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EFFECT OF PRETREATMENT METHODS, CHAMBER PRESSURE AND SUBSTRATE TEMPERATURE ON MORPHOLOGY, QUALITY, ADHESION AND CUTTING PERFORMANCE OF HFCVD DIAMOND COATED TOOLS IN MACHINING ALUMINIUM ON CEMENTED CARBIDE INSERTS

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ABSTRACT

The wide applications of aluminium in different industries have increased the necessity for researching on suitable cutting tool. It is observed that, comparing to ferrous materials; dry machining of aluminium, exhibits a greater challenge in view of heavy material built-up at the cutting edge. This not only enhances cutting force but also causes poor quality surface finish on the workpiece.

This paper thoroughly investigated the effect of substrate treatments, micro structure, adhesive toughness and cutting force of Hot Filament Chemical Vapour Deposition (HFCVD) diamond coating on uncoated carbide cutting inserts (94%WC+6%Co). To restrict the formation of non-diamond carbon phases, such as graphite, and to improve the adhesion between diamond and WC substrates, etching with a combination of diluted HCL and HNO_3 are used as substrate pretreatments. The pressure in the CVD chamber are set at 0.666, 1.333, 2.666 and 3.999 kPa and the substrate temperatures are kept at 650, 700, 750 ^{0}C while maintaining the filament temperature at 2100 ^{0}C. Characterization and purity (sp^3 / sp^2) of the obtained coatings are duly evaluated by Scanning Electron Microscope (SEM) and Raman Spectroscopy. The mechanical characterization of the coating is investigated by Rockwell indentation test under loads of 294 N, 588 N and 980 N to assess the coating-substrate adhesion.

It is observed that the deposition pressure at 2.666 and 3.999 kPa and substrate temperature at 700 ^{0}C, coating with (111) habits is mostly preferred to achieve maximum coating substrate adhesion. These results suggest that during dry machining, compare to uncoated tool, a 4-5 µm HFCVD diamond coating exhibits remarkable inertness towards aluminium leading to substantial reduction of cutting force and improvement of work-piece surface finish.

Keywords: HFCVD diamond, Coating, Morphology, Adhesion, Machining

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1. INTRODUCTION

In the field of innovative cutting tool mechanism, the use of Chemical Vapour Deposition (CVD) diamond coating on tungsten carbide inserts is succeeded to attract more attention in world over. But more understanding is needed to analyze the relation between deposition parameters and the vital properties of diamond film [1-2].

In recent years, with the development of different coated tools, the efficiency of cutting processes has been improved. Diamond coating has proved its superiority in the case of non-ferrous metal, such as aluminium and magnesium. It results in good surface finish, lesser edge formation, chemical inertness and high wear resistance [3-6]. Effort has been made to improvise the adhesion of diamond coatings in substrate materials and further to optimize the interface strength of tool performance; chemical and mechanical substrate pretreatments are being done [7-8].

Cemented tungsten carbide (WC-Co) is considered as one of the most appropriate substrate material in the production of diamond coated tools. Though cobalt acts as a binder which provides additional toughness to tool, but it becomes antagonistic to diamond adhesion [9-11]. It is understood from the phase diagram of Co-C system that under typical diamond CVD temperatures (700-1000^{0}C), carbon is soluble in Co in the range of 0.2-0.3 wt % C [12]. In the preliminary stage of CVD process, WC-Co is succumbed to a radical rich atmosphere of hydrocarbon and carbon species start to diffuse into the greater part of the binder stage till the solubility of carbon exceeds the partially filled 3d shell transition metal, cobalt. The Co acts like a catalytic agent in the formation of graphite [13]. This restricts the deposition of adherent diamond coatings onto untreated WC-Co tools. Therefore, it is necessary to remove cobalt from the surface of the tools. Several literatures [14-17] have established the reports that the early formation of sp^2-carbon layer at the substrate surface is detrimental to the growth of diamond in the later stage.

The growth of polycrystalline diamond coatings on several substrates using CVD technique are presented in different research papers [18-20]. Polycrystalline diamond is further categorized into micro, nano and ultra-nano crystalline depending upon the average grain size. Similarly the properties of different coatings are to vary with degrees similar to single crystal diamond [18-21].

Generally, diamond coatings on cutting tools are employed to increase their performances. The increase of performance for the conventional (macroscopic) size cutting tools using these coatings are necessitated for the different types of reasons which are listed below:

- Extremely high hardness (80-100GPa) which helps reduce tools wear [18-23].
- Low coefficients of friction against various work-piece materials, which decreases cutting and thrust forces and also reduced heat production [24].
- Chemical inertness and low adhesion to must work-piece materials, which prevents built-up-edge (the build-up of work-piece material on the tool rake face), dead metal zones and clogging of flutes [25].

