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Diamond-Coated Cutting Tools for Biomedical Applications

M.J. Jackson, L.J. Hyde, W. Ahmed, H. Sein, and R.P. Flaxman

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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 as a method to improve cutting performance and tool life. WC-Co burs containing 6% Co and 94% WC substrate, with an average grain size of 1-3 µm, were used in this study. To improve the adhesion between diamond and WC substrates, it is necessary to etch away the surface Co and prepare the surface for subsequent diamond growth. Hot filament chemical vapor deposition (HFCVD), with a modified vertical filament arrangement, has been used for the deposition of diamond films. Diamond film quality and purity has been characterized using scanning electron microscopy (SEM) 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, borosilicate glass, and acrylic teeth. Flank wear has been used to assess the wear rates of the burs when machining biomedical materials such as those just described.

Keywords biomedical applications, CVD diamond, dental burs, microtools, wear

1. Introduction

Chemical vapor deposited (CVD) diamond films have attracted considerable interest in recent years for cutting-tool applications, including rotary tools and inserts, due to their excellent physical and chemical properties. However, the process has proved to be problematic for the deposition of adherent high-quality diamond films onto substrates, such as cemented carbides, stainless steel, and various metal alloys containing transition elements. 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 (Mo), tungsten (W), niobium (Nb), hafnium (Hf), tantalum (Ta), and titanium (Ti). The carbide layer formed allows diamond to form on it, because 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]

Table 1 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}

Substrates made from metals of the first transition group, such as iron (Fe), cobalt (Co), and nickel (Ni), are characterized by high dissolution and diffusion rates of carbon into those substrates (Table 1).[12] Owing to the absence of a stable carbide layer, the incubation time required to form a diamond is higher and depends on substrate thickness. In addition, these metals catalyze the formation of graphitic phases. This concept is reflected in the graphite-diamond ratio during the deposition process, yielding 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 Fe to Ni, 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.

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M.J. Jackson and **L.J. Hyde,** Center for Manufacturing Research, Tennessee Technological University, P.O. Box 5014, Cookeville, TN 38505; **W. Ahmed** and **H. Sein,** Department of Chemistry and Materials, Manchester Metropolitan University, Chester Street, Manchester, M1 5GD, UK; and **R.P. Flaxman,** Cavendish Laboratory, University of Cambridge, Madingley Road, Cambridge, CB3 0HE, UK. Contact e-mail: mjackson@tntech.edu.

The present work is concerned with diamond deposition on tungsten carbide cemented with 6 wt.% Co. 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 Co binder that is present to increase the toughness of the tool. Much effort has been directed at increasing the adhesion strength of the diamond films to the WC-Co substrates, including decarburizing the surface prior to deposition,[15] seeding WC-Co with diamond powder and annealing prior to deposition,[10] removing Co atoms at the surface using Co-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 considerably prolong the lifetime of WC-Co dental burs when applied to the machining of highly abrasive nonferrous metallic alloys, borosilicate glass, porcelain or acrylic teeth, natural human teeth, and ceramic materials. The presence of Co(Co) provides additional tool toughness, but it has an adverse effect on diamond film adhesion. The Co binder can suppress diamond growth, favoring the formation of nondiamond carbon phases, and resulting in poor adhesion between the diamond coating and the substrate.[20] Most importantly, it is difficult to deposit adherent diamond to untreated WC-Co substrates. Various approaches have been used to suppress the influence of Co and to improve adhesion. A significant factor for the adhesion of CVD diamond to WC-Co substrates is the mechanical interlocking that occurs at the coating-carbide interface. Therefore, it is essential to pretreat substrates for both significantly reducing the surface Co concentration and achieving a proper interface roughness, which represents an important step prior to coating process.[21] Chemical treatment using Murakami's reagent and acid etching has been used successfully for removal of the Co binder from the substrate surface, resulting in adherent diamond films.[22-23]

This paper reports on the results of an investigation of diamond film deposited on WC-Co dental burs using a hot filament chemical vapor deposition (HFCVD) system and describes the subsequent machining results on human teeth, borosilicate glass, and acrylic teeth. Even though considerable work has been done on CVD diamond deposition, very little work has been done in applying the process to cylindrical-shape substrates such as dental burs. Even less has been done on the performance and characterization of CVD diamond-coated dental burs.

2. Experimental

2.1 Substrate Preparation

For the CVD diamond-deposition process, the authors used two sets of laboratory tungsten carbide (WC-Co) dental burs (AT23 LR) with fine WC grain sizes (1 μm) 20-30 mm in length and 1.0-1.5 mm in diameter (supplied by Metrodent Ltd, UK). Prior to pretreatment, the cutting tools were ultrasonically cleaned in acetone for 10 min to remove loose residues from the surface. The following two-step chemical pretreatment procedure was used. The first step was etching, using Murakami's reagent ([10 g $K_3Fe(CN)_6$] + 10 g KOH + 100 mL water), and this procedure was carried out for 10 min in an ultrasonic bath to etch the WC substrate, followed by a rinse with distilled water. The second step was again etching for 10 s, using an acid solution of hydrogen peroxide [3 mL (96 wt.%) H_2SO_4 + 88 mL (30% wt.%) H_2O_2] 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 characterized by scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS).

2.2 CVD Diamond Deposition

Diamond films were deposited onto the cutting edge of the burs at 5 mm distances from the Ta wire filament, which measured 0.5 mm in diameter and approximately 10-12 cm in length. The coiled filament was held vertically within the vacuum-deposition chamber, as opposed to the commonly used horizontal filament position as used in HFCVD 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 CVD diamond deposition, the Ta filament was carburized 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 were carried out for substrate temperatures of 800-1000 °C. The filament temperature was measured using a two-color optical pyrometer and found to be between 1800-2100 °C, depending upon the filament position. Diamond films were characterized by SEM and EDS. Micro-Raman spectroscopy measurements were performed in backscattering geometry at room temperature using a Dilor XY (Spectro Analytical Instruments GmBH, Kleve, Germany) triple spectrometer equipped with a liquid-nitrogen-cooled, charge-coupled device detector and an adapted Olympus microscope.

