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# A COMPARATIVE STUDY OF METHODS FOR OBTAINING SILICON CARBIDE CERAMIC MATERIALS

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To date, about ten different methods for producing silicon carbide ceramic materials have been developed. This study presents the mechanisms and describes the advantages and disadvantages of the most commonly used methods for forming and sintering SiC powders. The article describes the basic properties and the application areas of resulting ceramic articles.

**Keywords**: silicon carbide, hot slip casting, reaction sintering, siliconization, hot isostatic pressing, spark plasma sintering

## INTRODUCTION

SiC-based materials have a wide range of properties, such as low density, good mechanical properties over a wide temperature range, and a low thermal coefficient of linear expansion (TCLE). Due to these properties, silicon carbide ceramics are used as highly rigid and wear-resistant materials that operate when exposed to aggressive media at high temperatures [1, 2].

To date, various methods for producing SiC-based materials have been developed. They are aimed at obtaining products with different properties to be used in many technical fields. However, due to the great complexity of sintering pure SiC, all methods employ either special additives that activate the sintering process or exposure to high temperatures and pressure. Thus, obtaining a high-density material from pure SiC with maximum physical and mechanical characteristics is virtually impossible [3].

This article discusses the most common methods for producing silicon carbide materials, their sintering mechanisms, benefits and drawbacks, as well as the properties of resulting products.

## POWDER FORMING METHODS

Technologies for producing silicon carbide materials consist of several stages: initial powder preparation, its molding into a compact with the addition, if necessary, of sintering additives and/or a temporary process binder, excess plasticizer removal (drying), and compact sintering. Depending on the production method, one or more stages can be excluded or combined into one. For example, during hot pressing, pressing itself and sintering are performed simultaneously.

Preparation of the initial powder may involve its additional activation by increasing dispersity (grinding), increasing surface roughness and the number of defects in the structure, removing moisture (during calcination) and oxide layers from the surface of particles [4]. The properties of the green body affect the sintering process parameters (mainly temperature and pressure), and accordingly, the resulting sintered material characteristics. In addition, it is at the stage of compact forming when the final shape of the product is often set precisely. We will consider two primary methods of forming green bodies from silicon carbide powders: cold pressing and hot slip casting.

### Cold pressing

Pressing was and still is the most common method of compacting powders. Cold static pressing in closed, mostly metal, press dies is a technologically simple and low-cost

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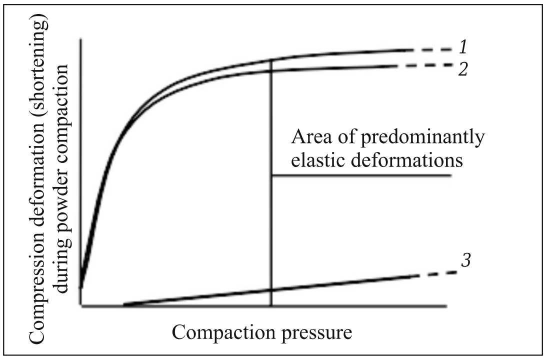

**Fig. 1.** Nature of the system deformation during powder mixture compaction: *1* ) general deformation during pressing; *2* ) residual deformation after pressure relief; *3* ) elastic deformation [8].

method for obtaining articles of various simple shapes. This method is versatile as it allows using powders of any composition as a starting material, which determines its wide application [5, 6].

The main types of pressing are uni- and bilateral static pressing in rigid metal dies, isostatic pressing, and hot pressing. The principle of the method is that the powder gets compacted under pressure due to the motion of particles relative to one another and their subsequent deformation.

The obtained green bodies are porous. To reduce the resulting compact porosity, it is necessary to increase powder compactability which depends on many factors. These are the initial powder or powder mixture dispersity, pressure and pressing direction, as well as the presence, quantity and nature of the liquid binder.

To obtain the most close-packed structure of green bodies, a two- or three-component powder mixture with different fractional compositions is used [6, 7]. A few finer fractions are introduced to fill the empty spaces between the powder particles of a coarse fraction during pressing, thus reducing the overall porosity of the compact and increasing its density. In addition to the grain sizes of green bodies, their morphological characteristics also influence the final compact density. Compacted powders with spherical particle shapes have a high bulk density, but they are difficult to compact. At the same time, irregularly shaped grains cause a low bulk density of powders, but they can increase the internal cohesive forces in the pressed semi-finished powder compact [6].

An important component of the starting powder is a temporary binder used as a binding agent between the solid powder particles. Its introduction significantly affects the compaction process and the final density of the semi-finished product. The presence of a film of water or another liquid between the powder particles reduces their mutual friction, thereby facilitating the powder compaction process. Moreover, the redistribution capability of the powder under pressure is enhanced, which leads to density equalization over the compact volume. A small amount of temporary binder in the powder may not provide the necessary elasticity of the compact and lead to the appearance of cracks. However, too much liquid binder increases the trapped air pressure in green bodies [6].

One should not overlook the phenomena affecting the liquid component of the system during the pressing process. Under pressure, the liquid contained in the layers between the particles is squeezed into the larger pores of the system. With a sufficiently large amount of binder, the liquid component of the powder can completely fill the total pore volume, which will lead to the ultimate compaction of the system. The pressure at which this phenomenon occurs is called critical. It is during the transition beyond the critical pressure that further compression of the system entirely reduces to reversible elastic deformation (Fig. 1). Thus, at a certain pressure, the reversible deformation of the system becomes predominant, and then practically the only one. When selecting the compacting pressure magnitude, one should avoid moving into this region [6, 8].

Thus, to obtain high-density green bodies, it is necessary to select the optimal fractional composition of the starting powders, temporary binder amount, and compaction pressure.

