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Short communication

Laser irradiation of α-SiC ceramics

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Abstract

The process of laser irradiation of a surface of SiC ceramics in air was investigated. As a result of SiC oxidation and evolution of CO_2, porous SiO_2 forms on the target surface. During deposition of ablation products on the substrate, a loose SiO_2 film forms.

Keywords: SiC ceramics; Laser irradiation; Oxidation

1. Introduction

Due to a combination of unique properties, silicon carbide (SiC) ceramics find extensive application in several fields of engineering [1–6].

Much attention is focused on the laser treatment of materials and the physicochemical and other processes accompanying it [7–13]. Laser treatment in air of SiC ceramics is accompanied by both surface oxidation and the occurrence of oxidation products in the gaseous phase [3,8,14]. In this connection, the aim of the present work is to determine main reactions occurring during surface oxidation of SiC ceramics sintered in air under high pressure. Laser-induced corrosion of SiC ceramics can be one of the methods for making silicon oxide films on the surface of silicon carbide and for the deposition of ablation products (SiO_2) on substrates.

2. Experimental

α-SiC ceramic specimens 5 mm in diameter and 10 mm in length were used as a target. The ceramics was obtained at the following sintering parameters: $T_s = 1800$ °C, $P = 4$ GPa, and $t_s = 60$ min.

The laser irradiation of the surface was realized in ''low-power'' and ''high-power'' continuous irradiation regimes at $\lambda = 1064$ nm in air. The diameter of the laser spot was 0.3 mm. In the low-power regime ($P = 240$ mW), the irradiation time was changed up to 30 min. In the high-power regime ($P = 170$ W), laser beam passed over the surface of the target three times. The traverse speed of a coordinate table was 0.1 mm/s. After irradiation, a formed white layer of the material was removed from the upper part of the specimen.

The X-ray analysis of the specimens was performed with a Siemens D-500 diffractometer with Cu Kα radiation. The electron microscopy study and an electron-probe X-ray microanalysis of deposited products were performed with a HU-200F type scanning electron microscope and LEO 1450 VP unit. The atomic force microscopy (AFM) study (Digital Instruments Nanoscope IV in Tapping mode with a silicon nitride tip) was performed in the height (topography) and phase regimes. IR spectra on M 80 spectrometer were obtained for products deposited in the irradiation chamber to rule out the possibility of superposition with the IR spectrum of the quartz plate.

3. Results and discussion

3.1. Surface of α-SiC ceramics

The ceramic specimen consists of sintered α-SiC grains (Fig. 1a) with small SiC grains, cracks, and pores located along

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Table 1
Contents of elements in different layers of specimens.

Place of investigation	Element, wt.% (at.%)			Compound correlation
	Si	C	O	
Initial surface of a SiC specimen	40.05 (22.22)	59.95 (77.78)	Not	SiC
White porous layer on a SiC specimen after irradiation	24.72 (15.29)	8.22 (11.88)	67.07 (72.83)	SiO_2
Layer under a white porous layer	33.44 (19.08)	42.84 (57.15)	23.73 (23.77)	SiO_2–C mixture
Ablation products	47.72 (26.47)	Not	52.28 (73.53)	SiO_2

Table 2
IR absorption bands observed in the frequency range 400–2000 cm^{-1} in silica specimens.

Material	IR-frequency (ν) (cm^{-1})				Ref.
Cristobalite	487m	654w	800m	1100s	[15]
Quartz	465–470m		{798 780 m	1084s	[15]
Fused quartz	476s		802m	1114s	[15]
Silicon oxide films	460–449m	560w	783–826w	1026–1055s 1163–1179sh	[16]
Ablation product	475m	630w	800w	1125s	This work

Note: s – strong, m – middle, w – weak intensity of adsorption band, sh – shoulder on the main band.

