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Calculating the penetration depth of reaction in chemical gas-phase deposition of boron nitride within porous bodies

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Abstract. The thermodynamic calculations conducted using the TERRA software package for the B–Cl–N–H system revealed that the inclusion of hydrogen into the B–Cl system significantly diminishes the thermodynamic stability of BCl_3 with the possibility of boron formation in the condensed phase. On the other hand, the introduction of ammonia, which includes hydrogen, results in the synthesis of boron nitride across a broad temperature spectrum. The analysis of kinetic relationships uncovered three distinct regions in the boron nitride deposition process: *K* – kinetic region (up to 1400 K), *D* – diffusion region (above 1800 K) and *T* – transition region. The activation energy for the kinetic region was calculated as E_a = 134 kJ/mol. Within the temperature range of 1023–1123 K, linear dependences were observed. The computation of the penetration depth for the boron nitride deposition process assumed a gas mixture of boron trichloride, ammonia, and argon ($BCl_3 + NH_3 + 30Ar$). The results indicated that boron trichloride governs the extent of penetration. The depths of penetration for the chemical vapor infiltration boron nitride (CVI-BN) process, conducted at 0.1 kPa within the temperature range of 1100–1400 K, were determined for pore diameters of 1, 10, 30, 100, 200 and 300 μm. When porosimetry data for a specific preform is available, the acquired penetration depth relationships for the CVI-BN process under specific parameters and process temperatures facilitate the estimation of essential parameters for interphase formation using pyrolytic boron nitride.

Keywords: pyrolytic boron nitride (PBN), interphase coating, thermodynamic calculation, chemical vapor infiltration boron nitride (CVI-BN)

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Расчет глубины проникновения реакции при химическом газофазном осаждении нитрида бора в пористых телах

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Аннотация. Проведенные с помощью программного комплекса «TERRA» термодинамические расчеты системы B–Cl–N–H показали, что введение водорода в систему B–Cl резко снижает термодинамическую устойчивость BCl_3 с возможностью образования бора в конденсированной фазе, а добавка аммика, который содержит водород, приводит к образованию нитрида бора в широком интервале температур. Анализ кинетических зависимостей показывает наличие трех областей процесса осаждения нитрида бора: К – кинетическая область (от 1400 К и ниже), Д – диффузионная область (от 1800 К и выше) и П – переходная область. Для кинетической области определена энергия активации процесса E_a = 134 кДж/моль. В диапазоне температур 1023–1123 К получены линейные зависимости. Для расчетов глубины проникновения процесса осаждения нитрида бора в пористое тело было принято, что газовая смесь состоит из трихлорида бора, аммиака и аргона ($BCl_3 + NH_3 + 30Ar$). Результаты расчетов показали, что реагентом, лимитирующим глубину проникновения, является трихлорид бора. Были определены глубины проникновения процесса химического осаждения нитрида бора из газовой фазы (CVI-BN – *chemical vapor infiltration boron nitride*) при давлении 0,1 кПа в температурном интервале 1100–1400 К при диаметрах пор 1, 10, 30, 100, 200 и 300 мкм. При наличии результатов порометрии конкретной преформы полученные зависимости глубины проникновения процесса CVI-BN в пористое тело от температуры при определенных условиях позволяют оценить необходимые параметры процессов формирования интерфазы из пиролитического нитрида бора.

Ключевые слова: пиролитический нитрид бора (ПНБ), интерфазное покрытие, термодинамический расчет, химическое осаждение нитрида бора из газовой фазы

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Introduction

Interest in pyrolytic boron nitride (PBN), which boasts a range of exceptional properties (high dielectric properties, chemical resistance in aggressive gases, liquids, and metal melts, heat resistance, low microwave energy absorption coefficient), remains unabated to this day [1–5]. Due amalgamation of these favorable attributes and their endurance at elevated temperatures and in deep vacuum conditions, this material has found extensive utility in electronics, electrical engineering, nuclear power engineering, the semiconductor industry, high-temperature technology, and the fabrication of containers and protective screens for synthesizing ultra-pure compounds.

