

Article

Structure and Properties of $\text{Ti}_3\text{AlC}_2\text{-SiC}$ and $\text{Ti}_3\text{AlC}_2\text{-TiC}$ Materials Obtained by Powder Injection Molding Technology

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Abstract: Powder injection melting (PIM) and material extrusion additive manufacturing (MEAM) are promising production technologies that allow us to obtain products from modern composite materials. These technologies make it possible to obtain products of complex shape from ceramic composite materials, which is a non-trivial task. A highly filled polymer feedstock is used as a source material in such technologies. In this work, a study of the structures and properties of samples obtained from $\text{SiC-Ti}_3\text{AlC}_2$ and $\text{TiC-Ti}_3\text{AlC}_2$ feedstocks by the PIM method was performed. The main purpose of this work was to study the influence of the powder compositions in feedstocks on the rheological properties and the structures of the obtained samples, as well as to determine the sintering parameters of samples of these compositions. In the future, it is planned to use the received and studied feedstocks in material extrusion additive manufacturing (MEAM). It was found that the investigated compositions had different MFI values depending on the composition. The effect of the sintering parameters on the structure and properties was shown. During the sintering of $\text{SiC-Ti}_3\text{AlC}_2$ ceramics in the temperature range of 1200–1400 °C in a vacuum, the main components of the mixture interacted with the formation of Ti_3SiC_2 and TiC. For materials of the $\text{TiC-Ti}_3\text{AlC}_2$ composition, partial oxidation of the material and the formation of titanium and aluminum oxides was observed.

Keywords: MAX phase; ceramic composite; nanolaminates; feedstock



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1. Introduction

Additive technologies (AT), also known as 3D printing technologies, are developing rapidly today. With the development of AT based on the selective sintering or melting of powder materials with laser or electron beams, the production of materials and products from them, with predetermined properties, depends not only on the properties of the initial material but also on the properties of the remelted or sintered material. The properties of such materials strongly depend on the structure and phase composition, which are determined by the elemental and chemical compositions of the initial powder material, the composition of the sintering environment, and the thermal conditions during high-energy treatment [1].

AT have a number of advantages over “classical” metalworking technologies—a high material utilization rate, the ability to create products of complex geometry, the absence of welded joints in complex products, and so on. To date, the range of high-temperature materials used in AT is very narrow; meanwhile, when using AT, materials that can withstand high operating temperatures and other extreme conditions are of great interest.

Ternary carbides and nitrides, called MAX phases, form a new class of materials that have very specific properties, combining the properties of both metal alloys and ceramic materials [2]. Similar to metals, these compounds show high thermal and electrical conductivity, as well as high resistance to thermal shock. At the same time, as with ceramics, they have a high modulus of elasticity, a low coefficient of thermal expansion, and high

heat resistance. The resistance of MAX phases to cyclic loads at temperatures above 1000 °C exceeds that of most known heat-resistant materials, including nickel alloys and alloys based on intermetallic compounds. Together with a low specific weight, these properties of MAX phases imply broad prospects for their application in various industries [3].

Promising MAX phases are formed in the Ti-Al-C system. The materials of this system offer high strength and deformation resistance, including cyclic loading [4]. Chemical etching of aluminum results in the formation of so-called MX-enes, which, due to their nanolaminate structures, can be used for the storage of electrical energy or hydrogen [5,6]. In addition, the MAX phases of the Ti-Al-C system are resistant to high-temperature oxidation and retain their strong mechanical properties even at high temperatures [7].

To date, the main research in the field of MAX phases of the Ti-Al-C system can be divided into two main groups: the synthesis and the creation of composite materials based on MAX phases. Synthesis studies imply the optimization of the experimental parameters to obtain MAX phases, and research in this area is associated with high-temperature processes, such as self-propagating high-temperature synthesis (SHS) [8–10] and spark plasma sintering (SPS) [11–13], as well as synthesis processes under pressure, such as hot isostatic pressing (HIP) [14–16]. Such technologies have a number of disadvantages—in particular, low productivity at a high cost.

The direction of the study of composites includes complex structural studies and mechanical testing of materials containing the Ti-Al-C system in the composition of the MAX phase together with other compounds, mainly including TiC [17] and intermetallic compounds of the Ti-Al system [15,18]. These compounds are used because they can be obtained as reaction products in the Ti-Al-C system, which simplifies the synthesis of a composite material. There are also composites that, in addition to the MAX phase, contain various metals (copper [19], silver [20], etc.), nickel alloys [21,22], silicon carbide [23], aluminum oxide [24], and others. These works imply that the technologies described above can be used for the synthesis of MAX phases (SHS, SPS, HIP) or simple sintering with long exposures.

A major feature of the described works is the study of the formation of phases and structure-forming processes in simple samples, without describing the formation of complex sample geometry, which is a non-trivial task.

Methods for the extrusion production of products (powder injection molding-PIM) are also actively being developed today; however, such works are most often devoted to metallic materials (MIM) [25–27]. In ceramic casting technologies (CIM), ceramics of the construction [28] and medical [29] fields are mainly used. At the same time, the additive (3D-PIM) formation of these products has been poorly studied, since research teams are mainly focused on the more expensive and less reliable InkJet 3D printing technology [30].

In this work, a study of the structures and properties of samples obtained from feedstocks by the PIM method is performed. In the future, it is planned to use the received and studied feedstocks in material extrusion additive manufacturing (MEAM). The feedstocks use mixtures of Ti_3AlC_2 powder (TAC) with ceramic powders of TiC and SiC at different ratios. The effect of the sintering parameters on the formed structures and properties is shown.

2. Materials and Methods

The Ti_3AlC_2 MAX-phase powder (Jiangsu XFNANO Materials Tech Co., Ltd, Jiangsu, China; 98% purity, $d_{50} = 5 \mu\text{m}$), SiC powder (DENSITEC L, SIKA, Oslo, Norway, $d_{50} = 5 \mu\text{m}$), and TiC powder (Reakhim, Smolensk, Russia; $d_{50} = 5 \mu\text{m}$) were used in this work. The methodology used to obtain the powders, as well as their characterization, is described in [31,32].

Mixing of the powders was carried out by the dry method in a turbula-type mixer (Turbula S2.0, Vibrotechnik JSC, Saint-Petersburg, Russia). Powder mixtures Ti_3AlC_2 -TiC and Ti_3AlC_2 -SiC were prepared using different ratios: 30 to 70, 50 to 50, and 70 to 30 by volume.