2. EXPERIMENTAL PROCEDURES AND CONDITIONS

The substrates used for CVD diamond deposition were cemented carbide turning inserts of geometry SPUN120308, ISO K grade containing 6wt% cobalt. Prior to deposition the samples were cleaned with trichloroethylene and acetone followed by isopropyl alcohol to remove contaminates from the surface. Samples were etched with $HCl+HNO_3+H_2O$ (1:1:1) for 15 minutes ultrasonically at room temperature to change the chemical composition and to roughen the surface. Then the inserts were seeded with diamond powder (0.2-1 µm) by ultrasonic agitation for two minutes in solvent 2-propanal, so that the seeds would enter the voids and act as nucleating site during deposition. The schematic diagram of the CVD chamber is shown in Fig.1.

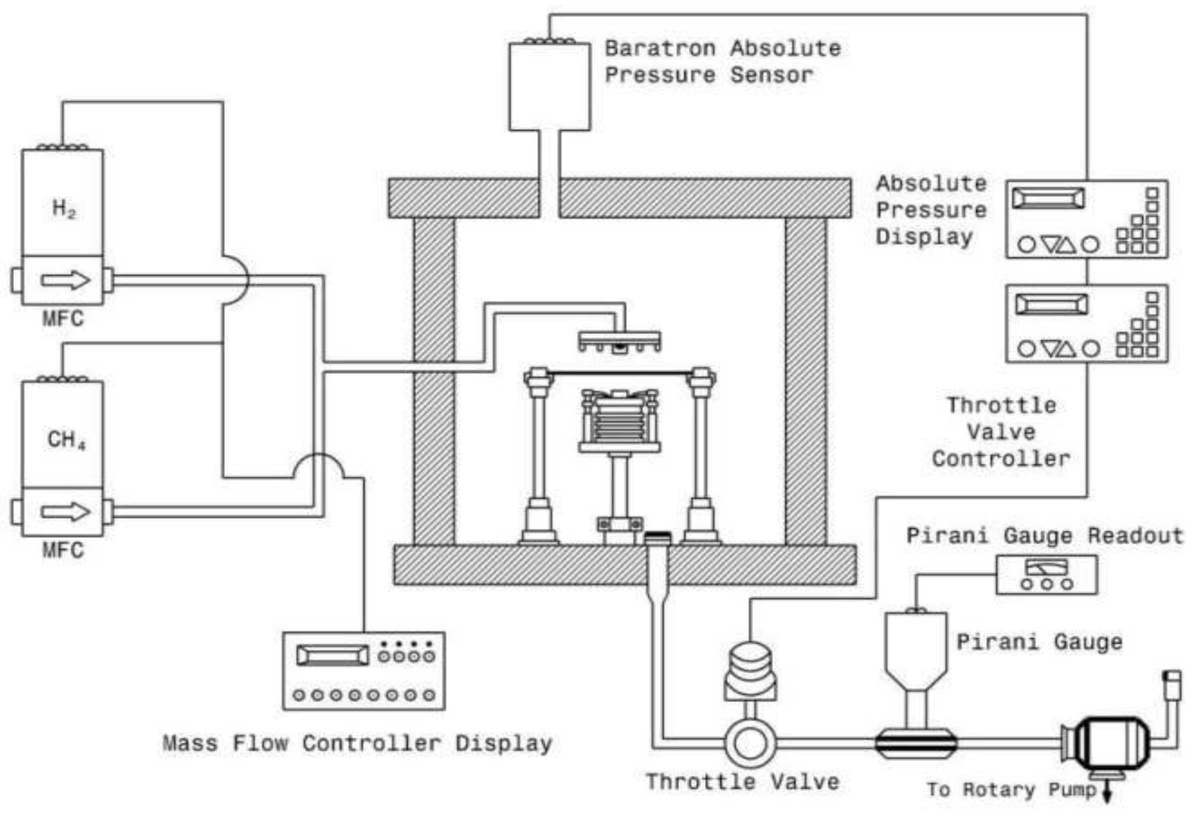


Figure 1 Schematic view of the CVD chamber

Table 1 Deposition parameters

Substrate (Sandvik cemented carbide inserts)	WC-6wt% Co ISO K10
Filament	Tungsten wire (ϕ0.25 mm) carburized
Filament temperature	2000-2200 ^{0}C
Substrate temperature	650, 700, 750 ^{0}C
Filament to substrate distance	5 - 6 mm
Chamber pressure	0.666, 1.333, 2.666 and 3.999 kPa
Gas consumption	0.5% CH_4 in H_2
Deposition time	480 min.

