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Production and investigation of physical-mechanical properties of syntactic carbon foams

E R Galimov[1], V L Fedyaev[1,4], N Ya Galimova[1], V M Samoylov[2], E A Danilov [2], E I Timoshchuk [2], D V Ponomareva[2], A V Nakhodnova [2] and M A Orlov[3]

[1]Kazan national research technical university named after A N Tupolev, K. Marx St. 10, Kazan, 420111 Tatarstan, Russia
[2]JSC NIIgrafit, 2 Elektrodnaya str., Moscow, 111524, Russia
[3]Bauman Moscow state technical university, Baumanskaya 2-ya str., building 1, house 5, Moscow, 105005 Russia
[4]IME - Subdivision of FIC KazanSC of RAS, Lobachevsky str. 2/3, Kazan, 420111 Tatarstan, Russia
engelgalimov@yandex.ru

Abstract. The production technology of syntactic carbon foams based on the phenol-formaldehyde binders and microspheres was developed. The influence of the composition and regime technological parameters on the change of the physical-mechanical properties of foams was investigated.

1. Introduction

Syntactic carbon foam (SCF) is a perspective material of ordered structure possessing a number of unique properties: high specific physical-mechanical characteristics, adjustable coefficients of thermal conductivity, electrical conductivity, high heat and thermal resistance, resistance to hostile environment, adjustable specific surface and etc. [1-4]. The above operational properties of syntactic carbon foams make these materials extremely interesting for application in many fields of engineering.

The paper considers the production technology of SCF based on the phenol-formaldehyde resin (PFR) filled with phenolic microspheres. This technology includes mixing of the initial components, pressing the mixture, heat treatment of the pressed workpieces, their pyro-compaction and subsequent mechanical processing for making test specimens. Investigations of the physical-mechanical properties of the SCF were carried out on the prismatic samples with dimensions 10×10×20 mm. Microscopic studies were performed by the scanning electron microscopy method on fresh cleavages of destroyed samples using a Hitachi TM3000 (figure 1).

2. Body text

The hollow microspheres based on the PFR with a wall thickness of 1-2 μm and characteristic defects in the form of cavities were used as filler. According to the laser diffraction data (Fritsch Analysette 22 Compact), the average outer diameter of the microspheres was 34 μm.

The novolak type PFR brand SF-012A (СФ-012А) was the binding agent. The filler content in the SCF was varied between 33-85 wt. %. The mixing was carried out by a liquid-phase method in a three-necked glass reactor with a reflux condenser. The filler and the binder material were placed in a reactor a solvent (acetone) was added in an amount of 10 ml per 1 g of the binder. The mixture was heated with stirring to boiling, and then it was boiled under reflux for 90 minutes. From this

composition, the solvent was removed by distillation under vacuum. The reaction mixture was transferred in portions of 200-400 ml to a 500-1000 ml round-bottomed flask and evaporated on a rotary evaporator IR-2M. Distillation conditions: residual pressure 36-39 kPa, temperature 40-45 °C.

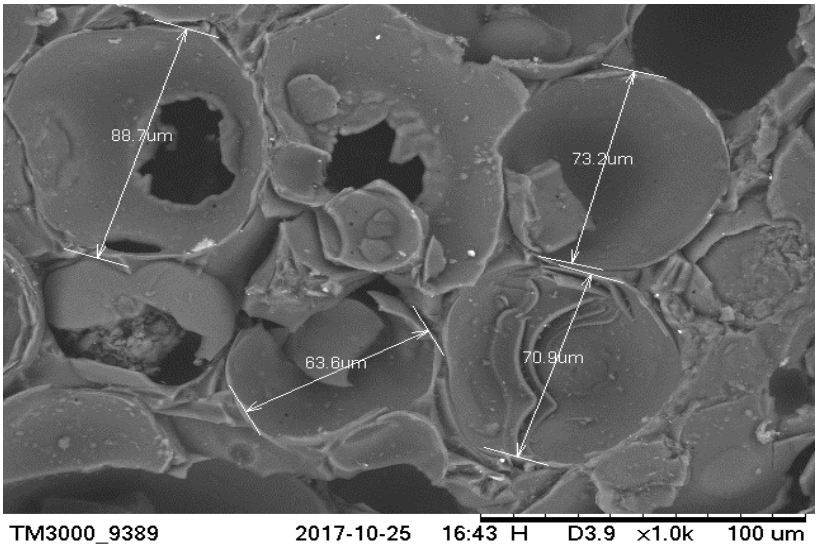


Figure 1. Characteristic linear dimensions of phenolic microspheres in SCF measured by scanning electron microscopy