Cold static pressing in dies has several advantages. The main benefit is the feasibility of obtaining simply shaped compacts with high dimensional accuracy quickly. Compared to single- or biaxial pressing, isostatic pressing enables one to obtain powder compacts with a more uniform packing density. Thus, the method can be used to produce preforms of complex shapes. However, the powder compacts obtained by this method have less accurate dimensions and often require additional mechanical processing due to the presence of defects and surface inaccuracies [3].

There is also a method for fabricating complex geometry ceramic products. According to this technique, it is proposed to mechanically process prepared ceramic green bodies using a temporary organic binder before high-temperature sintering [9].

## Hot slip casting

Fabricating ceramic products using the slip casting method involves consolidation of particles suspended in slurries which are homogeneous suspensions consisting of a plasticizer and powdered material. SiC casts are produced by hot slip casting of thermoplastic slurries into predominantly metallic molds. These slurries are made on the basis of an organic binder which has a melting point of about 50 – 70°C and is capable of reversibly transitioning from a liquid to a solid state when cooled to room temperature. Paraffin wax and compositions based on it are most often used as a plasticizer.

To prepare a slurry, it is necessary to use well-dried powders, since the presence of even 0.1 – 0.2% moisture significantly worsens the wetting of the mineral particle surfaces with paraffin wax, which leads to a decrease in slurry fluidity [8].

Paraffin wax is utilized as a slurry dispersion medium; its melting point is 50 – 55°C. The properties of paraffin wax determine its selection as a dispersion medium. They are as follows: chemical inertness, a melting point convenient for practical purposes, a low viscosity in the molten state, and the presence of a softening range [8, 10].

Not pure paraffin wax, but paraffin-based compositions with other thermoplastic additives such as wax, stearin, ceresin are often used to prepare a slurry. This reason is that molten paraffin wax does not wet the mineral powder particles well enough, which makes it difficult to cover the entire particle surfaces with a limited amount of the liquid binder. Consequently, it is impossible to obtain highly concentrated slurries with good casting properties using pure paraffin wax without additives. To improve wetting of the particle surfaces, surfactants containing polar groups and nonpolar (hydrocarbon) radicals are introduced into the paraffin wax. Due to the presence of polar groups, oriented surfactant molecules adsorb on mineral particles, while outwardly directed nonpolar radicals interact with the paraffin wax. The most commonly used surfactant additives are beeswax, oleic and stearic acids. The introduction of a surfactant also makes it possible to significantly reduce the total volume fraction of the plasticizer [8, 10].

To prepare a slurry, all the necessary components are mixed in a tank heated above the optimum casting temperature, which is about 70°C. This is done to reduce the viscosity of the system. As a result, slurry homogenization accelerates significantly. With vigorous stirring, the slurry is vacuumized at a residual pressure of 10 – 15 mm Hg; the process is necessary to remove air bubbles from the slurry [11].

Hot slip casting process is performed in cold or continuously cooled metal molds under a pressure of 3 – 5 bar. The pressure is maintained throughout the entire time required for the solidification of all casting body parts. The holding duration varies depending on the size and shape of the cast and can reach several minutes. Maintaining pressure for some time is necessary to fill the voids that result from the internal shrinkage with new incoming slurry portions. Such defects arise due to slurry shrinkage by 3 – 5% in the product volume, which is caused by paraffin wax solidification. The outer cast layers harden fast because they are adjacent to the walls of the mold, which causes enhanced heat transfer. The external casting dimensions are quickly fixed, and the external linear shrinkage rate (related to the mold dimensions) is 0.2 – 0.3%, which is sufficient for the smooth removal of the cast. When the entire poured slurry mass is cooled, internal shrinkage predominates with the formation of shrinkage voids in the body of casting [8, 11].

The temperature of the poured slurry, which determines its viscosity, has a strong influence on the casting quality. At lower temperatures, the viscosity and solidification rate of the slurry increase, which can lead to incomplete filling (especially of thin-walled elements). Too high a slurry temperature leads to a decrease in the casting density and an increase in the likelihood of the formation of shrinkage voids. The optimal slurry temperature is 65 – 75°C. However, in each individual case, all the main parameters of the hot slip casting process (the slurry and mold temperature, pressure, holding time) are selected experimentally, considering the properties of a given slurry, as well as the size and geometry of produced articles. The casting mold design, in particular the sprue system location, also has a significant influence. It is important to provide simple solutions for mold separation and casting removal [8, 10 – 13].

The resulting molded articles contain a large plasticizer amount which, with the inevitable heating during the product firing, begins to soften again and change to the liquid state. To retain the final article integrity, it is necessary to remove part of the plasticizer from the molded preform volume before sintering. This leads to the appearance of another processing stage — drying. The drying process parameters crucially depend on the initial plasticizer concentration in the slurry, as well as the molded preform size and shape. Thus, these parameters are selected experimentally for each specific case.

The disadvantages of this method include the high labor intensity of the manufacturing process, in particular, the long and multistage process of slurry preparation, as well as the need for a long drying process of the cast article [5, 8, 10 – 13].

## Methods for sintering silicon carbide materials

The properties of SiC ceramics are mainly determined by the sintering technique. High-purity starting SiC powders show only limited sinterability due to the low self-diffusion of the particles. To activate the sintering process, sintering additives are introduced into the charge. They subsequently remain in the structure of the sintered ceramic article in the form of glassy or crystalline phases and have a significant impact on the final product properties [3].

According to the sintering technique, silicon carbide can be denoted as follows [14, 15]: ceramic-bonded (CSiC), recrystallized (RSiC), reaction-bonded (RBSiC), reaction-sintered or siliconized (SiSiC), solid-state sintered (SSiC), liquid-phase sintered (LPSSiC), hot-pressed (HPSiC), manufactured by hot isostatic pressing (HIPSiC) and by spark plasma sintering (SPSSiC).

