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The Study on Diamond-Coated Insert by DC Plasma Jet CVD

Zhou Kesong*, Dai Mingjiang*, Song Jinbing*, Kuang Tongchun+, Liu Zhengyi+
*Guangzhou Research Institute of Nonferrous Metals, Guangzhou, PR. China
+South China University of Technology, PR. China

Summary

Diamond coating were deposited on cemented carbide inserts by DC plasma jet CVD. The cemented carbide inserts were pretreated by methods including chemical etching of Co, Ar/H_2 plasma etching. The characteristics of diamond film, interface structure, adhesion strength and film stress were analysized by different methods such as SEM, XRD, Raman spectrum etc. A comparing experiment of cutting Al - 22% Si alloy was carried out with diamond-coated cemented carbide inserts and uncoated cemented carbide inserts. The results show that the diamond-coated cemented carbide insert has a great advantage for cutting abrasive high content Al - Si alloy.

Keywords

Diamond, coatings, DC plasma jet, cemented carbide, inserts

1. Introduction

Low pressure CVD diamond coating can prolong the lifetime of cemented carbide cutting tools applied for working on nonferrous materials due to its excellent mechanical properties, especially its extreme high hardness, low friction coefficient as well as good chemical and wear resistance. Diamond-coated tools are suitable for dry machining without expensive coolants, which are hazardous to the environment (1). However it's well known that cobalt in cemented carbide has a negative effect on the deposition of diamond coating, resulting in low nucleation rates, formation of graphite carbon and poor

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coating adhesion (2). Therefore, cobalt is normally removed from the surface region of the cemented carbide inserts prior to the deposition of diamond coating. Even so under the high temperature of diamond deposition process and long deposition time, cobalt can migrate to the interface of the diamond coating/substrate forming cobalt droplets (2). So the high diamond deposition rate CVD method of DC plasma jet has its advantage over other CVD technologies due to its short diamond deposition time (3).

This paper describes the research on diamond-coated cemented carbide inserts by DC plasma jet technology. The pretreatment methods of cemented carbide inserts and the deposition process were researched. The diamond coated inserts were characterized by SEM, TEM, XRD and EDX analysis and Rockwell indention test. The cutting application of diamond coated inserts were also carried out by cutting aluminum alloys and copper alloys.

2. Experimental

Standard cemented carbide cutting inserts (WC-6%Co, similar to ISO K10 and WC-3%Co) have been used as substrates to be coated with diamond. To enhance nucleation and adhesion of the diamond coating, mechanical and/or chemical pretreatments were performed on the as-ground inserts. All inserts in this paper subjected etching in 25% HNO_3 for 1-15 minutes. Then they were conditioned by Al_2O_3 blasting or scratching with diamond powder. Deposition of diamond was carried out by a DC plasma jet CVD system described elsewhere (4). The process parameters were listed in Table 1. Diamond coated cemented carbide inserts were characterized by SEM, TEM, Raman analysis, XRD, Rockwell indention test, EDX analysis. Lab and field cutting test were also performed.

Table 1 DC plasma jet CVD process for coating cemented carbide inserts

Substrate	WC-6%Co, WC-3%Co							
Pretreatment	Co etching, scratching with diamond or Al_2O_3 blasting, plasma etching							
Deposition process parameters	Ar l/min	H_2 l/min	CH_4/H_2 %	Voltage V	Current A	Chamber pressure kPa	Substrate temperature	Deposition time, min
	6	5	1-5	80-170	70-200	2-8	700-1100°C	20-60

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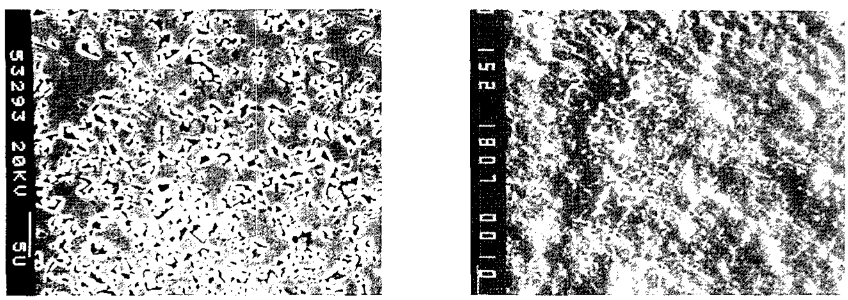
3. Results and discussion

