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Controlled Synthesis of TiB_2-TiC Composite: Substantiation of the Homogenizing Joule Thermostatting Efficiency and Improvement of SHS-Compaction Technology in a Vacuum

Jandieri G.*[0000-0003-2976-1201], Sakhvadze D.[0000-0001-9780-7027]

Rafiel Dvali Institute of Machine Mechanics, 10, Elizbar Mindeli St., 0186, Tbilisi, Georgia

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***Corresponding email:**
gigo.jandieri@top.ge

Abstract. This research aims to improve and substantiate the efficiency of homogenization heat-stabilizing Joule heating on ceramic-matrix composites of TiB_2-TiC system with a 2:1 component ratio during its synthesis. For this purpose, an improved technological approach is proposed, which is based on the known method of SHS-compacting but differs by the possibility of controlled Joule influence on the synthesis products, which is achieved by the use of a special electrothermal vacuum press-mold functioning according to a particular control algorithm. The task of controlled Joule heating is a compensation of the temperature gradient formed in the synthesized workpiece, which is solved by passing in it a direct current directed in line with the vector of propagation of the combustion wave. An indicator of assessment of the degree of compensation of the noted temperature gradient is the Seebeck effect, excited between the upper and lower surface of the SHS workpiece, which should be brought to zero in the process of Joule thermostatting. It was experimentally revealed that compensation of the noted temperature gradient with heat released predominantly by electrically conductive and Joule-heated TiC grains leads to their softening, which contributes to more uniform compaction of the workpiece due to diffusion coalescence of these grains around prism-shaped hard TiB_2 crystals. Such consolidation leads to a significant increase in the quality of structural packaging and a reduction in the number and volume of micropores, as a result of which the performance properties of the composite improve on average by 10–15 %.

Keywords: ceramic-matrix composite, Joule heating, Seebeck effect, microstructure homogenization.

1 Introduction

Ceramic-matrix composites based on titanium diboride (TiB_2) and titanium carbide (TiC) have attracted great interest in recent years since they have superior properties compared to single-phase ceramics TiB_2, involving high hardness, good wear resistance, and high fracture toughness [1]. Research on creating new critical materials based on the noted ceramic-matrix compounds is intensive. The achievements outlined in the publications can be distinguished [2, 3]. According to these studies, composites based on titanium borides and carbides, with different ratios of these components, are promising materials for their use in producing wear-resistant and shock-resistant parts, such as dies of industrial press machines and cutting tools for machine tools. They also demonstrate good characteristics, such as high-temperature structural components in nuclear reactors, heat exchangers, and engines.

According to the study [4], to ensure the best mechanical strength and fracture toughness, it is recommended to keep the optimal TiB_2:TiC ratio equal to 1:1. According to the authors of this study, only in this case is available homogenization of the crystal structure and reduction of concentrations of non-equilibrium thermal stresses which is caused by the predominant presence of unevenly distributed needle-shaped grains of TiB_2 in the structure. However, this approach is not always acceptable, as it is associated with the need to use reduced concentrations of boron and reduce the number of borides formed. Boron is essential for the targeted composite's heat, radiation, and corrosion resistance [5, 6].

Compared to conventional ceramic-metal materials, ceramic-matrix composites based on TiB_2 and TiC

compounds have higher hardness and chemical stability at high temperatures and are considered a good alternative for known wear-resistant metal-cutting tools based on tungsten, cobalt, and nickel carbides WC-Co-Ni [7].

On average, the Vickers hardness of the composite metal-ceramic workpieces of the noted system is 20 GPa. In contrast, this mechanical strength index of the much cheaper in-production ceramic-matrix composite TiB2-TiC, according to current synthesis technologies, which ensure forced compaction of blanks using gravitational overloads (500–2500 g), can reach 25–30 GPa, and it still has a reserve of increase [8, 9].

Notably, unlike cutting tool materials based on tungsten carbides with cobalt and nickel, the TiB_2-TiC composite, depending on the concentration and topology of the TiB_2 compound distribution, does not show a significant deterioration of hardness up to heating at 950–1150 °C, whereas, in products made of tungsten carbide with cobalt and nickel bonding, Vickers hardness the heat resistance temperature, depending on the content of the noted metals, can deteriorate already in the temperature range of 800–1000 °C. Simultaneously, attempts to improve the operational properties of this cermet by adding various other refractory compounds (for example, $MoSi_2$) can lead to a deterioration in its oxidation resistance [10], which is also not always an acceptable possibility.

Despite the apparent advantages of ceramic-matrix composites of the TiB2–TiC system over metal-ceramic ones, their widespread use as machine-building workpieces is still limited due to the complexity of their final homogenizing thermomechanical processing and giving them the condition of a finished product [11, 12]. This problem is especially manifested when it is necessary to manufacture precision, completely interchangeable parts or components of high-responsibility machine-building units with a complex configuration of working surfaces, which need incredibly accurate basing, uniformity of structural and mechanical properties, and purity of actuating surfaces, which is practically unattainable at a stage of their free synthesis and requires additional operational intervention with the use of different methods of physical and mechanical processing. Among these methods, the technological approach using laser-induced controlled oxidation by spark plasma in combination with the micro-milling of critical surfaces of the target workpiece can be distinguished by its complexity and laboriousness [13].

Based on the above, it can be emphasized that a significant part of future research should be focused on improving existing methods for the synthesis of this material, the main goal of which should be to ensure the direct production of products ready for use, homogenized in structure and morphology, eliminating the need for subsequent heat treatment and the use of labor-intensive special methods of mechanical, electrical discharge, ultrasonic or laser finishing.

2 Literature Review

Regarding technical and economic efficiency and production flexibility, the most acceptable method for synthesizing refractory and composite metal-ceramic materials can be considered the reactionary, self-propagating high-temperature synthesis (SHS). This method has also been successfully tested and is used to obtain powder materials of the TiB_2–TiC system, intended as raw materials for further production of ceramic-matrix composite products [14]. However, from the point of view of the possibility of directly producing compacted, integral structural products from this two-component composite, due to its manufacturability and prospects, the SHS-compaction technology is considered to have no alternative [15].

The advantages of SHS compaction technology include combining the exothermic autowave process of directed solid-phase reaction combustion with pressure processing of materials, which directly produces carbide materials and products, ensuring the desired density and geometric dimensions of the executive surfaces. The SHS compaction process itself, including assembly and disassembly of the mold, takes place in 2–3 minutes, which makes this technology especially promising and puts it in an advantageous position over traditional methods of manufacturing similar products, which involve the preliminary production of briquettes with the external shape of the target workpiece, followed by hot, long-term sintering of the compacted powder mixture in a vacuum or in an atmosphere of inert gases [16]. The absence of such necessity and the possibility of a direct operational synthesis process without the use of energy-intensive furnace equipment and complex, expensive systems of their technical operation are the main factors in the higher technical and economic efficiency of a method of SHS-compacting.

However, there remains one significant unsolvable problem, the essence of which is that after the completion of the combustion process inside the powder mixture and the synthesis of the target ceramic-matrix material located between the punch and the matrix of the press-mold used, an unevenly distributed temperature field is formed. The temperature of the zone of primordial initiation (start) of high-temperature self-propagating combustion always differs from that of the opposite (finishing) zone of combustion completion. It is perceptibly lower than the latter. The temperature gradient inside such a billet, depending on its thickness and the composition of the initial reaction mixture, can reach 200–300 °C.

Consequently, when pressed in a mold, the synthesized workpiece is compacted unevenly due to the significant difference in plastic properties across the cross-section. The resulting error in dimensions and the final product's physical and mechanical properties is an irreparable defect. The greater the TiB_2 boride phase formed, the more distinctly this problem is revealed.

The problem of a gradient temperature field and the associated increased heterogeneity of the physical and mechanical properties of the resulting SHS workpiece is

especially revealed when it is necessary to increase the specific amount of boron-containing component in it, for example, when the component ratio TiB_2:TiC is higher than 1:1 (TiB_2 > 50 % wt). It is necessary to note this problem because materials with a high boron content are highly resistant to extremely high-temperature loads and radiation. This is important since the duration of their life cycle inside nuclear reactor vessels is determined (regulated) based on the temperature and radiation resistance of structural and protective boron-containing ceramic-matrix materials.

The irradiation of materials at high temperatures during reactor operation causes changes in microstructure, leading to the deterioration of mechanical properties and the distortion of geometrical shape and external dimensions. As a result, all this leads to embrittlement, increased creep, swelling, and other highly undesirable defects of the used protective ceramic-matrix materials [17].

American researchers are developing [18], the essence of which is to combine the SHS-compaction with the instantaneous cumulative (shockwave) compaction of synthesis products. However, this development path of SHS technology could not become generally available, technically safe, and commercially acceptable since its implementation was associated with many features available only for a few military-industrial complexes of some highly developed countries. In addition, due to the ultra-high pressing speeds, the problem of degassing the resulting billet remained unresolved. Subsequently, many unevenly distributed gas voids remain inside the product, which gives it increased brittleness and instability to sign-variable shock-cyclic thermophysical loads.