## Ceramic-bonded silicon carbide (CSiC)

Silicon carbide ceramics, obtained with a ceramic binder, are SiC grains bound together by an aluminosilicate, silica, silicon nitride or silicon oxynitride phase. When this sintering technique is employed, physical and chemical processes only affect the binders. As a result, no changes occur in the primary silicon carbide phase. Thus, the properties of such materials are determined mainly by the properties of the ceramic binders [2].

The initial components during sintering are mainly coarse fractions of SiC powder, pure oxides ($SiO_2$, $Al_2O_3$)

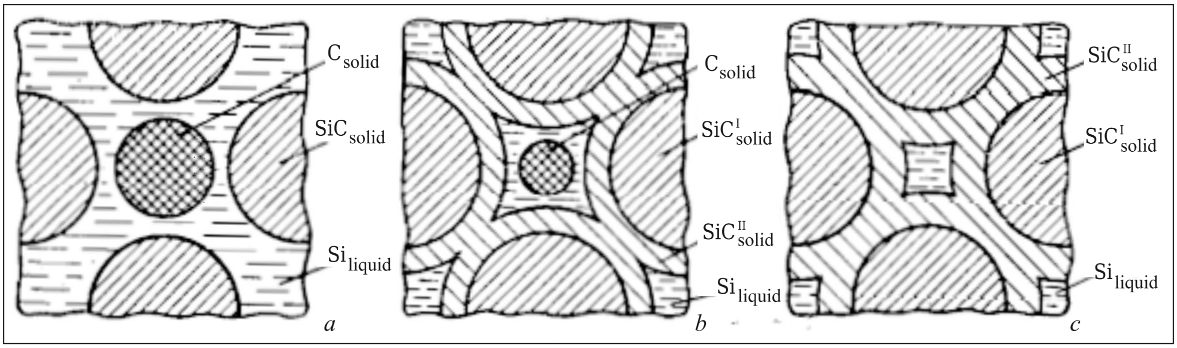

**Fig. 2.** Diagram of phase distribution at individual stages of the reaction sintering process.

and/or clay [16]. Pre-pressed compacts are fired at 1400°C in nitrogen. The resulting materials are highly porous (apparent porosity can reach 20 vol.%), low-density (2.55 g/cm$^3$) and are characterized by poor mechanical properties [17].

It is worth singling out the material obtained by sintering SiC in air (in an oxidizing atmosphere). In this case, the ceramic binder is formed by partial oxidation of primary SiC grains. This material is characterized by low thermal conductivity and high electrical resistance [2]. Silicon carbide materials with a ceramic binder are mainly used as refractory products for furnace linings, as well as abrasive discs.

### Recrystallized silicon carbide (RSiC)

Recrystallized SiC is obtained by free sintering of coarse SiC particles at 2100 – 2500°C in vacuum [18]. The sintering process proceeds according to the SiC evaporation-condensation mechanism [16, 19]. A two-component system is often used as the initial mixture of powders [6, 18].

Since no activating additives are introduced during sintering, the resulting material has high purity. Consequently, the SiC specific properties such as high chemical durability and high temperature creep resistance at a relatively low density (about 2.5 g/cm$^3$) are most fully realized in such products. The porosity of recrystallized SiC is up to 20 vol.%. It is impossible to obtain a material with a density close to theoretical using this synthesis method. Some reduction in porosity can be achieved through an additional compaction process by repeated impregnation of green bodies with polycarbosilane and polyvinylsilane solutions with subsequent carbonization and formation of secondary SiC in the pores of the material [13].

The advantage of this method is the possibility of manufacturing articles with a high degree of dimensional accuracy. As a result, such products do not require additional machining since this sintering method there does not involve shrinkage.

Silicon carbide materials obtained by this method are often used as high temperature resistant kiln furniture such as crucibles, plates, saggers, and others for furnaces whose operating temperature can reach 1600°C [20]. In addition, recrystallized SiC is widely used to produce heating elements for electric resistance furnaces with operating temperatures up to 1400°C [17].

### Reaction-bonded silicon carbide (RBSiC)

When shaping preforms from SiC powders, a certain amount of carbon component is additionally introduced in the form of soot or graphite.

Preforms may also contain residual carbon resulting from burning out the organic binder if it was used in powder molding. The obtained green bodies are sprinkled with high-purity silicon lumps and sintered in a vacuum at 1500 – 1650°C. When reaching 1400°C, silicon begins to melt and change to the liquid state. Porous powder compacts are impregnated with liquid silicon. After that silicon reacts with the carbon previously introduced into the compacts forming secondary $SiC^{II}$ in the intergranular space of primary $SiC^{I}$ particles. Thus, secondary SiC fills up the free pore space and binds the primary SiC particles, thereby forming a continuous polycrystalline SiC framework [21 – 25].

The most common mixture ratio for this sintering method is 90 vol.% SiC and 10 vol.% Si. The porosity of the resulting materials is about 20 – 30 vol.% [22]. The advantage of this method is almost no shrinkage, which makes it possible to obtain products of complex geometries.

### Reaction sintered (siliconized) silicon carbide (SiSiC)

This method is not fundamentally different from the previous one. The main dissimilarity is that the porous powder compact is infiltrated with excess liquid silicon to completely fill all pores with silicon melt [2, 26 – 33]. Figure 2 shows the mechanism of the reaction sintering process of silicon carbide.

Initially, this method was used to produce materials by infiltrating carbon-containing materials such as soot, charcoal, and graphite with silicon melt or silicon vapor. Formaldehyde resins, dextrin, and pulverbakelite acted as binders. Sintering was carried out in an argon atmosphere at 2050°C or in a vacuum when heated to 1600°C [34].

