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Recrystallization of amorphized Si during micro-grinding of RB-SiC/Si composites

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Abstract

X-ray diffraction (XRD) was performed to investigate the phase transformation of Reaction-Bonded SiC/Si composites (RB-SiC/Si) induced by micro-grinding. The results showed that amorphization (High Pressure Phase Transformation, HPPT) occurred for both SiC and Si phases in the outmost layer, and the amorphization degree dropped as the feed rate changed from 3 mm/min to 0.1 mm/min. Moreover, we firstly found that recrystallization of amorphized Si appeared in preferred orientation under grinding. Specifically, preferred Si(111) growth occurred at a lower feed rate attributed to the more obvious annealing effect, while preferred Si(220) recrystallization developed at higher feed rate due to the greater strain. Theoretical analysis based on the crystal structure of Si yield good consistence.

Keywords: X-ray diffraction; Phase transformation; Recrystallization; Silicon; Micro-grinding

1. Introduction

For the high efficiency, ultra-precision grinding of reaction-bonded silicon carbide (RB-SiC/Si) has been extensively conducted to achieve smooth surface of good integrity [1-3]. Nevertheless, the surface damage mechanism became more complicated with the existence of free silicon in the bulk materials, even though extra Si could densify the bulk materials. Actually, high pressure phase transformation (HPPT) of Si and SiC would be produced under mechanical loading [4-12]. Specifically, the transformation of diamond cubic Si (Si-I) to high pressure phases (β-Si (Si-Ⅱ), Si-Ⅴ, Si-Ⅵ, Si-Ⅶ, Si-Ⅸ and Si-Ⅺ) might occur under increasing pressure, while Si-Ⅻ, Si-Ⅲ or amorphous Si could be produced from the high pressure phases depending on the unloading condition [5, 7]. The rapid unloading directly resulted in the amorphization of β-Si [7]. This transition was found to bear great impact on the conductivity, surface integrity and machinability [10, 11, 13, 14]. On the other hand, crystallization of the amorphous Si has been identified by Raman spectra and Transmission Electron Microscope (TEM) techniques even at a lower temperature (200℃) [7, 15-17]. Therefore, the complex environment of high pressure and temperature in the contact zone between machining tools and workpiece surface should cause greater concern during micro-grinding. However, few study has been performed to investigate the combining effects of pressure and temperature on phase transformation of Si during grinding.

Based on the above discussion, investigation on phase transformation of Si under grinding was performed by XRD technique in the present work, with the heat and pressure effects during grinding discussed.

2. Experiments

Micro-grinding of RB-SiC/Si (~90wt.%SiC, ~10wt.%Si) composites was conducted on an

ultra-precision grinding machine (Moore Nanotech 450UPL), with minimum quantity of oil coolant (CLAISOL 350). Detail information of the material properties and grinding parameters could be found in our previous study [18]. Phase transformation of the machined RB-SiC/Si surface was characterized by Bragg-Brentano X-ray diffraction (BBXRD) with CuK_α radiation (Rigaku SmartLab). According to the standard PDF2-2004 card installed in Jade 6.5 software, the achieved spectra was analyzed, where the reference number of the powder diffraction for Si is PDF#27-1402. The standard XRD patterns of Si and SiC were also derived from PDF2-2004 database.

3. Results and discussion

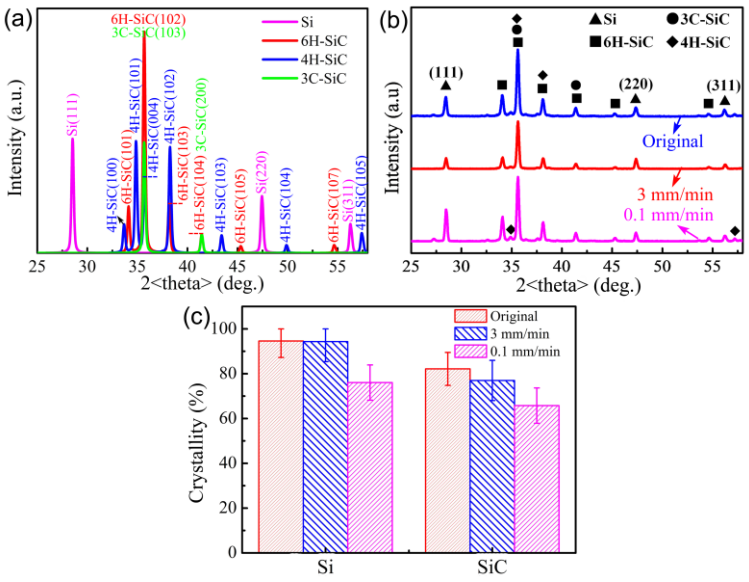

Fig. 1 (a) Standard XRD patterns of Si and SiC; (b) Bragg-Brentano X-ray diffraction spectra; (c) Calculated crystallinity for Si and SiC