The analysis of SHS-compacting process peculiarities shows that the main difficulty in controlling this process is both excessively high rate of exothermic autowave synthesis reaction (front advance rate of 5–7 cm/s) and rapid cooling of the billet obtained after completion of this process (150–200 °C/min). The importance of this factor is thoroughly emphasized in the study [19], the purpose of which was to clarify the influence of the cooling rate on the formation of carbides in the Ti-C-Al system. According to the results of the noted study, high cooling rates prevent the formation and preservation of carbide phases (TiC), which is very undesirable in this case since in order to obtain the target, high-quality, structurally homogenized ceramic-matrix TiB_2-TiC composite, the necessary condition is to ensure the maximum possible formation and homogeneous distribution of the TiC phase between TiB_2 crystals.

Based on this feature, the methods of impact action on the surfaces of synthesized workpieces proposed in the above study [18] are not rational solutions in terms of controlling the structure, phase composition, and physico-mechanical properties of the target product since these effects last for a very short time (fractions of a second) and set itself as the primary goal to solve only one problem - the maximum possible compaction of the target product.

Based on the above analysis, it is evident that obtaining the possibility of flexible control of the SHS-compacting process in the synthesis of ceramic-matrix composites with increased content of borides and carbides of titanium is an urgent problem. Its successful solution can lead to significant technical progress in directly producing high-quality, structurally homogenized products stabilized by properties products from bicomponent ceramic-matrix material of TiB_2-TiC system with an increased ratio of boride phase.

Notably, the continuum theory of plastic flow of compressible media is used for the theoretical description of the process of deformation and compaction of powder bodies [20]. Many scientists have proposed numerous rheological models reflecting the complex dilatancy character of the deformation of quasi-continuous bodies. At the same time, there is no theoretical justification for structural models and plasticity conditions that consider the unbound initial state and localization of plastic deformation in the contact volumes of powder particles.

As a rule, the object of the continuum theory of plasticity of quasi-continuous bodies is a two-phase material consisting of a solid phase and pore space. In contrast, as a rule, hot SHS products at the deformation temperature, in addition to solids and pores, can contain a liquid phase in the form of a melt of fusible components in a short period. However, known models do not consider the effect of the liquid phase on the rheological properties of porous bodies with a viscous substance. The shortcomings of the general theory of plasticity of compacted bodies determine the relevance of developing new and refinement of known continuous models of deformable solid and solid-liquid powdered bodies.

The importance of the noted is proved in the study [21], which is devoted to the study of the problems of compaction of Ti-B compounds obtained using SHS technology. Here, due to the improved thermodynamic modeling of the diagram of state and phase transformations of compounds of the Ti-B system, the necessity of considering and practical use of the phenomenon of short-term formation of the liquid-phase component was proved. It is shown that this can lead to an increase in the uniformity of axial compaction of titanium-boron-containing functional blanks obtained by SHS-compacting. The result is achieved by lengthening the lifetime of the highly plastic (softened) liquid crystalline non-equilibrium mesomorphic phase (mechanical solution of Ti_3B_4-Ti), formed in the aftermath of high-temperature synthesis and maintained by Joule heating, with the simultaneous influence of external pressing pressure. Experimental studies [22, 23] showed that the most reliable method of stable Joule heating and heat conservation is the contact electric resistive internal heating of the synthesized sample, for which an electric current of low voltage is sufficient. It is established that in the range of boron concentrations of 18–32 % wt., the lower permissible values of SHS-workpiece pressing temperature are in the temperature range of 1537–2167 °C, and the upper ones in the range of 2727–3217 °C, respectively.

Equalization and retention of the noted temperature range can be achieved by applying an electric voltage of

3–4 V to the synthesis products with a current of 600–550 A, with a frequency of 50 Hz and below.

The mentioned study [24] proved the fundamental possibility and efficiency of improving the quality of SHS-compaction products by optimizing the compression temperature modes and controlling the cooling rate of the functional-gradient workpiece of the Ti-B binary system after the completion of its synthesis. This study shows the effectiveness of lengthening the duration of the stage of structure-phase formation, thereby achieving its transformation from a dynamically directed one, accompanied by the combustion front, into a volumetric multi-axis (i.e., homogenized). In parallel, the forced compaction process is extended to achieve the maximum possible effect of infiltration and extrusion of the mechanically mixed liquid crystalline mesomorphic phase Ti_3B_4-Ti, retained by the Joule thermostatting influence around solid TiB_2 crystals. This is important since only in this way is it possible to fill the pores and heal intergranular microcracks formed due to the high-speed passage of the high-temperature wave of reaction combustion with the intense release of gases desorbed from the surfaces of the fused powder particles. During Joule thermostatting, in a workpiece with a preserved highly pliable liquid-crystalline mesomorphic phase, due to the equalization of the internal temperature field (minimization of the gradient), homogenization of plastic properties is also ensured, which leads to a more uniform compaction of the target product. In this case, the degree and uniformity of forced force compaction are guaranteed to improve, ensuring dense and durable products are produced without open macro and micropores, cracks, and significant residual deformation stresses. It has been established that, from the point of view of controllability of the SHS-compaction process of the Ti-B system composite, to maintain high compliance to compaction, preference is given to reaction mixtures with excess titanium, capable of forming the Ti_3B_4 compound. Excess titanium guarantees the obligatory formation and subsequent Joule maintenance of a highly pliable, mesomorphic liquid-crystalline phase.

Study [24] also indicates the effectiveness of energy stimulation of SHS processes, but unlike the noted experience, the approach proposed by the authors involves preliminary Joule heating of the reaction mixture to bring it to the explosive ignition temperature. At the same time, to obtain the effect of electrical breakdown and achieve the desired result of explosive ignition, an alternating electric current with a high-frequency voltage of 10 kHz is used.

There is also a known method involving the use of preliminary induction (or ultra-wave) heating of a workpiece from a pre-briquette reaction mixture, followed by shockwave (explosive) initiation of the process of self-propagating high-temperature synthesis [25]. After completing this process, it also becomes possible to influence the resulting product's cooling parameters and control its phase composition, microstructure, and mechanical properties. However, due to the need for remote exposure to an inductive electric field and indirect heating of powder particles in the reaction mixture, as well as due to the need to use non-metallic, non-magnetic structural materials (glass ceramics, quartz, corundum) in the press-mold, energy and technical-economic effectiveness of this approach is significantly inferior to the proposed method - direct Joule thermostatting.

Remarkably, inductive stimulation of SHS processes can only be carried out if the reaction mixture is dominated by the amount of metal part with high electromagnetic (inductive) permeability, for example, metallic nickel, titanium, and aluminum [25]. In the case of direct electrical contact, this limitation has disappeared.

In contrast to the mentioned technological solutions of Joule influence, requiring the use of special expensive equipment and technical tooling, with combined complex electrical circuit and implemented by special electrical materials, this study proposes a slightly different, technologically improved approach based on the use of rectified (direct) electric current, with the possibility of changing its polarity to direct it precisely to compensate for the undesirable temperature gradient, formed in the SHS-workpiece. The proposed approach should fulfill the requirement to be more economical, safer, technologically flexible, easily controllable, and thus more reliable regarding its stable serial industrial application.

Based on those mentioned above, the main aim of this study is to identify the technological features and substantiate the effectiveness of controlled energy-thermostabilizing Joule impact on the process of SHS-compacting in the synthesis of the ceramic-matrix composite of TiB_2-TiC system with the ratio of components 2:1.

3 Research Methodology

3.1 Theoretical substantiation of the approach's effectiveness

To theoretically substantiate the effectiveness of the proposed approach, it is necessary to show the sequence of analytical determination of the work *dA* performed by electric field forces on current carriers in a conventional section 1–2 of the SHS-sample during the time interval *dt*.

For this purpose, Figure 1 illustrates a simplified graphical model of the conditional elementary volume of the synthesized ceramic-matrix workpiece.

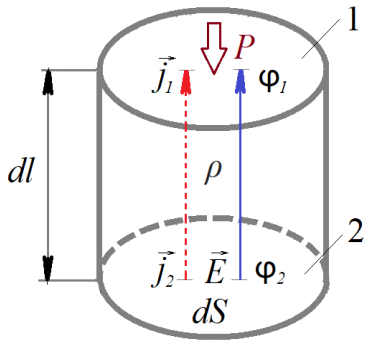


Figure 1 – Schematic model of Joule compensatory heating of representative-elementary volume of the SHS-synthesized workpiece: 1, 2 – surfaces

In the diagram (Figure 1), the following parameters are introduced: dS – the cross-sectional area of this representative-elementary volume of the workpiece; dl – height of the same volume of the workpiece; P – pressing pressure directed against the propagation of the combustion front (not shown in the diagram); ρ – density of the synthesized material; $\varphi_2 - \varphi_1$ – the potential difference between surfaces 2 and 1; j – a vector physical quantity characterizing the flux density of electric charge passing through surfaces 2 and 1.