Reaction-sintered silicon carbide contains a silicon phase, which significantly affects its high-temperature characteristics [7]. The density of the obtained silicon carbide ceramics is 85 – 95% of the theoretical [35]. The determining factors that make it possible to produce a material with a density close to the theoretical one are the particle size of the starting SiC powder, the correctly selected disperse composition, and the optimum pressure of molding powder compacts. The mechanical and thermophysical properties of silicon carbide materials significantly depend on the density of the sintered articles. The higher the density of ceramics, the better mechanical properties they possess [7].

Using the reaction sintering method, one can obtain various complex-shaped and large-sized non-shrinkable silicon carbide products. This method is also distinguished by its high productivity. Currently in Russia, more than 70% of all silicon carbide materials are produced using siliconization [33].

Siliconized silicon carbide has excellent physical and mechanical properties. Thus, products made from it are widely used in mechanical engineering (units and parts of gas turbine engines, radial and end-face plain bearings, mechanical seal rings), chemical technology (linings, on-off valves), instrument making (electrical insulators). Also, these materials are often used to manufacture thermal equipment (welding and gas nozzles, heaters, thermocouple protection tubes), armor plates, and so on [34].

## Solid-state sintered silicon carbide (SSiC)

The sintering process of silicon carbide powders is possible at high temperatures (2050 – 2500°C) [36]. The solid-state sintering process is based on the material mass transfer at high temperature and high pressure. The driving force during solid-state sintering is a decrease in the free surface energy of particles. Energy reduction is achieved either through the diffusion of atoms during matter transport from the grain volume to fill the pores or through the rearrangement of particles into the pore space [3].

Solid-state sintering can be divided into three stages. In the initial stage, the powder particles are brought together with the further formation and rapid growth of a neck between them, which is accompanied by shrinkage. These phenomena occur due to solid-state diffusion, diffusion through the gas phase (evaporation/condensation), and plastic or viscous flow. In the intermediate stage, the system compaction occurs due to the gradual reduction in the porosity and subsequent pore channel closure resulting in the formation of isolated pores [3]. The total porosity is reduced to approximately 10 vol.%. This stage usually covers the major part of the sintering process. In the final stage of sintering, the closed porosity decreases to 3 – 5 vol.% (at complete material densification) [37].

The main factors influencing the material microstructure and densification are the initial SiC grain size, sintering temperature and applied pressure. Better material densification is facilitated by using mechanically activated fine silicon carbide powder fractions (less than 1 micron) having frequent defects in the crystal lattice a high value of surface free energy. Products obtained from such powders are characterized by higher density [38, 39].

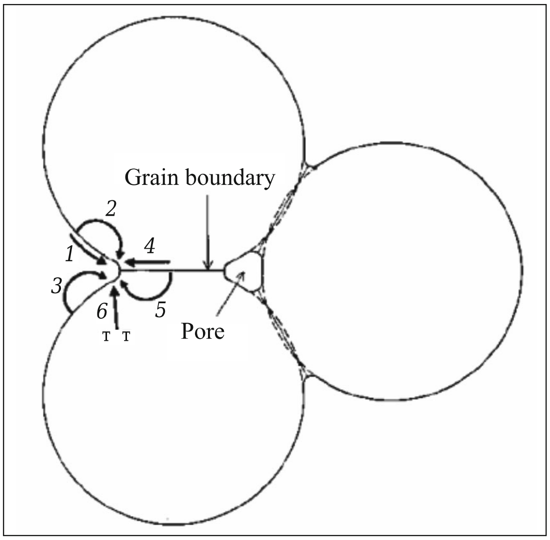

**Fig. 3.** Diagram of six mechanisms which contribute to the sintering of crystalline particles: *1*) surface diffusion; *2*) volume diffusion from the surface; *3*) vapor-phase diffusion; *4*) grain boundary diffusion; *5*) volume diffusion from the grain boundary; *6*) plastic flow. Only mechanisms *4* – *6* lead to densification.

Since the covalent bond in SiC is 80%, its self-diffusion is difficult. In this regard, mass transport during solid-state sintering of SiC is extremely low as it is carried out only due to the atomic diffusion in the solid state. Thus, pure SiC cannot be sintered to the theoretical density. Moreover, in highly covalent systems, one of which is SiC, grain growth often dominates over densification during solid-state sintering [34]. Consequently, a need arises to add sintering additives. Such additives, which form solid solutions, intensify diffusion, and slow down grain growth or accelerate sintering so much that the process of pore removal outpaces crystal growth [40 – 42].

Fig. 3 schematically shows the mass transport mechanism during sintering. The first three sintering stages are surface diffusion, lattice diffusion from the particle surface to the intergranular neck, and vapor transport due to neck growth without densification of the material (see Fig. 3) [3].

An increase in the process temperature does not lead to SiC densification since under such conditions sintering proceeds predominantly due to matter transport during its evaporation from the surface of powder particles and condensation on the contact surfaces between them (the so-called evaporation-condensation mechanism) [38, 43]. This process is not accompanied by redistribution of the matter. The distances between the particle centers do not change, and the pores practically do not heal.

The first materials obtained by pressureless (conventional or free) sintering were based on β-SiC and then on α-SiC with the addition of 2% boron and carbon [44, 45].

