Adhesion Improvement of CVD Diamond Coatings on WC-Co Substrates for Machining Applications

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ABSTRACT

In order to improve the performance of WC-Co cutting tools, high quality microcrystalline diamond coatings were produced using microwave plasma enhanced chemical vapor deposition (MPECVD) method. The adhesion of the diamond film deposited on the substrate has been considered to play an important role in the performance of the cutting tools in machining applications. A thin layer of Cr was coated on the WC-Co substrate before the diamond deposition; 75 µm diamond powders were sandblasted on the surface at 40 Psi to increase the nucleation density. Diamond film has been successfully deposited on the substrate at temperature around 750ºC with 1.5 % CH_4 in Hydrogen plasma. Scanning electron microscopy (SEM) has been used to study the surface morphology and Raman spectroscopy has been performed to characterize the quality of the diamond films and measure the residual stress. The adhesion of the diamond film has been evaluated by Rockwell indentation test. The results indicated that film grown on the Cr interlayer with diamond powder sandblasting has much better adhesion strength.

INTRODUCTION

Diamond Coating is an industrial solution to improve the surface properties in a wide variety of wear resistant applications because of its outstanding properties. Along with great wear resistance, the advantages of diamond coating include high surface hardness, high thermal conductivity, reduced friction, better corrosion protection and improved optical properties.

Cemented carbide is a material made by "cementing" very hard tungsten monocarbide (WC) grains in a binder matrix of tough cobalt metal by liquid phase sintering. The cemented tungsten carbide cutting tools are widely used all over the word in automotive industry, metal machining, mining and stone cutting industry. However, without a hard coating such as diamond, the cemented WC tools are found to wear rapidly when machining some particular materials such as green ceramics, abrasive composites or high silicon-filled aluminum. Successful development of diamond-coated tools will significantly benefit the performance of WC-Co cutting tools. As a result, diamond coated tools will have reduced down time, better cutting performance and less defective machined surface.

However, the poor adhesion of the diamond film on the substrate is the main cause of tool failures and becomes the main technical barriers for commercialization of diamond-coated tools [1-4]. The reason behind is that the deposition of diamond films on cemented carbides is strongly hindered by catalytic effect of cobalt. During the deposition, the Co leaching from the WC-Co substrate favors the formation of sp^2 carbon (non-diamond) phase instead of the sp^3 carbon [5]; this is considered the result why diamond films have poor adhesion to the WC-Co substrates. Various approaches have been reported to avoid or reduce the catalytic effect of cobalt on the growth of diamond films [6-10]. In our study, a thin Cr interlayer has been implanted between diamond film and substrate. Sandblasting with diamond powder on the Cr coated substrate was employed to improve the adhesion strength before the deposition.

EXPERIMENTAL

Cemented carbide coupon samples (Co 6%) were used as the substrates for diamond film deposition. In order to prevent the catalyst effect of Co, several surface pretreatments have been done prior to the deposition of diamond coating. Substrate surface was first cleaned using acetone and methanol, then a thin Cr layer (~5 μm) was coated on the WC-Co by unbalanced magnetron sputtering system. After that, the substrates were sandblasted by 75 μm diamond powder at pressure 40 Psi. During diamond powder sandblasting, half of the coupon sample was masked to compare the coating quality and morphology. This sandblasting procedure is believed to help the initial nucleation to get better adhesion strength. This treated substrate was then seeded by methanol solution containing 1μm diamond powder in an ultrasonic bath for 5 hours to further nucleate the surface. Diamond film deposition was carried out in the MPECVD system. Hydrogen and methane were used under the following conditions: microwave power: 800 W; substrate temperature, 750 °C; pressure, 80 Torr; gas composition, H_2:CH_4 (mass flow rate: 500/7.5 sccm); deposition time, 6 h. The surface morphology of diamond film was examined by scanning electron microscopy (SEM). The residual stress of the diamond film was estimated by Raman peak shift and curvature method. Rockwell indentation test was used to evaluate the adhesion of the diamond film. Pin-on-disk test was performed to investigate the wear resistance of the coating.

RESULTS AND DISCUSSION

Effect of Cr interlayer and diamond sandblasting

Cemented carbide substrate, polished to a mirror finish, was coated with a thin Cr interlayer (~5 μm) using sputtering system and sequentially coated with diamond film (~8 μm) at temperature around 750 °C. Figure 1 shows the SEM image of microcrystalline diamond films grown on bare WC-Co substrate. The crack of the film was observed right after the cooling down due to the catalyst effect of Co and the mismatch of coefficient of thermal expansion (CTE).

