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UNCONVENTIONAL MICROWAVE HEAT TREATMENT TECHNIQUE FOR PRODUCING CELLULAR GLASS BY SINTERING/FOAMING THE RECYCLED GLASS WASTE WITH SILICON CARBIDE AND COAL FLY ASH

BY

LUCIAN PAUNESCU[1,*], SORIN MIRCEA AXINTE[1,2] and FELICIA COSMULESCU[3]

[1]Daily Sourcing & Research SRL Bucharest, Romania
[2]University “Politehnica” of Bucharest,
Department of Applied Chemistry and Materials Science
[3]Cosfel Actual SRL Bucharest, Romania

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Abstract. The paper presents experimental results obtained in the microwave field manufacturing of an ecological cellular glass-ceramic using clear flat glass waste and coal fly ash as raw materials and silicon carbide as a foaming agent. The experiments have shown that under the influence of microwave irradiation, foamed products have a good microstructural homogeneity, although it is known from the literature that the flat glass is not suitable for a foaming with uniform pore distribution. The optimal sample of cellular glass-ceramic sintered at 977ºC had thermal insulation properties (apparent density of 0.42 g/cm^3 and thermal conductivity of 0.085 W/m·K) as well as high compressive strength of 2.4 MPa, being suitable for using as a thermal insulation building material.

Keywords: cellular glass-ceramic; flat glass waste; microwave heating; silicon carbide; microstructural homogeneity.

*Corresponding author; *e-mail*: lucianpaunescu16@gmail.com

1. Introduction

Recycling the glass waste has become a common practice in the world for the last 40-50 years. Initially, recycled waste was used only as a raw material in the industrial process of manufacturing the new glass allowing significant energy savings. Later, the high cost of the selecting process of glass waste by colour, determined the industrial manufacturers refocusing to use this waste for producing different types of glass foam as replacements of existing building materials.

Generally, the post-consumer packaging bottle is the most used type of glass waste (soda-lime glass) as a raw material for the glass foam manufacture, being practically, free of dangerous contaminants. Also, large quantities of glass waste are available in the world and their growth trend is obvious, the annual generation rate being very high.

One type of glass waste that exists massively in the world, although less available compared to packaging bottles, is the flat glass, mainly including window glass cullet, but also other forms of glass (sealed window units, laminated glass, mirrored glass, tinted glass, printed glass, old glass from wooden frames) (Dragoescu *et al*., 2018a). The United Kingdom produces 750,000 tons of flat glass annually, of which three quarters go into glazing products for buildings (Collection, 2008). The mass proportion of recycled flat glass in this country reaches 20-30%.

According to (Llaundis *et al.*, 2009), the flat glass waste is less interesting for the production of glass foam due to the difficulty of obtaining an adequate structural homogeneity. The literature presents various methods of improving the quality of glass foams made from flat glass waste by using oxygen suppliers (MnO_2) to intensify the foaming (Llaundis *et al*., 2009). The addition of MnO_2 improves the foaming ability of Si_3N_4 as a foaming agent, the expansion of the glass occurring at relatively low temperatures (800-850ºC) and the mechanical strength of the foam significantly increases up to 4.4 MPa.

According to the literature (Cosmulescu *et al*., 2020; Recycled glass, 2016; Geocell, 2017), the industrial use of the flat glass waste is practiced to a lesser extent compared to the post-consumer packaging bottle. The Austrian company Geocell Schaumglas currently uses a raw material mixture consisting of 90 wt.% coloured packaging bottle and 10 wt.% flat glass waste and the German company Glapor Werk Mitterteich uses the two glass waste either separately or together, the agent of foaming being predominantly glycerol.

The heating techniques used both in the industrial processes of glass foam manufacturing and in the experiments mentioned above were conventional. The Romanian company Daily Sourcing & Research, which in the last four years has focused its experimental activity on the use of the unconventional technique of microwave heating, also including testing the foaming of flat glass waste. In the work (Dragoescu *et al*., 2018b) two test groups were performed using clear flat

