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Thin Film Adhesion and Cutting Performance in Diamond-Coated Carbide Tools

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The effects of surface conditions of the C-2 cemented carbide substrate on the adhesion of diamond film were investigated The substrates were pretreated for different times with Murakami's reagent and then the acid solution of an H_2SO_4-H_2O_2. The adhesion strength was estimated by a peeling area around the Rockwell-A indentation The cutting performance of the diamond-coated tools was evaluated by measuring flank wears in dry turning of Al-17% Si alloy The morphology of deposited diamond crystallites was dominated by (111) and (220) surfaces with a cubo-octahedral shape. The diamond film quality was hardly affected by the surface conditions of the substrate. The variation of tool life with longer substrate etching times resulted from a compromise between the increase of film adhesion at the interface and the decrease of toughness at the substrate surface. The coated tools were mainly deteriorated by chipping and flaking of the diamond film from a lack of adhesion strength, differently from the wear phenomena of PCD tools.

Key words : Thin film diamond-coated tool, Co depletion layer, Adhesion strength, Cutting performance, Tool life

I. Introduction

The diamond is of great interest due to its unique properties[1-3] such as superior hardness, very high thermal conductivity and extremely low expansion coefficient etc.. The wear-resistant applications of diamond, however, are limited to nonferrous materials because of the high-temperature chemical interactions with ferrous alloys. The diamond tools are usually divided into PCD (high pressure polycrystalline diamond) tool and diamond-coated tool, which are used in the automotive industry to machine hypereutectic aluminium-silicon alloys. PCD is brazed to a cutting edge of cemented carbide insert for the application. The advantage of the diamond-coated tool over the PCD tool is that all cutting corners are usable and various tool geometries are available.

Although diamond film can be adhered well to silicon nitride, such a tool is applied only in the machining of graphites and polymer composites owing to the poor fracture strength of ceramics.[4] Attempts to coat diamond on cemented tungsten carbide revealed that the cobalt binder promoted the formation of graphite and eventually prevented the adhesion of deposited diamond to the substrate.[4,5] The film adhesion, therefore, should be improved through effective substrate pretreatment, by which microdefects must be created on the surface together with the removal of Co.[6] In order to obtain good diamond film on carbide tool, the substrates are in general etched and scratched,[6,7] or coated with buffer layers.[8-10]

The diamond layers are synthesized from gaseous phase mostly by hot filament CVD[11,12] and microwave plasma CVD[13,14] for thin film (less than 50 μm) and DC arc jet[14,15] for thick film.

In this study, the inserts of C-2 carbide were used as substrates, and the diamond thin films were deposited onto the substrates pretreated with chemical solutions by microwave CVD process. Morphology, crystallinity and texture of the films are observed, and the film adhesion is examined by means of Rockwell-A point indentation. The performance of the diamond-coated tools is also described with the results of cutting test for Al alloy.

II. Experimental

The substrates were the cemented carbide inserts of SPGN 120308 type with the composition of 92WC-2{TiC+Ta(Nb)C}-6Co (K10 grade from Korea Tungsten Mining Co.). From the surface of the substrates, first a small amount of the carbides were etched by Murakami's reagent for the scratching effects and subsequently the remaining Co binder was removed by the acid solution of an H_2SO_4-H_2O_2.[16] Table 1 exhibits the surface modification parameters for the substrates. The Co depletion at the surface was observed with the cross-section of substrate by energy dispersive X-ray spectroscopy (EDS).

The diamond deposition was carried out on the pretreated substrates in an CH_4-H_2 atmosphere using a microwave CVD apparatus, as shown in Fig. 1, where a maximum power of 1.5 kW (2.45 GHz) could be offered from the microwave generator (ASTeX S-1500). The deposition temperatures were measured through a circular (ϕ70 mm) quartz window with optical pyrometer

Table 1. Parameters for Surface Pretreatment of Substrates

Sample index	Etching time (min)	
	Murakami's reagent	H_2SO_4-H_2O_2 solution*
A	1	5
B	5	5
C	5	10

*H_2SO_4 : H_2O_2=1 : 9.[17]