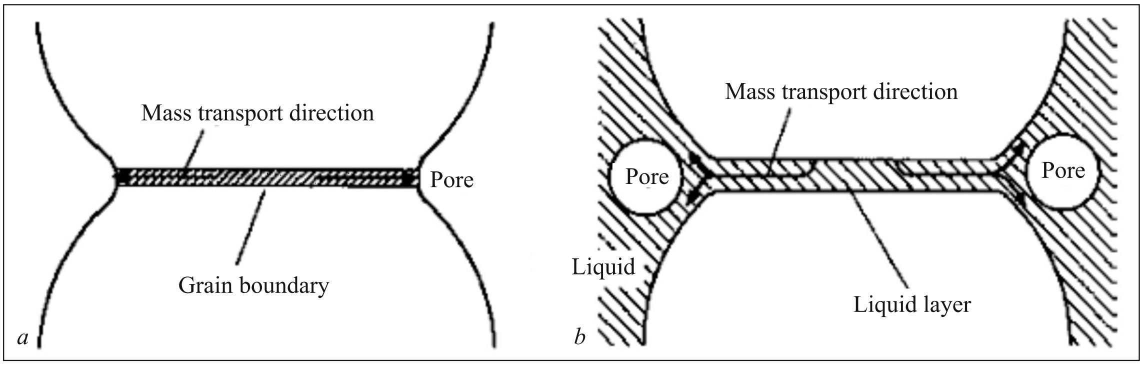

**Fig. 4.** Idealized two-sphere model comparing (*a*) solid-state and (*b* ) liquid-phase sintering.

During solid-state sintering, boron is introduced into the SiC lattice in place of silicon, thus forming a defect structure, which leads to an increase in volume diffusion [3, 44, 46 – 48]. Carbon interacts with the oxide film formed on the surface of SiC grains, reducing it [3]. The increased carbon content helps to slow down the growth of SiC grains and obtain a microstructure with rounded grains. Besides the additions of boron and its compounds, additives of aluminum, beryllium and their compounds are often employed [49, 50]. Elevated pressure in the sintering process increases the densification of silicon carbide materials, but it does not affect the grain growth rate [3].

The advantage of this sintering method is the feasibility of obtaining any geometry in a wide range of sizes without additional mechanical processing. The disadvantages of this method are significant shrinkage of the final product due to the densification process, as well as significant weight loss during sintering caused due to the decomposition of carbides caused by reactions of interaction of SiC with oxides [34].

## Liquid-phase sintered silicon carbide (LPSSiC)

Liquid-phase sintering is widely used to produce SiC materials. The principle of the method is to introduce additives into the original SiC powder. The additives melt at the sintering temperature to form a liquid phase between the SiC grains. Such a component is introduced into the system to increase the low self-diffusion of SiC grains due to accelerated mass transport through the liquid phase, which leads to an increase in the densification rate by facilitating the rearrangement of solid phase particles and ultimately obtaining a dense resultant material [3]. Figure 4 compares the microstructural aspects of solid-state and liquid-phase sintering on an idealized two-sphere model.

The liquid-phase sintering process can be divided into three stages. In stage one, the liquid formed in the sintering process wets the solid phase grains and covers them. Thus, the liquid layer separates the particles, which reduces their friction coefficient and simplifies their displacement relative to each other under the influence of compressive capillary stresses generated by the liquid. Solid particles also begin to dissolve in the liquid, thereby decreasing in size, which accelerates their rearrangement. In this process, part of the liquid is released and, under the influence of capillary forces, begins to fill the small pores between the particles. As the system densifies, its overall viscosity increases, which slows down the rearrangement process and leads to the second sintering stage.

The second stage is called the solution-precipitation stage. In regions of high chemical potential at the solid-liquid interface, the solid phase dissolves. The released solid atoms diffuse through the liquid phase into the regions of lower chemical potential, where they are deposited on the particle surfaces. The increased chemical potential regions where dissolution occurs are mainly the contact areas of solid particles that are under the influence of capillary and/or externally applied stresses. In two-component systems with the presence of particles of two different sizes, a coarsening of large particles occurs due to mass transport from smaller particles. This process is called Ostwald ripening. Thus, in the second stage of sintering, the microstructure is coarsened, and the particle surfaces are smoothed.

The last sintering stage starts when a rigid skeletal network of contacting solid grains is formed. Further densification occurs slowly due to long diffusion distances and the presence of a rigid skeleton. The main processes in stage three are Ostwald ripening and solid skeletal network sintering. Under the influence of pressure, the liquid from more densely packed areas can move into previously isolated pores, which also leads to an increase in packing density [3]. The liquid-phase sintering stages are shown schematically in Fig. 5.

The following parameters influence the process of liquid-phase sintering most strongly: the dispersion and particle morphology of the starting SiC powder, the nature and amount of the liquid component, powder activation methods, firing atmosphere (nitrogen or argon), sintering temperature and time [51]. Materials synthesized from fine powders with a rough surface possess the best properties. A decrease in the particle size and an increase in the number of surface and structure defects of a SiC powder increase the surface energy, which leads to an increase in the diffuse atomic mobil-

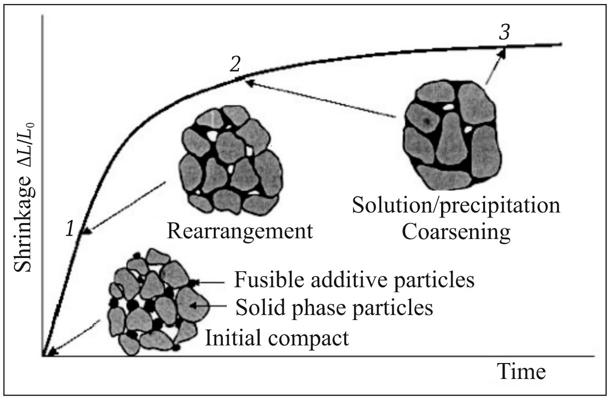

**Fig. 5.** Diagram of the evolution of a powder compact in the liquid-phase sintering process.

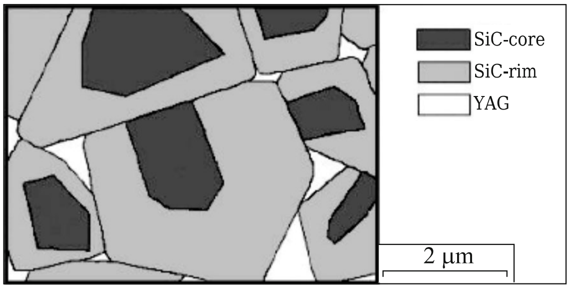

**Fig. 6.** Sketch of the liquid-phase sintered SiC structure.

ity, their degree of interaction, and sintering process acceleration. Silicon carbide materials synthesized from fine powders have high density and strength, as well as improved electrophysical properties [52].

