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The laser-prepared SiC nanocoating: preparation, properties and high-temperature oxidation performance

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Abstract

In this study, SiC nanocoatings were prepared on graphite substrates via a laser treatment process. Different laser energy densities were employed in the laser treatment. The surface morphology and elemental composition were systematically studied by scanning electron microscopy (SEM) and energy dispersive spectrometer (EDS). X-ray diffraction (XRD) and high-resolution transmission electron microscopy (TEM) were used to characterize the phase composition and coating microstructure. A high-temperature oxidation test was also conducted to determine their anti-oxidation performance. The results show that laser irradiation triggers the transformation from micro SiC particles into SiC nanocoating consisting of numerous polycrystalline SiC nanoparticles. At the laser energy density of 10.42 kJ cm$^{-2}$, the prepared SiC nanocoating reveals the best oxidation resistance at a high-temperature environment in tested samples. In addition to its dense and crack-free surface morphology, the formation of SiO$_2$ in the SiC nanocoating also helped to enhance the high-temperature oxidation resistance as a self-healing agent. Laser preparation of SiC nanocoating enhances the high-temperature oxidation resistance and protects the underneath graphite substrate, which serves as an efficient and effective manufacturing method for SiC protective coatings.

1. Introduction

The corrosion attack and high-temperature oxidation process cause serious damage to the graphite materials applied in nuclear reactors and scramjet engines [1–3]. Silicon carbide (SiC) materials have been extensively accepted as a protective coating on those graphite materials, due to their high-temperature strength, radiation tolerance, neutronic transparency, and oxidation resistance [4, 5].

Previous studies have fabricated SiC–based protective coatings via different methods including slurry, sol-gel, vapor deposition, embedding, and thermal spraying methods [6–14]. For instance, Hui Yang and co-workers [15] used the embedding method and prepared SiC/YSiC composite coatings with improved oxidation resistance under a high-temperature at 1000 °C–1100 °C. Lianfeng Huang and co-workers [16] fabricated Si–SiC and SiC–Si–ZrB$_2$ coatings by similar methods, where the authors reported compact surface structure and great adhesion of prepared coating. In other reports [17–20], chemical deposition was employed to prepare a SiC coating, yet it encounters problems from the time-consuming and high-temperature preparations. The recently developed chemical vapor reaction (CVR) avoided these drawbacks and provided strong adhesion and great oxidation resistance, while the coating quality largely depends on the surface conditions of graphite substrates [21–23]. However, the above processes reveal similar drawbacks such as long preparation periods and complicated process procedures.
Laser technology has been adopted to prepared SiC nanocomposite coatings in recent reports [24–27]. For instance, G Socol [24] employed pulse laser irradiation to prepare SiC films on silicon substrate under different methane gas pressures. In another report, C Y Cui [25] assembled SiC nanoparticles on aluminum substrates and fabricated SiC nanocomposite coatings by melting the substrate with laser irradiation. In general, the SiC preparation by laser technology possesses the advantages such as ultrafast heating, good controllability, excellent repeatability, high instantaneous energy, and environmental friendliness [26–28]. However, rare studies have been conducted for the SiC coatings directly made on graphite substrates. Inspired by this, this study proposes a laser technique that directly prepares SiC nanocoatings on graphite substrates. Compared with the traditional coating methods, the proposed laser treatment method should give rise to efficient preparation process and controllable properties. The almost in situ phase transition under laser irradiation straightforwardly generates nanocoating on a graphite surface. This study systematically discusses the properties of the laser-prepared SiC coating on graphite substrates, which would serve as good reference for future research on nanostructured SiC coatings and laser-assisted nanocoating preparation techniques.

2. Experimental

2.1. Sample preparation

SiC powders (40 μm average size) with 99.9% purity were used. A pure graphite sheet at the dimension of 10 mm × 10 mm × 4 mm was employed as substrate loading these micro-size SiC particles. Figure 1 presents the surface morphology of SiC powders loaded on a graphite substrate. The thickness of the particle layer is ~0.3 mm.

To prevent the graphite substrate from being oxidized by high energy laser, argon was used as the protective gas in the reaction chamber. A lens was placed on the upside of reaction chamber for laser irradiation. To remove oxygen, the argon wash procedure was conducted three times. Also, laser treatment was conducted on a titanium plate to eliminate the oxygen in the reaction chamber, before the preparation process for SiC coatings.

