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Research article
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Investigating the impact of the porous structure of needle-punched preform-based carbon-carbon composites on the completeness of liquid silicon infiltration

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Abstract: Currently, siliconized carbon-carbon composites (C/C composites) hold a significant position among materials used in non-ferrous metallurgy. The process of Liquid Silicon Infiltration (LSI) for porous C/C composites is strongly influenced by their microstructural characteristics. Studying the effect of the porous structure of various C/C composites on the completeness of silicon infiltration can enable the regulation of the phase composition of siliconized materials over a wide range, as well as the physical, mechanical, and thermophysical properties of C/C–SiC composites. This paper presents the results of analyzing the porous structure and strength characteristics of C/C composites based on needle-punched preforms with different types of carbon matrices (pyrocarbon, natural and synthetic pitch coke, and phenol-formaldehyde resin coke) and the C/C–SiC composites derived from them. Due to the specific features of carbon matrix formation from liquid or gas phases, differences in pore size distribution were observed. A carbon matrix formed by the gas-phase method exhibits fewer nanoscale pores compared to one formed by the liquid-phase method. The influence of the pore structure and the nature of the matrix carbon in various needle-punched preforms on the degree of saturation during LSI, infiltration depth, and mechanical properties was determined.

Keywords: carbon-carbon composites, porous structure, carbon matrix, carbon preform, liquid silicon infiltration (LSI), C/C–SiC composites.

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Определение влияния пористой структуры углерод-углеродных композиционных материалов на основе иглопробивной преформы на полноту пропитки расплавом кремния

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Аннотация: В настоящее время особое место среди материалов, используемых в цветной металлургии, занимают силицированные углерод-углеродные композиционные материалы (УУКМ). На процесс силицирования пористого УУКМ значительно вли-

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яют его микроструктурные характеристики. Изучение влияния пористой структуры различных УУКМ на полноту пропитки расплавом кремния может позволить регулировать фазовый состав силицированных материалов в широком диапазоне, а также физико-механические и теплофизические свойства углерод-керамического композиционного материала (УККМ). Описаны результаты анализа пористой структуры и прочностных характеристик УУКМ на основе иглопробивной преформы с различными типами углеродных матриц (пироуглеродная, кокс натурального и синтетического пеков, кокс фенолформальдегидной смолы) и УККМ на их основе. В силу особенностей формирования углеродной матрицы из жидкой или газовой фаз наблюдается отличие по границам диапазонов пор. Углеродная матрица, сформированная газофазным методом, оставляет меньше наноразмерных пор в сравнении с матрицей, полученной жидкофазным методом. Установлено влияние структуры порового пространства и природы матричного углерода различных УУКМ на основе иглопробивных преформ на их степень насыщения расплавом кремния, глубину пропитки, а также определены механические свойства.

Ключевые слова: углерод-углеродные композиционные материалы, пористая структура, углеродная матрица, углеродная преформа, силицирование, пропитка расплавом кремния, углерод-керамические композиционные материалы.

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Introduction

The modern development of materials science is inseparable from the creation and implementation of high-temperature materials that combine heat resistance, excellent thermal stability, high mechanical properties, wear resistance, and durability in aggressive gaseous and liquid environments at elevated temperatures. Siliconized graphite largely meets these requirements, which explains its widespread use in the chemical, metallurgical, and energy industries. Siliconized graphite is utilized as components in friction assemblies (such as sealing rings and sliding bearings) for pumps and reactors, as protective sheaths for immersion thermocouples, and as bottom pouring refractories or stopper rod systems for metallurgical furnaces. Ensuring and enhancing the strength properties of materials under high-temperature conditions necessitates the development of new structural materials [1—3].

Currently, siliconized carbon-carbon composites (C/C composites) hold a significant position among materials used in non-ferrous metallurgy. Liquid silicon infiltration (LSI) is one of the most effective and rapid methods for forming a ceramic matrix. This method involves applying a slip coating based on silicon-containing powder to the surface of a porous C/C composite semi-finished product. At temperatures exceeding 1414 °C, the coating decomposes, producing liquid silicon. The LSI process for porous C/C composites is significantly influenced by their microstructural characteristics. The completeness of silicon infiltration is determined by the nature of the porous structure and the pore distribution in the C/C composite (including pore volume, size, configuration, distribution throughout the material, and accessibility to liquid silicon). Studying the influence of the structural features of porous C/C composites on their reactivity with liquid silicon and the completeness of infiltration enables regulation of the phase composition of siliconized materials over a wide range, as well as the physical, mechanical, and thermophysical properties of C/C—SiC composites [4—14].

