

Article

Novel Method for Deposition of Gas-Tight SiC Coatings

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Abstract: A new high-temperature method for the deposition of gas-tight silicon carbide protective coatings with low gas permeability has been developed. The free carbon atoms form during the high-temperature pyrolysis of hydrocarbon molecules. In turn, carbon reacts with molten silicon contained in the subsurface substrate layers and/or with silicon vapor. The source of silicon vapor serves the molten silicon in the heated zone of a reactor furnace. Such coatings effectively protect SiC-C-Si and SiC-C-MoSi₂ ceramics, carbon-carbon composite materials, structural graphite, and refractory metals and alloys from oxidation. The conducted tests show the high thermal oxidation and thermal shock stability of deposited protective coatings, as well as their good adhesion to the substrates.

Keywords: silicon carbide; coatings; deposition; high-entropy alloys; gas permeability; thermal oxidation; thermal shock stability



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1. Introduction

Graphite, carbon-carbon composite materials (CCCMs), and silicon carbide-based composites containing carbon are widely used in the design of various devices, machines, and mechanisms. In particular, they are used in the development of aviation and rocket technologies. The application of these materials is limited to temperatures below 450–500 °C. At higher temperatures, carbon is oxidized by the oxygen contained in the air. High-density silicon carbide (SiC) ceramics (such as reaction-bonded silicon carbide) usually contain some residual silicon. This fact limits their applicability due to thermal oxidation or degradation when operating in an alkaline environment.

Therefore, the development of high-temperature protective coatings on carbon- and silicon-containing materials is an urgent task [1]. These so-called “functional coatings” can be used for the following purposes:

- Isolation of the substrate material from contact with an oxidizing atmosphere in order to prevent corrosion;
- Increasing the wear resistance of the surface of the product;
- Increase in the strength of the product;
- Surface engineering, etc.

Silicon carbide is one of the most commonly used materials for protective coatings due to its resistance to temperature variations, high corrosion resistance, and good wear resistance at high temperatures in an oxidizing environment [2–4]. SiC thin films and coatings on high-temperature materials can be deposited by chemical vapor deposition (CVD) [5–8], metal organic chemical vapor deposition (MOCVD) [9], and reactive chemical vapor deposition (RCVD) [10]. SiC coatings are also deposited by high-speed plasma deposition [11–13], thermal evaporation [14], and magnetron sputtering [15,16]. Chemical surface modification [17], sol-gel technology [18], electrolytic and electrophoretic deposition from liquid solutions [19,20], plasma chemical vapor deposition [21–23], and reactive diffusion coating [24] are also used to deposit SiC.

CVD is the most common method for the deposition of protective SiC coatings on carbon-containing materials. This is because of its versatility and technological ripeness. CVD allows for gas-tight, homogeneous coatings to be obtained (on large-dimension parts) with precise thickness control and improved surface morphology. These properties ensure the good protective ability of SiC coatings. The disadvantages of CVD include the high cost of reagents, low deposition rates, and difficulties with adhesion when applied to some materials due to the impossibility of forming buffer layers.

There are also other methods for the deposition of SiC coatings. Thus, to protect carbon fibers and nanotubes from oxidation, as well as to form a buffer layer, the coating can be deposited in such a way that the surface of the carbon material does not deteriorate. This can be achieved if carbon is excluded from the reaction. For example, fibers can be impregnated with mixtures of reagents containing carbon and silicon subjected to hydrolysis, followed by heat treatment such as a gradual temperature increase up to 1400 °C [25]. Additionally, to obtain a composite material, the carbon powders can be coated with a SiC layer before subsequent sintering [26].

On the contrary, the participation of silicon in the formation of a coating on SiC ceramics with a high content of residual silicon permits the production of continuous SiC coatings when only a carbon-containing component is supplied. This approach ensures not only the protection of silicon-containing material, but also the manufacturing of hermetic (gas-tight) joints for ceramic parts [27]. Pastes or slurries consisting of a finer carbon filler fraction than the siliconized graphite being coated can also be used for deposition of SiC coatings. In this way, after siliconization, the dense SiC layer is formed on the surface of the product [28]. Due to the high content of SiC and reactive bonding with the protected material, such SiC coating exhibit good adhesion and wear resistance.

In this paper, we consider a new method for the deposition of tight SiC coatings with low gas permeability. Such SiC coatings permit improvements to the thermal oxidative resistance of ceramic materials containing residual carbon and silicon, as well as carbon–carbon composite materials, especially graphites and refractory metals and alloys. We also characterized the properties of materials, coatings, and products obtained using the developed deposition technology.

2. Materials and Methods

The technique for applying a gas-tight silicon carbide coating to a substrate material is based on the direct interaction of carbon formed during the high-temperature pyrolytic decomposition of hydrocarbon molecules (for example, methane) with a silicon melt contained in the surface layer of the material to be coated and/or silicon vapor. The source of silicon vapor is the silicon melt placed in the thermal zone of the furnace [29].

The layout of the reactor for deposition of a gas-tight SiC coating is shown in Figure 1. The process was carried out in a vacuum high-temperature furnace with a graphite heater and carbon thermal insulation. In the lower part of the thermal zone of the furnace, a crucible with silicon was located on a support, above which the coated part was placed upon special tooling. The furnace was evacuated using a fore-vacuum pump and was smoothly heated to a temperature of 1500–1800 °C until silicon melted in the crucible. After that, with the help of a gas flow regulator, methane was supplied to the thermal zone of the furnace. After reaching the required thickness of coating, the supply of methane to the thermal zone of the furnace was stopped and the heater was turned off. The coating growth rate can vary from 10 to 100 µm/h depending on the application mode and the type of surface to be protected. The vacuum supply system was left on until the furnace was completely cooled down.

