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Sandblasting pretreatment for deposition of diamond films on WC-Co hard metal substrates

Xiaotian Shen, Xinchang Wang, Fanghong Sun *, Chaoyue Ding

School of Mechanical Engineering, Shanghai Jiao Tong University, Dongchuan Road 800, Minhang District, Shanghai 200240, China

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ABSTRACT

Sandblasting combined with acid etching was utilized as pretreatment of Co-cemented tungsten carbide substrates for the deposition of chemical vapor deposition (CVD) diamond films. Diamond films were deposited on the pretreated surface as well as on substrates treated by a two-step alkali-acid pretreatment as a contrast. The field emission scanning electron microscopy (FESEM), X-ray diffraction (XRD), Raman spectroscopy, Rockwell hardness tester and turning test were respectively conducted to characterize the pretreated substrates and deposited diamond films. Surface roughness of the treated surface was controlled by adjusting sandblasting parameters. An enhancement of surface roughness and removal of the binder phase were detected on the treated surface. The porous Co-depleted carbide layer caused by sandblasting and acid-etching pretreatment was obviously thinner compared to the two-step pretreatment. The nucleation stage of CVD process was investigated and the nucleation density was obviously enhanced by the sandblasting pretreatment. Indentation tests exhibited an improvement of adhesive strength compared to the two-step pretreatment. Moreover, the XRD patterns showed that the residual stress of diamond films was decreased. The turning tests showed that the diamond coated tools with sandblasting-acid pretreatment exhibited less area of flank wear and film shedding.

1. Introduction

Co-cemented tungsten carbides are widely used in the machining and petrochemical industry. Owing to the outstanding characteristics of chemical vapor deposition (CVD) diamond films, including low thermal expansion, low friction, good thermal conductivity, excellent wear resistance and high hardness, the deposition of CVD diamond coatings on WC-Co cemented carbides is a highly effective procedure that can improve their cutting performances as well as their lifespan [1], and large attention has been paid in recent studies [2–5]. Yet, the applications of CVD diamond coatings on Co-cemented tungsten carbides are affected by several drawbacks which would lead to the failure of CVD diamond coatings. Due to the high temperature of CVD process, the outermost layer of the Co matrix of the WC-Co substrates can catalyze the formation of graphite, leading to the formation of sp^2-carbon layer and low film-substrate adhesion [6–9]. Besides, the mismatch of thermal expansion coefficient between diamond coatings and WC-Co substrates leads to high residual stress which further compromises the lifespan of diamond coatings [10].

Various pretreatment methods have been reported to avoid the graphitization effect of Co binder on the deposition of CVD diamond films, including chemical etching, interlayer diffusion barriers [11,12], thermal treatments [13], etc. Multi-step chemical etching using Murakami's reagent and acid is a wide-spread pretreatment to remove the Co binder from substrates [14]. However, the etching of Co can result in tiny holes in the outermost layers and the holes still exist after deposition [15]. Consequently, mechanical properties and toughness of the substrate surface are compromised.

Rough and corrugated WC-Co surface with high defect density is widely believed to contribute to the enhancement of nucleation density in CVD process as well as adhesive strength by mechanical interlocking [16]. Thus, mechanical treatments to scratch or corrupt substrate surface such as mechanical peening and sandblasting are conducted to pretreat the WC-Co substrates in order to modify the nucleation and adhesion. F. Deuerler et al. reported a complex pretreatment method including sandblasting, two-step pretreatment, shot peening process with hard particles (SiO_2 or Al_2O_3)/ultrasonically induced cavitation, and nanostructured diamond coatings with modified adhesion were deposited on the pretreated cemented carbide [17]. R. Polini et al. adopted fluidized bed treatment after acid etching as pretreatment and diamond films with high nucleation density were deposited. Moreover, the film-substrate adhesion proved to be very close to that of the two-step pretreatment [18]. Z. Xu et al. adopted short peened Cr/CrN/Cr multi-interlayer coated WC-Co as substrates to deposit CVD diamond films [19], and results showed that mechanical peening with diamond powders on interlays can also scratch and roughen the surface of interlays and enhance the nucleation density during the deposition of CVD diamond, thus promoting the film-substrate adhesion. Nonetheless,

* Corresponding author.
E-mail addresses: 742941666@qq.com (X. Shen), wangxinchangz@163.com (X. Wang), sunfanghong@sjtu.edu.cn (F. Sun), 806187385@qq.com (C. Ding).

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Table 1
Deposition parameters of diamond films.

	Nucleation	Growth
Filament temperature/°C	2000–2200	2000–2200
Substrate temperature/°C	800–900	800–900
H_2 flow rate/sccm	100	80
V(Acetone)/V(H_2)/%	1.2	1
Chamber pressure/kPa	1.6	4
Bias current/A	5	3
Duration/h	0.5	8.5

Table 2
Sandblasting parameters of the samples.

Sample	Nozzle-substrate distance/mm	Air pressure/MPa	Duration of sandblasting/s	Surface roughness μ/σ/μm
MC	–	–	–	0.173/0.015
S1	100	0.1	90	0.192/0.021
S2	75	0.3	30	0.242/0.008
S3	75	0.2	90	0.287/0.011
S4	50	0.2	60	0.343/0.021

diamond nucleation on cermet interlayers such as TiN/TiCN are relatively rare and slow, leading to low growth rate and poor adhesion of the diamond films. Besides, thick interlays may result in the dulling of cutting edges of diamond coated tools.