Depending on the intended purpose, structural features of the components, and their operating conditions, various methods are used to manufacture reinforcing preforms and form carbon matrices for the production of C/C composites. Each type of carbon-reinforcing framework has its own structural features, and together with the different technological processes for densifying them with a carbon matrix, they exhibit specific ranges of pore sizes and configurations. Over the past decade, significant attention has been devoted to the development of high-speed, fully automated technologies for creating needle-punched reinforcing frameworks from carbon fibers. The emergence of new needle-punched frameworks has necessitated the development of porous C/C composites based on these frameworks and the adjustment of LSI process parameters to produce high-density C/C—SiC composites with uniformly distributed SiC [15—20].

To saturate the reinforcing framework with a carbon matrix, various carbon-containing substances are used. In the case of forming the carbon matrix by the gas-phase method, a carbon-containing gas undergoes pyrolysis, resulting in carbon deposition between the fibers of the framework. The advantages of this method include ensuring uniform distribution of the carbon matrix, high density of deposited carbon, strong adhesion between the matrix and the fibers, and enhanced mechanical and strength characteristics of the C/C composites. However, the gas-phase method is characterized by its long processing time, low raw

material utilization efficiency, and consequently, high cost [10; 19; 21—24].

An alternative method for forming the carbon matrix is the impregnation of the framework with a polymer binder. This approach is faster and more cost-effective. The liquid-phase method of forming a carbon matrix involves impregnating carbon frameworks with polymer resin, followed by pyrolysis, carbonization, and high-temperature treatment (HTT). Precursors for this method can include various thermosetting (e.g., phenol-formaldehyde, furan) and thermoplastic (e.g., coal tar pitch, petroleum pitch) resins. The mechanical and thermophysical properties of the resulting composites largely depend on the chemical and physical structure of the coke residue from the polymer binder. The advantages of using pitches include their high coke density, good graphitization tendency, and the elimination of solvents from the technological process. However, their drawbacks include thermoplasticity, which leads to binder migration during heat treatment, and the presence of carcinogenic compounds in pitches, which negatively impacts working conditions. In industrial applications, phenol-formaldehyde resins and coal tar pitches are most commonly used to form the carbon matrix via the liquid-phase method [19; 25—28].

The objective of this study is to determine the influence of the pore structure and the nature of the matrix carbon in various needle-punched preform-based C/C composites on their degree of saturation with silicon melt, infiltration depth, and mechanical properties.

1. Research methodology

For the study, C/C composites were manufactured based on needle-punched preforms (NPPs) produced by JSC "Kompozit" (Korolev, Russia). The needle-punching technology enables the production of layered fibrous preforms with the required level of mechanical properties. NPPs made from continuous carbon fibers were fabricated by sequentially laying the tapes with rotation at a specific angle to reduce anisotropy in the properties. After preparing the reinforcing preform, the space between the fibers was filled with a carbon matrix formed using gas-phase and liquid-phase methods.

Phenol-formaldehyde resin of the BZh grade, produced by LLC "Naukom" (Nizhny Novgorod, Russia), natural and synthetic coal tar pitches produced by LLC "Mini-Max" (Moscow, Russia), and the carbon-containing gas methane (CH_4) were used as precursors for the carbon matrix.

The total open porosity and apparent density were determined using the hydrostatic weighing method in accordance with GOST 15139-69.

Data on the pore size and volumetric content of pores in the studied materials were obtained by the standard contact porometry (SCP) method using the "Porosimeter 3.2" instrument. The SCP method allows for the evaluation of the integral and differential porosity of materials, as well as their density. The measurement range of the pores is from 1 nm to 500 μm. The SCP experiment involves measuring the equilibrium relative moisture content curve (the ratio of the volume of liquid—octane—inside the pores to the weight or volume of the porous sample) between a standard and the test sample. The equilibrium relationship between the relative amount of octane in the test sample and its quantity in the standard, for which the porometric curve is pre-determined, is calculated. From this dependence and the calibration porometric curve of the standard (the distribution curve of pore volumes by their radii), the porometric curve of the test sample can be derived. The obtained integral and differential pore distribution data make it possible to evaluate the volumetric pore content and the porous structure of the material.

To study the porous space and microstructure of C/C composites and C/C—SiC composites, microstructural analysis of cross-sections of the samples was performed using a JCM-6610 LV scanning electron microscope equipped with an "Advanced Aztec" energy-dispersive analyzer. The investigation was conducted at various magnifications under an accelerating voltage of 20 kV. Surface topography analysis was carried out based on the contrast of the microstructure images obtained using secondary electrons (SEI). Imaging with backscattered electrons (BEC) was used to determine the elemental composition and morphology of the sample surfaces based on differences in the electron density distribution of the elements. Heavier elements appear brighter (e.g., Si), while lighter elements appear darker (e.g., C).

Tests to determine the ultimate tensile and compressive strength in the primary reinforcement direction, as well as compressive and shear strength perpendicular to the reinforcement direction, were conducted using a UTS-111 universal testing machine in accordance with OST 92-1459-77, 92-1460-77, and 92-1472-78, respectively. The loading range varied from 50 N to 50 kN. The method involves applying loads at fixation points according to various test schemes at a speed of 2—5 mm/min. The measurement error is ±0.5 %.

