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Chemical vapour deposition of microdrill cutting edges for micro- and nanotechnology applications

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Conventional cemented tungsten carbide-cobalt (WC-Co) microdrills generally have a low cutting efficiency and short lifetime mainly because they operate at very high cutting speeds. Since it is relatively expensive to make microtools it is highly desirable to improve their lifetime and in-service performance. Microtools used to make microelectronic and mechanical systems (M.E.M.S) devices with sharp cutting edges, such as milling or drilling tools need protective coating in order to extend life and improve performance. One method of achieving this objective is to use a suitable surface engineering technology to deposit a hard wear resistant coating, such as diamond. Diamond has excellent mechanical properties, such as ultra-high hardness and a low friction coefficient. One of the most promising surface treatment technologies for depositing diamond onto complex shaped components is chemical vapour deposition (CVD). However, CVD of diamond coatings onto the cemented WC-Co tool has proved to be problematic. Binder materials such as cobalt can suppress diamond nucleation resulting in poor adhesion between the coating and substrate. In this paper the effects of pre-treated substrate material on the coating structure are reported. The morphology and the crystallinity of the as-grown films was characterised by using scanning electron microscopy (SEM). Raman spectroscopy was used to assess the carbon-phase purity and give an indication of the stress levels in the as-grown polycrystalline diamond films. The diamond coated tools have potential applications in micro- and nanomachining of micro- and nano-sized components used in M.E.MS.

1. Introduction

Diamond has a unique combination of excellent physical and chemical properties making diamond a promising material for numerous applications [1]. For example, diamond films are of interest for tribological applications because of their high hardness, low friction coefficient, high wear resistance and chemical inertness. Diamond coatings find use in cutting tools and biomedical applications. Recently, CVD has been used for the fabrication of new dental burs [2] with continuous diamond film offering improvement in cutting efficiency and longer life. Much of the work on the CVD of diamond has been carried out on flat substrates. Although, cutting tools such as drill bits, hacksaw blades and tool inserts have been successfully coated with diamond-based coatings; there are only a few reports of diamond deposition onto micro-tools, such as cylindrical abrasive pencils and small spiral drills [3]. In this study, we report the deposition of uniform diamond films onto the cutting edges of cemented WC-Co microdrills used in manufacture of micro and nanoscale components using a modified hot-filament chemical vapour deposition (HFCVD) system. The filament is mounted in a vertical arrangement with the microdrill 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, ensuring higher nucleation densities, higher growth rates and uniform coating covering [4]. These cemented tungsten carbide microdrills operate at extremely high cutting speeds in

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the range between 10,000 to 300,000 rpm [5]. Such high operating speeds impose stringent demands on the cutting surfaces and the coating deposited. It is required that the coating deposited is tough, adherent, hard and wear resistant in order to enhance overall tool performance and lifetime.

2. Experimental

The WC-Co microdrills used were 20 mm in length and 1 mm in diameter. The Co concentration was about 6%. Prior to diamond deposition the microdrills were ultrasonically cleaned in acetone for 10 minutes to remove any loose residues or surface impurities. The poor adhesion of deposited diamond films onto cemented tungsten carbide surfaces can lead to catastrophic film failure in metal cutting [6]. The cobalt binder suppresses diamond nucleation and causes deterioration of diamond film adhesion [7]. To eliminate this problem, it is usual to pre-treat the WC-Co microdrill surface prior to CVD diamond deposition. The WC-Co microdrills were etched in Murakami solution (10 g $K_3(Fe(CN)_6)$ + 10 g KOH + 100 ml water) for 20 minutes in an ultrasonic bath. The surface cobalt was removed by a 10 second acid etch (3 ml H_2SO_4 (96%) + 88 ml (30%) H_2O_2) followed by ultrasonically cleaning in distilled water [8].

Diamond films were deposited onto the tip of the microdrills. The microdrill-to-filament distance was typically 4–5 mm with the spiral diameter of 10 mm and length 25 mm. A schematic diagram of the HFCVD system is presented in Fig. 1. The system had the facility to carry out bias-enhanced nucleation experiments. The chemical treatment alone was sufficient to induce diamond nucleation and growth without the need for abrasive pre-treatment prior to coating. However, such experiments were not performed in this study. The gas sources are 1% methane in excess hydrogen with hydrogen flow rate of 100 sccm and methane flow rate of 1 sccm. The deposition time and pressure in the vacuum chamber was 5 hours and 26.6 mbar respectively. The filament temperature was measured using an optical pyrometer and found to be between 1480–2100°C depending upon the filament position. The as-grown films were analysed for crystallinity using scanning electron microscopy (SEM) (Jeol JSM-5600LU). In addition, Raman spectroscopy (Kaiser Holoprobe) was used to monitor the carbon-phase purity of the deposited films. Chemical compositions of the microdrills were obtained using EDS (Oxford Pentafet).