A feedstock was prepared with the MC2162 polymer binder (Emery Oleochemicals, Dusseldorf, Germany). The MC2162 binder is a mixture of polyester and polyamide with a ratio of 1:5 by mass. The powder content was 35 vol.% for all feedstocks. For MFI investigations, feedstocks with different powder:polymer ratios were obtained. The dry pre-mixture of powder mixtures with the polymer binder was prepared in a Z-blade mixer SPQ-10 (Welber, Moscow, Russia) with a 60 rev/min rotational speed and a mixing time of 6 h. Premixed material was then extruded in a single-screw extruder LE45–30 (Scientific, Saint-Petersburg, Russia) at 145 °C to obtain a homogeneous feedstock.

The melt flow index (MFI) for all feedstocks was measured with a plastometer IIRT-M (LOIP, Saint-Petersburg, Russia) with a load of 5 kg at 150 °C. The measurement of MFI was carried out according to ISO 1133. The TG-DSC analysis of feedstocks was carried out on the STA 449 F3 Jupiter setup (NETZSCH, Selb, Germany) to record the binder decomposition behavior. The analysis was performed from room temperature up to 1100 °C at the heating rate of 10 K/min in an argon atmosphere.

Test PIM samples were obtained in a FORMOLDER PRO injection molding machine (Novosibirsk, Russia). The feedstock was heated to a temperature of 140–145 °C before injection. The injection was carried out at 145–150 °C at the pressure of 120–130 bars. The samples were held for 80 s in the machine after injection at 40–45 °C. Samples were then removed from the machine and cooled down to room temperature. Debinding of the samples was carried out in acetone for 24 h, followed by drying of the samples in an oven at 60 °C for 6 h. The density was measured by the hydrostatic weighing method.

The samples were sintered in a vacuum furnace (Nabertherm GmbH, Lilienthal, Germany) at temperatures of 1200, 1300, and 1400 °C on a corundum substrate. The heating rate was 5 K/min, and the isothermal holding time at the sintering temperature was 1 h. The density of sintered samples was determined by hydrostatic weighing.

The microhardness of the samples was measured on a microhardness tester (Duramin-500, Struers, Denmark) by the Vickers method (HV) at a load of 200 gf. The samples were studied by optical metallography (Axiovert 200-MAT, Carl Zeiss GmbH, Germany) and scanning electron microscopy (Apreo S, Thermo Fisher Scientific, Waltham, MA, USA). For the study, cross-sections were prepared on a disc grinding-polishing machine (Saphir 520, ATM Qness GmbH, Mammelzen, Germany) using SiC paper with a 400–1600 mesh size and diamond discs with a 2000–4000 mesh size.

The phase composition of the samples was studied using an XRD-7000S X-ray diffractometer (Shimadzu, Japan) in the Bragg-Brentano configuration using CuK α radiation with a long wavelength $\lambda = 0.154$ nm at X-ray tube parameters of 40 kV and 30 mA. For each of the samples, a spectrum was collected with the following parameters: scanning step—0.0143°; sample scanning speed—10°/min; range of angles in 2θ —30–90°.

3. Results and Discussion

3.1. Melt Flow Index of the Feedstocks

The melt flow index (MFI) is an important technical characteristic of feedstocks. MFI values for feedstocks applicable in PIM and MEAM technologies should be no more than 60 g/10 min, since, at higher MFI values, a stable shape cannot be achieved in the forming processes. In addition, other defects may appear, such as weld lines, flash, and others [33]. If the feedstock has an MFI below 20 g/10 min, the PIM process fails to effectively fill the injection mold, and the main defects in this case are voids. It should be noted that for the MEAM technology, the limitation of the MFI is significantly lower—about 2–5 g/10 min—and the main condition is the extrusion of the material through the nozzle, which is also possible for feedstocks with a low MFI.

The measurement of the MFI for feedstocks showed similar dependences (Figure 1)—in composites with both SiC (Figure 1a) and TiC (Figure 1b), an increase in the MFI occurred with an increase in the amount of TAC and with an increase in the polymer content. The fluidity of the feedstocks was primarily determined by the polymer content; therefore, with

an increase in the polymer content in the feedstock, the MFI increased. The growth of the MFI with an increase in the content of TAC was not trivial.

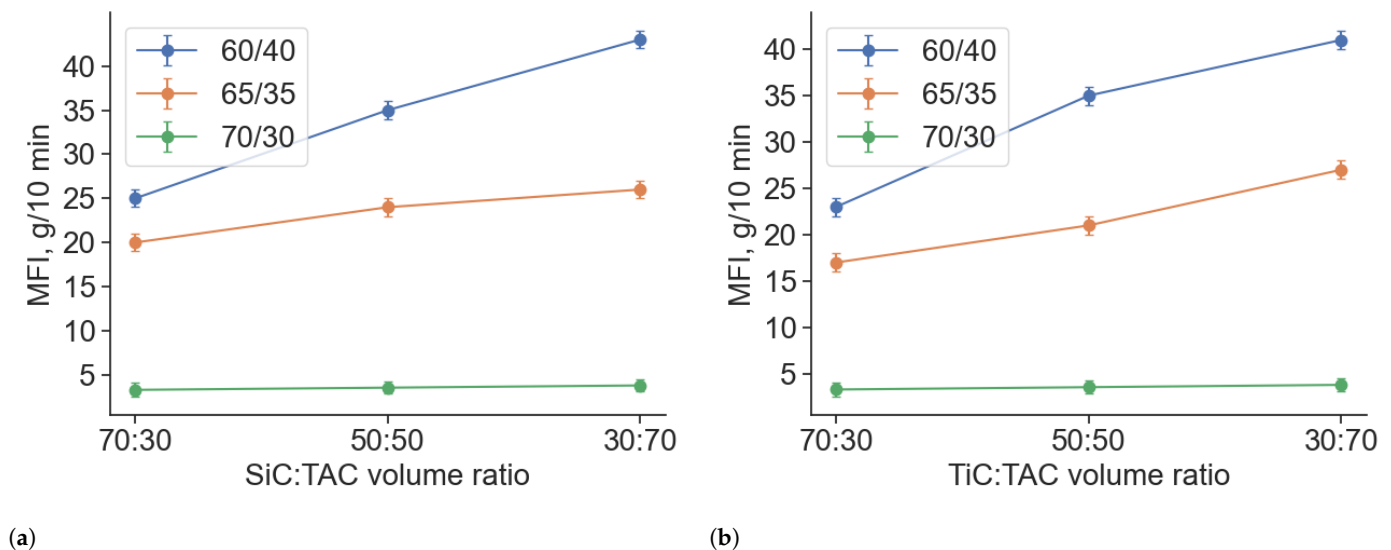


Figure 1. Melt flow indexes (MFI) of the SiC-TAC (a) and TiC-TAC (b) feedstocks with different SiC:TAC, TiC:TAC, and powder:polymer ratios.