The structure of C/C—SiC composites was examined for the absence of hidden macro-defects (such as

cracks or delaminations) and to assess the depth of LSI using *X*-ray tomography with an XT H 320 LC *X*-ray tomograph. As *X*-ray radiation passes through each part of the examined object, it loses intensity, which is then recorded by the cells of the receiver matrix. Each element (pixel) of the receiver records the intensity of the *X*-ray radiation. The calculated grayscale values, ranging from 0 (black) to 65536 (white), are proportional to the *X*-ray density of the examined object. This density, in turn, is directly proportional to the atomic numbers of the elements comprising the object, as listed in the Periodic Table of Elements, and to the physical density of the object.

2. Results and discussion

The main characteristics (apparent density — ρ, kg/m^3, and open porosity — OP, %) of various types of C/C composites depending on the type of matrix are presented in Table 1. The initial density of the NPP framework is 720 kg/m^3, and the volumetric fraction of the reinforcing filler is 50 %.

2.1. Porometric analysis of C/C composites

The analysis of the pore space can help in selecting the optimal mode for the subsequent formation of the ceramic matrix. To investigate the volumetric content, distribution, and size of pores, porometric analysis was performed on samples with different carbon matrices, ensuring comparable open porosity. The integral and differential pore distributions as a function of the logarithm of their radius are presented in Fig. 1.

The predominant pore size ranges from 1 to 15 μm, accounting for over 50 % of the total pore volume in the material. At the same time, due to the specific features of carbon matrix formation via liquid- or gas-phase methods, differences are observed in the pore size distribution boundaries. The carbon matrix formed by the gas-phase method contains fewer nanoscale pores (up to 9 %) compared to the matrix formed by the liquid-phase method

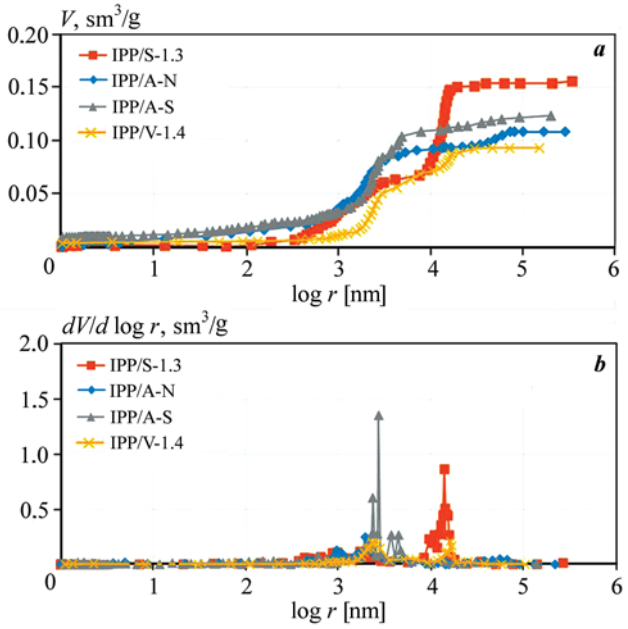

Fig. 1. Integral (***a***) and differential (***b***) pore distributions as a function of the logarithm of their radius in the studied samples

Рис. 1. Интегральное (***a***) и дифференциальное (***b***) распределения пор в зависимости от логарифма их радиуса в исследуемых образцах

(ranging from 17 to 36 %). For IPP/V, a higher number of closed pores is characteristic compared to C/C composites with a carbon matrix formed by the liquid-phase method, due to the partial blockage of pores during the deposition of pyrocarbon from the gas phase.

2.2. Microstructural analysis of C/C composites

The microstructural analysis of the pore space in C/C composites of grades IPP/S-1.3, IPP/A-N, and IPP/V-1.4 is shown in Fig. 2. The IPP/A-S sample is not included due to the absence of distinctive structural features in the coke on the microphotographs, regardless of the type of pitch used.

Table 1. **The main characteristics of CCCM**
Таблица 1. Основные характеристики УУКМ

Type of precursor	Material grade	ρ, kg/m^3	OP, %
Phenol-formaldehyde resin	IPP/S-1.3	1360	12.1
Natural pitch	IPP/A-N	1590	15.1
Synthetic pitch	IPP/A-S	1580	15.5
Pyrolytic carbon	IPP/V-1.4	1480	15.3

Table 2. **Quantitative analysis of pore distribution in C/C composites**

Таблица 2. Количественный анализ распределения пор УУКМ

Material grade	Pore distribution in range, %			
	1 nm–1 μm	1–15 μm	15–70 μm	>70 μm
IPP/S-1.3	21.4	65.9	10.9	1.8
IPP/A-N	35.8	49.6	13.4	1.2
IPP/A-S	17.3	69.4	10.5	2.8
IPP/V-1.4	9.1	74.2	13.5	3.2

