dentine.

A sequence of fifty drillings was employed in each drilling experiment. The sharpness and initial condition of the burs were inspected by an optical method after the burs had drilled ten holes in sequence. An abrading coefficient of drilling, C_a, has been defined as a quality criterion for small drilling tools [31]. It is defined as the ratio between the bur's total abraded area, S, and the effective coated area of the bur used during the drilling process. The effective coated area is given by the difference of the nominal coated bur area, D_b, and the area of the bur consumed during drilling, W_b. This can be written as:

$$C_a = S/(D_b - W_b) \qquad (4)$$

A high quality coated dental bur is one that produces accurate drilling that has an area S close to D_b and does not lose its coating during the machining process, i.e. $W_b \approx O$. The cutting will therefore have an abrading coefficient close to unity. It must be remembered that the quality of machining is dependent on the cutting speed, V_c. A comparative figure of merit (F) for the dental bur can be defined as:

$$F = C_a/V_c \qquad (5)$$

where F is directly related to the lifetime of the dental bur for a specific drilling process. Figs. 9–11 show the results of drilling the dental materials with the three types of burs described.

Fig. 7(b) shows the morphology of a sintered diamond bur after being tested on borosilicate glass at a cutting speed of 30,000 rpm for 5 min with an interval at every 30 s. It is clearly evident that there is significant removal of diamond particles from the surface of the tool after 50 holes. As expected there is the deterioration of the abrasive performance of the PCD sintered diamond dental burs. Borges *et* alia [32] also reported that the significant loss of diamond particles occurred during cutting with the commercial sintered diamond bur. In addition, the metallic nickel binder shows major defects generated by pulled-out particles [33].

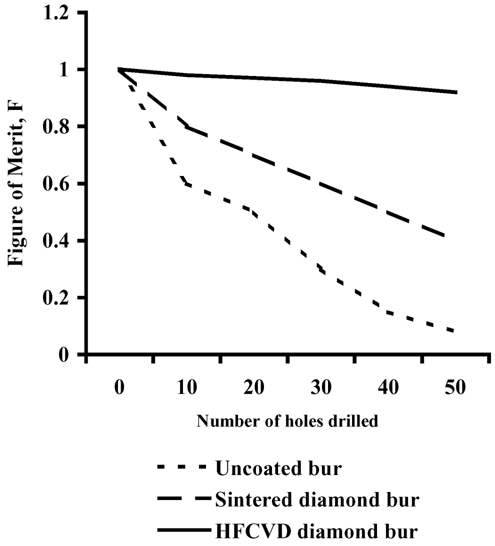


Figure 9 Figure of merit for dental burs drilling borosilicate glass.

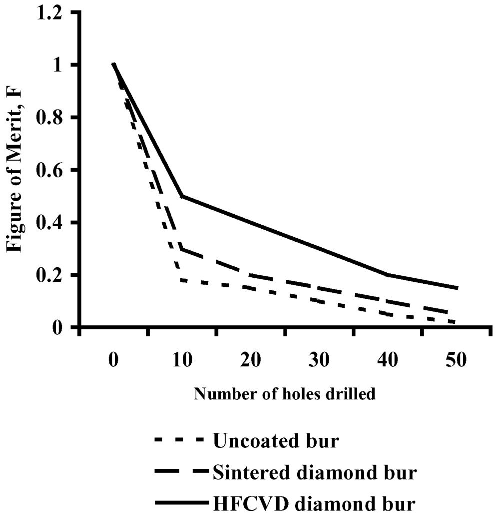


Figure 10 Figure of merit for dental burs drilling acrylic material.

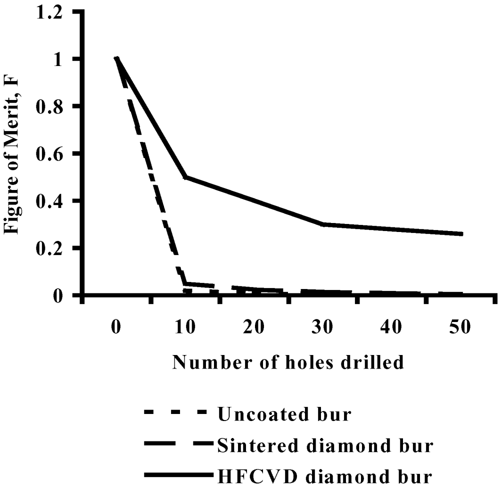


Figure 11 Figure of merit for dental burs drilling human tooth material.