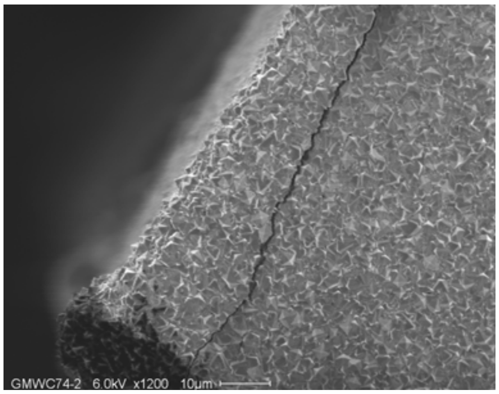
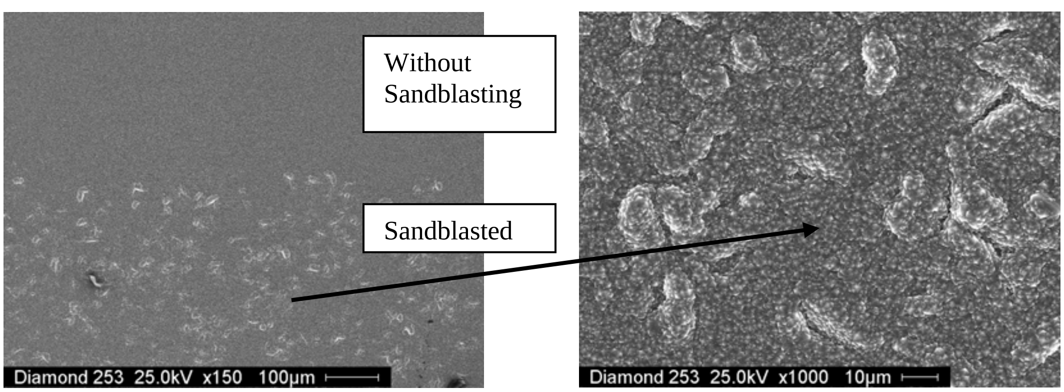


Figure 1. SEM image of diamond coating on bare WC-Co substrate.

Figure 2a shows the SEM image of diamond film on a Cr coated WC-Co substrate with two regions, the top part was masked during sandblasting and the bottom part was blasted by the diamond powder. Both samples have much better adhered coatings than the bare WC-Co substrate. The 5 µm Cr layer is good enough to inhibit the catalyst effect of Co and improves the adhesion strngth. Figure 2b shows the high resolution image of the coating in the blasted region. Much more diamond crystals grew in the blasted region and small diamond particles formed together like cauliflower in some places. This indicates that sandblasted surface has much higher nucleation rate. If diamond nucleation is low, the individual diamond crystals can increase in size before they grow together and form the layer. Voids between the crystals are formed at the interface, which makes the interface weaker. In the case of high diamond nucleation rate, the voids between the crystals remain much smaller and thus improve the adhesion. The adhesion strength of these two regions was evaluated by Rockwell indentation test.

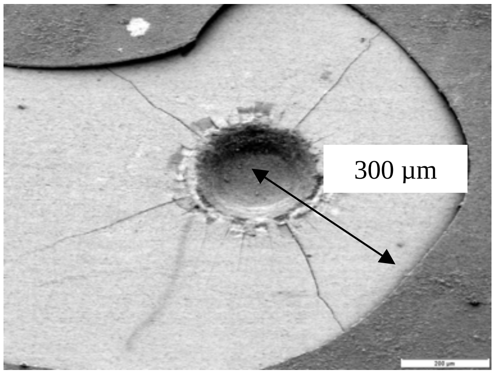
Adhesion evaluation and residual stress measurement

Coating adhesion can be investigated qualitatively using Rockwell indent method [11, 12]. The diamond-coated surface was indented by a Rockwell indenter with a force of 150 kgf. Figure 3a and 3b show the SEM images of indentation area of two samples, with sandblasting and without sandblasting, respectively. Figure 3a shows the indentation area of the sample only with the Cr layer interlayer. Diamond delamination is observed and the average radius of the delamination area is around 300 µm. Figure 3b shows the SEM image of the indentation test done on the surface sandblasted with diamond powder. It is observed that the average delamination radius reduced to 180 µm. and the diamond film adhereed very well with the Cr interlayer along the edge where the coating delaminated. In the first sample, on the contrary, the diamond film peeled off along the edge, indicating that film deposited on the blasted WC-Co substrate has much stronger adhesion strength.

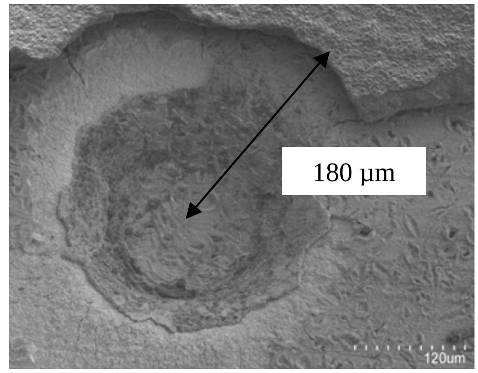


(a) (b)

Figure 2. (a) SEM images of diamond coating on Cr coated WC-Co substrate with (bottom part) or without (top part) diamond powder sandblasting. (b) High resolution SEM image of diamond coating on the sandblasted region.



(a)



(b)

Figure 3. Effect of sandblasting with diamond powder on Cr coated WC-Co substrate. (a) delamination area without sandblasting; (b) delamination area with sandblasting.

