Nonconventional Technologies Review
Romania, June, 2020

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SILICON CARBIDE CERAMIC FOAM PRODUCED BY DIRECT MICROWAVE HEATING

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ABSTRACT: The paper presents results of the research of manufacturing silicon carbide ceramic foam applying the microwave energy as a power source for the heat treatment at very high temperature (up to 1610 ºC). On the other hand, aluminosilicate waste (clay recovered from recycled brick from building demolition as well as coal fly ash) as silica suppliers, has been used to replace some materials such as quartz or sand, with very high silica content. The obtained product had a very high compressive strength (up to 58 MPa), but the porosity had lower values than those obtained from the commonly used raw material. The most important characteristic of the ceramic foam obtained by the nonconventional method is the extremely low specific energy consumption (maximum 1.42 kWh / kg) required for sintering the raw material at very high temperature, with at least 50% lower than the values obtained by the conventional methods.

KEYWORDS: silicon carbide ceramic foam, microwave, silica, compressive strength, specific energy consumption.

1. INTRODUCTION

An unique simultaneous combination of the physical and mechanical characteristics of silicon carbide ceramic foams (low density, porosity that can reach very high values, excellent mechanical, chemical and thermal shock resistances, etc.) recommends this material as being suitable for a wide variety of industrial applications as filters (for molten metals, hot gases, corrosive substances, water), membrane supports, thermal and acoustic insulators, catalyst support, high-temperature structural materials, porous burners, solar receivers, fusion reactors, etc. [1-6]. Numerous ways of producing silicon carbide ceramic foam have been tested, involving a large number of raw materials (natural or synthetic). According to [1, 7, 8], the following main ways are known and used: partially sintering, replica, sacrificial template, direct foaming and bonding methods. All these methods allow to be obtained porous structures with open cells, the pore size varying in a very wide range: between 0.1 μm and 5 mm. Open pores are important for filtration or separation applications. The porosity of the ceramic foam has values between 15-97%, depending on the method type, and the nature of the raw material. The first four methods mentioned above require very high temperatures (over 1500 ºC) exceeding even 2100 ºC [9], if the starting raw material is silicon carbide, due to its strong covalent bonds.

According to [1], the bonding method proposes more material types for bonding silicon carbide ceramic foams: mullite, silica, silicon carbide, silicon nitride, alkali, cordierite, silicon, silicon oxycarbide, etc., which were experimentally tested. From this large list of bonding agents, the authors' attention was focused only on silica, given that initially there has been the intention to use the red clay of the recycled brick waste from demolition of buildings, with a high content of silica (over 60 wt.%) [10, 11].

Several works in the literature refer to the addition of silica in the silicon carbide compact powder and firing at high temperature in the unprotected atmosphere of the oven. Silica binds the silicon carbide particles to each other, contributing to the formation of ceramic foams with very high mechanical strength. According to [12], a silicon carbide ceramic foam with spherical pores and high porosity was made by preparing a mixture between silicon carbide and a microporous polymer, and sintering it in an unprotected atmosphere. The silica produced by oxidation binds the silicon carbide particles to each other. Depending on the content of the micropores and the sintering temperature (between 1100-1400 ºC), the porosity of the foam is between 19-77%. The flexural strength and the compressive strength, corresponding to a porosity of 40%, can reach 65 MPa and 200 MPa respectively, due to the homogeneous distribution of the small pores (below 30 µm) and the dense struts into the porous silicon carbide ceramics. Generally, the porosity decreases with increasing the temperature in the range mentioned above, and it increases with decreasing the grain size of the silicon carbide powder [1].

In another paper [13], the infiltration technique of a liquid precursor (a silica supplier) into the compact mass of silicon carbide powder was tested. The silica resulting during the pyrolysis process at low temperature, in the presence of air, can act by forming bonds between neighboring silicon carbide particles at their contact points. A process of sintering at 1300 ºC of these materials led to the obtaining of ceramic foams, in which only silicon carbide and cristobalite were detected as crystalline phases. Cristobalite significantly influenced the porosity and mechanical characteristics of the samples. At a porosity of 26%, with the pore size around 5 µm, the flexural strength had reasonable values around 48 MPa.