The MFI values for the studied feedstocks did not exceed 43 g/10 min. This value was achieved in feedstocks with the highest content of polymer binder (40 vol.%). For the composites that we studied earlier, the MPI values for similar materials were higher and reached 2500 g/10 min for some compositions [34].

The MFI data do not fully depend on the structure of the samples. Samples with larger agglomerates should have a lower MFI. The size of the agglomerates is approximately the same for systems with SiC and TiC; however, the trend of change in the MFI is different in these systems. When the feedstock flows, it is necessary to expend energy in the destruction of the agglomerates, including due to the interparticle shear action. In this regard, the larger the agglomerates, the more energy is required for their destruction, and the smaller the MFI. However, if only structural features influence the MFI, then the dependences for systems with TiC and SiC will be similar. Thus, the structure, particularly the size of the agglomerates, does not have a predominant effect on the MFI of the samples.

A feature of the feedstocks studied in this work is the filling of the polymer with non-spherical particles. When filled with spherical particles, the fluidity of the feedstock is higher due to the higher fluidity of the spherical powder without a polymer binder. In bimodal (nano-micro) powders, the ball bearing effect can additionally appear [35], further increasing the fluidity of the feedstock. The used SiC and TiC powders are not characterized by a spherical shape, while the TAC powders are characterized by sharp edges due to the nanolaminate structure.

However, an increase in the content of coarse TAC particles leads to an increase in the MFI of the feedstocks. The reasons for this phenomenon are not entirely clear, since the dispersed composition of the used powders is close, but the shape of the SiC and TiC particles is closer to spherical. Presumably, in addition to mechanical effects associated mainly with the size and shape of particles, catalytic effects (chemical interaction with the polymer) are manifested in TAC [36,37].

3.2. Microstructure

An important feature of the sintering of PIM and MEAM samples is the need to remove residual polymer. The polymer is preliminarily removed at the debinding stage. However, the backbone polymer remains, which is removed at a temperature of 500–600 °C. After removing the backbone polymer, the product becomes brittle, so it is necessary to sinter the

product. Ceramic composites require a high sintering temperature, but the MAX phases decompose at high temperatures. Thus, the sintering temperatures of 1200, 1300, and 1400 °C were chosen as the temperatures that ensured the sufficient sintering strength of SiC and TiC without leading to the mass decomposition of the MAX phase.

The microstructures of the sintered SiC-TAC samples were represented by large pre-sintered agglomerates of particles (Figure 2). The sintering mechanism consisted of the sintering of particles at the contact points with the necking processes; both particles of the same composition and SiC-TAC particles were sintered. The fracture surfaces of the samples also showed insufficient sintering of the powder. With an increase in the content of SiC in the material, the structure was refined, and the size of the agglomerates decreased from 11 ± 3 to 3 ± 1 μm . The reason for the refining of agglomerates is in the finer size of the SiC powder—with an increase in the content of SiC, the agglomerates begin to contain more fine powder. Agglomerates are represented by a mixture of SiC and TAC powder. An increase in the sintering temperature does not lead to a significant increase in the size of the agglomerates; however, stronger necks between the particles appear, and the agglomerates themselves become more monolithic.