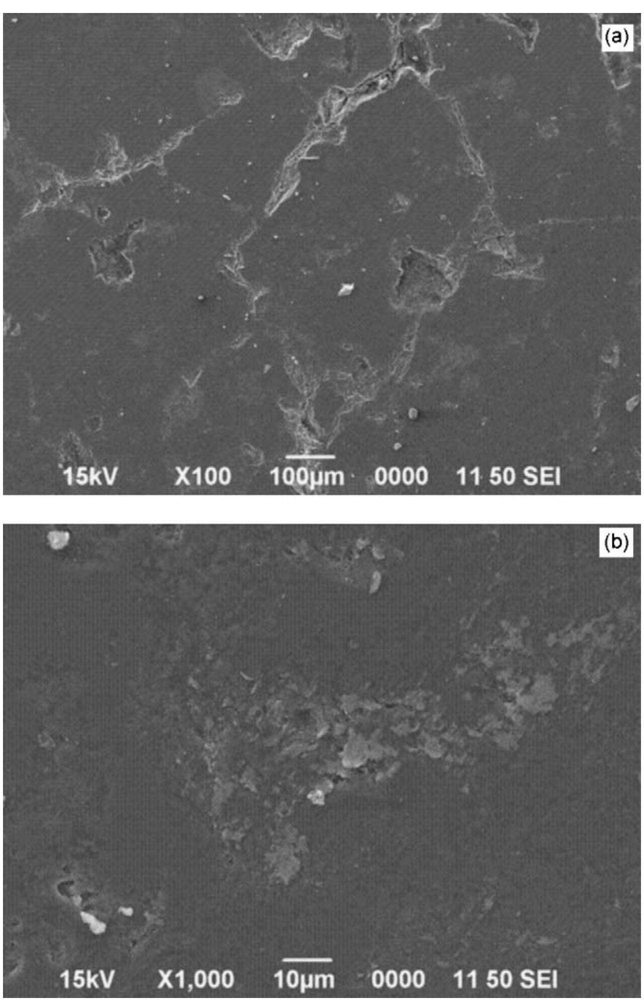

Fig. 1. Micrograph of a surface of α-SiC ceramics (a and b); (b) zone between large grains.

grain boundaries (Fig. 1b). The formation of small grains was caused by the fracture of large α-SiC grains during the high-pressure sintering. The electron-probe X-ray microanalysis detected only Si and C (Table 1).

3.2. "Low-power" laser irradiation

After irradiation, AFM micrograph shows that a loose deposit forms (Fig. 2b) on the surface of the α-SiC (which consists of α-SiC microplates, Fig. 2a). According to X-ray microanalysis, after irradiation in air and in a (N_2–O_2) medium, Si and O are present on the surface, i.e., the silicon carbide ceramics is oxidized. As the irradiation time increases, the surface of the ceramics is cleaned (see Fig. 2c) and then is covered with a SiO_2 layer. This means that evaporation–sublimation of SiO_2 occurs along with the oxidation of the SiC-ceramic surface.

On the surface of a collective plate, a SiO_2 film forms [15–19]. This is substantiated by the IR-spectroscopy data (Fig. 3, Table 2). The presence of an absorption band at $\nu \sim 800$ cm^{-1} indicates the formation of quartz. In an ATM image (Fig. 2d), it is seen that the film consists of clusters of different size.

3.3. "High-power" laser irradiation

During high-power irradiation, a white deposit in the form of a dome is obtained on the surface of a SiC-target. Fibers (whiskers) of different thickness originate from the dome (see Fig. 4). It should be noted that the formation of thick fibers is most probably a result of the deposition of products from the gaseous phase on thin fibers.

