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Production technology of heat-resistant high-strength syntactic carbon foams for operation in extreme conditions

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Abstract. Production technology of products based on the syntactic carbon foams designed to operate in extreme conditions is proposed. The components and regime parameters for the production of foams of an open-pore cellular structure with specified thermophysical and strength characteristics for manufacturing the large-sized products of complex shape are determined.

1. Introduction

Syntactic foams are composite materials consisting of a polymer, ceramic or metal matrix and hollow particles in the form of microspheres [1]. The term "syntactic" means the structure regularity when an elementary cell can be isolated the material structure can be described by its repetition [2-3].

Among the syntactic foam materials, carbon foams which have increased thermophysical and strength properties have a special place in terms of effectiveness and prospects of use [4].

The purpose of this work is to develop the production technology of syntactic carbon foams with an open-pore cellular structure for manufacturing the articles including large-sized ones with complex geometry.

2. Body text

Phenol-formaldehyde resin (PFR), solvent (acetone), and hollow glass microspheres were used as initial components. The preparation of the compositions was carried out by mixing the PFR, the solvent and the finished hollow glass microspheres at certain ratios. Manufacturing of the workpiece was carried out in shape-generating molding tools under pressure and elevated temperature with the simultaneous action of ultrasonic vibrations with a frequency of 15-18 kHz. Then, the workpiece was

carbonized in an electrovacuum furnace with a removal of the gas phase, and the article was exposed to pyro-compaction.

Figure 1 shows the diagram of the processing line in which the articles are produced. The ground granules of PFR 70-80 μm in size are loaded in the supply tank 1 of the auger-type dispenser machine 2 for feeding in the mixing unit 3. The solvent from the container 4 is also supplied by the membrane dispensing pump 5 to the mixing unit 3 in which the binder material is prepared. Mixing of the binder with microspheres is carried out by feeding them from the supply tank 6 equipped with a pneumatic vibrator 7 through the heating device 8 into the mixer 9. The prepared binder material with viscosity required for mixing with the filler is supplied by a dispensing pump 10, and the mixing unit 9 has a chamber 11 with an ultrasonic generator built into it at the output flow part.

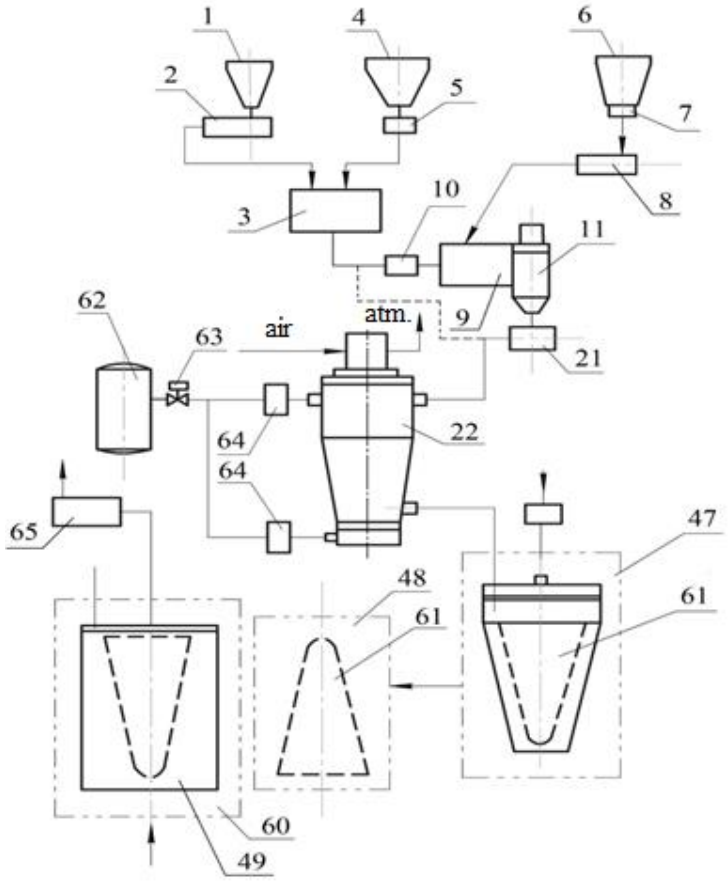

Figure 1. Diagram of the processing line

The mixing unit 9 (figure 2) of the binder material with the filler is a cylindrical body 12 inside which the core 13 with the Archimedes helical line 14is placed motionlessly and without tip clearance. The waveguide 15 of the ultrasonic generator 16 is located in the chamber 11 along the outflow axis of the mixed components therefrom. The binder material through the inlet opening 17 through the conical gap 18 passes the helical channel 19 into the chamber 11 acquiring a rotational movement. In the inlet part of the mixing unit 9 at the angle parallel to the inclination angle of helical line 14, the filler is supplied through the channel 20 in the direction opposite to the rotation of the binder composition. The filler is crushed and turbulized in the binder because of the different moving directions of the flows of binder and filler in the binder volume. The turbulization effect of the filler in the binder is enhanced by the additional influence of the ultrasonic vibrations primarily on the filler particles which have a density different from the density of the binder composition.

