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Article Title *: Removal and fracture characteristics of cemented tungsten carbide under nanoindenting and
Removal and Fracture Characteristics of Cemented Tungsten Carbide under Nanoindenting and Nanoscratching

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Keywords: Grinding; Tungsten carbide; Elastic modulus; Hardness; Removal mechanism

Abstract: Nanoindenting and nanoscratching were used to investigate removal and fracture characteristics of cemented tungsten carbide (cWC). Nanoindentation results indicated that the elastic modulus and hardness of WC grains were significantly greater than those measured in cobalt binder rich regions, respectively. Few evidences of cracking or fracture were observed on the indented surfaces using both in-situ atomic force microscopy and scanning electron microscopy. However, the pop-in events were observed from indenting load-displacement curves and the corresponding acoustic emissions were detected, indicating the occurrences of brittle fracture. Nanoscratch results demonstrated that similar removal characteristics existed, but cracking was observed in both surface and subsurface of the scratched samples.

Introduction

Cemented tungsten carbide (cWC) is a material of great technological interest due to its excellent mechanical properties such as high hardness, excellent corrosion resistance, chemical stability and attractive high temperature wear resistance. Recently, cWC with fine microstructure is commonly used for making glass moulds or mould inserts. The fabrication of such products is usually via nanogrinding [1].

Nanogrinding is a process that has a material removal unit at nanometric scale and is capable to machine components with feature sizes ranging from several to several hundreds of microns. Compare to other mechanical machining methods, grinding has unique ability to machine hard and brittle materials [1-3]. The machined components are of high dimensional accuracy and high surface and subsurface integrity. To successfully develop a nanogrinding process for cWC, it is essential to gain a comprehensive understanding of the interaction mechanism of abrasives with the work material involved. This requires systematic investigations of the mechanical properties of the material and the effect of its microstructure on material deformation and removal under the nanogrinding conditions [4,5]. However, these investigations are extremely challenging due to the complex nature of the grinding.

The penetration of a diamond abrasive into the work material in the nanogrinding of brittle materials can be envisaged as a nanoindenting process, which has been extensively used as a method to study the mechanical properties of those materials with fine microstructure where highly localized properties need to be measured [6-11]. Nanoscratching appears to be a better simulation of a grinding event as it is closely analogous to a single-grit scratching in a grinding process, but with parameters, including load, velocity, and depth of penetration, under controlled [12-18]. Nanoscratching was also used to investigate fundamental micro-fracture in brittle materials.

This study investigated the material removal and fracture characteristic of cWC, by the use of nanoindenting and nanoscratching. Hardness and elastic modulus of WC grains and binder areas were measured. Topographies of impressions and subsurface of the scratches were examined using in-situ atomic force microscopy (AFM) and scanning electron microscopy (SEM). Fundamental removal mechanisms were discussed with respect to the microstructure of the material.
Cemented tungsten carbide with fine microstructure (supplied by Toshiba Tangaloy, Kawasaki, Japan) was the work material in this study. Cemented tungsten carbide, also named hard metal, is a material made by cementing hard tungsten mono-carbide (WC) grains in a binder matrix of cobalt metal by liquid phase sintering. The nominal elastic modulus (E) of the bulk material used in this study is 560 GPa [19]. Hardness (H) for the bulk material measured using a Mitutoyo microhardness tester, is 18.1 GPa. The specimens were lapped using diamond films of grit sizes ranged from 30 to 0.5 μm, then followed by the final polishing using silica suspension with particle size of 0.1 μm on a soft pad. The microstructure of polished specimen was examined using SEM (JEOL 6300LA).

Nanoindenting experiments were conducted using a Hysitron Triboindenter®. A Berkovich indenter with a tip radius of 100 nm was used to measure mechanical properties of the specimen. The indentation loads were varied from 2 to 30 mN. Nanoindenting was purposely made either on the grain or binder rich areas. AFM was used to examine the surface topographies prior to and after indenting. Determination of E and H was undertaken from the initial part of the unloading segment of load-displacement curve. Pop-in event, which is associated with acoustic emission (AE) occurrence, was investigated using an AE transducer embedded with a 90° cube corner indenter tip with radius of 50 nm.

Nanoscratching experiments were performed on the Triboindenter®, on the same specimen for nanoindenting. A cone-shaped conical indenter with the tip radius of 3 μm and include angle of 120° was used. The scratching experiments were carried out at linear velocity of up to 1 μm/s with a length of 12 μm. The normal load remained as constant during scratching, which varied from 1 to 5 mN. The coefficient of friction, which is defined as the ratio of lateral force over normal force involved in nanoscratching, was measured. Top and cross-sectional surfaces of the scratches were examined using SEM (JEOL 6300LA). For the cross-sectional surface examination, a bonded-interface sectioning technique [13] was used.

Results and Discussion

Microstructure. A typical SEM image of microstructure of cWC is shown in Figure 1. The microstructure consists of WC grains (grey phase) and cobalt binder (black phase). The WC grains are randomly distributed within the matrix of binder, exhibiting to have nearly triangle, rectangular and trapezoid shapes with the average grain size of 0.32 μm [17-18].

![Figure 1. A typical SEM image showing the microstructure of cWC.](image)

Indented Surface. Indentations were made on a WC grain, as shown in Figure 2. A greater indentation load used resulted in a larger impression. There were no obvious cracks observed from the AFM and SEM examinations, for the loads up to 30 mN.
Figure 2. AFM images of surfaces (a) prior to and (b) after indentation at load of 2 mN, (c) prior to and (d) after indentation at 6 mN.