At the turn of the century, with the advancement of silicon carbide fibers and materials derived from them, PBN began to serve as an interphase between fibers and matrices, imparting robust mechanical properties to SiC_f/SiC_m composite material [6–10]. The evolution of ceramic fibers encompassing diverse compositions (Si_3N_4, AlN, BN, etc.) and the composites founded upon them will further solidify the demand for PBN, functioning both as an interphase and matrix.

Simultaneously, it's imperative to acknowledge that the parameters for establishing the interphase on fibers or the matrix of composite materials via the chemical vapor infiltration (CVI) technique within a porous structure are fundamentally influenced by the preform's porous structure (pore size, tortuosity, etc.). In contemporary contexts, diverse reinforcement schemes and techniques for fabricating fiber preforms are employed in structures [11–13], each delineating a unique porous structure. This accentuates the necessity to tailor CVI process parameters according to the specifics of each structure.

This paper presents an analysis of the admissible parameter regions for the boron nitride deposition process via the CVI method, taking into account varied pore sizes and thicknesses of porous bodies.

Thermodynamic analysis of the B–Cl–N–H system

In order to determine the acceptable parameters ranges for the chemical deposition of boron nitride from the gas phase (temperature (T), pressure (P), and the ratio of initial reagents in the gas mixture), the TERRA software package (Russia, Moscow, Bauman Moscow State Technical University), was employed for thermodynamic analysis of the B–Cl–N–H system.

In the initial stage, equilibrium compositions of the B–Cl, N–H, B–Cl–N, and B–Cl–H systems were scrutinized to approximate the initial temperatures of boron trichloride and ammonia disproportionation. The impact of hydrogen and nitrogen on this process under varying pressures within the system was also evaluated. The analysis, illustrated in Figure 1, *a*, indicates that at $P = 10$ kPa, BCl_3 remains stable up to $T = 1800$ K. Beyond this threshold, the process of disproportionation into boron dichloride and monochloride is initiated. In the N–H system, where ammonia is a key component, its decomposition into nitrogen and hydrogen is nearly complete at $T = 600$ K and $P = 10$ kPa (Figure 1, *b*).

The introduction of nitrogen into the B–Cl system does not lead to the formation of boron nitride and does not significantly impact the thermodynamic stability of boron trichloride (Figure 1, *c*). However, the inclusion of hydrogen in the B–Cl system markedly diminishes the thermodynamic stability of BCl_3 (Figure 1, *d*). This situation introduces the potential for boron to form in the condensed phase, with its proportion increasing alongside higher hydrogen content in the mixture. The analysis also indicates that decreasing pressure within the system decreases the thermodynamic stability of boron trichloride and ammonia.

The introduction of ammonia, which contains hydrogen, into the B–Cl system reduces the thermodynamic stability of boron chlorides and leads to the formation of boron nitride across a wide temperature range.

