EFFECT OF PRESSURE ON MORPHOLOGY AND ADHESION OF HFCVD DIAMOND COATING ON CEMENTED CARBIDE INSERTS

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Abstract: In the present investigation, effect of chamber pressure on morphology, quality and adhesion of hot filament chemical vapour deposition diamond coating on cemented carbide cutting inserts were studied. Flow rate of methane and hydrogen were kept at 0.5 and 100 SCCM. The tungsten filament temperature was kept at 2100°C, while substrate at 700°C. The chamber pressure was set at 1, 2, 5, 10, 20 and 30 Torr. Deposition was continued for 8 hours. Pressure of 5 and 10 Torr gave good crystal habits with (111) plane on the cutting edge. While deposition at chamber pressure of 20 and 30 Torr gave crystal habits of cubo octahedral nature. In the case of deposition at 20 and 30 Torr, coating-substrate adhesion was good enough for machining performances. The crystal planes and purity (sp³/sp² content) of the coatings at different pressures were evaluated by XRD and Raman spectroscopy respectively before going for actual mechanical characterizations. CVD diamond coated inserts were indented under Rockwell C scale to assess the coating-substrate adhesion under three loads of 30, 60 and 100 kgf respectively. The adhesion between the coating and the substrate were observed under SEM. These results suggest that deposition at high pressure was more favourable in terms of adhesion, than that at low pressure.

Keywords: CVD diamond, Pressure, Morphology, Adhesion, Machining

1. INTRODUCTION

The application of CVD diamond coating on tungsten carbide inserts has already drawn much attention for use as advanced cutting tool. However, the understanding of the correlation between deposition parameters and the ultimate properties of the diamond film is of particular importance.

One of the most important parameters in CVD diamond process is the system pressure. However, only few investigations have been reported about the effect of pressure on CVD diamond film. Also most of the work done so far used silicon or molybdenum as the substrate. It was possible to have efficient nucleation of diamond following HFCVD route at a system pressure as low as 5 Torr on silicon substrates without any pre-seeding with diamond particles. However, it is reported that diamond film deposited at a low pressure contained more non-diamond components than deposited at high pressure [1, 2, 3]. It has also been shown that in microwave plasma enhanced CVD of diamond, low pressures always decreased the film quality [4]. In HFCVD of diamond at higher system pressure, (100) have been favourable growth surfaces, whereas (111) growth habits is favoured at low pressure [5]. In HFCVD of diamond at 1100°C substrate temperature with the increase of system pressure from 40 Torr to 300 Torr, the structure of the film gradually changed from microcrystalline to nanocrystalline and also non-diamond phases increased [6]. Investigation on HFCVD of diamond film revealed that diamond growth rate did not depend on pressure or
mass flow rate only. It was suggested that residence time could also control the growth rate substantially [7, 8]. Another investigation on HFCVD of diamond on cemented carbide substrates revealed that among three pressure ranges 3, 20 and 50 mbar, secondary nucleation is predominant at 3 and 50 mbar compared to 20 mbar [9]. Most of the investigation on system pressure has been done using silicon as the substrate [10].

In the present work, an attempt has been made to study the influence of system pressure on nucleation, growth rate, morphology, quality of the HFCVD diamond coating and finally its adhesion to the cemented carbide substrates.

2. EXPERIMENTAL PROCEDURES AND CONDITIONS

Cemented carbide turning inserts of geometry SPUN 120308, ISO K10 grade containing nominal 6wt.% of cobalt were used as the substrate for CVD diamond deposition. Prior to deposition, the samples were cleaned with trichloroethylene and acetone to remove contaminants from the surface. Samples were etched with HCl+HNO₃+H₂O (1:1:1) solution and K₃[Fe(CN)₆]+KOH+H₂O (1:1:10) solution for 15 minutes each at room temperature under ultrasonic vibration to remove cobalt and WC phases respectively. Then the inserts were seeded with diamond powder (0-2 μm) ultrasonically, so that each diamond seed will enter the voids and will act as nucleating site during deposition. The schematic diagram of the CVD diamond set-up is shown in Fig.1.

Table 1 shows deposition parameters on the carbide inserts at different reaction pressure of the CVD chamber. Reaction pressure plays a big role in nucleation of the crystals and then growth of diamond. The flow rate of hydrogen and methane were kept at 100 and 0.5 SCCM respectively by Mass flow controllers of MKS make. The purity level for hydrogen and methane were 99.995% and 99.9995% respectively. The pressure of the CVD chamber was regulated by MKS Baratron pressure sensors (range-1-100 Torr). The surface roughnesses of the inserts were measured by Talysurf (Talyor-Hobson Surfomatic 3P, diamond stylus tip radius 5 μm). Ra, Rz and Rmax. were measured with a computer interface in μm.

<table>
<thead>
<tr>
<th>Substrate</th>
<th>WC-6wt% ISO K10 Sandvik cemented carbide insert</th>
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</thead>
<tbody>
<tr>
<td>Filament</td>
<td>Tungsten wire(Ø0.25mm) carburised</td>
</tr>
<tr>
<td>Filament temperature</td>
<td>2000-2100°C</td>
</tr>
<tr>
<td>Filament to substrate distance</td>
<td>5-6 mm</td>
</tr>
<tr>
<td>Substrate temperature</td>
<td>700°C</td>
</tr>
<tr>
<td>Gas composition</td>
<td>0.5% CH₄ in H₂</td>
</tr>
<tr>
<td>Chamber pressure (Torr)</td>
<td>1, 2, 5, 10, 20, 30</td>
</tr>
<tr>
<td>Deposition time</td>
<td>15, 30, 480 min.</td>
</tr>
</tbody>
</table>

The crystal morphology were characterized by SEM (model no: JEOL 5800). The crystal planes of carbides and diamond were measured by X-ray diffraction by Philips 1710 machine using nickel-filtered Cu Kα (λ=1.54178Å) produced by 40kV/20 mA). The purity of the diamond coatings was analyzed by micro-raman spectroscopy (model: RENISHAW 20 MW Argon laser, wavelength-514.5nm, beam diameter-1.5μm). The mechanical characterization was done by indentation test under 30, 60 and 100kgf load in Rockwell hardness tester.