2.3 Dental-Bur Machining: Drilling Experiments

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

After the dental burs were coated and examined for adhesion, the coated burs were used to machine a number of dental materials. The coated burs were compared with uncoated burs to distinguish them in terms of their drilling behavior. The drilling center, shown in Fig. 1, is constructed using three principal axes, each controlled using a direct current (DC) motor connected to a Motionmaster (GlobalSpec, Inc., Troy, NY) controller. A laser light source is focused onto the rotating spindle to measure the speed of the dental bur during drilling. Postmachining analysis was performed using SEM to detect wear on the flanks of the cutting edges.

The flank wear of the burs was estimated by SEM analysis at a selected time interval between 1 and 3 min. Prior to SEM analysis, diamond-coated burs were ultrasonically washed with 6 *M* H_2SO_4 solution to remove any unwanted

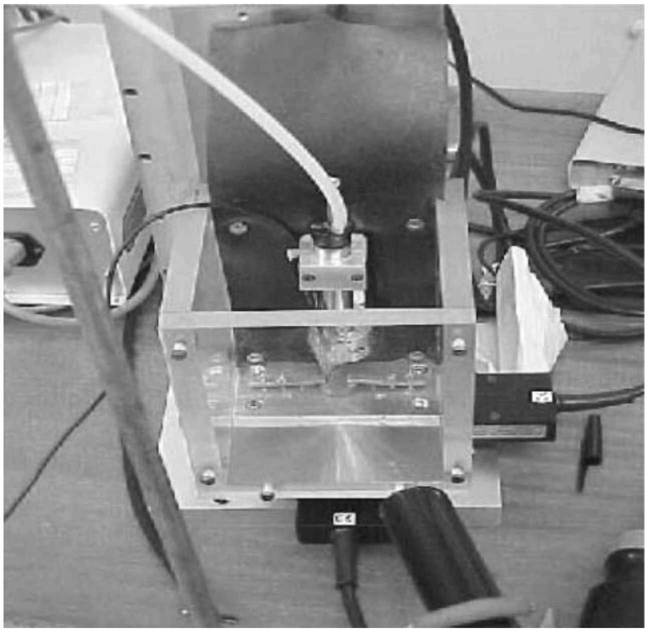

Fig. 1 Dental bur drilling machine

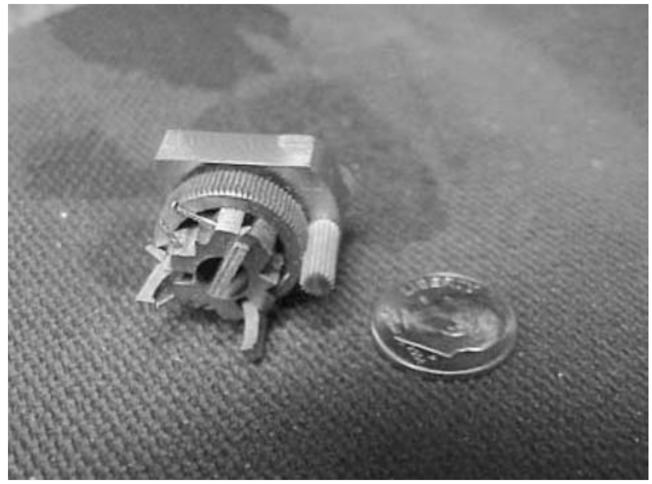

Fig. 2 Human tooth-clamping device

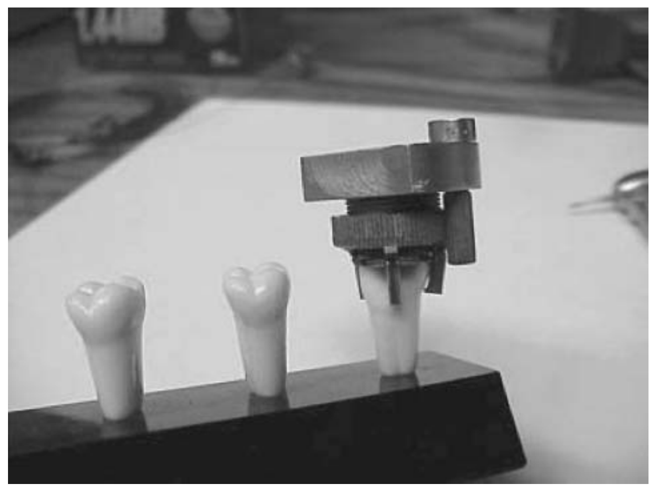

Fig. 3 Tooth-clamping device and driving stage used to locate clamping device onto surface of the tooth

machining material, which eroded onto the surface of the CVD diamond-coated bur. For comparison, conventional PCD (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. Figure 2 shows the basic construction of the clamping device.

The clamping device was affixed to the tooth to be machined and allowed 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. Figure 3 shows the driving mechanism attached to the clamping device and a tooth. The driving mechanism is attached to the air-operated handpiece that provides the power to drive the mechanism, clamp, and dental bur. Figure 4 shows the construction of the full assembly that allows machining of the tooth to take place. The bur was rotated at 20 000-30 000 rpm, with a feed rate between 0.2-0.5 mm/rev without water cooling. The uncoated and coated dental burs were compared with sintered diamond burs machining acrylic teeth, human teeth, and borosilicate glass (used to simulate the machining of porcelain teeth). Flank wear of the dental burs was estimated using SEM.