Figs. 12 and 13 show SEM images of CVD diamond coated laboratory bur after drilling experiments

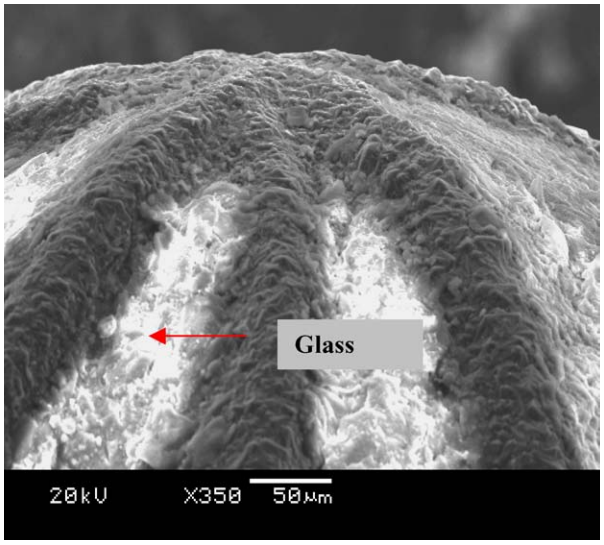


Figure 12 Cutting edge of dental bur after drilling borosilicate glass showing adhesion of glass on the flute of the bur.

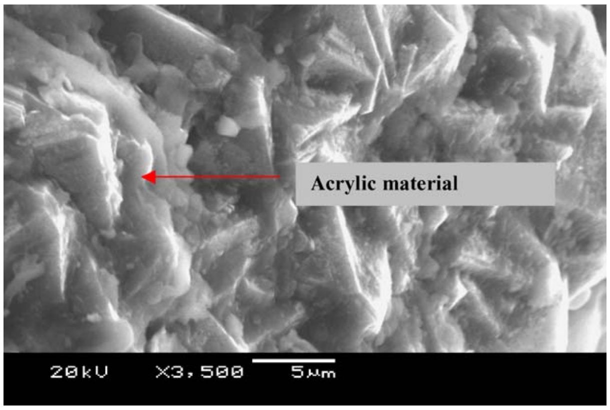


Figure 13 Magnified image of the dental bur after drilling acrylic material.

on borosilicate glass and acrylic teeth respectively, for 5 min at a cutting speed of 30,000 r.p.m. After machining, it is clearly evident that the diamond films are still intact on the pre-treated WC substrate and diamond coating displayed good adhesion. Also, there is no indication of diffusion wear (which is characterized by the formation of craters on the cutting edge of the bur) after the initial test for 50 holes. However, the machined materials such as glass pieces erode the cutting edge of the diamond dental bur and adhere to the flutes of the bur. Fig. 12 shows the adhesion of glass to the flutes of the dental bur. After conducting experiments on acrylic materials the mechanisms of wear experienced by the bur involve adhesion as well as abrasive wear. Fig. 13 suggests that inorganic components from acrylic teeth adhered to the cutting tool surface in localised areas when increased rate of abrasion was used [34].

Fig. 14 shows that a micrograph of uncoated WC-Co dental bur tested on the borosilicate glass using the same machining conditions. The uncoated WC-Co burs displayed flank wear along the cutting edge of the bur. The areas of flank wear were investigated at the cutting edge of the dental bur. Fig. 15 shows flank wear as a function of cutting time when drilling borosilicate

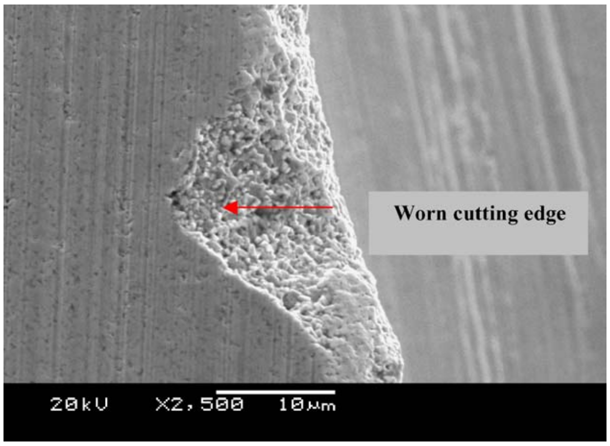


Figure 14 Worn cutting edge of an uncoated WC-Co dental bur after drilling borosilicate glass.