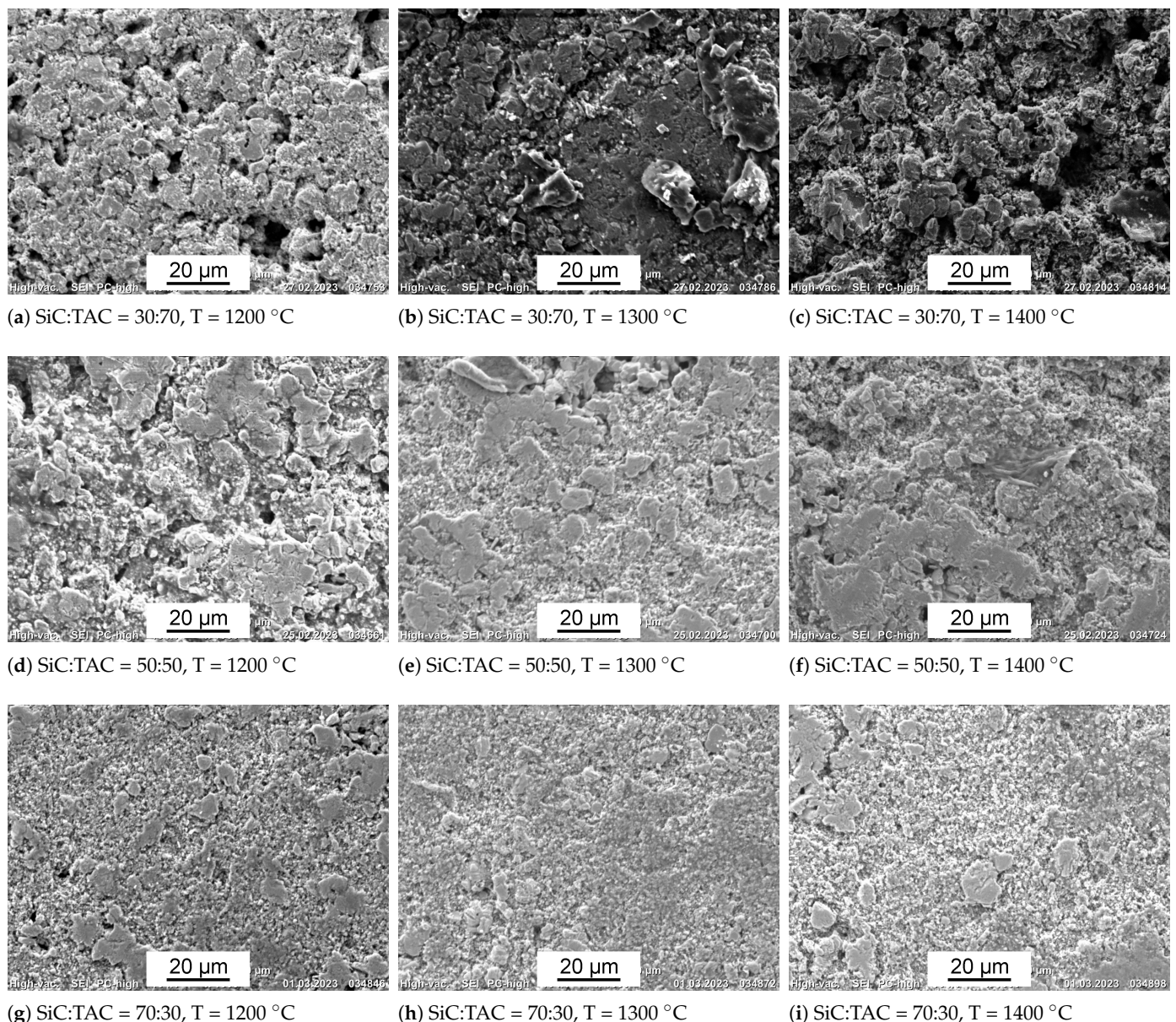


Figure 2. Structures of the SiC-TAC samples with different SiC:TAC ratios and sintering temperatures.

The microstructures of the sintered TiC-TAC samples are characterized by high porosity and the presence of larger agglomerates at all temperatures (Figure 3). The mechanism of agglomerate formation is similar to that for the SiC-TAC system. In samples with a TAC content of 70 vol.%, there are agglomerates larger than 20 μm . With an increase in the TiC content, the size of the agglomerates decreases (from 15 ± 3 to $5 \pm 1 \mu\text{m}$), and with an increase in the sintering temperature, the density of the agglomerates increases for all compositions. The structure of TiC samples is looser due to the presence of large pores between agglomerates.

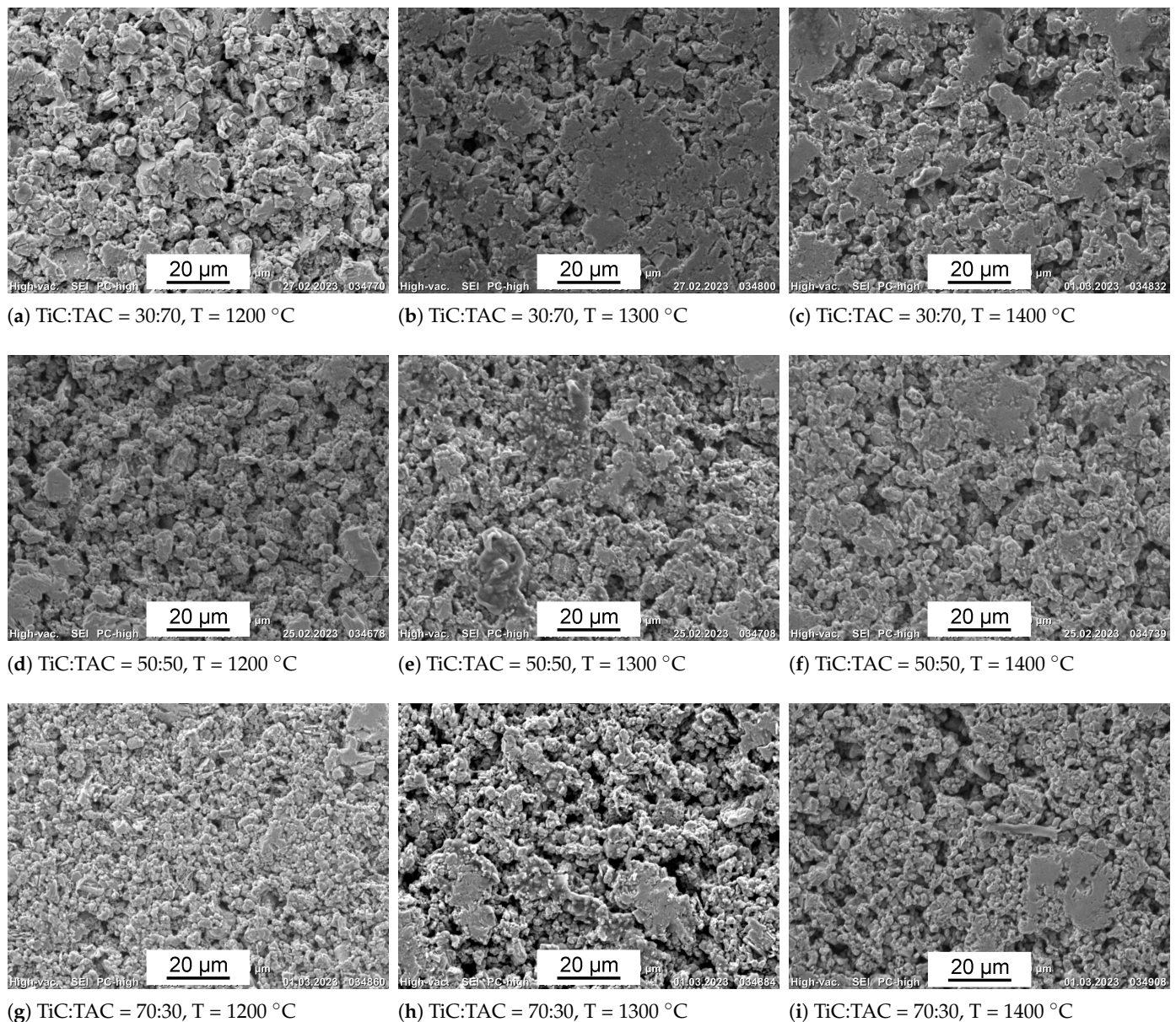


Figure 3. Structures of the TiC-TAC samples with different TiC:TAC ratios and sintering temperatures.

