Advances in nanotechnology education and research: Surface Engineering of Surgical and Dental Tools using Diamond Films

W. Ahmed¹, H. Sein², J.O. Uhomoibhi³, M.J. Jackson⁴

 ^{1.2}School of Computing, Engineering & Physical Sciences, University of Central Lancashire, Preston, PR1 2HE. UK,
³Faculty of Computing and Engineering, University of Ulster, Newtownabbey, Northern Ireland, BT37 0QB, UK,
⁴Department of Mechanical Engineering and Centre for Manufacturing Research, Tennessee Technological University, Cookeville, TN 38505, USA

> wahmed4@uclan.ac.uk¹, j.uhomoibhi@ulster.ac.uk³, jacksomj@purdue.edu⁴

Abstract

Dental and surgical tools play a critical role in the removal of parts and unwanted impurities from the head, mouth and the body. Often during operation these are damaged, have reduced lifetime or poor performance. For example, conventional dental burs are made of a tool with diamond particles embedded in the surface with binder materials. Often these metal containing binders are toxic and may harm the patient. However, we can improve performance, lifetime and eliminate harmful effects by using a technology known as chemical vapour deposition to deposit high quality adherent diamond films onto the surface. In this paper, we present work on the process optimization of growing diamond films onto dental and surgical tools. The performance, lifetime and process characteristics have been investigated and results show that the CVD diamond coated tools exhibit superior properties. The results obtained are generic and may be applied to a wide range of cutting tools for different applications.

Introduction

Diamond is one of the most interesting carbon polymorphs due to its extreme chemical and physical properties [1,2]. Diamond cutting tools are often one of the choices for machining of high-strength and high abrasive non-ferrous alloy [3]. Chemical vapour deposition (CVD) of diamond films on cobalt-cemented tungsten carbide (WC-Co) has been the subject of commercial interest in the last decade. The possibility to combining the toughness of cemented carbide with the hardness of diamond results in outstanding wear resistance.

The most commonly used dental diamond burs are manufactured by imbedding diamond particles onto a metal matrix using a suitable binder material containing nickel ions. These burs have several limitations mainly due to the heterogeneity of diamond crystallites, contamination of oral tissue and variation in the product performance. There is no universal specification of the diamond particles sizes imbedded into the binder matrix in order to ensure reproducible and consistent cutting performance. Recently, CVD has been used for the fabrication of new generation dental burs [4] with continuous diamond film offering improvement in cutting efficiency, longer life and better technical performance are expected for the components made of diamond –coated carbide. Much of the work on the CVD of diamond has been carried out on flat substrates. Although, cutting tools such as drills and inserts have been successfully coated with diamond-based coatings; there are only a few reports of diamond deposition onto rotary cutting tools, such as cylindrical abrasive pencils and small spiral drills [5].

In this study, we report the deposition of uniform diamond films onto the cutting edges of WC-Co dental burs used in dental laboratory and clinical surgery using a modified hot-filament chemical vapour deposition (HFCVD) system. The filament is mounted in a vertical arrangement with the dental bur held concentrically in between the filament coils, as opposed to the horizontal position commonly used in the HFCVD system configurations. This new vertical

filament arrangement used in the modified HFCVD system enhances the thermal distribution and ensures uniform diamond coating [6]. The Co cemented dental burs operate at extremely high cutting speeds in the range between 3,000 to 300,000 r.p.m [7]. Such high operating speeds impose stringent demands on the cutting surfaces. The diamond coatings must be tough, adherent, hard and wear resistant in order to enhance overall performance and life of the tools.

Material Preparation, Deposition and Testing

The present studies involved preparation of substrates, the deposition process of samples and the testing. The (WC grain preferable grade 1-3 mm size- 6 wt.% Co) dental burs used were 20-30 mm in length and 1-1.5 mm in diameter. Prior to diamond deposition the dental burs were ultrasonically cleaned in acetone for 10 minutes to remove any surface impurities. The poor adhesion of deposited diamond films on WC-Co surfaces can be caused by mismatch of thermal expansion coefficient and interaction between carbon and thin layer of cobalt on the surface. It can lead to catastrophic film failure in metal cutting due to presence of Co binder [8]. The Co binder in WC-Co burs suppresses diamond nucleation and causes deterioration of diamond film adhesion due to the large solubility of carbon and diffusivity in cobalt. These prolong the incubation period for diamond nucleation and enhance the accumulation of sp2 graphite at diamond-carbide interface [9]. To eliminate this problem, it is usual to pre-treat the WC-Co dental bur surface prior to CVD diamond deposition. The WC surface was etched away with Murakami solution [K3Fe(CN)6: KOH: H2O=1:1:10] and surface Co was removed by acid etching followed by ultrasonically washed in distilled water [10].

