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Friction measurements on hot filament CVD diamond films deposited on etched tungsten carbide surfaces

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Abstract

Diamond-coated cemented carbides are a promising candidate for cutting tools, but a critical problem is the poor adhesion strength between the coating and the substrate. We have grown diamond films on WC–6%Co and WC–10%Co using hot filament CVD. Acid etching was necessary to remove Co from the substrate surfaces, and Co was more easily removed from the 10% Co sample. Scratch testing with a diamond stylus gave friction coefficients of 0.4–0.6. The standard deviation in the coefficient of friction during a pass was used as a measure of the adhesion quality when comparing films of similar morphology.

Keywords: Friction; Diamond films; Heated filament CVD; Adhesion

1. Introduction

The recent development of CVD methods for the production of diamond films has opened the way for a variety of engineering applications. The high strength and hardness, coupled with the low coefficient of friction, make diamond an attractive proposition for coatings on cutting tools for advanced non-ferrous alloys [1–5]. Tungsten carbide is a possible substrate for such tools, but the poor adhesion strength of the films is a critical problem [2]. We have used scratch testing to examine adhesion problems and the effect of surface morphology on the coefficient of friction. (The coefficient of friction has been linked to the cutting characteristics [3] and is an easy test to perform on relatively small samples.)

2. Experimental details

The substrates used were specimens of tungsten carbide (10 mm × 10 mm; thickness, 1 mm) with either 6% Co or 10% Co, which had been surface ground with diamond wheels (grit size, 90–106 μm) and mirror polished using 1 μm diamond paste. The samples were etched in concentrated nitric acid for 10 min (10% Co substrates) or 15 min (6% Co substrates) to remove Co from the surface. Just before deposition, the samples were abraded with 1–3 μm powder.

A hot filament CVD reactor (Ta filament at 2000 °C) was used to deposit the diamond film on the tungsten carbide substrate (substrate to filament distance, 5–6 mm). The gas mixture used was 1% methane in hydrogen, and the chamber pressure was kept constant at 30 Torr. The substrate temperature was in the range 800–900 °C. Diamond growth rates were typically 0.5 mm h^{-1} on abraded and etched samples.

The diamond coatings were characterized by scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX). X-Ray diffraction (XRD) analysis was carried out on a Rigaku Geigerflex machine with 2θ values between 0° and 80°. The coefficient of friction and adhesion were investigated using a purpose built scratch tester [6]. A 120° conical diamond stylus (tip radius, 200 μm) was loaded at the end of an aluminium beam load, using a spring and micrometer arrangement. Loading and sideways forces were measured with strain gauges on the beam, whilst the sample was moved sideways underneath the stylus. The sample movement rate was 0.15 mm s^{-1}, and four passes along the same track (two in each direction) were made at each load used (25 and 100 gf). Each pass was 5–7 mm long and thus yielded 250–300 data points (which were themselves each the average of 100 data readings). In order to eliminate frictional changes caused by starting and stopping the scratch, only the central 80% of the data was used to calculate the mean coefficient of friction for each pass.

3. Results and discussion

3.1. Effect of Co content

In common with other workers [2,3,5], we found that the Co binder in the tungsten carbide samples inhibited the diamond nucleation rate, and it was necessary to etch out Co from the substrate surface to obtain satisfactory diamond films. Continuous adherent films were produced by 4 h deposition on acid etched and abraded samples. Previous work on our substrates has shown that Co is more easily removed from the 10% Co samples than the 6% Co samples because, in the former case, the Co is concentrated as large pools between the WC multiple grain junctions [7]. Hence it is more difficult to etch out the Co from the 6% Co samples and a longer etching time was used. Even so, Co particles were present on the films grown on the 6% Co samples (Fig. 1(a)). Some of the thicker films (6 h or more deposition) on the 6% Co samples spontaneously delaminated. Small Co particles could be seen on both sides of the delaminated films, and it seems likely that poor film adhesion is linked to the presence of these particles. The delamination occurred by a mixture of interfacial failure and cohesive failure in the etched substrate layer, since EDX of the underside of the film showed tungsten carbide grains to be present (Fig. 1(b)).