At the specified interval dl, the following can be considered:

$$j_1 = j_2 = nq\upsilon = IS^{-1}, \quad (1)$$

where n – the concentration of charge carriers, i.e., electrically conducting particles (mainly TiC grains); q – the charge of these particles, according to another, the amount of electricity (i.e., a physical scalar value showing the ability of the body to be a source of electromagnetic fields and take part in electromagnetic interaction); υ – the average velocity of the current-conducting particles (in this case, the velocity of free electrons in the TiC compound); I – the current strength, is a scalar physical quantity that is equal to the ratio of the electric charge dq that has passed through the surface dS in an infinitesimal time dt corresponding to the duration of this period ($I = dq/dt$).

In the diagram of Figure 1, $E(\uparrow)$ – the electric field strength, i.e., a vector physical quantity characterizing the electric field at a given point. It is equal to the ratio of the current I acting on a stationary small point charge placed at a given point to the value of this charge dq).

The work dA, which is performed by the electric field forces over the charge carriers $dq = Idt$ on the conventional segment 2–1, is equal to

$$dA = (\varphi_2 - \varphi_1)dq = I(\varphi_2 - \varphi_1)dt, \quad (2)$$

According to the law of conservation of energy, the energy equivalent to work dA is released in the form of heat Q if the conductor is motionless and no chemical transformations occur in it, i.e., the conductor heats up (in our case, this corresponds to the period of completion of the reaction synthesis). Current carriers (electrons), as a result of the work of electric field forces E, acquire additional kinetic energy and then spend it to excite vibrations of the crystal lattice when colliding with its nodes (atoms). Then considering Ohm's law ($\varphi_2 - \varphi_1 = IR$) obtained:

$$dA = I(\varphi_2 - \varphi_1)dt = I^2Rdt, \quad (3)$$

but since the heat generated is equal to

$$dQ = dA = I^2Rdt = IUdt = U^2R^{-1}dt, \quad (4)$$

and the parameters that determine its quantity are equal to

$$I = jdS;\ R = \rho dl/dS;\ dSdl = dV, \quad (5)$$

the following differential form of the Joule-Lenz law can be obtained:

$$dQ = dA = I^2Rdt = (jdS)^2\rho(dl/dS)dt = \rho j^2dVdt, \quad (6)$$

To more accurately describe the process of heat release during SHS-thermostatting, it is necessary to consider the phenomenon of the formation of additional Thomson heat dQ^T, which will depend on the temperature gradient formed in the body of the sample immediately after the passage of the combustion front and the direction of the electric current passed through it [26].

According to the Thomson effect, if the direction of the current passing through the synthesized sample is opposite to the vector of the temperature drop inside this workpiece, the heat release will increase. In this case, the amount of additional heat released will be equal to

$$dQ^T = \tau\Delta T_{2-1}jdtdV, \quad (7)$$

where τ – the Thomson coefficient, which has the same dimension as the thermoelectromotive force, V/K; ΔT_{2-1} – gradient of the temperature field between conventional surfaces 2 and 1 of the synthesized workpiece (Figure 1).

Therefore, the total heat release will be equal to

$$Q^\Sigma = dQ + dQ^T = \rho j^2dVdt + \tau\Delta T_{2-1}jdtdV; \quad (8)$$

$$Q^\Sigma = (\rho j^2 + \tau\Delta T_{2-1}j)dVdt. \quad (9)$$

Based on the noted Thomson effect and the corresponding expression (7), it follows that for the effective use of additional Thomson heat, the cathode electrode for supplying a direct current to the sample should always be based on the side of the surface where the synthesis process is initiated, since this is the side that is the starting one for the propagation of the reaction combustion wave. It is this that will cool down first (predominantly). In the scheme shown in Figure 1, such a surface is plane 2. Consequently, the electromotive force to be excited between surfaces 1 and 2 ($T_1 > T_2$) will be directed from top to bottom $E(\downarrow)$ and will impede the motion of electrons driven by the DC electric field $E(\uparrow)$ applied from bottom to top. Consequently, this will result in the additional beneficial use of the Thomson effect and maximize the efficiency of Joule thermostatting.

From expression (6), it is clear that the thermophysical process of electro-resistive Joule thermostatting of a synthesized workpiece, in addition to the electrical parameters of the influence, can depend on the density (concentration) of the current-conducting structural-phase component in the synthesized material j, on its active volume dV, which falls in the zone of electrical influence and duration of Joule thermostatting dt. Consequently, in our case, with the accompanying pressing of the sample obtained after completion of the synthesis stage with a force P, a decrease in the volume dV and, as a consequence, a gradual increase in the distribution concentration of the conductive medium TiC, the amount of heat generated will change.

However, after reaching the maximum possible (close to theoretical) density value, the significance of the influence of the pressure factor P disappears. The variables remain the temperature gradient ΔT_{2-1}, the Thomson coefficient τ, and the duration of Joule thermostatting dt. Hence, the following statement follows: the external pressing pressure P and the supply of electrical energy

must be stopped when the termination of the increase in the power consumption indicator is recorded (displayed in the termination of the increase in the current consumption). Otherwise, due to local overheating of the central zone of the sample (the maximum amount of heat will begin to accumulate here), infiltration extrusion from it overheated (liquefied) conducting phase TiC and, consequently, gradual loss of active volume (concentration) of charge carriers, the opposite effect is obtained, which is expressed both in the reduction of released Joule heat and in the violation of the proportion of the ratio TiB_2:TiC. For example, a similar effect is fixed at excessive thermomechanical influence on synthesis products of a sample from a layer-by-layer functional ceramic-matrix composite of $TiB_{0.6}$-TiB_2 system, where a predominantly current-conducting component was a solid solution of free (stoichiometrically excessive) Ti with titanium monoboride TiB.

The effect of infiltration squeezing out over softened as a result of overheating of this conductive phase (Ti-TiB) is illustrated in Figure 2.

Figure 2 – A round disk-shaped sample obtained by SHS compaction in a vacuum with accompanying Joule heating (top and side views): 1–1' – sample without overheating (duration of Joule thermostatting is 1.5 min at a pressing pressure of 150 MPa); 2–2' – the same after overheating up to 1800–2000 °C (duration of thermostatting is 3.5 min)

In image 2–2', the softened mechanical solution of Ti-TiB ($TiB_{0.6}$) extruded from the central zone of the observed sample can be seen.

On the upper and lower surfaces of the resulting sample, thin residual layers from the burning of the graphite current-carrying electrodes are also clearly visible. Removing the latter and cleaning the surfaces of the sample to the required condition does not present any difficulty. Surfaces are quickly cleared when processed by grinding.

3.2 Used materials and technical equipment for Joule thermostatting

The materials and their proportions during the experiments were selected based on the need to solve the task of providing a one-stage, direct production of a ceramic-matrix composite of the TiB_2-TiC system, ready for operation, homogenized according to physical and mechanical characteristics, with the ratio of these components 2:1. Meanwhile, the initial particle size of titanium powder used for the synthesis of the above ceramic-matrix composite was in the range of 50–100 μm. The particle size of amorphous boron was 5–10 μm, and graphite – 2–4 μm.

Before the reaction mixture (charge) was prepared, the powders were subjected to desorption heat treatment in a PM-12M muffle furnace at a heating temperature of 150 °C (calcination in an inert gas atmosphere - argon). After cooling, the powders were subjected to mechanical activation treatment in a laboratory ball mill WZM-1L for 30 min.

After this treatment, the powders' dispersibility was reduced to 20–25, 1–2 and 1 μm, respectively. After that, they were homogeneously mixed and pre-briquetted under pressure of 10 MPa in the form of flat discs with a diameter of 30 mm and thickness of 10 mm.

For experimental studies of the SHS-compacting process, an improved design of the previously developed [23] vacuum press mold was used with the function of Joule termostatting (Figure 3).

The improved design of thermovacuum mold features the ability for smoother, more uniform electrothermal heating and thermostatic retention of softened mesomorphic phases in a high-temperature billet synthesized by SHS and ready for force compaction by vertical pressing.

A special thermal vacuum press-mold consists of a punch 1, a matrix 2, mutually parallel channels for vacuum suction of reaction gases 3, a vacuum chamber 4, a fitting 5 for connection to a vacuum pump, a steel pan 6, a thermodielectric gasket 7 for electrical insulation of the pan 6, contact current supply electrodes, dielectric spacers-fixers in the form of semi-ring segments 8–8' – 8''–8''' thermodielectric gasket 9 for insulating the matrix 2, contact sleds 10, side segment-end current collectors 11–11' attached to them, sandwiched between perforated flat graphite disks 12 with a pyrographite mesh, a powder billet (briquette) for SHS-compaction 13, a tungsten spiral for initiating a thermal shock (one-time stimulating thermal pulse) 14, electrical and thermal insulating quartz sand 15, a dielectric spacers-retainers 16 located between segment electrodes 8–8''', a sealed flexible polyethylene plate 17, a rectified pulse power supply system 18, with the ability to reverse the polarity "RP" of current-carrying segments 8'–8'', and a computer unit for operational control and regulation of the Joule thermostatting process.