The structure of C/C composites with a matrix formed by the liquid-phase method predominantly exhibits high porosity, with large pores located along the boundaries of fiber bundles and the carbon matrix formed during the pyrolysis of the polymer binder at the carbonization stage (Fig. 2, *a*, *b*). The primary channels in the needle-punched preform are through interbundle pores as well as pores aligned with the needle-punching direction. The interfiber space in C/C composites with a liquid-phase-formed matrix is significantly filled with coke, with the presence of submicron-sized pores. Additionally, in C/C composites with a resin coke matrix (Fig. 2, *a*), as well as those with a pyrocarbon matrix (Fig. 2, *c*), the interbundle pores are clearly visible. In C/C composites with a matrix based on coal tar pitch coke (Fig. 2, *b*), the interbundle pores are largely filled with coke, which may hinder the penetration of silicon melt during subsequent LSI. In C/C composites with a gas-phase-formed matrix (Fig. 2, *c*), interbundle pores with a radius exceeding 15 μm are distinctly observed.

2.3. Evaluation of strength characteristics

The initial C/C composites with various types of carbon matrices were tested to determine the ultimate tensile and compressive strengths in the primary reinforcement direction (*X*), as well as compressive and shear strengths in the plane perpendicular to the primary rein-

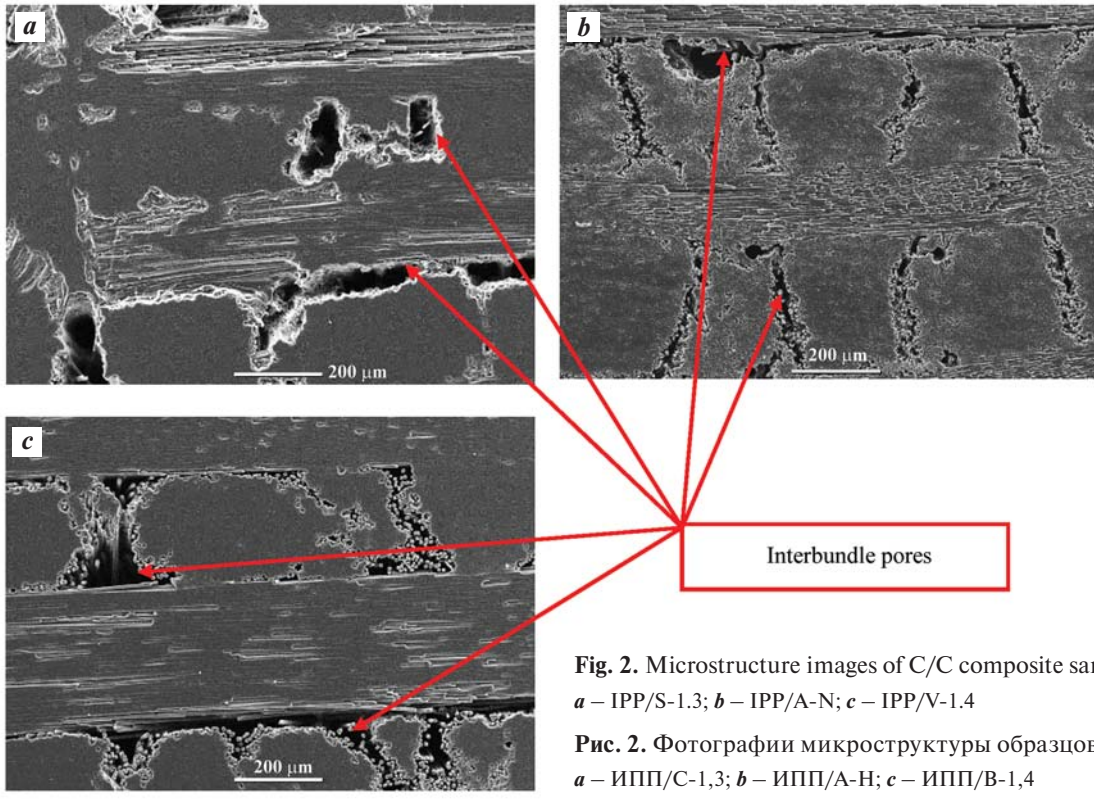

Fig. 2. Microstructure images of C/C composite samples
a – IPP/S-1.3; ***b*** – IPP/A-N; ***c*** – IPP/V-1.4

Рис. 2. Фотографии микроструктуры образцов УУКМ
a – ИПП/С-1,3; ***b*** – ИПП/А-Н; ***c*** – ИПП/В-1,4