Measurement of the microhardness of the samples (HV 0.5/10) showed that all the samples were not completely sintered. For all samples, the microhardness values are in the range of 200–500 HV, which is significantly lower than the values for pure components (SiC and TiC) sintered in a vacuum at the same temperature. A feature of SiC and TiC is the need for long-term exposure or sintering at a higher temperature. On the other hand, the possible decomposition of TAC during high-temperature sintering is undesirable; therefore, the sintering temperature was not increased in this work. Increasing the sintering time

can make it possible to achieve denser sintering of the samples. The sintering parameters given in this work are sufficient for the formation of intermediate properties of products from SiC-TAC and TiC-TAC composites; then, the products can be finished, including, for example, by hot isostatic pressing (HIP) to reduce the porosity. Intermediate-stage sintering is important for PIM and MEAM products, as it allows the complete removal of the residual polymer.

3.3. Sintering of SiC-Ti₃AlC₂

The used commercial silicon carbide powder (β -SiC, polytype 6H, Figure 4) contained additives of Y₂O₃ and Al₂O₃, which provided the liquid-phase reaction sintering of SiC due to the eutectic formation in the Y₂O₃-Al₂O₃ system at temperatures above 1700 °C [38,39].

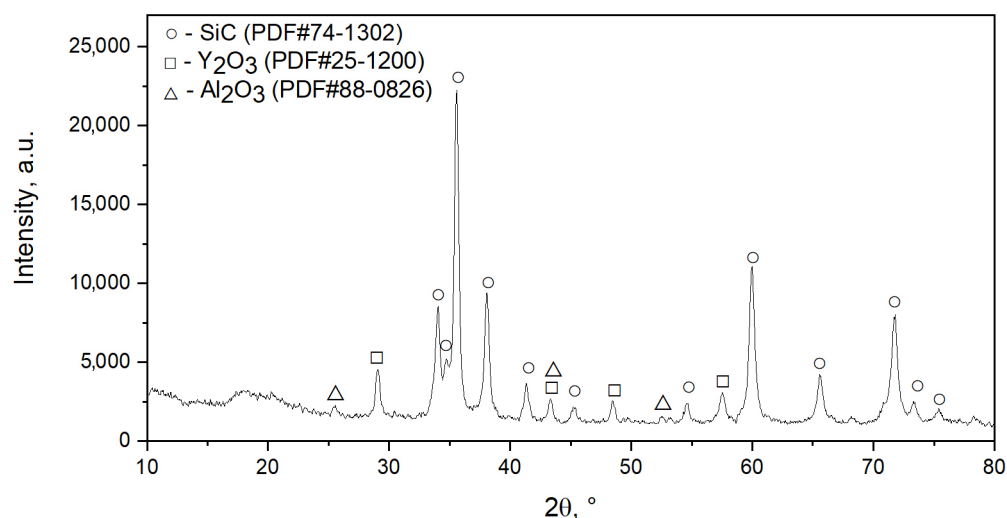


Figure 4. X-ray diffraction pattern of SiC starting powder.

During the sintering of SiC-Ti₃AlC₂ ceramics in a vacuum at a temperature of 1200 °C, the interaction of the main components of the mixture occurs with the formation of a new MAX phase, Ti₃SiC₂. A direct reaction between SiC and Ti₃AlC₂ is unlikely due to the complexity of the structures of the reactants and the reaction product. According to [40], the interaction of Ti₃AlC₂ macroparticles with SiC at temperatures above 1200 °C results in the formation of TiC.

Moreover, when Ti₃AlC₂ is sintered in a vacuum, thermal dissociation with the formation of TiC and Ti is possible, while Al is removed from the reaction volume into the gas phase [41]. The described mechanism partly explains the absence of Al-containing phases (metallic aluminum, aluminum carbides, etc.) in the compositions of the materials. Thus, the most probable sequence of processes is as follows: (i) the decomposition of Ti₃AlC₂ with the formation of active TiC and Ti; (ii) the interaction of TiC and Ti with SiC to form Ti₃SiC₂, as one of the most thermodynamically favorable processes in the synthesis of Ti₃SiC₂ [42].

The formation of metallic tin (Sn) at a temperature of 1200 °C is proof that the formation of Ti₃SiC₂ occurs due to the decomposition of Ti₃AlC₂. Tin is a common additive that activates the synthesis of Ti₃AlC₂ [43].

The presence of metallic and gaseous aluminum in the reaction volume probably mineralizes the synthesis of yttrium aluminum garnet Y₃Al₅O₁₂, the presence of which is recorded already at a temperature of 1200 °C. This agrees with the literature data [44,45]: aluminum is able to wet the Y₂O₃ surface with the formation of the AlYO₃ intermediate compound, which is more actively involved in the process of garnet synthesis.

The presence of metallic tin and titanium in the reaction volume leads to the formation of Sn₃Ti₅, which is first seen in the ceramic composition in a low-crystalline X-ray amorphous form, and when the temperature is increased to 1400 °C, it develops a crystalline form [46].

With an increase in the content of Ti_3AlC_2 in the mixture up to 50 vol. %, there is a significant increase in the yield of yttrium aluminum garnet with the increasing temperature, which is due to an increase in active aluminum in the reaction volume. In this case, a small amount of the independent TiC phase is also formed, probably due to the limited mutual diffusion of ions at the $\text{SiC}/\text{Ti}_3\text{SiC}_2/\text{TiC}$ phase boundary, which limits the amount of the formed Ti_3SiC_2 MAX phase (Figure 5). In ceramics with the highest content of Ti_3AlC_2 (70 vol.%), the smallest amount of the yttrium aluminum garnet phase is observed due to the low content of the corresponding oxides in the initial mixture.