Table 1 show all the process parameters and conditions of CVD diamond on the cemented carbide inserts at different pressure and temperature in the CVD chamber. Reaction pressure and temperature play a major role in nucleation and growth of each diamond crystal on carbide substrate. The flow rate of hydrogen (99.995% pure grades) and methane (99.95% pure grades) were maintained constant at 100 and 0.5 SCCM respectively by mass flow controllers (MFC) of MKS make. The pressure in the CVD chamber was controlled by MKS Baratron pressure sensor of (0.1333-13.332 kPa). The surface roughness of the carbide substrates was measured by 3P-surtonic Taylor Hobson instrument. The roughness parameters R_a, R_z and R_{max} were measured on tungsten carbide substrates at different stages with computer interface in µm.

The crystal morphology and the content of cobalt / tungsten were characterized by SEM (model no: JEOL 5800 OXFORD ISIS 300) with an attachment of EDAX (Energy Dispersive Analysis of X-ray: Detector-LiSi crystal, Element analysis above atomic number 10, OXFORD-ISIS 300). The purity, orientation and individual defects states were evaluated by Micro-Raman Spectroscopy (Renishaw Laser Argon ion, Power: 8mW, Wave length: 5140Å, beam diameter 1.6µm). The mechanical characterization of the coating was studied by Rockwell indentation test under loads of 294 N, 588 N and 980 N. The indented samples were seen under optical microscope (Olympus Z4045ST) for primary study and finally these samples were studied under SEM for nature of failure. The interface strength / lateral crack radius was calculated by measuring cracked area at a particular load. A comparison has been made among various chamber pressure, substrate temperature, indentation load and crack indentation diameter.

Before machining test, indentation tests confirmed the nature of interface adhesion by changing various process parameters. The machining performances of the uncoated carbide tool and diamond coated tools were carried out at cutting speed of 365m / min., feed 0.1 mm / rev. and depth of cut 0.5mm in a combination turret lathe. The tool was held in a standard Sandvik tool holder mounted on a 3-D piezoelectric dynamometer (KISTLER 9257B Switzerland) for measurement of axial (P_x) and tangential forces (P_z). The signals were amplified by charge amplifiers (KISTLER 5070A, Switzerland) with the help of data acquisition card, NI-9205 and LabView software 8.6.1(USA). They were also used for recording both static and dynamic characteristics of the cutting force components during machining. Comparison has been made between axial force, tangential force,surface roughness and coating fracture.

3. RESULTS AND DISCUSSION

3.1. On Substrate Treatment

The SEM micrographs of the as received uncoated tool show fine grinding marks in Fig. 2. Cutting edge shown in SEM micrograph does not seem to very sharp; rather it shows traces of fracture. Few attempts have been made in past [26] to use mixture of HNO_3+HCl+H_2O (1:1:1) as the etching medium for efficient removal of surface Co from the carbide substrate and enhancement of roughness of the etched surface. The present investigation quantitatively established that carbide substrate treated with solution of HNO_3+HCl+H_2O (1:1:1) (Treat 1) could bring down Co as low as low as 0.5% and surface roughness has gone up. These features are clearly shown in Fig. 3. SEM micrograph of the etched surface in Fig. 2 clearly indicates that not only grinding marks have been completely removed but also surface carbide grains were clearly exposed as a result of strong etching. In Fig.4 EDAX results established the percentage of surface cobalt and WC content.

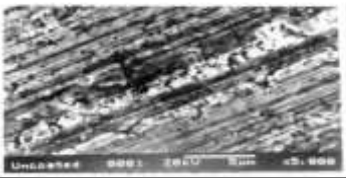
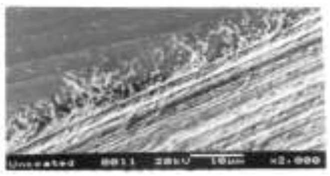
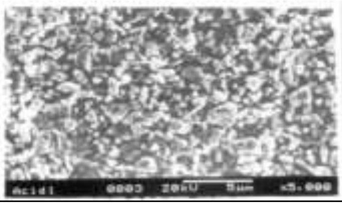
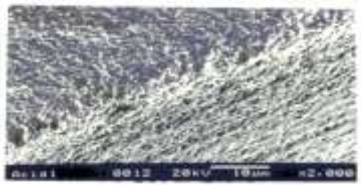
Carbide Inserts	 × 5000	 × 2000
As received		
Treat 1		

Figure 2 SEM micrographs showing the surface and cutting edge morphology of tool before and after various pretreatments.

