JOURNAL LA MULTIAPP
VOL. 01, ISSUE 04 (007-017), 2020
DOI: 10.37899/journallamultiapp.v1i4.228

# Manufacturing Ceramic Foams at Very High Temperature by the Unconventional Process of Direct Microwave Heating

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Article Info
*Article history:*
Received 01 September 2020
Received in revised form 13 September 2020
Accepted 25 September 2020

*Keywords:*
Ceramic Foam Silicon Carbide
Direct Microwave Heating
Silica
Compressive Strength

**Abstract**

*SiC ceramic foams were manufactured by direct microwave heating up to 1520 ºC. Silicon carbide (42-68 wt.%), quartz sand as a silica supplier (20-38 wt.%), coal fly ash (12-20 wt.%) and a constant water addition of 15 wt.% were used as starting materials. The ceramic foam samples had semi-open microstructures in which neighboring cells are partially connected to each other and partially closed. Due to the very dense cellular walls and the very low cells size (below 21 µm), the compressive strength had very high values (41.3-56.5MPa), the porosity was within an average value range (52.4-57.6%) and the thermal conductivity and the apparent density had relatively high values. In energy terms, the technique of direct microwave heating was very advantageous, the specific energy consumption being very low (1.04-1.21 kWh/kg) compared to the consumptions achieved by conventional methods. The application field of SiC ceramic foams obtained by the bonding method and using silica as a bonding agent includes hot gas or molten metal filters, porous burners, catalytic supports and others. From the four tested experimental variants, it could be concluded that the optimal sample was that achieved at 1520 ºC with 68% silicon carbide, 20% quartz sand, 12% coal fly ash and 15% water addition, having the porosity of 57.6%, thermal conductivity of 0.174 W/m•K, compressive strength of 56.5 MPa and the equivalent pore size between 9-21 µ m.*

## Introduction

Silicon carbide (SiC) ceramic foams are porous materials (with porosities in a very wide range reaching even very high values) that combine this physical property with excellent mechanical, chemical and thermal shock resistances. These simultaneous characteristics of ceramic foams are very suitable for thermal and acoustic insulating materials, high- temperature structural materials, catalytic supports, reinforcement of composites, hot gas, molten metal and water filters, porous burners, solar receivers, fusion reactors, etc (Eom et al., 2013; Mao, 2017; Saxena & Saxena, 2015; Mollicone et al., 2014; Werschy et al., 2011). Several methods of manufacturing SiC ceramic foam have been experimented in recent decades, being used



*ISSN: 2716-3865 (Print), 2721-1290 (Online)*

various types of raw materials (natural or synthetic). The known techniques are: partially sintering, replica, sacrificial template, direct foaming and bonding method (Eom et al., 2013; Mao, 2017). The most common technique for making ceramic foams is the polymeric sponge replication method, which facilitates obtaining ceramics with reticulated structure starting from polymeric sponge with open pores immersed in a ceramic suspension. After drying and pyrolysis, the material is sintered at high temperature. By the direct foaming method, ceramic foams are obtained due to the air incorporation in a suspension or a liquid, inside which air bubbles are formed and kept. The consolidated foams are sintered at high temperature. Generally, the foamed products have closed cells or partially closed (Mao, 2017). The sacrificial template method is applied by gel-casting using sacrificial polymeric spheres as the template. According to Zhang et al. (2019) the porous alumina ceramic obtained by this method has microstructural uniformity and a high density of cell walls, which leads to high mechanical strength (28MPa). By controlling the sintering temperature, the proportion of the solid load of the ceramic suspension and the size of the polymeric spheres, the compressive strength of the porous ceramic material can increase. The partial sintering is the most conventional method of making porous ceramics. Sintering combined with "in-situ" chemical synthesis lead to a very homogeneous porous structure with extremely small pore sizes (below 10 μm). The mechanical characteristics of the porous ceramics reach high values, which cannot be obtained not even in dense materials (Ohji & Fukushima, 2013).