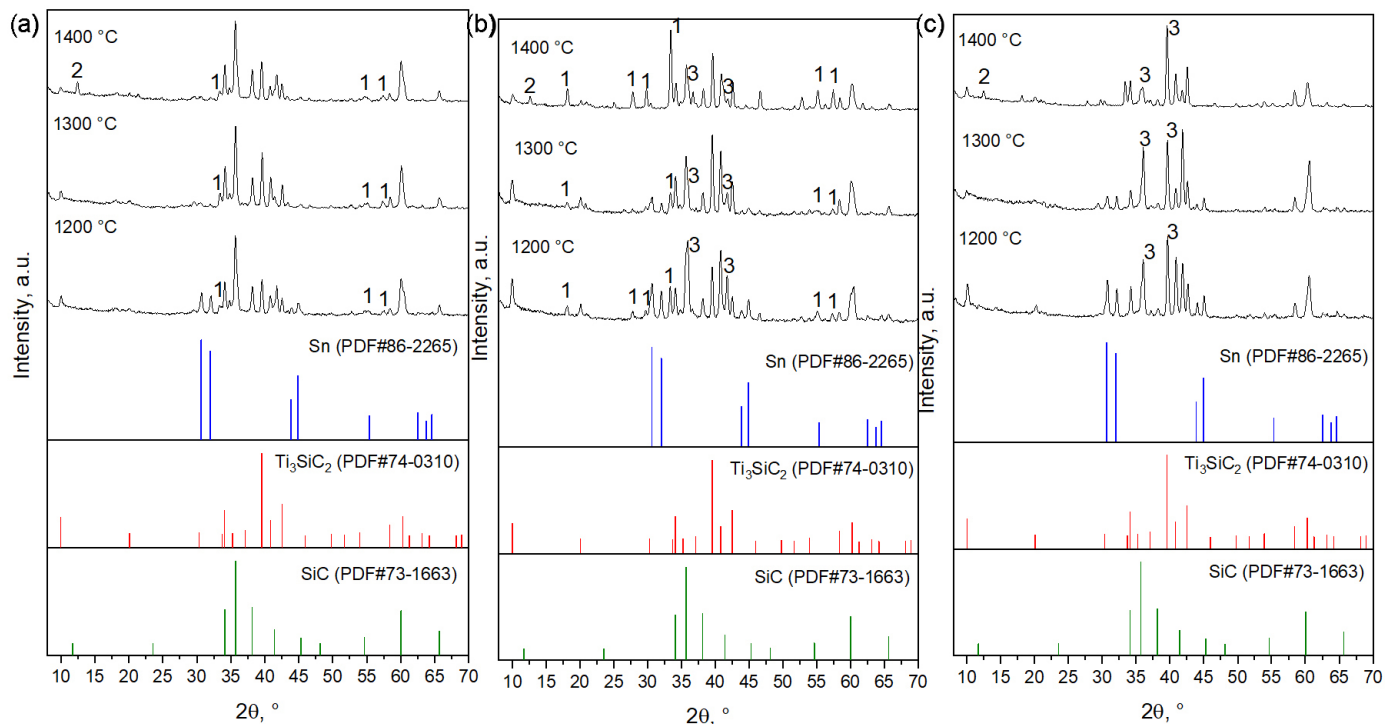


Figure 5. X-ray diffraction patterns of $\text{SiC-Ti}_3\text{AlC}_2$ ceramics, sintered in vacuum, with different Ti_3AlC_2 content: (a) 30 vol.%; (b) 50 vol.%; (c) 70 vol.%. 1— $\text{Y}_3\text{Al}_5\text{O}_{12}$ (PDF#71-0255); 2— Sn_3Ti_5 (PDF#65-3605); 3—TiC (PDF#65-8807).

Thus, ceramics are mainly represented by the Ti_3SiC_2 , TiC, and SiC phases. The decrease in the apparent density of materials (Figure 6) with an increase in the sintering temperature is mainly due to an increase in the amount of evaporated Al, which is formed during the decomposition of Ti_3AlC_2 , as well as to the formation of a difficult-to-sinter phase, Ti_3SiC_2 [47].

3.4. Sintering of $\text{TiC-Ti}_3\text{AlC}_2$

The initial titanium carbide powder is represented by a single TiC phase (Figure 7). During the sintering of $\text{TiC-Ti}_3\text{AlC}_2$ ceramics in a vacuum, the formation of aluminum oxide in the form of corundum is observed; most likely, this is due to the contact reaction of the samples with the aluminum oxide substrate. In a vacuum, as mentioned above, the thermal dissociation of Ti_3AlC_2 occurs with the formation of TiC, as well as active Ti and Al, which probably exhibit a mineralizing effect with respect to the interaction between TiC and Al_2O_3 [48], which also explains the presence of the TiO and TiO_2 phases in the compositions of composites with an increase in temperature up to 1400 °C.

TiC and Ti_3AlC_2 are probably inert components with respect to each other at a temperature of 1200 °C; however, with an increase in the sintering temperature, and, accordingly, the degree of dissociation of Ti_3AlC_2 , intermetallic compounds are formed in the Ti-Al system of various compositions (Figure 8). The formation of intermetallic and oxide com-

pounds contributes to an increase in the density of ceramics with increasing temperature (Figure 9), since they are more sinterable than TiC and Ti_3AlC_2 .