glass waste and successively calcium carbonate ($CaCO_3$) between 1.2-1.4 wt. % and silicon carbide (SiC) between 3.5-3.6 wt. % together with coal fly ash between 8.5-10.5 wt. %. The sintering temperatures varied from 820-833ºC (first group) to 980-995ºC (second group). The foamed products had an apparent density between 0.32-0.39 g/cm^3 and 0.32-0.42 g/cm^3 respectively, a compressive strength between 1.2-1.3 MPa and 1.4-2.1 MPa and homogeneous microstructures with pore sizes between 0.6-2 mm and 0.9-3 mm, respectively. In another paper (Dragoescu *et al*., 2018a), the flat glass waste foaming was performed with SiC as a foaming agent (between 2.5-3.5 wt. %) and coal fly ash (between 9.5-10.5 wt. %). The sintering/foaming temperature had values between 978-992ºC, the heating rate being between 13.1-13.6ºC/min. The characteristics of cellular glass-ceramics were: apparent density between 0.25-0.31 g/cm^3, compressive strength between 1.27-1.35 MPa, thermal conductivity between 0.038-0.041 W/m·K, pore size being in the range 0.5-2.5 mm. The microstructure of the samples was characterized by a very good homogeneity.

The current work aims to improve the characteristics of cellular glass-ceramic samples made from clear flat glass waste with the addition of coal fly ash and SiC as a foaming agent. The heating technique adopted by the authors was unconventional, using the 0.8 kW-microwave oven in the Daily Sourcing & Research Company described also in other previous works presented in the literature.

2. Methods and Materials

The basic method of foaming a glass-based powder mixture is to incorporate a foaming agent (solid or liquid) which releases at a high temperature a gas (or gaseous compound) into the thermally softened material with a suitable viscosity so that the gas is trapped under the shape of bubbles. By cooling after stopping the heating, the bubbles turn into a network of pores that characterizes the structure of a glass foam (Scarinci *et al*., 2005).

The use of various silicate wastes (metallurgical slag, coal fly ash, mud from zinc hydrometallurgy, different sludge types, etc.) together with glass waste and a foaming agent favors the controlled crystallization (devitrification) of the glass forming cellular glass-ceramics (Scarinci *et al*., 2005; Rawlings *et al*., 2006). Uniquely combining adequate physical, thermal and mechanical characteristics, the cellular glass-ceramics are attractive as alternative building materials to similar materials on the market.

The foaming agent adopted was SiC. By its oxidation reaction (1) in the oxidizing atmosphere of the oven, which has the most favorable conditions in thermodynamic terms at temperatures around 900ºC (Scarinci *et al*., 2005), carbon dioxide (CO_2) which contributes directly to the foaming process of glass and silicon dioxide (SiO_2) that is incorporated into the molten mass of the glass are released.

$$SiC + 2O_2 = SiO_2 + CO_2 \quad (1)$$

As noted above, the heating method adopted in experiments was the microwave irradiation. Although known for over 80 years, this technique recognized in the literature for its energy efficiency (Kharissova *et al*., 2010) has only been applied in drying or low temperature heating processes. The use of microwaves in industry is still in different experimental stages, although it has been found in the last decades that several types of materials (ceramics, organics, metals, glasses, polymers, etc.) are suitable for an efficient microwave heating (Kharissova *et al*., 2010).

Due to the very high SiO_2 content of the glass waste and to a lesser extent the coal fly ash that make up the raw material, the use of microwaves at low temperatures (up to 500ºC) should not be adequate due to the microwave transparency of SiO_2 at these temperatures. However, this disadvantage is offset by the high presence of iron oxide (Fe_2O_3) in coal ash, which, although is a contaminant for the glass foam, has a good microwave susceptibility at room temperature (Jones *et al*., 2002), the efficiency of the microwave heating being very high since the beginning of the thermal process.

The constructive solution of the experimental microwave equipment was influenced by the fact that the recycled commercial glass (soda-lime glass) is not suitable for a completely direct microwave heating, the microwave flux that irradiates the material subjected to heating tending to destroy its core structure at the foaming temperature (Paunescu *et al*., 2017). According to the literature (Jones *et al*., 2002; Kitchen *et al*., 2014), the direct microwave heating is initiated in the core of the irradiated material by converting the microwave energy into heat and its volumetrically propagating from the inside to the peripheral areas. Another feature of the direct microwave heating is the selectivity of this process avoiding the heating of other massive components of the oven. It has been experimentally found that a screen made of a microwave susceptible material based on SiC allows the intensity of the direct contact of the microwave flow with the material to be reduced to a level at which its foaming takes place properly, part of the emitted microwave field being absorbed by the thin wall of the screen (2.5-3.5 mm), converted into heat and transferred to the material by thermal radiation.