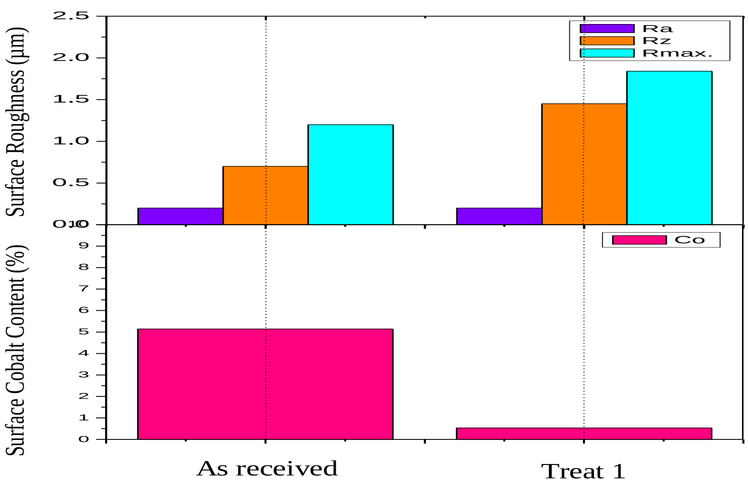
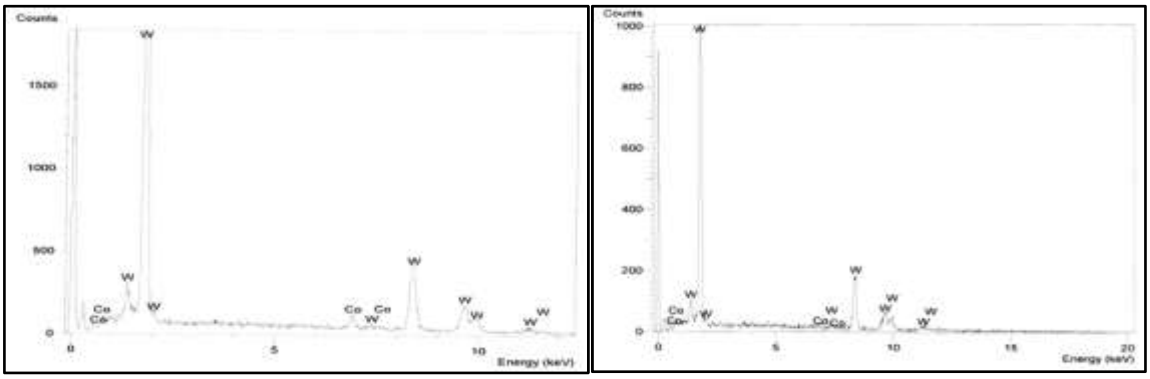


Figure 3 Surface roughness and cobalt content of WC-Co inserts surface before and after various treatments.

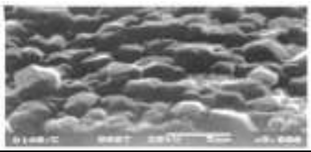
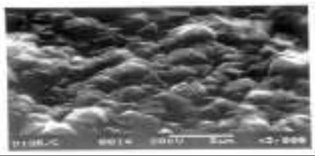
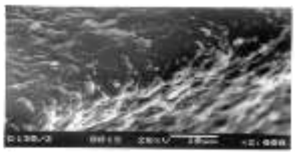
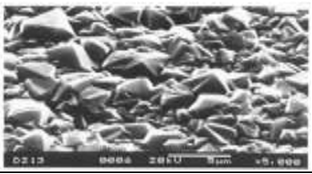
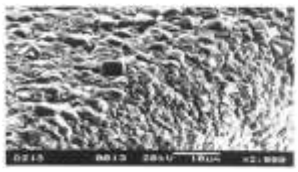
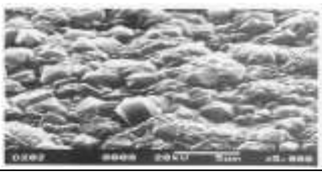
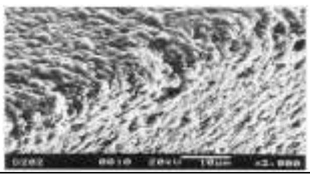


As received Treat 1

Figure 4 EDAX spectra of the surface of as received substrate and substrate surface have undergone various treatments.

3.2. Effect of Deposition Pressure on Morphology of Coating Deposited on Pretreated and diamond seeded substrates

With the increase of deposition pressure to 0.666 kPa typical growth habit can be immediately seen in grown diamond crystals. The size of crystal has also increased. The deposited crystals appear to form a continuous coating with efficiently covered face and flank including the cutting edge as shown in Fig.5. However, same pinhole sites can also be observed on the cutting edge which does not appear to be uniform after coating.

Deposition Pressure (kPa)	 × 5000	 × 2000
0.666		
1.333		
2.666		
3.999		

Treat 1,Seeding: Diamond powder

CH_4 / H_2= 0.5 SCCM / 100 SCCM

Deposition temperature:700 ^{0}C,Deposition time:8 hours

Figure 5 SEM micrographs showing the effect of deposition pressure on morphology of coating deposited on carbide inserts pretreated with Treat 1.