According to the literature Eom et al. (2013) the bonding method uses several materials as bonding agents, experimentally tested: mullite (3Al2O3•2SiO2), silica (SiO2), silicon carbide (SiC), silicon nitride (Si3N4), alkali (Na2O), cordierite (2MgO•2Al2O3•5SiO2), silicon (Si), silicon oxycarbide (SiOC) and fritted phases. Generally, ceramic structures with open cells are obtained and the porosity values vary widely (17-87%). The bonding methods are cheap, with processing temperatures between 800-1550 ºC depending on the nature of the bonding agents, significantly lower compared to the other methods described above characterized by values of at least 1500 ºC reaching even above 2000 ºC. The SiC porous ceramics have variable porosity between 15-60%. Using a geopolymer as the bonding phase, the manufacture of the ceramic foam with alkali as a bonding agent can be performed at room temperature and the porosity is very high (78-87%) (Eom et al., 2013). The ceramic foams have structures composed of solid struts or walls and empty cells surrounded by them. Most often, the structure of ceramic foams is semi-open, in which neighboring cells are partially connected to each other and partially isolated (Mao, 2017). Referring to the use of silica (SiO2) in a silicon carbide powder corresponding to the bonding method, by firing at high temperature (between 1100-1400 ºC) in the oxidizing atmosphere of the oven, SiO2 binds together the fine particles of silicon carbide leading to the production of a ceramic foam with very high mechanical strength due to the uniform distribution of very fine pores (below 30 μm) and dense struts or walls existing into the porous ceramic. According to Eom et al. (2013) in the temperature range mentioned above the porosity decreases with increasing the temperature. It is also favorably influenced by a very fine granulation of silicon carbide powder.

Because the starting material is silicon carbide, the sintering temperature of the material mixture is very high due to its strong covalent bonds. It should be noted that all high



*ISSN: 2716-3865 (Print), 2721-1290 (Online)*

temperature sintering processes corresponding to the manufacturing methods mentioned above are performed by conventional heating techniques requiring very high energy consumption which at least theoretically (in the absence of information from the literature) could exceed 5 kWh/kg for a heat treatment of over 1500 ºC. A very energy efficient solution could be the use of microwave heating by direct irradiation of the aluminosilicate raw material with electromagnetic waves. Worldwide, this fast, "clean" and economical technique is not used especially in processes that occur at high temperatures. Although the microwaves have been known for more than 70 years, their application has been limited to drying and low-temperature heating processes of solid or liquid materials. Only in the last two decades, it has been found experimentally that several types of materials (ceramics, organics, polymers, metals, glass, etc.) are suitable for efficient microwave heating, but the application of this technique is still in various experimental stages (Kharissova et al., 2010). Applied research of microwave heating of silicate waste (especially glass) for the production of cellular glass was carried out in the last four years in the company Daily Sourcing & Research Bucharest. The results showed that glass-based waste is not suitable for the direct heating, mixed heating techniques being adopted (direct and indirect) (Axinte et al., 2019). But the use of aluminosilicate materials waste, especially clay and coal ash, has allowed to reach high temperatures with low energy consumption by direct microwave heating, without affecting the internal structure of the material (Paunescu et al., 2020).

## Methods

The method adopted for the experimental manufacture in microwave field of SiC ceramic foam was the bonding method. In the particular case of ceramic foam obtained by bonding SiC particles with $SiO_2$ as a result of the reaction between SiC and $SiO_2$ at 1270-1430 ºC (Pultz & Hertl, 1966), products with high porosity are obtained containing open pores in an intercommunication structure and having a high mechanical strength.

The reaction leading the SiC ceramic foam manufacturing process between quartz sand silica and silicon carbide takes place at over 1270 ºC. The reaction products: silicon, silicon monoxide and carbon monoxide remain in the adsorbed state (being noted by the index "ad") on the surface of fine silicon carbide particles. The desorption of the reaction products on the surface of silicon carbide and their conversion into gases are determinant for the reaction rate. The process is characterized by the high pressure value of SiO + CO gaseous mixture on the solid interface between SiC and $SiO_2$.

The reaction between SiC and $SiO_2$ has two stages:

$$2SiC + SiO_2 = 3Si_{ad} + 2CO_{ad} \quad (1)$$

$$2Si_{ad} + SiO_2 = 2SiO_{ad} \quad (2)$$

The reaction in its first stage (1) produces silicon, which subsequently volatilizes at 1412 ºC. According to Ferguson & Nuth (2012) being removed. The evaporation of silicon monoxide resulting from reaction (2) occurs in the temperature range 1160-1335 ºC. (Lourenco et al., 2010) and the silicon dioxide volatilization occurs at over 1600 ºC (Ferguson & Nuth,



*ISSN: 2716-3865 (Print), 2721-1290 (Online)*

2012). The experimental process of manufacturing SiC ceramic foam was performed in the Daily Sourcing & Research company on a 0.8 kW-microwave oven of the type used in the household to prepare the food, adapted for operation at very high temperatures (Figure 1a). Because the microwave heating process is initiated in the core of the material, where the highest temperature develops the heat being propagated from the inside to the peripheral areas, it is important to thermally protect the hot material with several ceramic fiber mattresses resistant to 1600 ºC (Figure 1b). In this way, the tin walls of the oven are required at acceptable temperatures (below 80 ºC), even if the temperature of the hot material reaches 1600 ºC.