Figure 1 presents the components of the experimental microwave equipment. The used oven was a 0.8 kW-microwave oven (1) adapted for operation at high temperature (up to 1200ºC). The oven was powered by only a microwave generator, whose waveguide (8) was placed in one of the side walls of the oven. The screen mentioned above was a ceramic tube made of a SiC and Si_3N_4 mixture with an outer diameter of 125 mm, a height of 100 mm and a wall thickness of 2.5 mm (5). The wall thickness of the ceramic tube decreased from 3.5 mm (Dragoescu *et al*., 2018a; Dragoescu *et al*., 2018b) to 2.5 mm contributed

to the increase of the direct microwave heating ratio. The pressed powder mixture (3) was freely deposited on a metal plate (4) placed on a bed of ceramic fiber mattresses (7) at the base of the oven. The pressed material was protected in the inner space of the ceramic tube covered with a ceramic lid (2). A very efficient thermal insulation from the ceramic fiber mattresses (7) was used around and above the ceramic tube and lid, avoiding the loss of heat outside. The radiation pyrometer (9) mounted above the oven on a vertical central axis allowed the temperature control of the irradiated material surface, visualized through holes provided on the central axis in the upper metal wall of the oven, ceramic lid and thermal insulation of the lid.

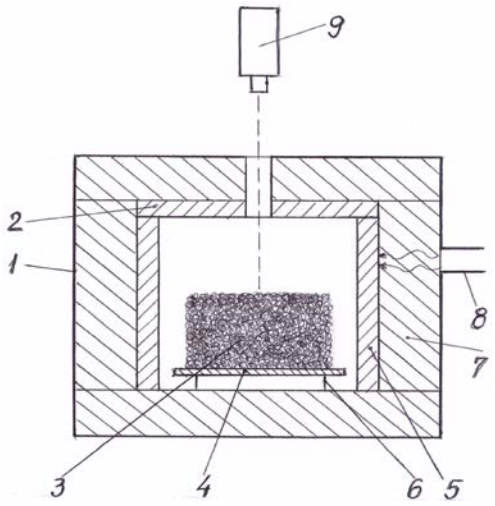


Fig. 1 – The experimental microwave equipment (constructive scheme of the equipment):
1 – microwave oven; 2 – ceramic lid; 3 – pressed powder mixture;
4 – metal plate; 5 – ceramic tube; 6 – metal support;
7 – ceramic fiber thermal insulation; 8 – waveguide; 9 – pyrometer.

The solid components of the materials mixture used in the experiments were: clear flat glass cullet recycled from the demolition or rehabilitation of buildings, coal fly ash captured in electrofilters at the Paroseni (Romania) thermal power station and silicon carbide as a foaming agent. The glass waste was cleaned by washing, dried, broken, ground in a ball mill and sieved at a grain size below 100 μm at the Romanian company Bilmetal Industries SRL Popești-Leordeni (Ilfov). The coal fly ash purchased from the Paroseni station at a grain size below 300 μm was ground in a ball mill and sieved at a grain size below 130 μm.

A typical chemical composition of the flat glass waste (Collection, 2008) and the chemical composition of coal fly ash are shown in Table 1.

Table 1
Chemical Composition of Flat Glass Cullet and Coal Fly Ash

Component	SiO_2	Al_2O_3	CaO	MgO	Na_2O	K_2O	Fe_2O_3
Clear flat glass cullet (wt. %)	70.0-73.0	max. 1.5	8.0-9.7	3.5-4.5	13.4-14.6	-	max. 0.20
Coal fly ash (wt. %)	46.5	23.7	7.9	3.2	6.0	4.1	8.6

The silicon carbide was purchased from the market with a very fine granulation below 40 μm and was used in experiments at this dimension.

According to the literature (Kolberg and Roemer, 2001), a factor favoring the absorption of microwaves and implicitly, intensifying the foaming process is the presence in the raw material composition (flat glass waste and coal fly ash) of a rather high content of alkali metal oxides (Na_2O, K_2O). Thus, the coal fly ash content (8.5-13.5 wt. %) of the starting material was considered sufficient without the need of an oxygen supplier addition to intensify the foaming.

The composition of the adopted experimental variants containing clear flat glass waste, coal fly ash as raw materials, silicon carbide as a foaming agent and water addition as a binder to facilitate the cold mixture pressing is shown in Table 2.