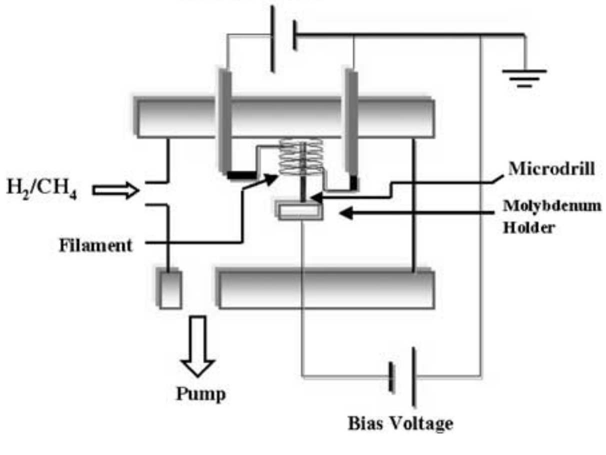


Figure 1 Schematic diagram of the modified HFCVD system.

3. Results and discussion

The filament material and its geometry were important factors to consider in order to obtain improved coatings using the new CVD method. Therefore, in order to optimise both the filament wire diameter and the filament assembly/geometry it was necessary to understand the temperature distributions of the filament. Fig. 2 show the way in which coil filament temperature changes from the end to the centre of the filament, which was 12.5 mm from either of the clamped ends. Using two different diameters of tantalum wire (0.25 and 0.5 mm) the results obtained gave similar trends. The best thermal distribution was obtained at the centre of the filament coil where highest temperature was measured. The existence of a thermal gradient in between the coiled filament can affect a number of film properties, such as adhesion, stress and hardness. Trava *et al.* [9] indicated that there could be a variation in the substrate temperature from the end to the centre of the filament. This could be due to the heat conduction through the substrate and the heating distribution from the hot filament. Our results indicated that the best thermal distribution and diamond growth uniformity was obtained using tantalum wire of 0.5 mm in diameter. To ensure uniform coating all around the microdrill was positioned centrally and coaxially within the coils of the filament [10], the six-spiral (coil) filament was made with 1.5 mm spacing between the coils.

Fig. 3a shows a SEM micrograph of an uncoated microdrill. The WC-Co cutting edges are welded onto the steel shaft (Fe-Cr). The cutting tip is about 4 mm in length and 0.8 mm in diameter. It can be seen that the microdrill has six cutting edges Fig. 3b. The sharp cutting edges of the tool are clearly visible.

Fig. 4 show the SEM micrographs and the corresponding EDS spectra of the WC-Co microdrill before and after the chemical etching process. Before etching, the EDS spectrum Fig. 4a show the peaks for cobalt (Co), carbon (C) and tungsten (W). It is known that the high cobalt content inhibits diamond deposition