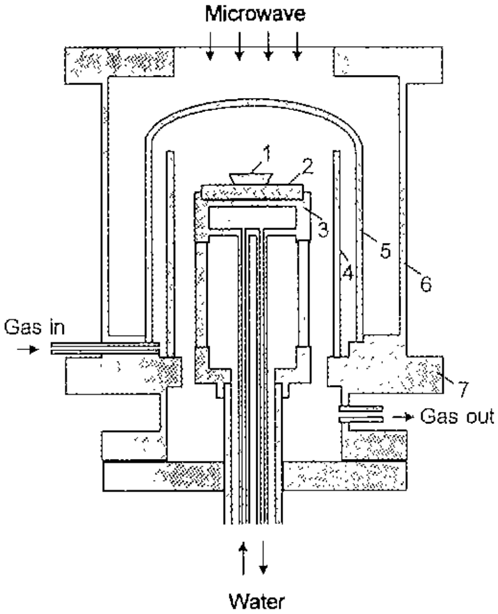

Fig. 1. Reactor configuration of microwave plasma CVD apparatus (1: substrate, 2: SiC board, 3: SS holder, 4: quartz gas guide, 5: quartz bell jar, 6: chamber, 7: base plate).

Table 2. Experiment Condition for Diamond Deposition

Deposition parameter	Values
Substrate temperature (°C)	900
System pressure (torr)	50
H_2 gas flow rate (sccm)	500
Methane concentration (vol.%)	1.0 in H_2
Reaction time (hr)	9~13.5

(Minolta TR-630). SiC disk (ϕ100 mm×T5 mm) was used as a suscepter on the substrate holder with water cooling. The deposition parameters are mainly substrate temperature, system pressure and gas flow rate. The experimental conditions are given in Table 2.

The deposited diamond films were characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD) and Raman spectroscopy. The film adhesion was estimated by measuring the maximum length from the center to the edge of film (peel-off radius) delaminated by the indentation of Rockwell-A-Scale hardness tester with the load of 60 kg.[18] The cutting performance of thin film diamond-coated inserts was tested by dry turning of 390 aluminium (Al-17%Si). The thickness of the diamond films was about 10 and 15 μm. The workpiece was in the form of a cylinder (ϕ200 mm×L300 mm). The cutting condition was as follows: cutting speed, 275-365 m/min; depth of cut, 0.5 mm; feed rate, 0.1 mm/rev; entrance angle, 75°. The test was carried out in 5 minute increments to investigate the wear procedure. The flank wear of inserts was measured by optical microscopy (×100).

III. Results and Discussion

1. Surface modification of cemented carbide substrates

In the surface pretreatment of the substrates, Murakami's reagent was used for etching carbide components, by which the exposure of cobalt was increased, so that the Co binder could be easily removed with the acid solution. Figure 2 shows the surface characteristics of the substrates etched for different times by Murakami's reagent and the acid solution. The microstructure of the substrate surfaces becomes porous gradually with increasing etching times, at which the carbide grains are less connected in order of sample A, B, and C. It is evident that the increase of etching depth decreases the detrimental influence of cobalt on diamond coating but results in more porous substrate surfaces.[19] In EDS analysis, no cobalt component was detected at the surfaces after the etching treatment. The results suggest that these pretreatment methods are very effective in removing the binder phase from the surface of cemented

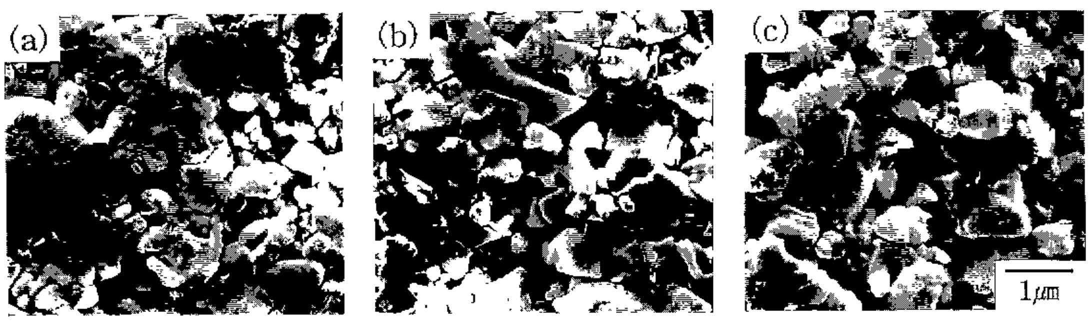

Fig. 2. Surface morphologies of cemented carbide substrate after surface pretreatments: (a) sample A, (b) sample B, (c) sample C.

carbides. However, an excessive Co etching is not desirable because it can significantly weaken the substrate.[1]

2. Characterization of deposited diamond films

With the deposition condition of Table 2, diamond film was deposited on samples A, B and C. The films all exhibited similar characteristics irrespective of the substrate pretreatment. The film morphologies also were uniform, but the crystal size at the corners of the coated tool was slightly larger than that at the central region. This phenomenon might be caused by high plasma density at the periphery of a tool insert during deposition.