The paper [14] presents a technique for the production of silicon carbide ceramic foam by recrystallization at high temperature in the presence of silica. By this method, silica reacts with silicon carbide at temperatures between 1270-1430 ºC, resulting gases in silica in the liquid state. These lead to the formation of a porous structure by cooling. The ceramic foam is composed of directional and interconnecting silicon carbide crystals and numerous intercommunication pores, which are positioned between them. The crystalline phase of the foam was identified as 6H-SiC (hexagonal unit cell, wurtzite crystal structure [15]) without the presence of silicon dioxide. So, the silicon dioxide particles reacted completely with the silicon carbide particles, resulting gases (carbon monoxide) released in the mass of the sample [16]. The porosity of the ceramic foams was between 61-81% and the bending strength in the range 1.5-4,8 MPa.

Porous silicon carbide ceramics were manufactured by the same bonding process [8], the compact powder being heated into an oven with unprotected atmosphere at 1100-1400 ºC. The presence of the amorphous silicon dioxide is necessary for its crystallization during the sintering process into cristobalite. Thus, the fracture strength of the ceramic foam can be maintained to relatively high values at high temperatures. For a very low grain size of the silicon carbide powder (about 0.6 µm), the fracture strength reached 185 MPa, corresponding to a foam porosity of about 31%.

The research, whose results are presented in the present work, aimed the achievement in economic conditions of a silicon carbide ceramic foam, with open pores and high temperature resistance, by the bonding method. Unlike the commonly used manufacturing methods, the technique experimentally tested in the Romanian company Daily Sourcing & Research was based on the direct microwave heating of the powder material. This heating mode is a fast, clean and economical process, too few used industrially so far [17].

2. METHODS AND MATERIALS

2.1 Methods

From the point of view of the energy source used for the thermal treatment of the raw material in order to manufacture silicon carbide ceramic foam the adopted method is an original one. To obtain a high energy efficiency, the direct microwave heating technique has been adopted. A 0.8 kW-domestic microwave oven adapted to high thermal requirement has been used. Thus, the components of the homogenized powder mixture of materials, wetted with water, were pressed into a metal mold. After pressing, the material removed from the mold was placed on a bed made of ceramic fiber mattresses at the bottom of the microwave oven and wrapped in ceramic fiber mattresses. Also, the upper area of the thermal insulation package thus formed was covered with ceramic fiber mattresses. Both the upper metal wall of the oven and the mattresses positioned in the upper area of the package containing the pressed mixture, were provided with a Ø 30 mm hole, which allows viewing the mixture surface with a radiation pyrometer mounted above the oven at 400 mm, in its central axis. Figure 1 shows the experimental microwave equipment.

a b

Figure 1. The experimental microwave equipment
a – 0.8 kW-experimental microwave oven; b – ceramic fiber thermal protection of the pressed sample.

The use of electromagnetic waves with frequencies between 300 MHz and 300 GHz (usually 2.45 GHz) is the basis of microwave heating of materials susceptible to these waves, called dielectrics. They have very few free charge carriers. The molecules or atoms of the dielectric have a dipole motion, representing two equal and opposite electrical charges. The distortion of the electron cloud around the nonpolar molecules or atoms generates friction inside the dielectric, the energy being subsequently dissipated as heat [18]. The dielectric materials absorb in the entire volume the microwave energy and are heated very quickly [19, 20]. Theoretically, the content of microwave transparent materials (SiO_2, Al_2O_3) of the clay waste and coal fly ash should create difficulties when the heating starts at ambient temperature due to the reduced electrical conductivity [21, 22]. In fact, the presence of Fe_2O_3 in the composition of the two wastes, but, especially, the large use of silicon carbide cancels the disadvantage mentioned above, and the material is heated with maximum efficiency even at room temperature [23]. Also, the oxide content of the alkali metals (Na_2O, K_2O) in the composition of the material favors the microwave heating at low temperature due to their electrical conductivity [24].

One reason for the remarkable energy efficiency of the use of microwaves compared to the application of the conventional processes is the selective mode of heating, which allows that the thermal energy to be assimilated only by the material subjected to this process, and not by the massive construction elements of the oven [18].

The technological method adopted for the manufacture of silicon carbide ceramic foam was that of the bonding method. In the particular case of the ceramic foams obtained by bonding the silicon carbide particles with silicon dioxide, as a result of the chemical reaction between the two components at high temperatures (1270-1430 ºC), high porosity ceramic products are obtained, containing open pores of equivalent diameter below 110 μm in an intercommunicating structure, and having a very high mechanical strength. The characteristics mentioned recommend these ceramic foams as being suitable, especially, as filters (for molten metals, hot gases, corrosive materials, etc.), catalyst support, porous burners, etc.

