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Microstructure and phase state of a composite based on silicon carbide irradiated with krypton ions

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Abstract. Composite based on silicon carbide was irradiated with krypton ions with an energy of 280 keV fluence $1 \cdot 10^{13}$–$5 \cdot 10^{15}$ cm^{-2}. Irradiation leads to irradiation growth of the crystal lattice, as well as amorphization of the near-surface layer.
Keywords: silicon carbide, SiC-6H, Kr, amorphization, irradiation growth.

1. Introduction

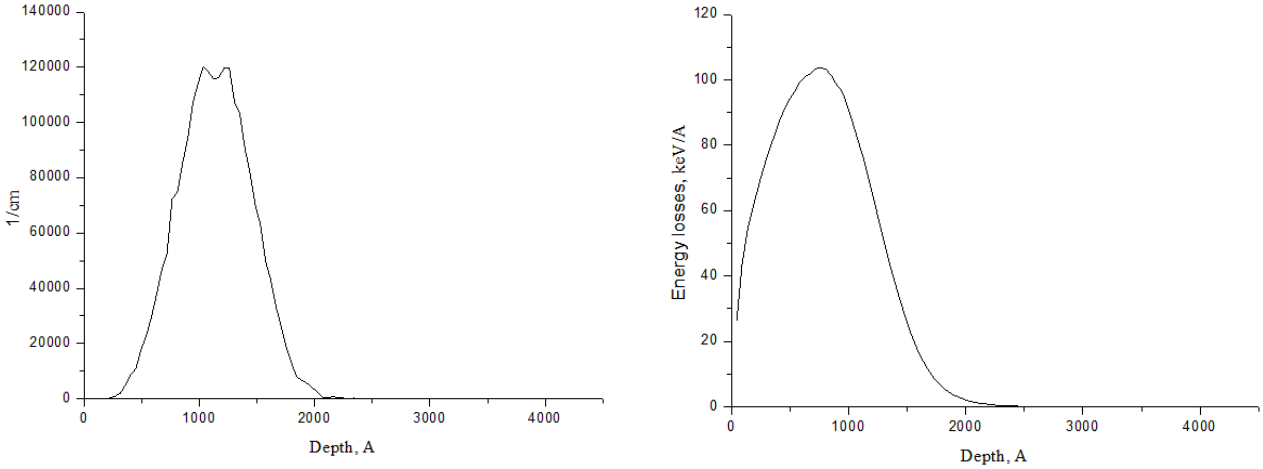
Due to its wide band gap, high thermal conductivity, good stability, high strength and radiation resistance, silicon carbide is a promising material for use as structural element in thermonuclear reactors, fission reactors and gas-cooled fission reactors, as well as in the burial of radioactive nuclear waste. [1] Thus, the study of the radiation resistance of the structural-phase state and the nature of the evolution of defects in silicon carbide after simulated irradiation of fission fragments is an urgent task.

2. Experimental setup

The samples were prepared at the Lykov Institute of Heat and Mass Transfer. Two commercially available fractions of silicon carbide powder were used as raw materials: coarse M50 grade with characteristic grain size of 50 μm and fine M5 grade with grain size of 5 μm (Volzhsky Abrasive Works, Russia). The ratio of large and small fractions is 5:3. Silicon carbide powder (88 wt.%) was mixed with a thermoplastic binder based on paraffin P-2 (12 wt.%) and was cast into the mold. Thermal removal of the binder was carried out in air at 600°C. Bakelite varnish based on resole resins LBS-1 (plant named after Yu.M. Sverdlov, Russia) was used for impregnation of a porous SIC matrix after removal of the binder. The impregnation temperature is 40°C, the pressure is 0.5 MPa. After the impregnation stage, the workpiece was dried in the air at 160 °C for 4 hours. Then the workpiece was subjected to pyrolysis in a vacuum furnace (VacETO, Russia) at a temperature of 1200°C and a pressure of 0.13 Pa for 2 hours. The same vacuum furnace was used for the final siliconizing stage, which was carried out at a temperature of 1800°C and a pressure of 0.13 Pa for 4 hours. For this purpose, the sample was placed in a closed graphite crucible and covered with a homogeneous layer of electronic purity silicon powder (Semiconductor Plant, Ukraine), which melted when heated and penetrated into the porous structure of the C/SiC workpiece. A chemical reaction took place between liquid silicon and carbon in the pores with the formation of secondary silicon carbide, which bound the initial particles of primary silicon carbide powders. The remaining space remained filled with silicon. After siliconizing, the sample surface became rough and inhomogeneous, so mechanical grinding and polishing were used to remove residual silicon and create appropriate conditions for further study of ceramics. [2] The samples were irradiated with Kr^+ ions with an energy of 280 keV at RT at the DC-60 linear heavy ion accelerator (Institute of Nuclear Physics, Nur-Sultan, Kazakhstan). Irradiations with krypton ions were carried out with fluences $1 \cdot 10^{13}$, $1 \cdot 10^{14}$, $5 \cdot 10^{15}$, $5 \cdot 10^{15}$ sm^{-2}.