Again, the H_2SO_4 solution was used to remove machining detritus from the surface of the dental burs.

3. Results and Discussion

3.1 Substrate Preparation and Diamond Deposition

The crystallinity of as-grown films was analyzed using SEM. In addition, Raman spectroscopy (Kaiser holoprobe conventional Raman spectrometer) (Kaiser Optical Systems, Inc., Ann Arbor, MI) was used to monitor the carbon-phase purity of the deposited films. The chemical composition of the WC-Co surface was analyzed using EDS. The effects of chemical etching can clearly be seen on the WC-Co substrate surfaces (Fig. 5). Murakami's solution has chemically attacked the WC-Co substrate. Figure 5 shows that Co etching was achieved by the (H_2SO_4 + H_2O_2) solution, and it was apparent that no Co peaks could be detected in the EDS spectrum. In addition to this effect, acid etching produced a roughened surface.

Diamond layers were deposited on pretreated WC-Co dental burs. The diamond deposition rate on Mo wire, coated under identical condition, is about 1 μm/h. Thus, after a 5.5 h deposition time, a diamond layer of 5-6 μm thickness was obtained. On the surface of WC-Co dental burs, the deposition rate is expected to be much higher because the surface is much rougher at the cutting edge and on the tip of the burs compared with smooth substrates.

In addition, WC has a much higher affinity for diamond

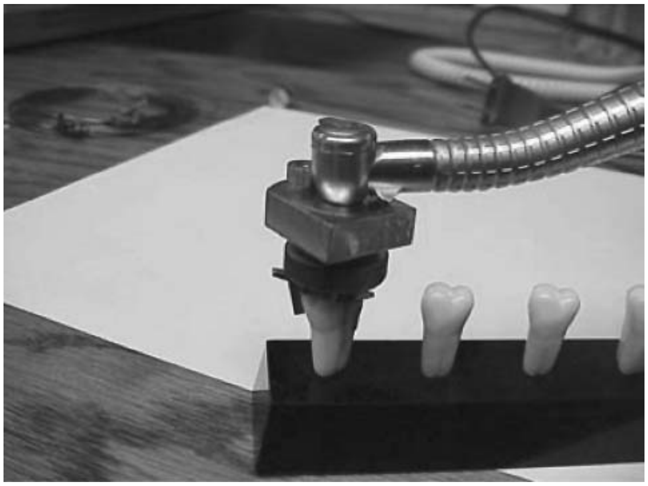

Fig. 4 Air-operated spindle unit attached to the clamping device and driving unit attached firmly to the tooth

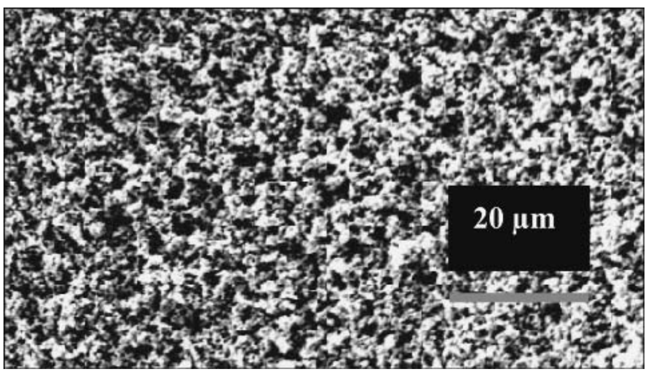

Fig. 5 The cutting edge of a dental bur after etching with acid etchant and Murakami's solution

nucleation than Mo, because the Murakami's solution and acid etching had etched Co away from the surface. Adherent diamonds on WC-Co dental burs, consisting of mainly (111) faceted diamond crystals were deposited, as shown quite clearly in Fig. 6. Micrographs of the burs show that the diamond film is uniformly coated onto the cylindrical substrate surface. 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 extremely desirable for dental machining applications. Raman analysis was performed to evaluate the quality and stress imparted in the CVD diamond films. The Raman spectrum shown in Fig. 7 shows a single peak at 1335 cm^{-1} (at the middle and at the end of the dental bur). The Raman spectrum also gives information about the stress in the diamond coating. The diamond peak 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 finding indicates that the stress is compressive. The results of Raman analysis on

