

# **Application of X-ray diffraction to study the grinding induced surface damage mechanism of WC/Co**

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## **Abstract**

X-ray diffraction was utilized to examine the WC/Co surface after high spindle speed grinding (HSSG) to get a further insight into the machining induced surface damage mechanism. The results showed that grinding induced reorientation and preferred {100}/{10-10} growth of WC particles occurred in the deformed surface, while the crystallinity of WC(001) increased. Based on the analysis of the penetration depth of X ray in WC and Co, grazing incidence X-ray diffraction (GIXRD) showed that the grinding induced preferred crystal growth occurred only in the outmost layer (~3.324 nm), but the compressive stress was caused to a certain depth of the subsurface (>756.18 nm).

**Keywords:** X-ray diffraction; Preferred orientation; Compressive stress; WC/Co; Grinding

## **1. Introduction**

Grinding of cemented carbides has been widely conducted for the extensive application of WC/Co in molding industry, automatic field and cutting tools [1-5]. To improve the life span of the devices or tools, the (sub-)surface damage induced during the machining is of great concern. It has been reported that the varied worn modes occurred for the machined surface, such as surface deformation, fracture, grain pull-outs, and residual stress [3, 5-7].

To characterize the surface damages, different methods have been applied to examine the machined surface, such as Electron Backscattering Diffraction (EBSD) [7, 8], Transmission Electron Microscope (TEM) [9-12], Scanning Electron Microscope (SEM) equipped with Energy Dispersive Spectra (EDS), Focused Ion Beam (FIB) [6], as well as X-ray Diffraction (XRD) [1, 13, 14]. In comparison, X ray diffraction (XRD) technique provides us an effective and nondestructive approach to detect the structure transformation of the machined surface.

In the present work, XRD (Bragg-Brentano X-ray diffraction and Grazing incidence X-ray diffraction) is utilized to characterize the grinding induced crystallinity change and preferred orientation of WC/Co, and the corresponding theoretical analysis is given based on the specific crystal structure of WC.

## **2. Experiments**

High spindle speed grinding (HSSG) of WC/Co (~10 wt.%Co) composites was conducted on an ultra-precision grinding machine (Moore Nanotech 450UPL, USA), with minimum quantity of oil coolant (CLAISOL 350). Detail information of the materials properties and grinding setup could be found in our previous studies [5, 15]. The micro-structure change of the machined WC/Co surface was characterized by Bragg-Brentano X-ray diffraction (BBXRD) and Grazing

Incidence X-ray diffraction (GIXRD) with  $CuK_{\alpha}$  radiation (Rigaku SmartLab) in a scanning angle range of 20 to 80°. The spectra was analyzed by Jade 6.5, compared with the standard PDF2-2004 database, and the reference number of the powder diffraction file for WC is PDF#73-471.

### 3. Results and discussion

To eliminate the impact of the rough surface after sintering, the WC/Co was firstly polished with the diamond paste of 1  $\mu\text{m}$  diameter to remove the surface layer and reduce the impact of the surface defects. The surface morphology, as shown in Fig. 1(a), of the polished WC/Co composites indicates that there are no obvious defects in the original bulk material and the average grain size of WC is about 2  $\mu\text{m}$ . The obtained surface roughness ( $R_a$ , arithmetic mean of the departures of the profile) was about 3 nm for the workpiece after polishing, as shown in Fig. 1(b). Fig. 1(c) and Fig. 1(d) shows the surface morphology and surface topography of WC/Co after grinding. It can be readily seen that the machined surface is covered by scratching grooves, WC grain dislodgement, as well as the extrusion of binder. In addition, the surface roughness reached 5.43 nm ( $R_a$ ). Compared with the original surface, the XRD pattern of the machined surface, as shown in Fig. 2, shows some reduction in the peak intensity, which is firstly attributed to the formation of a densified layer that is seriously deformed under the dynamic pressure of diamond grits [16]. Specifically, due to the extrusion of cobalt binder, the surface deformation was induced during grinding, where the mean free path of WC grains decreased, resulting in the densification of the deformed surface [1, 14, 15]. In addition, amorphization of WC could be induced under the shear stress of the dynamic diamond grits during grinding, which has been reported by Stoyanov et al [17], and this could also lead to the decreasing peak intensity.