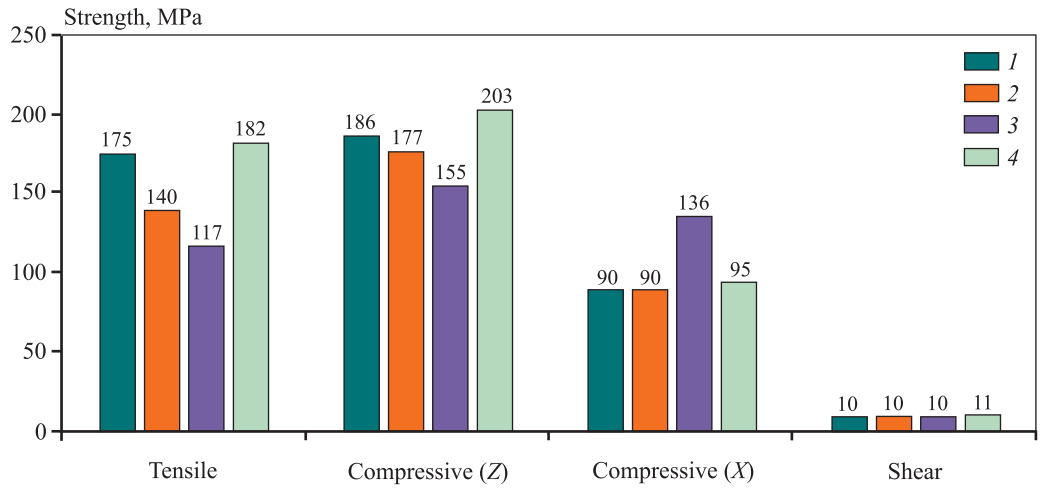

Fig. 3. Tensile, compressive, and shear strengths for various grades of C/C composites
1 – IPP/S-1.3; ***2*** – IPP/A-N; ***3*** – IPP/A-S; ***4*** – IPP/V-1.4

Рис. 3. Пределы прочности при растяжении, сжатии и сдвиге для различных марок УУКМ
1 – ИПП/С-1,3; ***2*** – ИПП/А-Н; ***3*** – ИПП/А-С; ***4*** – ИПП/В-1,4

forcement direction (*Z*). The results of the strength tests for the initial C/C composites are shown in Fig. 3.

Overall, the obtained values of the physical and mechanical characteristics for all C/C composite semi-finished products are relatively high, indicating their suitability for producing C/C—SiC composites with superior strength properties. For all grades of C/C composites, the tensile strength ranges from 117 to 182 MPa, compressive strength along the *X*-axis ranges from 90 to 136 MPa, compressive strength along the *Z*-axis ranges from 155 to 203 MPa, and shear strength ranges from 10 to 11 MPa.

2.4. Results of liquid silicon infiltration (LSI)

During the determination of technological parameters and optimization of the LSI process, adjustments were made to the coefficient of the applied slip, consisting of a silicon-containing powder and binder. The amount of slip was calculated based on the initial characteristics of the C/C composite. Increasing the coefficient allowed for obtaining a denser material in a single infiltration cycle. However, it could result in the formation of residual silicon build-ups, tightly bonded to the sample surface, representing an excess of the melt. The evaluation parameters included weight gain as a percentage of the initial mass and final open porosity not exceeding 5 %. The results of LSI for various samples are presented in Table 3.

C/C—SiC composites of grades IPP/S-1.3 and IPP/A-N exhibit identical weight gains (22.6 and 22.7 %, respectively) and a residual open porosity slightly above 5 %. The lowest residual open porosity (4.1 and 3.3 %) was observed for C/C—SiC composites with initial pyrocarbon and synthetic pitch coke-based matrices, respectively. The highest weight gain and SiC content were observed in C/C—SiC composites with initial pyrocarbon (31.0 and 12.9 %, respectively) and synthetic pitch coke-based matrices (26.0 and 12.2 %, respectively). Similar SiC phase volume fractions were also observed in C/C—SiC composites with matrices based on phenol-formaldehyde resin coke and natural pitch coke (9.6 and 11.2 %, respectively). The dependence of weight gain and silicon carbide content on the type of carbon matrix is shown graphically in Fig. 4.

The intensity and completeness of the bulk LSI process for C/C composites are significantly influenced by the nature of their porous structure, the total pore volume, pore size and configuration, their distribution throughout the entire volume of the material, and their accessibility for liquid silicon infiltration. It is most likely that interbundle pores, once filled, allow the silicon melt to flow further into interfiber pores. The C/C—SiC composite of grade IPP/V-1.4, with a pyrocarbon matrix, exhibits the highest weight gain and SiC content, which is attributed to its more favorable porous structure (interfiber and interbundle pores ranging from 1 to 15 μm account for approximately 75 % of the total pore volume). In the case of carbon matrices formed by the liquid-phase method, the submicron pore fraction for IPP/A-S, IPP/S-1.3, and IPP/A-N materials is 17 %, 21 %, and 36 %, respectively. This high proportion of submicron pores can lead to premature pore blockage

Table 3. **Results of liquid silicon infiltration using the developed technology**