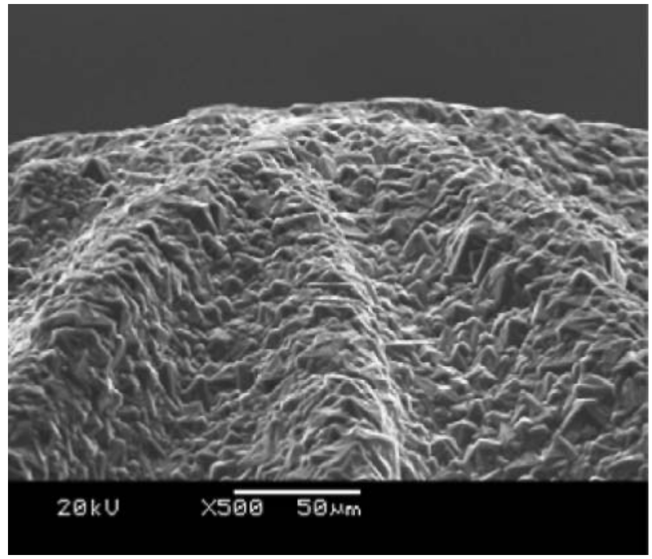

Fig. 6 (111) faceted CVD diamond-coated dental bur

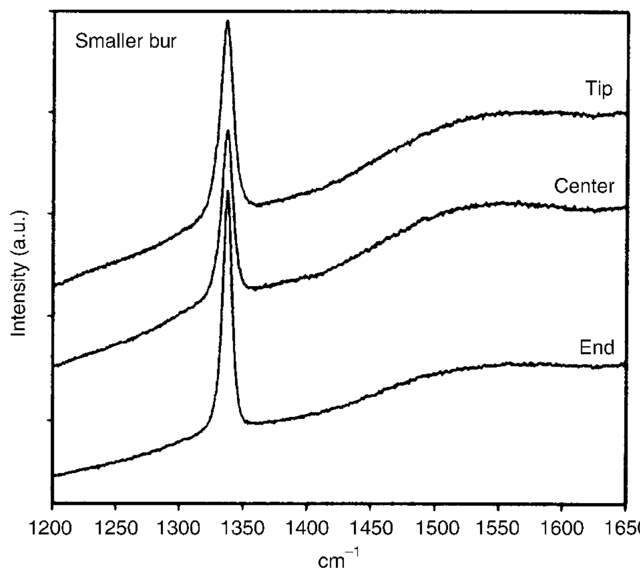

Fig. 7 Raman spectrum of the diamond film deposited onto the surface of a dental bur

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, which are uniformly distributed.

In contrast, Fig. 8 is a close-up view from a SEM micrograph of a conventional PCD sintered bur. The diamond particles are embedded onto the surface with a suitable binder matrix material such as Ni(Ni^{2+}). Typically, the surface is inhomogeneous and the sizes of particles range from 50-200 μm, causing considerable variation in the cutting performance of the tool. Figure 9 shows that the filament coil temperature changes from the end to the center of the filament. The diameter of Ta wire used in the experiments was 0.5 mm. It is assumed that the best thermal distribution is obtained at the

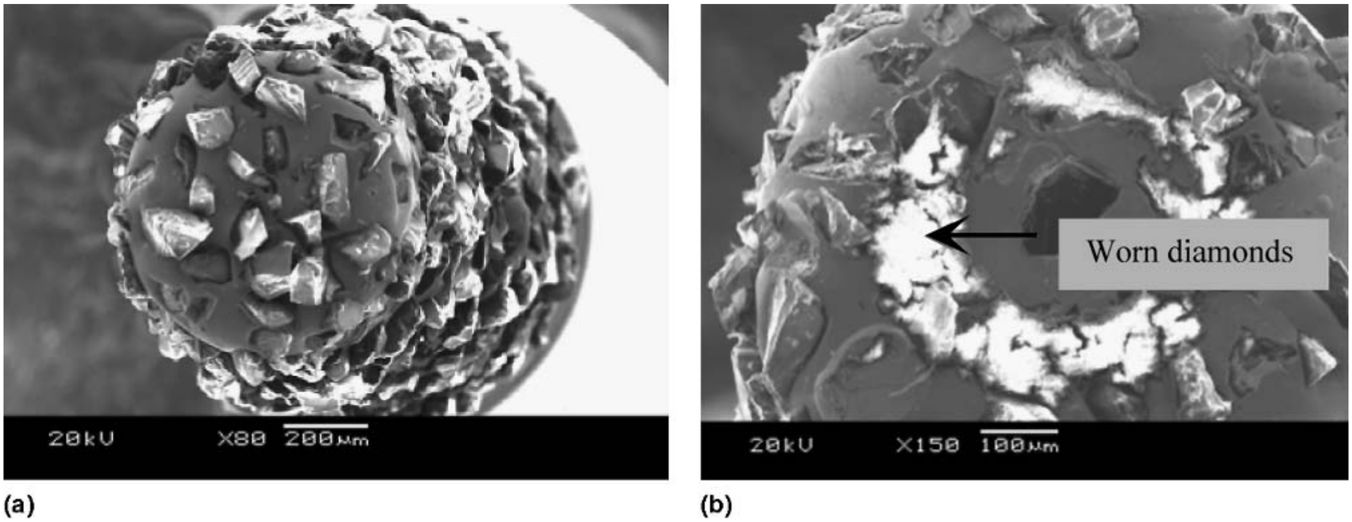

Fig. 8 **(a)** Inhomogeneous surface of PCD diamond-sintered bur; **(b)** PCD diamond-sintered bur after testing with glass

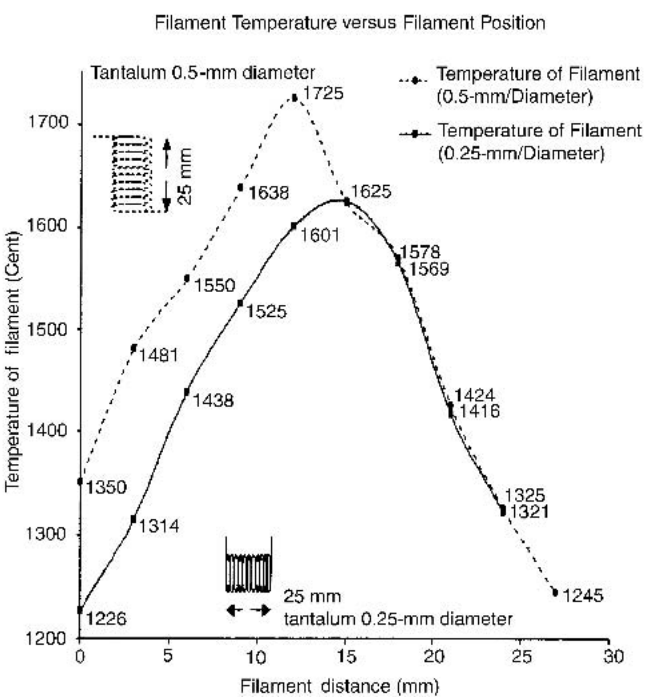

Fig. 9 Measurements of in-process filament temperature during the diamond-deposition process

center of the filament coil where the highest temperature was measured. Trava-Airoldi et al.[27] indicated that substrate temperatures can be different from the end to the center of the filament, and is more accentuated for Mo wire with a smaller diameter. This result could be due to heat being conducted through the substrate and 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 nonplanar dental burs.