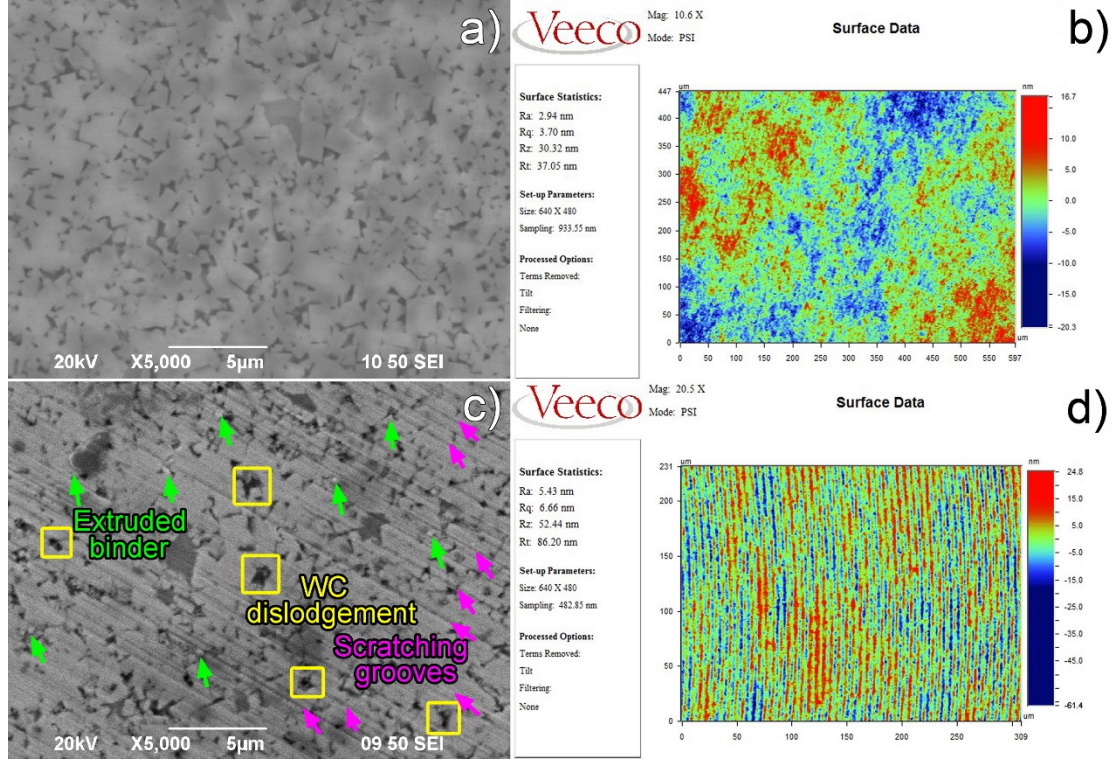


Fig. 1 Surface morphology and topography of WC/Co after polishing and grinding: (a) and (b) after polishing, (c) and (d) after grinding

Crystallinity is specified as a percentage of the volume of the material that is crystalline, and the crystallinity of the different crystal planes for WC is calculated by Eq. (1):

$$Crystallinity = \frac{I_{\text{Intensity of crystalline peaks}}}{I_{\text{Total intensity of spectrum}}} \times 100\% \quad (1)$$

In the present work, the calculated crystallinity of WC, as shown in Fig. 2(c), indicated that the crystallinity for WC(001), WC(100) and WC(101) all decreased, and this is attributed to the formation of amorphous WC [17]. In addition, the texture coefficient of different crystal planes for WC could also be obtained referring to [18, 19], which was calculated based on Eq. (2),

$$T_c = \frac{n I_{hkl} / I_{hkl}^0}{\sum I_{hkl} / I_{hkl}^0} \quad (2)$$

where  $I_{hkl}$  is the measured peak intensity of  $(hkl)$  plane,  $I_{hkl}^0$  is the peak intensity of the same plane as indicated in the standard sample (PDF#73-471),  $n$  is the total number of reflections observed

( $n=3$ ). Based on the crystal structure of WC, as shown in Fig. 2(e), it can be easily seen that the close-packed crystal plane is  $\{0001\}$ , and the close-packed orientation is  $\langle -12-10 \rangle$ . To low the system energy, preferred WC $\{0001\}/\langle -12-10 \rangle$  growth is expected during sintering, so the preferred orientation for the original workpiece is WC(001), as shown in Fig. 2(d).