3.2. Texture of films

We compared the XRD peak counts of five films with the XRD peak intensities of isotropic standards (see method in Ref. [8]). The analysis showed a substantial diamond {111} peak, but no detectable diamond {220}

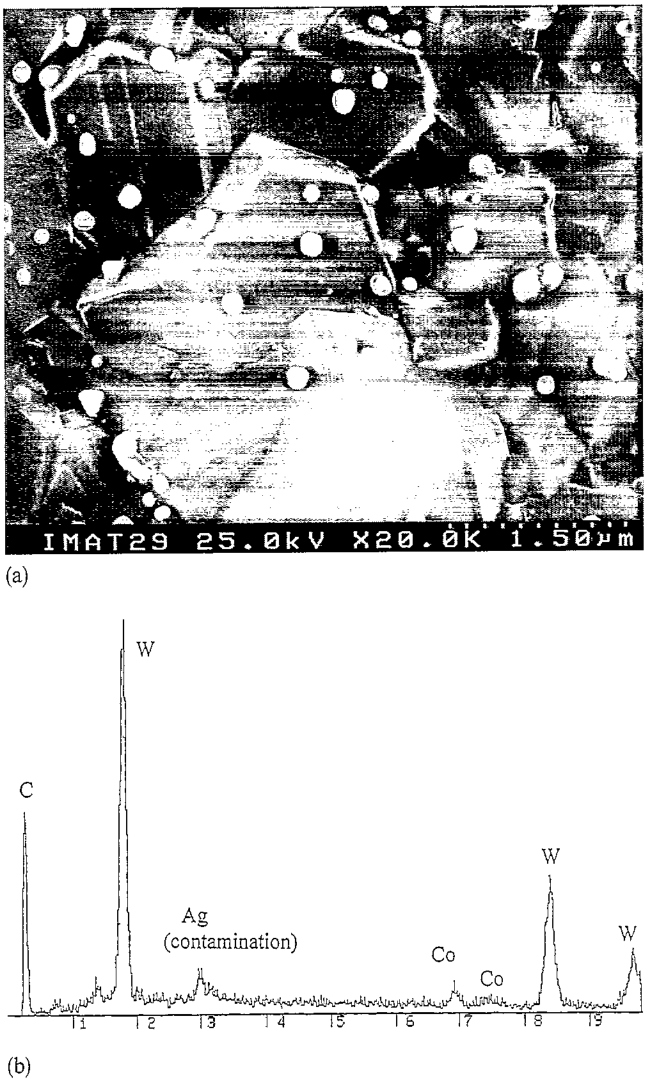


Fig. 1. Diamond film produced by 6 h deposition on an acid etched and abraded WC–6%Co substrate: (a) Co particles on the top of a film; (b) EDX analysis of WC area of the underside of a delaminated film.