The resulting bulk product was dried under vacuum using a rotary evaporator (residual pressure 1.3 kPa, temperature 110-120 °C) to obtain a granulated molding powder with a particle size of the order of 10 mm. The molding powder was crushed to a size of <500 μm and pressed 2-5 days after. Molding of workpieces was carried out using a hydraulic press PG-10 at room temperature in a mold. Figure 2 shows the dependence of the density of the pressed workpieces on the compacting pressure.

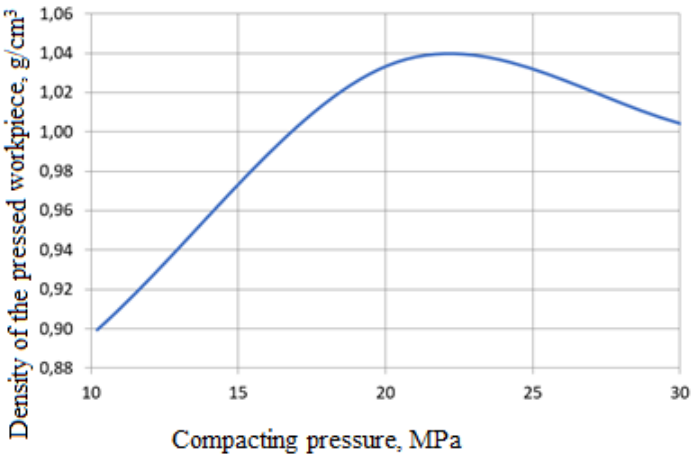


Figure 2. Dependence of the density of the pressed workpieces on the compacting pressure (mixture 50 wt. % PFR)

It can be seen that the density maximum of the “green” workpieces falls on the compacting pressure of about 21 MPa. Cracking of the samples is observed at a further increase of the compacting pressure. With this consideration in mind, pressing of samples was carried out at a pressure of 20.5-21.0 MPa, the rate of pressure increase and release of about 10 MPa/min and holding time at an end pressure of 3 min. Further, the workpieces were cured at a temperature of 150 °C with a holding time

of 180 minutes in a vacuum oven. Subsequent carbonization was carried out in a laboratory muffle furnace the workpieces of "green" foams were wrapped in paper and placed in a steel container with graphite filling up. The rate of temperature elevation was 2.5 deg/min, the holding time at an end temperature 900 °C was 180 minutes. Carbonized workpieces were exposed to pyro-compaction at a temperature of 1050 °C and a methane pressure 1.1-1.4 kPa in an industrial furnace in several stages.

Figure 3 shows the diagram of the density change of the SCF workpieces during the processing. These data are typical for high-porosity samples of carbon materials: the workpiece has a high apparent density (about 1 g/cm^3) after pressing, during the curing process the workpiece expands and a partial loss of binder mass occurs this is accompanied by a drop of apparent density. At carbonization, the loss of mass because of the release of low-molecular volatile products at the thermal destruction of the binder dominates the shrinkage phenomena. Then, in the pyro-compaction process, a slow increase of mass occurs due to the deposited pyrolytic carbon.

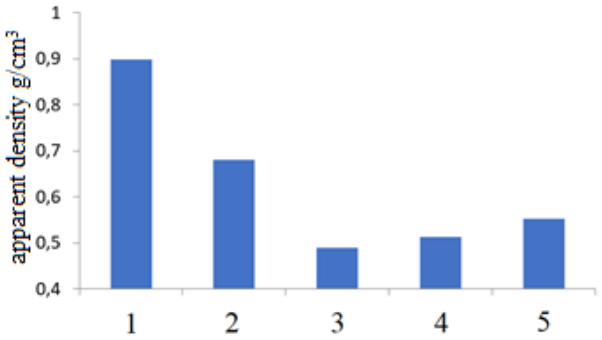


Figure 3. Change of the apparent density of the initial samples during processing: 1 – pressing, 2 – heat treatment at 150 °C, 3 – carbonization at 900 °C, 4 – pyro-compaction at 1050 °C for 20 hours, 5 – pyro-compaction at 1050 °C for 44 hours

Figure 4 illustrates the dependence of the samples apparent density on the PFR content in the initial molding powder after firing and pyro-compaction. It can be seen that an increase of the PFR content leads to an increase of the carbonized material density and this dependence is linear for large changes of PFR concentration. It follows that SCF density can be regulated in a wide range.