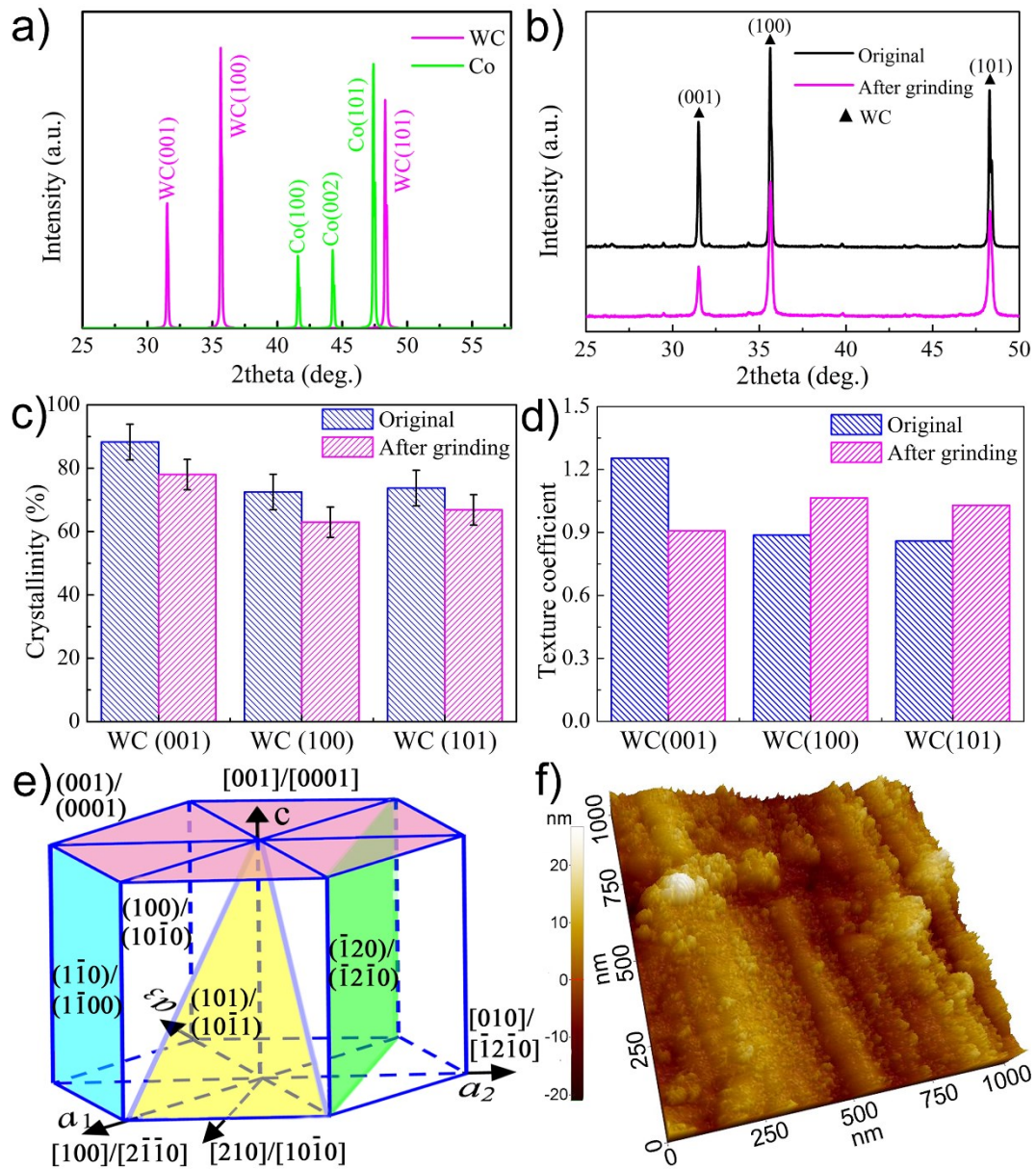


Fig. 2 (a) Standard XRD pattern for WC and Co phases, (b) XRD pattern of the original material and machined surface, (c) the calculated crystallinity of the original and machined WC/Co, (d) the calculated texture coefficient, (e) typical crystal plane and orientation of WC, (f) surface

topography of the machined WC/Co measured by AFM

However, the preferred orientation of WC(100)/(10-10) appeared for the machined surface, as indicated in Fig. 2(d). As far as the high grinding temperature and dynamic pressure of the diamond grits were concerned, the grain growth of WC could be induced, which promoted the further growth of WC grains, leading to the preferred growth of WC(100)/(10-10) for the machined surface. The surface topography of the machined WC/Co measured by AFM in a range of  $1\ \mu\text{m} \times 1\ \mu\text{m}$  is shown in Fig. 2(f). It can be readily seen that the machined surface is covered by many nano grains. According to the previous experimental and theoretical study, the instantaneous temperature of the grinding zone, which is called ‘spike’ or ‘flash’ temperature, could be close to the melting point of the workpiece material with minimum quantity lubrication, especially at a very small depth of cut where the friction heats resulted in the high temperature [20-22]. Therefore, the preferred growth for the nano grains could be induced during the grinding process under this consideration.