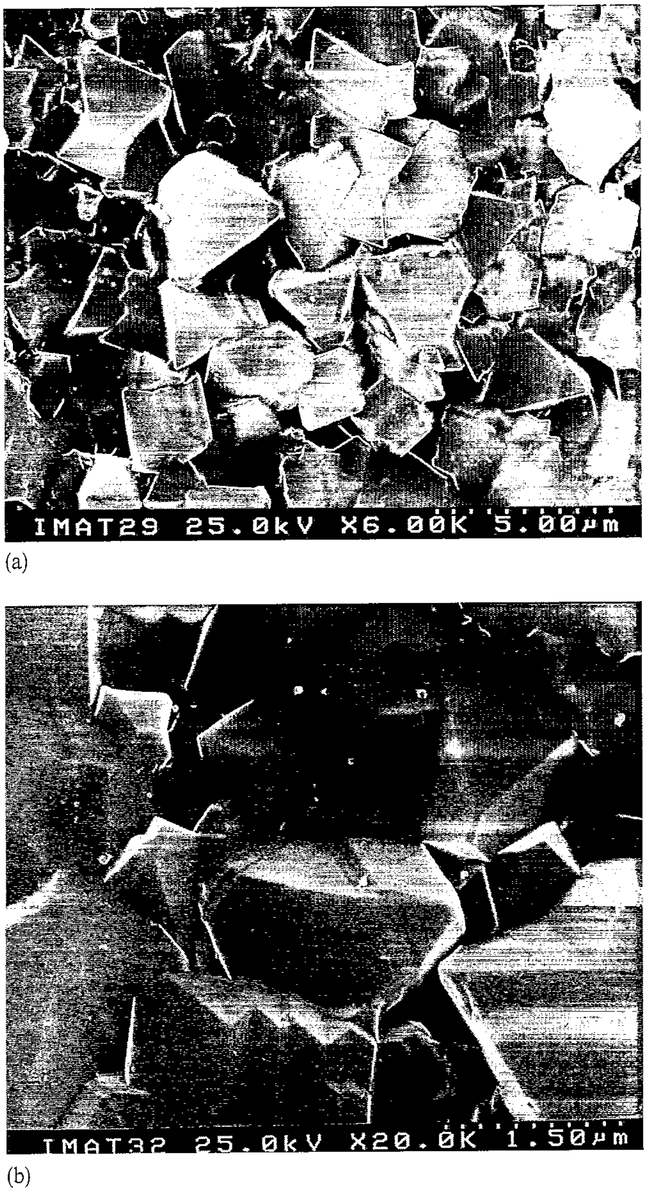


Fig. 2. Diamond film produced by 8 h deposition on an acid etched and abraded WC–10%Co substrate: (a) low magnification illustrating {111} facets; (b) high magnification illustrating multiple twinning.

peak above the WC {200} peak at the similar wavelength. We conclude that the diamond films are predominantly of {111} texture. The majority of surface grains appear to be in the {111} orientation, i.e. triangular facets uppermost in the plane of the film (Fig. 2(a)). All the films also showed multiple twinning (Fig. 2(b)).

3.3. Scratch testing and coefficient of friction

Table 1 shows a summary of the scratch testing results. Each quoted result is the average of four passes unless otherwise stated. Fig. 3 shows representative results of the scratch test behaviour of diamond films on each type of substrate.

Table 1
Summary of scratch testing results on diamond film

Amount of Co in bulk substrate (%)	Time of diamond deposition (h)	Average coefficient of friction at 25 gf load, and the mean standard deviation for a pass		Average coefficient of friction at 100 gf load, and the mean standard deviation for a pass	
6	6	0.44[a]	0.012[a]	—	—
6	8	0.46[a]	0.017[a]	0.57	0.10
10	6	0.53	0.010	0.53	0.009
10	8	0.42	0.007	0.49	0.005
10	Etched and abraded, but uncoated, WC–Co substrate	0.24	0.001	0.26	0.001

[a] Only three passes recorded, instead of four.

The coefficients of friction of our films are in the region of 0.4–0.6. This is higher than that for diamond on natural diamond (about 0.1 in air, but dependent on the orientation [9]), but consistent with the results of previous work on hot filament films [10–12]. A higher surface roughness tends to lead to higher coefficients of friction [13]. Thus the roughness of the film morphology is the most probable reason for the coefficients of friction which are comparatively high compared with smoother morphologies [10] or polished films [11,12]. The lower coefficients of friction for the 8 h deposition on the 10% Co substrate, compared with the 6 h film on the 10% Co substrate, suggests that a longer deposition time gives a smoother surface. Scratch testing with the 100 gf load gave a larger value for the coefficient of friction than testing with the 25 gf load in all but one case.