Таблица 3. Результаты пропитки расплавом кремния по отработанной технологии

Material grade	$\rho_{initial}$, kg/m^3	OP_{init}, %	Weight gain Δm, %	ρ_{final}, kg/m^3	OP_{final}, %	V_{SiC}, vol. %
IPP/S-1,3	1360	12.1	22.6	1740	5.3	9.6
IPP /A-N	1590	15.1	22.7	1950	5.5	11.2
IPP /A-C	1580	15.5	26.0	2010	3.3	12.2
IPP /V-1,4	1480	15.3	31.0	1870	4.1	12.9

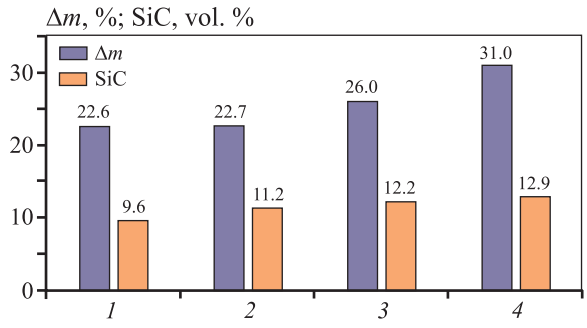

Fig. 4. Dependence of silicon carbide content on the type of carbon matrix

Matrix type: ***1*** – phenol-formaldehyde resin coke; ***2*** – natural pitch coke; ***3*** – synthetic pitch coke; ***4*** – pyrocarbon

Рис. 4. Зависимость содержания карбида кремния от типа углеродной матрицы

Тип матрицы: ***1*** – кокс фенолформальдегидной смолы; ***2*** – кокс натурального пека; ***3*** – кокс синтетического пека; ***4*** – пироуглерод

and unreacted carbon matrix volumes, reducing the effectiveness of LSI.

After LSI, the strength characteristics of the C/C—SiC composites were determined. The test results for the ultimate tensile and compressive strengths in the primary reinforcement direction (*X*), as well as compressive and shear strengths in the plane perpendicular to the primary reinforcement direction (*Z*), are shown graphically in Fig. 5.

The tensile strength of all types of C/C—SiC composites showed only a slight decrease after LSI compared to the initial C/C composites before infiltration, indicating minimal carbidization of carbon fibers. In contrast, the compressive strength along the primary reinforcement direction (*X*-axis) more than doubled, while compressive strength perpendicular to the reinforcement direction (*Z*-axis) increased by up to 70 %. Interlayer shear strength improved by 70—80 %. These significant enhancements in the mechanical properties of all C/C—SiC composite types relative to their initial C/C composite precursors are attributed to the formation of the silicon carbide matrix.

To evaluate the infiltration depth, a tomographic study was conducted on cubic samples measuring 15 × 15 × 15 mm. The tomographic images, presented in Fig. 6, show the non-infiltrated regions marked with dashed lines, facilitating the assessment of infiltration completeness.

Tomographic analysis revealed that samples of C/C—SiC composites with initial pyrocarbon and phenol-formaldehyde resin coke-based matrices were infiltrated to their full depth (7.5 mm from the surface), compared to C/C—SiC composites with initial pitch-based matrices, which showed infiltration depths of up to 5 mm. This difference can be attributed to the porous structure formed during carbonization and high-temperature treatment, characterized by a high fraction of submicron pores and low interbundle porosity, which hinders silicon infiltration.

Notably, a dependence of infiltration depth on the type of pitch was identified. For example, C/C—SiC composites with a synthetic pitch-based matrix exhibited an infiltration depth 1.5 times greater than those with a natural pitch-based matrix, due to a higher proportion of pores with radii ranging from 1 to 15 μm.

For a detailed analysis of the microstructure and overall composition of the C/C—SiC composites, a microstructural analysis was conducted. The microstructural images are shown in Fig. 7.

The distribution of SiC in C/C—SiC composites with a carbon matrix formed by the liquid-phase method is uniform across all pore size ranges. In contrast, C/C—SiC composites with an initial pyrocarbon matrix contain closed macropores (10—50 μm in diameter) that remain unfilled with SiC. This is due to the characteristics of carbon matrix distribution during deposition. Nevertheless, the highest weight gain after LSI in composites with an initial pyrocarbon matrix