3.1 Influence of Co etching and plasma etching on the substrate

Surface morphology of substrate after Co etching is showed in Fig. 1a. It can be seen that many holes were left after Co etching. EDX analysis indicated that cobalt on the surface was nearly completely removed after 5 minutes etching (Table 2). Fig. 1b illustrates that substrate surface is roughened and no holes are left after plasma etching. Cross-section SEM image showed in Fig. 2 indicates that there are 3 layers in the substrate after fully pretreatment, including cobalt etching, mechanical pretreatment and plasma etching: the first layer is dense (about 4μm), the second is porous (about 70μm) and the third is unaffected substrate. EDX and hardness test indicated that the first layer was abound with tungsten and was formed due to the decarbonization of WC under hydrogen atmosphere. The second layer was Co-depleted and slightly carbon-abound. Fig. 4 showed that after 2 minutes plasma etching WC of the surface region had nearly completely converted to tungsten or Co-W-C compounds and no single phase Co or WC existed any more.

Table 2 Cobalt content of the substrate at different etching time

Etching time, min	Co, wt%	W, wt%	C, wt%
0	13.73	85.27	1.00
2	2.71	96.17	1.12
5	0.27	98.55	1.18
10	0.14	98.73	1.13



(a) (b)

Fig.1 Surface morphology of substrate: (a) after Co etching; (b) after Co etching and plasma etching

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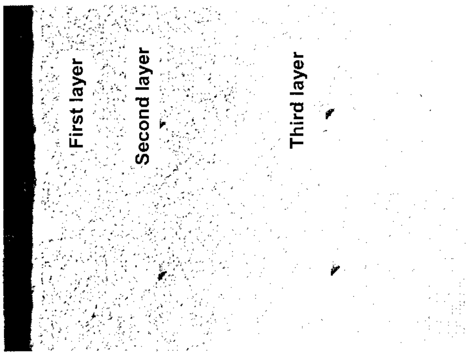

Fig. 2 Cross-section of substrate after Co etching and plasma etching

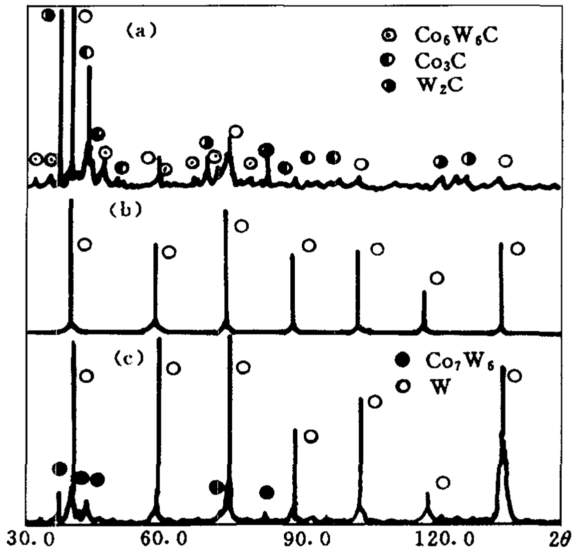

Fig. 3 XRD plot of substrate after: (a) 2min plasma etching without chemical etching; (b) 2min plasma etching after chemical etching; (c) 5min etching after chemical etching

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3.2 Influence of substrate pretreatment on the diamond nucleation

Table 3 listed the diamond nucleation densities deposited under the same depositing process parameters with different pretreatments. It can be seen from Table 3 that the nucleation density could be 2 or 3 orders higher with diamond scratching compared with other pretreatment methods. While large size diamond powders mixed with small size diamond powders (named as B) using for scratching substrate, the nucleation could be an order high than just using large size diamond powder (named as A). It is generally believed that submicroscopic fragments of the diamond left on substrate during scratching will serve as starting points for the nucleation process (5).