Grain size of the SiC coating (from microcrystalline to nanoscale), the fraction of void, the atomic structure of grain boundaries, the composition of polytypes, and the morphology of SiC depend on the temperature in the interaction zone and the pressure of hydrocarbon gas. In this case, depending on the deposition conditions, one can controllably obtain either

coating from SiC nanoparticles with a controlled content of nanopores or poreless coatings consisting of nano- or microcrystals with uniform granulometric composition.

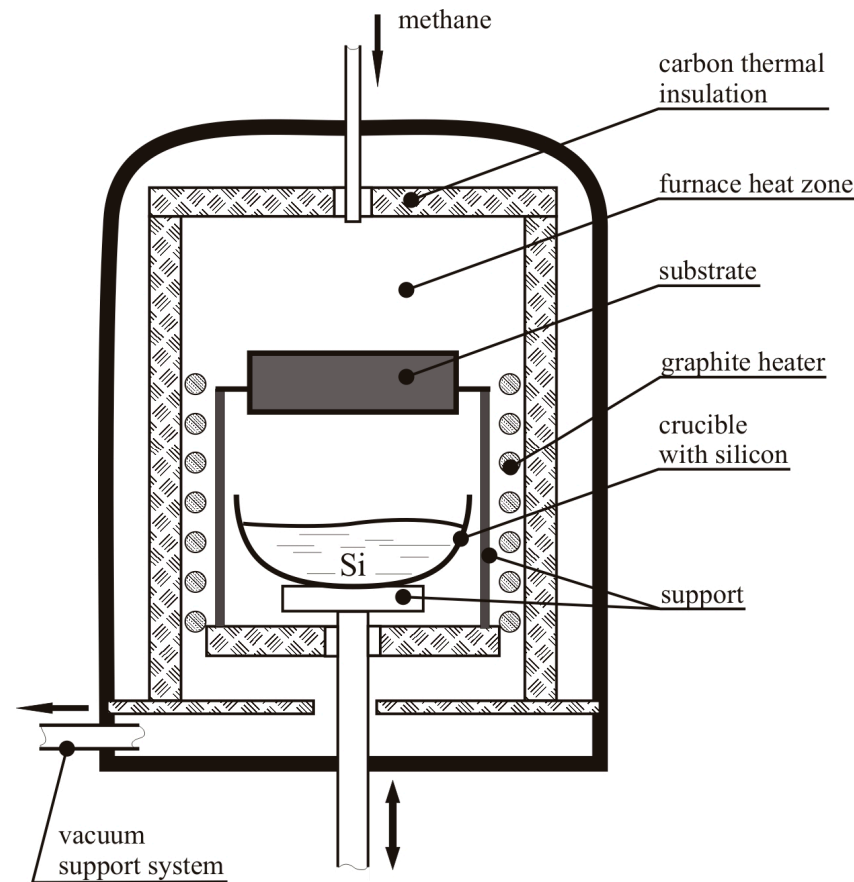


Figure 1. The layout of the reactor for deposition of gas-tight SiC coatings.

At low temperature (1500–1600 °C) in the reaction zone, the coating structure was formed of SiC nanoparticles ranging in size from 10 to 80 nm, which were agglomerated into clusters up to several tens of microns in size (Figure 2a). These clusters compose an ordered structure on the surface of the material, forming a porous coating in one or more layers (Figure 2b,c). Such coatings have significant advantages for a number of specific applications. For example, for materials operating under conditions of intense radiation, a large positive role is played by the presence of nanocavities, which ensure the rapid emergence and annihilation of radiation defects.

At higher temperatures (1700–1800 °C), gas-tight protective coatings are formed from micro- and nanocrystals (Figure 3).

Depending on the composition of the protected material, carbon and silicon can be used to form a coating both from external or internal sources. External sources are either a crucible with a silicon melt placed in the thermal zone of the furnace or carbon from the thermal decomposition of a gaseous hydrocarbon. Internal sources are residual silicon and carbon contained in the near-surface substrate layer. Information on the components involved in the formation of coatings on various materials is given in Table 1.

Application of the developed technique makes it possible to obtain air-tight polycrystalline silicon carbide coatings with thicknesses from 300 nm to hundreds of micrometers and even several millimeters. Depending on the feed rate, pressure, and temperature of the components in the interaction zone, it is possible to change the grain size of the SiC coating from dozens of nanometers to hundreds of micrometers.

