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INFLUENCE OF THE CERAMIC SLURRY CHARACTERISTICS BY FORMING OF POROUS CERAMICS USING A REPLICATION METHOD

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Abstract: *It was found that the viscosity of the ceramic slurry highly affected the structure and physicochemical properties of the porous material. The content of refractory component in the ceramic slurry strongly affects the volume density and the structure of the samples obtained by the replica technique. At 70 mass% content of refractory component (SiC), the slurry spreads uniformly on the polymer substrates and the volume density of the samples was 0,42 g/cm^3.*

Keywords: *Porous ceramics, Polymeric sponge method, Slurry, Density.*

INTRODUCTION

The polymeric sponge method is one of the most effective ones used to synthesize open pore ceramics [1]. The porosity of these materials can reach 95% abd cell size - 200μm-3mm [2]. The cellular materials have unique structure – a network of interconnected polyhedral cells and properties like high permeability, high thermal stability and wear resistance [3, 4].

Unlike other methods where the structure and properties of the end products can be regulated by varying certain technological parameters [5], by the polymeric sponge method it is achieved by varying the pore size of the polymeric substrate and the viscosity of the ceramic slurry [6].

The aim of the present work is to study the influence of ceramic slurry viscosity on the structure and properties of the synthesized materials.

MATERIALS AND METHODS

Polyurethane substrate "Buplen S" (Italy) sized 50x50x22 was used for the experiments. The cross-section of the fibers building polyurethane structure is “X” shaped and the porosity was 10 ppi (pores per inch).

The refractory component used was silicon carbide – SiC (China), density 3200 kg/m^3, dispersity 40μm. Kaolin ($Al_2O_3.2SiO_2.2H_2O$) and bentonite ($Al_2(Si_4O_{10})(OH)_2.nH_2O$) were used as binders and gummi arabicum as adhering agent. For the preparation of ceramic slurry, the main

component (highly refractory component) and the clay materials were mixed at ratios 60:40, 70:30 and 80:20%. The compositions of the initial slurries prepared are presented in Tabl 1

Table 1. Composition of the initial slurries, mass.%

Composition Raw materials	A (HRc):(Cm) 60:40	B (HRc):(Cm) 70:30	C (HRc):(Cm) 60:20
$SiC_{40\mu m}$	36	42	48
Kaolin	18	13,5	9
Bentonite	6	4,5	3
Gummi arabicum	20	20	20
Water	20	20	20

The homogenization was carried out with a propeller mixer. The polyurethane substrates were immersed in the ceramic slurry, then pressed on a device to remove the excess slurry. The samples were dried in air for 24 h. They were sintered in electric resistance super kanthal furnace "Naber"-Germany. The decomposition and burning of the polymer at temperatures up to 600ºC was carried out at heating rate of 1º/min, with two isothermal periods: at 100ºC – 1 h and at 600ºC – 2,5 h. The final sintering temperature was 1400ºC where the isothermal period was 2,5 h and the heating rate was 6ºC/min.

RESULTS AND DISCUSSION

During the impregnation of the polymeric substrate with ceramic slurry, the latter should be viscous enough to penetrate and fill polymer cells and get uniformly deposited onto polymer surface. The slurry must have optimal viscosity under static conditions in order to stay within the polymer substrate. It means that the slurry should have optimal rheological characteristics. The main factor affecting the viscosity of the thixotropic ceramic slurry is the ratio between the highly refractory SiC particles and the clay slurry. Fig.1 shows the dependence of the amount of slurry deposited on the polymeric struts on the content of the highly refractory component.