An IPG continuous fiber laser (PS-YLR-500) source with 500 W maximum output and a galvanometer scanning system was used in this experiment. The laser treatment was conducted at 20 kHz frequency, 120 μm spot size, and 298 mm field mirror focal length. The selected laser wavelength is 1060 ∼ 1090 um in our experiments. The graphite sheet loaded with micro SiC particles was placed in the objective table, and argon gas flow of 10 l min⁻¹ was applied to the whole device. In laser treatment, the empirical equation of the energy density output during laser irradiation is as follows:

\[ Ps = \frac{P}{dv} \]  

(1)

\( Ps \) stands for energy density (kJ/cm²), and \( P \) is output power (W). \( d \) is laser spot diameter (mm), and \( v \) is scanning speed (mm/s). According to the above equation, the laser output power is positively proportional to its energy density. In our experiments, the range of laser power was between 180 W to 260 W, and the laser scanning speed was 20 mm s⁻¹ with a scanning distance of 0.06 mm. Table 1 summarizes the laser treatment parameters employed in our sample preparation process.
Table 1. The selected laser treatment parameters.

| Sample number | Scanning speed (mm s⁻¹) | Laser power (W) | Energy density (kJ cm⁻²) |
|---------------|-------------------------|----------------|-------------------------|
| 1             | 20                      | 180            | 7.50                    |
| 2             | 20                      | 200            | 8.33                    |
| 3             | 20                      | 210            | 8.75                    |
| 4             | 20                      | 220            | 9.17                    |
| 5             | 20                      | 230            | 9.58                    |
| 6             | 20                      | 250            | 10.42                   |
| 7             | 20                      | 260            | 10.83                   |

2.2. Sample characterization

The high-temperature oxidation resistance was tested in a muffle furnace. The samples were taken out from the muffle furnace at the specific temperature (600 °C, 625 °C, 650 °C, 675 °C, 700 °C, 725 °C, 750 °C) every 20 min and cooled at room temperature. The mass change was measured by electronic balance (0.01 mg). The mass change rate (ΔX) was calculated as follows:

\[
ΔX = \frac{m_2 - m_1}{m_1} × 100\%
\] (2)

Where \( m_1 \) and \( m_2 \) represent the coating mass before and after high-temperature oxidation. The oxidation resistance of the coating was characterized according to its mass change rate for prepared samples.

A field emission scanning electron microscope (SEM, Hitachi SU3900, Japan) was used to observe surface microstructures. The embedded energy dispersive spectrometer (EDS) was used for element analysis. The phase composition was detected by an x-ray diffractometer (XRD, PANalytica X’Pert PRO, the Netherlands). A transmission electron microscopy (TEM, Philips–FEI Tecnai G2, the Netherlands) was used for further microstructure analysis.

3. Results and discussion

3.1. Structures and phase constituents

Figure 2 shows the morphology of SiC nanocoatings prepared under different laser energy densities. The nanostructured SiC nanocoatings were fabricated despite the distinct morphologies. In figure 2(a), SiC nanoparticles with an average diameter of 60–70 nm were generated on the graphite substrate at the laser energy density of 7.5 kJ cm⁻². The nanoparticle size gradually increased as the laser energy density increased, as shown in figures 2(b) and (c). The SiC nanoparticles with irregular shape were observed at 9.17 kJ cm⁻² as shown in figure 2(d). It is assumed that grain growth directions were irregular due to the uneven distribution of laser energy under this condition. Further increasing the energy density led to a compact SiC coating consisting of sphere SiC nanoparticles, as indicated in figures 2(e) and (f). Meanwhile, the aggregation of nanoparticles was found in the SiC coating by applying 10.83 kJ cm⁻² energy density in figure 2(g). The rather high laser energy caused excessive surface energy for the generated SiC particles which then aggregate with each other to maintain the system stability.

Figure 3 illustrates the formation pathway of SiC nanocoatings in laser treatment process. In the reaction chamber, the micro SiC particles irradiated by pulse laser energy were transformed into the Si and C plasma [29] which aggregated near the graphite surface. Under this condition, the pressure increased and reached a supersaturation state in the airtight reaction chamber, along with a rising Gibbs free energy. When laser irradiation was finished, it is assumed that the SiC nucleus was generated and then grown up from the supersaturated Si and C plasma, along with the decreased system Gibbs free energy and chamber pressure. By reaching a new balance in the reaction chamber, a protective nanocoating consisting of numerous SiC nanoparticles was formed on the surface. Herein, the SiC nanocoating was successfully prepared on the graphite substrate. It is noted that some newly generated SiC particles were attached on the chambers after the laser treatment, which further confirms the gasification/solidification process during the generation of SiC nanocoating.