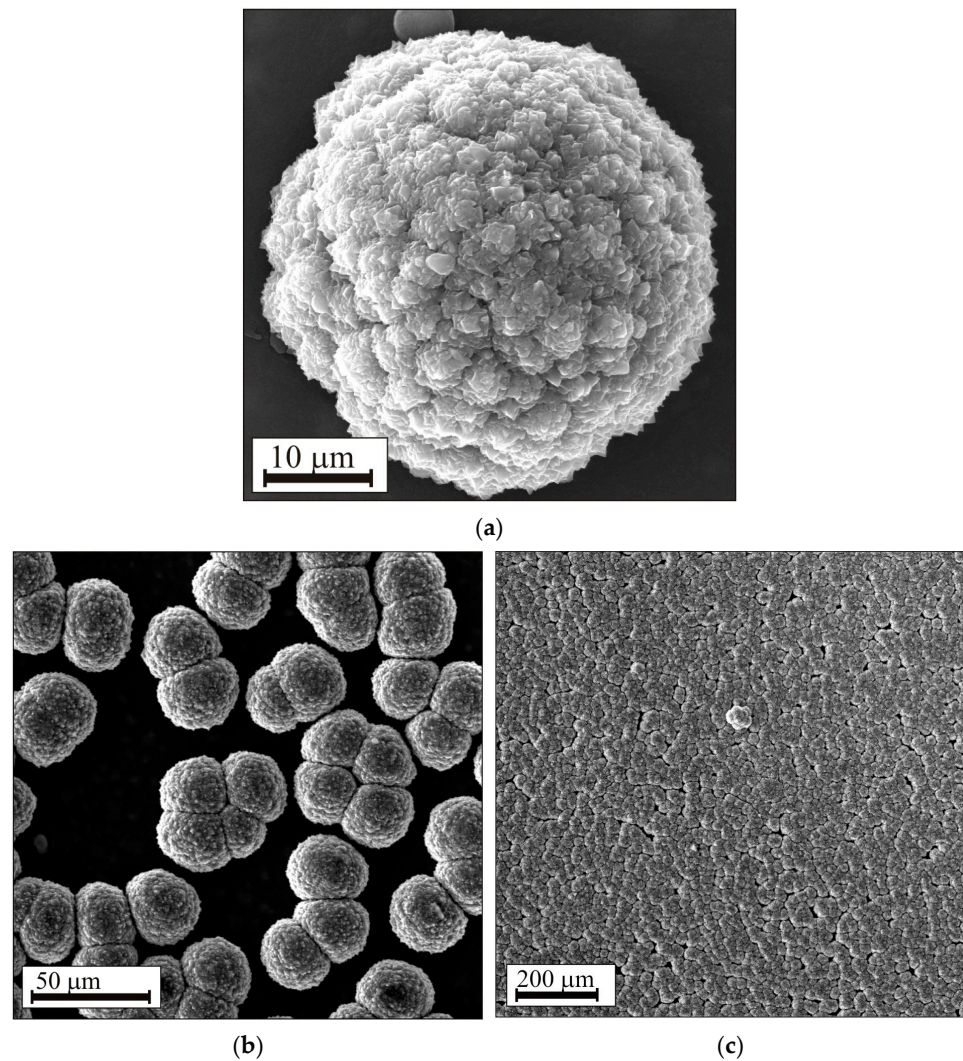


Figure 2. Porous coating formed of SiC nanoparticles from 10 to 80 nm in size, agglomerated into clusters. (a) SiC nanoparticles ranging in size from 10 to 80 nm agglomerated into clusters up to several tens of microns in size; (b) clusters in an ordered structure on the surface of the material; (c) resulted porous coating in one or more layers.

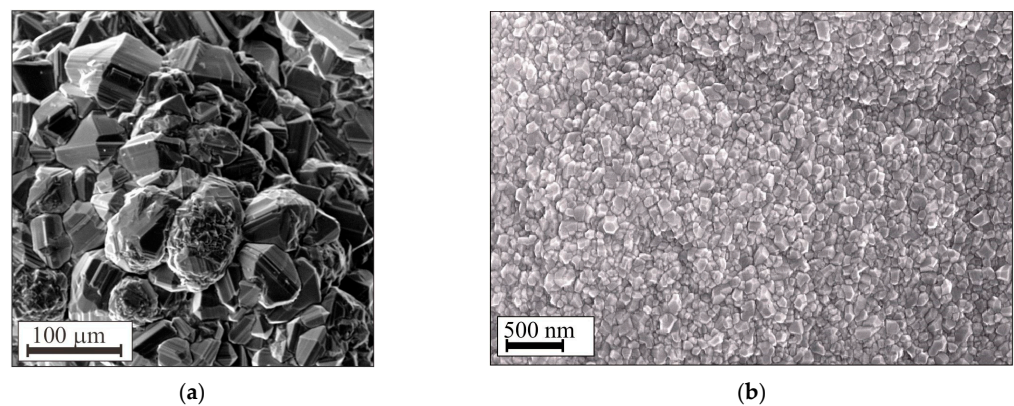


Figure 3. Gas-tight protective coatings formed by micro-F (a) and nanocrystals (b).

Table 1. Features of SiC coating deposition on various refractory materials.

Substrate Material	Carbon from the Substrate	Silicon from the Substrate	Carbon from the External Source	Silicon from the External Source
CCCMs and graphite	yes	no	yes	yes
SiC-C-Si, siliconized graphites	yes	yes	yes	yes
SiC-C-MoSi ₂	yes	no	yes	yes
Modified RSiC	no	yes	yes	yes
Metals and alloys	no	no	yes	yes

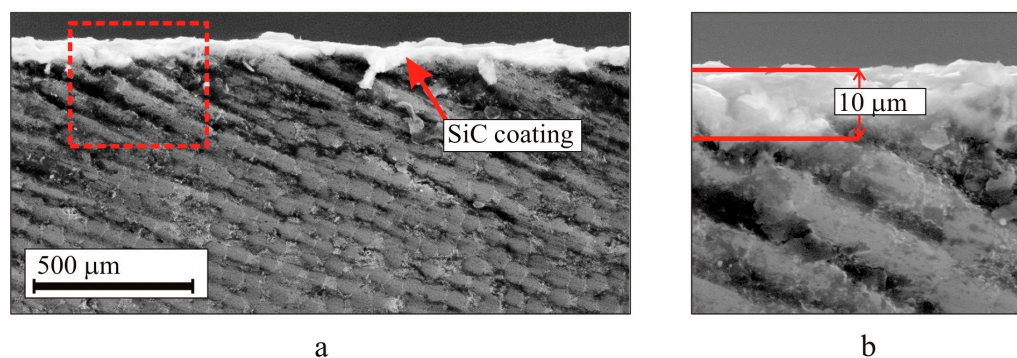
3. Results and Discussion

In this section we describe the deposition of protective SiC coatings on various high-temperature materials. The features of the formation of coatings on various materials and their influence on the characteristics of products are considered. Oxidation resistance at high temperatures, thermal shock resistance, and the influence on mechanical properties are described.