Table 2
Composition of the Experimental Variants

Variant	Clear flat glass waste (wt. %)	Coal fly ash (wt. %)	Silicon carbide (wt. %)	Water addition (wt. %)
1	82.5	13.5	4.0	10.0
2	85.4	11.0	3.6	10.0
3	86.5	10.0	3.5	10.5
4	87.0	9.7	3.3	10.5
5	87.7	9.2	3.1	11.0
6	88.5	8.5	3.0	10.5

3. Results and Discussion

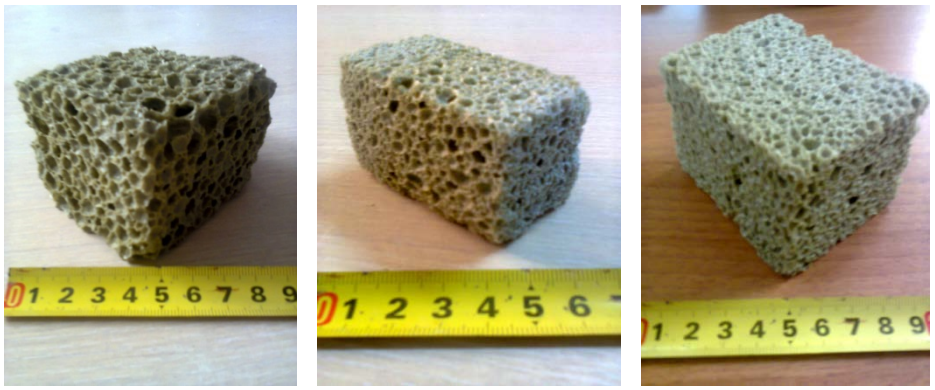
The heat treatment of the powder mixture (in six experimental variants) was performed on the 0.8 kW-microwave oven described above. The main functional parameters of the process are shown in Table 3.

Table 3
The Main Functional Parameters of the Manufacturing Process of Cellular Glass-Ceramics

Variant	Dry raw material/ glass foam amount (g)	Sintering/ foaming temperature (ºC)	Heating time (min)	Average rate, ºC/min		Index of volume growth	Specific energy consumption (kWh/kg)
				Heating	Cooling		
1	510/494	990	60	16.2	6.1	3.20	1.27
2	510/494	982	58	16.6	6.0	2.80	1.22
3	510/496	977	56	17.1	6.5	2.50	1.18
4	510/495	973	52	18.3	6.3	2.30	1.09
5	510/494	969	49	19.4	6.7	2.10	1.03
6	510/497	964	46	20.5	6.7	1.90	0.96

According to Table 3, the heating process for obtaining cellular glass-ceramics had a temperature range between 964-990ºC, the process duration varying between 46 - 60 min. The average heating rate increased from 16.2ºC/min corresponding to the heat treatment up to 990ºC (variant 1) to 20.5ºC/min corresponding to the minimum value of the process temperature of 964ºC (variant 6). The raw material expansion was maximum (3.20) in the case of variant 1 heated to 990ºC, while in the case of variant 6 heated to 964ºC the index of volume growth was much lower (1.90). Given that the level of maximum heating temperatures is high (almost 1000ºC), the values of the specific energy consumption can be considered very economical (between 0.96-1.27 kWh/kg).

Images of the appearance of the six cellular glass-ceramic samples are shown in Fig. 2. Samples heated to the highest temperatures (a and b) have the appearance of materials with coarse porosity, but with closed pores. The pore size of samples heated to lower and lower temperatures tends to diminish, so that the appearance of samples e and f is satisfactory in terms of porosity.



a *b* *c*

d *e* *f*

Fig. 2 − Overall images of cellular glass-ceramic samples:
a – sample 1 heated at 990ºC; *b* – sample 2 heated at 982ºC;
c – sample 3 heated at 977ºC; *d* – sample 4 heated at 973ºC;
e – sample 5 heated at 969ºC; *f* – sample 6 heated at 964ºC.

The cellular glass-ceramic samples were subjected to the common methods for determination of the physical, thermal, mechanical and microstructural characteristics. The apparent density was measured by the gravimetric method (Manual, 1999; Metrology, 2019). The porosity was calculated by the method of comparing the true and apparent density (Anovitz and Cole, 2005). The thermal conductivity was measured by the heat-flow meter method (ASTM E1225-04) and the compressive strength was determined using a TA.XTplus Texture Analyzer (ASTM C552-17). The water absorption was determined for a time of 24 hours by the water immersion method (ASTM D570). The microstructural configuration of the glass-ceramic foam samples was investigated with an ASONA 100X Zoom Smartphone Digital Microscope. The investigation of the crystallographic structure of the samples was performed with a X-ray diffractometer Bruker-AXS D8 Advance with CuKα radiation (EN 13925-2:2003 standard). The main physical, thermal, mechanical and microstructural characteristics of glass-ceramic foam samples are presented in Table 4.