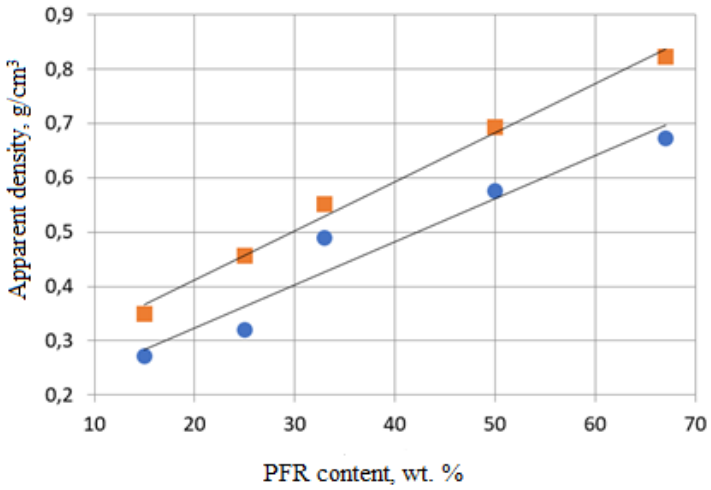
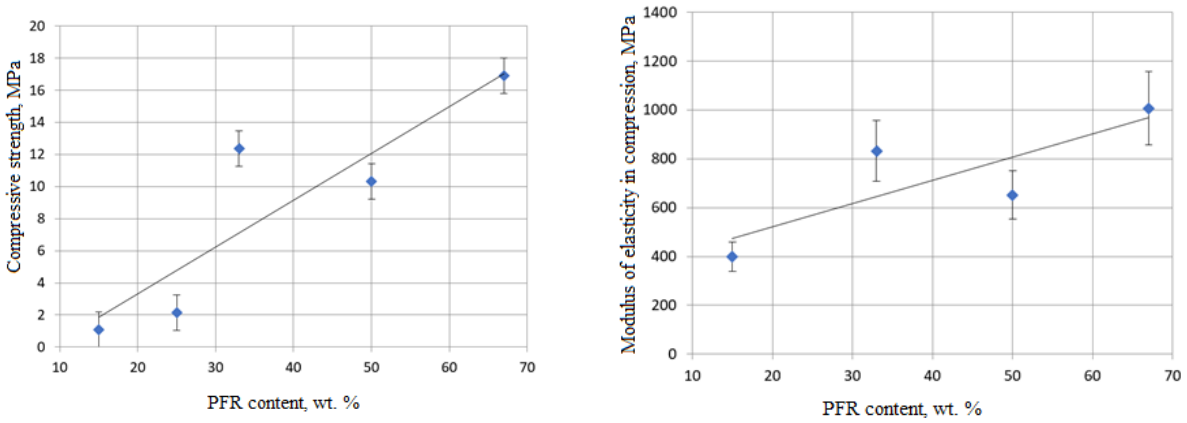


Figure 4. Dependence of the SCF apparent density based on the phenolic microspheres on the PFR content in the initial mixture for carbonized (●) and pyro-compacted (■) materials

Figure 5 shows, as an example, the change Dependencies of the SCF physical-mechanical properties on PFR content in the initial mixture.



a) b)

Figure 5. Dependencies of compressive strength (a) and modulus of elasticity in compression (b) of SCF samples on PFR content in the initial mixture

3. Conclusions

It can be seen that compressive strength of SCF containing more than 33 wt. % of PFR (in the initial mixture) stably stands at a level above 10 MPa, the modulus of elasticity – higher than 500 MPa approximately linearly increasing with an increase of PFR content in the sample. The achieved level of properties allows us to speak about the possibility of using the received SCF as a structural material.

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