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Microstructural Characterization of Thermal Oxide Scales Formed on Hexagonal Silicon Carbide

B.Chayasombat[1], T.Kato[2], Y.Sasaki[2], T.Hirayama[2], K.Sasaki[3], K.Kuroda[3]

[1]Graduate School of Engineering, Nagoya University, Nagoya 464-8603, Japan
[2]Nanostructures Research Laboratory, Japan Fine Ceramics Center, Nagoya, 456-8587, Japan
[3]Department of Quantum Engineering, Nagoya University, Nagoya 464-8603, Japan

Silicon carbide is adequate for variety uses in high temperature environment, such as high temperature structural material, heating element or coating material for nuclear fuel elements, because of its chemical stability, high sublimation temperature (approximately 3073K), and high oxidation resistance, etc. However, oxide scales are known to be formed on silicon carbide in certain oxidation conditions. Many studies on thermal oxidation of various types of silicon carbide in many oxidation conditions have been carried out previously in order to understand the oxidation mechanism [1]-[6]. The formed oxide scales have been reported as amorphous silica and/or various types of crystalline silica. However, the oxidation process of silicon carbide is not clearly understood.

In this study, we characterized the oxide scales thermally formed on the C-terminated face of single crystal 6H-SiC at 1473K for 20h in air using X-ray diffraction (XRD) and transmission electron microscopy (TEM). TEM specimens were prepared by focused ion beam (FIB) microsampling. The crystalline silica scales are highly sensitive to electron beam irradiation; accordingly low-dose TEM observation was employed.

Figure 1 shows XRD patterns from the oxide scale formed on the C-terminated face. The reflection peaks are as follows; the (201), the (402), the (-604) and the (020) of monoclinic tridymite, and the (110), the (220) and the (311) of cubic cristobalite. Fig. 2(a) shows a cross-sectional TEM image of oxide scale formed on the C-terminated face, and selected area electron diffraction patterns (SADP) corresponding to A and B region are shown in (a`) and (b`), respectively. The SADP in Fig.2(a`) indentifies the oxide scale as amorphous silica and Fig.2(b`) as crystalline silica. The thickness of the oxide scale in amorphous region and crystalline region are approximately the same. The crystalline silica is identified as cubic cristobalite which aligned in the [311] direction perpendicular to the silicon carbide substrate. Fig. 3 shows a cross-sectional TEM image of another region of oxide scale on the C-terminated face and inset is the SADP corresponding to the circled area. Micro-twins are observed in this region. The SADP identifies the oxide scale as cubic cristobalite aligning in the [110] perpendicular to the substrate. These results agree well with the XRD results from the C-terminated face shown in Fig. 1.

In summary, the thermally formed silica scales were characterized by XRD and electron diffraction pattern. The formed oxide scale was composed of amorphous and crystalline silica. The TEM electron beam diffraction patterns from the crystalline silica formed on the C-terminated face specimen whom oxidized at 1473 K for 20 hours in air were identified as cubic cristobalite. The crystals were alligned in the [311] and the [110] directions perpendicular to the substrate which agreed well with the XRD result. These results indicated that the thermally formed crystalline silica on the C-terminated face of single crystal hexagonal silicon carbide was oriented.

References

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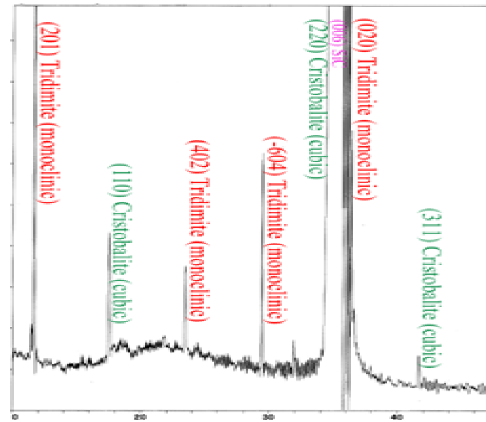

FIG. 1: XRD patterns from the C-terminated face of sample oxidized at 1473 K for 20 hours in air.

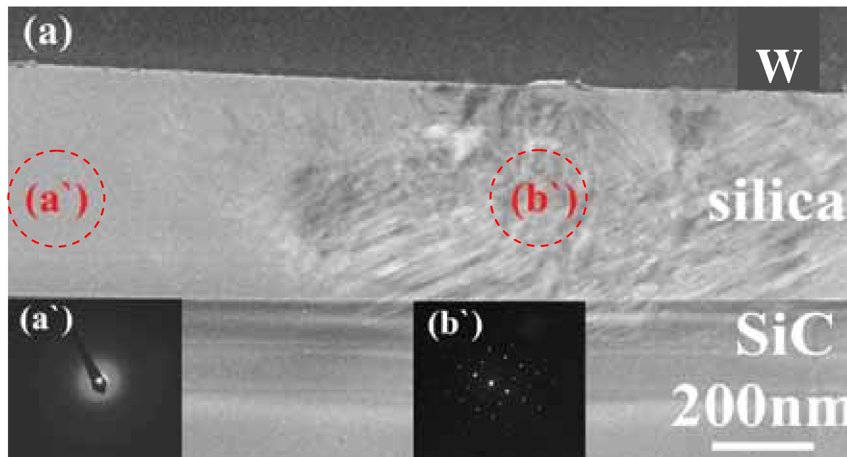

FIG. 2: Cross-sectional low-dose TEM image of oxide scale formed on the C-terminated face after oxidized at 1473 K for 20 hours in air. Insets are the SADP of each circled area.

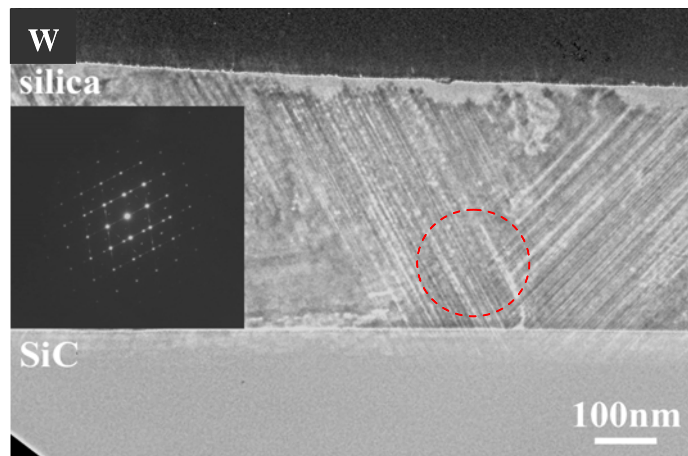

FIG. 3: Cross-sectional low-dose TEM image of oxide scale formed at 1473K in air on the C-terminated face and inset is the SADP of indicated area.