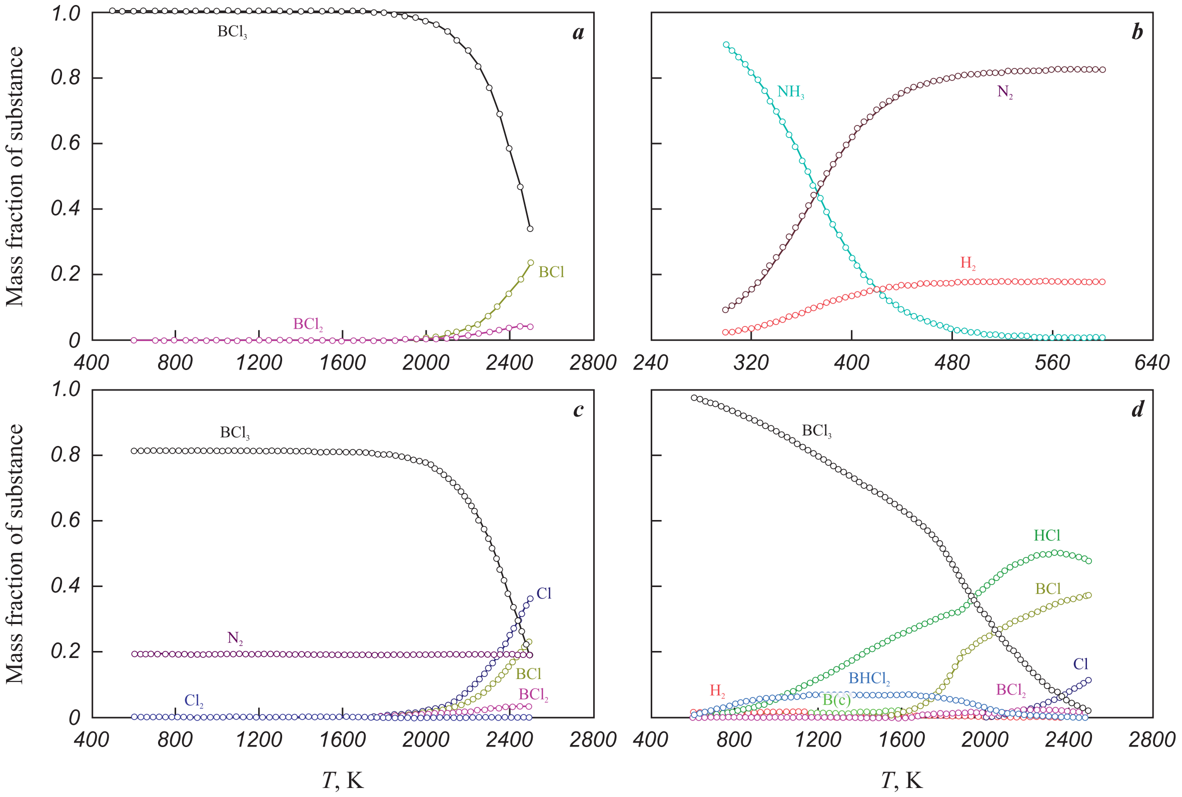

Fig. 1. Equilibrium composition of systems at 10 kPa pressure
a – B–Cl system formed by the BCl_3 gas mixture; ***b*** – B–N formed by NH_3;
c – B–Cl–N formed by $BCl_3 + N_2$; ***d*** – B–Cl–H formed by $BCl_3 + H_2$

Рис. 1. Равновесный состав систем при давлении 10 кПа
a – система B–Cl, образованная газовой смесью BCl_3; ***b*** – B–N, образованная NH_3;
c – B–Cl–N, образованная $BCl_3 + N_2$; ***d*** – B–Cl–H, образованная $BCl_3 + H_2$

Figure 2 illustrates the analysis results, showcasing the relationship between the yield of condensed boron nitride in the B–Cl–N–H system and temperature at a pressure of 10 kPa, while varying the initial reagent ratios. The findings reveal (Figure 2, *a*) that when BCl_3:NH_3:H_2 = 1:1:0, the thermodynamic stability of boron trichloride is sustained nearly until 2200 K, but this doesn't yield the maximum output of boron nitride. The introduction of hydrogen into the $BCl_3 + NH_3$ gas mixture promotes an escalation in the extent of boron trichloride decomposition, subsequently enhancing the theoretical yield of boron nitride (Figure 2, *b*, *c*). However, elevating hydrogen concentration beyond the ratio of 1:5 (BCl_3:NH_3:H_2 = 1:1:5) does not yield further increases in boron nitride yield, as near-complete decomposition of boron trichloride is already achieved.

It's noteworthy that augmenting the hydrogen content within the system could be accomplished by increasing the amount of ammonia in the mixture. For instance, with the BCl_3:NH_3 = 1:3 (Figure 2, *d*), the theoretical yield of condensed-phase boron nitride becomes nearly equivalent to the situation where hydrogen is introduced into the system with a BCl_3:NH_3:H_2 = 1:1:5 (as shown in Figure 2, *b*).

Consequently, the thermodynamic analysis of the equilibrium composition within the B–Cl–N–H system, generated from the gas phase through the utilization of initial reagents BCl_3, NH_3 and H_2, facilitated the assessment of the admissible parameter range for the chemical deposition process of boron nitride. The acquired outcomes provided the foundation for constructing the experimental matrix for PBN) production.