Due to the proportioning by volume of the binder fed to the special mixing unit of the components 9 the material viscosity up to the chamber 11 with the waveguide 5 is maintained within 10-15 Pa·s and after preliminary evacuation of the volatiles from the chamber 11 under the influence of the ultrasonic vibrations the viscosity increases to 18-20 Pa·s.

The resulting composition using the airless spraying device 21 is fed to the drying unit 22 (figure 3) of the shells of the microspheres encapsulated in the binder. The drying unit 22 is a cylindrical body 23 with a conical bottom in the lower part of which there is a pseudofluidization chamber 24 with a filter baffle 25 above which there is a suction pipe of the ejector device 26 for supplying the microspheres with the dried shell into the tooling of the product forming.

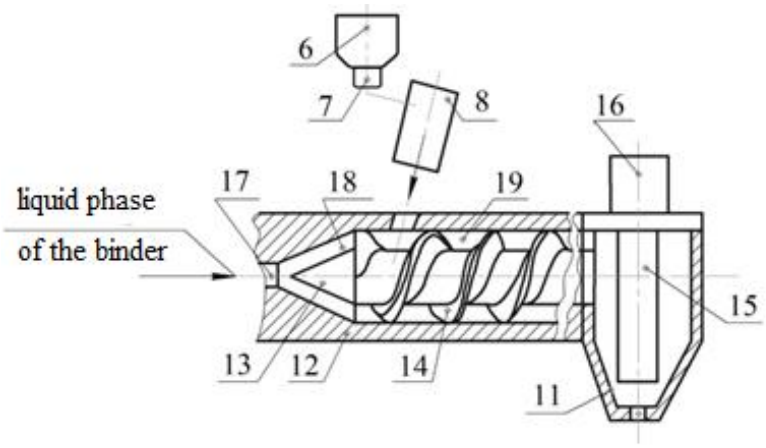

Figure 2. Mixing unit

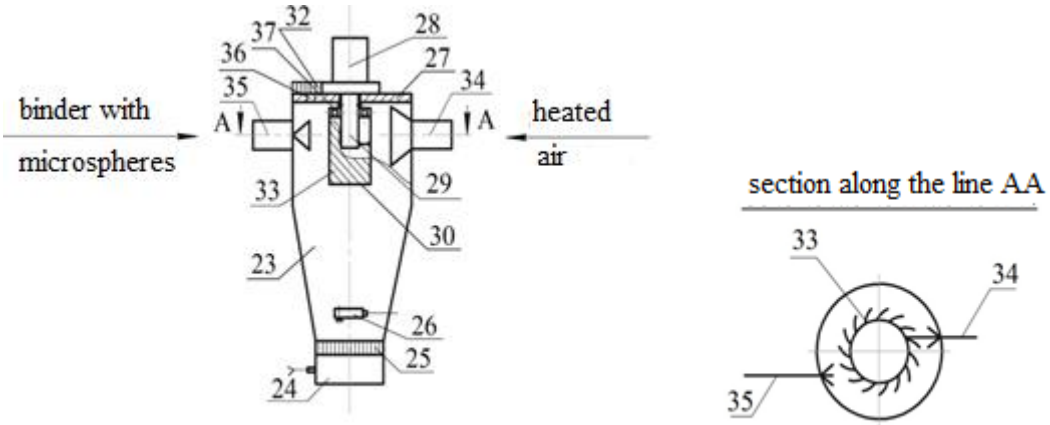

Figure 3. Drying unit

The pneumatic engine 28 is mounted in the upper part of the cylindrical body 23 closed by the lid 27, on its shaft 29 the cylinder shell 30 with the pneumatic vibrator 31 is fixed, and the compressed air is supplied to it through a central channel 32 on the shaft of the pneumatic engine 28. The blades 33 are positioned at an angle to the rotation axis on the outer surface of the shell 30. Under the lid 27 of the cylindrical body 23, the crevice nozzle 34 is positioned for supplying the heated air to the cylindrical shell surface and from the side opposite to it the nozzle 35 of the airless spraying device 21. The excess air from the drying unit 22 is removed through the channel 36 with the filter 37. A jet of viscous mass, sprayed by the airless spraying device, breaking into small particles falls on the surface of the rotating cylindrical shell with blades, meets with a stream of heated air, and falls down

along the blades. The microspheres that have fallen from the surface meet with the stream of heated air from the pseudofluidization chamber which is constantly in a bubbling state forming a dry shell on them. The drying unit operates in a cyclic mode for a definite volume of encapsulated microspheres. The air is supplied from the air heater 62 through the valve 63 and is controlled by the chokes 64.