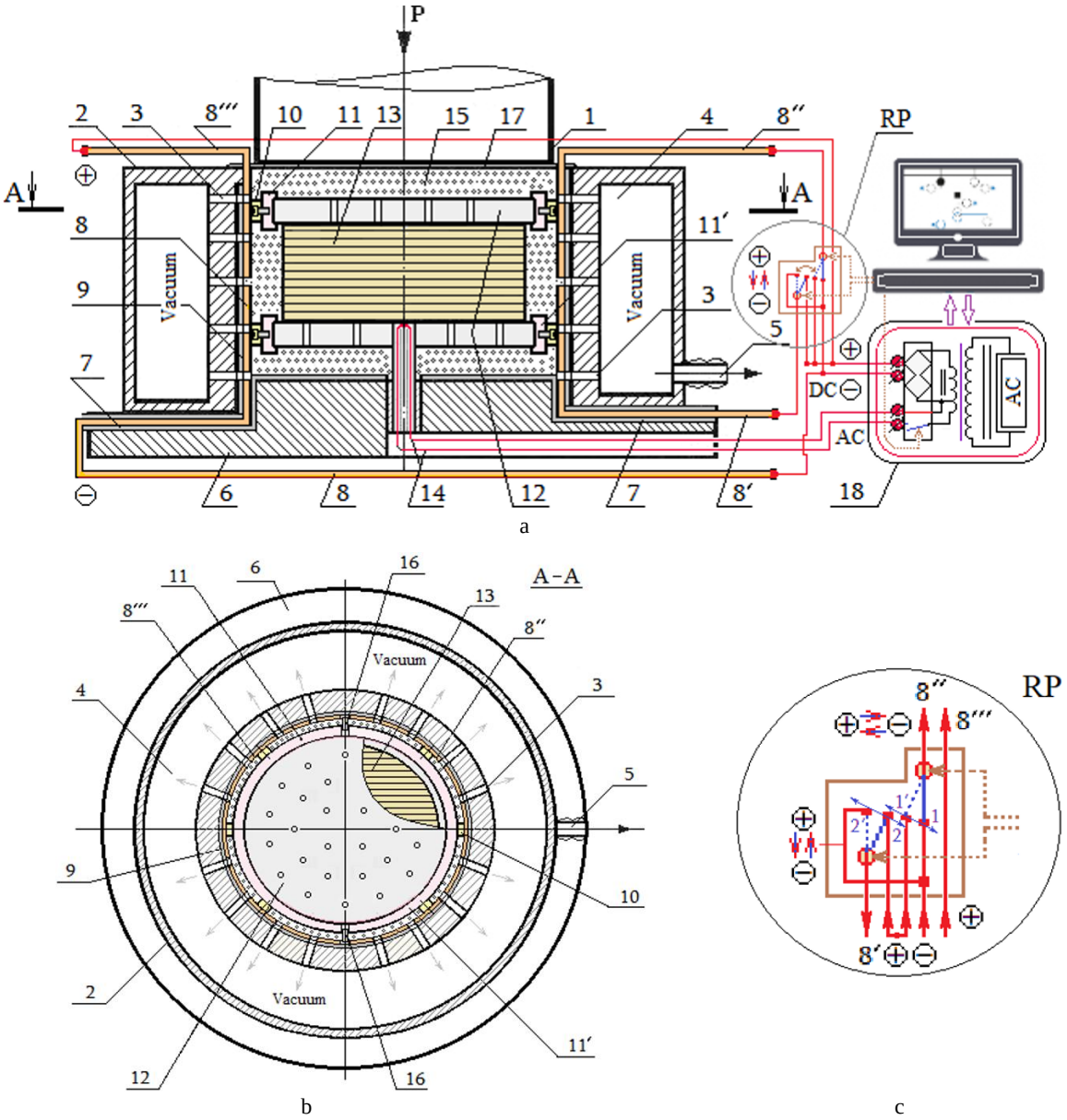


Figure 3 – Structural diagram of a special thermal vacuum press mold to increase the efficiency of SHS-compaction of ceramic-matrix composites: a – cross-section; b – section A–A; c – polarity reversal unit RP

The design of the press mold is solved so that the current is supplied to the synthesis zone before initiating the SHS process. Consequently, the appearance of predominantly conductive phase TiC is passed exclusively through the graphite disc planes 12, gradually heating them. After the initiation of the SHS process from pallet 6 and the formation of the noted conductive phase TiC, the need for electrical heating of the graphite discs is eliminated, and the advantage belongs to the passage of current through the synthesized billet 13. In order to obtain the maximum effect of joule thermal influence, which requires the helpful use of the Thomson effect (equation (7)), the electrical circuitry of the special press mold provides the possibility of reversing the polarity switching of the current-conducting segments 8'–8''. Polarity switching should be done immediately after the complete passage of the reaction combustion wave and the end of the synthesis process, simultaneously with the start of the forced compaction process using vertical force movement of punch 1 from top to bottom.

Initial heating of graphite discs 12 solves the problem of minimizing thermal losses from the surfaces of workpiece 13 on punch 1 and matrix 6 or the intermediate quartz layer 15 between them. In turn, by regulating the power of the supplied electrical energy and the duration of holding under pressure, the thermophysical state of the synthesized TiB_2-TiC compounds is controlled, achieving the maximum possible preservation of the conditions necessary for their complete compaction, structural homogenization, and thermostabilizing.

Standard methods and measuring instruments were used to evaluate the accuracy and efficiency of the experimental data. In particular, to measure the vacuum power of the vacuum chamber 4 of the press-mold used

(Figure 4), a Leybold THERMOVAC TTR 91 vacuum gauge was used (measurement range 0.5–1.0 μbar), connected to fitting 5 perpendicularly (in the diagram of Figure 4 not shown).

A hydraulic press model D-1932 with a force of $1 \cdot 10^6$ N, adapted to SHS processes, with original mechanical equipment and digital pressure indication output, was used for vertical pressing of the synthesized samples.

The dynamics of changes in the temperature field along the SHS workpiece's lateral perimeter were measured using an original optical pyrometry complex through a sealed transparent quartz window.

Structural and morphological studies of the resulting products were carried out using a field emission scanning electron microscope JSM-7800F. The Carl Zeiss Neophot 32 optical microscope was used for metallographic studies.

Standard test methods were also used to investigate the physical and mechanical properties of the obtained products. In particular, the bending strength was determined according to ISO 7438:2020 "Metallic materials. Bend test". Vickers hardness was determined according to ISO 6507-1:2023 "Metallic materials. Vickers hardness test". Palmqvist method was used to evaluate the fracture toughness according to ISO 28079:2009 (Hardmetals, Palmqvist toughness test). Microporosity was evaluated according to ISO 4499-4:2016 "Hardmetals. Metallographic determination of microstructure. Part 4: Characterization of porosity, carbon defects and eta-phase content".

4 Results

As a result of the studies, it was found that for the analytical description of the SHS-compaction process with simultaneous Joule thermostatting of the synthesized sample, graphic expressions presented in the form of curves illustrated in Figure 4 can be used.

From the characteristic graphical dependences shown in Figure 4, it is clear that the pressing pressure *P* used in SHS-compaction, which, together with the applied current *I* and the electrical resistance of the product *R* (depending on its thickness), is the main factor affecting the change in volume *V*, should be controllable and not maximum possible (shock-impulsive), as it was achieved by researchers [18, 24].

In this case, the main control criterion should be the index of the degree of compaction ρ and the amount of heat *Q* released by the sample, which is very important from the point of view of solving the target problem of structural homogenization of the synthesized sample. In practice, as an indicator of the degree of compaction of the synthesized sample, the shrinkage of the mold punch for SHS-compaction can be used, and the change in the amount of heat generated can be judged by the temperature of the gases escaping in the vacuum chamber of the same press-mold.