Fig. 1(a) shows the standard XRD patterns of Si and SiC, while the XRD spectra of the machined and original material is shown in Fig. 1(b). Except for Si, there are diffraction peaks of 6H-SiC, 4H-SiC and 3C-SiC polytypes in the bulk material. Compared with the original material, the crystallinity dropped and amorphization occurred for both SiC and Si, which can be seen in Fig. 1(c). Moreover, it could be

found that the amorphization seemed to become more serious with the reduction of feed rate. According to Equs. (1) and (2) [19], the grinding force exerted by the diamond grits during grinding was proportional to the feed rate (f_r: feed per revolution). Therefore, the amorphization should be more severe at the higher feed rate (3 mm/min) induced by the higher pressure [4, 20]. Nevertheless, the serious surface fragmentation resulted in the removal of metamorphic material and the exposure of original material, leading to the stronger influence of the original material.

$$F_t = \frac{e_c \cdot a_e \cdot v_w \cdot f_r}{v_s} \tag{1}$$

$$F_n = \frac{F_t}{\mu} \tag{2}$$

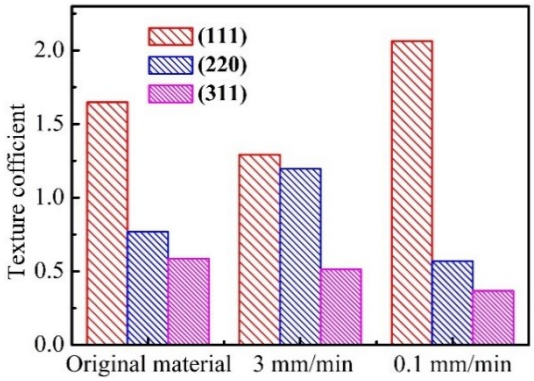

Fig. 2 Texture coefficient of Si for crystalline planes (111), (220) and (311)

Moreover, it is quite interesting to find that the peak intensity of Si at around 2θ=28.46 deg. became stronger after grinding at 0.1 mm/min than the original material. To illustrate this phenomenon, the texture coefficient (T_c) of Si phase was calculated based on Equ. (3) [21, 22]

$$T_c = \frac{n I_{hkl} / I^0_{hkl}}{\sum I_{hkl} / I^0_{hkl}} \tag{3}$$

Where I_{hkl} is the measured peak intensity of (hkl) plane, I^0_{hkl} is the peak intensity of the same plane as indicated in a standard sample (PDF#27-1402), n is the total number of reflections observed (n=3).

The maximum value of T_c is supposed to be 3 for a perfectly oriented surface, while T_c =1 means its randomly oriented nature. Calculated texture coefficients for the crystal planes are shown in Fig. 2. Compared with the original material, texture coefficient of Si(220) for workpiece machined at 3 mm/min became more notable, while Si(111) dominated for RB-SiC/Si machined at 0.1 mm/min (T_c>2). The inconspicuous change in texture coefficient for Si(311) led us to focus only on the variation of Si(220) and Si(111). As has been known, Si(111) is of isotropic elasticity and Si(220) is anisotropic [23]. During micro-grinding, the sliding and scratching between the diamond grits and workpiece surface were more evident, and it would prompt the temperature rise in the contact zone. The maximum and effective temperature during grinding might resulting in the melting of Si [5, 24]. Then, the temperature could be higher than 200℃[7], and it will promote the recrystallization of amorphized Si. The finer feed rate used, the more evident is the tempering effect. Under the assumption that the growth of crystal texture minimize the total energy of the system, the presence of the increasing (220) orientation indicates the strain energy dominated growth, which corresponds with the higher grinding force at the feed rate of 3 mm/min. On the other hand, Si(111) is of the lowest surface energy (1.14 J/m^2 for Si(111) and 1.9 J/m^2 for (110)) [25]. Under the more obvious annealing effect at 0.1 mm/min, preferred (111) orientation growth is expected to develop when the surface energy is the dominant contribution to the total energy [22].