Sandblasting is a safe, low-cost technology with geometric simplicity and working stability, which is suitable for commercial pretreatment in the CVD process. In the present work, sandblasting followed by acid etching was utilized as pretreatment of the WC-Co substrates, and microcrystalline diamond films were deposited on such substrates by the CVD method. FESEM and EDX were adopted to characterize the pretreated WC-Co surface. SEM, XRD, Raman spectroscopy, and Rockwell hardness tester were adopted to characterize the morphology and adhesion of the as-fabricated diamond films. Turning tests were conducted to investigate the cutting performance of diamond coated inserts with sandblasting-acid pretreatment.

2. Experimental

2.1. Pretreatment of WC-Co substrates

WC-6 wt.% Co cuboid samples (13 mm × 13 mm × 3 mm) were adopted as substrates and cleaned in ultrasonic bath with deionized water and acetone separately to remove contaminations on the substrates. Prior to the acid etching, all of the samples were submitted to a sandblasting process. Thus, the surface of substrates was roughened for the sake of removing Co binder efficiently in the following acid etching process and enhancing nucleation density during CVD deposition [17]. The sandblasting pretreatment on WC-Co substrates was conducted on an air-sand blasting rig. Angular SiC sands of 320 mesh (~45 μm) were chosen to impact the surface of the WC-Co substrates. The sands were carried by compressed air in pipelines and impacted the samples through a nozzle. Samples were fixed perpendicularly to the nozzle by a clamp. The scattering angle of the nozzle was about 6° which meant most of the SiC sands would impact the substrates perpendicularly. The pressure of compressed air could be adjusted at the range of 0–0.5 MPa and nozzle-substrate distance could be fixed at 5–100 mm. Several samples were treated with different blasting parameters in order to control the surface roughness of the samples. As there might be remaining SiC particles lodged into the blasted substrate which could affect the deposition of diamond films, the blasted substrates were rinsed by deionized water and cleaned in an acetone ultrasonic bath for 10 min to remove remaining SiC sands as well as WC-Co fragments contributed to the sandblasting process. EDX was adopted to identify the washed surface. After that, samples were etched by Caro's acid solution (3 mL 96 wt.% H2SO4 + 88 mL 40% w/v H2O2) for 30 s to remove the Co phase from the outermost layers.

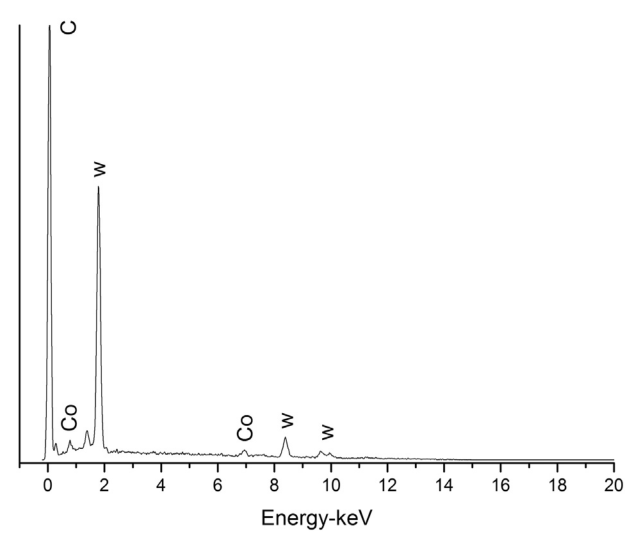

Fig. 1. The EDX pattern of the substrate surface treated by sandblasting and washing.

As a comparison to the sandblasting-acid pretreatment in this paper, several WC-Co substrates denoted as MC samples, were treated by a two-step pretreatment, including ultrasonic treatment in Murakami's reagent ([10 g $K_3Fe(CN)_6$] + 10 g KOH + 100 mL water) for 30 min and Caro's acid solution for 30 s. The FESEM (Zeiss ULTRA55) and EDX (D8 ADVANCED) were adopted to characterize the surface of WC-Co substrates after acid etching process.

2.2. Deposition and characterization of diamond films

In this work, the hot filament CVD was adopted due to its geometric simplification and large deposition area. All the samples were deposited in the same deposition process in order to avoid the imprecise control of other variable deposition parameters, so any change of the properties of as-fabricated diamond films was considered to be induced by sandblasting process. Six tantalum wires (Φ 0.5 mm) with an interval of 35 mm were chosen as hot filaments, all of which were dragged to be straight and fixed 10 mm above the substrates. A positive bias current was applied from filament to substrates for the sake of enhancing the growth rate of diamond films [20,21]. Acetone and hydrogen gas were adopted as source materials. Acetone was used as carbon resource mainly for two reasons: The growth rate of diamond films adopting acetone as carbon resource is higher than that of other common carbon sources such as methane, methanol [22]; It would be convenient to fabricate B/Si-doping diamond films in further work by mixing acetone with trimethyl borate/tetraethyl orthosilicate. The detailed deposition parameters of diamond films were listed in Table 1. As-deposited diamond films were characterized by FESEM, Raman spectroscopy and X-ray diffraction (XRD). The Hoytom Rockwell hardness tester with a diamond indenter (angle = 120°, radius = 0.2 mm) was adopted to obtain their indentation morphologies under a 1000 N load and accordingly ranks of their film-substrate adhesion were estimated.

2.3. Turning test

To evaluate the cutting performance of the diamond films deposited on the pretreated surface, diamond coated WC-Co inserts were adopted to turning Al-15 wt.% Si alloys with bar capacity of 70 mm. The cutting conditions for all the experiments were cutting velocity, 470 m/min; feed rate, 0.1 mm/rev; and depth of cut, 0.5 mm. The scars that appeared on the cutting edges were examined with optical microscope.