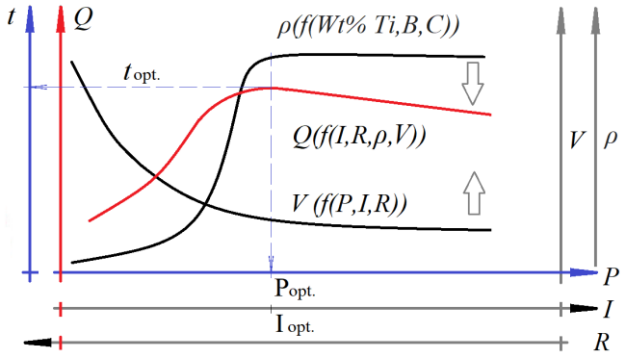


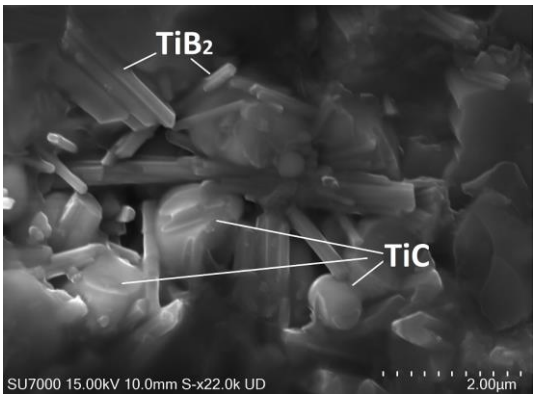
Figure 4 – The characteristic curves of the interdependence of technological parameters of Joule thermostatting during SHS-compacting of a sample from a ceramic-matrix composite of TiB_2-TiC system

The importance of the above-proposed approach is also reasonably emphasized in the study [27], which, using the example of single-phase ceramics sintered from TiB_2 powder synthesized in advance, shows the significance of the degree of force pressing and its effect on the structure formation process. The study shows that if increased pressing forces are applied (150 MPa ≤ 1.5 GPa), the sample compaction mechanism transforms into plastic deformation of sintered grains, leading to grain-boundary diffusion. Dislocation cells are also formed since these grains are excessively ground. Substructural zones with increased dislocation concentrations are formed. With this result, the authors prove the possibility of controlling the microstructure of single-phase TiB_2 ceramics by its partial substructural transformation (grinding) at the high-temperature (1900 °C) sintering and force compacting stage. The task that is posed in this study has the opposite goal. It consists of providing the effect of regulating the microstructure of a two-phase ceramic-matrix billet obtained by the SHS-compacting method due to its homogenization and grain enlargement, which should be carried out by moderate boundary diffusion and fusing of softened TiC grains with TiB_2 grains.

In order to avoid overloads of pressing pressure and excessive duration of exposure to the direct current supplied to the sample, a conventional vertical line is highlighted in the diagram of characteristic dependencies shown in Figure 4, indicating the optimal (maximum permissible) values of the pressing pressure and Joule thermostatting of the synthesized workpiece. In turn, the horizontal line shows the value of the time sufficient to achieve such an effect (t_{opt}). Further holding of the workpiece, increasing the pressing force or the strength of the electric current passing through the workpiece will lead to a deterioration in the efficiency of the temperature control process.

It should be noted that since the optimum values of pressing pressure, electric current strength, or holding time of the thermomechanical action depend on the geometrical dimensions and composition of the synthesized composite, these parameters should be determined previously, in each case by experimental means.

Analysis of the experimental results obtained, the central part of which is given below, showed that the process of SHS-compaction of a ceramic-matrix composite of the TiB_2-TiC system with Joule thermostatting, in addition to compensation of the existing temperature gradient, structural homogenization and uniform compaction of the workpieces, really ensures the enlargement (consolidation) of the grains formed during synthesis, mainly due to infiltration-extrusion impregnation and fusion (coalescence) of the carbide phase TiC, relatively softened from the prevailing Joule heating, with a harder boride TiB_2. To prove this, Figure 5 compares the morphology of workpieces obtained by SHS compaction in a developed special vacuum press mold with a vacuum degree of 0.9 bar before and after Joule thermostatting.



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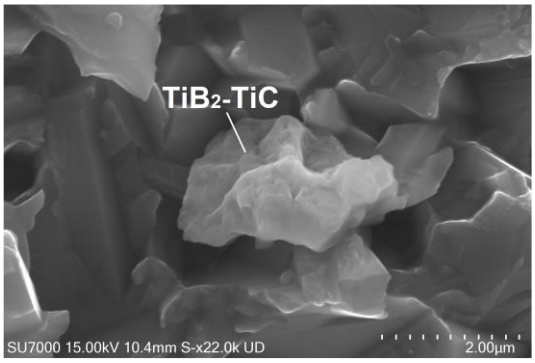


b

Figure 5 – Morphology of the ceramic-matrix composite $TiB_2(TiC)_{0.5}$ before and after Joule thermostatting: grain coalescence and rigid, relatively equiaxed workpiece formation (technological parameters of Joule heating: current strength – 570 A (0.8 A/mm^2); voltage – 3.2 V; heating duration – 2 min; pressing pressure – 150 MPa)

From Figure 5, it is clear that the morphology of the ceramic-matrix composite obtained as a result of Joule thermostatting with the TiB_2:TiC phase ratio 2:1, which means that stoichiometrical $TiB_2(TiC)_{0.5}$, in contrast to the usual one obtained without thermostatting, consists of shapeless fused conglomerates of enlarged dimensions, formed as a result of the intergrowth of globular TiC crystals with needle-shaped TiB_2 crystals, forming a rigid skeleton of a predominantly equilibrium position. This is mainly due to the increased electrical conductivity of the TiC phase and, consequently, to the advanced accumulation of heat Q^{Σ} (equation (9)) generated by the superposing Joule–Lenz and Thomson effects.

From the study [28], the resistivity of TiC, depending on the size of the formed crystals, can vary in the range of 60-86 μΩcm. In turn, the resistivity of the TiB_2 compound, depending on the content of boron in it ($1.2 \leq B \leq 2.8$), can vary in the range of 133–413 μOhm·cm [29]. That is, the electrical resistance of the latter is several times higher than that of the former. These data confirm our statement about the preferential Joule heating of the TiC phase. At the same time, according to the study [30], the heat capacity of TiC in its carbon concentration decreases from 3 to 30 % of stoichiometric carbon concentration (19 %) and increases from 29.4 to 34.2 J/(mol·K). The heat capacity also increases as its temperature increases from 41 J/(mol·K) at 400 °C to 52 J/(mol·K) at 1200 °C [31]. The heat capacity of the TiB_2 boride phase is also variable, which varies from 217 J/(mol·K) at 400 °C to 275 J/(mol·K) and above at 1200 °C [32].

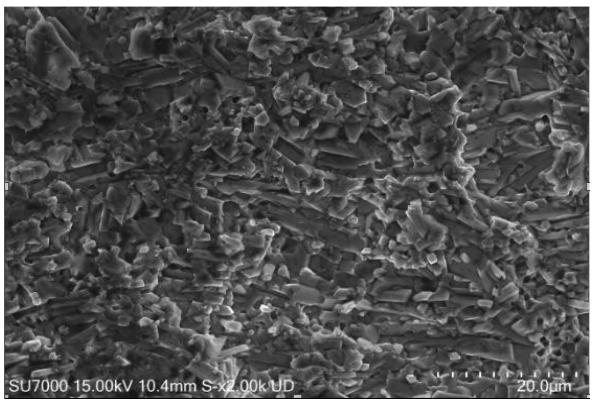
The almost 5-fold higher resistivity and heat capacity of TiB_2 compared to TiC makes it considerably inertial in terms of external energy stimulation and heating. Therefore, the main work, which is used by passing electric current through the lower and upper surfaces of the synthesized billet, is mainly accumulated and converted into thermal energy precisely due to heating and softening of the carbide phase of TiC.

The main characteristics of the process are as follows: initial particle sizes of mechanically activated titanium powder not more than 25 μm; graphite and boron – amorphous, with particle sizes not more than 2 μm; the velocity of combustion front propagation 3–4 cm/s; applied current – 550–600 A, corresponding to the specific current passing through the workpiece – 0.85–0.9 A/mm^2; voltage – 3.5–3.0 V, duration – 1.5 min at temperature 2230–2160 °C and pressure 150 MPa.

The results of investigating the morphology of the structure of the ceramic-matrix composite $TiB_2(TiC)_{0.5}$ obtained after Joule temperature control by scanning electron microscopy, as well as by energy dispersive X-ray spectroscopy and elemental mapping, are illustrated in Figure 6.

Analysis of the research results shown in Figure 6 shows that the morphology of the $TiB_2(TiC)_{0.5}$ composite obtained as a result of SHS-compaction and Joule-vacuum thermostatting is predominantly homogeneous (Figure 6a), crystals and elements containing in them (Ti, B, and C) are distributed relatively evenly (Figure 6b).

The presence of a small amount of oxygen and its scatter indicates an insufficient depth of evacuation and an insufficient degree of desorption of gases introduced into the reaction mixture from amorphous boron or carbon. However, it is inevitable and within the bounds of what is acceptable.