Experimental procedure

The following reagents were employed to investigate the kinetic patterns of the PBN deposition process:

BCl_3 – grade A boron trichloride (TU1-00-07526811-23-92);

– NH_3 – ammonia (GOST 6221-90);

– Ar – high-purity argon (GOST 10157-79).

In order to explore the kinetic trends of PBN coating formation on impermeable substrates, we utilized samples crafted from C-3 grade graphite, manufactured by JSC "Karbotek" (Moscow, Russia), featuring pyrolytic carbon coating. For porous substrates, we employed preform (skeleton) samples generated through the hydrajet method from Keralan silicon carbide ceramic

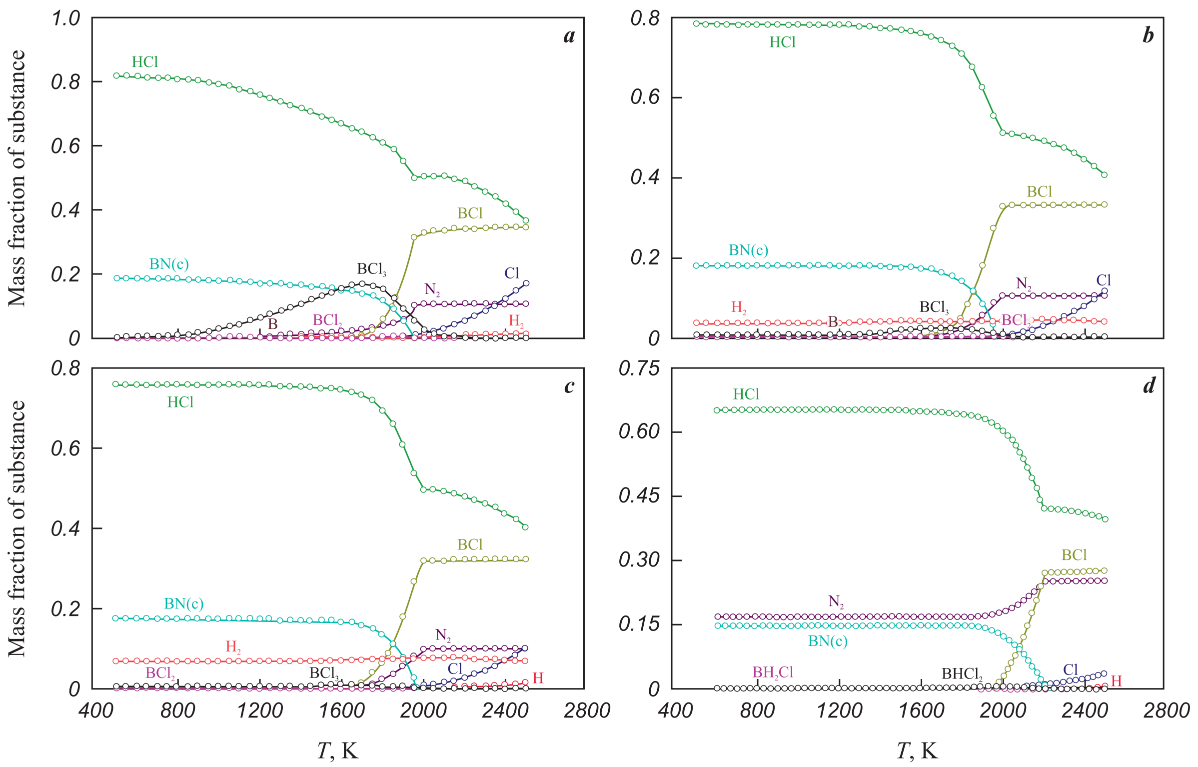

Fig. 2. Equilibrium composition in the B–Cl–N–H system at 10 kPa pressure,
formed by gas mixtures $BCl_3 + NH_3$ (***a***), $BCl_3 + NH_3 + 5H_2$ (***b***), $BCl_3 + NH_3 + 10H_2$ (***c***) and $BCl_3 + 3NH_3$ (***d***)

Рис. 2. Равновесный состав в системе B–Cl–N–H при давлении 10 кПа,
образованной газовыми смесями $BCl_3 + NH_3$ (***a***), $BCl_3 + NH_3 + 5H_2$ (***b***), $BCl_3 + NH_3 + 10H_2$ (***c***) и $BCl_3 + 3NH_3$ (***d***)

filament (TU 20.60.13-952-56897835-2022), produced by JSC "Kompozit" [14].