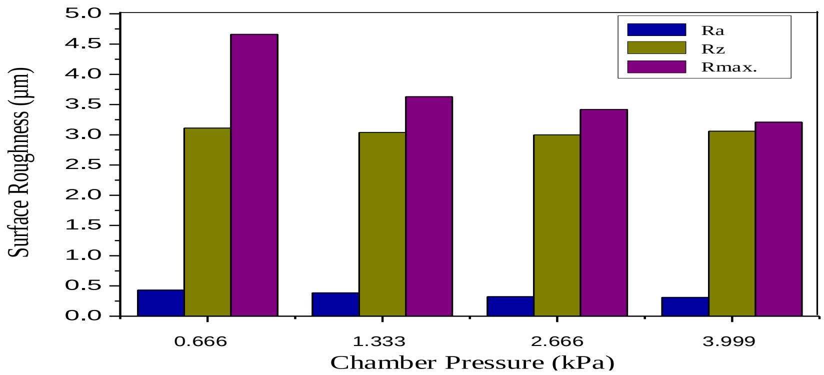


Figure 6 Variation of the surface roughness (R_a, R_z and $R_{max.}$) of the coated inserts pretreated with Treat 1 with deposition pressure.

With further rise of pressure to 1.333 kPa, diamond coating appears to be denser. Crystal size also slightly reduced than what can be seen in the coating deposited at 0.666 kPa. Less dense coating with large crystal at 0.666 kPa deposition pressure made it rougher than the diamond coating obtained at 1.333 kPa. This is shown as the bar diagram in Fig. 6.

Quality of the coating in terms uniformity and compactness has shown the sign of further improvement near the cutting edge when the deposition pressure was raised to 2.666kPa. A structure very close to ‘cauliflower’ like developed on the coated surface which is in total contrast to a well-developed dense crystalline coating that deposited on the acid solution treated surface as can be seen in Fig. 5. Coating morphology at pressure of 3.999 kPa does not show any momentous difference from that of the coating deposited at 2.666 kPa. The most important feature which draws immediate attention is that pitting and crater formation grown on the diamond crystals throughout the coated surface at 3.999 kPa. Upon reviewing the SEM pictures presented in Fig. 5 one may reasonably come to the conclusion that the diamond coating deposited at 2.666 kPa on acid solution etched (Treat 1) carbide inserts happened to be the best in terms of compactness, uniformity, coverage of rake, flank and cutting edge. It can be concluded that a single step etching with HNO_3+HCl+H_2O (1:1:1) solution is more effective and economic.

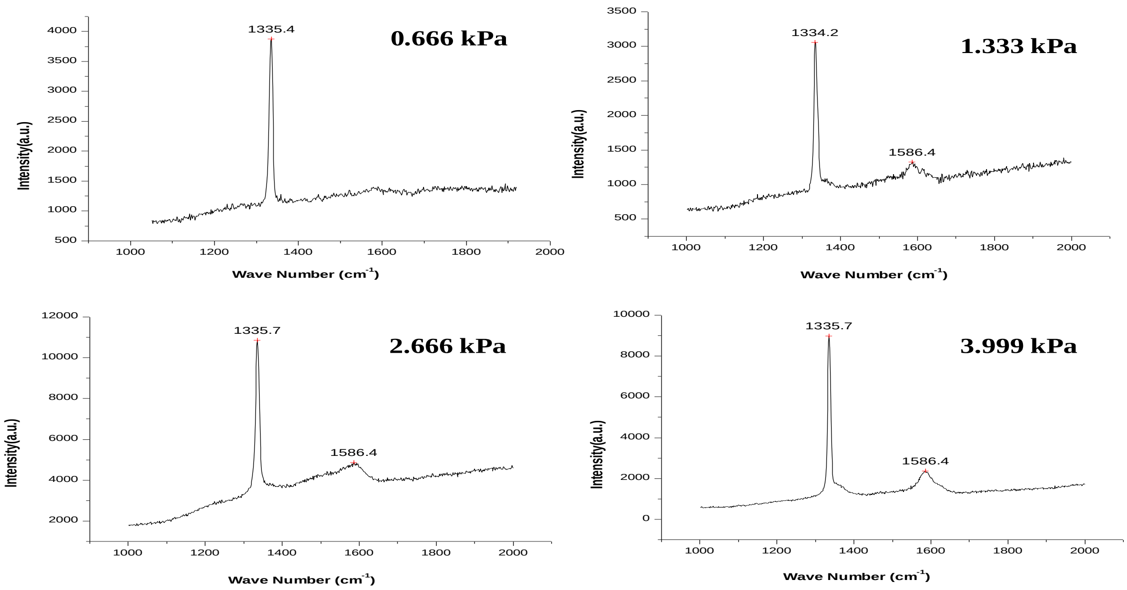


Figure 7 Raman peaks showing the sp^3 / sp^2 content with variation with deposition pressure.