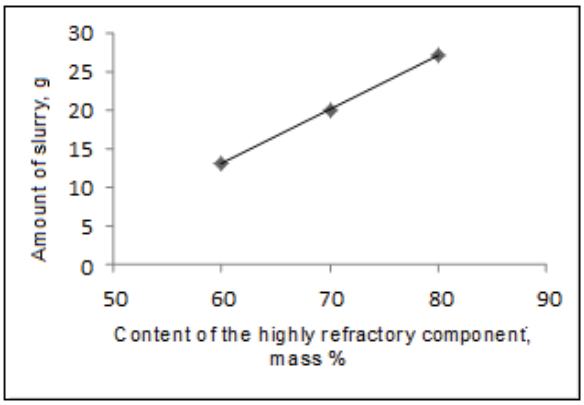


Fig.1. Dependence of the amount of deposited slurry on the content of the refractory component

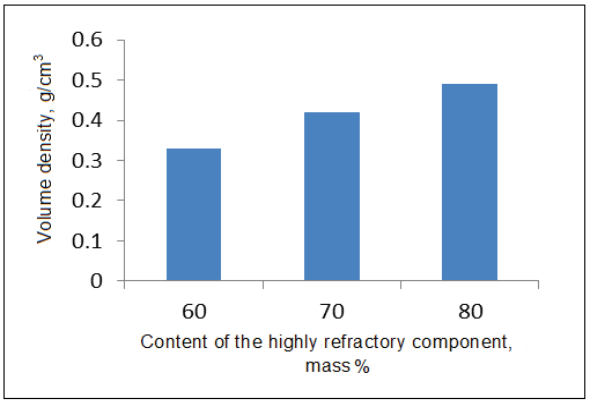


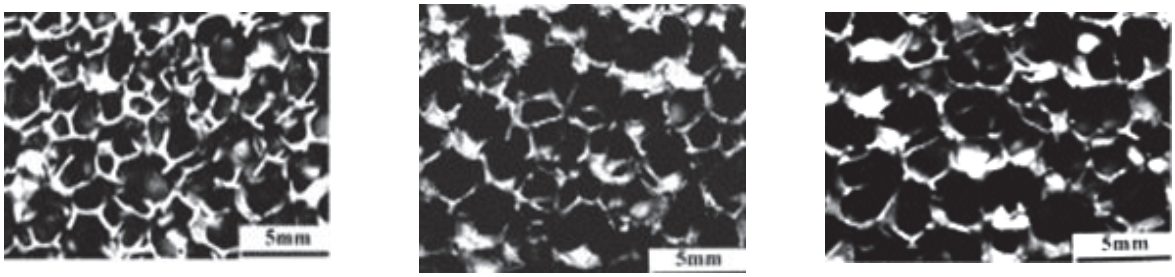
Fig.2. Dependence of the volume density on the content of the refractory component

The amount of the deposited ceramic slurry increased with the increase of the content of refractory component which results in increased viscosity. The infiltrate weight is an important variable which determines the porosity, the permeability and the strength of the ceramic synthesized. It depends on the method of substrate impregnation, ceramic slurry viscosity and the number of passes through the compressing device.

The volume density increased with the increase of the ceramic slurry viscosity (Fig.2). The samples density changed from 0,35 g/cm^3 to 0,47g/cm^3 with the increase of the content of refractory component in the ceramic slurry from 60 to 80 mass.%.

Fig.3 shows photographs of samples impregnated with different amounts of refractory component (SiC). In Fig.3b, a multitude of filled cells can be observed, as well as the thick layer formed. It indicates that the slurry can be uniformly deposited onto polymer substrates at 80 mass% content of refractory component but the layer formed on the cell struts was thick due to the high viscosity.

In Fig.3b, only a few filled cells can be observed in the lower part of the polymer (70 mass. % SiC). With the decrease of SiC content in the ceramic slurry to 60%, the ceramic layer formed was thin and non-uniform. No filled cells were observed (Fig.3a).



a) b) c)

Fig.3. Macrostructure of samples impregnated with different amounts of refractory component (SiC) a) 60mass.%; b) 70mass.%; c) 80mass.%

To study the effect of the number of passes through the compressing device on the quality of the ceramic layer deposited on the polymeric substrate, the following experiments were carried out. The samples covered with ceramic layer were rolled different number of times through the rollers. The number of passes was varied from 1 to 5. After passing, the samples were estimated by four aspects: large scale uniformity, small scale uniformity, covering the edges of the intercellular walls, presence of filled cells of cell surfaces. The dependence of the amount of slurry deposited on the polymer substrate on the number of passes through the rollers is presented in Fig.4.