Figure 3 shows a typical scanning electron micrograph and the corresponding Raman spectrum of diamond film at rake surface near the cutting edge. From the film morphology, well-faceted cubo-octahedral diamond crystals[2,20] are clearly identified with a rough surface. The grain size is in the range of 6.5 to 11.5 μm. The film was deposited for 13.3 hr, and the thickness was about 15 μm. The Raman lines demonstrate sp^3-bonded crystalline diamond and sp^2-bonded amorphous carbon, correspondingly to a sharp peak in the vicinity of the standard 1332 cm^{-1} and a broad peak around 1500 cm^{-1}, respectively.[21] It could be noted that the particles of Co binder migrated on the surface due to the higher deposition temperature at the tool corners, which dissolved the deposited diamond partially.[22]

As shown in Fig 4, XRD analysis revealed that the diffraction lines of diamond originated from the (111), (220), (311), and (400) crystallographic planes and their relative intensity was 100, 45, 6, and 3 in that order. The diamond crystallites were dominated by the (111) and (220) faces. Therefore, the film surface is closer to rather rough (111) facet than smooth (100) facet.[23]

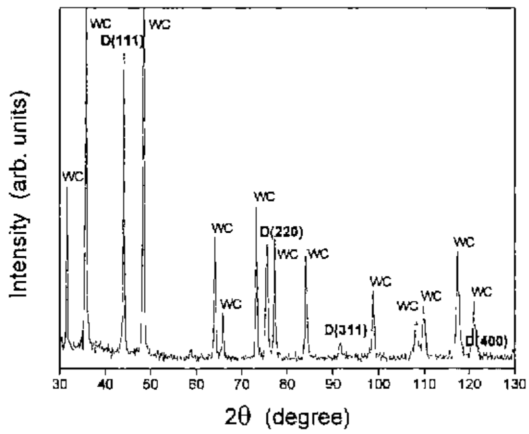

Fig. 4. A typical X-ray diffraction pattern of diamond-coated tools (deposition condition: T_s=900°C, P=50 torr, C_{CH4}=1% in H_2, t=13.3 hr).

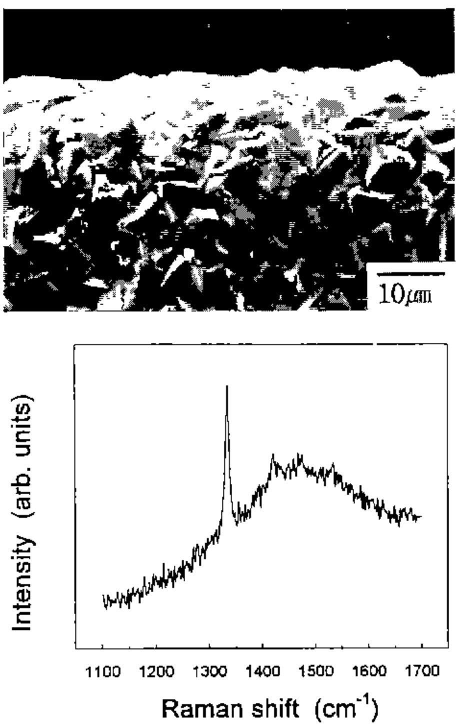

Fig. 3. A typical morphology and corresponding Raman spectrum of diamond films at cutting edge of coated tool (deposition condition: T_s=900°C, P=50 torr, C_{CH4}=1% in H_2, t=13.3 hr).

3. Film adhesion and cutting performance

The indentation was tested more than two times for each specimen. The deviation of peel-off radii was within ±5 μm for the same specimen. In Table 3, the indentation results are given as the mean value of peel-off radius together with coating thickness. The radii for the sample A and B are much the same at the film thickness of 10 μm but considerably increased with the thickness of 15 μm. From the fracture area of specimens, Co depletion depth was measured as 16, 20, and 25 μm for the sample A, B, and C, respectively. Such a decrease in adhesion strength for the thicker film may be ascribed to more increased stress with longer deposition times because the coating adhesion of cemented carbide substrates is influenced by the thermally induced stress[8,22] as well as the Co binder phase. With specimen 4 for the sample C, however, the peel-off radius is dramatically decreased, representing much better adhesion of diamond to the substrate surface

The cutting test was carried out with two edges for each of these tool specimens. The criterion for a worn-