Determining the characteristics of the cellular glass-ceramic samples highlighted the possibility of obtaining porous materials with compressive strength that can reach 4.6 MPa when using 8.5 wt. % coal fly ash and 3.0 wt. % SiC. Increasing the proportions of coal fly ash and SiC to 13.5 wt. % and 4.0 wt. % respectively, significantly reduces the compressive strength to a minimum of 1.3 MPa. The apparent density and thermal conductivity, whose values determine the thermal insulation character of the analyzed samples, are satisfactory in the range of samples 1-3. An apparent density between 0.30-0.42 g/cm^3 and a thermal conductivity between 0.063-0.085 W/m·K are considered excellent for a thermal insulation material. The compressive strength of samples 1-3 increases sharply

from 1.3 to 2.4 MPa and the value corresponding to sample 3 (2.4 MPa) in combination with the density (0.42 g/cm^3) and thermal conductivity (0.085 W/m·K) are optimal characteristics for a very good thermal insulation material for buildings.

Table 4
Main Physical, Thermal, Mechanical and Morphological Characteristics of Cellular Glass-Ceramic Samples

Variant	Apparent density (g/cm^3)	Porosity (%)	Thermal conductivity (W/m·K)	Compressive strength (MPa)	Water absorption (vol. %)	Specific energy consumption (kWh/kg)
1	0.30	85.7	0.063	1.3	3.20	1.27
2	0.36	82.9	0.075	1.8	2.80	1.22
3	0.42	80.0	0.085	2.4	2.50	1.18
4	0.48	77.1	0.099	3.2	2.30	1.09
5	0.54	74.3	0.109	4.0	2.10	1.03
6	0.56	73.3	0.114	4.6	1.90	0.96

For a deeper analysis of the characteristics of the cellular glass-ceramic samples, it was necessary to investigate their microstructural configuration presented in Fig. 3.

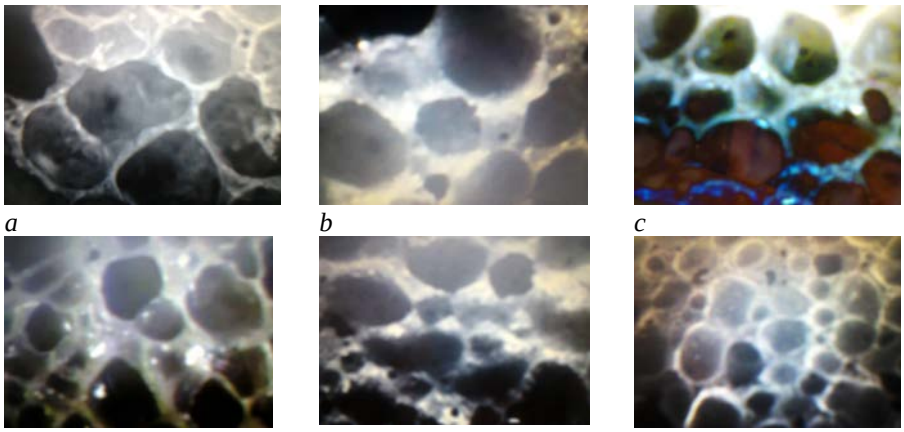


Fig. 3 − Microstructural configuration of the cellular glass-ceramic samples
a – sample 1, *b* – sample 2; *c* – sample 3; *d* – sample 4; *e* – sample 5; *f* – sample 6.

Analysing the pictures in Fig. 3, it is observed that the microstructural configuration of the samples is homogeneous with closed pores. The pore size is quite large, practically only samples 5 and 6 having normal dimensions (between 1-2.4 mm and 0.7-2 mm, respectively). The expanding intensification of the raw

material due to the influence of the high content of alkali metal oxides in the composition of flat glass waste and coal fly ash was highlighted by the formation of microstructures with large pores. According to Fig. 3, the sample 3 made of 86.5% glass waste, 10% coal fly ash and 3.5% SiC, considered optimal, has a homogeneous microstructure with pore size between 2-3.6 mm.