An investigation of the deposit (the formed "hat") showed that it consists of a porous sponge (Fig. 5). According to X-ray

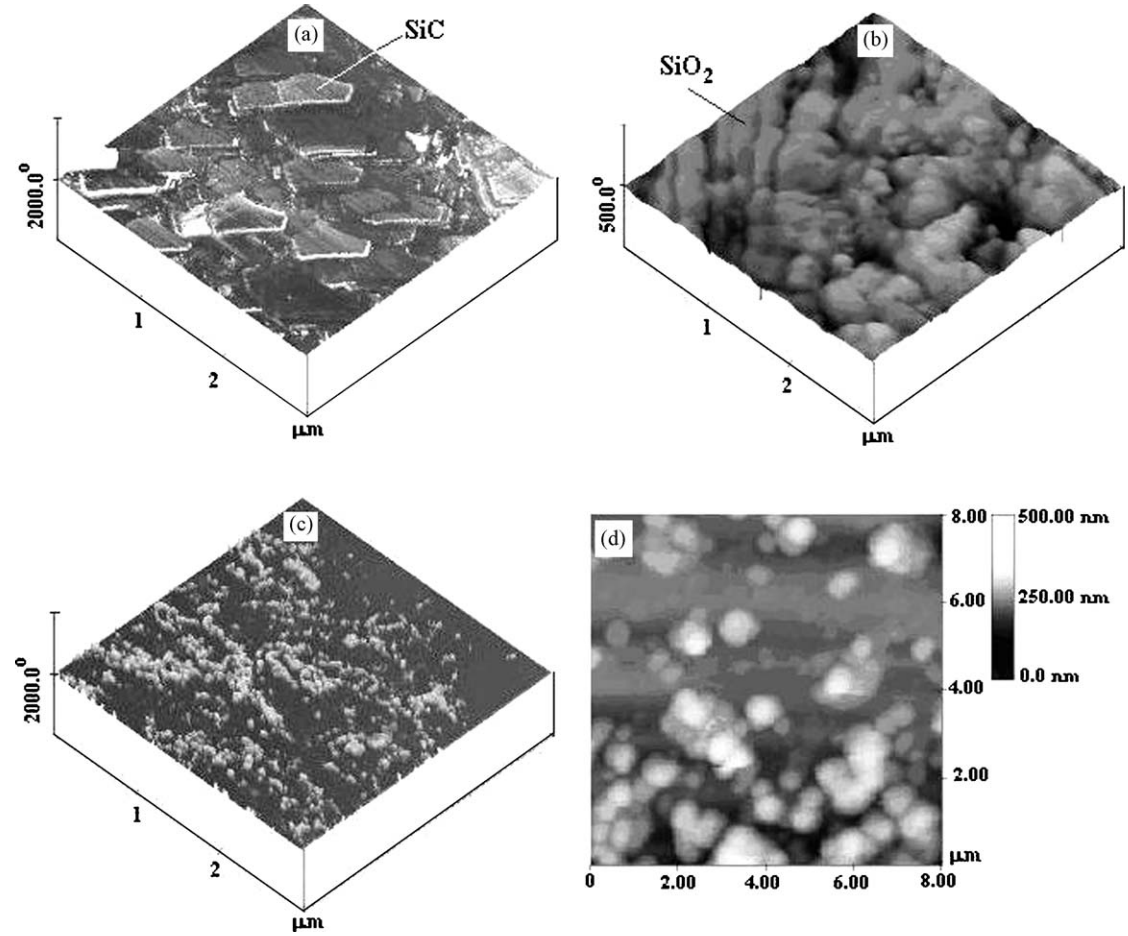

Fig. 2. AFM images of a surface of SiC ceramics obtained in the phase regime (a–c) and a deposited film obtained in the height (topography) regime (d). (a) Initial specimen; (b) specimen irradiated for 15 min; (c) and (d) specimen irradiated for 30 min.