The porosity of the preform was evaluated using reference porosimetry via the reference contact porometer Porotech 3.2 at the Leonov University of Technology (Korolyov, Moscow Region, Russia).

Deposition of pyrolytic boron nitride was conducted using the BN 2.0 plant (Leonov University of Technology). The presence of the interphase BN coating and its thickness on framework fibers were assessed via scanning electron microscopy (SEM) utilizing a JSM-6610 LV device (JEOL, Japan).

The phase composition of PNB was ascertained using an *X*-ray diffractometer EMRYREAn (PANalytical, the Netherlands).

Results and discussion

The chosen parameters for the chemical vapor deposition (CVD) process were based on thermodynamic analysis:

$P = 0.1$ kPa for the temperature range of 1023–1123 K at a ratio of reagents of $BCl_3:NH_3 = 1:1$;

$P = 0.2$ kPa for $T = 2023 \div 2123$ K at $BCl_3:NH_3 = 1:3$.

In both cases, argon was employed as the carrier gas for boron trichloride.

Since the commencement temperatures for the decomposition of ammonia and boron trichloride are notably disparate (as seen in Figure 1, *a*, *b*), it is prudent to separately introduce them into the reaction zone to prevent premature interaction. This was accomplished in the BN 2.0 plant through a coaxial tube.

Figure 3 depicts the dependency of the boron nitride coating deposition rate constant on temperature, as obtained from [3] (where hydrogen was employed as the carrier gas), alongside the results derived by the authors of this paper. Thermodynamic analysis revealed that within the low-temperature range (1000–1200 K), the theoretical yield of condensed-phase boron nitride in the is relatively consistent for a $BCl_3:NH_3$ mixture ratio of 1:1, both with and without hydrogen. Meanwhile, within the high-temperature range, the introduction of hydrogen into the mixture or an increase in ammonia content amplifies the theoretical yield of condensed-phase boron nitride. Similar conclusions were reached in experimental studies conducted by [15–19].

The analysis of kinetic dependences acquired from [3] uncovers the existence of three process regions in boron nitride deposition:

K – kinetic region (1400 K and below);
D – diffusion region (1800 K and above);
T – transient region.

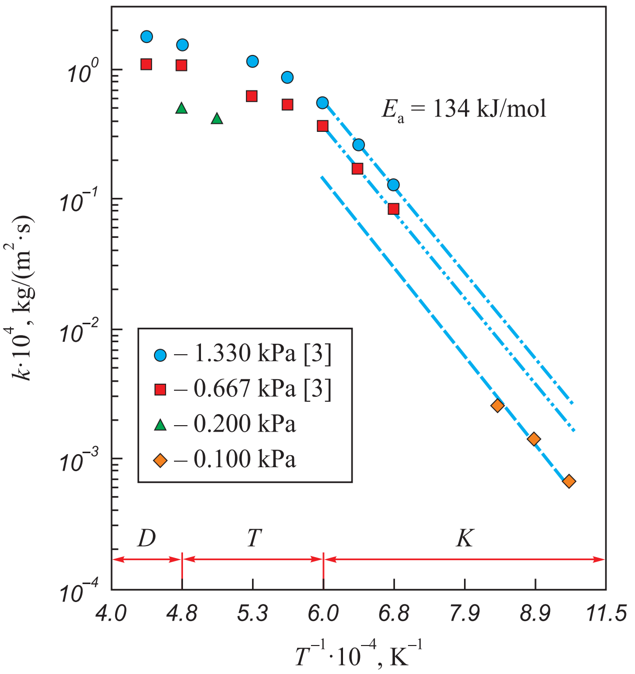

Fig. 3. Dependence of the deposition rate constant of a pyrolytic boron nitride coating on temperature