The phase constituents were characterized for the prepared SiC nanocoatings as shown in figure 4. There appeared several \( β\)-SiC diffraction peaks in all XRD patterns, where the sharpest SiC peak was detected at 35.6°. It should be noted that no C peak was detected in the phase patterns, which means the good coverage of prepared SiC coatings on graphite substrates. It indicates that, at the laser energy density of 7.5 \( \sim \) 10.42 kJ cm⁻², a nanocoating made up of \( β\)-SiC was generated on the surface of graphite substrate.
Figure 5 presents the HRTEM images for the prepared SiC nanoparticles. SiC nanoparticles were densely packed and formed a nanocoating as observed in figure 5(a). Higher magnification in figure 5(b) revealed that lattice fringes with an interplanar distance of 0.261 nm. This confirmed the formation of SiC, which was consistent with the XRD analysis. The embedded electron diffraction pattern showed many bright spots, indicating its polycrystalline structure. An increase in laser energy density resulted in larger particle size and more uniform distribution, as indicated in figure 5(c). The HRTEM image showed the interplanar distance was 0.265 nm, confirming the formation of SiC nanoparticles with a polycrystalline structure.

Figure 6 shows the TEM images of SiC nanoparticles prepared at a higher laser energy density. Figure 6(a) and (b) present the samples irradiated at 9.17 kJ cm$^{-2}$, where a larger particle size was observed. Figures 6(c) and (d) presents the samples irradiated at laser energy densities of 10.42 kJ cm$^{-2}$. Under this condition, the generated
nanoparticle had a uniform shape and a further increased size. It should be noted that a small amount of SiC nanofibers along the heat dissipation direction was also formed due to the high laser energy in figure 6 (c).

The TEM analysis confirmed the formation of polycrystalline SiC nanoparticles after the laser treatment. The increase in laser energy density causes an increase in nanoparticle size. The free energy in the isolated chamber becomes increasingly higher under the laser treatment, while the generation and growth of SiC nanocoating can offset this trend. At a high laser energy, the high free energy gives large driving force for grain growth of SiC nanocoating and therefore causes the larger crystals.

3.2. High-temperature oxidation performance
The anti-oxidation capability of as-prepared samples at high temperatures was tested through high-temperature oxidation experiments. Figure 7 exhibits the curves of the oxidation weight-loss rate to an escalating temperature, in which the weight-loss rate of both coated and uncoated graphite substrates fabricated at different energy densities was discussed. Since the heat-treatment below 600 °C showed negligible variation in sample mass, only weight-loss curves above 600 °C are provided in this paper. As shown in the curves, the existence of SiC nanocoating improved the high-temperature oxidation resistance of the graphite materials. The weight-loss rate of pristine graphite (that is, without coating) was 15.6% at 750 °C, while that of the coated sample, prepared with a laser energy density at 8.33 ∼ 10.42 kJ cm⁻², decreased by 7 ∼ 11%. Therefore, the graphite substrates coated with a nano-SiC layer could be protected from oxidation.

The improved oxidation resistance can be attributed to the barrier effects of nano-SiC layer to O₂ and the filling of micropores and micro-cracks with SiO₂. A rational explanation of the latter is that the SiC on surface is partially oxidized into SiO₂ in the high-temperature oxidation process. The as-oxidized SiO₂ can fill the micro-
pores and cracks on the substrate. As the eroded SiC coating exhibits such self-healing effects, the SiC nanocoating layer shows superior inhibition against the erosion from O$_2$, thereby boosting the high-temperature oxidation resistance and protecting graphite substrates. At a laser energy density of 10.42 kJ cm$^{-2}$, the as-prepared coating had the strongest resistance towards oxidation. This is because, under this condition, the silicon carbide particles with appropriate density could be obtained, which effectively circumvents the invasion of oxygen molecules into the graphite substrate.