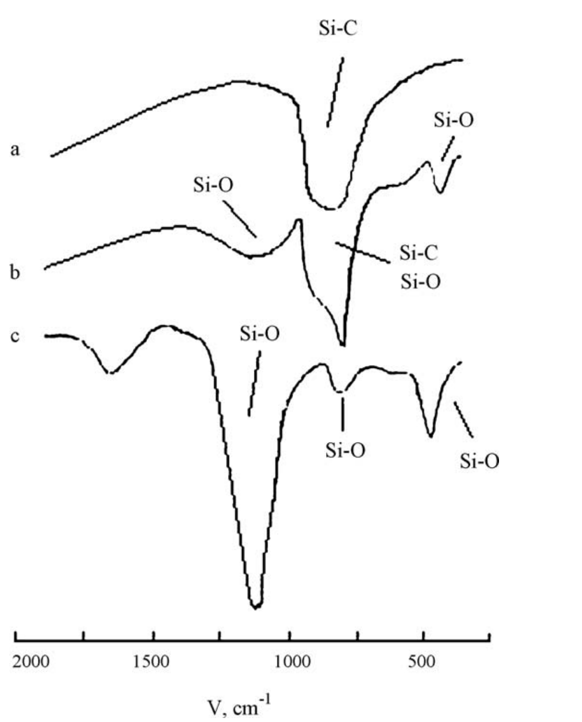

Fig. 3. IR spectra of untreated SiC ceramics (a), SiC ceramics irradiated for 15 min (b), and deposited films formed on the base of ablation product in air (c).

microanalysis, the deposit contains Si and O (see Table 1). The formed product can be assigned to SiO_2.

A layer pierced with pores was observed under the ''heat'', on the surface of the ceramic specimen (Fig. 6). The surface shows signs of the solidified melt. The presence of pores (cavities) at the apexes of domes indicates that gaseous products evolved from the depth (lower layers) and penetrated through the viscous melt. These products overblew the melt forming a dome and then broke it through. In cooling of the specimen, circular cracks formed on the smooth surface around the cones with pores. According to the electron-probe X-ray microanalysis data, Si, O, and C are present in this layer (see Table 1). It can be concluded that, along with the formed silica, the layer contains carbon.

A loose layer, which contains Si and O (see Table 1), is deposited on the surface of a collective plate (substrate) under irradiation (Fig. 7). The ratio of the Si content to the O content is close to the ratio of these elements in SiO_2. In the peripheral regions of the collective plate and inside the irradiation chamber, whiskers and fibers were observed. Particles deposited on thin fibers, which led to the formation of fibers of different thickness (Fig. 8). Note that when the layer of particles deposited on fibers increased in thickness, the morphology of the surface of fibers and the film became similar (see Figs. 7 and 8).

Fig. 4. Photograph of a SiC specimen laser-irradiated at $P = 170$ W.

Absorption bands characteristic of cristobalite, fused quartz, and silicon oxide films are observed in an IR-spectrum of the ablation product, in the frequency range 400–2000 cm^{-1} (Table 2 and Fig. 9).

Above results show that, in laser irradiation of the SiC target in air, the main processes are heating of the surface, oxidation–corrosion of silicon carbide, and ablation of oxidation products.

It is known that in heating of SiC ceramics in the temperature range 800–1600 °C in an oxygen-containing atmosphere, its surface oxidizes to SiO_2, and CO_2 is released [3,19–21].

At $T > 1600$ °C, the interaction of SiC with SiO_2, SiC evaporation, and silica dissociation [3,19–21] are initiated,