The pressed powder mixture was placed on a thick bed of ceramic fiber mattresses at the base of the oven. Also, the upper area of the sample wrapped in ceramic fiber was protected with mattresses of the same material provided with a hole of 30 mm diameter. The hole in the upper mattresses was positioned on the same vertical axis with a similar hole in the upper wall of the oven, through which a radiation pyrometer mounted above the oven at about 400 mm can measure the temperature of the heated material. The distribution of the microwave field was performed by a single waveguide provided in one of the side walls of the oven. The microwave power was kept constant during the heating process.

(a) (b)

Figure 1. Experimental Microwave Equipment

a = 0.8 kW-microwave oven;
b = ceramic fiber protection of the pressed powder raw material.

The materials used in the experiments performed for the manufacture of silicon carbide ceramic foam were: silicon carbide, quartz sand and coal fly ash. Silicon carbide falls into the category of ceramic materials suitable for the manufacture of ceramic foams ($Al_2O_3$, $ZrO_2$, $TiO_2$ and $SiO_2$) (Ahmad et al., 2013) having a very good resistance to high temperature. It has also been found experimentally that this material has a very high susceptibility to microwaves, being a very active absorber of the electromagnetic radiation (Axinte et al., 2020). Silicon carbide was used at a grain size below 6.3 μm without any additional processing compared to the product purchased on the market.

The quartz sand was adopted as a raw material in experiments due to its high $SiO_2$ content. The sand was ground in a ball mill several times to obtain a fine granulation. The grain size



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after sieving was below 63 μm. The chemical composition of quartz sand determined by X-ray diffraction analysis is shown in table 1.

Table 1. Chemical composition of quartz sand and coal fly ash

| **Chemical composition** | **Raw material, wt.%** | |
|---|---|---|
| | Quartz sand | Coal fly ash |
| $SiO_2$ | 87.11 | 53.1 |
| $Al_2O_3$ | 3.64 | 23.7 |
| $Fe_2O_3$ | 1.63 | 8.6 |
| CaO | 1.87 | 7.9 |
| MgO | 0.40 | 3.2 |
| $Na_2O$ | 0.56 | Total 3.5 |
| $K_2O$ | 0.89 | |
| Other oxides | 1.30 | - |
| LOI | 2.60 | - |

The coal fly ash purchased from the Paroseni Romania) thermal power station had a grain size below 250 μm. By grinding in a ball mill, the ash granulation was reduced below 80 μm for use in the ceramic foam manufacturing process. The chemical composition shown in Table 1 and the particle size make the coal ash suitable for direct incorporation into a ceramic powder without prior processing. According to the literature (Yao et al. 2015; Kolberg & Roemer, 2001) the coal ash can partially substitute kaolin, feldspar and quartz. Although in qualitative terms $Fe_2O_3$ is a contaminant for ceramic foam, its presence in the starting mixture is beneficial being microwave susceptible at room temperature and ensuring the start of the high efficiency microwave heating process at ambient temperature (Knox & Copler, 1997).

In order to experiment the manufacture of silicon carbide ceramic foam in the microwave field, four compositional variants were adopted containing silicon carbide (between 42-68 wt.%), quartz sand (between 20-38 wt.%), coal fly ash (between 12-20 wt.%) and a constant water addition (15 wt.%) (Table 2). The limits of proportions of the mixture components were established based on the own previous experimental results (Axinte et al., 2020).

Table 2. Experimental variants for producing the ceramic foam

| **Variant** | **Silicon carbide wt.%** | **Quartz sand wt.%** | **Coal fly ash wt.%** | **Water addition wt.%** |
|---|---|---|---|---|
| 1 | 42 | 38 | 20 | 15 |
| 2 | 50 | 35 | 15 | 15 |
| 3 | 56 | 25 | 19 | 15 |
| 4 | 68 | 20 | 12 | 15 |

## Result and Discussion

As mentioned above, the four experimental variants were tested in the 0.8 kW-microwave oven, the samples being prepared by dry mixing and pressing the wet powder material. In all four cases the amount of dry material was 495 g and that of wet material was 569 g. Table 3 presents the main functional parameters of the manufacturing process of ceramic foams.