Figure 8 exhibits the morphology of coating surfaces treated at different laser energy densities before and after oxidation at 750°C. Figure 8(a) presents the SiC coating obtained by applying a laser energy density at 10.42 kJ cm$^{-2}$. We should note that similar surface morphologies were observed for all the SiC coatings prepared by laser treatment, revealing the flat surface with no appreciable cracks and pores. Notably, after a heat treatment at 750°C for 30 min, micro-sized pores and cracks emerged on the surface of SiC coating treated under 8.33 kJ cm$^{-2}$ irradiation, figure 8(b). The micro-sized pores and cracks were relatively rough, leading to a decrease in the density of coating layers and the subsequent invasion of oxygen into graphite substrate. The overall oxidation erosive behavior on graphite substrate gives rise to a considerable weight-loss rate. Figure 8(c) shows the surface morphology of irradiated samples with a laser energy density at 9.17 kJ cm$^{-2}$ after high-temperature oxidation. Apparently, fewer cracks were observed on the SiC coating and the coating surface was relatively flat, which is consistent with the slight improvement in high-temperature anti-oxidation performance. The surface morphology of SiC coating irradiated with a laser energy density at 10.42 kJ cm$^{-2}$, as displayed in figure 8(d), showed even fewer pores and cracks. Under this condition, the laser irradiated SiC coating was rather dense and compact, compared with other samples.

Combined with the corresponding curve in figure 7, when a laser energy density of the irradiation was 10.42 kJ cm$^{-2}$, the SiC coating’s functionality that preserves graphite substrates from being oxidized at a high temperature was improved most. One possible explanation is the proper density and compactness of the coating obtained under this condition. More specifically, the coating layer treated with lower laser energy density is less compact, and it is prone to form micro-sized pores and cracks in SiC coating at high-temperature oxidation.
Figure 6. TEM images of prepared SiC nanoparticles after the laser treatment of (a) and (b) 9.17 kJ cm$^{-2}$, (c) and (d) 10.42 kJ cm$^{-2}$.

Figure 7. Weight loss range of the uncoated graphite substrate and SiC nanocoating heated above 600 °C.
With the heating time prolonged, cracks are much likely to spread out and eventually turn into devastating fractures, thereby rendering the oxidation resistance of coating deteriorated.

In order to further analyze the coating surface after high-temperature oxidation, samples treated after the oxidation tests were systematically characterized by high magnification SEM images, as depicted in figure 9. Many spherical aggregates can be observed after the oxidation tests, as shown in figures 9(a), (c) and (e). However, the SEM images as shown in figures 9(a) and (c) reveal the apparent groves in both SiC nanocoatings irradiated at 8.22 and 9.17 kJ cm\(^{-2}\), after the high-temperature oxidation at 750 °C for 30 min Figures 9(b), (d) and (f) exhibit the high-magnification (that is, 50 K times) images of these spherical aggregates, it can be noted the morphology of SiC particles did not change significantly and remained at nanoscale after high-temperature oxidation.

By applying a relatively low laser energy density (e.g. 8.22 and 9.17 kJ cm\(^{-2}\)), the laser scanning causes different thermal expansion coefficients in the scanned areas and the transition areas between laser trajectories, due to the non-uniform heat distribution in such process. The generated SiC undergoes an irreversible growth during the whole high-temperature laser treatment. Such mismatch of thermal expansion coefficient finally leads to increased surface stress and the formation of cracks in as-prepared SiC nanocoatings. During the high-temperature oxidation, the dramatic high-temperature thermal expansion enlarges the cracks and then oxygen invades the inner graphite substrate. This results in the formation of surface grooves shown in the SEM images, which indicating their inferior high-temperature oxidation resistance.

Meanwhile, the higher energy density of 10.42 kJ cm\(^{-2}\) gives increased surface energy to these generated SiC nanoparticles during the laser treatment and the high residual heat remaining in the generated particles after the treatment. This decreases the possibility of crack formation and results in a dense and compact SiC nanocoating. This optimal coating, which is robust enough to avoid the formation of cracks, is effective to function as a barrier against O\(_2\) in subsequent high-temp oxidation. Thus, a distinct surface morphology with no deep grooves could be observed after high-temperature oxidation tests in figure 9(e), indicating the superior high-temperature oxidation resistance of SiC nanocoating.