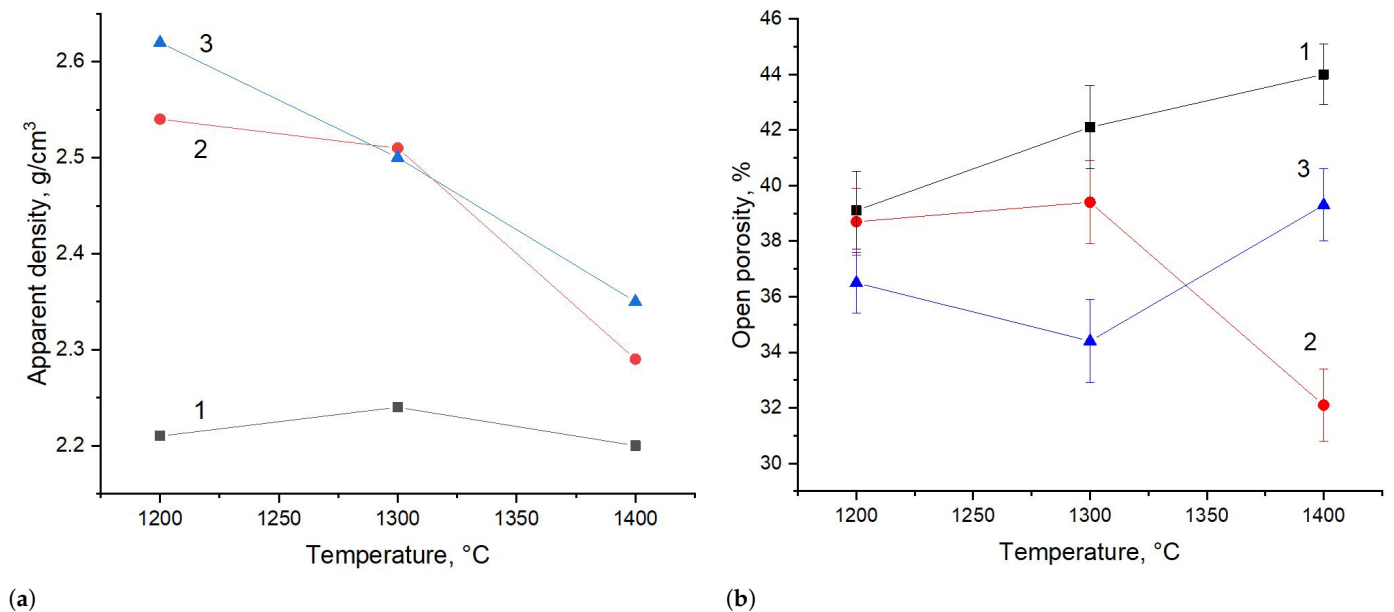


Figure 6. Apparent density (a) and open porosity (b) of SiC- Ti_3AlC_2 ceramics against sintering temperature with different Ti_3AlC_2 content: (1) 30 vol.%; (2) 50 vol.%; (3) 70 vol.%.

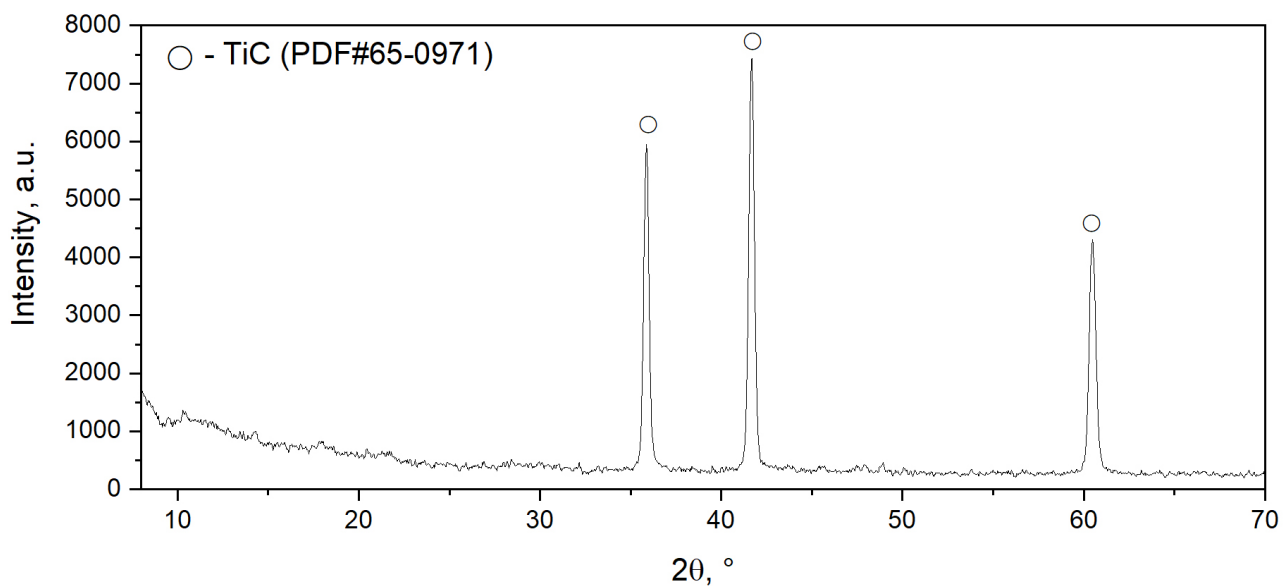


Figure 7. X-ray diffraction pattern of TiC starting powder.

Increasing the content of Ti_3AlC_2 in ceramics up to 70 vol.% significantly intensifies the processes of interaction between TiC and the corundum substrate, resulting in the formation of aluminum oxide already at a temperature of 1200 $^\circ\text{C}$. In this case, due to the high content of the MAX phase in the composition of the initial powder, the dissociation of Ti_3AlC_2 proceeds stepwise with the formation of Ti_2AlC and TiC, which is more susceptible to the contact oxidation reaction upon interaction with the substrate. The compositions of all ceramics at a temperature of 1400 $^\circ\text{C}$ include titanium oxides, mainly in the form of titanium monoxide and titanium dioxide, as well as aluminum titanides.

