euspen's 17th International Conference & Exhibition, Hannover, DE, May 2017
www.euspen.eu

Subsurface structure defects beneath fracture area of reaction-bonded silicon carbide in ultra-precision grinding

Feihu Zhang[1], Zhipeng Li[1], Xichun Luo[2]

1 School of Mechatronics Engineering, Harbin Institute of Technology, Harbin, China
2 Department of Design, Manufacture and Engineering Management, Faculty of Engineering, University of Strathclyde, UK

zplihit@gmail.com

Abstract
In this paper, cross-section transmission electron microscopy (TEM) was first time explored to investigate the structure defects beneath the fractured area of reaction-bonded silicon carbide (RB-SiC) induced by ultra-precision grinding. The results showed that there was no slip system glide occurred in SiC particle, just basal plane <a> dislocation was activated and dissociated into Shockley partial dislocations. It is proposed that the blocking effect on sliding motion caused by cross propagated dislocations, phase boundary, and sintering agents play an important role in the evolution of brittle fracture.

Key Words: RB-SiC; TEM; basal plane <a> dislocation; sintering agents; phase boundary

1. Introduction

Reaction bonded silicon carbide (RB-SiC) has attracted much attention as a prospective material to be used in a high temperature and corrosive environment due to its high thermal conductivity, thermal shock resistance and high chemical inertness [1]. However, on account of sp3 orbital hybridization, the tetrahedral covalent bonds of SiC makes it a difficult to machine even using the hardest diamond tool. Besides, limited yield deformation characteristic of SiC leading brittle fracture which was always inevitable occurred during the machining process. Hence, the induced surface and subsurface damage posed an obstacle to the valid performance of RB-SiC based devices. Therefore, the mechanisms of micro-defects in the subsurface of fractured RB-SiC composite during machining need to be deeply understood. Early studies were mainly focused on the ductility response of 6H-SiC, 4H-SiC and 3C-SiC single crystal based on compression, nanoindentation /scratching experimental techniques or molecular dynamics (MD) simulation [2,3].

While composited RB-SiC material composed of α-SiC, β-SiC and Si, grain/phase boundary microstructure exhibit more complicated deformation mechanism than single crystal. Recently, micro grinding RB-SiC study have proposed that amorphization (High-Pressure Phase Transformation, HPPT) occurred for both SiC and Si phases in the outmost layer based on X-ray diffraction (XRD) detection [4]. Nevertheless, there are no reports on the direct observation of the fractured area of machined RB-SiC surface in atomic level. Is there exist structure defects underneath the brittle fracture area? What's type? what relationship it with brittle fracture? This paper aims to investigate the subsurface structure defects and formation mechanisms underneath brittle fractured area of RB-SiC ground by ultra-precision grinding through TEM observation.

2. Experiment details

The dimension of RB-SiC ceramics was 12.5×12.5×5 mm, which offered by Goodfellow Cambridge Ltd., UK. The grinding experiments were carried out on a Moore Nanotech 350FG ultra-precision freedom grinder with water-based coolant (CHALLENGE 300-HT). The grain size of the diamond wheel is 7μm. A wheel velocity v_s of 5000 rpm, a feed rate v_w of 2mm/s, a depth of cut a_p of 2 μm are used in the grinding trial. After grinding experiments, the machined surface morphology was characterized by scanning electron microscope (SEM) (Dual beam FEI Helios Nanolab 600i). The cross section lamellae for scanning transmission electron microscopy (TEM) analysis was prepared by focused ion beam (FIB) in situ etching (Helios Nanolab600i). The detail of the lattice defects beneath the fractured surface was investigated by TEM using a FEI Talos F200x, operated at 300 kV.