Previous studies have also shown that reorientation of grains could be induced in the seriously deformed layer [23-25], and the reaction indicated by Eq. (3) occurred [26]. In addition, a tendency of fracture path occurred on  $\{10-10\}$  has been proposed during the indentation of WC/Co [27]. Therefore, the expected orientation alignment and grain fracture could lead to the increasing amount of prismatic planes (WC $\{100\}/\{10-10\}$ ), resulting in the preferred WC(100)/(10-10).

$$\frac{1}{6}[11\bar{2}\bar{3}](\bar{1}\ 100) + \frac{1}{6}[2\bar{1}\ \bar{1}\ 3] \rightarrow \frac{1}{2}[10\bar{1}0] \quad (3)$$

As is known, the machined surface of WC/Co after grinding is always covered by a deformed layer of about  $2\ \mu\text{m}$  or more [23-25], and it could be seen as a thin film. Compared to BBXRD,

the outmost layer could be characterized by GIXRD with a controlled incident angle [28-30]. Fig. 3 shows the XRD instrument and the schematic of GIXRD geometry performed in out of plane mode. A critical incident angle  $\alpha_c$  is believed to be of great significance for GIXRD and it could be determined by Eq. (4) [30],

$$\alpha_c = \sqrt{2 \times \frac{N_A r_e \rho}{2\pi A} \lambda^2 (f_0 + f')} = \sqrt{\frac{N_A r_e \rho Z}{\pi A} \lambda} \quad (4)$$

where  $\alpha_c$  is the critical incidence angle,  $\alpha_i$  is the incidence angle,  $r_e$  is the classical electron radius,  $N_A$  is the Avogadro constant,  $A$  is the mean atomic weight,  $\rho$  is the mass density,  $\lambda$  the wavelength of X ray,  $f_0$  is the atomic scattering factor,  $f'$  is the dispersion correction term for  $f_0$ ,  $Z$  is the atomic number of workpiece. Based on Eq. (4), the critical incident angle for WC and Co was achieved to be  $0.5181^\circ$  and  $0.414^\circ$ . When the incident angle is less than the critical angle ( $\alpha_i < \alpha_c$ ), the total external reflection of X ray would occur and the penetration depth is limited to the outmost surface layer; if  $\alpha_i \geq \alpha_c$ , the depth of penetration would rise rapidly, as illustrated by Eq. (5) and Eq. (6) [30].

$$L = \frac{\lambda}{2\pi \sqrt{\alpha_c^2 - \sin^2 \alpha_i}}, \quad \alpha_i < \alpha_c \quad (5)$$

$$L = \frac{1}{2\mu} \cdot \sin \alpha_i, \quad \alpha_i \geq \alpha_c \quad (6)$$

where  $L$  is the penetration depth and  $\mu$  is the linear absorption coefficient. Therefore, GIXRD could serve as an effective tool in the study of subsurface damage, including phase structure and residual stress.

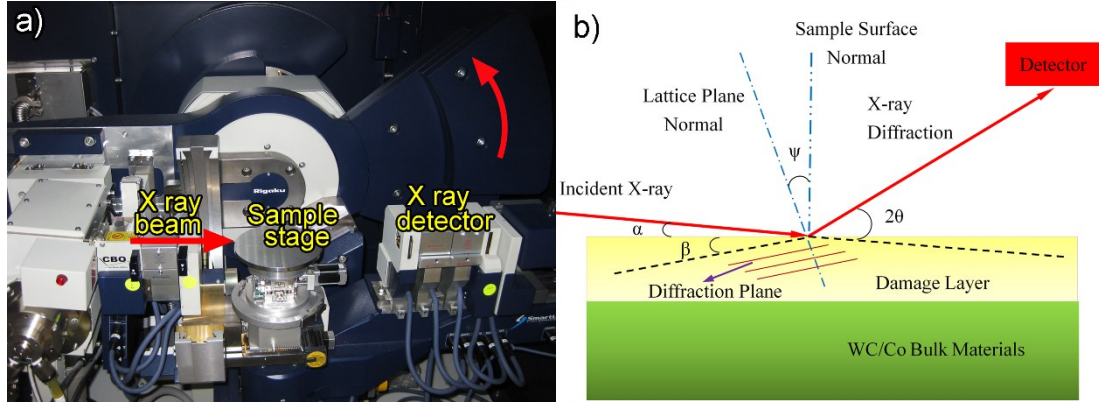


Fig. 3 (a) Illustration of the XRD instrument, (b) schematic of the GIXRD geometry for out of plane mode, where the damage layer is assumed to be a thin film isolated from bulk material