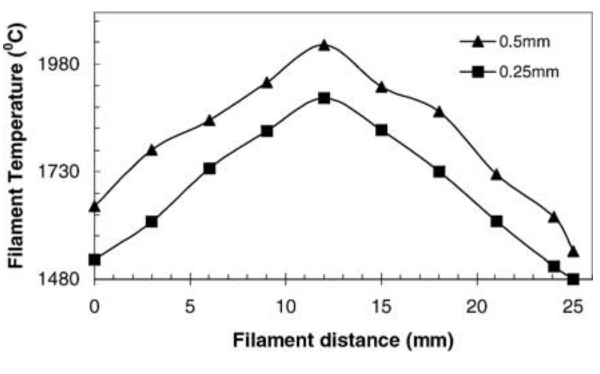
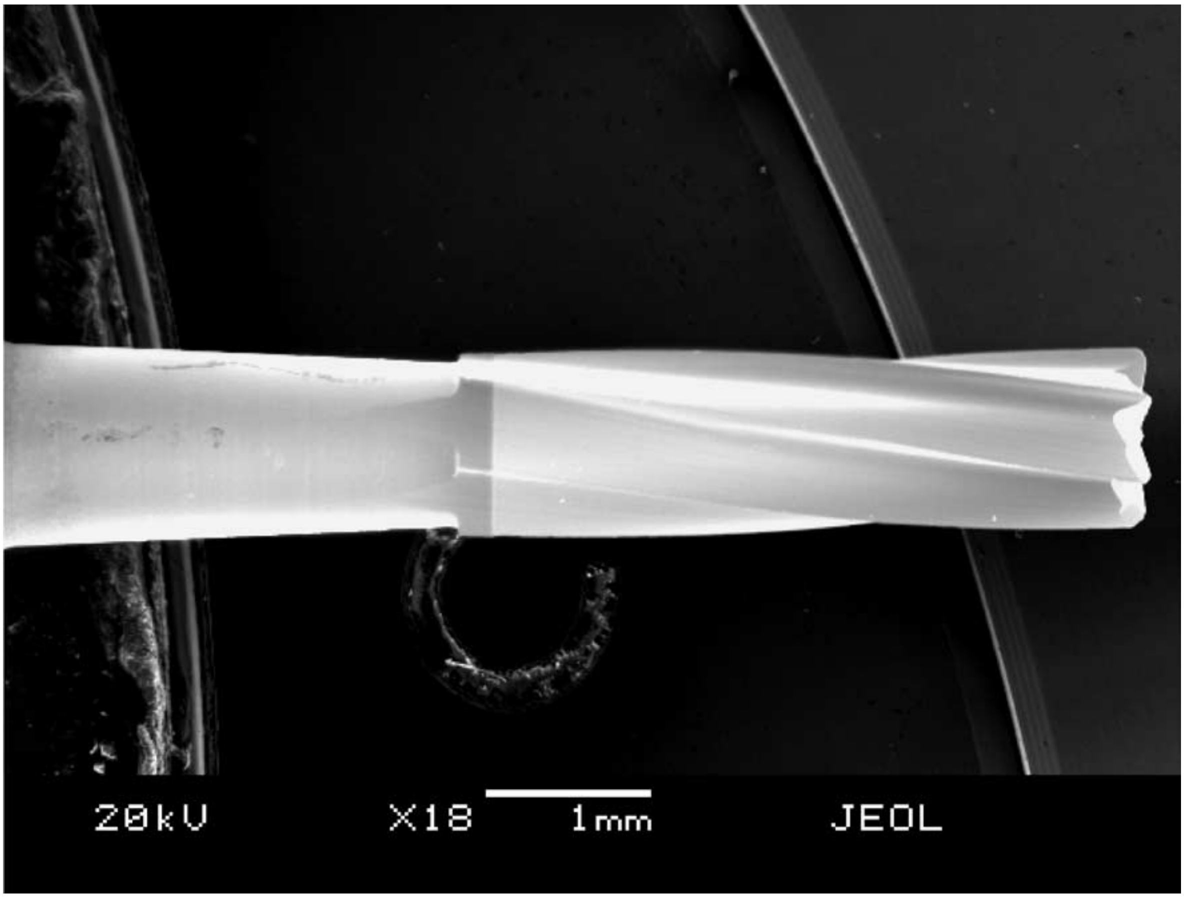