3. Surface characteristic by SEM

Fig. 1 shows a SEM micrograph of the fractured zone induced in grinding process, which first appeared at SiC phase and the fractured core was mainly restricted in a SiC particle. The EDS spectra illustrate that dark and grey phase corresponding to SiC and Si particles, respectively. A tiny crack can be identified at the initiate sites of the fracture and impeded by the grain boundary. Besides, most areas of the fractured surface were almost smooth. Those observed features indicate that fractured occurred in a transgranular manner. Tearing behavior caused drape fracture characteristics are also noticed as marked by the dash lined square. Moreover, it is interesting to found that on Si particles cracks are absent, meaning the ductile removal took place under the same machining condition compared with SiC particles.

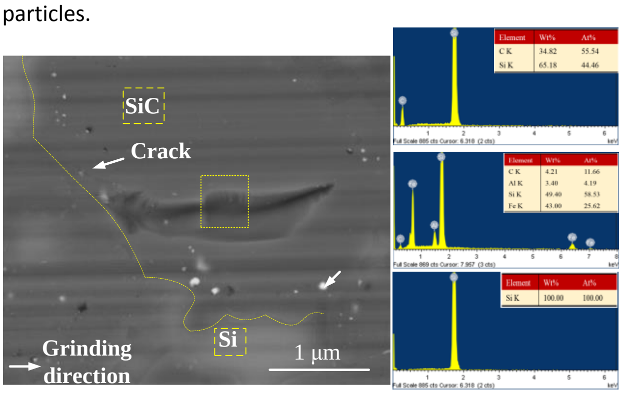

Figure 1. SEM image and EDS spectra of fractured surface morphology.

4. Subsurface structure defects

As shown in Fig. 2(a) TEM cross sectional image, there are mainly contained three particles i.e. two 6H-SiC particles and one diamond cubic Si-I particle. No crack were observed. It is noteworthy that the upper part of grinding-induced deformation zone in 6H-SiC grain containing a high density of dislocations. The dislocation-dislocations interaction leads the formation of nodes or dislocation locks due to cross propagation as denoted by red arrows. No slip bands can be found in this region. Under the dashed line, another type of dislocations that emitted from high energy interface of SiC and Si caused by interface atoms deviated from the equilibrium position and terminated within the SiC grain. Fig. 2(b) also shown that a dislocation wall was generated and some dislocation half loops were distributed along the sides of dislocation wall. Both end points of half loops are pinned by the residual sintering agents, which evidence suggests that the activation of dislocations is likely Frank-read mechanism. Future more, the independent residual sintering agents embedded in ‘g1’ can also serve as initiated sites of dislocation and those dislocations propagated to the surroundings as shown in Fig. 2(c). Thus, it can be concluded that the place with lattice defects such as phase boundary and impurities always act as the preferable nucleates sites of dislocations. While ‘g3’ contained lots of stacking faults stripes which were parallel to the basal plane. This observed phenomenon was presumed to be induced in the sintered process of material because 6H-SiC has lower stacking faults energy.

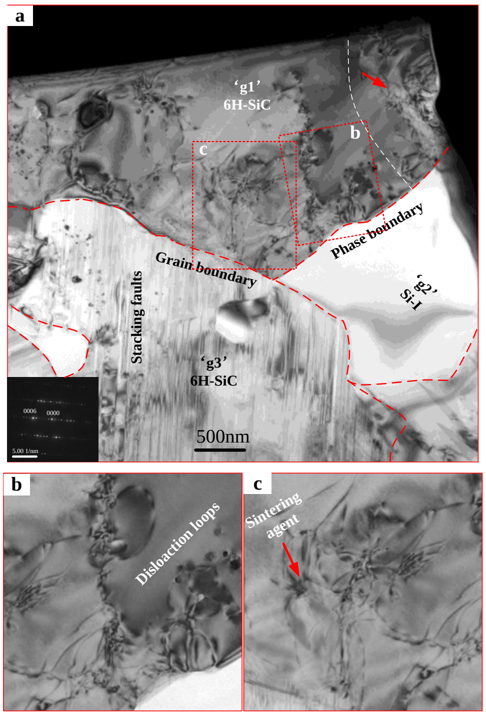

Figure 2. (a) Cross-sectional Bright field (BF) TEM image of the subsurface damage beneath fracture area (b-c) high magnifications corresponding ‘b’ ‘c’ marked in (a), respectively.