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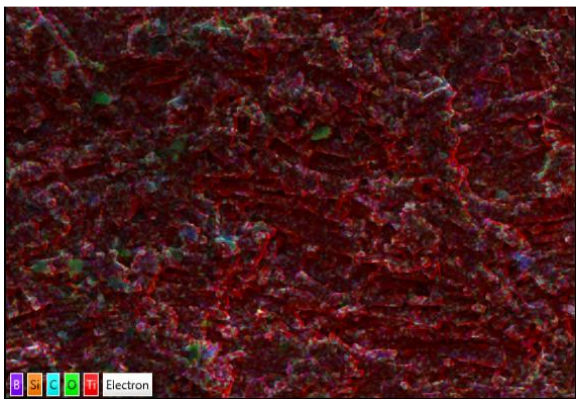

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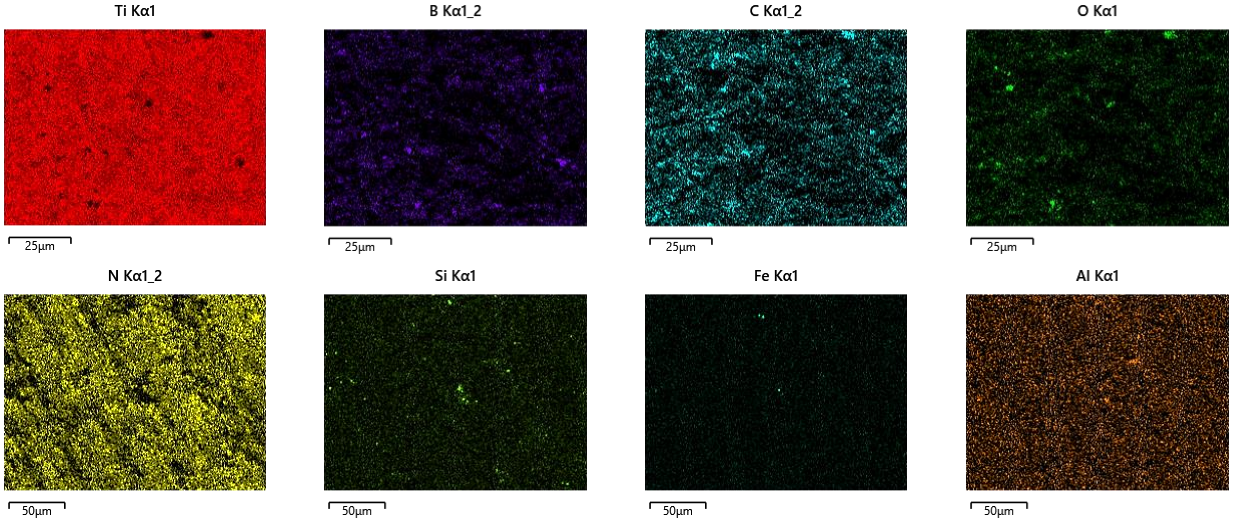

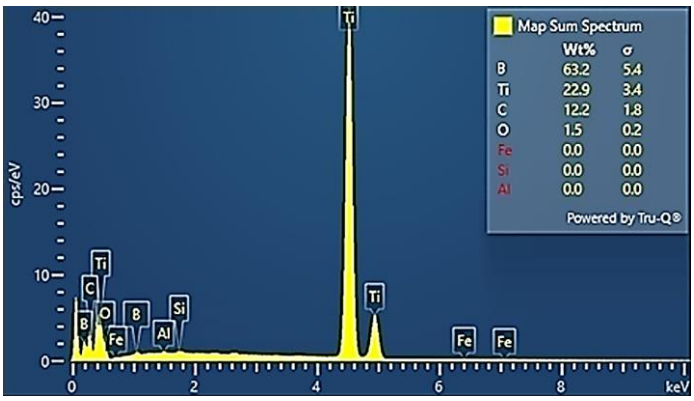

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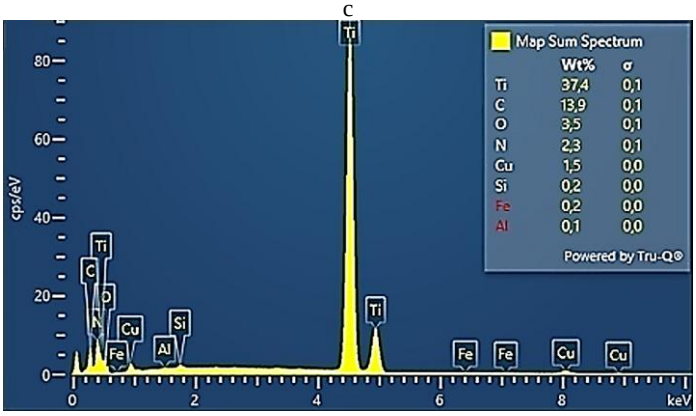


d

Figure 6 – SEM image, EDS X-ray maps, elemental spectrum, and quantification results of the $TiB_2(TiC)_{0.5}$ composite obtained during SHS-compaction with Joule thermostatting

Technological parameters of Joule heating are as follows: current strength – 600 A (0.85 A/mm^2); voltage – 3.0 V; heating duration – 1.5 min, pressure – 150 MPa

According to this elementary map (EDS maps in Figure 6, Ti, B, C, O, N, Si, Fe, Al), the main reason for the appearance of a small amount of oxide inclusions in the marked area of the resulting composite may be atomic oxygen absorbed in graphite particles. This is evidenced by a comparative analysis of images mapping the elements Ti, C, and O. The possibility of oxidized particles of titanium powder getting into the workpiece is immediately excluded since, on the distribution map of this element, areas of oxygen dislocations are undarkened, the radiation intensity is minimal (i.e., there is no oxygen) and vice versa, the oxygen mapping coordinates, expressed as light points on the distribution map (O), coincide as much as possible with the carbon location coordinates (C). This indicates that in the process of high-temperature self-propagating synthesis, a small part of the carbon when combined with titanium, managed to capture a small amount of oxygen absorbed from the air, which could have occurred at the stage of preliminary preparation (forming briquetting) of the workpiece before its placement in a special vacuum press-form. This did not have a noticeable impact on the quality of the synthesized ceramic-matrix composite since it did not cause any undesirable structural or morphological changes.

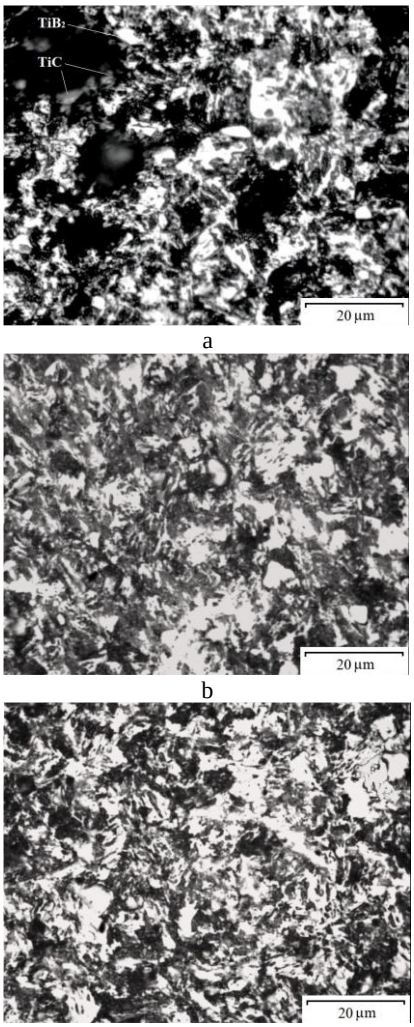
The presence of elements such as silicon, copper, nitrogen, iron, and aluminum are also observed in the obtained sample, which results from quartz thermal insulation sand, copper current-carrying segment-shaped semi-annular electrodes, and corresponding auxiliary accessories in the press-mold used.

The quantitative characteristics of the distribution of elements in the conglomerate of coalescenced TiB_2 and TiC crystal grains displayed on the EDS spectrograms illustrated in Figures 6c–d show that the total chemical formula of the resulting composite, according to the weight content of the leading components, can be written as follows – $Ti_{1.5}B_2C_{0.5}$, which corresponds to targeted stoichiometry $TiB_2(TiC)_{0.5}$.

The comparative analysis of the physical and mechanical properties of the obtained ceramic-matrix composite of TiB_2-TiC of disc-shaped form with dimensions D30×10 mm, before and after Joule-vacuum heat treatment showed a significant advantage of the latter. In particular, the volume of open porosity of the heat-treated composite was less than 0.5 %, against 3–4 % of the conventional one, the Vickers hardness HV at room temperature was in the range of 27 GPa, against the hardness of 23 GPa in the conventional (not thermostatted sample), the bending strength was 480 MPa, instead of 380–400 MPa in the conventional one, and the fracture toughness was 5.7 MPa·m$^{1/2}$, against 3.5 MPa·m$^{1/2}$ in the conventional one.

Multiple repetitions of the experiments at different energy parameters of joule temperature control showed the significance of the influence of the duration of this process on the size and shapes of the formed crystalline conglomerates. In particular, it was found that the longer the Joule heating process and the pressing duration of the synthesized products lasts, the degree of enlargement of crystalline conglomerates of TiB_2-TiC solid solution increases (Figure 5b), but after reaching the equilibrium point, further growth of conglomerates stops. The process passes into the mode of volumetric plastic deformation, similar to the characteristic curve shown in Figure 4. Such a point of conditional equilibrium at static vertical compression of the sample D30×10 mm with a pressure of 150 MPa was a thermostatting temperature of 2450 K (2280 °C) with a holding time of 1.5 min.