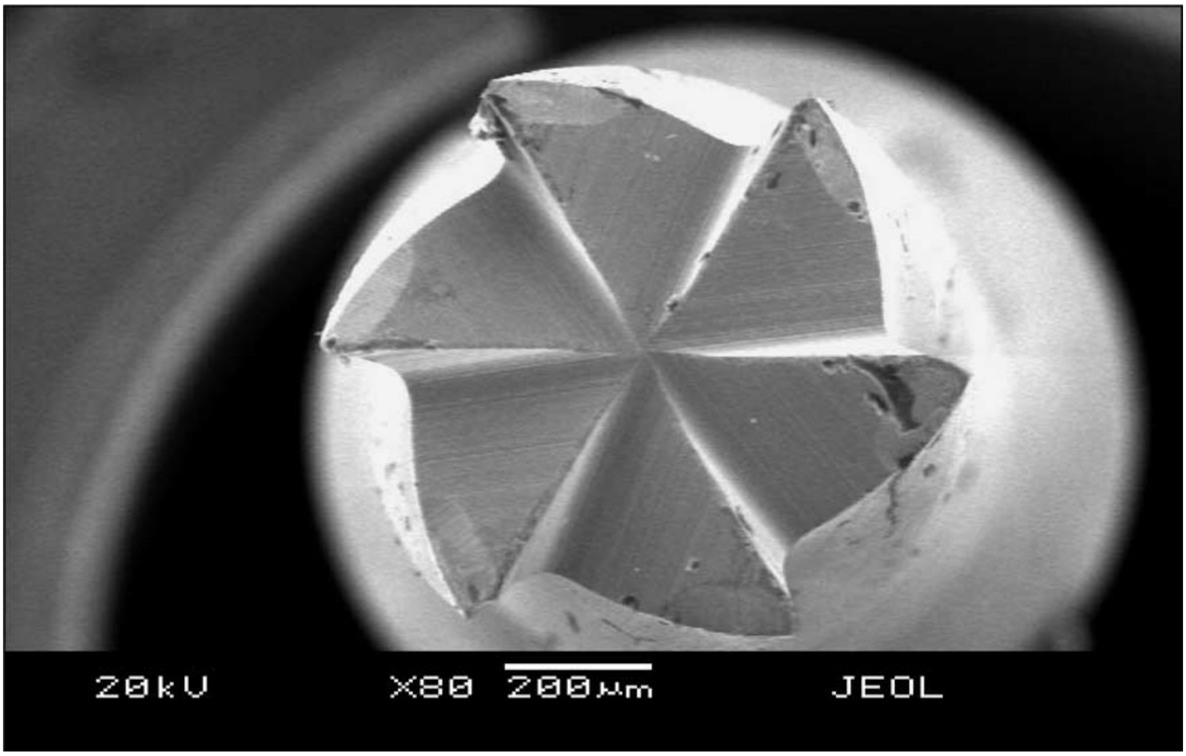