3.2 Stress Analysis

Raman analysis was performed to evaluate diamond phase purity and the level of stress in the diamond film. The Raman spectrum shown in Fig. 7 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 result indicates that a compressive stress exists in the coating. Ager and Drory[30] investigated biaxial stresses in diamond film grown on Ti alloy by Raman spectroscopy and developed a model that describes the relationship between singlet and doublet photon scattering and the biaxial stress:

$$\sigma = -1.08(\upsilon_s - \upsilon_o) \text{ GPa for singlet phonon} \quad \text{(Eq 1)}$$

$$\sigma = -0.384(\upsilon_d - \upsilon_o) \text{ GPa for doublet phonon} \quad \text{(Eq 2)}$$

where υ_o is 1332 cm^{-1}, υ_s is the observed maximum of the singlet in the spectrum, and υ_d is the observed maximum of the doublet in the spectrum. From Eq 1 and 2 one obtains

$$\sigma = -0.567(\upsilon_m - \upsilon_o) \text{ GPa} \quad \text{(Eq 3)}$$

where the observed peak position υ_m is assumed to be between the singlet and the doublet, i.e., $\upsilon_m = \upsilon_s + \upsilon_d$.

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

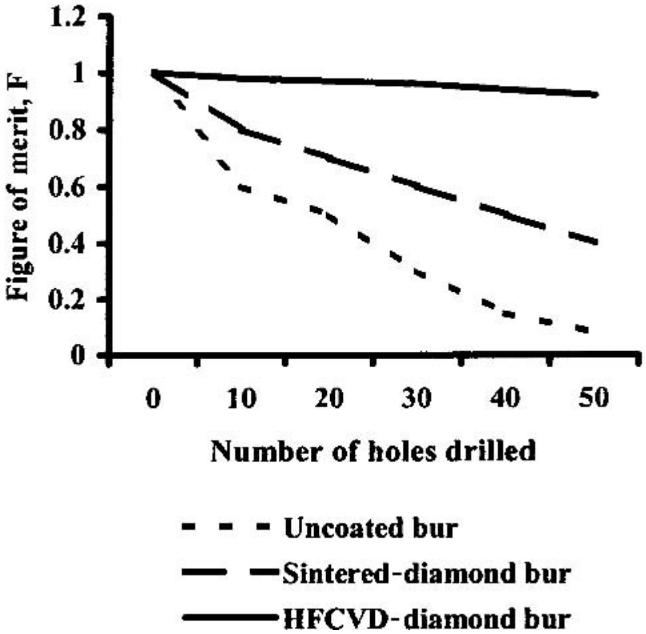

Fig. 10 Figure of merit for dental burs drilling borosilicate glass

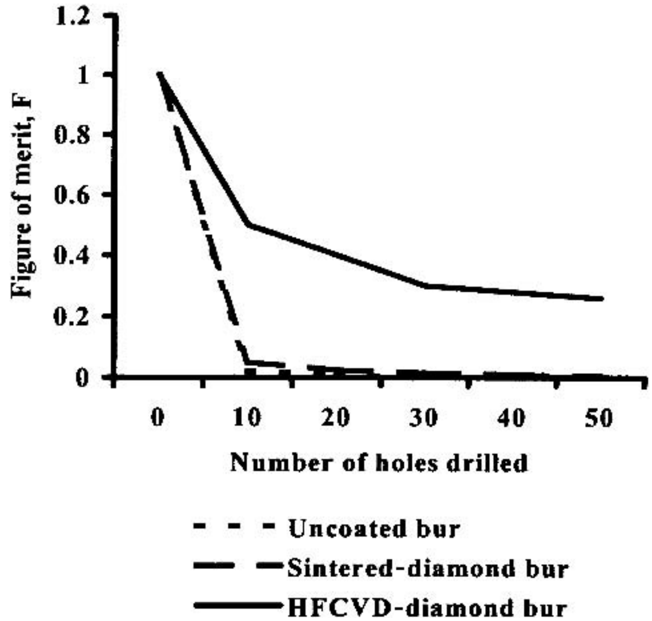

Fig. 12 Figure of merit for dental burs drilling human tooth material