The study of the structural-phase state of the initial and irradiated SiC samples was carried out by X-ray diffraction analysis (XRD) and scanning electron microscopy (SEM).

The calculation of energy losses was carried out in the SRIM 2013 program. Fig.1 shows the results of modeling implanted krypton ions in SiC samples. The maximum damage is observed at a depth of 165–175 nm. The greatest value of the displacements is: 0.03 dpa for a dose of $1 \cdot 10^{13}$, 0.3 dpa for a dose of $1 \cdot 10^{14}$, 3 dpa for a dose of $1 \cdot 10^{15}$ and 15 dpa for a dose of $5 \cdot 10^{15}$ cm^{-2}. The concentration of implanted Kr^{+} ions does not exceed 1%.



(a) (b)

Fig.1. Profiles of the distribution of implanted Kr ions (a) and energy loses (b) in SiC samples.

3. Results and discussion

Fig.2 shows an X-ray pattern of an initial silicon carbide sample.

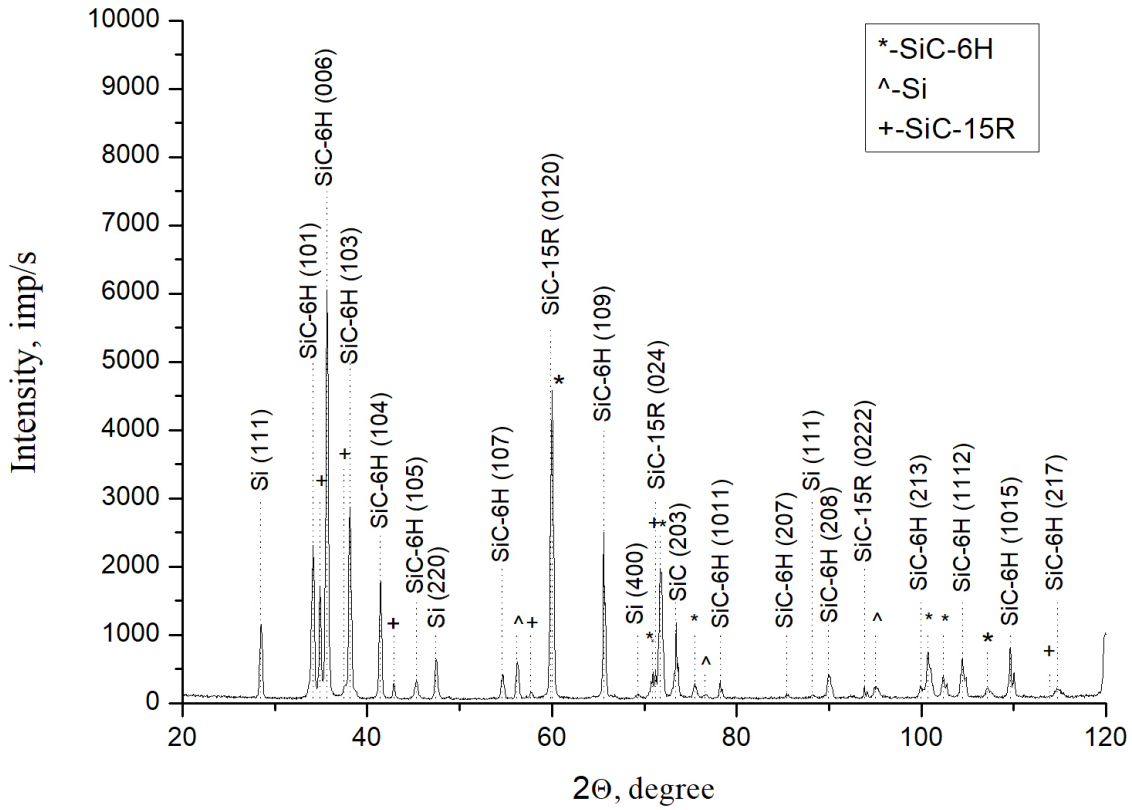


Fig.2. XRD pattern of the initial SiC.

The results of studies of the phase composition showed that the initial samples are a multiphase system: SiC-6H – hexagonal (P63mc) syngony, Si – cubic (Fd-3m) syngony, SiC-15R – trigonal (R3m) syngony. The main phase is SiC-6H (about 80%).