Diamond synthesis was performed in a stainless steel HFCVD chamber of internal diameter of 200 mm. The gas phase mixture of hydrogen (purity, 99.99%) and methane (purity, 99.99%) with CH4/H2 volume ratio fixed at 1.0 % CH4 with excess hydrogen) was activated by tantalum filament (0.5 mm in diameter) wound in a 10 mm internal diameter spiral. The dental bur was positioned centrally and coaxially within the spiral vertical filament at 5 mm distance (Fig.1). The filament temperature (1800-2100°C) was monitored by a two-color pyrometer. The substrate temperature was measured to be between 800-950 °C. The total pressure of the gas mixture in the reactor was 2.66 kPa (20 Torr) and flow rate was 200 sccm. The diamond deposition was carried out for 5-15 hrs with a single used filament. The deposited diamond films were analyzed by scanning electron microscopy (JEOL JSM-5600LU) and 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.

Erosion tests were carried out in a high velocity air-sand erosion facility capable of attaining impact velocities of up to 360 ms-1. In the sandblasting rig, sand was injected into an air stream and accelerated down to a 10 mm diameter stainless steel tube, 0.8 meter in length, into the erosion chamber where it strikes the diamond deposited substrate. The sand used was a blend of dental laboratory grade. The broad size distribution in the range 100-400 μ m and average diameter of sand was 250 μ m.

Feed rate of 5.9 kgm-2 s-1 was employed for test. The nozzle to stand off distance was 40 mm and test on the substrate area was average 10 mm in diameter. All tests were conducted using air-sand jet impact angle of 90° as the erosion of brittle materials is at normal impingement. The diamond-coated bur has to be rotated 120° at a time for three intervals. The tests were interrupted every 10 min in order to monitor the diamond coating [11].

Figure 1: HFCVD Chamber and its schematic





The machining unit was specifically constructed with a water-cooling system so that maximum spindle speed of 250,000 revolutions per minute (rpm), feed rates of between 5-20 μ m per revolution and cutting speeds in the range 100 to 200 m/min for cutting human teeth with clinical bur. Separate set up for laboratory bur was used to operate at 3,000-30,000 rpm with a feed rate of 0.2-0.5 mm/rev without water-cooling. The flank wear of the burs was estimated by SEM analysis at pre-selected time interval of one minute. Prior to SEM analysis diamond coated burs were ultrasonically washed with 6M H2SO4 solutions to remove any unwanted machining material, which eroded onto surface of CVD diamond coated bur. For comparison, conventional PCD (polycrystalline diamond) sintered bur as reference with different geometry were also tested on the same substrates materials.

bstract

The diamond coatings deposited by HFCVD on dental burs, whose surface was roughened by Murkami's solution prior to deposition. EDX spectra confirmed the removal of the Co binder from the surface. Surface morphology of predominately (111) faceted octahedral shape diamond films shown in fig.2 (a) was obtained after deposition for 10 hrs.



Fig 2: Topical view of diamond coated dental bur (a) and cross section of diamond film on coated dental bur (b)

In general, diamond film synthesized by vapour phase methods is polycrystalline and exhibit columnar crystal growth [12]. The SEM micrograph of cross section of 13μ m thick polycrystalline diamond is shown in fig.2 (b). Results of

the erosion tests on diamond coated dental bur were compared with the untested diamond coated bur [fig.3(a and b)]. The coating failure occurred, the only surface features observed were minor amounts of micro or sub-micro chipping of the crystal. Diamond has greatest resistance to plastic deformation of any material and damage is always of a wholly elastic nature, with no traces of plastic deformation [13].

Fig.4 (a) shows a SEM micrograph of a CVD diamond coated laboratory dental bur at the cutting edge. The film is homogeneous with uniform diamond crystal sizes; typically the crystal sizes are in the range of 6-10 μ m. As expected the surface morphology is rough making the



Fig 3: Diamond film before erosion test (a) and after erosion test (b)

dental burs extremely desirable for abrasive cutting applications. Fig.4(b) shows an SEM image of CVD diamond coated laboratory bur after machining tests on borosilicate glass for 5 min at a cutting speed of 3,600 rpm. It is clearly indicated 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 after the initial test for 500 holes. However, the machining materials such as glass pieces are eroded onto cutting edge of diamond coated dental bur as adhesive wear were observed. In contrast, fig. 5(a) is a close up view from SEM micrograph of a conventional polycrystalline diamond (PCD) sintered bur used as a reference. The diamond particles are imbedded onto surface with a suitable binder matrix material such as nickel (Ni2+).