The reaction between silica and silicon carbide occurs at over 1270 ºC. According to [16], the reaction products: silicon, silicon monoxide and carbon monoxide remain in the adsorbed state (noted below with the index "*ad*") on the surface of the silicon carbide particles. The determining stage of the reaction speed is the desorption of these products on the surface of silicon carbide and their conversion into gases. An important feature of the process is the high value of the SiO + CO pressure (gas) at the SiC/ SiO_2 interfaces (solid).

The initial reaction (1) produces silicon, which subsequently volatilizes (at 1412 ºC [25]), being removed. The evaporation of silicon monoxide, resulting from the reaction (2), occurs in the temperature range 1160-1335 ºC [26]. Silicon dioxide volatilizes at over 1600 ºC [25].

$$2SiC + SiO_2 = 3Si_{ad} + 2CO_{ad} \tag{1}$$

$$Si_{ad} + SiO_2 = 2SiO_{ad} \tag{2}$$

2.2 Materials

The materials used in the manufacturing process of silicon carbide ceramic foam by the bonding method are: silicon carbide as well as red clay from recycled brick waste and coal fly ash, due to their silica and alumina contents.

Along with alumina, zirconia, titania and silica, silicon carbide is one of the ceramic materials suitable for the manufacture of ceramic foams [7]. Extremely rare in nature, it is used in the form of powder produced by synthesis. The silicon carbide granules can be bonded together by sintering in the form of very hard ceramics. The material has a very good resistance to high temperature. In addition, silicon carbide is one of the most microwave susceptible materials, being a very good absorber of the electromagnetic radiation. It was purchased from the market with a grain size between 63-80 μm, reduced to below 40 μm by grinding in a laboratory electrical device.

The red clay from the recycled clay bricks recovered from demolition of buildings contains high proportions of silica [25, 26] (Table 1), and was used in the experiment after crushing and grinding in the laboratory device at a grain size below 180 μm. According to [27], the softening point of silica (quartz) is about 1250 ºC. Obviously, the softening point of the clay waste is lower.

Table 1. Chemical composition of the red clay brick and the coal fly ash

Chemical composition	**Raw material, wt.%**	
	Clay brick	Coal fly ash
SiO_2	60.6	53.1
Al_2O_3	19,2	23.7
CaO	2.6	7.9
MgO	2.9	3.2
Fe_2O_3	8,1	8.6
Na_2O	1.2	3.5
K_2O	3.9	
TiO_2	1.3	-

Table 1 also contains the chemical composition of coal ash, corresponding to the ash brought from Paroseni thermal power station. The grain size of this waste is below 180 μm, being reduced after grinding below 80 μm. It is known that in the process of manufacturing glass-ceramic foams with high mechanical strength is commonly used coal fly ash in fairly high proportions (generally between 10-40 wt.%), contributing to a significant increase of density and compression strength as well as a decrease of the porosity due to the increase of the viscosity of material and the obstruction of the foaming process. Implicitly, the temperature of the sintering/ foaming process is higher [28, 29].

2.3 Characterization of the samples

The silicon carbide ceramic foams experimentally obtained after the sintering processes previously described were subjected to tests for determining the physical, mechanical and structural features. The following features of materials were investigated in laboratory: apparent density, porosity, compressive strength, thermal conductivity, and crystallographic microstructure, using common methods of analysis. The samples features were investigated in laboratory in the company Daily Sourcing & Research SRL, as well as in the Department of Applied Chemistry and Materials Science of the University "Politehnica" of Bucharest and Metallurgical Research Institute of Bucharest. Thus, the apparent density was determined by the gravimetric method [30]. The porosity was calculated by the comparison method of true and apparent densities of the material, experimentally measured [31]. The compressive strength was measured with an uniaxial press, and the thermal conductivity was determined by the guarded-comparative-longitudinal heat flow technique, according to ASTM E 1225-04. To investigate the crystallographic microstructure of the ceramic foams, X-ray diffraction (XRD) was used according to the standard EN 13925 – 2: 2003 using a X-ray diffractometer Bruker-AXS D8 Advance with CuK α radiation.