3. RESULTS AND DISSUSSIONS

Fig.2 shows the untreated, etched with HCl+HNO₃+H₂O (1:1:1) solution, etched with K₃[Fe(CN)₆]+KOH+H₂O (1:1:10) solution and diamond seeded (0-2 μm) tungsten carbide substrates. The micro carbide grains were clearly visible after the surface treatments. Theoretically it is known that at very low pressure, mean free path is very large. So nucleation density also decreases. Fig.3 shows the surface roughness and Co% of the treated samples. Murakami treated samples have higher roughness since the WC grains were etched and the surface cobalt content was increased to 15%.

Fig.4 shows the diamond morphology after depositions for 8 hours at various pressures. At pressure of 1 Torr, no distinct crystals are observed. At pressure of 2 Torr some small crystals are observed. However at 5 Torr, the morphology shows well faceted diamond crystals of considerable size. Again the morphology tends to get poorer at higher pressure of 30 Torr. At very...
low pressure the species for diamond nucleation is very low. Hence the crystals are not significant in availability and size at 1 and 2 Torr respectively. However at pressure of 5 Torr the mean free path for radical adsorption on substrate surface is favourable for good nucleation. This was evidenced in the diamond morphology observed in Fig.4(c). Again the diamond crystals tend to get slightly smaller in nature at a relatively higher pressure of 20 and 30 Torr. This is due to excess availability of more methyl radicals on the substrate surface. Different peaks of WC planes and diamond were shown in Fig.5 with an uncoated and a coated sample. The surface roughness of diamond-coated samples were shown in Fig.6.

Fig.2: (a) Untreated Co = 6% wt (b) Treat 1 Co = 0.45% wt (c) Treat 2 Co = 15.0% wt (d) Treated with diamond seeds

Fig.3: Surface roughness and Co% of treated tungsten carbide substrates.

Fig.4: Diamond deposition for 8 hours (a) 1 Torr (b) 2 Torr (c) 5 Torr (d) 10 Torr (e) 20 Torr (f) 30 Torr

Fig.5: XRD study showing relative peak intensities for uncoated and diamond coated substrates.

Fig.6: Surface roughness of seeded carbide and diamond coated at different pressure ranges. At low pressure deposition, crystals were in spherical form and lead to low roughness. While at high-pressure deposition, low surface roughness was obtained due to smaller crystals with clear crystal habits. But the purity of diamond coating at different chamber pressures was determined by micro-raman spectroscopy. It has been observed that at very low pressure such as at 1, 2 Torr pressure, crystals are discrete, spherical and mostly amorphous carbon was present. But at 5 Torr pressure there was no humps in the coating and mostly sp3 phases were present. This was valid also for 10 Torr pressure but with slight humps. But with increasing more pressure such as at 20 and 30 Torr the amorphous carbon (G peaks-1582 cm\(^{-1}\)) phases increase due to high collision of gases at the surface and with slight change in crystal habit [11]. This broader peak observed at 1553 cm\(^{-1}\) can be correlated to the existence of amorphous carbon (a-C) in the grain boundaries of diamond crystals. This was an
The crack diameters of the indentation and the peeling of the coating are shown in Fig.8. This was an indication of the premature failure of the coating revealed by SEM. While at high pressure coating fracture has been minimized and there were grooves on the insert. These results give a clear indication that high-pressure depositions were favourable for better anchorage at the carbide interface and lead to better machinability of non-ferrous materials [12].

4. CONCLUSION

(i) The present study reveals the clear variation in morphology of diamond coating with chamber pressure. At a pressure of 1 Torr, the size of the crystal was rather small which grew to a larger size at chamber pressure of 2 Torr. However well developed crystals with (111) growth habits could be easily detected at a pressure of 5 Torr. Roughness of the coating was also substantially high at this condition. Coating deposited at 10 Torr also exhibited good crystal habits but with smaller size. With increase of pressure to 20 and 30 Torr, the crystal habits changed from octahedral (111) to cubo-octahedral nature. The deposition at high pressure resulted in more number of secondary nucleation. The coating obtained at 20 and 30 Torr are less rougher than that obtained at 5 Torr.

(ii) Coating deposited at 5 Torr hardly shows any presence of non-diamond phase. The quality of coating at 10 Torr was also as good as that obtained at 5 Torr. Traces of non-diamond phases could be detected at 20 Torr. The non-diamond phases increased as the pressure increased further to 30 Torr.

(iii) In terms of adhesion, the coating deposited at 20 and 30 Torr exhibited best result followed by that deposited at 10 Torr. The adhesion was found to be lowest with the coating deposited at 5 Torr.

(iv) Considering the morphology, roughness and adhesion characteristics, it appears that coating deposited at 20 Torr will be most favourable for cutting tool application.
REFERENCES