To compare the difference in the structural and chemical composition of nanocoating obtained before and after high-temperature oxidation, the SiC nanocoating exhibiting the best oxidation resistance (this is, the SiC irradiated with a laser energy density of 10.42 kJ cm\(^{-2}\)) was characterized, as shown in figure 10. From figures 10(a) and (c), it can be observed that the coating layer was more compacted before oxidation, while some shallow grooves formed in the transition area (that is, the area between laser trajectories) of laser treatment. As shown in figure 10(a), the original grooves were thinner and relatively smooth, being less likely to form cracks.
As can be seen from figures 10(b) and (d), the higher-magnification microstructure of the coating was quite similar before and after oxidation, despite there was a slight increase in particle size after oxidation. The variation of elements distribution caused by the high-temperature oxidation was also determined. Figure 11 shows the element mappings undertaken for SiC coatings obtained before and after high-temperature oxidation. Oxygen could be hardly detected in the coating obtained before oxidation, which indicates that the coating was composed of SiC particles. After the high-temperature oxidation, the oxygen content of coating surged to 40.49%. Such increase in oxygen content means that the co-existence of substantial SiO$_2$ in addition to SiC particles, despite the accurate quantitative detection is not applicable in EDS analysis. The coefficient of thermal expansion of SiO$_2$ ($0.5 \times 10^{-6}$ K$^{-1}$) is much smaller than that of SiC ($4.7 \times 10^{-6}$ K$^{-1}$), and SiO$_2$ could function as a self-healing agent at high temperature. Thus, the co-existence of SiO$_2$ in the coating layer could hinder the crack expansion and avoiding the further oxidation of the coating layers.

The above analysis is based on experiments and tastings. In addition to these, a theoretical discussion was also conducted regarding the prepared anti-oxidation coatings by applying the Arrhenius equation. Figure 12 shows the linear fitting curve plotted through the Arrhenius equation. Higher activation energy indicates a stronger antioxidant ability [28, 29]. Thus, the Arrhenius formula can be expressed as follows:

$$\ln k = -\frac{E_a}{RT} + \ln A$$ \hspace{1cm} (3)

where $T$ is the absolute temperature, $k$ represents the reaction rate constant at the temperature of $T$, $A$ refers to the Arrhenius constant, $R$ is the molar gas constant, and $E_a$ is called the experimental activation energy which can generally be regarded as a temperature-independent constant. The oxidation resistance of the coating can be
Figure 10. Surface morphology of SiC nanocoating (a) and (b) before, and (c) and (d) after high-temperature oxidation conducted at 750 °C for 30 min ($P_s = 10.42 \text{ kJ cm}^{-2}$).

Figure 11. Distribution of elements in the nanocoatings before and after the high-temperature oxidation conducted at 750 °C for 30 min ($P_s = 10.42 \text{ kJ cm}^{-2}$).
verified by calculating the activation energy of the corresponding reaction. According to equation (3), when \( P_s = 8.33 \text{ kJ cm}^{-2} \), the calculated \( E_a = 0.939 \times 10^5 \text{ J mol}^{-1} \). When \( P_s = 9.17 \text{ kJ cm}^{-2} \), \( E_a = 1.147 \times 10^5 \text{ J mol}^{-1} \). When \( P_s = 10.42 \text{ kJ cm}^{-2} \), \( E_a = 1.198 \times 10^5 \text{ J mol}^{-1} \). From the above calculations, the maximum activation energy was obtained when the \( P_s \) equals 10.42 J cm\(^{-2}\), which is consistent with the experimental results of high-temperature oxidation resistance tests.

4. Conclusion

In this study, the method of laser irradiation was employed to transform the micron SiC particles into SiC nanocoating on the surface of graphite substrate. In general, the high laser energy density resulted in densely coated SiC nanoparticles with a large size and a uniform shape. However, agglomeration of the coated nanoparticles was found when applying an excessively higher energy density. All the prepared SiC nanocoatings revealed a polycrystalline structure. The high-temperature oxidation tests showed the SiC nanocoating prepared at 10.42 kJ cm\(^{-2}\) possessed the best oxidation resistance in examined samples. In addition to its compact and dense coating surface, the formation of SiO\(_2\) in the SiC nanocoating also helped to enhance the high-temperature oxidation resistance as a self-healing agent. The theoretical analysis well-matches the obtained experimental results, showing the best oxidation resistance at the laser energy density of 10.42 kJ cm\(^{-2}\). This study proves that laser treatment is an efficient and effective manufacturing method for SiC protective coatings on graphite substrates.

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Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

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