Fig 4: Cutting edge of diamond coated dental bur (a) and after testing with glass (b)

Typically the surface is inhomogeneous and sizes of particles ranging from 50-200 μ m causing considerable variation in the cutting performance of the tool. Fig.5 (b) shows the morphology of a sintered diamond bur after being tested on borosilicate glass at a cutting speed of 3,600 rpm for 5 minutes with an interval at every 30 sec. It is clearly evident that there is significant removal of diamond particles from the surface of the tool after 500 holes. As expected there is the deterioration of the abrasive performance of the PCD sintered diamond dental burs. Borges et al also reported significant loss of diamond particles occurred during cutting with the commercial sintered diamond bur. In addition, the metallic nickel (Ni2+) binder shows major defects generated by pulled-out particles [4].



Fig 5: Inhomogeneous surface of PCD diamond sintered bur (a) and PCD diamond sintered bur after testing with glass (b)

Fig. 6(a) and (b) show SEM images of uncoated WC-Co dental bur before testing on the borosilicate glass. For machining test, a material workpiece (borosilicate glass) was prepared and aligned orthogonal to the dental cutting tool. Trench machining was the method used to generate wear on the tool with cutting speed of 3,600 r.p.m. After machining, fig.6(c) shows that the uncoated WC-Co bur has lost their cutting geometry on the bur head and displayed significant wear along the cutting edge of the bur fig.6 (d). The areas of flank wear were investigated at the cutting edge of the dental bur.

Fig. 6 (a) and (b): Uncoated WC-Co dental bur before machining with glass



Fig 6 (c) and (d): Uncoated WC-Co dental bur after machining with glass



Figure 7 shows that the areas of flank wear is plotted against time for the performance of 3 different dental burs after being tested on borosilicate glass. There is an indication of a longer duty cycle of machining that could cause higher rate of flank wear on the cutting edge of tool. It is evident that HFCVD diamond coated bur has significant wear resistant compared to other types of dental burs. Therefore, the cutting edge of WC-Co dental bur should have significantly thick of CVD diamond film, which will enhance not only quality of the cutting but also prolong the tool life [14].

Fig 7: Flank Wear (µm) versus cutting time (min) for 3 types of dental burs



Natural human teeth were cut using a diamond coated clinical bur. The cuts were made in the central groove of the teeth. This permitted cutting three grooves in each tooth. Fig. 8 shows a SEM image of diamond coated clinical WC-Co dental bur after testing on extracted teeth. It is evident from the SEM shown that the tooth materials such as dentine clog up the bur surface reducing its abrasive performance.



Fig 8: CVD diamond coated clinical bur after testing on human teeth

Raman analysis was performed in order to evaluate the quality and stress imparted in CVD diamond films. The Raman spectrum shown in fig. 9 shows that at the tip, centre and end of cutting tool a single sharp peak at 1336, 1337 and 1337 cm-1 respectively was observed for different positions. The result of Raman analysis on WC-Co substrates at several different locations on the tool has shown indications of compressive stress in the coating [15]. There are a lot of parameters associated with the manufacturing of dental tools. Diamond films do offer improvement on grounds of wear and tear and the engineering and understanding of some of the processes of coating remains important as the technology develops. Fig 9: Raman spectrum of diamond films



Conclusions

Erosion tests show that adherent diamond films offer significantly better erosion resistance. Therefore deposition of heteroepitaxial diamond on metal surface offers protective coating on uncoated substrate. Etching treatment of surface of the substrate and removing the surface cobalt resulted in rough and pitted surface, which create ideal condition for diamond deposition.

The PCD sintered diamond bur loses significant proportions of imbedded diamond particles during the abrasive machining procedure where CVD diamond bur remain intact with potential of prolong tool life. Thicker coating of CVD diamond at the cutting edges expected to give tool longer life and better quality of machining. The morphology of diamond films exhibits rough surfaces creating micro-cutting edge on cutting surface. We plan to engage in further works to encompass investigation and comparison with other types of films. The present research has shown the performance and lifetime of CVD diamond bur are much superior to the sintered bur and uncoated WC-Co bur.

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