As shown in Fig. 4(a), the peak intensity grows stronger with the increasing incident angle. Specifically, compared with the standard peak position (PDF#73-471), the peak position of the machined surface shows some right shift, indicating the decreasing crystal space and compressive stress, as shown in Fig. 4(b), and the compressive stress exists to some depth beneath the grinding surface, which is greater than 756.18 nm (the calculated penetration depth of X ray at  $\alpha_i=1.2^\circ$  by Eq. (6)). In addition, the texture coefficient for the outmost layer, the depth of which is calculated to be 3.324 nm at the incident angle of  $0.3^\circ$  based on Eq. (5), obviously differs with the subsurface over a certain depth, where preferred orientation of WC(100)/(10-10) appears. However, the further increase of incident angle shows that the texture coefficient does not change obviously, which indicates that the grinding induced preferred crystal growth occurred only in the outmost layer.

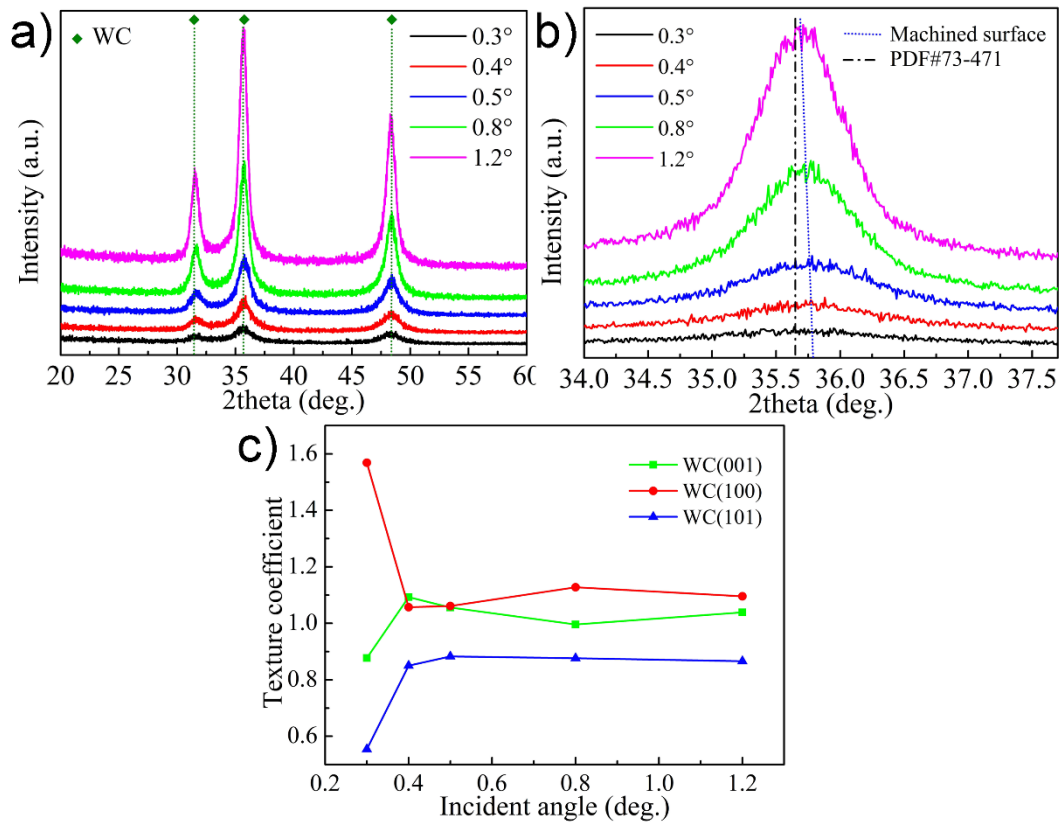


Fig. 4 (a) GIXRD pattern of the machined surface at different incident angle, (b) an enlarged view around the peak of WC(100), (c) the calculated texture coefficient for WC(001), WC(100) and WC(101)

#### 4. Conclusions

X ray diffraction is applied to examine the grinding WC/Co surface in the present work. The results showed that the preferred WC{100}/{10-10} growth occurred in the outmost surface (~3.324 nm), which is in good consistence with the theoretical analysis based on the crystal structure of WC. In addition, compressive stress was induced to some depth (>756.18 nm) during grinding.

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