Table 3 the nucleation density using different pretreatment methods

Substrate pretreatment	Process parameters	Nucleation densities
Al_2O_3 scratching	CH_4/H_2=4%	10^3-10^4
Al_2O_3 blasting	Substrate temperature = 800°C,	10^3-10^4
Diamond powder A	chamber pressure = 5kPa,	5-6X10^6
Diamond powder B	Deposition time = 5min	5X10^7

3.3 Influence of diamond deposition process on the diamond nucleation

Substrates were scratching with diamond powder B. Then diamond films were deposited under different process. The results were showed in Table 4. The results indicated that deposition process has some effect on the diamond nucleation density to some extent, but not as seriously as the pretreatment method. Medium high methane concentration and medium low substrate temperature resulted in high diamond nucleation density.

Table 4 Influence of diamond deposition process on the diamond nucleation

Deposition process parameters		Nucleation densities
CH_4/H_2, %	2	10^6
	4	5X10^7
	6	2X10^7
Substrate temperature, °C	700	1.5X10^7
	800	5X10^7
	900	5X10^6
Chamber pressure, kPa	5	5X10^7
	7	5X10^7

* Other parameters were the same as in Table 3.

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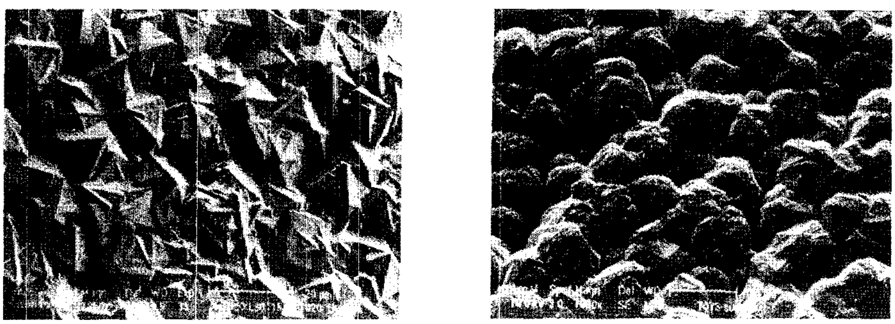
3.4 Influence of deposition process on the diamond coating quality

The methane concentration and the substrate temperature are the two main process parameters influencing diamond coating quality. Fig. 4 indicates the surface morphologies deposited under different methane concentrations. Diamond deposited under low methane concentration is faceted and very compact, while it is cauliflower-like under high methane concentration. Fig. 5 is the typical Raman spectrum of diamond coating deposited by DC plasma jet CVD. Substrate temperature in the range of 700-1100°C has no great effect on the diamond coating purity, but has effect on the diamond grain size. High substrate temperature leads to large grain size.

3.5 Cross-section structure of diamond-coated inserts

There are two kinds of cross-section morphology as shown in Fig. 6. Fig.6a indicates that WC grains near the diamond/substrate interface are fine and smooth which is formed through decarbonization and recarbonization of the substrate surface prior to diamond nucleation. These fine WC grains layer is favorable for diamond adhesion strength. TEM images in Fig. 7 illustrate the diamond/substrate interface. It shows a bright thin discontinuous gap between the diamond coating and the substrate as the result of ion bombardment. This layer is more easily sputtered than diamond and WC. It was confirmed to be graphite. So the cross-section structure can be considered as: diamond film/thin graphite layer/fine WC layer/residual Co-etching layer/substrate (6).



