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Microstructural Characterization of Oxide Scales Thermally Formed on Single Crystal Silicon Carbide

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Silicon carbide (SiC) has many excellent properties, such as, high sublimation temperature, high chemical stability, high thermal conductivity and low thermal expansion, which make it adequate for using as high temperature structural materials. At high temperature environment, the oxide scales, which are amorphous and/or various types of crystalline silica, are known to be formed on SiC [1-3]. In this study, we observed and characterized the microstructures of the thermally formed oxide scales on singly crystal 6H-SiC using optical microscopy and transmission electron microscopy (TEM).

Double-side polished single crystal 6H-SiC obtained from Cree Inc. were used in this study. The thermal oxide scale was formed on the Si-terminated face at 1473K for 25 hours in dry oxygen at 1 atm. Cross-sectional TEM specimens were prepared by focused ion beam (FIB) micro-sampling technique. Pt was deposited on the surfaces of the sample prior to the FIB milling to prevent ion charge-up. C was deposited by electron beam and W was deposited by Ga ion beam on the surface of the oxidized sample to protect the surface of the specimen during the FIB milling. Since the formed crystalline silica scales were highly sensitive to electron beam irradiation, low-dose TEM observation was employed.

Luster gloss spherical patterns were observed on less than 1% of the whole surface of the sample after the oxidation using optical microscope. Fig.1 shows one of the spherical patterns observed on the Si-terminated face of sample after being oxidized at 1473K for 25 hours. Fig.2 shows a cross-sectional TEM image of the sample taken from the spherical region pattern indicated by a broken line square in Fig.1. The oxide scale was composed of amorphous silica and crystalline silica. The upper layer of the oxide scale was composed of crystalline silica, while the lower layer of the oxide scale was amorphous silica. The thickness of each layer was not uniformed. Fig.3 (a) shows cross-sectional TEM image of the oxide scale and Fig.3 (b)-(g) show X-ray energy dispersive (XEDS) elemental maps of of Si, C, O, Pt, W and Ca corresponding to Fig.3 (a), respectively. A trace of calcium was observed in the crystalline silica scale, as shown in the region indicated by a circle in Fig.3 (g). The existence of impurity contamination, such as H_2O or sodium, is believed to strongly influence the crystallization of the oxide scale [4]. In this study, the existence of calcium impurity was believed to be related to the crystallization of the oxide scale.

In summary, the oxide scale formed on the Si-terminated face of the single crystal 6H-SiC were characterized using optical microscopy and TEM. Spherical patterns were observed on the surface of the samples after oxidation. The oxide scale in this region was composed of amorphous and crystalline silica. XEDS map showed a trace of calcium in the crystalline oxide scale.

References

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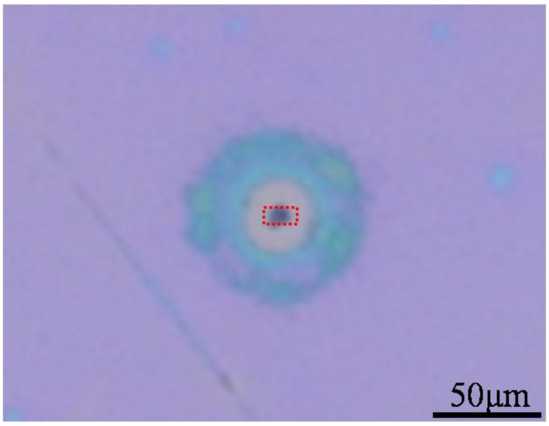

Fig.1 Optical microscope image of the Si-terminated face of the sample after being oxidized at 1473K for 25 hours.

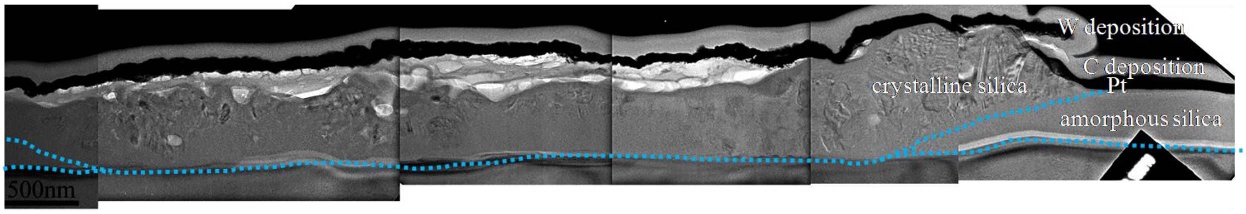

Fig.2 Cross-sectional TEM image of the region indicated by a broken line square in Fig.1

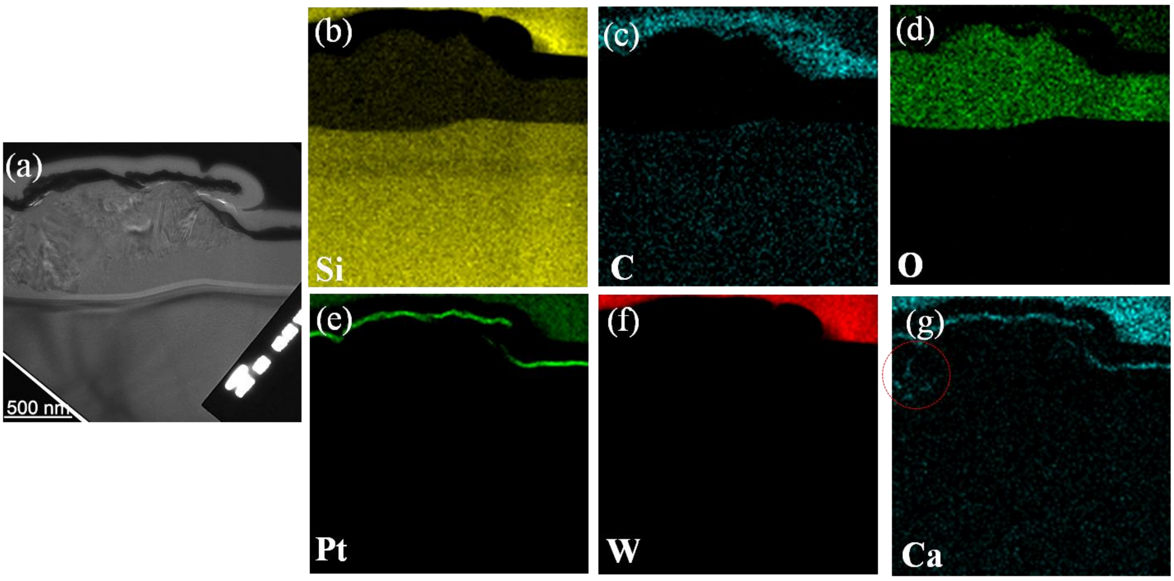

Fig.3 (a) Cross-sectional TEM image and (b)-(g) XEDS elemental maps of Si, C, O, Pt, W and Ca corresponding to Fig.3 (a), respectively.