Novel method for adherent diamond coatings on cemented carbide substrates

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Abstract

Adhesion of diamond films on cemented carbide substrates is a critical issue due to its large thermal expansion mismatch. This large difference in thermal expansion results in the generation of very high stresses in the coating that may lead to delamination, cracking, or other deleterious effects. A method to increase the adherence of diamond coatings on cemented carbide substrates is reported, based on a substrate-modification process that creates a three-dimensional thermally and compositionally graded interface. Indentation tests on diamond coated tungsten carbide substrate showed that the adhesion of diamond films significantly improved with increasing the surface roughness of cemented carbides. © 1998 Elsevier Science S.A.

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1. Introduction

Diamond is an attractive tool material for machining abrasive materials such as Al–Si alloys due to its high hardness and thermal conductivity, combined with a low coefficient of friction [1]. Diamond films have been grown on cutting tool inserts of cemented carbides and silicon nitride using low-pressure chemical-vapor deposition methods [2–5]. Conventional polycrystalline diamond (PCD) tools usually have a single tip as well as a limitation in geometrical styles of inserts. These aspects, combined with the high manufacturing costs, make the use of PCD tools an expensive choice. Diamond coated tools (DCT) offer a high quality, low cost alternative because DCTs have the advantages of using multiple tips and a flexible design of inserts with complex geometries [6].

At present, one of the critical barriers to be overcome for widespread application of this technology is the poor adhesion of a diamond film to a cemented carbide substrate (WC–Co). During tooling operations, the diamond film spalls due to poor interfacial adhesion with the substrate. The poor adhesion of diamond films is primarily due to large thermal expansion mismatch, and the formation of a graphite film between the substrate and the thin film at the WC(CO)/diamond interface [7–10].

Several methods have been adopted to enhance the adhesion of the diamond films on WC(0) substrates. These methods can be divided into two categories: removal of cobalt at the diamond–carbide interface using Co etching agents [11–13] and the deposition of an interlayer as a diffusion barrier [14,15]. In this paper, we have investigated a novel method to improve the adhesion of diamond films on WC/Co substrates. This method is based on morphological surface modification of the WC/Co substrate prior to etching and chemical-vapor deposition of diamond. The morphological surface modification results in the formation of a thermal and compositionally graded three-dimensional interface which leads to enhanced adherence [17].

2. Experimental

Cemented carbide inserts (SPG422, K313, Kennametal) having tungsten carbide particles of 1 μm with 6% cobalt binder were used as substrate materials. The surfaces of the cutting tool inserts were modified using a repetitive pulsed laser irradiation process ($\lambda = 248$ nm, $\tau = 25$ ns) [17]. Incident laser energies were varied from 1.7 to 7.6 J/cm$^2$ and the number of pulses from 90 to 800 were employed to control the morpholog-
tical roughness of the substrate. The error in the energy density measurement was within ±10%. The controlled laser irradiation resulted in the formation of a smooth undulating hill-valley structure on the substrate surface. The details of this surface modification process have been published elsewhere [16,17]. In this study, inserts of four different roughness were studied with peak-to-valley roughness ($R_a$) varying from 0.5 to 3.5 μm. Following the surface modification, the tool inserts were etched by Murakami agent: ($K_2$Fe(CN)$_6$·NaOH:H$_2$O=1:1:10 by weight) for 2 min to remove non-stoichiometric WC and provide further micro-roughness. This was followed by nitric acid treatment for 1 h to remove graphite formation at the diamond-carbide interface.

After surface pre-treatments, the carbide inserts were sonicated in a diamond suspension for 1 h to enhance diamond nucleation. Diamond was deposited by the hot filament chemical-vapor deposition system (HFCVD) process using 1% methane in hydrogen at a total pressure of 20 Torr with a flow rate of 200 sccm for 20 h [18]. Substrate and filament temperature were maintained at 960 and 2100 °C, respectively. The surface roughness of the tool inserts was measured with a Dektak surface profilometer having a mechanical stylus. Indentation was carried out to qualitatively analyze the diamond adhesion on the substrate.