Fig. 7 show Raman peaks of the diamond coated inserts pretreated with Treat 1 after deposition time of 8 hours. The SEM micrographs reveal nucleation density is less. Crystals were not developed to its size due to low pressure. So on broadening of the peaks can be seen sp^3 / sp^2 content is in equal portion. Due to the availability of large crystal tool deposited at 0.666 kPa pressure has shown very low intensity G-phase. The other carbide inserts deposited at higher pressures of 1.333, 2.666 and 3.999 kPa have shown same kind of peaks with slight disturbance at the grain boundaries.

3.3. Effect of Substrate Temperature on Nucleation and Growth of Diamond on Pretreated (Treat 1) Substrates

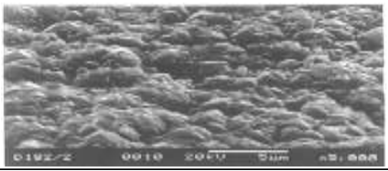
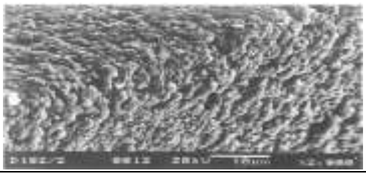
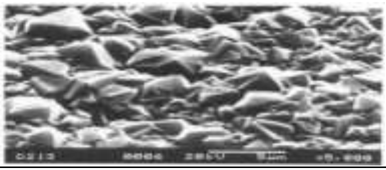
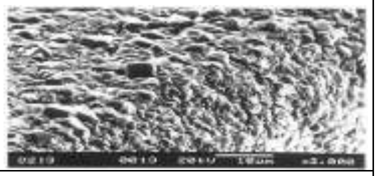
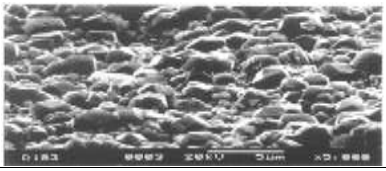
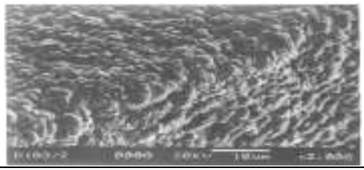
Temp. (^{0}C)	 × 5000	 × 2000
650		
700		
750		
Treat 1 CH_4 / H_2= 0.5 / 100 Pressure:2.666 kPa Seeding: Diamond powder Deposition time: 8 hours		

Figure 8 SEM micrographs showing the effect of substrate temperature on nucleation and growth of diamond on carbide substrates pretreated with Treat 1.

SEM micrograph of the Fig. 8 visualizes the morphology of diamond coating deposited at a low temperature of 650 ^{0}C. Both face and flank surfaces are well covered with the coating. SEM picture of the rake face does not show a well faceted morphology. The requirement of high substrate temperature is strongly felt for un-etched substrate wherein etching of Co by hydrogen needs a high temperature [27]. However the present investigation gives clear impression that a substrate temperature of 650 ^{0}C did not favour growth of well faceted diamond coating with the present configuration of the CVD set-up. This is in good agreement with other previously done investigations [28, 29, 30].

When the temperature was raised to 700 ^{0}C a compact diamond coating could be obtained. Comparing the morphology of the coating deposited at 650 ^{0}C with that grown at 700 ^{0}C, one can arrive at the conclusion that crystallinity of the coating with (111) habits could be effectively obtained. At elevated temperature effectual etching of amorphous phase by hydrogen could lead to very good crystallinity with high quality diamond. At elevated substrate temperature of 750 ^{0}C the coating effectively covered the rake, cutting edge and the flank. However, the cutting edge shows same 'pin hole' sites. Average size of crystals appears to be slightly less than that obtained at 700 ^{0}C. Hence at 750 ^{0}C the effect of atomic hydrogen on etching of not only amorphous phase but also diamond may be considered.

3.4. Effect of Deposition Pressure and Substrate Surface Treatment on Adhesion of Diamond Coating

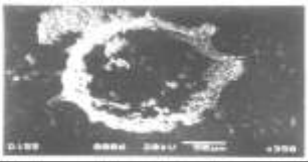
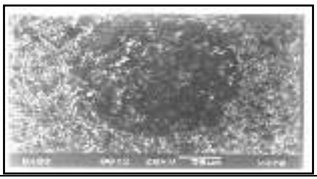
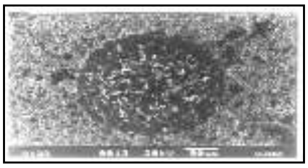
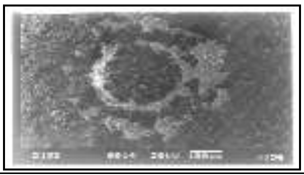
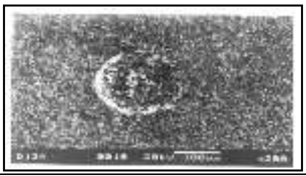
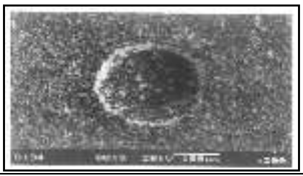
Deposition Pressure (kPa)	**294 N**	**588 N**	**980 N**
0.666			
1.333			
2.666			
3.999			
Treat 1 CH_4 / H_2 : 0.5 / 100 Substrate temperature :700 ^{0}C Deposition Time: 8 hours			

Figure 9 SEM micrographs showing the indentation crack diameter of diamond coated carbide inserts pretreated with Treat 1.