Рис. 3. Зависимость константы скорости осаждения покрытия из пиролитического нитрида бора от температуры

In the kinetic region, the activation energy of the process was determined to be $E_a = 134$ kJ/mol. In the temperature range of 1023–1123 K, linear dependences were also observed. Their similarity to the dependencies obtained in [3] suggests a comparable activation energy, despite the variations in process pressure. This could be attributed to the fact that within the considered pressure range of 0.1÷0.2 kPa, the deposition rate's correlation with pressure and, consequently, reagent concentration in the kinetic region is not pronounced. As a result, the process rate is dictated by the reaction rate, whose mechanism remains consistent between the conditions outlined by the authors in [3] and our study.

In the future, as we refine the results, adjustments might be made to the analysis of the activation energy. In that case, for the kinetic region, the boron nitride deposition rate constant (k, kg/(m^2·s)) at a pressure of 0.1 kPa, using a reaction mixture of $BCl_3 + NH_3$ in a 1:1 ratio, can be expressed as follows:

$$k = 0.24\exp\left(-\frac{134{,}000}{RT}\right), \tag{1}$$

where R is the universal gas constant, J/(mol·K); T is the temperature of the PBN deposition process, K.

It is worth noting that the density of PBN obtained within the temperature range of $T = 1023 \div 1123$ K is approximately $1.4\cdot10^3$ kg/m^3, while for the temperature range of 2023–2123 K, it is on the order of $2.0\cdot10^3$ kg/m^3. Drawing from the findings of [3], we can hypothesize that the dependence expressed by equation (1) remains valid up to at least 1400 K, with possible deviations linked to changes in PBN density.

Determining the kinetic region stands as a pivotal stride, enabling the progression to the modeling of the PBN deposition process on the surface of a porous preform's pores. The central objective in investigating the kinetics of PBN deposition within a porous preform is to ascertain the depth of the coating formation reaction's penetration on the pore surface. This, in turn, facilitates estimating the maximal thickness of the preform – a volume within which PBN will precipitate onto the pore surface. To achieve this estimation, the computational-theoretical model outlined in [20] can be effectively utilized. It can be expressed as follows, taking the form of a first-order reaction equation:

$$L_{\max} \approx \left[\frac{\lambda d\sqrt{\dfrac{RT}{3M}\left[1-\exp\left(-\dfrac{d}{\lambda}\right)\right]}}{4X^3k}\right]^{1/2}, \tag{2}$$

where $L_{\max}$ is the depth of the reaction's penetration into the porous preform, m; λ is the free path length of a gas molecule, m; d is the pore diameter, m; M is the mass of a gas molecule, kg; k is the rate constant of the heterogeneous reaction on the surface, kg/(m^2·s); X is the tortuosity coefficient, kg/m^3.

For a gas phase composed of a mixture of molecules from various substances 1, 2, 3 ..., the path length is equal to [19]

$$\lambda_1 = KT\left[\pi\left(N_1\sigma_1^2\sqrt{2} + N_2\sigma_2^2\sqrt{1+\frac{M_1}{M_2}} + N_3\sigma_3^2\sqrt{1+\frac{M_1}{M_2}} + \ldots\right)\rho\right]^{-1}, \tag{3}$$

where N is the mole fraction; K is the Boltzmann constant, J/K; σ is the molecule diameter, m; P is the pressure within the system, Pa; ρ is the density of the gas phase, g/cm^3.

For the analysis, it was considered that the gas mixture comprises $BCl_3 + NH_3 + 30Ar$ composition, wherein boron trichloride and ammonia are determined via thermodynamic analysis, and argon is employed as a diluent gas. In order to mitigate random errors arising from using molecule diameter (σ) values from disparate sources, a standardized calculation procedure was implemented employing the formula

$$\sigma = 1.329\cdot10^{-8}\left(\frac{M}{\rho}\right)^{1/3}, \tag{4}$$

where ρ is the density of the condensed phase, kg/m^3 ($\rho_{BCl_3} = 1.345 \cdot 10^3$ kg/m^3 [21]).