(a) (b)

Fig. 4 Influence of the methane content on the diamond coating morphology: (a) 1% CH_4/H_2; (b) 3% CH_4/H_2

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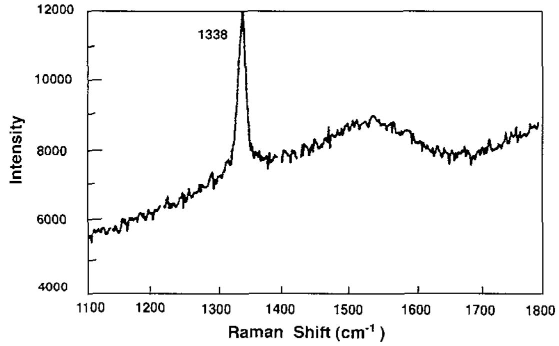
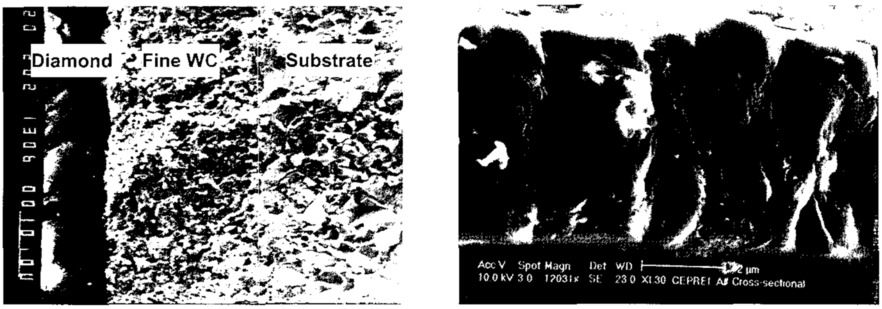


Fig. 5 Raman spectrum of typical diamond coating on cemented carbide inserts



(a) (b)

Fig. 6 Cross- section structure of diamond-coated inserts

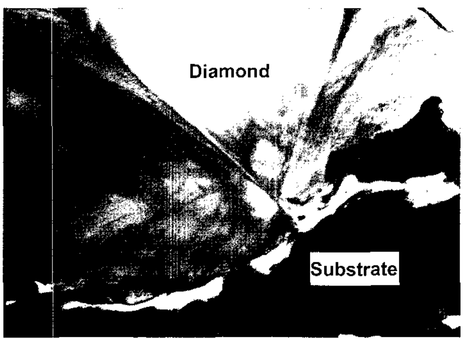


Fig. 7 Bright field TEM image showing diamond coating/substrate interface

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3.6 Factors influencing the diamond adhesion

The diamond coating adhesion was investigated by Rockwell indention test. The adhesion was characterized by the critical load, which causes the crack of diamond coating.
There are many factors, which influence the adhesion of diamond coating, such as coating thickness, when coating is too thick it will spall itself. So diamond coating thickness generally is controlled less than 20µm. Cobalt etching can also influence the adhesion of diamond coating. Very poor adhesion of coating was obtained without cobalt etching, which attributed to the detrimental effect of Cobalt. But too much Co etching weakens the substrate and leads to poor adhesion too. It is shown in Table 5.
Surface roughness has a great influence on adhesion strength of diamond coating; rougher substrate surface leads to higher adhesion strength, which is listed in Table 6. Unfortunately diamond deposited on rough surface is rough too and this is not good for the work-piece surface finishes. So improvement of coating adhesion by roughening substrate surface is very limited.
It's generally believed that proper compressive stress is favorable for the coating adhesion strength. Fig. 8 illustrates that the coating adhesion strength is best at compressive stress of –2Gpa. Too low or too high stress is not good for the adhesion strength.
The quality of the diamond coating also influences the coating adhesion. The coating adhesion is poor if diamond coating contains too much non-diamond carbon.

Table 5 Influence of Co etching on the coating adhesion

Etching time, min	Critical load Pc, kg/mm^2
0	30
15	125
30	60

Table 6 Influence of surface roughness on the coating adhesion

Roughness, µm	Pc, kg/mm^2	
	1.5% CH_4/H_2	2.0% CH_4/H_2
0.03	55	40
0.10	125	80
0.17	150	125