To prevent the ceramic green body from being destroyed during sintering due to excess liquid between the powder particles, a limited amount of a melting component is introduced.

In some cases, the liquid phase evaporates or crystallizes towards the end of sintering. As a result, the glass phase is either completely absent from the final product or is present as an inclusion in the solid phase when a solid solution is obtained. Such SiC is often classified as solid-state sintered. An example is the sintering of SiC doped with Al and C [53] or with the addition of $Al_2O_3$ [54]. The effectiveness of dopants is determined by the uniformity of their distribution between the sintered powder grains. $Al_2O_3$, $Y_2O_3$, MgO, $ZrO_2$, CaO, as well as AlN are often used as additives that activate the sintering of SiC powders [35, 41].

However, the advantages gained from introducing a liquid component during sintering make sense only if the properties of the resulting silicon carbide product remain within acceptable values. After cooling, the sintered material is a two-phase system. In it, the glass phase is located along the grain boundaries of the solid phase. Its presence can degrade the high temperature mechanical properties of the final product, such as strength, creep, and fatigue resistance [3]. A schematic representation of the microstructure of a ceramic material produced by liquid-phase sintering with the addition of yttrium aluminum garnet (YAG) as a dopant is shown in Fig. 6.

Liquid-phase sintering of SiC has several advantages over the solid-state method. The materials obtained by the former technique have a denser structure with a smaller grain size since the sintering process occurs at a lower temperature in the range of 1850 – 1950°C, which is 100 – 300°C lower than the temperature of solid-state sintering (2050 – 2150°C). Liquid-phase sintering also requires lower production costs compared to solid-state sintering [34].

Liquid-phase sintering can produce silicon carbide articles of various sizes with high productivity [48]. The products obtained by this method are used as parts and elements experiencing long-term static or short-term dynamic stresses [56]. These can be plain bearings and various friction units. Also, these silicon carbide materials are utilized as lining and armor materials [57].

## Hot pressed silicon carbide (HPSiC)

The principle of hot pressing is the simultaneous influence of elevated temperature and pressure in the sintering of a powder or a preform obtained by cold pressing. According to [36], highly dense (>95% of the theoretical) silicon carbide materials can be obtained at high temperatures of ~2,300 – 2,500°C and very high pressures reaching 1 GPa. In this regard, in practice, to reduce the process parameters, fine SiC powders are used, and sintering additives are introduced [6, 41]. Moreover, it is possible to physically activate powders further by explosive treatment to increase defect concentration, remove moisture and oxide layers from the surface, and so on [4].

Pressure applied to the material suppresses the internal pore pressure helping to reduce porosity without significantly increasing grain size [3, 58]. In the hot pressing process, matter transport occurs by a diffusion mechanism in conjunction with plastic deformation. The latter plays a significant role in the healing of isolated pores. However, without sintering additives, pure SiC does not have a sufficiently high plastic deformation up to the decomposition temperature, which necessitates the creation of stringent conditions to achieve a high density of the final material [36].

The molding pressure used in practice is 30 – 50 MPa as it is limited by the strength of the employed graphite die [59]. The temperature usually does not exceed 2000°C. At temperatures above 1200°C, the process is carried out in an inert or reducing atmosphere [3]. Figure 7 shows the diagram of a laboratory-scale hot press [60].

In several studies examining the production of silicon carbide materials by hot pressing, the hot-pressed material was additionally subjected to heat treatment after sintering to reduce stress concentrations in the material, which led to an improvement in fracture toughness [61 – 66].

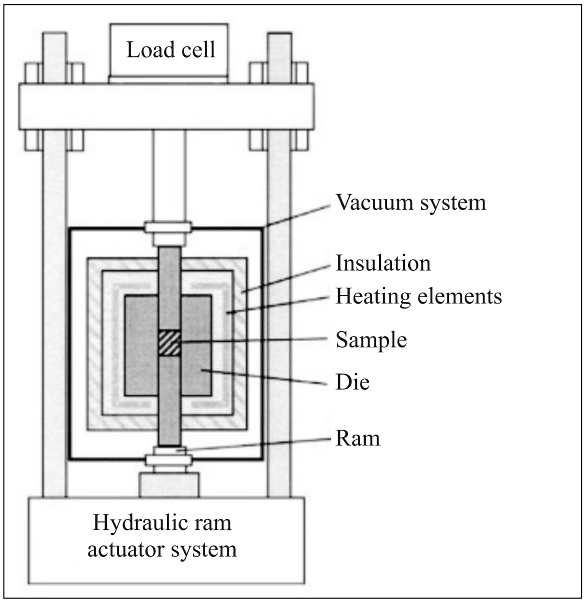

**Fig. 7.** Schematic of the hot pressing process.

The hot pressing method has a number of disadvantages. First, the high cost and energy intensity of the process coupled with its low productivity are worth noting. Therefore, this method is not used in industry. The resulting products are limited by the simple geometry of the dies used. Moreover, significant shrinkage of the material occurs. In the case of uniaxial hot pressing, compaction occurs only in one direction (pressing direction), which leads to anisotropic orientation of grains elongated perpendicular to the compacting pressure (Fig. 8), and consequently, to anisotropy in mechanical properties and wear of the material [3].

The main advantage of this method is the possibility of obtaining high-density SiC (porosity <5 vol.%) with excellent mechanical characteristics [67]. It is also worth noting that sintering time is reduced since the pressing and sintering processes occur simultaneously.