Fig.3 and Table 1 show the dependence of the relative change of lattice parameters ($\Delta a/a_0$) and ($\Delta c/c_0$) and on the dose of krypton ions irradiation. The lattice parameters a and c were calculated using the Rietveld method. The lattice parameters of the unirradiated SiC-6H were used as a_0 and c_0. The relative change in the lattice parameters a and c are associated with the deformation of the lattice.

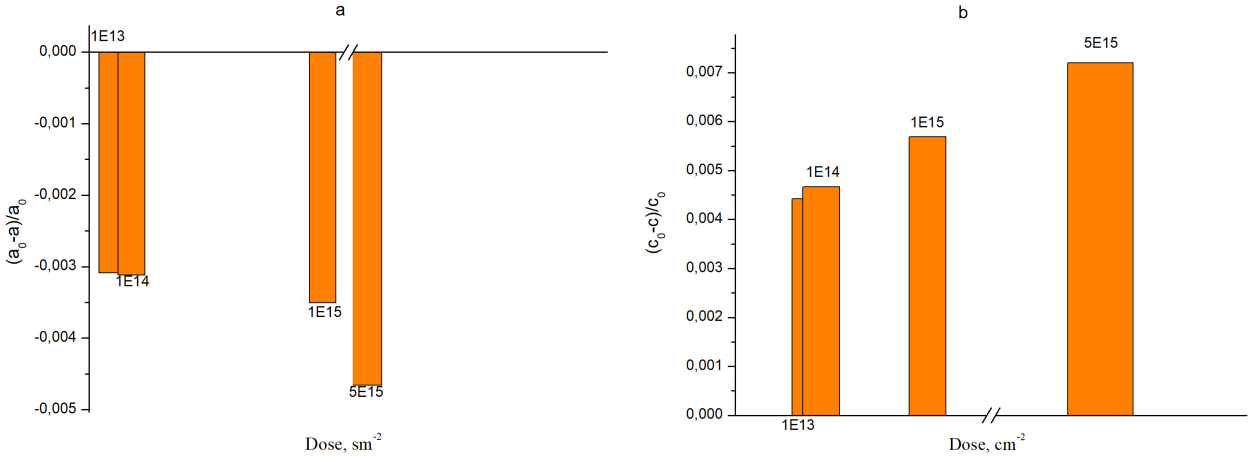


Fig.3. Dependence of relative change of lattice parameters a (a) and c (b) of 6H-SiC on the radiation dose of SiC samples irradiated with Kr ions.

Irradiation with Kr^+ ions leads to a significant increase in lattice deformation, which is caused by the formation of radiation defects and their clusters. It can be seen from the graphs that with an increase in the dose, the relative change in the lattice parameter a decreases, parameter c increases. Such a change in the crystal lattice occurs as a consequence of irradiation growth. This irradiation effect is characteristic of metals with a HCP, especially zirconium [3].

Table 1. Deformations of parameter a and c

Dose, cm^{-2}	**Relative change of lattice parameters**	
	$\Delta a/a_0$	**$\Delta c/c_0$**
$1\cdot10^{13}$	-0.00309	0.00442
$1\cdot10^{14}$	-0.00312	0.00467
$1\cdot10^{15}$	-0.00351	0.00569
$5\cdot10^{15}$	-0.00466	0.0072

Fig.4 shows the SEM images of the initial and irradiated samples. The depth of electron penetration is about 0.5 μm. On the initial sample, the elemental contrast makes it possible to identify grains of silicon carbide, and silicon between them (which is related to the technology of sample preparation). At a dose of $4\cdot10^{13}$ cm^{-2}, a grain structure is observed, but with a further increase in the dose, the amorphization of the near-surface layer occurs. Based on the data obtained and the calculated depth analysis of the sounding, it can be assumed that the thickness of the amorphous layer is approximately 0.5 μm.