SEM micrographs in Fig. 9 of the indentation crack morphology of the coating deposited on acid treated substrate (Treat 1) at 0.666 kPa show poor adhesion. Crater appears due to indentation with the diamond cone but the coating suffered lateral flaking beyond the crater even at 294 N indicating poor adhesion. With increasing load to 588 N followed that by 980 N the size of the crater as well as that of the flaked area around crater also increased. Deposition at 1.333 kPa caused marginal increment in size of the flaked area around the crater. Real improvement in coating adhesion could be achieved when the deposition pressure was set in the range of 2.666 and 3.999 kPa. SEM micrographs clearly suggest that at 294 N load the coating underwent deformation without experiencing crack formation. At 588 N load coating deposited at 2.666 kPa shows only a small crack which increased marginally when the deposition pressure was augmented to 3.999 kPa. Clear sign of flaking can be noticed in the coating when the indentation load was 294 N. However it was possible to assess the indentation crack diameter with μm marker provided with each SEM micrographs and results shown in Fig. 9 are displayed graphically in Fig. 10. It appears clearly from Fig. 10 that a deposition pressure in the range of 2.666 and 3.999 kPa is most preferred to achieve maximum coating substrate adhesion.

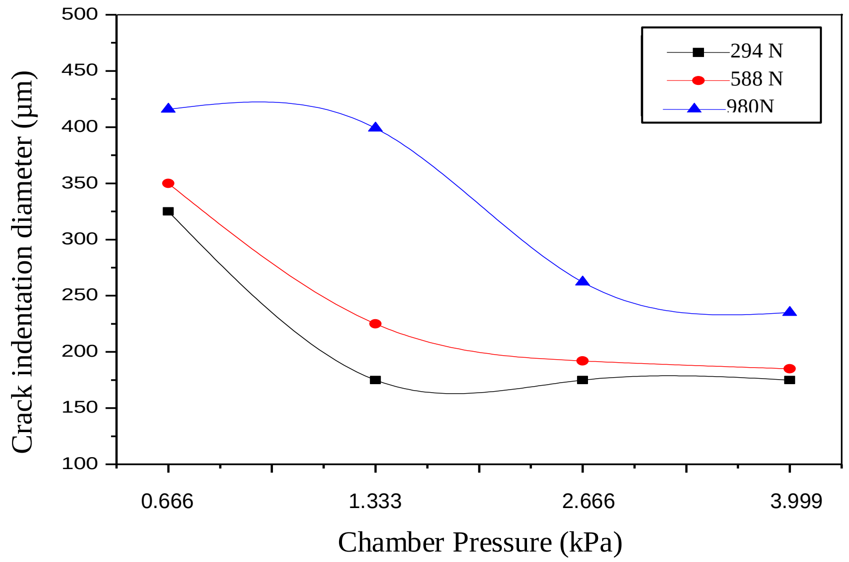


Figure 10 Crack indentation diameter of diamond coated carbide inserts at various chamber pressures.

3.5. On Performance of Diamond Coated Tools

Fig. 11 clearly reveals that fluctuation of both tangential(P_z) and axial(P_x) component of cutting force occurred over a broad band in comparison to what happened in case of a diamond coated tool (Treat 1, Pressure: 2.666 kPa, CH_4 / H_2= 0.5 / 100, Temp. = 700 ^{0}C, Time: 8 hrs). The tangential and axial cutting force was always greater for the uncoated carbide tool compared to the diamond coated tool.