3. Results and discussion

The surface morphology and roughness of cemented carbide tool inserts depend on the laser fluence and the number of laser pulses. The surface morphology of the laser-modified cemented carbides as a function of laser pulses is shown in Fig. 1. At 1.7 J/cm$^2$ with 90 pulses (Fig. 1a), surface features, like ripples due to laser etching processes, begin to develop. As the number of pulses increased up to 800, surface features continue to etch with a high degree of definition such as cone shapes. No further changes in features were obtained with increasing number of pulses. These structures have been speculated to develop because of non-uniform spatial etching at the surface within a laser energy windows [16,19]. Fig. 2 shows the roughness change as a function of laser pulse at fluence of approximately 1.7 J/cm$^2$. As the number of pulses and fluence of the laser increased, the surface roughness of cemented carbides increased.

Fig. 1. Surface morphology changes of WC–CO after laser irradiation at 1.7 J/cm$^2$ with different number of laser pulses. (a) 90 pulses; (b) 270 pulses; (c) 400 pulses; and (d) 800 pulses.
non-linearly. Thus, by varying the number of pulses, the average roughness of the cemented carbide inserts can be controlled. Fig. 3 shows XRD patterns of cemented carbide surfaces after laser irradiation at a fluence of 1.7 J/cm² with different number of pulses. The as-received sample shows only tungsten carbide and cobalt peaks (Fig. 3a). After laser irradiation with 90 pulses in Fig. 3b, a WC₁₋ₓ (x = 0–0.3) phase was observed. With an increase of the number of pulses, the formation of z-W₂C phase peak was observed. From these phase changes, we speculate that carbon in a cemented carbide reacted with oxygen in air to form volatile gases such as CO or CO₂ during the melting/ablation process on the WC/Co surface by laser irradiation, resulting in non-stoichiometric carbide phases. As the formation of non-stoichiometric carbon-deficient tungsten carbide phases is deleterious to tool performance, the modified surface was etched by a Murakami agent for 2 min.

Fig. 4 shows surface morphology of diamond films deposited on the modified cemented carbides with different surface roughness. The image of surfaces after indentation with 100 kg are also shown in this figure. The figure shows that as the modified surface roughness of cemented carbides was increased, the diamond films deposited on cemented carbides by HFCVD were also roughened as shown in Fig. 4a–c. Adhesion tests for each group of diamond coated cemented carbide samples were performed using a Rockwell C scale diamond sphero-conical indenter having a 120° tip angle at 100 kg as referred to in Jindal et al. [20]. During the indentation, the fracture of diamond films occur, leading to the initiation and propagation of lateral cracks along the film-substrate interface. The diameter of the lateral crack has been correlated to the adhesion strength of the film. It has been shown that films with poorer adherence exhibit larger crack diameters [20]. Depending upon the interfacial strengths, the delamination (lateral crack) diameters of a film are varied. The delamination diameter of diamond films deposited on the as-received cemented carbides was largest as shown in Fig. 4d. As the roughness of diamond-cemented carbide interfaces increased, the delamination diameter significantly decreased, which suggests the formation of adherent interfaces. In the case of Rₐ = 3.5 μm, no delamination of the diamond film was observed for an indent load of 100 kg. Thus these results clearly show that the laser morphological surface modification process results in improved adhesion of the diamond film on the cemented carbide substrate.

In conclusion, we have shown that an unique laser-based morphological surface modification process resulted in improved adhesion of the diamond film on the cemented carbide substrate. Indentation results showed conclusively that adhesion of the diamond films increased as the surface roughness of cemented carbides was increased.

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Fig. 4. Surface morphology of diamond films deposited on tungsten carbides having (a),(d) $R_t = 0.5 \ \mu m$ (as-received, nitric acid etching for 2 hrs); (b),(e) $R_t = 1.5 \ \mu m$; and (c),(f) $R_t = 2.5 \ \mu m$ before and after indentation with 100 kg.

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References