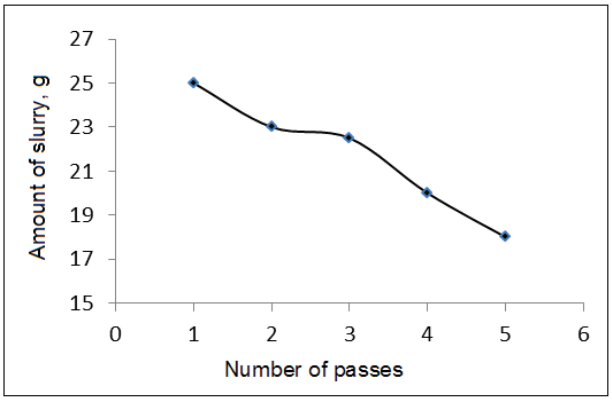


Fig.4. Dependence of the amount of slurry deposited on the polymer substrate on the number of passes through the rollers

It was found for 22 mm thick samples that the best results were obtained after 2-3 passes through the rollers giving 75% compression. Additional passes would remove smaller amounts of slurry but often redistribute the retained slurry, thus improving the small scale uniformity.

Some authors [7] suggested an improvement of the ceramic layer quality by second impregnation of the samples with slurry after the drying. The second layer is deposited aiming to remove various defects like areas covered with thinner layer during the first impregnation. In this respect, the following technique was employed. After drying in air for 5-6 h, additional amount of slurry is deposited using spray gun. The impregnated samples still preserve some elasticity and

this prevents formation of cracks by the deposition of thinner slurry. Thus, uniform thickening of the intercellular barriers is achieved and the openings of the spongy polyurethane are not filled with slurry.

The effect of the viscosity of the slurry for secondary deposition on the amount of its deposition onto the dried sample was studied (Fig.5).

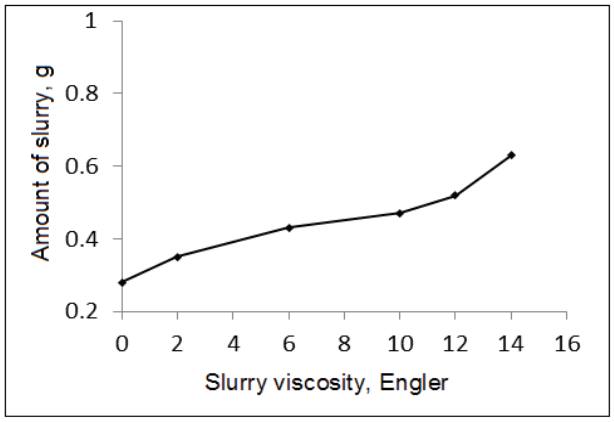
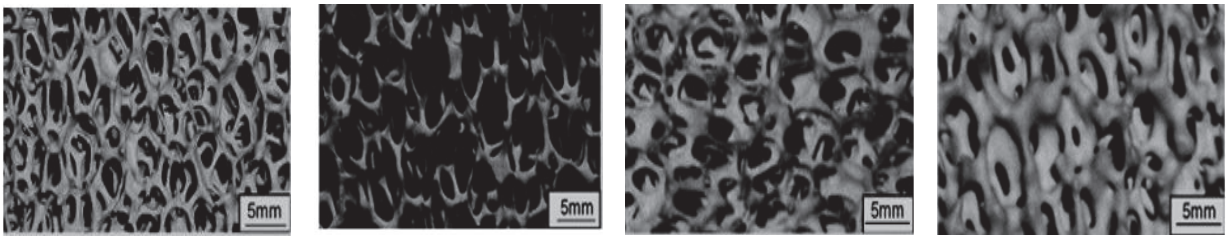


Fig.5. Dependence of the amount of deposited slurry by the second deposition on its viscosity

The value of 0 means no secondary layer. The amount of slurry deposited on the polymer increased with the increase of slurry viscosity. The amount of slurry with viscosity of 14 Engler degrees was twice more than that with viscosity of 4. The best covering was achieved with slurry viscosity 8 Engler degrees.