During tests on the 6 h film on WC–6%Co an audible "cracking" sound could be heard, which was probably related to the delamination of the film during the scratch test, since visible delamination could be seen after the test. It seems likely that the low coefficient of friction recorded is influenced by the stylus going right through the film and contacting the substrate underneath. From the large "spikes" shown in the trace in Fig. 3(a), it seems probable that delamination also occurred during the 25 gf test on the 8 h film. Thus, for films with poor adhesion, the coefficient of friction may be misleading because of delamination.

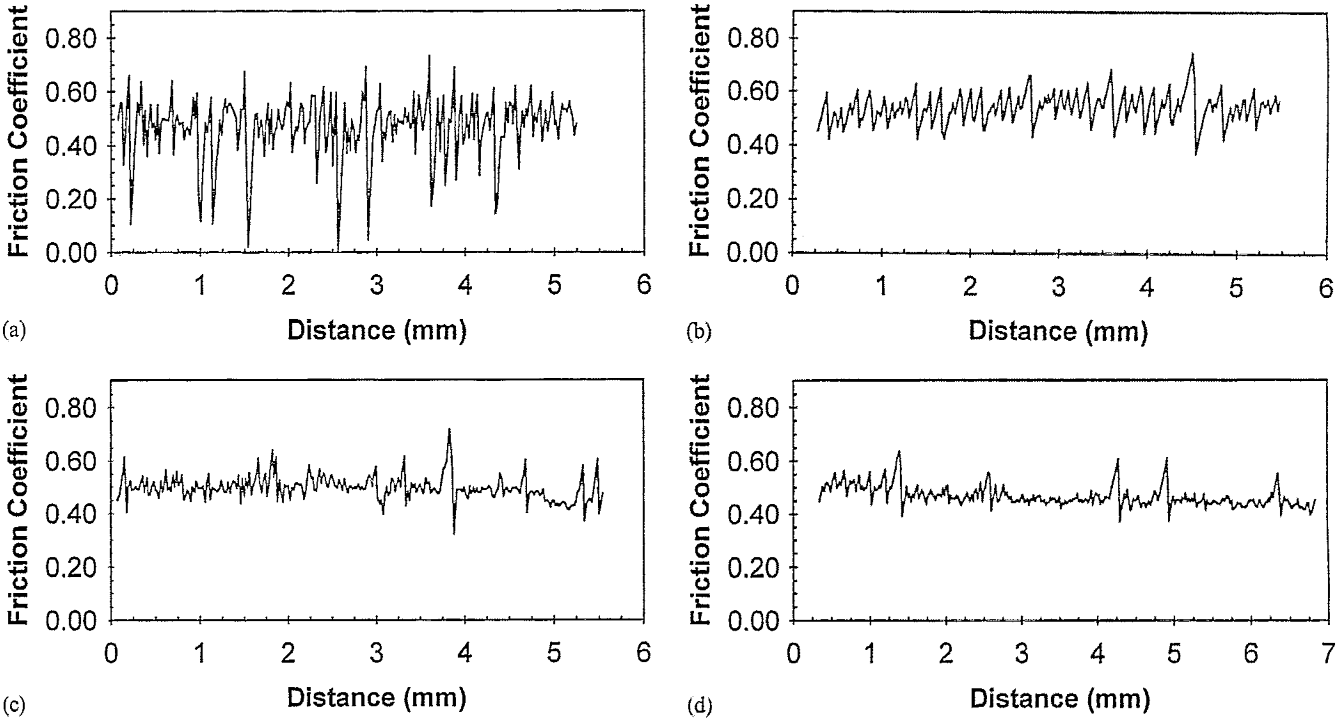


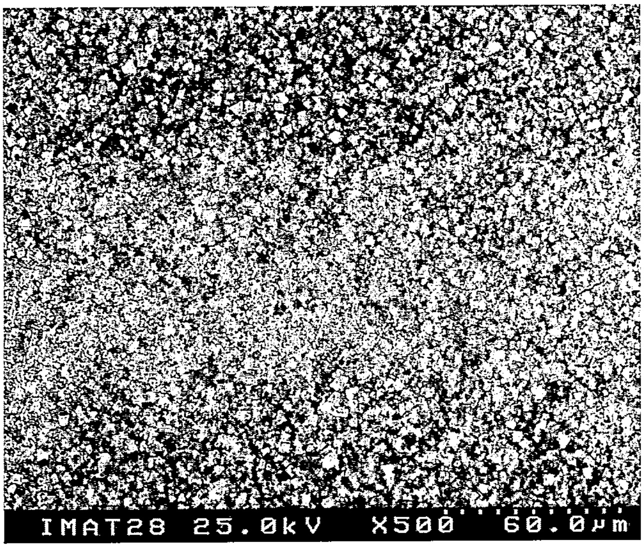
Fig. 3. Variation of the friction coefficient along the path of a scratch test on diamond films: (a) 8 h deposition on WC–6%Co, 25 gf load; (b) 8 h deposition on WC–6%Co, 100 gf load; (c) 8 h deposition on WC–10%Co, 25 gf load; (d) 8 h deposition on WC–10%Co, 100 gf load.

Another possible cause of discrepancy in the recorded friction coefficients is stylus wear. We found that the diamond stylus showed significant wear after a number of tests, particularly at higher loads. A seriously worn tip gave a different coefficient of friction, normally higher. However, the stylus tip was checked and renewed as necessary throughout this experimental work.

An interesting value is the standard deviation of the friction coefficient along a single pass. Difference of variance tests between the standard deviations of the friction coefficients for films of the same deposition time, scratch tested at the same load, on either WC–6%Co or WC–10%Co substrates showed that the standard deviations for the different substrates were different (to a 98% confidence limit). We therefore suggest that the standard deviation of the friction coefficient during a pass can be used as a comparative adhesion quality parameter when assessing diamond films of similar morphology.

For the films on WC–10%Co, scratch testing at 25 gf and 100 gf did not produce delamination. At higher loads, the scratches were just visible by a damage path in which the tips of diamond grains broke off and wear debris was generated, but grain pull out was not observed (Fig. 4).