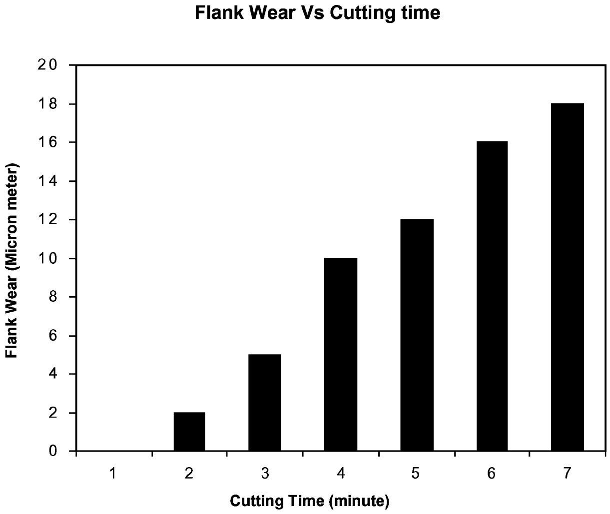


Figure 15 Flank wear chart as a function of cutting time for uncoated WC-Co dental burs drilling borosilicate glass.

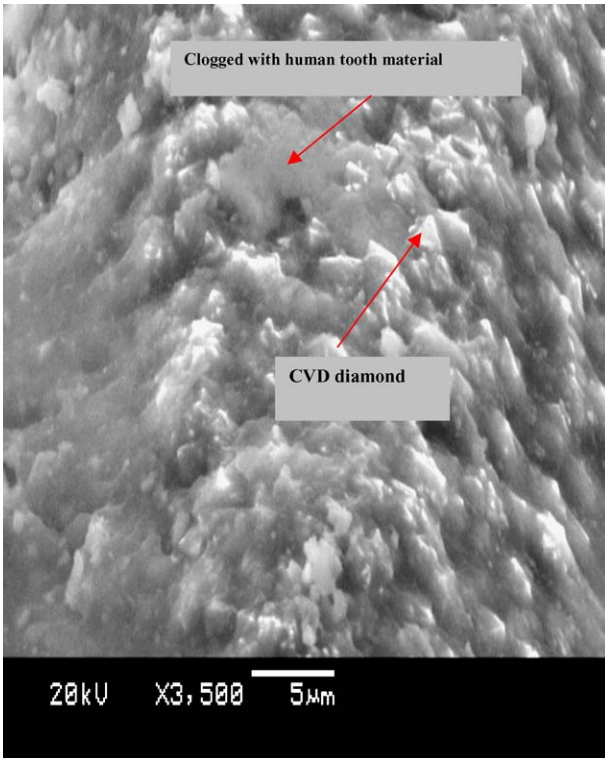


Figure 16 HFCVD diamond coated dental bur after drilling human tooth material.

glass. It is evident that the action of machining causes higher rates of flank wear on the cutting edge of a dental bur. Therefore, the cutting edges of WC-Co dental burs should have a minimum thickness of C.V.D. diamond of approximately 40 μm, which will enhance not only quality of cutting but also prolong the life of the bur [35].

Natural human teeth were drilled using a diamond coated dental bur. Previous studies have indicated that natural teeth should not be used for reduction tests because of the differences in hardness between enamel and dentine (Knoop hardness data: Enamel, 250–500 kg mm^{-2}; Dentine, 50–70 kg mm^{-2}) [36]. The cuts were made in the central fissure of the teeth. This permitted cutting three grooves in each tooth. Fig. 16 shows the S.E.M. image of diamond coated clinical WC-Co dental bur after testing. It is evident from the micrograph that materials such as dentine clog up on the bur reducing its abrasive performance. This observation explains why dentists use a fine water jet spray during the drilling and machining of natural teeth in order to remove dentine from the bur, and to keep the tooth cool in order to prevent overheating of the tooth pulp thus preventing the tooth from dying.