## Hot isostatic pressing of silicon carbide (HIPSiC)

A more high-tech synthesis method is hot isostatic pressing. The preconsolidated powder is placed in a deformable can which is subjected to isostatic pressing due to gas pressure. During the process, the initial pressure is maintained while the sample is heated to the sintering temperature. After reaching the required temperature, the gas pressure gradually increases to a fairly high value [3].

HIP systems, as a rule, can be operated at temperatures not exceeding 2,000°C and pressures up to 400 MPa, which distinguishes them favorably from hot pressing systems [41, 68]. Also, this method permits obtaining parts of complex geometries with a small density difference in the volume [6, 69 – 71].

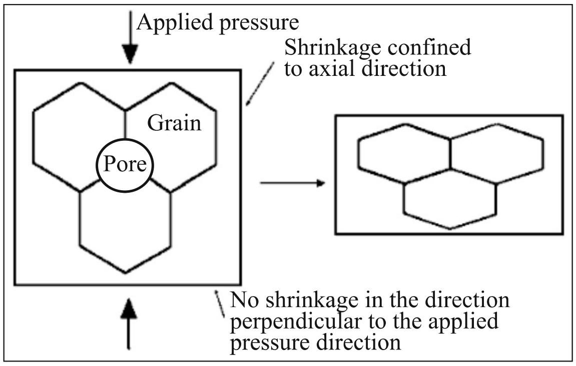

**Fig. 8.** Schematic diagram illustrating grain deformation during hot pressing.

## Spark plasma sintering of silicon carbide (SPSSiC)

The principle of spark plasma sintering (SPS) consists in simultaneous pressing and sintering of powders by passing short current pulses to a die made of a conducting material. The current pulses heat the conducting powders in the die. Non-conducting materials are heated from the walls of the heated conducting die. The duration of one dc current pulse can be from $10^{-5}$ to $10^{-3}$ sec. The heating rate in SPS systems can reach 1,000°C/min. In practice, a heating rate of about 100°C/min is usually used during sintering materials. The applied pressure, as a rule, does not exceed 100 MPa [34]. A schematic diagram of a spark plasma sintering system is shown in Fig. 9 [72].

To date, the spark plasma sintering mechanism has not been fully studied [72 – 75]. Most often, SPS is described as follows. Passing through the powder compact in the die, the current pulse produces two types of thermal effects [76]. Heating between powder particles in the presence of ohmic contacts between them occurs due to Joule heating. In the absence of such contacts, an electric charge buildup occurs at the interfaces between the particles and the voids. When a critical value is reached, a spark discharge occurs, i.e. a source of discharge plasma. The temperature in the discharge region can reach 7000 – 10,000°C [72], which leads to short-term but intense heating of the space between the particles. In the particle contact area, the powder surface is strongly heated, which causes the appearance of a temperature gradient. Under its influence, atoms diffuse from the hot region to the cold one. Gradually, atoms concentrate in the area with maximum surface curvature and form a neck between the particles (Fig. 10).

Thus, rapid heating results in surface diffusion, the main mechanism of matter transport during SPS. The impact of spark discharges also leads to the removal of the adsorbed gases, oxide layers, and contaminants from the powder surface, which significantly improves the sintering behavior of the material. Moreover, performing SPS generates an electromagnetic field which further enhances the diffusion of atoms due to electromigration effects [72].

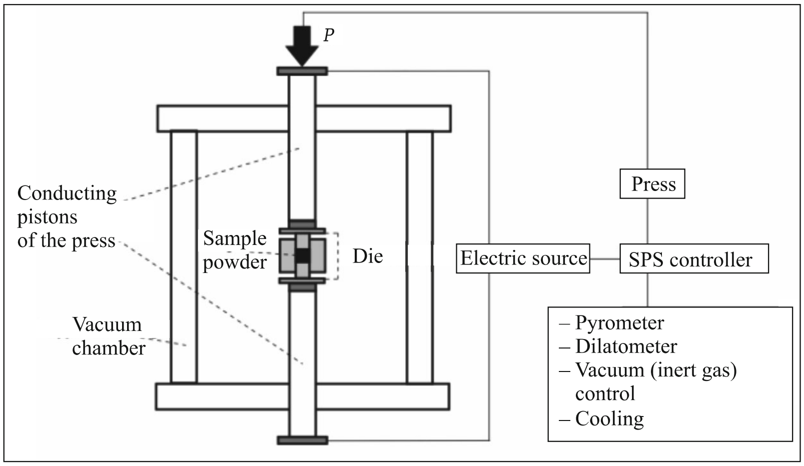

**Fig. 9.** Schematic diagram of a spark plasma sintering apparatus.

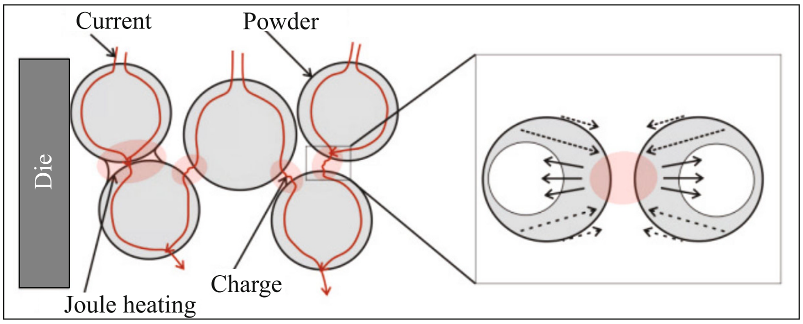

**Fig. 10.** Schematic diagram of a current pulse passing through a sintered powder in SPS [72, 76]: solid lines — movement of atoms from a heated region to a cold one; dotted lines — movement of atoms from the surface to the region with maximum curvature.