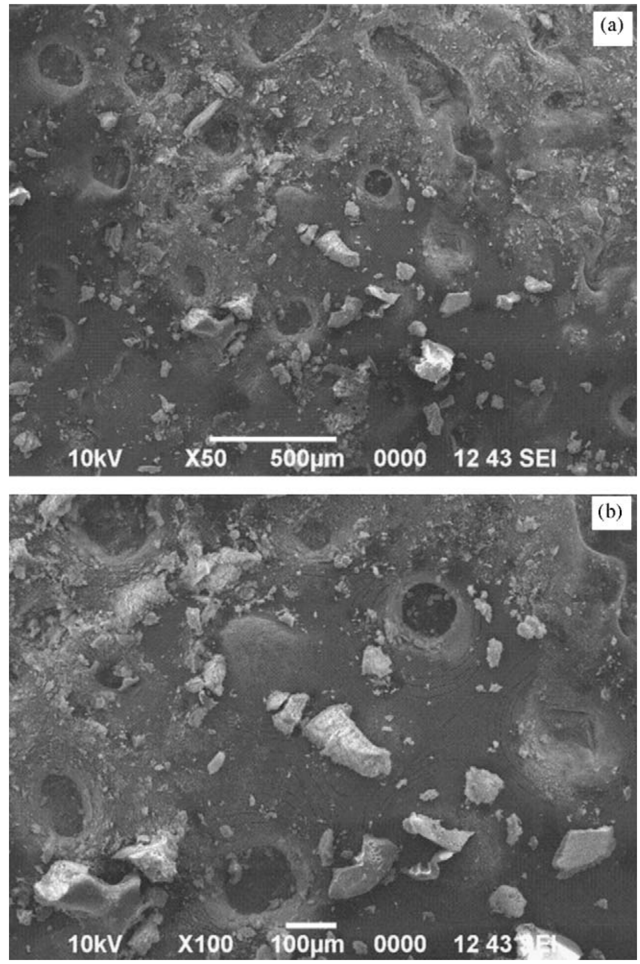

Fig. 6. Micrographs of a surface formed under a deposit on α-SiC ceramics (a and b).

which is accompanied by the appearance of SiC, Si, and SiO in the gas phase.

Analogous processes occur in laser irradiation of a SiC surface [22–24]. However, due to a substantial heat gradient from the specimen surface towards the interior, all processes are realized simultaneously. This leads to the ‘‘foaming’’ of the surface of melted SiO_2.

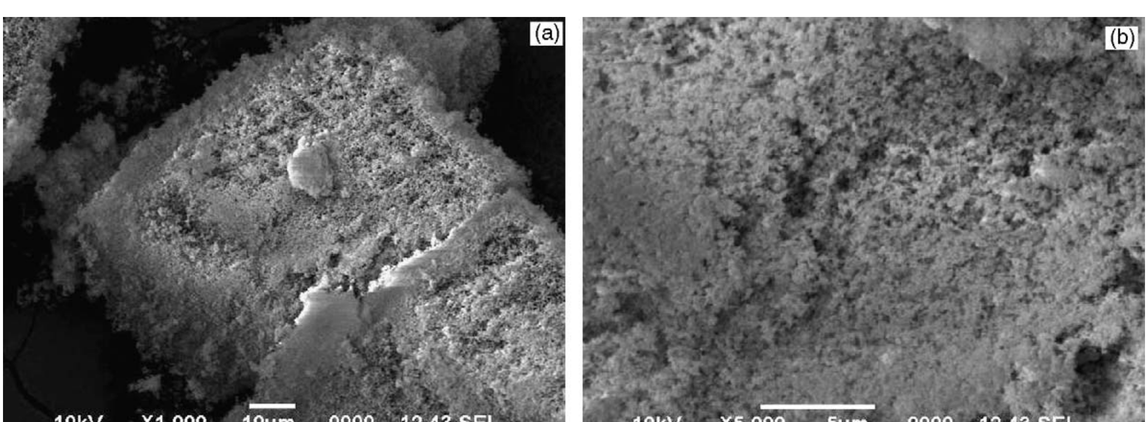

Fig. 5. Micrographs of a deposit formed on the surface of α-SiC ceramics (a and b).