As well as the Co content, previous work [3] has shown that the surface roughness of the substrate is important for the adhesion of diamond films on cemented carbide surfaces, and it has been suggested that there exists an appropriate range of substrate roughness for an optimum adhesion strength. Our substrate samples were originally polished and, even after acid etch and abrasion with fine diamond powder, are relatively smooth (see Table 1 for the coefficients of friction). This will have a deleterious effect on the adhesion strength of the diamond [5]; we would expect the adhesion on non-polished cutting inserts to be even better than achieved on our samples since additional mechanical bonding is provided.



(a)

(b)

Fig. 4. Scratch test on diamond film produced by 6 h deposition on an acid etched and abraded WC–6%Co substrate: (a) low magnification of scratch path (from left to right across the centre of the image); (b) higher magnification of abraded path region.

4. Conclusions

Diamond films were successfully deposited on etched and abraded cemented carbide using the hot filament CVD technique. The acid pre-etch was necessary to remove Co from the surface of the substrates, because it causes poor adhesion (and nucleation) of the diamond film. Of the two types of substrate used, WC–6%Co and WC–10%Co, it was easier to remove the Co from the 10% samples, so films on these substrates showed better adhesion. Work is continuing on the coating of cutting inserts which have a rougher surface finish than the polished samples reported here, and this should produce even better adhesion through mechanical coupling.

The diamond films had a predominantly {111} texture and gave friction coefficients of 0.42–0.57 when scratch tested with a diamond stylus and 25 or 100 gf load. The standard deviation in the friction coefficient can be used as a measure of the film adhesion if films of similar morphology are being compared.

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