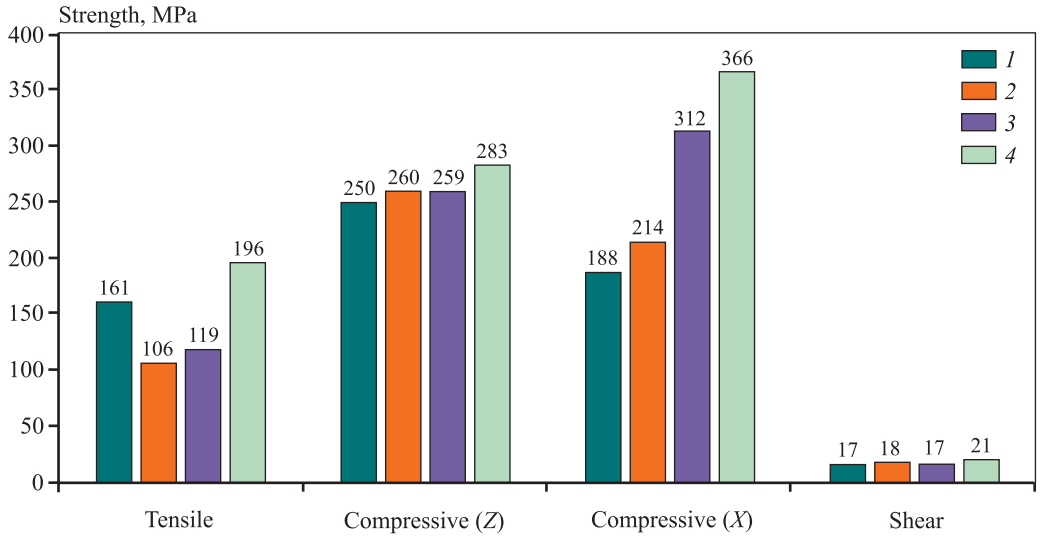

Fig. 5. Tensile, compressive, and shear strengths for various grades of C/C–SiC composites
1 – IPP/S-1.3; ***2*** – IPP/A-N; ***3*** – IPP/A-S; ***4*** – IPP/V-1.4

Рис. 5. Пределы прочности при растяжении, сжатии и сдвиге для различных марок УККМ
1 – ИПП/С-1,3; ***2*** – ИПП/А-Н; ***3*** – ИПП/А-С; ***4*** – ИПП/В-1,4

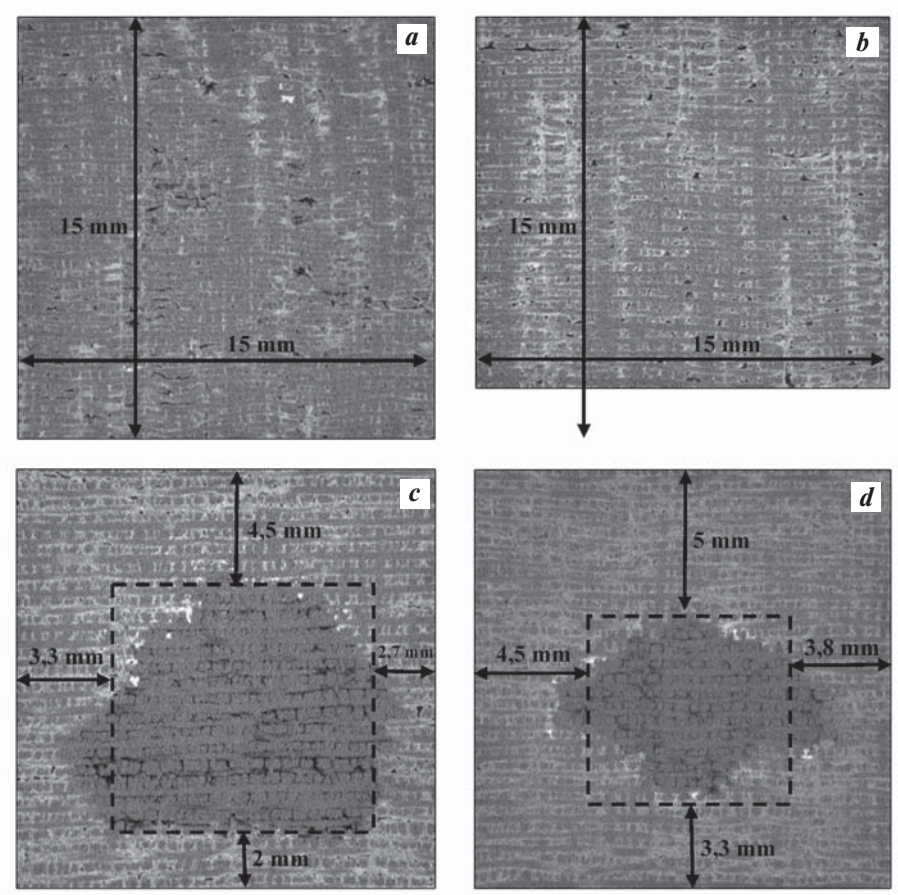

Fig. 6. Tomographic images after LSI of C/C–SiC composite samples
a – IPP/S-1.3; ***b*** – IPP/V-1.4; ***c*** – IPP/A-N; ***d*** – IPP/A-S

Рис. 6. Томографические изображения после силицирования образцов УККМ
a – ИПП/С-1,3; ***b*** – ИПП/В-1,4; ***c*** – ИПП/А-Н; ***d*** – ИПП/А-С