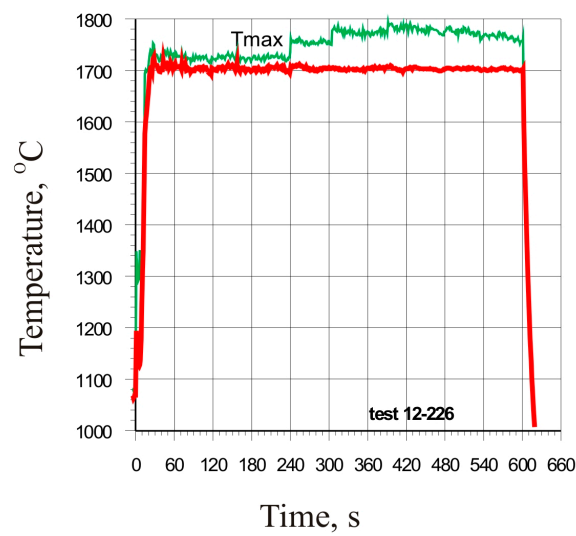
3.1. Deposition of Protective SiC Coatings on the Carbon–Carbon Composite Materials and Graphites

Carbon–carbon composite materials (CCCMs) and graphites have high strength and thermal stability in vacuum or inert environments. However, they can be oxidized by the oxygen in air at above 450–500 °C. The oxidation leads to corrosion and loss of strength and therefore limits the applicability of CCCMs and graphites. To protect CCCMs from oxidation, deposition modes were used, in which a dense SiC coating about 10 µm thick with a small grain size (80 nm) was formed. Such coating protects the substrate material from reactions with environmental components. The SiC coating also leads to a noticeable change in the size and mechanical characteristics of the coated composite material.

A CCCM with a SiC gas-tight coating is shown in Figure 4. The carbon from the surface layer of the CCCM partially participated in the formation of the SiC coating. This ensures the high adhesion of the SiC coating to the substrate and high protective properties with a thin coating.

**Figure 4.** Cross-section of a CCCM with a protective gas-tight SiC coating (a) and its enlarged fragment (b).

To determine the thermal oxidative resistance of the coatings, tests were carried out on a specially prepared CCCM sample with a SiC coating. The experimental conditions were as follows: air velocity head 4–6 GPa, temperature $T = 1700$ °C, test time of 10 min. Figure 5 shows the test thermograms for a CCCM sample with a protective SiC coating and its appearance before and after testing. Figure 6 shows the temperature distribution over the area of the sample. Despite the local overheating of the sample surface (Figure 4), the continuity of the coating was not disturbed. The weight loss of the sample after testing was 14.6 mg, which was less than 0.1% of the total weight of the sample. Tests for thermo-oxidative resistance demonstrated the promising features of the developed technique for deposition of silicon carbide coatings on CCCMs. The coating has good protective properties up to temperatures of 1700–1750 °C.



a



b



c

Figure 5. Test thermograms for a CCCM sample 50 mm in diameter with a protective SiC coating (a) and its appearance before (b) and after (c) testing.

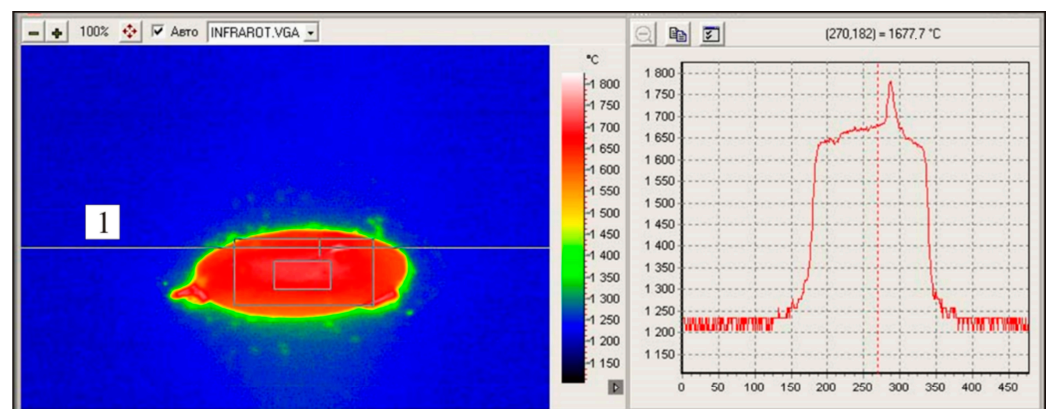


Figure 6. Temperature distribution over the area of the CCCM sample with a protective SiC coating.

Tight SiC coatings with low gas permeability were also deposited on graphite parts with various geometries to protect them from oxidation. The maximum dimensions of products to be coated are limited only by the dimensions of the thermal zone of the installation. Figure 7 shows examples of SiC coatings of uniform thickness that were deposited on graphite parts with various geometries.



Figure 7. Examples of gas-tight SiC coatings of uniform thickness on the large graphite parts with complicated geometries.

Graphite can be used as a removable substrate to obtain polycrystalline silicon carbide in complex shapes. Figure 8 shows SiC tubes and a magnetron sputtering target made by depositing a thick layer of silicon carbide on rectangular graphite rods and a plate. After coating, the graphite was removed by annealing in air at a temperature of 1100 °C. This technique allows you to obtain macroscopic articles of various configurations from nanostructured SiC.