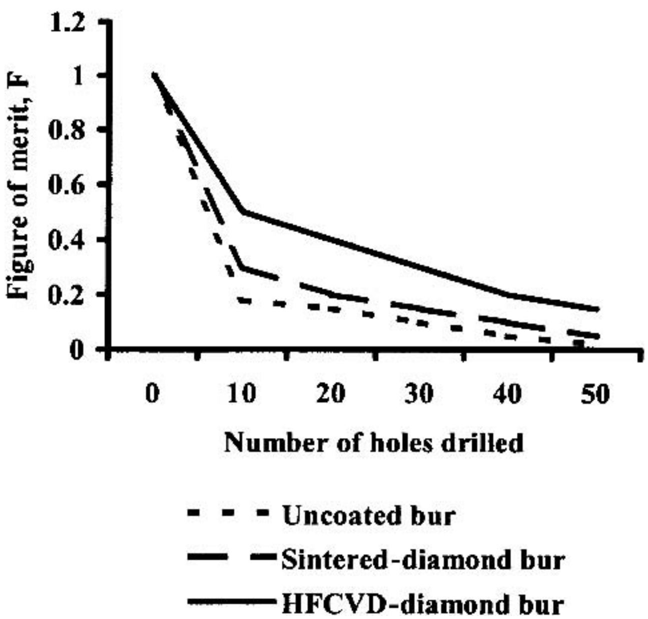

Fig. 11 Figure of merit for dental burs drilling acrylic material

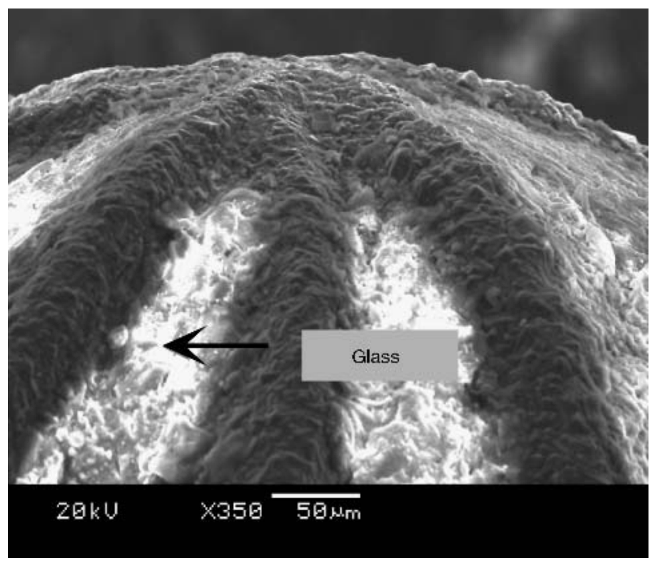

Fig. 13 Cutting edge of dental bur after drilling borosilicate glass

conventional CVD 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 teeth, and borosilicate glass.

A sequence of fifty drillings was used in each drilling experiment. The sharpness and initial condition of the burs were inspected by an optical method after the burs had drilled 10 holes in sequence. An abrading coefficient of drilling, C_a, has been defined as a quality criterion for small drilling tools and is defined as the ratio between the total abraded area of the bur, 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:

$$C_a = S/(D_b - W_b) \quad \text{(Eq 4)}$$

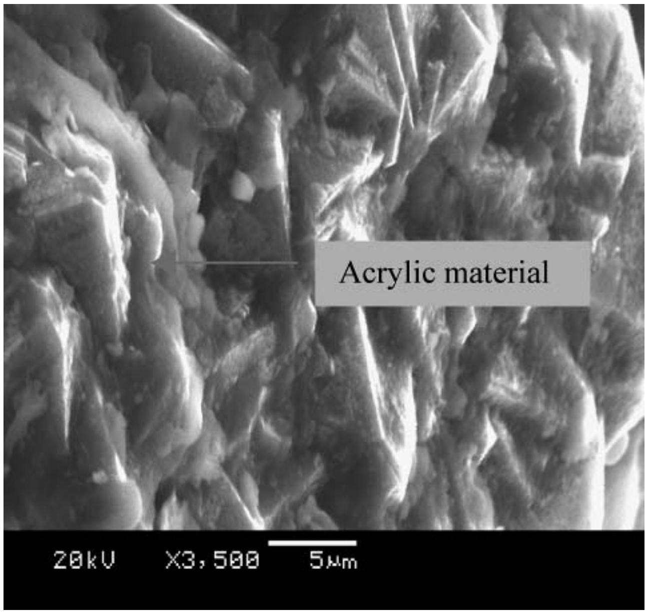

Fig. 14 Magnified image of the dental bur after drilling acrylic tooth material

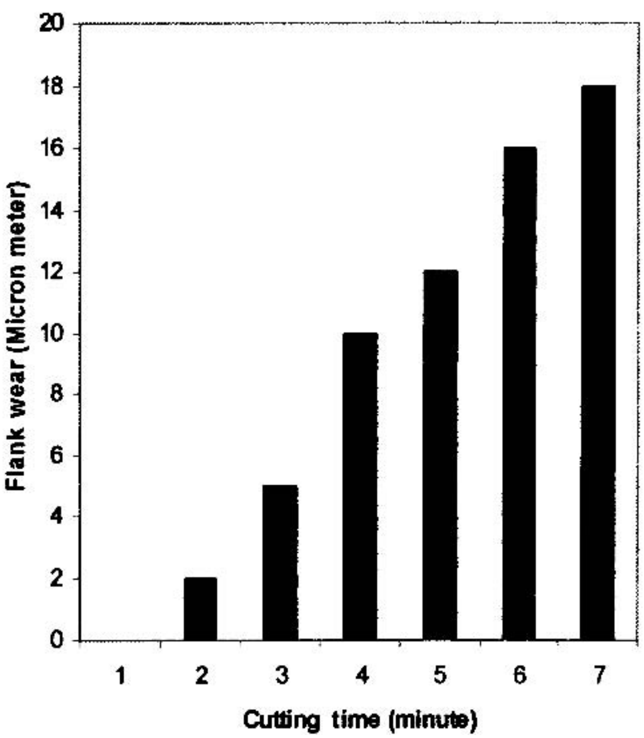

Fig. 15 (Flank wear versus cutting time) worn cutting edge of an uncoated WC-Co dental bur after drilling borosilicate glass

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., $W_b \approx 0$). The cutting will therefore have an abrading coefficient close to unity. One must remember that the quality of machining is dependent on the cutting speed, V_c, defined by the ratio of the