Figure 2 Filament temperatures against filament position for 0.25 and 0.5 mm (tantalum) diameter wires.



(a)



(b)

Figure 3 (a) The tip and cutting edges of microdrill without the deposition of CVD diamond. (b) The cutting edges of the microdrill before deposition with CVD diamond (topical view).

resulting generally in graphitic phases, which degrade the coating adhesion. The Co diffuses to the surface regions preventing effective bonding between the substrate surface and the film coating. To improve the coating adhesion of diamond on WC-Co tools, several approaches can be employed. For example, firstly, the use of interlayer material such as chromium can act as a barrier against cobalt diffusion during diamond CVD. Secondly, the cobalt from the tool surface can be etched using either chemical or plasma methods.

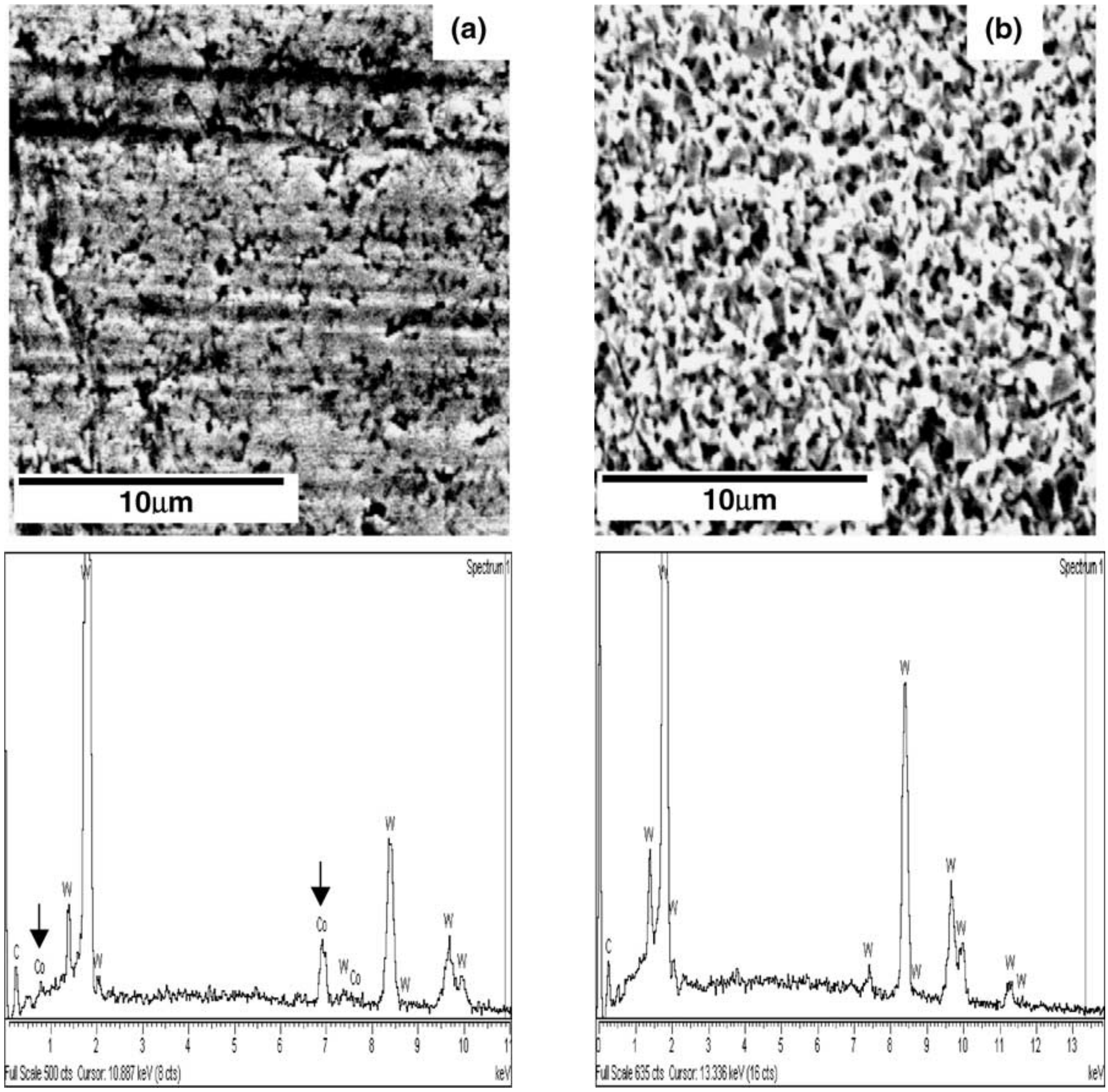


Figure 4 (a) WC-Co microdrill before Murakami and acid etching. (b) WC-Co microdrill after Murakami and acid etching.

Thirdly, the cobalt can be converted into stable intermediate interlayer cobalt compounds. These can act as a barrier to cobalt diffusion from the substrate during film growth [11]. In this study, Murakmi solution followed by H_2SO_4/H_2O_2 etch was used to chemically remove the cobalt from the bur surface. Fig. 4b display the EDS spectrum, which shows that the Co peak has disappeared after etching. This will hopefully prove to be beneficial in enhancing the coating adhesion. In addition, comparison of the SEM micrographs in Fig. 4a and b show that the surface topography is significantly altered after etching in Murakami and H_2SO_4/H_2O_2 solutions. The etching process makes the surface much rougher with a significant amount of etch pits. These pits act as low energy nucleation sites for diamond crystal growth.

Fig. 5 show the SEM micrograph of a diamond coated WC microdrill. It is important to note that all six cutting edges of the microdrill tip were coated with a polycrystalline diamond film with a film thickness of 10 μm grown for 5 hours using the modified vertical HFCVD method. SEM analysis showed that the coating uniformly covered the cutting edges as well as the nearby regions. This is the result of using a vertical filament arrangement and the placement of the microdrill within the coils of the filament. The diamond crystal structure and morphology were found to be uniform and adherent, as shown in Fig. 5a and b. It also shows a close up view of the diamond coated region of the microdrill in Fig. 5c. Typically the crystallite sizes are of the order of 5–8 μm. The visibly adherent diamond coatings on the WC-Co microdrills consisted of (111) faceted diamond crystals. The design of the filament and substrate in the reactor is highly suitable for small micro and nanotools and also offers the possibility of uniformly coating larger diameter cylindrical substrates. For large scale use the filaments can be arranged in parallel with one another and the deposition carried out simultaneously.