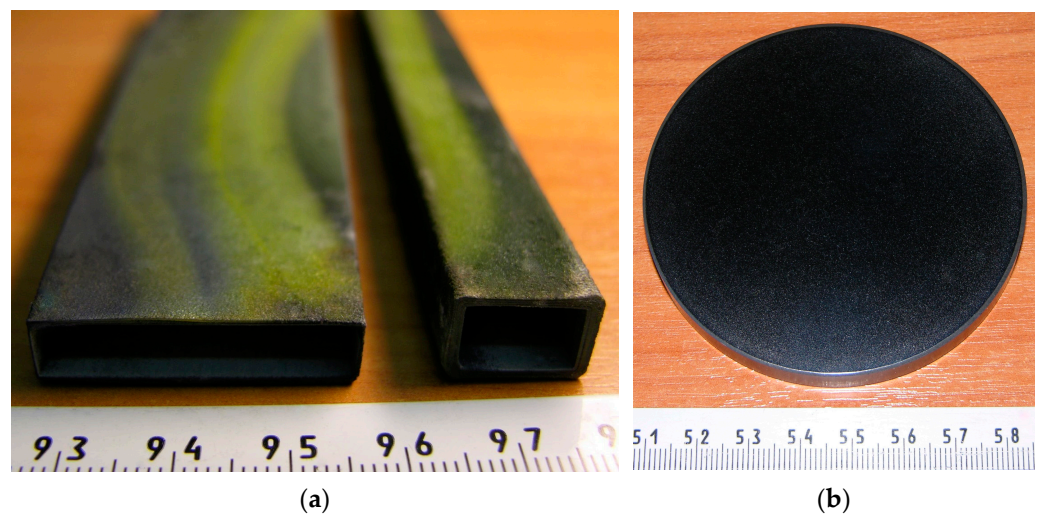


Figure 8. SiC rectangular tubes with a wall thickness of 1–1.5 mm (a) and a SiC magnetron sputtering target of 70 mm in diameter and 6 mm in thickness (b), which were obtained by deposition onto removable graphite substrates.

3.2. Deposition of Protective SiC Coatings on SiC-C-Si and Siliconized Graphites

Relatively light and wear-resistant SiC-C-Si ceramics are well suited for manufacturing complex-shaped parts. As for carbon materials, their operating conditions are limited to temperatures of 450–500 °C due to the oxidation of residual carbon. In the absence of residual carbon, their operating conditions are limited to temperatures of 1100 °C due to the oxidation of residual silicon. The atmosphere of water vapor and alkali metal salts accelerates the destruction of SiC-C-Si ceramics. Thus, a protective SiC coating should significantly limit the corrosion of such ceramics.

Polycrystalline SiC coatings with a thickness of 20–100 µm were deposited on the parts made of SiC-C-Si ceramics. These parts were produced by the impregnation of porous carbon matrices with liquid silicon. The porous carbon matrices had different residual silicon and carbon content in the volume [30,31]. It can be seen in the cross section of the coated substrate (Figure 9) that the part of the residual silicon in the near-surface open pores was involved in the formation of the coating. Single pores in the near-surface layer of the material do not have any noticeable effect on the mechanical properties of ceramics, especially in front of their significant increase from the presence of the SiC coating. The coating itself has multiple continuous bonds to the carbon and silicon carbide of the material, providing strong bonding to the substrate.

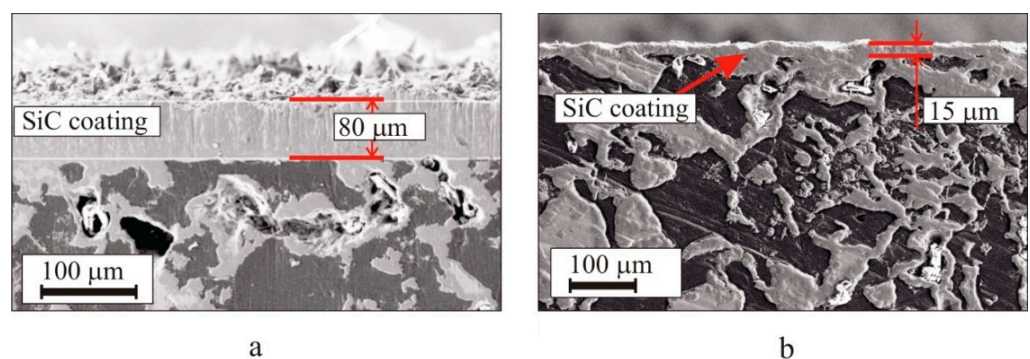


Figure 9. Cross section of a SiC-C-Si ceramic substrate with a deposited protective gas-tight SiC thick (a) and thin (b) coating.

To determine the temperature stability of protective SiC coatings on SiC-C-Si ceramics, the decrease in the mass of samples due to carbon burnout when exposed to high temper-

ature in an oxidizing atmosphere was evaluated. Figure 10 shows cross sections of the SiC-C-Si/50-40-10 ceramics without a protective coating (Figure 10a) and with a gas-tight SiC coating after annealing in air at 1000 °C for 60 h (Figure 10b). It can be seen that the structure of the material without a protective coating is severely damaged due to carbon burnout from the bulk of the material. Information about the changes in the weight of the samples is given in Table 2. The weight of parts with SiC coatings changed by less than 1%, which indicates that the coating has high functional quality.

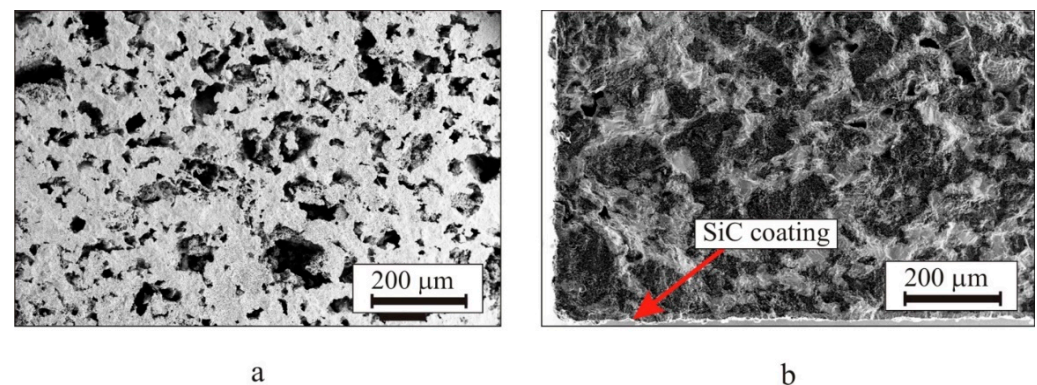


Figure 10. Microstructure of composite material SiC-C-Si/50-40-10 without a coating (a) and with a gas-tight SiC coating (b) after annealing at 1000 °C in air for 60 h.

Table 2. Weight of samples from SiC-C-Si/50-40-10 ceramics without a coating and with a protective SiC coating before and after annealing.