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Table 3. Functional parameters of the manufacturing process of SiC ceramic foam

| Parameter | Variant 1 | Variant 2 | Variant 3 | Variant 4 |
|---|---|---|---|---|
| Dry/wet raw material amount, g | 495/ 569 | 495/ 569 | 495/569 | 495/ 569 |
| Sintering temperature, ºC | 1455 | 1475 | 1500 | 1520 |
| Heating duration, min | 43 | 45 | 48 | 50 |
| Average rate, ºC/min<br>-heating<br>-cooling | <br>33.4<br>6.5 | <br>32.3<br>6.6 | <br>30.8<br>6.1 | <br>30.0<br>7.0 |
| Ceramic foam amount, g | 483 | 482 | 480 | 481 |
| Specific energy consumption, kWh/kg | 1.04 | 1.09 | 1.17 | 1.21 |

According to the data in table 3, the required temperature for sintering and foaming the silicon carbide ceramic samples reached high values (between 1455-1520 ºC), the maximum temperature corresponding to variant 4 which contains the highest proportion of SiC (68 wt.%). Due to the excellent efficiency of the direct microwave heating process and the high ability to absorb microwaves of the components of the raw material mixture, the heating rate reached very high values (between 30.0-33.4 ºC/min) and the process duration was very short (43-50 min) despite the very high level of the thermal regime. The energy efficiency of the microwave heating process involved very low values of the specific energy consumption (1.04-1.21 kWh/kg), at the level of conventional industrial processes for the manufacture of glass foam at much lower temperatures of 800-1000 ºC.

The physical, mechanical and microstructural characteristics of SiC ceramic foam samples were determined by common methods described in previous papers (Paunescu et al., 2020). The apparent density was measured by the gravimetric method (Manual, 1999). The porosity was calculated by the method of comparing the true and apparent density (Anovitz & Cole, 2005). The compressive strength was determined using a Stable Micro Systems TA XT Plus Texture Analyzer and the thermal conductivity by the guarded-comparative- longitudinal heat flow (ASTM E1225-04 standard). The water absorption was measured by the water immersion method (ASTM D570 standard). The samples microstructure was examined with a Smartphone Digital Microscope. The main physical, mechanical and microstructural characteristics of the ceramic foam samples are presented in table 4.

Table 4. Physical, mechanical and microstructural characteristics of the ceramic foam samples

| Var. | Apparent density g/cm$^3$ | Porosity % | Thermal conductivity W/m·K | Compressive strength MPa | Water absorption % | Pore size M m |
|---|---|---|---|---|---|---|
| 1 | 0.95 | 52.4 | 0.186 | 44.8 | 6.5 | 1-8 |
| 2 | 0.92 | 54.0 | 0.180 | 41.3 | 8.0 | 4-12 |