The XRD analysis has identified wollastonite ($CaSiO_3$) as the main crystalline phase and to a lesser extent SiC and cristobalite (SiO_2).

The method of manufacturing a cellular glass-ceramic from flat glass waste using the conversion of microwave energy into heat through predominant direct contact with the irradiated material, the raw material being glass waste and coal fly ash, showed that the conditions were met to obtain a product with a homogeneous microstructure. The intensification of the microwave absorption by the starting powder mixture due to the sufficiently high proportion of alkali metal oxides (Na_2O, K_2O) had an important role in achieving a uniform foamed structure. Given the difficulty reported in the literature of producing foamed materials with a uniform pore distribution using flat glass waste as a raw material, the ability to manufacture homogeneous microstructural products due primarily to use the microwave energy is a solution that can satisfactorily solve the inadequacy of flat glass for foaming processes.

The cellular glass-ceramic experimentally made have specific characteristics of thermal insulation materials (low apparent density, low thermal conductivity and high mechanical strength) and can successfully replace existing building materials on the market.

4. Conclusions

Six experimental variants of powder mixtures containing clear flat glass waste (between 82.5-88.5 wt. %), coal fly ash (between 8.5-13.5 wt. %), SiC (between 3.0-4.0 wt. %) as a foaming agent and water addition (between 10.0-11.0 wt. %) as a binder were sintered at 964-990ºC in a 0.8 kW-microwave oven to obtain cellular glass-ceramics.

The aim of these experiments was to manufacture a porous material with specific characteristics of a thermal insulator (low apparent density, low thermal conductivity and high compressive strength) under the conditions that it was known from the literature that the flat glass is not suitable for obtaining a homogeneous microstructure of the foamed product.

The originality of the experiments by comparison with the conventional heating methods previously used was the application of the unconventional microwave heating technique, the absorption of electromagnetic waves in the irradiated material favoring the intensification of its foaming process.

The flat glass waste and coal fly ash as an industrial by-product used as raw materials have the peculiarity of a fairly high content of alkali metal oxides,

which facilitates the absorption of microwaves by intensifying the foaming process.

The compressive strength of the cellular glass-ceramic was strongly influenced by the decrease of coal fly ash and SiC contents and implicitly, of the sintering/foaming temperature in the ranges of tested values leading to a maximum of 4.6 MPa.

The microstructural configuration of the product in all six experimental variants showed a good homogeneity, the pores having a uniform distribution.

Analyzing the physical, thermal, mechanical and microstructural characteristics, variant 3 made of 86.5 wt. % flat glass waste, 10.0 wt. % coal fly ash, 3.5 wt. % SiC and 10.5 wt. % water addition by sintering at 977ºC was adopted as the optimal sample. This sample characteristics were: apparent density of 0.42 g/cm^3, porosity of 80.0%, thermal conductivity of 0.085 W/m·K, compressive strength of 2.4 MPa, water absorption of 6.4% and pore size between 2.0-3.6 mm.

The product has the required characteristics of a thermal insulation material usable in the building construction as a replacement for existing materials on the market.

The advantages of using this product are: the low cost of the raw material consisting of a waste and an industrial by-product, the favorable ecological consequences and the low energy consumption of the unconventional manufacturing process.

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TEHNICĂ NECONVENȚIONALĂ DE TRATARE TERMICĂ CU MICROUNDE A PRODUCERII STICLEI CELULARE PRIN SINTERIZARE/SPUMARE A DEȘEULUI DE STICLĂ RECICLAT CU CARBURĂ DE SILICIU ȘI CENUȘĂ DE CĂRBUNE ZBURĂTOARE

(Rezumat)

Lucrarea prezintă rezultate experimentale obținute la fabricarea în câmp de microunde a unei vitroceramici celulare utilizând deșeu de sticlă plată incoloră și cenușă de cărbune zburătoare ca materii prime și carbură de siliciu ca agent de spumare. Experimentele au arătat că sub influența iradierii cu microunde produsele spumate au o bună omogenitate microstructurală, deși este cunoscut din literatura de specialitate că sticla plată nu este adecvată pentru spumare cu distribuție uniformă a porilor. Proba optimă de vitroceramică celulară sinterizată la 977ºC a avut proprietăți termoizolante (densitate aparentă de 0,42 g/cm^3 și conductivitate termică de 0,085 W/m·K), precum și rezistență înaltă la compresiune de 2,4 MPa, fiind adecvată pentru utilizarea ca material de construcție termoizolant.