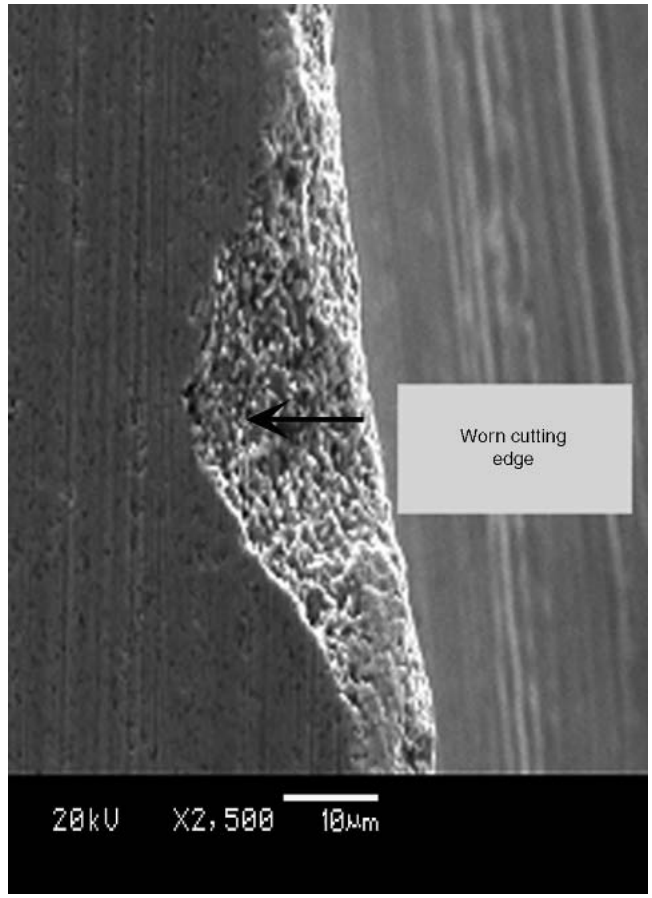

Fig. 16 Flank wear chart as a function of cutting time for uncoated WC-Co dental burs drilling borosilicate glass

drilling depth by the drilling duration. A comparative factor of merit for the dental bur is defined as:

$$F = C_a/V_c \quad \text{(Eq 5)}$$

In Eq 5, F is directly related to the lifetime of the dental bur for a specific drilling process. Figures 10-12 show the results of drilling the dental materials with the three types of burs described.

Figure 8(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. Clearly 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 al.[31] also reported that the significant loss of diamond particles occurred during cutting with the commercially sintered diamond bur. In addition, the metallic Ni(Ni^{2+}) binder shows major defects generated by pulled-out particles.[32] Figures 13 and 14 show a SEM image of CVD diamond-coated laboratory bur after drilling experiments on borosilicate glass and acrylic teeth, respectively, for 5 min at a cutting speed of 30 000 rpm.

After machining, it is evident that the diamond films are still intact on the pretreated WC substrate and that the diamond