**Mechanical Properties.** Averages values of elastic modulus ($E$) and hardness ($H$) obtained from the nanoindenting tests are listed in Table 1. The results suggest that $E$ of the WC grains is significantly higher than that measured in the binder-rich region. As mentioned previously, the cemented WC consist of WC grains as the stronger phase in the microstructure, and cobalt binders as the weaker phase. Therefore, it is expected that the $E$ of the bulk material should be smaller than $E$ of the WC grains, but greater than that of the binder-rich region. $H$ values of the WC grains are greater than that measured in binder-rich region too. However, the $H$ values measured in binder-rich region are slightly greater than the nominal value.

<table>
<thead>
<tr>
<th>Microstructure phase</th>
<th>Elastic modulus (GPa)</th>
<th>Hardness (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>WC grain</td>
<td>$606.3 \pm 41.1$</td>
<td>$25.9 \pm 3.1$</td>
</tr>
<tr>
<td>Binder-rich region</td>
<td>$484.1 \pm 26.5$</td>
<td>$20.9 \pm 2.4$</td>
</tr>
<tr>
<td>Nominal value for bulk cemented WC</td>
<td>560</td>
<td>18.1</td>
</tr>
</tbody>
</table>

The hardness was slightly affected by the indenting load. As shown in Figure 2, when the indenting load was higher, the indent would cover more WC grains, and the influence of WC grains was thus greater, resulting in slightly larger hardness than its nominal. However, this tendency is not apparent in the elastic modulus. This result may suggest that the local hardness is more sensitive to the cWC microstructure than the elastic modulus.

**Pop-in Events.** No pop-in phenomena was observed when indenting using Berkovich tip. However, pop-in followed by acoustic emission signals were observed when indenting using cube corner indenter tip at the same load range. Pop-in events occurred randomly at any penetration depth of the loading period. AE events were investigated by varying indentation loads. Table 2 shows the effect of load on the occurrence rate when the loading rate was kept to 8 mN/s. It is apparent that a greater load resulted in a high occurrence rate of the pop-in events. Previous studies have also shown that a pop-in event in nanoindentation was often associated with a fracture event in the indentation of brittle materials [20]. However, when the indented surfaces were examined, there was no evidence of cracking observed, which suggests that fracture possibly occurred in the specimen subsurface.

<table>
<thead>
<tr>
<th>Indentation load (mN)</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
</tr>
</thead>
<tbody>
<tr>
<td>Occurrence rate (%)</td>
<td>3</td>
<td>17</td>
<td>23</td>
<td>37</td>
<td>53</td>
<td>57</td>
<td>63</td>
<td>70</td>
</tr>
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</table>

**Coefficient of Friction.** The lateral force in nanoscratching is an important parameter to characterize the removal and deformation characteristics of material being scratched. During nanoscratching, the coefficient of friction was obtained by taking the ratio of the lateral force ($F_L$) over the normal force ($F_N$) used. Figure 3a and 3b are the typical lateral force histories of scratched WC grains and binder-rich region, respectively. Figure 3a shows the lateral force on a WC grain.
slightly varied with the progress of scratching. In Figure 3b, the lateral force was discontinuous at some points along with the tip movement. This abrupt drop was corresponded to the cobalt binder area between adjacent WC grains. The binder material was softer, which caused the tip experienced sudden drop (indicated by the arrow). The lateral force then was stable as the tip passed through the surface of subsequent grain. The relationship of lateral force and normal force obtained from scratching WC grains was linear. Thus, an increase in normal force resulted in an increase in lateral force. The coefficient of friction of WC grains slightly varied, with average value of 0.18.

Figure 3. Lateral force during scratching on (a) WC grain and (b) binder-rich region.

Fracture Characteristic. Figure 4 shows a typical SEM images of the scratched surface under a normal load of 4 mN. A relatively smooth scratching groove with clear edges was observed. Relatively small debris are visible along the scratch edges, and then a build-up is seen at the end of the scratch. The fractured grains and cracks were observed along the scratch.

Figure 4. SEM image of scratched surface at a load of 4 mN. The arrow indicates scratching direction.

Figure 5 shows the SEM cross-sectional view of a scratched sample at load of 8 mN. Fractures under the scratch impression were observed. The fragmented WC grains into smaller size grains in the contact area (i), and a fractured WC grain underneath the scratch groove (ii) were observed. The fragmented grains at the contact area were apparently contributed to the high stress generating during scratching. The fractured WC grain under the groove was evidently caused by the moving adjacent grains with sharp corners due to squeezing caused by scratching. The concentrated pressure from sharp corners caused the WC grain cleaved.

Figure 5. SEM images of cross-sectional view of a scratched specimen showing (a) the scratch impression, as pointed by the arrow, and (b) fractures underneath the impression.

Concluding remarks

Nanoindentation showed that elastic modulus and hardness of individual WC grains were greater than the respective values obtained in the binder rich region. No pop-in phenomena was observed when indenting using Berkovich tip. However, pop-ins and the corresponding acoustic emission
signals were observed when indenting using cube corner indenter tip in the same load range. This is a clear indication of the effect of tip sharpness on fracture characteristics of cWC. Different friction coefficients were obtained when scratching on a WC grain and binder rich region too. The scratch-induced deformation patterns were strongly influenced by the applied load. Fracture characteristics were observed on both surface and subsurface of the scratched samples. In summary, the microstructure of cWC has significantly affected its mechanical properties and removal and deformation characteristics. Such influence should be taken into account to determine the critical conditions for damage-free grinding.

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