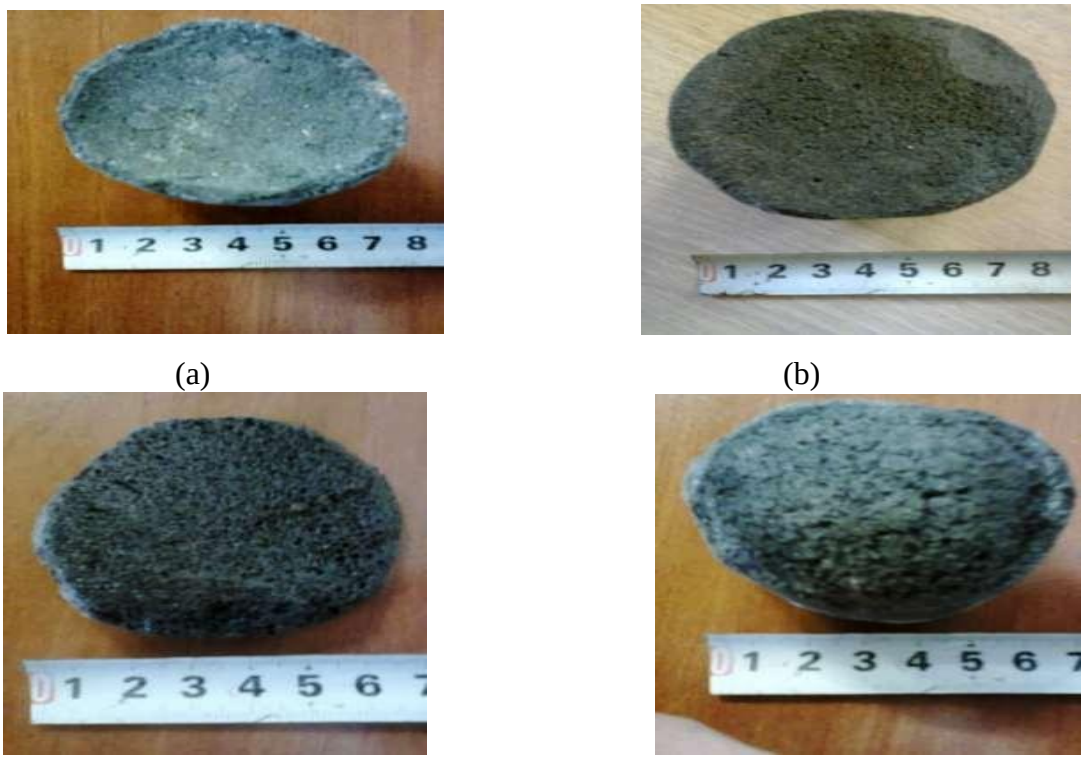


*ISSN: 2716-3865 (Print), 2721-1290 (Online)*

| | | | | | | |
|---|---|---|---|---|---|---|
| 3 | 0.89 | 55.5 | 0.178 | 42.0 | 6.8 | 7-15 |
| 4 | 0.85 | 57.6 | 0.174 | 56.5 | 9.6 | 9-21 |

Analyzing the data in Table 4, it should be noted firstly the high values of compressive strength (41.3-56.5 MPa) and the very low pore size (below 21 μm). The porosity of the ceramic foam samples had values at an average level (52.4-57.6%) compared to samples obtained by bonding methods for producing SiC ceramic foam (Eom et al., 2013). Water absorption was moderate (below 9.6%), as a general characteristic of foams made from aluminosilicate materials (Paunescu et al., 2019).

Pictures of the four samples of silicon carbide ceramic foam are presented in Figure 2 and microstructural images of these samples are shown in figure 3.



(a) (b)

(c) (d)

Figure 2. Pictures of the appearance of ceramic foam samples

a = sample 1, sintered at 1455 ºC;

b = sample 2, sintered at 1475 ºC;

c = sample 3, sintered at 1500 ºC;