Sample Description	Weight of Sample before Annealing, g	Weight of Sample after Annealing, g	Weight Loss, %
Sample 1, without coating	29.35	18.76	36.08
Sample 2, without coating	25.19	16.33	35.17
Sample 3, with protective SiC coating	28.82	28.68	0.48
Sample 4, with protective SiC coating	24.21	24.05	0.66

The deposition of a SiC coating on SiC-C-Si ceramics can also improve the mechanical properties of the material by reducing the contribution of surface defects during bending and compression testing. Table 3 shows the measurement results of mechanical testing of the uncoated and coated SiC-C-Si specimens.

Table 3. Comparison of the strength characteristics of SiC ceramics with SiC-C-Si/50-40-10 composition without coating and with a gas-tight SiC coating.

Property	Without Coating	With Coating, 20 °C	With Coating, 1000 °C
Compression strength, MPa	398 ± 19	472 ± 11	483 ± 8
Bending strength, MPa	126 ± 6	204 ± 11	203 ± 7

The SiC film deposited according to the developed method is sufficiently conformal. In other words, its thickness on the walls of complex structures is comparable to the thickness of the film on the horizontal sections of these structures. This means that the method can be used to apply protective coatings on parts that are very complex in shape.

Using the developed method for manufacturing ceramic parts with complex shapes and the technique of applying protective SiC coatings, high-temperature equipment was manufactured. Namely, inserts for thermocouples were produced and used in the test bench for a gas turbine engine (Saturn, Rybinsk). Figure 11 shows the water-cooled metal case of a gas flow thermometer with five SiC inserts for the placement of thermocouples [32,33].

In the test bench for the gas turbine engine, a test cycle was carried out where the total operating time of the equipment was about 8 h; the thermogram (Figure 12) shows that the maximum measured temperature was 1465 °C.

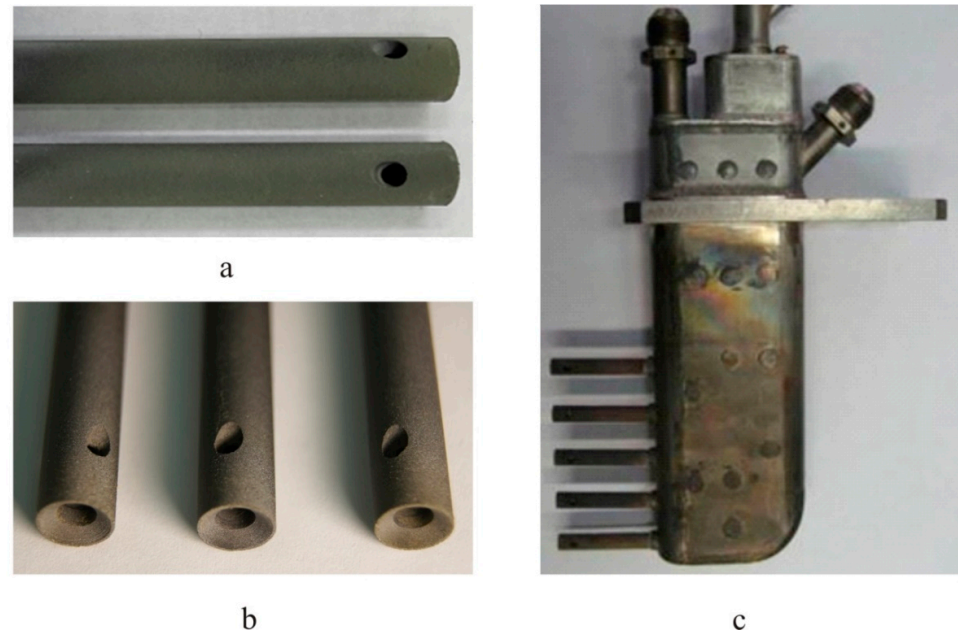


Figure 11. Thermal probes for temperature measurement in a high-temperature gas flow with brake chambers (a,b) and a comb with thermal probes as an assembly (c).

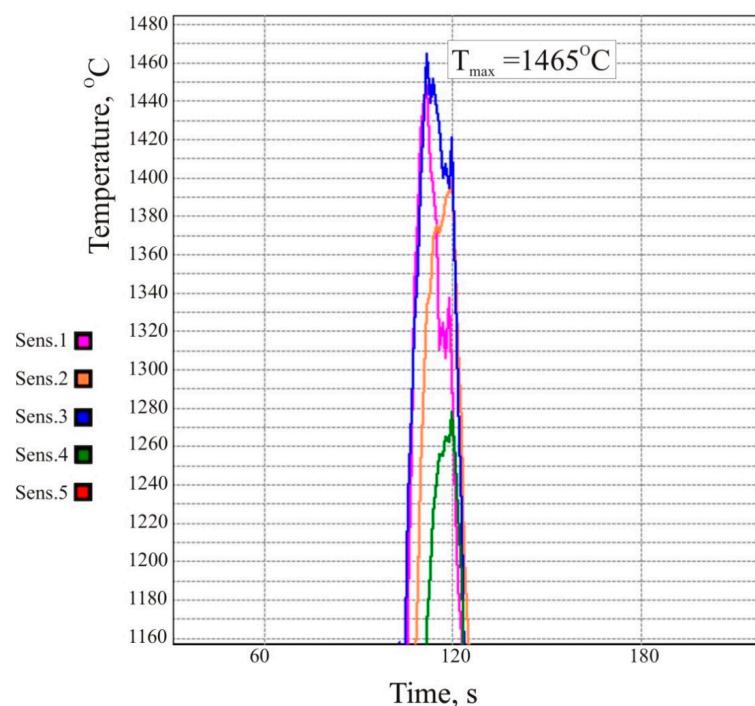


Figure 12. Thermogram recorded by thermocouples placed in silicon carbide inserts with a protective SiC coating.