The main advantage of this method is the possibility of obtaining ceramics from nanosized particles with excellent mechanical properties. Since the pulsed nature of heating and the absence of holding at high temperatures ensure rapid powder sintering in a short period of time, SiC grains do not have time to greatly increase in size [76 – 78]. The disadvantages include the possibility of producing simply shaped articles as only axisymmetric dies should be used in a SPS apparatus [3].

## COMPARISON OF THE PHYSICAL AND MECHANICAL PROPERTIES OF SILICON CARBIDE CERAMICS PRODUCED BY DIFFERENT PROCESSING METHODS

Table 1 compares the physical and mechanical properties of SiC-based ceramic materials obtained by using different processing methods discussed above.

When choosing a method for producing structural articles, there are other important aspects to consider in addition to its operational properties. These include the possibility of obtaining products with complex shapes and/or large dimensions, products with precise geometric parameters (which is affected by the presence or absence of shrinkage), labor intensity, and cost of production (Table 2).

Ceramic-bonded and recrystallized SiC have low strength characteristics due to their low density and weak bonds between SiC particles. However, they have high refractoriness (operating temperature reaches 1600°C) and are easy to obtain. These materials are used as refractory loading and structural elements of furnaces.

Silicon carbide ceramics produced by hot pressing and spark plasma sintering have the best properties. Thus, they are promising candidates for manufacturing structural materials which are expected to operate under aggressive conditions at high loads and temperatures. However, the high cost of production and the difficulty of obtaining parts with complex geometries make it possible to produce only a limited number of articles. Currently, these sintering methods are mainly used to obtain laboratory samples for research. In this regard, a promising area for development is the production of articles using the labor-intensive method of hot isostatic pressing. Employing it, one can obtain products with more complex shapes.

Another promising area for developing silicon carbide technologies is the optimization and improvement of the production of solid-state and liquid-phase sintered, as well as siliconized SiC. Solid-state sintering is inferior to the liquid-phase method in several parameters: high sintering temperature (2050 – 2150°C), labor-intensive production, worse mechanical properties at low temperatures. However, SSiC materials have high strength at ambient as well as at elevated temperatures, which is due to the low-melting phase absence in the ceramic microstructure. Consequently, it is logical to systematically design specific products from this material for their operation in conditions requiring increased strength and heat resistance.

Materials based on liquid-phase sintered SiC have a complex of high strength characteristics and seem to be the most versatile ones in terms of practical application.

Products made from siliconized SiC are currently very widely used in various fields of mechanical engineering. The reaction sintering method for producing heat-resistant special engineering products based on SiC has several advantages. They are as follows: maintaining high strength characteristics at a significantly lower production cost by reducing the sintering temperature; no change in the geometric dimensions of the material after sintering (non-shrink technique); obtaining a non-porous material using various types of molding (pressing, casting, additive technologies) and, as a conse-

**TABLE 1.** Average physical and mechanical characteristics of SiC-based materials [34, 79, 80]

| SiC-based material | Density $\rho$, g/cm$^3$ | Porosity $\Pi$, % | Elastic modulus $E_{\text{elas}}$, GPa | Thermal conductivity at 600°C $\lambda$, W/(m·K) | Bending tensile strength at 20°C $\sigma_{\text{bend}}$, MPa |
|---|---|---|---|---|---|
| CSiC (ceramic-bonded) | 2.55 | 20 | 100 | 16 | 30 |
| RSiC (recrystallized) | 2.60 | 20 | 250 | 28 | 100 |
| RBSiC (reaction-bonded) | 2.60 | 20 | 250 | 25 | 250 |
| SiSiC (reaction-sintered or siliconized) | 3.10 | <1 | 400 | 60 | 400 |
| SSiC (solid-state sintered) | 3.15 | <2 | 410 | 50 | 430 |
| LPSSiC (liquid-phase sintered) | 3.21 | <2 | 420 | 50 | 450 |
| SPSSiC (obtained by spark plasma sintering) | 3.18 | <1 | 440 | — | 490 |
| HPSiC (hot-pressed) | 3.20 | 0 | 450 | 55 | 640 |
| HIPSiC (obtained by hot isostatic pressing) | 3.21 | 0 | 450 | 75 | 640 |

**TABLE 2.** Comparison of technological characteristics of SiC-based materials

| SiC-based material | Shrinkage | Ability to form complex shapes | Labor intensity |
|---|---|---|---|
| CSiC (ceramic-bonded) | No | Yes | Low |
| RSiC (recrystallized) | No | Yes | Low |
| RBSiC (reaction-bonded) | No | Yes | Low |
| SiSiC (reaction-sintered or siliconized) | No | Yes | Low |
| SSiC (solid-state sintered) | Significant | Yes | Average |
| LPSSiC (liquid-phase sintered) | No | Yes | Low |
| SPSSiC (obtained by spark plasma sintering) | Significant | No | High |
| HPSiC (hot-pressed) | Significant | No | High |
| HIPSiC (obtained by hot isostatic pressing) | Significant | Yes | High |

quence, the possibility of manufacturing a geometrically complex product with thin blades.

The main disadvantage of this material is the operating temperature (1200 – 1300°C) limited by the beginning of residual silicon melting. A way to improve this technique may be a reduction of the amount of silicon in the final material. This can be achieved through a more complete process of secondary SiC formation and filling the pores with it, including by varying the carbon component content and decreasing the total pore volume in the green body.

## CONCLUSION

The performed literature analysis shows that silicon carbide ceramic materials are promising candidates to be used in knowledge-intensive industries (energy, space, military, etc.) which require structural materials capable of withstanding increased loads in the conditions of aggressive environments and high temperatures.

This article briefly presents various methods for producing ceramics. It is worth singling out liquid-phase and reaction-sintered SiC as the most promising ones. They are distinguished by the technology simplicity, high productivity, and high performance characteristics of the resulting silicon carbide materials.

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