Raman analysis was performed in order to evaluate the diamond carbon-phase quality and film stress in the deposited films. The Raman spectrum in Fig. 6 shows a single peak at 1335 cm^{-1} for the tip of the diamond-coated microdrill. The Raman spectrum also

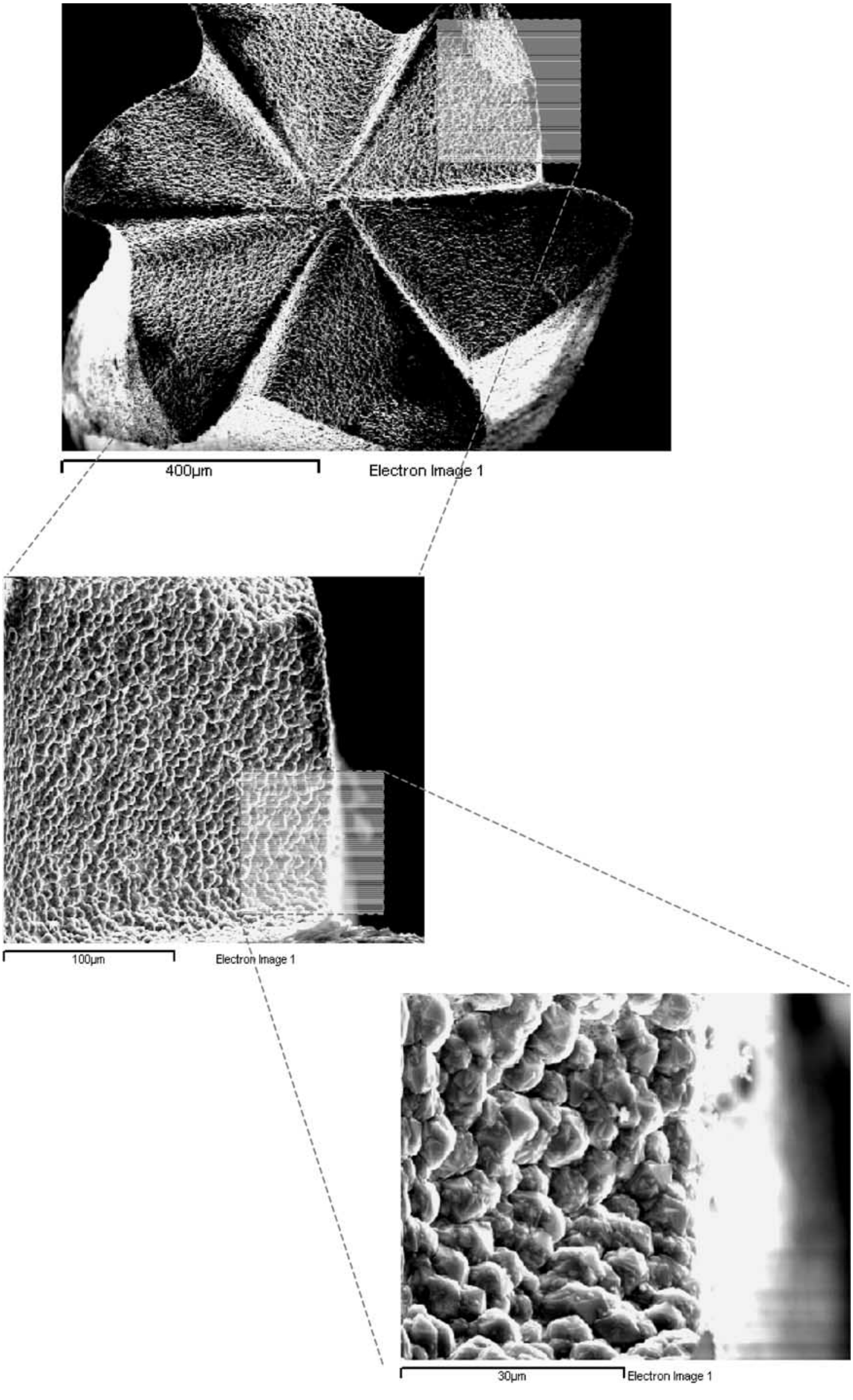


Figure 5 (a) Cutting edge of microdrill after depositing with CVD diamond (topical view) (b) Cutting edge of microdrill uniformly coated with CVD diamond. (c) SEM of microdrill after depositing with CVD diamond. (Close up view).