As a result, the following values were obtained: $\sigma_{BCl_3} = 5.89 \cdot 10^{-10}$ m; $\sigma_{NH_3} = 3.88 \cdot 10^{-10}$ m; $\sigma_{Ar} = 4.15 \cdot 10^{-10}$ m and $\lambda_{BCl_3} = 9.05 \cdot 10^{-6}\, T/P$ m; $\lambda_{NH_3} = 21.5 \cdot 10^{-6}\, T/P$ m.

The tortuosity coefficient for woven and needle-punched frameworks with a density of (0.4–0.5)·10^3 kg/m^3 was approximated to be 1.3 [22].

The analysis revealed that the limiting reagent dictating the extent of penetration of the PBN deposition reaction into the porous body $L_{\max}$ is boron trichloride. Figure 4 depicts the calculated relationships of the depth of PBN deposition penetration onto pores of varying diameters within the temperature range of 1100–1400 K, at a pressure of 0.1 kPa.

The pore diameter in preforms of ceramic matrix composite materials is contingent on the reinforcement schemes and potential subsequent process stages aimed at obtaining porous blanks. Thus, within the pore space of 3D and 4D structural reinforcing frameworks, distinct types of porosity emerge: inter-strand (inter-rod) and inter-filament. In the former scenario, the dimension of inter-strand (inter-rod) pores is determined by the diameter of the strand (rod). For instance, in a 4D framework crafted from rods of 0.7 mm diameter, the inter-rod pore diameter measures 135.45 μm [22].

In preforms showcasing a 2.5D structure anchored on UT-900P carbon fabric interwoven with Ural-NSh24 carbon thread, three categories of pores have been discerned [22]:

1) pores in the range of up to 0.8 μm in diameter originated from fiber imperfections and resin coke and pyrocarbon porosity;

2) the set of pores sized between 0.8÷3.6 μm primarily arises from inter-filament pores;

3) the assortment of pores encompasses both inter-strand pores ($d = 3.6$ to 36.0 μm) and interlayer pores ($d > 36.0$ μm).

In a similar preform, albeit fashioned from Ural-TM/4 carbon fabric coupled with Ural-NSh24 carbon thread, the prevalent pores possess radii of 0.1÷1.0 μm (coke pores and inter-filament pores), and 1–10 μm (inter-filament (inter-strand) pores). Pores with a radius less than 0.1 μm or exceeding 10 μm are less abundant in the material. Discrepancies in the porous structure's characteristics are ascribed to the distinct properties of Ural-TM/4 and UT-900P fabrics. Furthermore, the filament diameter, strand diameter, and framework density exert an impact on the pore size.

In [14], three primary observable pore sizes that contribute to the overall porous structure were identified for the nonwoven framework crafted from Siqulox silicon carbide fibers (JSC "GNIIChTEOS", RF): 15, 40 and 250 μm. Similar findings were also obtained for frameworks relying on Keralan carbide-silicon fibers (JSC "Komposit", RF) [14].