3. RESULTS AND DISCUSSION

3.1 Results

The process of sintering/ foaming the powder mixture of raw material composed of silicon carbide as the main material of the process, the red clay from recycled brick waste and the coal fly ash, as mainly silica suppliers, and the water addition as a binder, was carried out in the 0.8 kW-microwave oven, adapted for operation at very high temperature, described above. According to the adopted experimentation methodology, four compositional variants were used, the silicon carbide successively representing 40, 50, 65 and 75 wt.% of the amount of dry raw material. Correspondingly, the clay waste had ratios of 40, 40, 23 and 15 wt.%, respectively, and the coal fly ash represented 20, 10, 12 and 10 wt.%. The water addition was maintained at a constant weight proportion of 17%. Table 2 shows the percentage distribution of the mixture components for each variant tested.

Table 2. Composition of the powder mixture of raw material

Variant	Silicon carbide	Red clay waste	Coal fly ash	Water addition
1	40	40	20	17
2	50	40	10	
3	65	23	12	
4	75	15	10	

The functional parameters of the sintering/ foaming process are presented in Table 3, and the results of the physical, mechanical and morphological characterization of the ceramic foam samples are shown in Table 4.

Analyzing the data in Table 3, it results firstly the high energy efficiency of microwave heating, far superior to conventional methods. The specific energy consumption values are between 1.29-1.42 kWh/ kg and the average heating speeds are very high (over 30.6 ºC/ min), under the conditions where the required maximum temperatures reach 1560-1610 ºC.

The energy requirements being met, samples of silicon carbide ceramic foam with very high compressive strengths (up to 58.0 MPa) and relatively low apparent densities (maximum 1.39 g/ cm^3) were obtained (see Table 4). The high values of mechanical strength are due to the homogeneous distribution of very low size pores (below 110 μm) with dense struts in the silicon carbide ceramic foam. It is noted that by increasing the proportion of silicon carbide (to 75%) at the expense of silica supply materials (clay and coal ash) reduced to 15% and 10% respectively, the compressive strength of ceramic foam decreases significantly (up to 47.6 MPa) compared to the sample made with 65% SiC.

Figure 2 shows pictures of the four variants of ceramic foam samples with contents of silicon carbide between 40-75 wt.%.

Table 3. The functional parameters of the sintering/ foaming process

Variant	**Raw material amount, g**		**Sintering temperature ºC**	**Heating time min**	**Average rate, ºC/ min**		**Ceramic foam amount g**	**Specific energy consumption kWh/ kg**
	Dry	Wet			Heating	Cooling		
1	490	573	1560	47	32.8	6.8	487	1.29
2	492	576	1590	50	31.4	6.6	488	1.37
3	491	574	1600	51	31.0	8.1	486	1.40
4	492	576	1610	52	30.6	7.9	489	1.42

Table 4. Physical, mechanical and morphological characteristics

Variant	**Apparent density g/ cm^3**	**Porosity %**	**Thermal conductivity W/ m · K**	**Compressive strength MPa**	**Pore size µm**
1	1.05	51.4	2.34	28.3	< 110
2	1.22	44.5	5.21	40.5	< 90
3	1.39	36.8	6.85	58.0	< 85
4	1.38	37.1	6.85	47.6	< 70

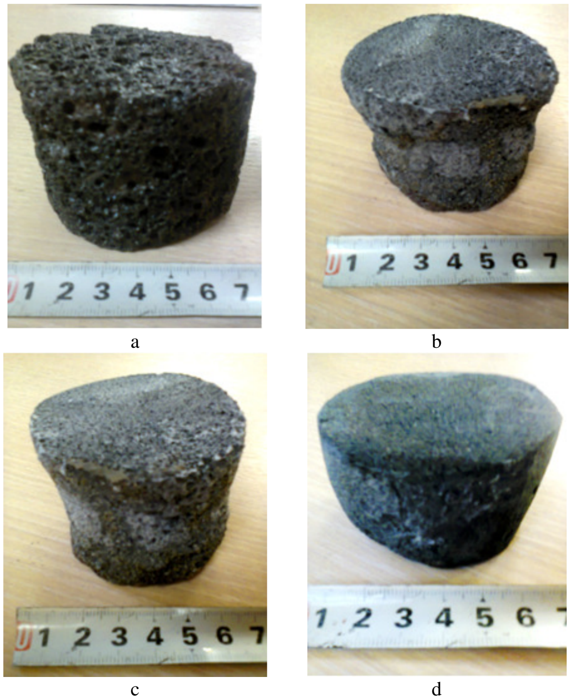

Figure 2. Pictures of the ceramic foam samples
a – sample 1; b – sample 2; c – sample 3; d – sample 4.