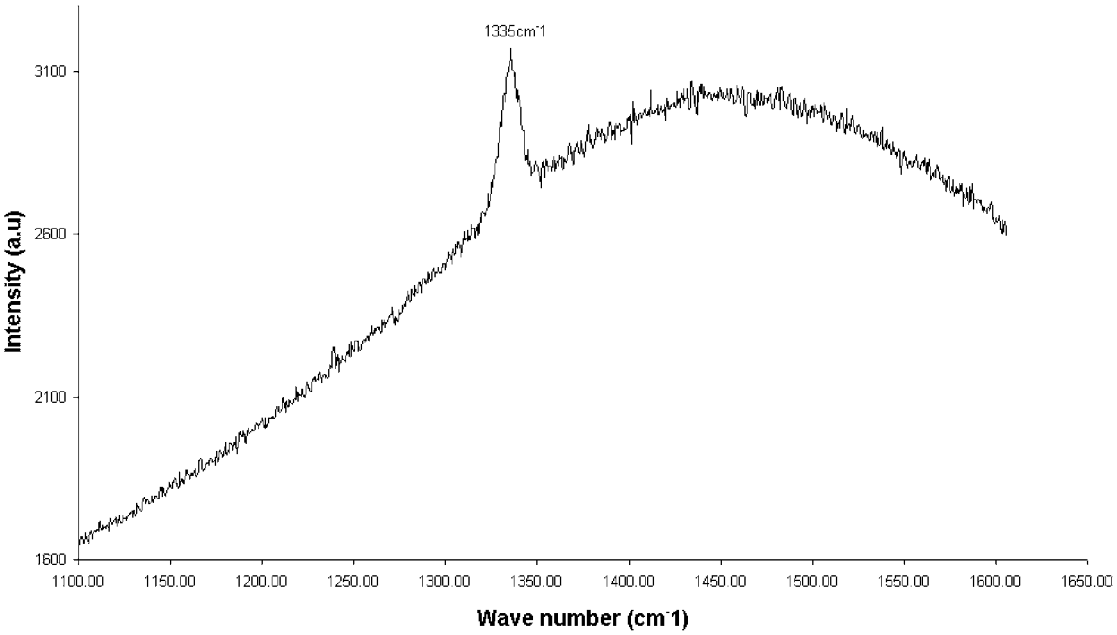


Figure 6 Raman spectra of diamond coated WC-Co microdrill.

gives information about the stress in the diamond coatings. The diamond peak is shifted to a higher wave number of 1335 cm^{-1} than that of natural diamond peak 1332 cm^{-1} indicating that stress, which is compressive in nature, exists in the resultant coatings [12]. The Raman diamond peak position can be used to calculate the film stress. Ager and Drory [13] investigated residual biaxial stress in diamond films grown on titanium alloy by Raman spectroscopy and developed a general model, which describes quantitatively the relations between singlet or doublet phonon scattering and the biaxial stress σ as follows:

$$\sigma = -1.08(\nu_\text{s} - \nu_0)\ (\text{GPa}) \quad \text{for singlet phonon,} \quad (1)$$

$$\sigma = -0.384(\nu_\text{d} - \nu_0)\ (\text{GPa}) \quad \text{for doublet phonon,} \quad (2)$$

Where ($\nu_0 = 1332\ \text{cm}^{-1}$, ν_s is the observed maximum of the singlet in the spectrum and ν_d the maximum of the doublet. In the case when the splitting of the Raman line is not so obvious, the observed peak position ν_m is assumed to be located at the centre between the singlet ν_s and the doublet ν_d, i.e., $\nu_\text{m} = (\nu_\text{s} + \nu_\text{d})$ [16]. From Equation 1 and 2 we obtain

$$\sigma = -0.567(\nu_\text{m} - \nu_0)(\text{GPa}) \quad (3)$$

The stress values can be calculated using Equations 1–3. The coating deposited onto the microdrill exhibited a stress value of −1.7 GPa, in compression. The results of Raman analysis on WC-Co substrate for several different locations on the microdrill have shown 1335 cm^{-1} consistently, indicating largely compressive stress, which is uniformly distributed throughout the cutting tip area. Although, the film was under compressive stress, the coating withstood the stress and refrained from delaminating from the microdrill.

Machining results obtained from a specially constructed machining centre operating at 500,000 rpm using the diamond uncoated and coated tools tested on an aluminium alloy showed a 300% improvement in performance. Uncoated tools drilled an average of 8,000 holes before breakdown and the CVD diamond coated tools drilled an average of 24,000 holes.

4. Conclusion

Diamond films were successfully deposited onto chemically etched cemented WC-Co microdrills using a modified HFCVD process. Pre acid-etching of the microdrills essentially removed the cobalt binder from the drill surface. The modified HFCVD process proved to be a successful method of uniformily coating cemented WC-Co microdrills with polycrystalline diamond films. Raman spectroscopy showed that then as-grown films were subject to compressive stress. However, the diamond films accommodated the stress and refrained from delaminating from the microdrills.

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