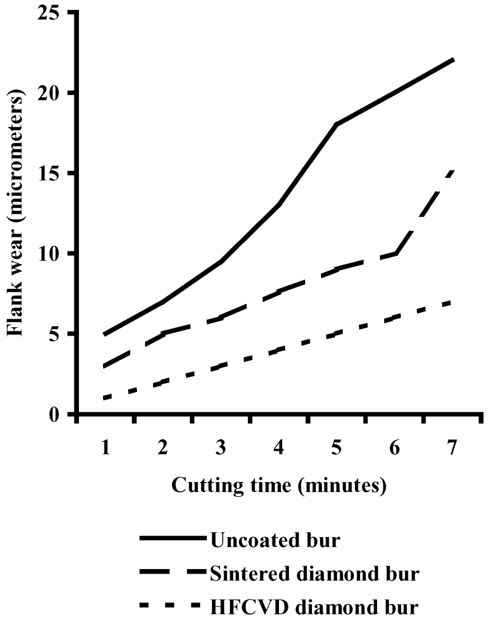


Figure 17 Flank wear of burs machining borosilicate glass.

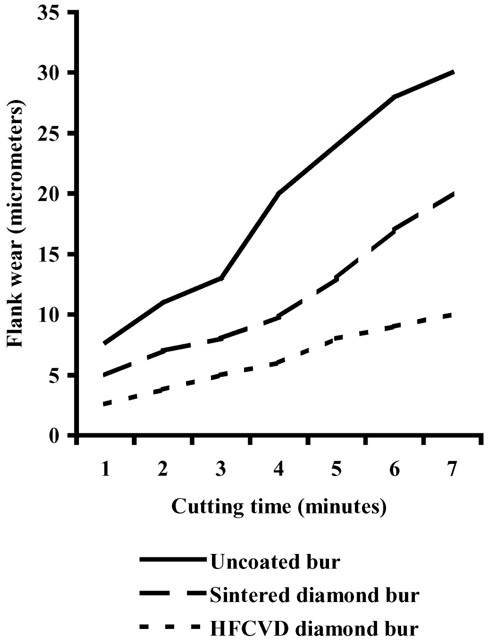


Figure 18 Flank wear of burs machining acrylic tooth material.

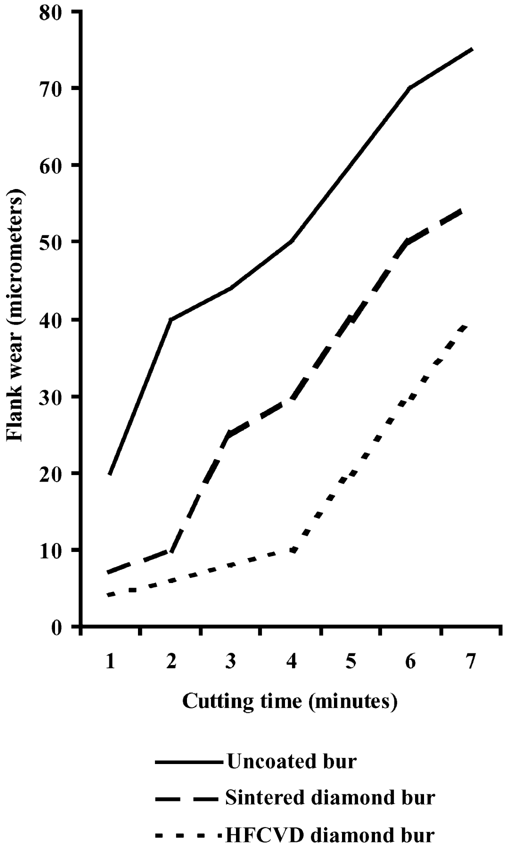


Figure 19 Flank wear of burs machining human tooth material.

Flank wear was measured at time intervals of 2, 3, 4, 5, 6, and 7 min machining duration. Flank wear on dental burs was measured from photomicrographs obtained using optical and scanning electron microscopes. Figs. 17–19 show flank wear measurements for each bur machining different dental materials.

4. Conclusions

PCD sintered diamond dental burs lose a significant proportion of their embedded diamond particles when they are used to cut both natural and synthetic dental materials. CVD diamond coated tungsten carbide-cobalt burs, on the other hand, remain intact and have the potential for a prolonged life.

Retention of the CVD diamond coating has been shown to be enhanced by an etching treatment, which removes some of the cobalt binder and thus provides better retention of the diamond coating. It is suggested that the cutting edge of these burs should have a diamond coating of up to 40 microns if they are to have an improved performance and an extended life.

Acknowledgments

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