The images of the microstructural configuration of the samples viewed with a Smartphone Digital Microscope are shown in Figure 3.

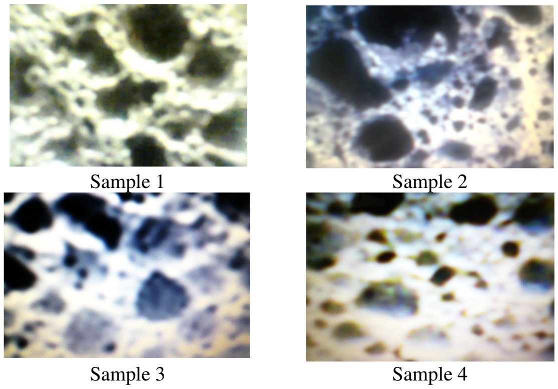

Figure 3. Microstructural configuration of the samples

The samples were subjected to the XRD analysis. In all cases, the main crystalline phase identified after the heat treatment between 1560-1610 ºC was silicon carbide (SiC) and traces of cristobalite and quartz.

3.2 Discussion

The present paper is the result of a research that involved two types of original approaches by comparison with the known techniques of manufacturing silicon carbide ceramic foam. The first, and the most important, refers to the use of the microwave energy in the heating process of the raw material at very high temperature. The second original approach is based on the use of aluminosilicate waste (clay recovered from the bricks recycling after demolition of buildings as well as fly ash recovered from coal burning in thermal power plants) as a silica supplier for its reaction with silicon carbide.

A special attention should be paid to the very low level of specific energy consumption values corresponding to the direct microwave heating technique. The maximum sintering temperature (1610 ºC) of the powder mixture containing 75% silicon carbide, 15% clay waste and 10% coal fly ash (variant 4) corresponds to a specific consumption of 1.42 kWh/ kg. An own theoretical calculation shows that the minimum required energy consumption for the sintering process at 1610 ºC is 0.74 kWh/ kg. Under the conditions where the thermal efficiency of the conventional industrial technological process is 0.40-0.50, it results that the real specific energy consumption has values between 1.48-1.85 kWh/ kg, higher up to 32%. The paper [17] considers that, generally, a microwave heating process on an industrial scale could be more effectively with up to 25% compared to a similar one carried out in a small capacity microwave oven, such as the case of the experimental oven.

From a strictly technological point of view, the use of aluminosilicate waste, silica representing between 14.4-34.9%, as a replacer for the material (quartz or sand) very rich in silica (over 96%), has led to obtaining different microstructures . Thus, the reference ceramic foam [14] consisted of directional and interconnecting silicon carbide crystals and numerous intercommunicating pores positioned between them, while the ceramic foam experimentally produced was characterized by open pores with size below 110 µm in an intercommunicating structure. The XRD analysis of the reference ceramic foam revealed the presence of SiC in the form of wurtzite (6H-SiC) as a crystalline structure, without the presence of SiO_2 [14], while the XRD analysis of the experimental foam allowed the identification as the main crystalline phase of SiC, but also traces of cristobalite and quartz, suggesting that SiO_2 did not fully react with SiC, although its mass ratio in the mixture was lower compared to that corresponding to the reference sample. Under these conditions, the porosity of the reference sample was much higher (61-81%) compared to that of the experimental sample (36.8-51.4%) and also the mechanical strength was slightly higher.

However, the physical, morphological and, especially, mechanical characteristics recommend the silicon carbide ceramic foam produced in the microwave field as a filter for hot liquid or gaseous materials.

4. CONCLUSION

Original techniques for applying microwave energy in sintering processes at very high temperatures, and for using aluminosilicate wastes (recycled clay and coal fly ash) in the manufacturing process of silicon carbide ceramic foam constituted the basic elements of the research.

The silicon carbide ceramic foam experimentally produced had physical, mechanical and morphological characteristics suitable for its using as filters for hot liquid or gaseous materials.

The main characteristic of the ceramic foam was the very high compressive strength (up to 58 MPa) despite the relatively low value of the apparent density (1.39 g/ cm^3). The ceramic foam with the highest mechanical strength was the sample 3.

The experimentally produced ceramic foam had a microstructure characterized by numerous open pores with the size below 110 µm in an intercommunicating structure.

The specific energy consumption was very low (maximum 1.42 kWh/ kg) considering the very high value of the process temperature (1610 ºC). This parameter has demonstrated the remarkable efficiency of the use of microwave energy in processes of direct heating at very high temperature.

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