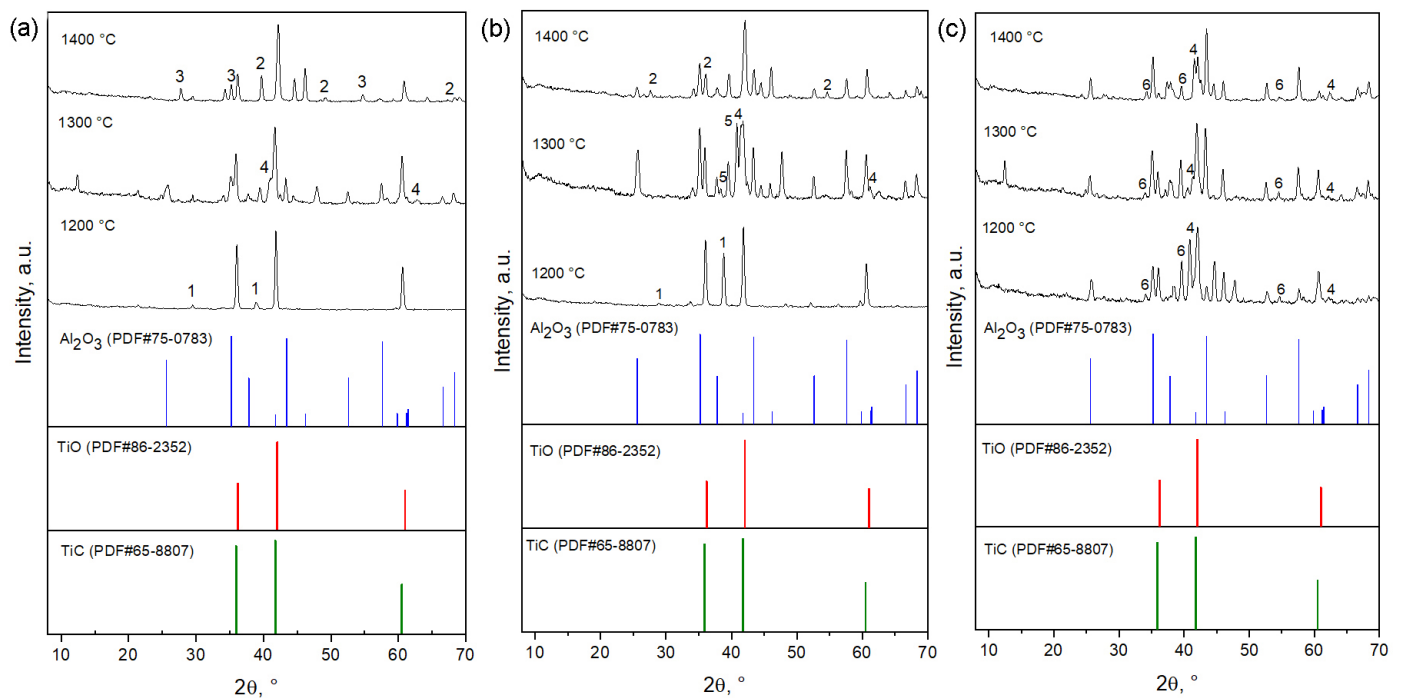


Figure 8. X-ray diffraction patterns of TiC-Ti₃AlC₂ ceramics, sintered in vacuum, with different Ti₃AlC₂ content: (a) 30 vol.%; (b) 50 vol.%; (c) 70 vol.%. 1—Ti₃AlC₂ (PDF#52-0875); 2—TiO₂ (PDF#88-1173); 3—Ti₂O (PDF#73-1582); 4—AlTi₃ (PDF#52-0859); 5—Al₆Ti₁₉ (PDF#65-8567); 6—Ti₂AlC (PDF#73-1512).

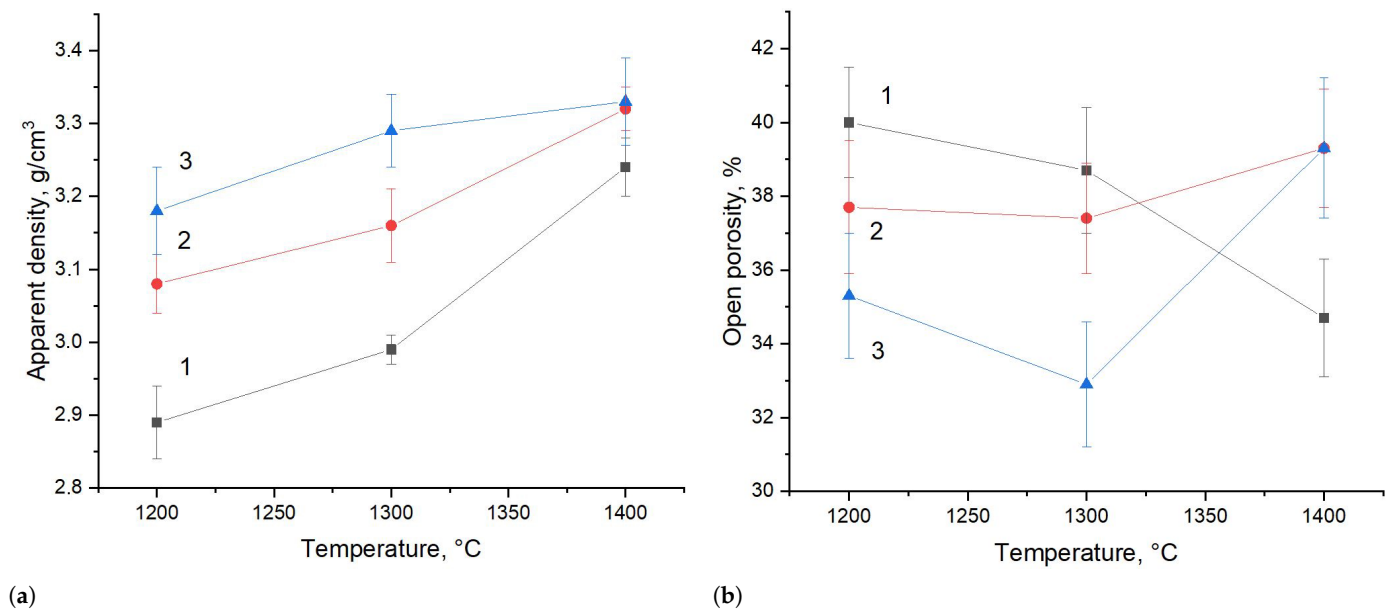


Figure 9. Apparent density (a) and open porosity (b) of TiC-Ti₃AlC₂ ceramics against sintering temperature with different Ti₃AlC₂ content: (1) 30 vol.%; (2) 50 vol.%; (3) 70 vol.%.

4. Conclusions

In the present study, samples from ceramic mixtures SiC-Ti₃AlC₂ and TiC-Ti₃AlC₂ were successfully obtained by the PIM technology with different ratios of components. It was possible to preserve the Ti₃AlC₂ MAX phase in all samples at the investigated sintering parameters. Additional heat treatment may be carried out to improve the mechanical properties of the samples.

With an increase in the sintering temperature and the content of the MAX phase, the density of the samples decreases in the SiC-Ti₃AlC₂ system and increases in the TiC-Ti₃AlC₂ system. The main reason is the various reactions occurring in these systems during the sintering process. In particular, the formation of oxide with an increase in the sintering temperature in the TiC-Ti₃AlC₂ system leads to a significant change in the phase composition and density.

During the sintering of SiC-Ti₃AlC₂ ceramics in the temperature range of 1200–1400 °C in a vacuum, the main components of the mixture interact with the formation of the Ti₃SiC₂ and TiC phases, the presence of which makes it difficult to sinter materials to a non-porous state.

For materials of the TiC-Ti₃AlC₂ system, an important feature is the chemical activity during their contact interaction with an aluminum oxide substrate, which leads to the partial oxidation of the material and the formation of titanium and aluminum oxides. The increase in the density of ceramics during sintering in a vacuum is associated with the formation of intermetallic compounds in the Al-Ti system, which provide the more intensive sintering of the ceramics.

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