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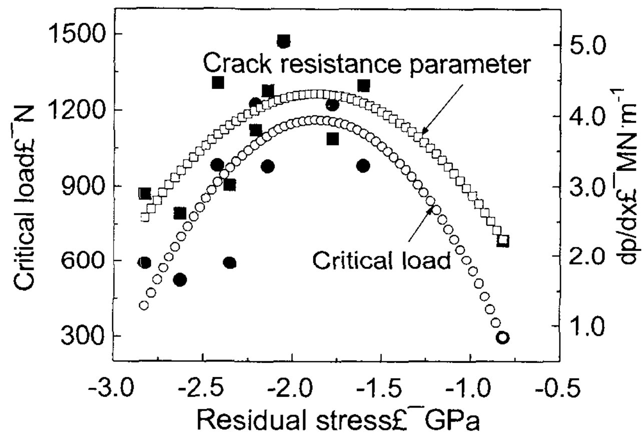

Fig. 8 Residual stress of diamond coating on cemented carbide inserts

3.7 Machining test of diamond coated inserts

Lab machining test of diamond-coated inserts was conducted with a CNC machine. Work-piece material was Aluminum-Silicon alloy, which contains 22%wt silicon. Most silicon phase sizes were larger than 60μm. The applied cutting conditions were: cutting speed = 180m/min, cutting depth = 0.2mm, feed = 0.1mm/rev., dry machining. It was found that tool life was greatly increased to about 55 times longer than uncoated inserts, as shown in Fig. 9. It was also found that the work-piece surface finishes were improved and the cutting temperatures were decreased [7].

Field dry machining tests were carried out on the Aluminum bronze alloy worm production line. The parameters were: cutting speed = 150m/min, cutting depth = 2-3mm, feed = 0.2mm/rev. The field cutting tests showed that the tool life of the coated inserts were dozens of times longer than that of the uncoated inserts. The work-piece surface finishes were also improved.

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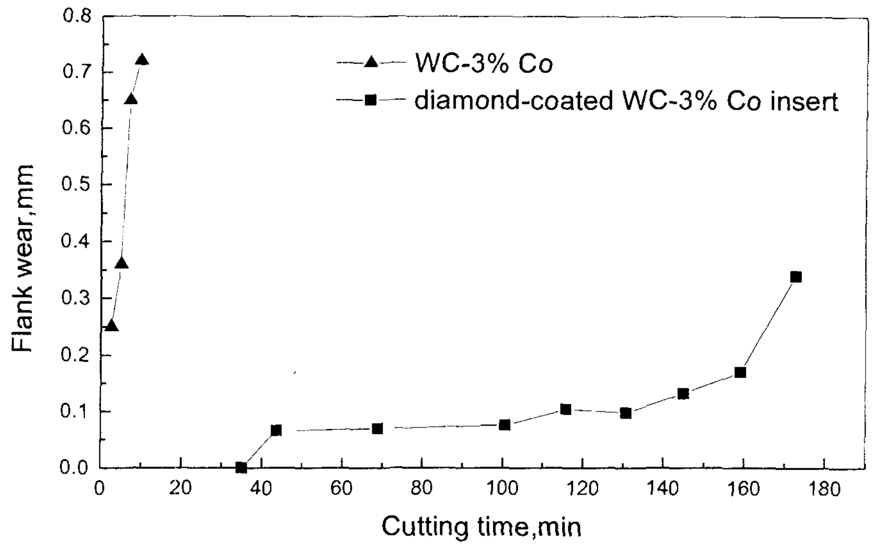

Fig. 9 Cutting performance of diamond-coated inserts

4. Conclusion

1. Pretreatment on cemented carbide substrate has great effect on substrate, diamond nucleation and diamond coating adhesion;
2. Deposition process parameters, especially substrate temperature and methane concentration, influence diamond nucleation and diamond quality.
3. Diamond coating thickness, stress, substrate pretreatment and coating quality influence diamond coating adhesion;
4. Diamond coating/substrate interface formed by DC plasma jet technology is diamond/thin graphite/fine WC layer/residual Co-etching layer/substrate;
5. Diamond coating greatly prolongs the tool lifetime of cemented carbide inserts when cutting aluminum alloy, the surface finishes also improved and the temperatures of the diamond coated inserts and the work-piece was decreased.

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