Table 3. Mean Peel-Off Radius of Specimens Measured by Indentation Test

Specimen No	Substrate index	Film thickness (μm)	Peel-off radius (μm)
1	A	10	325
2	B	10	335
3	B	15	415
4	C	10	215

out tool was set on with the flank wear of 250 μm because of poor surface finish of the workpiece in the values over the maximum wear. Figure 5 exhibits the mean life time of the tools. The cemented carbide tool was uncomparable with the coated tools because it resulted in a critical wear already during the early stages of test. The tool of specimen 1 indicates the life time of 82 min with the best performance, whereas the tool life at specimen 2 is greatly decreased to 35 min, which is somewhat improved with the increase of film thickness. Also compared specimen 2 with specimen 4, the lives make only a little difference although the adhesion strength is much higher in case of the latter owing to the increase of the etching time by the acid solution. Therefore, the variation of the tool lives is likely to result from a compromise between the increase of adhesion at the film-substrate interface and the decrease of toughness at the substrate surface region.

Figure 6 shows that damage to the edge of tested inserts was caused by chipping and flaking of the diamond film at relatively short cutting times. This result coincides with the fact that as the cutting proceeds, the failure of diamond-coated tools is due to microfractures developed by crack,[24] differently from the wear of conventional PCD tools which gradually occurrs at the cobalt grain boundaries.[25]

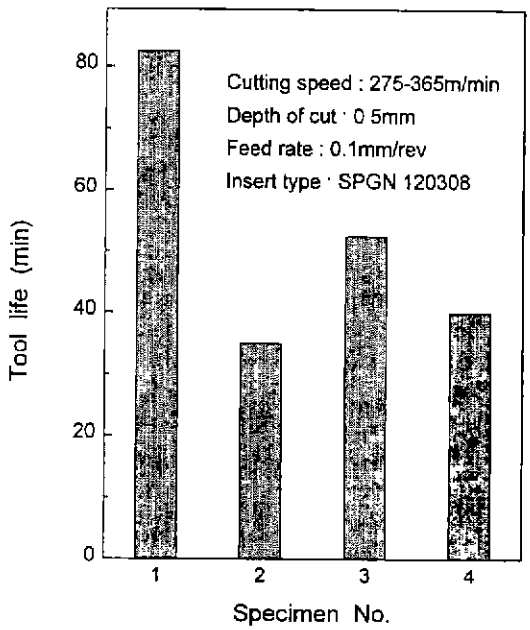

Fig. 5. Comparison of tool lives at flank wear 250 μm during turning test of an Al-17%Si alloy with diamond-coated tool specimens.

IV. Conclusion

Thin film diamond-coated tools were studied with the expectation of high performance in cutting of Al-17%Si alloy. The diamond films were deposited onto differently pretreated C-2 cemented carbides under an CH_4-H_2 atmosphere in a microwave CVD system.

An effective pretreatment of the substrate surfaces could be achieved by using Murakami's reagent for 1 min and then the $1H_2SO_4$-$9H_2O_2$ solution for 5 min. The deposited diamond crystallites were grown in a cubo-octahedral shape and dominated by (111) and (220) surfaces. The film morphology and the diamond quality were hardly affected by the surface conditions of the substrate at the region of experimental parameters, but the cutting performance of the coated tools was markedly improved with the Co depletion of 16 μm from the effectively pretreated surface.

Tool life over substrate etching time varied with a compromise between the increase of film adhesion at the interface and the decrease of toughness at the substrate surface. The tool failure was caused by chipping and flaking of the diamond film, compared with the gradual wear of conventional PCD tools.

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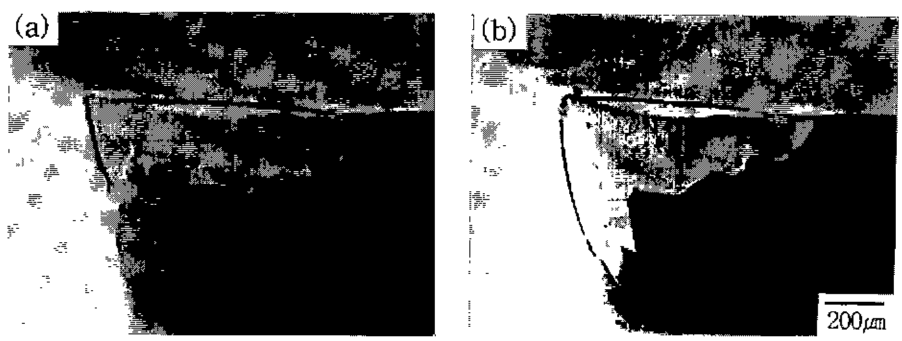

Fig. 6. Damage to tool edges after cutting test for (a) 115 min with specimen 1 and (b) 40 min with specimen 2.

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