3.3. Deposition of SiC Coatings on SiC-C-MoSi₂ Ceramic Products

SiC has good chemical compatibility and thermal stability with MoSi₂, so the strength and toughness of SiC-based composites enriched in the MoSi₂ phase can be greatly improved. Figure 13 shows the microstructure of a SiC-C-MoSi₂ composite material with

a silicon carbide protective coating. The coating makes it possible to prevent both the high-temperature oxidation of carbon particles (which are present in the material's structure) and the low-temperature oxidation of molybdenum disilicide. Such stability is necessary during the long-term operation of such a material in the temperature range of 600–900 °C [34,35]. The coating was deposited by the gas-phase method through the exposure of the composite sample in the thermal zone of the furnace, where silicon vapors and products of high-temperature pyrolytic decomposition of methane molecules were present.

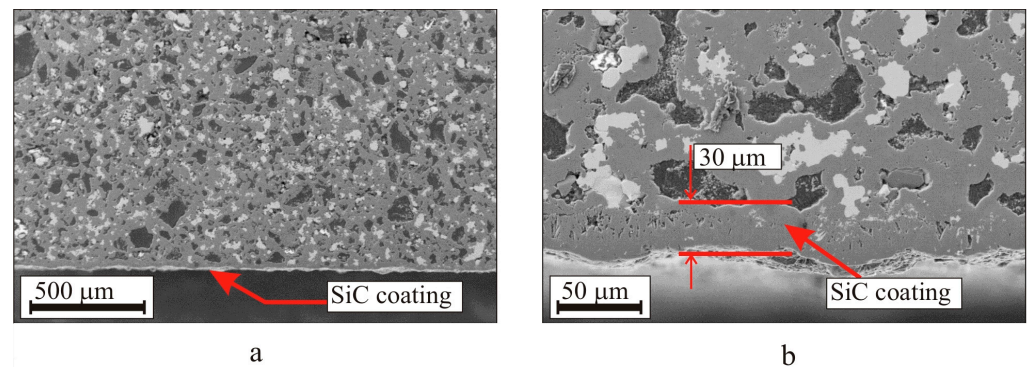


Figure 13. Micrograph of the structure of a SiC-C-MoSi₂ ceramic composite material with a protective SiC coating: black areas represent carbon, dark gray represents SiC, and light gray represents MoSi₂. (a) low magnification; (b) high magnification.

Additionally, this method can be used to obtain protective coatings for SiC-based composites reinforced with refractory metal wire, such as molybdenum, tungsten, niobium, etc. [36,37].

3.4. Deposition of SiC Coatings on Products from SiC-Si Material

A silicon carbide gas-tight coating can be deposited on materials containing residual silicon, such as modified recrystallized silicon carbide (mRSiC) or reaction-bonded silicon carbide (RBSiC). Such protective SiC coatings significantly increase the thermal and oxidative resistance of mRSiC or RBSiC (Figure 14) due to the protection against oxidation of residual silicon contained in the material.

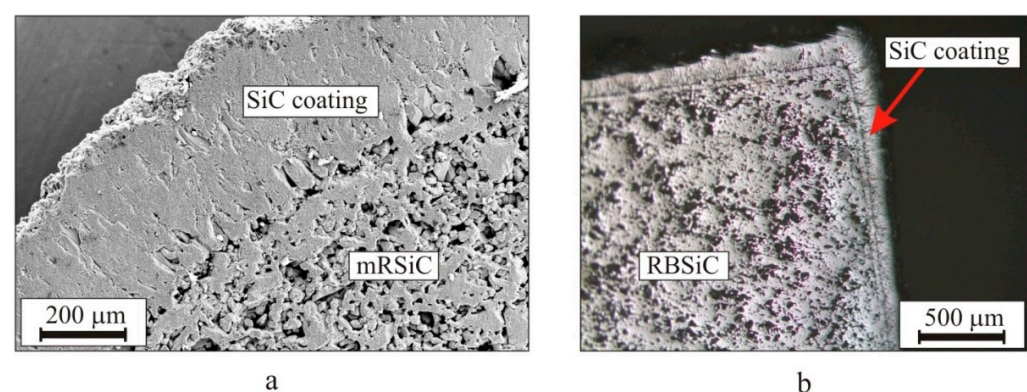


Figure 14. Protective SiC coating on recrystallized (a) and reaction-bonded (b) SiC.

Heaters made of recrystallized SiC ceramics “age” during operation. Due to oxidation, the cross section of the working part of the heater decreases and its electrical resistance increases. As a result, zones of local overheating are formed. In places of overheating, more intense oxidation occurs, which in turn causes a further decrease in the cross section of the heater and an increase in temperature. The process proceeds at an increasing rate and leads to the burnout of the heater. We have developed a technique for modifying RSiC [38]. This technology increases the mechanical strength and thermal shock resistance of the material.

The dependence of electrical resistivity on temperature becomes more stable. Therefore, the manufacture of an mRSiC heater with a protective SiC coating is of great interest as this would increase the resistance of the heater to oxidation.

To confirm the effectiveness of the above method for manufacturing heating elements from mRSiC, a prototype heater was prepared and tested. A heating element made of mRSiC with an outer diameter of 12 mm and a length of 180 mm was made from the working parts of a standard heating element. The protective silicon carbide coating was deposited on its surface (Figure 14a).

The tests were carried out on a stand (see the scheme in Figure 15). A computer-controlled power supply heated the heater up to $\sim 1600\text{ }^{\circ}\text{C}$ for two minutes, which was followed by cooling for one minute. The cycle was repeated afterwards. At the end of each heating cycle, the values of the current flowing through the heater and the voltages on the current leads were recorded.