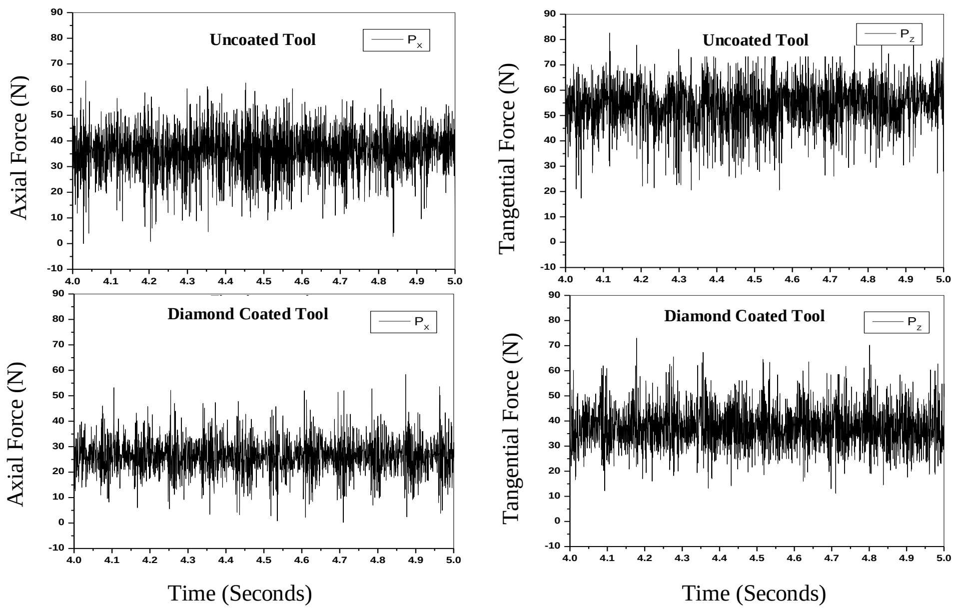


Figure 11 Comparison of cutting forces (axial and tangential components) of uncoated and diamond coated carbide insert (Treat 1, Pressure: 2.666 kPa, CH_4 / H_2= 0.5 / 100, Temp. = 700 ^{0}C, Time: 8 hrs).

The surface roughness produced by the uncoated tool on the aluminium work-piece was quite high compared to that produced by various diamond coated tools prepared under various deposition pressures as shown in Fig.12. It is interesting to note that surface roughness produced by different coated tools is mainly influenced by the roughness of the coating at the flank near the cutting edge. In machining of aluminium by uncoated carbide and diamond coated carbide tool, affinity of the work-piece towards the tool surface played an overriding influence in affecting surface roughness of the work-piece.

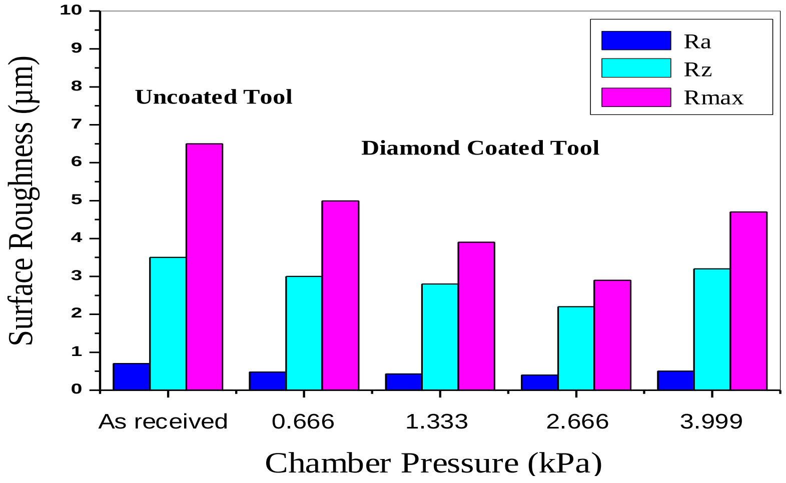


Figure 12 Surface roughness of work-piece machined by uncoated and diamond coated inserts produced at the different deposition pressure, pretreated with Treat 1.

4. CONCLUSIONS

1. Treat 1 could reduce the concentration of surface cobalt of WC+Co substrate to as low as 0.5%. The surface roughness was also increased.
2. Dense coating with desired morphology and growth habit could be obtained on acid treated (Treat 1) surface which was seeded with diamond. At 0.666 kPa, good crystallinity was observed, but the coating appeared to be somewhat porous. At 1.333 and 2.666 kPa pressure, the morphology obtained was very good with clear facets. At 3.999 kPa, coating was found to lose its crystallinity.
3. At a low substrate temperature of 650^0C, the crystals formed were small containing more amorphous phases. With increase in temperature to 750^0C, the crystal became discrete and mostly cubo-octahedral. The medium temperature of 700^0C showed improved morphology.
4. Diamond coating on acid treated (Treat 1) insert along with diamond seeding provided greater adhesion strength. Among the acid treated specimen, diamond coating deposited on 2.666-3.999 kPa range has resulted in highest adhesion.
5. The adhesion test results confirmed that diamond coated tool obtained by Treat 1 can be utilized for turning tests.
6. During dry machining, compared to uncoated tool, a 4-5µm HFCVD diamond coating exhibited remarkable inertness towards aluminium leading to substantial reduction of cutting force and improvement of work-piece surface finish. Tools coated at higher pressure produced smoother surface on the work-piece than obtained at lower pressure.

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