The structure of samples impregnated with ceramic slurries of different viscosities is shown in Fig.6.



a) b) c) d)

Fig.6. Structure of SiC ceramics :
(a) without second layer; (b) slurry with viscosity 6 Engler degrees;
(c) slurry with viscosity 8 Engler degrees; (d) slurry with viscosity 10 Engler degrees

The microstructure of SiC ceramic without deposition of second layer is shown in Fig.6. Severe flaws can be observed like side cracks on the polymeric struts, which worsen the mechanical properties, as well as areas with quite thin covering. The structure became stable after deposition of second layer of ceramic slurry. Rather thin cell walls can be observed even after deposition of slurry with viscosity 6 Engler degrees (Fig.6b). At higher viscosity (10 Engler degrees), despite the better uniformity of the layer, the thickness of the polymeric struts increased and the size of the openings decreased (Fig.6d). it can be seen that the best structure had the samples with second layer deposited with slurry viscosity 8 Engler degrees (Fig.6c).

The properties of the SiC ceramics sintered at 1400°C are presented in Table 2. The volume density of the samples without second layer was lower than that of the samples with second layer. The volume density increased also with the increase of the viscosity of the ceramic slurry used to deposit the second layer. After depositing the second layer, the compression strength of the SiC replica significantly increased.

Table 2. Properties of SiC ceramics

Viscosity of the slurry for the second layer, Engler	Volume density, g/cm^3	Compression strength, MPa
0	0,21	0,32
6	0,37	0,78
8	0,41	0,80
10	0,44	0,88
12	0,49	1,20
14	0,65	1,59

The compression strength of the samples impregnated with slurry with viscosity 6 Engler degrees (0,78 MPa) was about twice higher than that of samples without second deposition (0,32 MPa). This was due to the removal of the cracks on the cell struts. With the increase of the viscosity of the slurry for the second deposition, the volume density increased from 0,37 to 0,65 g/cm^3. The size of the openings decreased which results in increased strength. The viscosity of the second layer is very important for the improvement of the mechanical properties of the SiC ceramics. The compression strength of SiC increased from 0,78 MPa to 1,59 MPa with the increase of viscosity from 6 to 14 Engler degrees.

CONCLUSION

It was found that the content of refractory component in the ceramic slurry strongly affects the volume density and the structure of the samples obtained by the replica technique. At 70 mass% content of refractory component (SiC), the slurry spreads uniformly on the polymer substrates and the volume density of the samples was 0,42 g/cm^3. The samples for which the second layer deposited with slurry of viscosity 8 Engler degrees were found to have the best quality.

REFERENCES

[1] Woyaansky S.J., C.E.Scottetal. Processing of porous ceramics, Am. Ceram. Soc. Bull., 1992, 71 (11), 1674-1682.

[2] Studart A.R., T.Gonzenbach, E.Tervoort, L.J.Gauckler. Processing Routes to Macroporous Ceramics: A Review, J. Am. Ceram. Soc., 2006, 89 (6), 1771–1789.

[3] Sharif S.M., Z.A.Ahmad, M.R.Othman. Preparation and characterization of tubular ceramic support for use in transesterification process, Science of Sintering, 2012, 44 (2), 187-195.

[4] Jones I.R., L.L.Hench. Regeneration of trabecular bone using porous ceramics, Solid State Mater Sci., 2003, 7 (20), 301–307.

[5] Colombo P. Conventional and Novel Processing Methods for Cellular Ceramics, Philos. Trans. Roy. Soc. A, 2006, 364, 109–24.

[6] Tripkovic D., V.Radojevic, R.Aleksic. Factors affecting the microstructure of porous ceramics, J. Serb. Chem. Soc., 2006, 71 (3), 277–284.

[7] Zhu X.,D.Jiang, S.Tan, Z.Zhang. Improvement in the Strut Thickness of Reticulated Porous Ceramics, J. Am. Ceram. Soc., 2001, 84(7), 1654–1656.

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