Encapsulated in a casing made of binder material and dried fillers are transported by the ejector device 26 to the molding unit (figure 4) which is a split housing 38 with its inner cavity having an external configuration of the product being manufactured. The punch 39 is installed in the split housing 38, on the outer side it has a flexible silicone casing 40 which is tightened by a lid 41 to a split housing 38. The configuration of the punch 39 with the casing 40 is equidistant to the configuration of the inner surface of the split housing 39 with preservation of the channel 42 the size of which corresponds to the design thickness of the article 61. The grooves 43 connected with the central channel 44 are made from the outer surface of the punch 39, and the lid 41 is provided with openings 45 for air-filling of the encapsulated in the casing made of binder material fillers into the working volume 42 and the outlet channel 46 with filter cell (not shown) for the removal of compressed air at pneumatic transport.

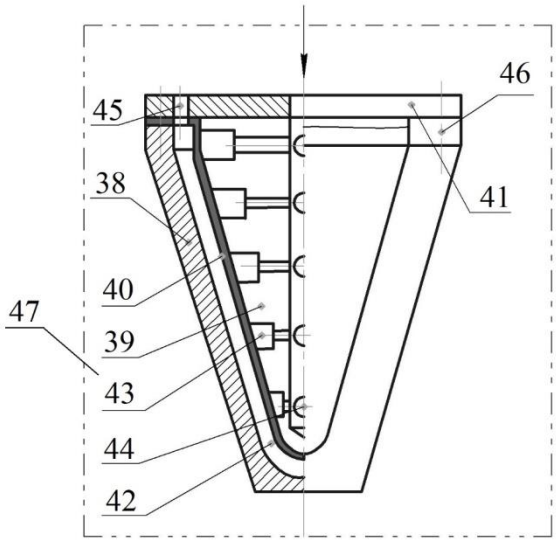

Figure 4. Molding unit

The preshaping of the workpiece is performed in the muffle furnace 47 at atmospheric pressure with a premolding of the silicone casing backfill at a compressed air pressure from 0.15 to 1.5 MPa through the central channel 44 and at temperatures up to 200-250 °C. After cooling of the formed workpiece, it is carbonized in the free state in an electrovacuum industrial furnace 48 at a vacuuming pressure of 10-80 mm Hg and temperature up to 900 °C with a rate of temperature elevation of 5 °C/min.

To perform the pyro-compaction of the outer working surface, the carbonized article 38 is loaded into the special tooling 49 (figure 5) its inner cavity is identical to the outer configuration of the pre-carbonized article.

The special tooling 49 is a set of rings 50 hermetically connected and tightened together by a stud. Each ring has an internal bore 51 and an opening 52 mated with the outer contour of the article. In this case, the series of openings 54 for supplying the gas phase are made on each ring's walls 53 sealing the product contour, and openings 54 are located in the horizontal plane and shifted by 180° relative to each other starting from the gas phase supply channel 55. The general sealing of the set of tooling rings is carried out by tightening them with top 56 and bottom 58 lids through the seal 57. For pyro-compacting, the tooling with the product 61 is loaded in the electrovacuum furnace 59. The pyro-

compacting process of the pre-carbonized polymeric matrix is carried out by a known method of supplying the natural methane gas on the heated surface of the article from the outer side by infiltrating the gas in the presence of a pressure gradient. This method of depositing the pyrolytic carbon on the outer surface of the product with an open-pore cellular structure at a certain depth allows providing high-temperature properties of the outer layer while maintaining the strength and heat-shielding characteristics of the entire product. In this variant, the reactant gas passes through the porous structure in the radial direction to the internal cavity of the article. Vacuuming [5-12] of the internal cavity through the channel 59 is carried out by the vacuum station 65.

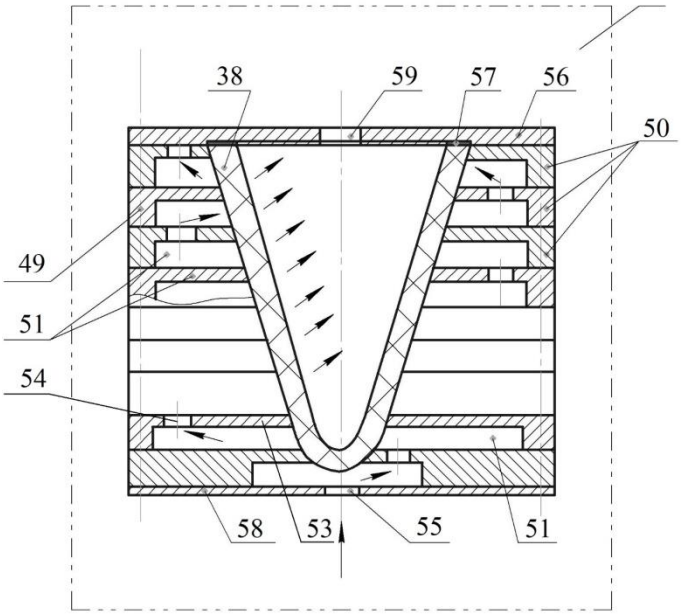

Figure 5. Tooling for product forming

3. Conclusions

Thus, the proposed technology ensures production of an article with a two-layer structure where the outer working layer compacted by pyrolytic carbon provides heat-shielding properties at operation up to temperatures of 3000 °C while maintaining the strength properties of the internal layers obtained by the carbonization of the carbon foam.

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