Besides, in order to examine the nature of dislocation structure in 6H-SiC particle, a two beam analysis using invisible criterion $g \cdot b = 0$ (g is diffraction vector, b is Burgers vector) was carried out with different diffraction conditions. Most of dislocations are out of contrast with the diffraction g=[0-110], g=[-1100] and no stacking faults fringes contrast can be observed. Therefore, those dislocations are nearly perfect basal plane dislocations with a Burgers vector b1=(1/3)[-2110] and b2=(1/3) [11-20].

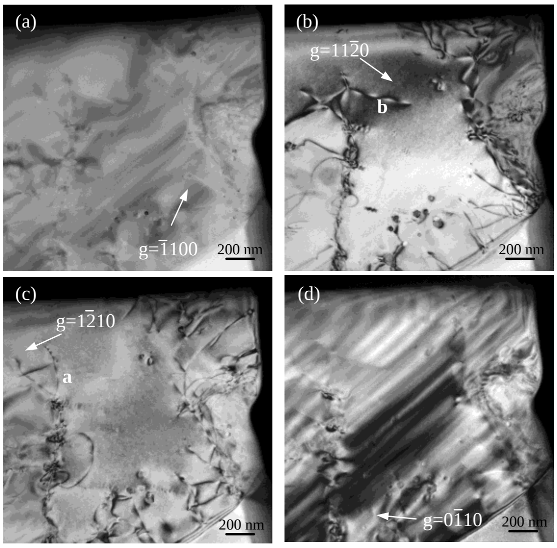

Figure 3. TEM BF images in two-beam conditions of dislocation underneath 6H-SiC particle (a) g=[-1100] (b) g=[11-20] (c) g=[1-210] and (d) g=[0-110].

Owing to HCP structure features, 6H-SiC has less dislocation slip system and the basal plane <a> dislocation activation cannot fulfill von Mises criterion as least five independent slip systems are needed to achieve the plastic deformation [23]. The interaction between basal plane dislocations results in the formation of junctions (Fig. 2). The constraining effect of dislocation junctions on dislocation motion becomes increasingly more important with the increasing strain. It provides intrinsic resistance to the movement of dislocations. Hence, there is no way to accommodate the strain along other directions, the fracture was induced on the surface of SiC grain.

On the other hand, the interaction between elastic field originating from the interface lattice mismatch of residual sintering agents/SiC particles and stress field offered by dislocation will increase CRSS needed to active the slip system. Thus, the critical stress need for the crack propagation was presumed to be achieved ahead of stress need for macroscopic sliding plastic deformation.

5. Conclusions

In summary, this study has first time adopted TEM as a direct observation method to explore the fracture damage mechanism of RB-SiC in ultra-precision grinding. Beneath SiC particle, basal plane <a> dislocation was activated and dissociated into Shockley partial dislocations were the only subsurface damage types. The blocking effect on sliding motion caused by cross propagated dislocations, phase boundary, and sintering agents play an important role in the evolution of brittle fracture.

Acknowledgement

This research is financially supported by the Major State Basic Research Development Program of China (973 Program, Grant No. 2011CB013202).

References

[1] Goel S, 2014. The current understanding on the diamond machining of silicon carbide. Journal of Physics D: Applied Physics, 243-001.

[2] Yan J, Gai X and Harada H, 2010. Subsurface damage of single crystalline silicon carbide in nanoindentation tests. Journal of nanoscience and nanotechnology, 7808-7811.

[3] Noreyan A. and Amar JG, 2008. Molecular dynamics simulations of nanoscratching of 3C SiC. Wear, 956-962

[4] Zhang Q, To S, Zhao Q and Guo B, 2015. Amorphization and C segregation based surface generation of Reaction-Bonded SiC/Si composites under micro-grinding. International Journal of Machine Tools and Manufacture, 78-81.