The diamond coating delamination around the Rockwell indent indicates high level of residual stress in the coating. Residual stress of the coating could be measured by Raman shift. The Raman shift on diamond peak at 1332 cm^{-1} is proportional to the film stress according to Ralchenko et al. [13]. From Figure 4, we observed that the diamond peak 1337 cm^{-1} has 5 cm^{-1} shift, which is correlated to compression stress around 2.5 GPa. The residual stress has also been estimated by curvature method. Measured by WYKO optical profilometer, the curvature of the as deposited sample is about 11.5 µm. The curvature of the sliced sample, thickness is 250 µm, is about -0.43m. Using stoney's equation we estimated that the residual compression stress of the diamond coating is around 1.9 GPa.

Pin-on-disk test

The diamond coated sample on blasted WC-Co/Cr substrate was also tested on a pin-on-disk machine. This pin-on-disk machine was used to test the wear resistance of the coating. The sample was fixed on the machine and rotated continuously. An aluminum pin rode on a diamond-coated surface is used to test the wear resistance of the sample. Figure 5 shows the wear track covered by aluminum after the test.

Aluminum was observed filling the valleys between the diamond grain peaks (Figure 5a). Small chips and chunks of aluminum stuck between the diamond grains. After more than 100,000 rotations of pin-on-disk machine there was no wear of the diamond coating. The smallest wear that could have been detected in our SEM observations was less than 20 to 30 nanometers. However, no wear was observed, and the diamond grains remained very sharp after the test (Figure 5b). This suggests that the diamond coating on a machining tool is likely to last for a long time.

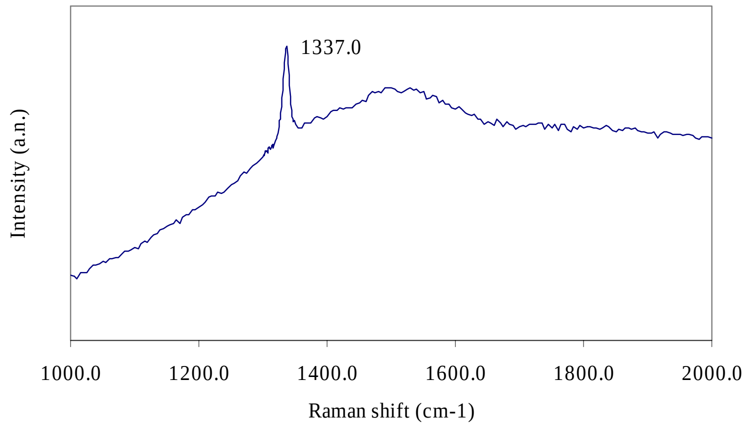
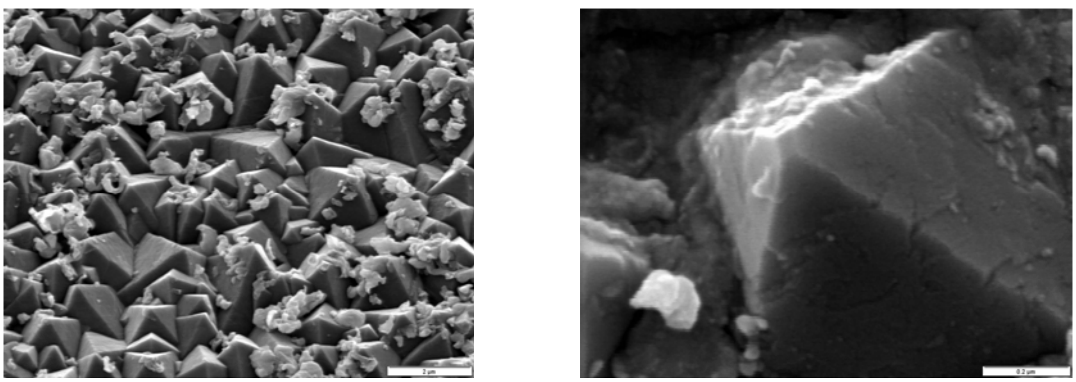


Figure 4. Raman spectrum of diamond film on blasted WC-Co/Cr substrate.



(a) (b)

Figure 5. SEM micrographs of (a) diamond coating on cemented carbide substrate with a wear track; (b) diamond grain in the wear track after 100,000 rotations against an aluminum pin.

CONCLUSIONS

In our study, microcrystalline diamond films were deposited on WC-Co cemented carbide substrates using Cr interlayer to improve the adhesion strength. A thin Cr layer around 5 µm was coated on the substrate before diamond deposition, this layer were used as the diffusion barrier layer to inhibit the Co migration during CVD process. Continuous diamond film has been grown on the substrate and adheres well after the deposition. Sandblasting with 75 µm diamond powder has been performed to improve the adhesion and Rockwell indentation test shows that film grown on sandblasted substrate has much less delamination area and stronger adhesion strength. The diamond coating performs very well during the pin-on-disk test and no wear was observed. The residual stress was calculated based on the diamond peak shift by Raman spectroscopy measurement and curvature method. Compression stress around 2 GPa was obtained.

ACKNOWLEDGEMENTS

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