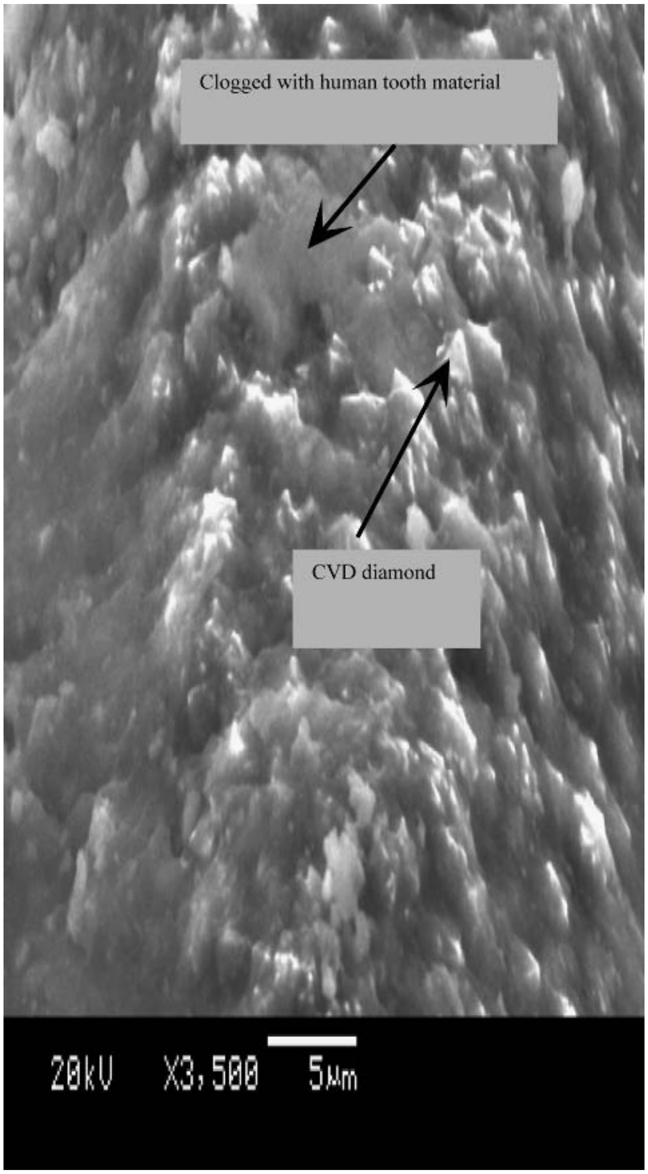

Fig. 17 HFCVD diamond-coated dental bur after drilling human tooth material

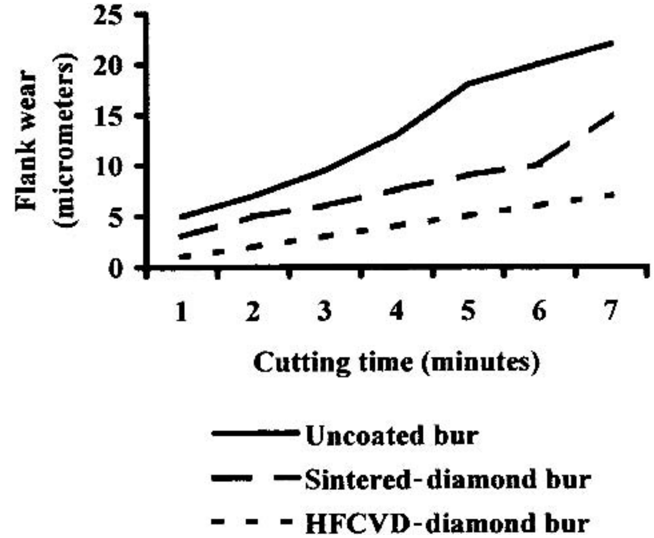

Fig. 18 Flank wear of burs machining borosilicate glass

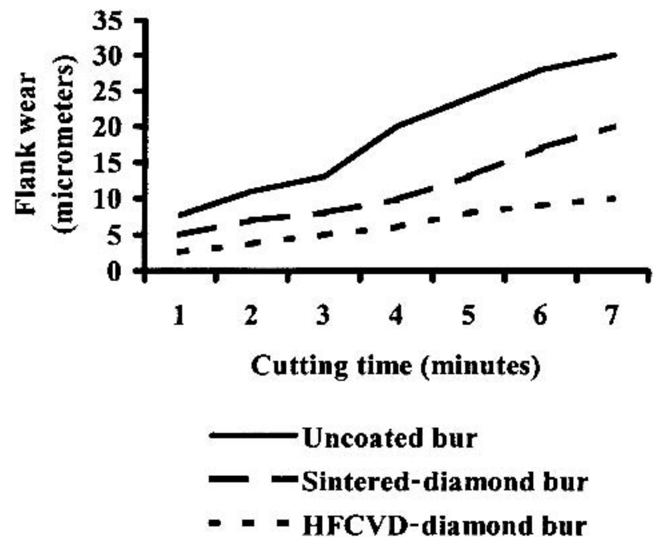

Fig. 19 Flank wear of burs machining acrylic tooth material

coating displayed good adhesion. Also, there is no indication of diffusion wear after the initial test for 50 holes. However, the machined materials, such as glass pieces, erode the cutting edge of the diamond dental bur as adhesive wear was observed (Fig. 13). After conducting experiments on acrylic teeth, the mechanisms of wear involve adhesion as well as abrasion. Figure 14 shows that when an increased rate of abrasion was used, inorganic fillers from acrylic teeth adhered to the cutting-tool surface in localized areas.[33]

Figure 15 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 flank wear along the cutting edge of the bur. The areas of flank wear were investigated at the cutting edge of the dental bur. Figure 16 shows flank wear as a function of cutting time when drilling borosilicate glass. It is evident that the action of machining causes higher rates of flank wear on the cutting edge of dental bur. Therefore, the cutting edges of WC-Co dental burs should have a minimum thickness of CVD diamond, which will enhance not only quality of cutting but also prolong the life of the bur.[34]

Natural human teeth were drilled using a diamond-coated dental bur. Previous studies have indicated that teeth should not be used for reduction tests due to the differences in hardness between enamel and dentine (Knoop hardness data: enamel, 250-500 kg/mm^2; dentine, 50-70 kg/mm^2).[35] The cuts were made in the central groove of the teeth. This strategy permitted cutting three grooves in each tooth. Figure 17 shows the SEM image of diamond-coated clinical WC-Co dental bur after testing on extracted teeth. It is evident from the micrograph that the tooth materials such as dentine clog interstices on the bur, reducing its abrasive performance.

A series of machining experiments were conducted using

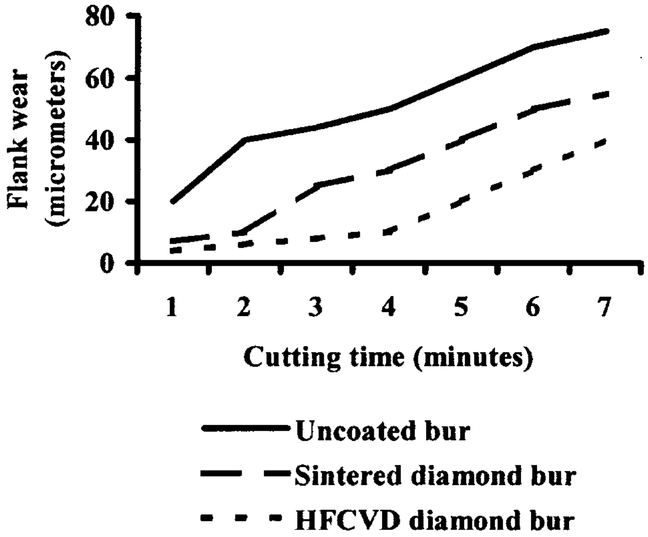

Fig. 20 Flank wear of burs machining human tooth material

uncoated burs, HFCVD dental burs, and sintered diamond burs when machining extracted human teeth, acrylic teeth, and borosilicate glass. The teeth were machined using the apparatus shown in Fig. 2-4. The life of the burs in the machining sense was measured by comparing the amount of flank wear exhibited by each type of dental bur. The flank wear was measured at machining time duration intervals of 2, 3, 4, 5, 6, and 7 min. Figure 18-20 show the flank wear measurements for each bur machining different dental materials.

Once again, the dental burs were examined using optical and SEM techniques and were found to exhibit trends similar to those burs associated with drilling experiments.

4. Conclusions

The etching treatment of the surface of the substrates described here to remove the Co surface layer results in much better adhesion of diamond. The PCD-sintered diamond bur loses significant proportions of embedded diamond particles during the abrasive machining procedure, whereas CVD diamond-coated diamond burs remain intact with the potential of prolonging tool life. A thicker coating of CVD diamond at the cutting edges is expected to give longer bur life and a much better quality of drilling and machining. The performance and lifetime of CVD-coated dental bur is far superior to the sintered bur and the uncoated WC-Co bur. Further work is required to study the effects of diamond film adhesion and thickness of the coating at the cutting edge on dental tool life.

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