Further holding (thermostatting) of the workpiece resulted in significant plastic deformations and increased intergrainal (intercrystalline) diffusion. Deformation traces of compaction and the effect of intergrainal diffusion coalescence of TiC compound with TiB_2 are clearly visible in the metallographic image of microstructures presented in Figure 7. This is also shown in Figure 8 – a diagram showing the difference between the effects obtained from the Joule thermostatting times of 1.5 and 2.5 min at different pressing speeds of 1.00 and 1.66 MPa/s, respectively.



a

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Figure 7 – Evolution of microstructure of the obtained $Ti_{1.5}B2C_{0.5}$ ceramic-matrix composite: a – without Joule termostatting; b, c – after Joule exposure with a duration of 1.5 and 2.5 min, respectively

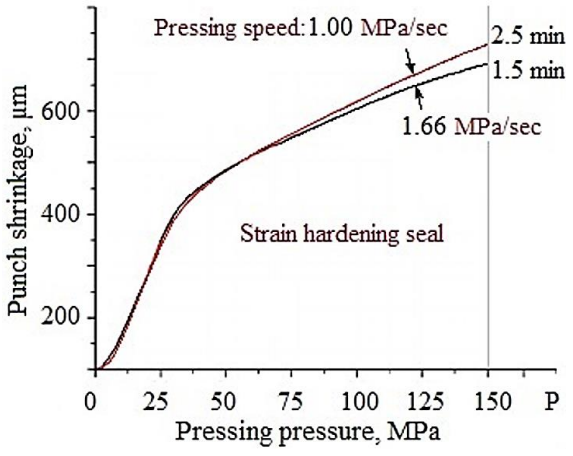

Figure 8 – Degree of compaction of the microstructure depending on pressing speed and duration of thermostatting

The proposed approach minimized porosity in the structure stabilized the morphology, and eliminated the problem of uneven compaction and violation of the geometric accuracy of the resulting composite workpiece.

Figure 7 shows that the use of pressing pressure for compacting hot products of synthesis of TiB_2-TiC composite in the 150 MPa range with a duration of Joule thermostatting during 2.5 min led to a more intense decrease of the number and size of existing pores and compaction of the microstructure, and a deeper diffusion coalescence of softened carbide grains TiC, featuring a characteristic grey color with relatively light crystals of TiB_2. This is also confirmed by the diagram in Figure 8, which shows the amount of shrinkage of the punch of the special press mold and, consequently, the degree of strain-hardening compaction of the synthesized composite as a function of the duration of the Joule thermostatting. Here it can be seen that the degree of vertical shrinkage of the press-mold punch at an increased duration of Joule thermostatting of the SHS-workpiece (2.5 min) and reduced speed of its pressing (1 MPa/s) is 6–7 % higher than that of the workpiece with increased pressing speed (1.66 MPa/s) and shorter duration of thermostatting (1.5 min).

From the comparative analysis of the microstructures shown in Figures 7, 8 it follows that at energy-stimulated thermophysical deformation-strengthening compaction and diffusion coalescence of mainly softened TiC grains with TiB_2 crystals, their coarseness on average decreased by 30–35 %, from about 7–8 to about 4–5 μm, which was achieved due to their segregationally displacement to the zones of reduced pressure, i.e., to the pores located near TiB_2 crystals.

The effect obtained from the micro-extruded filling of intergranular pores with TiC-softened mesomorphic crystals led to the formation of newly enlarged conglomerate TiB_2-TiC grains with zones of cementation hardening of the matrix skeleton.

It is noteworthy that experimental studies of the peculiarities of obtaining composites with different ratios of synthesized components TiB_2 and TiC unambiguously confirmed the special significance of the influence of the concentration (distribution density) of TiC grains at the initial stage of pressing and Joule thermostatting of the target workpiece. In particular, precisely the concentration of TiC grains, and hence – the number of contacts formed directly between them, taking on the function of carriers of electric charge and determining the density of the transmitted charge through the contact surfaces of the workpiece (Figure 1, equation (1)), are among the essential factors in selecting the necessary electrical power Joule heating, sufficient to create the desired thermal effect. The above-mentioned is entirely consistent with the Joule-Lenz law written down by us in differential form (equation (6)), once again proving our proposed approach's effectiveness and emphasizing its advantage in high controllability.

The analysis of the obtained experimental data showed that the dependence of the electric power applied for Joule's impact on the SHS workpiece on the content of the TiC carbide phase has an inversely proportional character, with a correlation coefficient of 0.85. Consequently, this means that the power of Joule electrothermal influence should be decreased when expecting the formation of increased amounts of TiC compound and, conversely, increased at reduced concentrations of the noted phase. Planning or forecasting of the amount of TiC carbide phase formation can be carried out by preliminary selection/calculation of the stoichiometric ratio of reacting elements (Ti and C) and estimation of the expected thermodynamic effect created as a result of self-propagating reaction combustion volition between them.

It is also evident that the content of TiC should be regulated based on the final purpose of the synthesized ceramic-matrix composite and its operating conditions.

Moreover, in manufacturing critical parts intended for thermocyclic alternating loads used for producing radiation-resistant protective shields with reduced magnetic permeability and high dielectric properties, minimizing the presence of the TiC component is necessary.

It has been found that the allowable TiC concentration range for the proposed Joule homogenization thermostatting approach, depending on the dispersion of the titanium powder used and the expected grain sizes resulting from its combination with carbon, is in the range of 35–25 %. In particular, - the carbide phase TiC concentration should be 35–30 % at the relatively raised size of titanium powder particles – 50–25 μm. When using titanium powder with a dispersity of 25–20 μm, due to the increase of its specific surface and the total number of particles mutually reacting with the smallest graphite particles (1 μm), preliminary stoichiometric calculation of initial components for the synthesis of TiB_2-TiC composite can be carried out to obtain a composite with the possibility of obtaining in its TiC compound within 30–25 %. Using a reaction charge designed to obtain a lower concentration of TiC, less than 25 %, does not give the desired result because there is almost complete blockage of conductive TiC grains by TiB_2 crystals. The system analysis of the results of our experimental research work has shown that the chosen proportion of ceramic-matrix composite TiB_2-TiC with the ratio of components 2:1

(corresponds to the concentration of TiC equal to 33.3 %) is optimal for the technological process of SHS-compacting in vacuum with homogenizing Joule impact on the synthesized and densified workpiece.

Experimental work has shown that the complexity, and in many cases the lack of technical capabilities of high-precision operational control of the temperature gradient formed in the volume of the synthesized workpiece ΔT_{2-1}, assessment of the degree of its electrothermal Joule compensation, the achieved level of microstructural homogenization, and therefore, determination of the moment of removal of the applied thermophysical effect, causes necessary the development of a special functional algorithm for diagnosing and controlling the homogenization thermostatting efficiency.

In order to flexibly solve the problems that have arisen in the process control, on the one hand, and the general improvement of the SHS-compaction technology, on the other, based on the theoretical and experimental justifications given in chapters 4 and 6 for the features of electro contact Joule thermostatting of the synthesized workpiece with a gradual increase in the pressing pressure, have developed a generalized algorithm for controlling the SHS-compaction process (Figure 9).

