GLASSY-CARBON AND SILICON-CARBIDE HIGH-POROSITY PERMEABLE CELLULAR MATERIALS

V. N. Antsiferov¹ and N. M. Avdeeva¹

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Consideration is given to the production of glassy-carbon and silicon-carbide high-porosity cellular materials by resort to thermochemical transformations in the foamed polyurethane–phenol-formaldehyde resin–silicon system. The physicochemical properties of the materials thus synthesized are examined. Their microstructures are demonstrated.

High-porosity permeable cellular materials (HPCM) have a well-developed specific surface area and a low apparent density. Anywhere from 80 to 98% of a HPCM volume is taken up by macropores (cells) bounded by bridges which act as structural elements (Fig. 1). An HPCM owes its properties to both its structural characteristics and those of the matrix. In recent years, many sectors of the ceramic industry have been attaching an ever greater importance to high-temperature HPCM, of which the one of particular interest from the viewpoint of properties and practical value is silicon carbide. A major roadblock in the manufacture of silicon-carbide HPCM is that it is difficult to sinter. Silicon carbide is a compound with a covalent-ionic bond and will not sinter under ordinary conditions; that is, it fails to be densified by the viscous flow diffusion mechanism [1]. For this reason, the fabrication of silicon-carbide HPCM needs specialized methods involving chemical processes of phase formation and unorthodox reactants.

Research Institute of Powdered Metallurgy and Coatings, Russian Research and Technology Center for Powder Metallurgy, Perm, Russia.

The silicon-carbide HPCM has in the end been developed using constituents able to change into silicon carbide without disturbing the network-cellular structure of the material. These components are foamed polyurethane (FPU), phenol-formaldehyde resin (PFR), and fine-grained powdered silicon (Si). Each of these constituents has a particular function to perform in the formation of silicon carbide. FPU provides a shape-imparting substrate, PFR is the principal source of glassy carbon, and Si is the essential constituent in the synthesis of silicon carbide.

The formation of silicon carbide from the above constituents is a complex, multistage process which includes polymerization, carbonization of the organic constituents, and, finally, the chemical sintering of glassy carbon and silicon [2].

High-porosity cellular glassy carbon, which duplicates the network-cellular structure of the FPU, is produced by the co-destruction of the FPU – PFR polymer system in a non-oxidizing atmosphere [3]. Figure 2 shows a fracture in a structural element of high-porosity glassy carbon. The central

¹ Research Institute of Powdered Metallurgy and Coatings, Russian Research and Technology Center for Powder Metallurgy, Perm, Russia.
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Fig. 3. Section of a structural element in a high-porosity cellular silicon carbide preform: 1) channel pore; 2) silicon; 3) glassy carbon; a) $\times 200$; b) $\times 500$, reflected light.

Fig. 4. Structure of high-porosity cellular silicon carbide: a) section of a structural element, $\times 200$, reflected light; b) electron photomicrograph of a fracture in a structural element, $\times 500$.

part of the structural element is taken up by a channel pore produced upon the removal of the substrate. No other pores can be observed in the structure.

To convert the high-porosity glassy carbon to silicon carbide, powdered silicon particles are dispersed in the glassy-carbon matrix [4]. The microstructure of a preform composed of silicon and glassy carbon is shown in Fig. 3. The useful surface of the structure is formed by glassy carbon on which silicon stands out as isolated light-colored inclusions. When heated to 1600°C in an inert atmosphere, the glassy carbon reacts with the silicon to form silicon carbide. Figure 4a shows a section of a structural element in synthesized silicon carbide. As found by experiment, sintering leaves the size and shape of the HPCM unchanged. Because of this, the changes in volume that take place when the silicon and glassy carbon are converted to silicon carbide give rise to capillary micropores. They run through all of the synthesized silicon carbide and take up about 37% of the volume in the structural elements of the HPCM [5]. The manner in which micropores are distributed in chemically sintered silicon carbide can be assessed from the photomicrograph of an HPCM structural element (Fig. 4b).

Due to the presence, in high-porosity silicon carbide, of communicating micropores produced as the silicon carbide is formed and of channel pores left upon the removal of the shape-imparting FPU, it is possible to fabricate a composite material by filling the micropores with silicon [6]. The structure of such composite material is shown in Fig. 5. The background of the structure is silicon against which gray pore-free patches of silicon carbide can be seen. The pore-free patches result from the recrystallization of the silicon carbide which occurs as the fine silicon carbide grains dissolve and then are deposited on monolithic areas.

In this way, the various stages of thermochemical transformations in the FPU – PFR – Si system produce three kinds of high-porosity permeable cellular materials based, respectively, on glassy carbon (C-HPCM), silicon carbide (SiCHPCM), and a silicon carbide – silicon composition (SiC – Si-HPCM). Their physicochemical characteristics as found in this study are summed up in what follows.

The specimens were tested for mechanical properties on an Instron-1195 machine. The thermal conductivity of the materials was determined by the steady-state heat flux method, and their TCLE on a horizontal quartz dilatometer. The specific surface area of the materials was measured with a Sorbtometer K202 tester. Photomicrographs of the structures were taken using a NEOPHOT-21 optical microscope and an REM-200 scanning electron microscope.

For a proper comparison of characteristics, we examined materials having the same total porosity of 95%, although