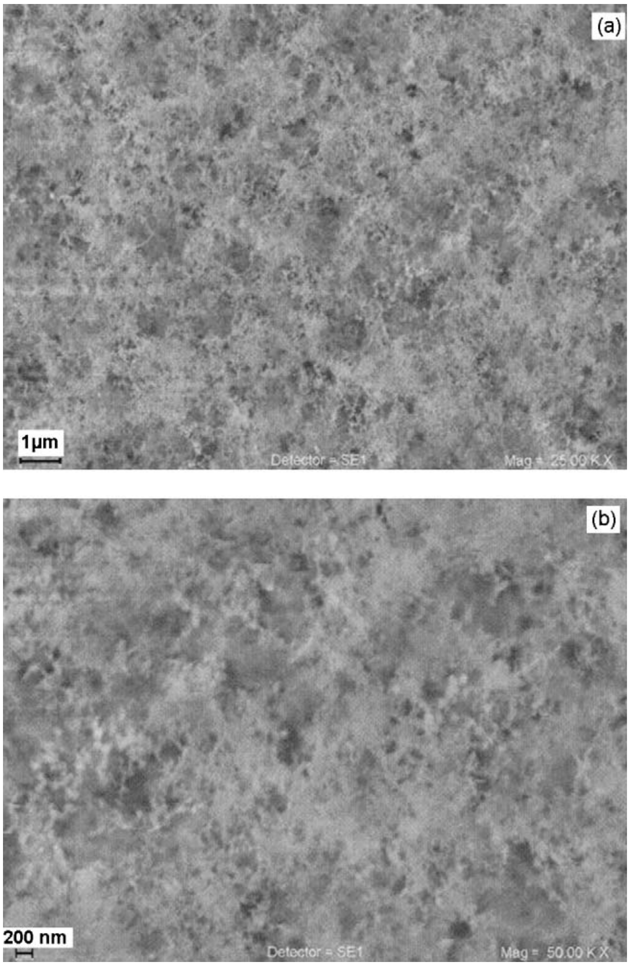

Fig. 7. Micrograph of a precipitated film formed on SiC ceramics under irradiation (a and b).

The formation of fibers in the periphery of the collective plate and in cooler zones of the irradiation chamber agree with data of [25], according to which the concentration of clusters in the periphery of laser plasma attains a critical value. The dynamics of clusters leads to their joining, a change in the aggregate state, the formation of cluster chains and fibers.

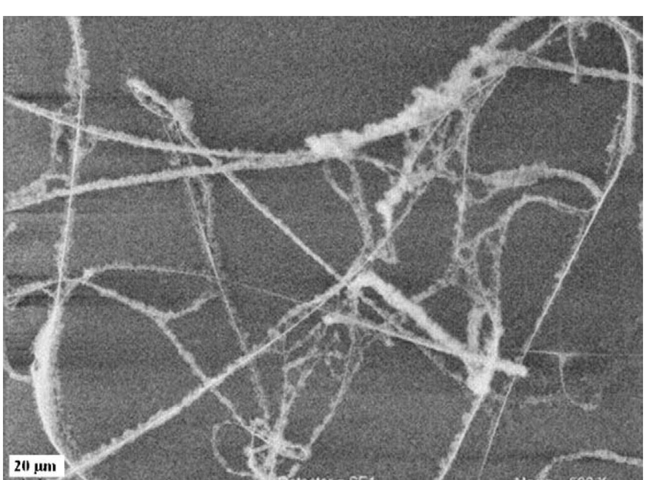

Fig. 8. Micrograph of fibers and whiskers formed under laser irradiation of SiC ceramics.

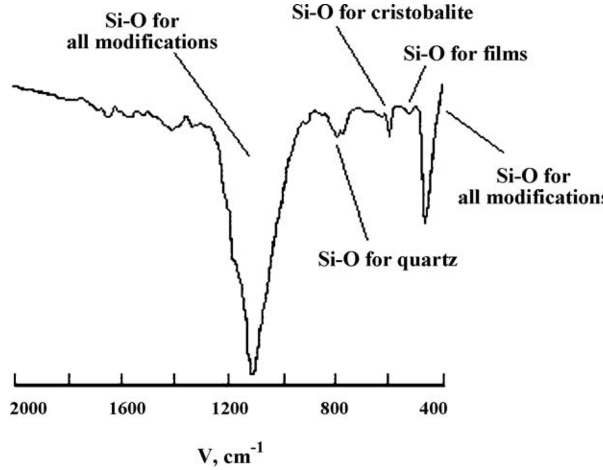

Fig. 9. IR absorption spectra of ablation products obtained in air at P = 170 W.

4. Conclusions

In the laser irradiation of SiC ceramics in an oxygen-containing atmosphere, oxidation processes characteristic of its corrosion in the temperature range 800–3000 °C occur.The formation of porous silica on the surface of SiC ceramics is due to the release of gaseous products through the layer of SiO_2 melt.A porous SiO_2 film forms on the base of ablative products.

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