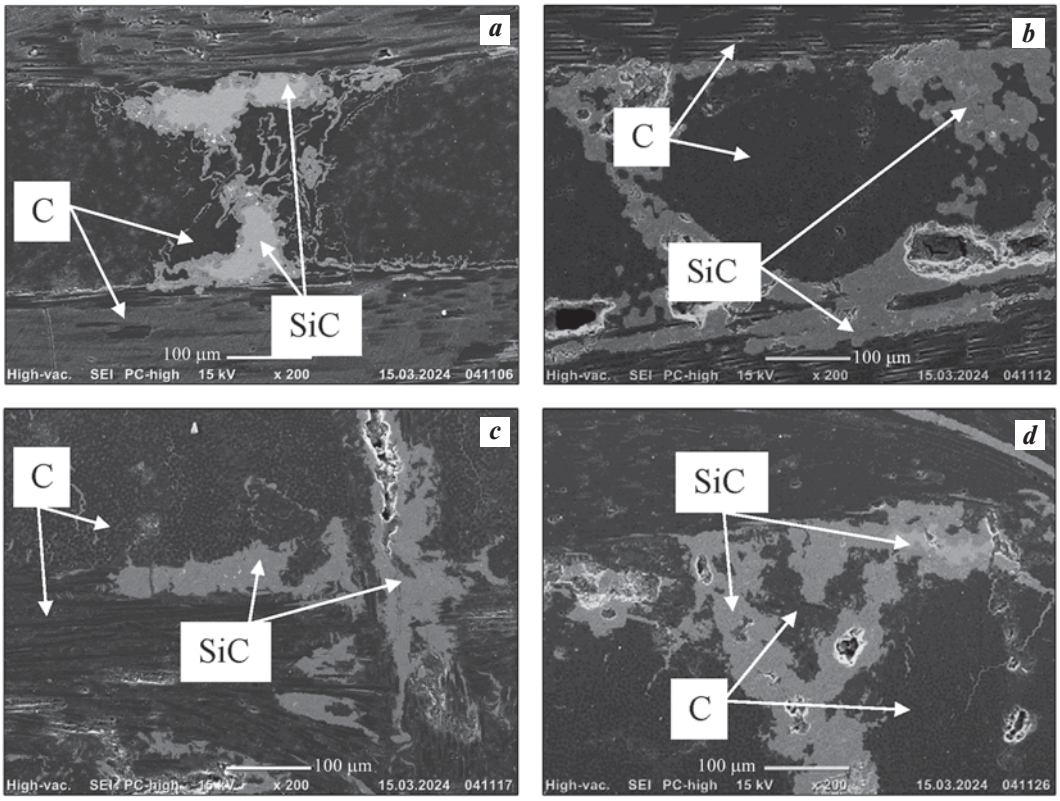

Fig. 7. Microstructural images of C/C–SiC composite samples
a – IPP/S-1.3; ***b*** – IPP/V-1.4; ***c*** – IPP/A-N; ***d*** – IPP/A-S

Рис. 7. Фотографии микроструктуры образцов УККМ
a – ИПП/С-1,3; ***b*** – ИПП/В-1,4; ***c*** – ИПП/А-Н; ***d*** – ИПП/А-С

is achieved due to the predominance of pores in the 1—15 μm range in the initial C/C composites before infiltration.

Conclusion

The microstructural, porometric, and tomographic studies of C/C composites with different initial carbon matrices reveal that the nature of the matrix carbon and the pore distribution in terms of volume and size significantly influence the degree of saturation and infiltration depth during liquid silicon infiltration (LSI). The highest saturation was observed in C/C—SiC composites of grades IPP/V-1.4 with a pyrocarbon matrix and IPP/S-1.3 with a phenol-formaldehyde resin coke-based matrix, owing to their favorable porous structures for siliconization. In composites with pyrocarbon matrices, infiltration depth reached 7.5 mm from the surface, with weight gain and SiC content values of 31.0 and 12.9 %, respectively. For liquid-phase carbon matrices, the greatest infiltration depth (7.5 mm from the surface) was achieved in the IPP/S-1.3 composite with a phenol-formaldehyde resin coke-based matrix, with weight gain and SiC content values of 22.6 and 9.6 %, respectively. For IPP/A-N and IPP/A-S composites, infiltration depths averaged 4.5 mm from the surface, with weight gain and SiC content values of 22.7 and 11.2 % for IPP/A-N (natural pitch-based matrix) and 26.0 and 12.2 % for IPP/A-S (synthetic pitch-based matrix). A direct dependence of the infiltration depth during LSI on the fraction of submicron pores was identified: the smaller the fraction of nanoscale pores, the higher the weight gain, SiC content, and infiltration depth. Mechanical tests of various C/C composites and their corresponding C/C—SiC composites demonstrated high physical and mechanical properties, confirming the applicability of C/C—SiC composites as structural materials. For all grades of C/C—SiC composites, tensile strength ranged from 106 to 196 MPa, compressive strength along the *X*-axis ranged from 188 to 366 MPa, compressive strength along the *Z*-axis ranged from 250 to 283 MPa, and shear strength ranged from 17 to 21 MPa.

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