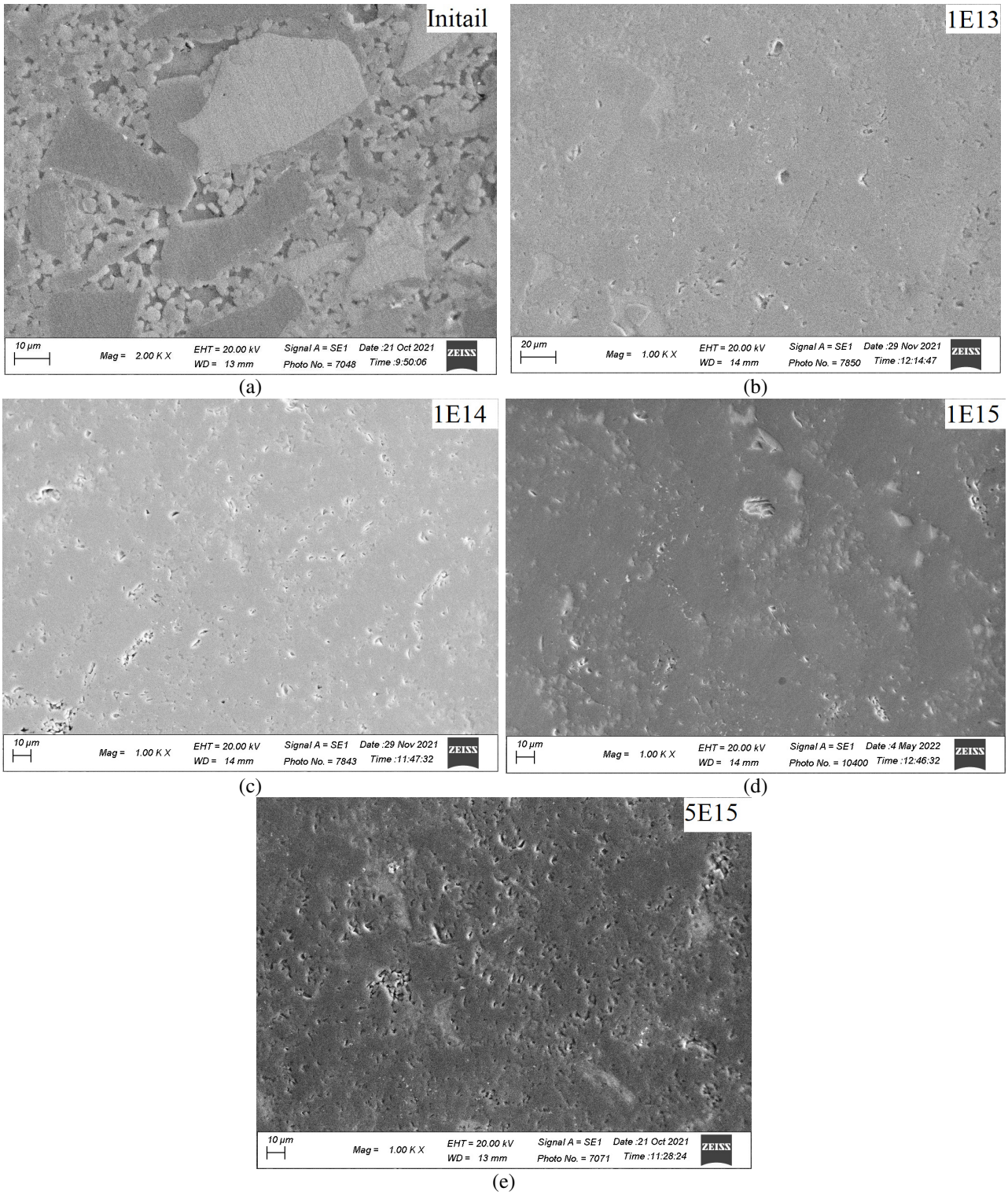


Fig.4. SEM images of initial (a) and irradiated by Kr ions with dose of 1×10^{13} (b), 1×10^{14} (c), 1×10^{15} (d), 5×10^{15} cm^{-2} (e) SiC samples.

4. Conclusion

Based on the results, several conclusions can be drawn:

- When silicon carbide is irradiated with Kr^+ ions (280 keV energy and doses up to $5\cdot10^{15}$ cm^{-2}), irradiation growth occurs, characteristic of the hexagonal type of crystal lattice;

- Irradiation leads to amorphization of the near-surface layer, which is confirmed by the electron microscopic studies.
- Based on the data obtained and the calculated depths of the depth analysis of the sounding, it can be assumed that the thickness of the amorphous layer is approximately 0.5 μm.

5. References

[1] Bus T., van Veen A., Shiryaev A., Fedorov A.V., Schut H., Tichelaar F.D., Sietsma J., *Mater. Sci. Engin.*, **B102**, 269, 2003; doi: 10.1016/S0921-5107(02)00712-2

[2] Grinchuk P.S., Kiyashko M.V., Abuhimd H.M., *J. Eur. Ceram. Soc.*, **38**, 4815, 2018; doi: 10.1016/j.jeurceramsoc.2018.07.014

[3] Li Yang, Ghoniem Nasr, *J. Nucl. Mater.*, **540,** 152312, 2020; doi: 10.1016/j.jnucmat.2020.152312