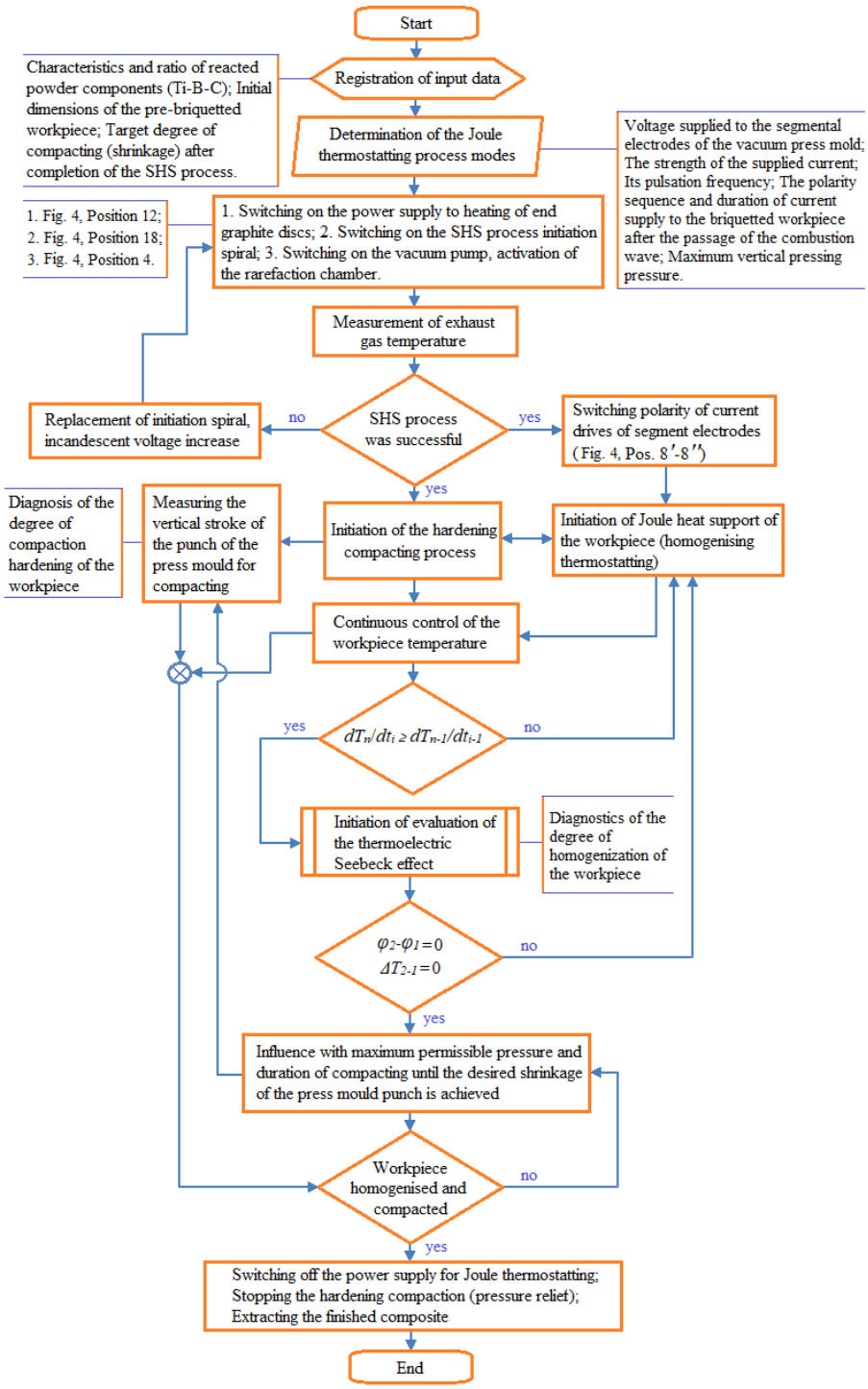

Figure 9 – A flowchart of the algorithm for the controlled synthesis of a TiB2-TiC-based ceramic-matrix composite

One of the primary reference signals for diagnosing the efficiency of the process is provided the use of such a relatively easy-to-measure and proportional to the temperature gradient ΔT_{2-1} as the value of the thermo-electromotive force, called the thermoelectric Seebeck effect (thermo-EMF) [33], excited between the upper, relatively hot and the lower, relatively cooled surface of the workpiece, by contact control of the difference of their potentials $\varphi_2 - \varphi_1$, according to Figures 1, 4.

The laboratory measurements have shown that the value of the arising thermal EMF in the obtained ceramic-matrix composite $Ti_{1.5}B_2C_{0.5}$, depending on the value of the temperature gradient ΔT_{2-1}, is 0.025 mV/(100 °C) on average.

5 Discussion

According to our proposed approach to algorithmizing, graphically presented in the form of a structural and functional block diagram (Figure 8), the billet, thermostatted by Joule heating in a special thermovacuum press-mold (Figure 4), can be considered homogenized if the temperature gradient formed in it is fully compensated by Joule heating and is equal to zero (i.e., the condition $\Delta T_{2\text{-}1} = 0$). In this case, of course, inside the workpiece will completely disappear the Thomson heat effect dQ_T (equation (7)) and, accordingly, the amount of total released heat Q_Σ, controlled through the temperature of exhaust gases, will stop growing (Figure 3, curve $Q_f(I, R, \rho, V)$), begins to decrease (equation (8)). The marked part in the block diagram of the presented algorithm is highlighted by the comparison block, designated by the condition: $dT_n / dt_i \geq dT_{n-1} / dt_{i-1}$. Theoretically, the moment of such a slowdown of temperature growth and the transition from constant growth to a temporary decline (fluctuation transition) can be considered as the point of homogenization of the thermostatted SHS workpiece. This means that the temperature field and, consequently, the distribution of compressive stresses inside the compacted billet are equalized. Consequently, the Joule thermophysical and force vertical compressive action on the workpiece can be canceled.

However, since temperature measurements in practice are inertial and can be determined indirectly, without direct contact with the workpiece, direct control of the potential difference between the upper and lower surfaces of the thermostatically controlled workpiece ($\varphi_2 - \varphi_1$) is used as the primary diagnostic function, for which a special highly sensitive digital microvoltmeter is used, capable of recording the thermoelectric Seebeck effect in the range 0–1 mV. The signal of complete compensation of the Seebeck effect, i.e., zeroing of the potential difference between the surfaces of the thermostatted billet ($\varphi_2 - \varphi_1 = 0$) through a particular controller connected to the microvoltmeter is fed to the computer, where the pre-recorded control algorithm accepts this fact as the moment of achieving the maximum effect of joule thermal stabilization and the desired degree of homogenization.

As a result, the pressing pressure is brought to the maximum value of the planned threshold. After the desired degree of sealing contraction of the workpiece (determined by the shrinkage of the press-mold punch) has been reached, the program gives a feedback signal to the controller to switch off the DC power supply for Joule heating, provided that the pressing pressure is maintained. The pressure is released after the workpiece has cooled down to 50–60 % below the solidus temperature of the TiC phase, to 820–980 °C.

Implementation of the proposed method of diagnostics and control of the process of Joule thermostatting can be achieved through the same semi-ring current-supplying electrodes 8'–8" (Figure 4), using which a direct electric current is supplied to the upper and lower surface of the synthesized workpiece, by connecting the above-mentioned microvoltmeter to them in parallel. The measurement process should be discrete and carried out with short interruptions of current supply on the applied half-ring electrodes 8'–8".

Based on the specifics of the developed multi-level algorithm, for a highly efficient practical implementation of the proposed approach of homogenizing Joule thermostatting and thereby improving the technological process of SHS compaction of ceramic-matrix composites of the TiB_2-TiC system, it is necessary to achieve a high degree of automated control and management of the operations provided for by the proposed production process, which can be achieved implemented using an adaptive neuro-fuzzy inference system (ANFIS), after deep learning of the latter. This task is the subject of separate research and will be implemented in the future.

6 Conclusions

Based on the system analysis of the given theoretical substantiations and the results of the experimental research, it is possible to draw the following conclusion.

First, an approach to homogenizing Joule thermostatting was proposed for a ceramic-matrix composite TiB_2-TiC during its synthesis by SHS compaction in a vacuum. It is achieved by passing a direct current through the body of the synthesized workpiece, directed in line with the propagation vector of the combustion wave (in our case - from bottom to top), ensuring the beneficial use of the Thomson and Seebeck effects, which appeared after the completion of the synthesis. Subsequently, the formation of an intrabody temperature gradient is effective. It can be used to improve the technological process for producing ceramic-matrix composites based on TiB2-TiC, with a component ratio of 2:1, ensuring a significant improvement in operational physical and mechanical properties and reducing geometric errors target workpieces.

Second, a prerequisite for implementing the proposed process is using a special electrothermal vacuum press mold developed for this purpose and an algorithm for controlling the technological modes of homogenizing Joule influence.

Third, the possibility of compensating thermal losses due to heat released mainly by electrically conductive TiC crystals leads to homogenization of the temperature field and provides compensation of the temperature gradient inside the synthesized workpiece, which creates convenient thermodynamic conditions for excluding the formation of internal (structural) thermal stresses. Moreover, the formation of a relatively softened TiC phase in the structure of the workpiece, during pressing, promotes a more uniform consolidation of the synthesis products due to plasticity-diffusion redistribution of relatively pliable TiC grains around prism-shaped rigid TiB_2 crystals. Such consolidation leads to a significant improvement in the quality of the structural packing of the workpiece, where the number and volume of both open and closed micropores are reduced to 0.5 %, which improves the composite's performance properties by an average of 10–15 %.

Fourth, electric power applied for Joule heating should be selected depending on the planned content of the electrically conductive TiC phase in the processed composite workpiece. The dependence of the electrical power of Joule heating on the TiC concentration is inversely proportional. The correlation coefficient is 0.85.

Also, the rational interval of TiC concentration, depending on the dispersibility of the used titanium powder and, consequently, on the expected size of sintered grains obtained after synthesis with the range of 50–20 μm, is 35–25 % wt. Using the reaction charge designed to obtain a lower concentration of TiC ≤ 25 % does not give the desired result, as there is almost complete isolation of electrically conducting TiC grains by highly resistive TiB_2 crystals.

Moreover, the concentration of TiC equal to 33.3 %, which corresponds to the ratio of components TiB_2:TiC = 2:1, is optimal for the implementation of homogenizing Joule thermostabilizing of the technological process of SHS-compaction in a vacuum.

Finally, with the noted weight ratio of the TiB_2-TiC components, the optimal electrical modes of homogenizing Joule thermostatting of the obtainable $Ti_{1.5}B_2C_{0.5}$ composite are specific current passed through the workpiece – 0.8–0.9 A/mm^2; voltage – 3.5-3.0 V; holding time at maximum pressing pressure 150 MPa – 2.0–2.5 min.

Acknowledgment

This research work is dedicated to the glowing memory of the honored scientists of their time, Professors Alexander Shteinberg and Archil Kostava.

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