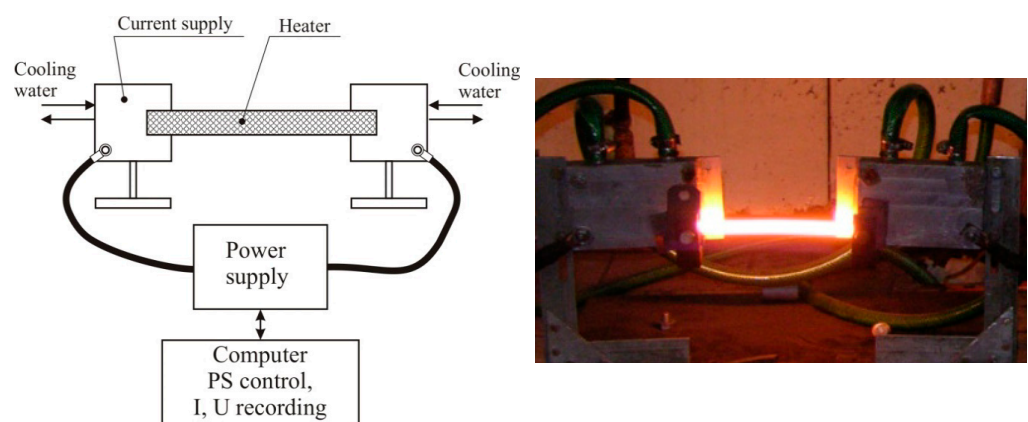


Figure 15. Scheme of the stand and a photograph of the testing process for the mRSiC heater with a protective SiC coating.

In Figure 16, the dependence of heater resistance vs. the number of warm-up/cool-down cycles is plotted. After 2000 cycles, the change in the electrical resistance of the heater was less than 5%. The stability of electrical resistance confirms the good protective properties of the SiC coating and its high adhesion to the heater surface.

3.5. Deposition of SiC Coatings on SiC-C-MoSi₂ Ceramic Products

The protective SiC gas-tight coating can also be deposited on refractory metals and metal alloys. Figure 17a shows a SiC-coated molybdenum plate that was used as a heater in an oxidizing atmosphere at a temperature of $1500\text{ }^{\circ}\text{C}$ for about 10 h. Heater contact zones at the edges were not protected by the coating.

To achieve good adhesion with the SiC coating and prevent its delamination and destruction during temperature cycling, an intermediate layer of silicide was formed on the surface of metal (Figure 17b). In order for this to be achieved before methane was supplied to the chamber, the metal was preliminarily held in silicon vapor to form a silicide layer on its surface. Further, after the start of methane supply to the interaction zone, a silicon carbide coating was formed on a silicide layer.

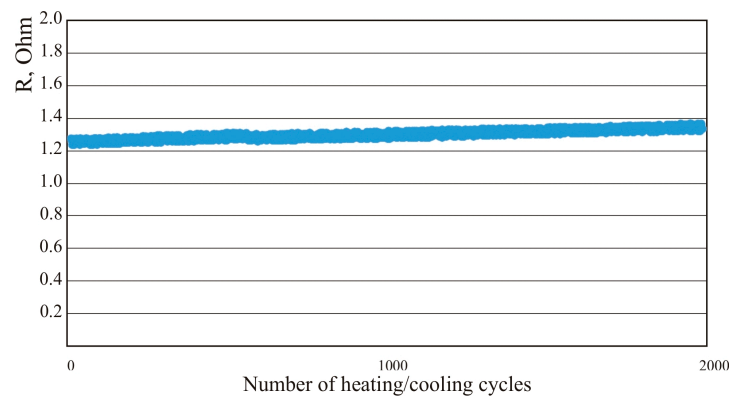
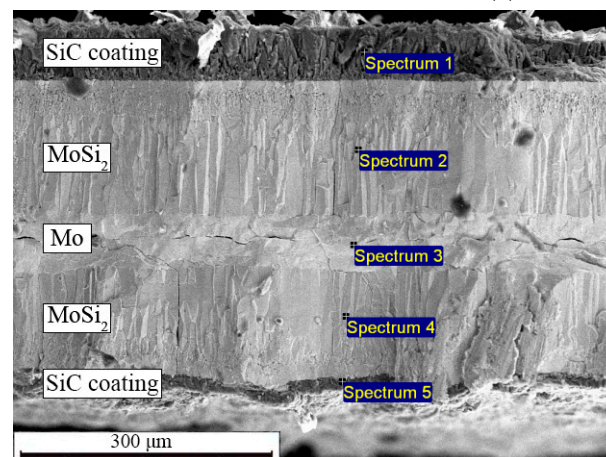


Figure 16. Dependence of heater resistance on the number of heating/cooling cycles.



(a)



(b)

Spectrum	C	Si	Mo
Spectrum 1	78.48	21.52	
Spectrum 2		64.83	35.17
Spectrum 3		2.03	97.97
Spectrum 4		64.64	35.36
Spectrum 5	57.52	41.73	0.75
Max.	78.48	64.83	97.97
Min.	57.52	2.03	0.75

All results in atomic%

Figure 17. Protective gas-tight SiC coating on a molybdenum heating element (a). Phase analysis of the heating element's cross section (b).

4. Conclusions

The conventional CVD method for SiC deposition includes various precursors (monomethylsilane, methyltrichlorosilane, tetramethylsilane, etc.). In contrast to CVD, the proposed high-temperature method of the gas-phase deposition of protective SiC coatings has a number of significant advantages. These are the following:

- The deposition rate is approximately two orders of magnitude higher;
- The adhesion of the protective coating to the surface of a carbon- and/or silicon-containing material is significantly higher. This is because the process is carried out at a high temperature with the participation of carbon and silicon located directly in the substrate material;
- Simpler technological equipment;
- Low cost due to the speed of coating deposition and the use of more accessible and cheaper components compared to silicon-containing precursors.

Thus, a high-temperature method for the deposition of protective SiC coatings onto SiC-C-Si and SiC-C-MoSi₂ ceramics, CCCMs, graphite, refractory metals, and metal alloys has been developed. The tests carried out showed high thermal-oxidative and thermal

shock resistance and good adhesion, with protective coatings also capable of withstanding harsh environments.

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