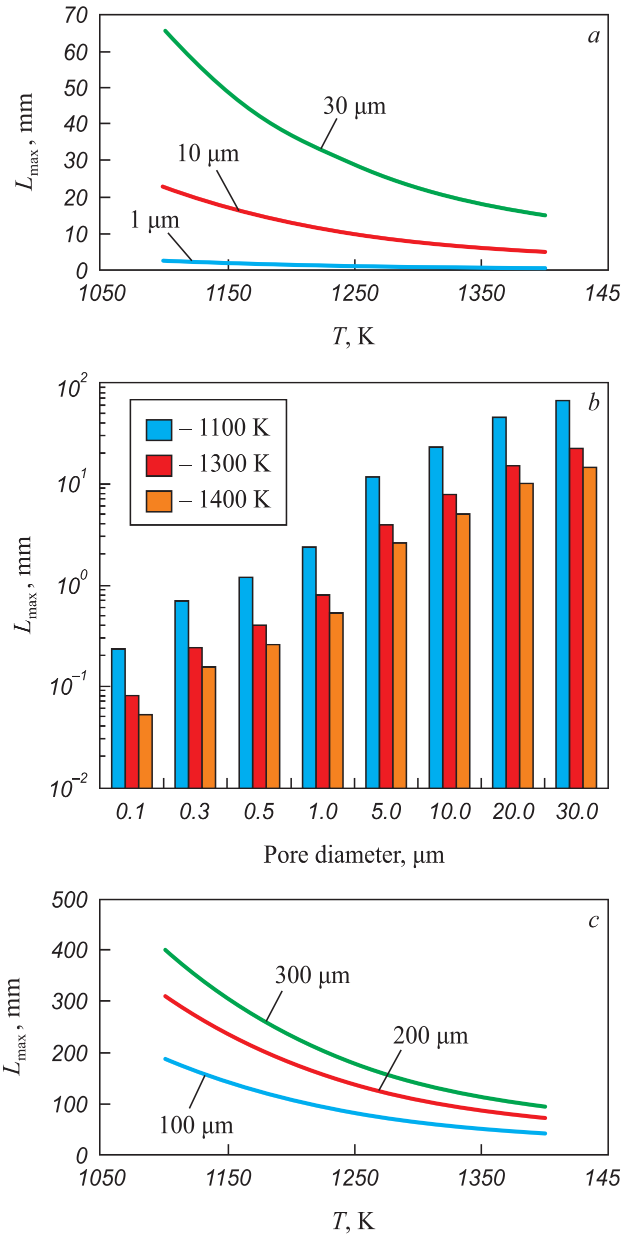

Fig. 4. Penetration depth of the CVI-BN reaction at $P = 0.1$ kPa into a porous body at $T = 1100–1400$ K (***a***), 1100, 1300 and 1400 K (***b***) and 1100–1400 K (***c***)
The numbers next to the curves are the pore diameter

Рис. 4. Глубина проникновения реакции CVI-BN при $P = 0{,}1$ кПа в пористое тело при $T = 1100÷1400$ К (***a***), 1100, 1300 и 1400 К (***b***) и 1100–1400 К (***c***)
Цифры у кривых – диаметр пор

Utilizing the outcomes of these computations, the PBN deposition mode at $T = 1100÷1400$ K and $P = 0.1$ kPa into the nonwoven framework founded on Keralan carbide-silicon fibers was subjected to testing. Figure 5 illustrates the microstructure of the acquired sample, showcasing the silicon carbide framework with a PBN interphase coating.

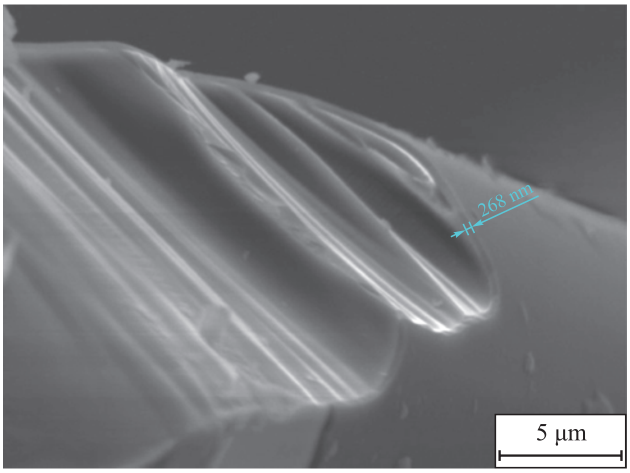

Fig. 5. Formation of a pyrolytic boron nitride interphase coating with a thickness of ≈250 nm on Keralan silicon carbide fiber

Рис. 5. Внешний вид интерфазного покрытия пиролитического нитрида бора толщиной ≈250 нм на карбидокремниевом волокне Кералан

The findings from determining the phase composition of the fiber sample reveal the existence of a hexagonal modification (*P6mc*) on the surface of PBN carbide-silicon fibers. This hexagonal modification possesses a low shear strength within its fundamental plane, which is crucial for facilitating the functionality of the interphase coating [8].

Conclusion

Hence, the outcomes of porosimetry conducted on specific preforms and the established relationships depicting the penetration depth of the CVI-BN process under specific conditions within the porous structure provide us with the means to estimate the essential parameters for crafting interphases using PBN and creating composites with a matrix composed of PBN.

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