Table 1 Atom density and bonds density of typical crystal plane for silicon

Crystal plane	Linear atom density	Plane atom density	Atomic bonds density
(110)	$\frac{1.4}{a}$/[110]	$\frac{2.8}{a}$	$\frac{2.8}{a}$
(111)	$\frac{1.17}{a}$/[111]	$\frac{2.3}{a^2}$	$\frac{6.9}{a^2}$ or $\frac{2.3}{a^2}$
(100)	$\frac{1}{a}$/[100]	$\frac{2}{a^2}$	$\frac{4}{a^2}$

* a is the lattice constant, and atom arrangement for Si(220) is the same with Si(110).

From the view of atomic structure, as is shown in Fig. 3, the atom density and bonds density of Si(100), (110), and (111) planes could be calculated, which is tabulated in Table 1, where *a* is assumed to be the lattice parameter of Si. It could be easily found that both the linear density along [110] direction and plane density of (110) is the largest one among the three special crystal planes of (100), (110), and (111). Therefore, it belongs to the close-packed plane of atoms, which is generally thought to be the preferred growth orientation. However, the special distribution of atoms on (111) plane make it quite distinctive. Specifically, the diagonal atom located at the quarter catercorner results in two different inter-planar atomic bonds density, ($6.9/a^2$ and $2.3/a^2$), respectively. The highest bond density indicates its highest stability and lowest formation energy, which make (111) plane become the much more preferred growth plane without outside interference, in spite of its relative lower plane atom density compared with (110). This yields good consistence with the experimental results and the thermodynamic analysis [22, 25].

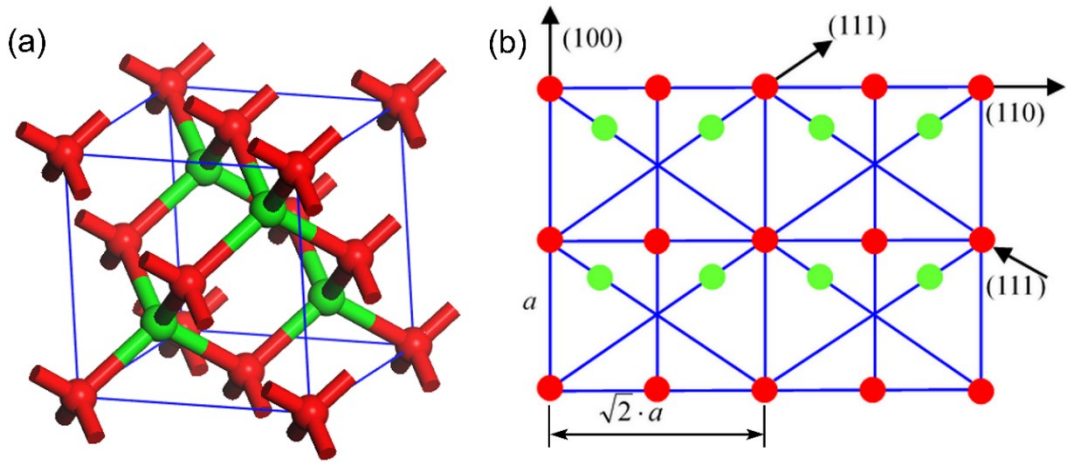

Fig.3 (a) Cubic diamond structure of Si; (b) Atomic arrangement in different crystalline plane for Si watched in parallel to the plane

4. Conclusions

In summary, phase transformation of Si during micro-grinding of RB-SiC/Si was investigated by X

ray diffraction (BBXRD). Amorphization firstly occurred for both SiC and Si phases during grinding. Considering the combining effects of high pressure and temperature in the contact zone between diamond grits and workpiece materials, recrystallization of amorphized Si was analyzed and discussed for the first time. Interestingly, preferred orientation growth of Si under different machining parameters appeared, corresponding to the strain state and annealing effects. Theoretical analysis was given based on the special lattice structure of Si, and it corresponded with the experimental results.

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