d = sample 4, sintered at 1520 ºC



*ISSN: 2716-3865 (Print), 2721-1290 (Online)*

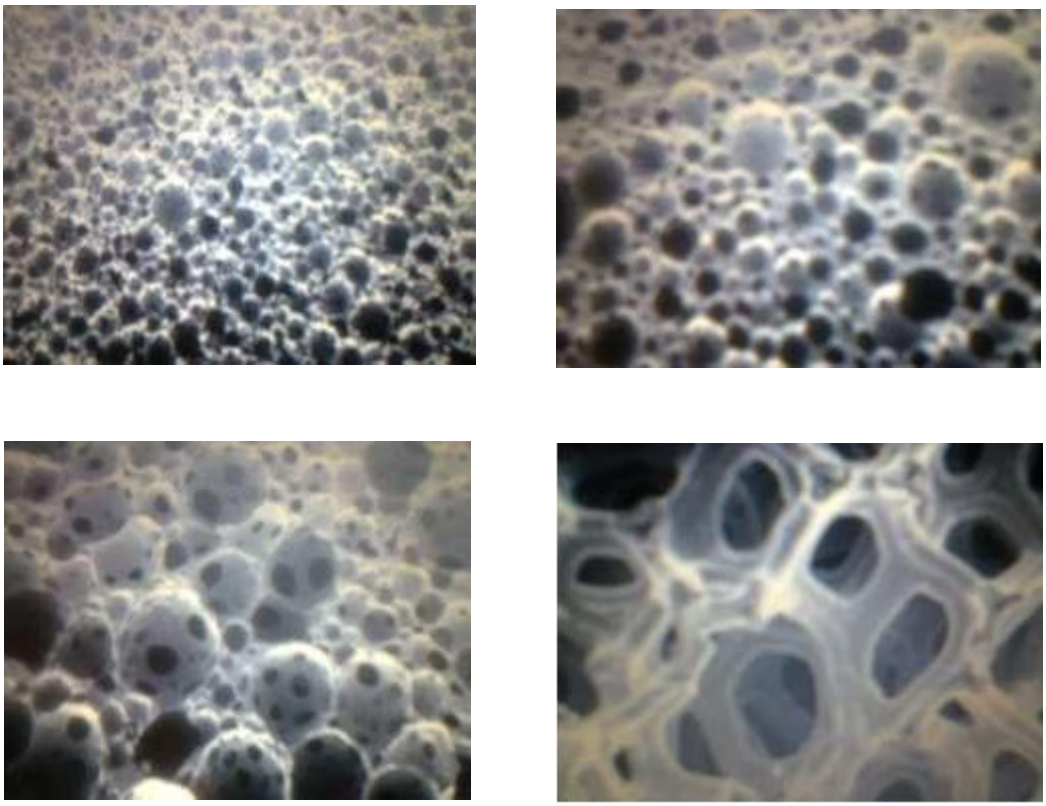


Figure 3. Microstructural images of the ceramic samples

Examining the images in figure 3, it can be observed that the samples microstructure changes significantly with the increase of the sintering/foaming temperature. Thus, sample 1 has a very fine microstructure with mostly closed cells, while samples 2 and 3 have microstructures characterized by the existence of semi-open cells, which in the case of sample 3 clearly become the majority compared to closed cells. The effects of these microstructural changes are increasing the porosity value of the samples and reducing the value of their compressive strength. At the maximum sintering temperature reached (1520 ºC) corresponding to sample 4, the existence of a predominantly open structure is obvious. The maximum equivalent diameter of pores was 21 μm and the porosity reached 57.6%. However, the compressive strength did not decrease compared to samples 1-3, but increased significantly to 56.5 MPa due to the structure with dense walls of the silicon carbide porous ceramic.

Considering the field of application of silicon carbide ceramic foams obtained by bonding technique and using silica as a bonding agent (hot gas or molten metal filters, porous burners, catalytic supports, etc.), the high compressive strength, predominantly open porosity and resistance to high temperature are required characteristics of this type of ceramic material. From the four tested experimental variants, it can be concluded that sample 4 meets the best physical, mechanical and microstructural characteristics (porosity of 57.6%, thermal conductivity of 0.174 W/m·K, compressive strength of 56.5 MPa, sintering temperature of 1520 ºC, equivalent diameter of open cells between 9-21 μm) being considered as the optimal



*ISSN: 2716-3865 (Print), 2721-1290 (Online)*

sample. Furthermore, the manufacture of this type of ceramic foam by the unconventional technique of microwave use requires a very low energy consumption (1.21 kWh/kg corresponding to sample 4), well below the theoretical consumption of its production by conventional heating methods (at least 5 kWh/kg, calculated in the absence of any information in the literature).

## Conclusion

The objective of the research was the manufacture of a silicon carbide ceramic foam by the bonding method using silica as a bonding agent in conditions of a high energy efficiency due to the application of the unconventional microwave heating technique. Based on the chemical reaction between silicon carbide and silica provided by the quartz sand, the manufacturing process occurred at very high temperatures (1455-1520 ºC) depending on the weight ratios of the components of the pressed powder mixture (42-68% silicon carbide, 20-38% quartz sand,

12-20% coal fly ash). The microwave heating has been facilitated by the existence in the load of some chemical compounds with high microwave susceptibility (SiC, Na2O, K2O) or other compounds (e.g. Fe2O3) with microwave susceptibility at room temperature, helping the heating with maximum efficiency starting even from the ambient temperature. Also, the technique of direct microwave heating, the selective and volumetric character of this heating mode, allowed to reach very high heating rates of over 30 ºC/min, short durations (43-50 min) and very low specific energy consumption (1.04-1.21 kWh/kg).

The ceramic foam sample with the highest proportion of silicon carbide in the powder mixture (68%) that reached the sintering temperature of 1520 ºC had the best physical and mechanical characteristics (porosity of 57.6%, thermal conductivity of 0.174 W/m·K, compressive strength of 56.5 MPa) as well as the most suitable microstructure (semi-open, with the equivalent pore size between 9-21 μm). The optimal experimental variant meets the requirements of porous ceramics used as hot gas or molten metal filters, porous burners, catalytic supports etc.

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*ISSN: 2716-3865 (Print), 2721-1290 (Online)*