Properties of composites SiC/SiCf obtained by hot pressing of SHS of silicon carbide powder

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Abstract. Dense ceramic samples based on SiC were obtained by hot pressing. We used the Use of silicon carbide powder as the initial components of SHS, due to the spherical shape of the particles, allows obtaining a denser workpiece. The reinforcing component in the form of silicon carbide fibers is obtained by siliconizing carbon fabric with SiO vapours. The phase composition of SiC fibers obtained by silicification by SiO vapour was determined. The properties of the obtained SiC / SiCf composites were studied: density 3.20 g/cm³, strength 520 MPa (for monolithic ceramics 390 MPa), the value of the crack resistance coefficient 5.9 MPa·m¹/².

1. Introduction

Modern technology, for its successful development and formation, is in dire need of materials capable of working in a wide range of operating temperatures and aggressive environments. High values of the physicochemical properties of silicon carbide ceramics make it widely applicable in various industries - rocket science, defense, chemical, metallurgy, oil industry, as well as cutting tools, heating elements, and abrasives, etc. However, brittleness is the major drawback of SiC ceramics and limits their uses as structural materials [1-3].

Monolithic ceramics low values of crack resistance can be improved with the help of reinforcing components in the form of fibers, fabrics and whiskers. The combination of ceramic matrix and reinforcing components allows to obtain ceramic materials composites with a high level of physico-chemical properties [4].

SiC/SiCf ceramic matrix composites (CMCs) are promising materials for replacing Ni-base super-alloys in high temperature applications as they exhibit as much as 1/3rd the density while maintaining sufficient strength and creep resistance to operate at significantly higher temperatures (i.e. >1000°C) [5].

The most successful for reinforcing silicon carbide ceramics are SiC fibers. The main manufacturers of SiC fibers are three main manufacturers found in the market are Nippon Carbon, COI Ceramics and Ube ind, producing the trade-marks Hi-Nicalon (Type-S), Sylramic (iBN) and Tyranno SA3 respectively, where the elemental composition of the precursors and the maximum production temperature varies [6]. However, the
process of obtaining SiC fibers is quite time-consuming and expensive, and in Russia it is insufficiently mastered. In modern conditions, there is a need to develop an alternative technology for the production of fibers based on silicon carbide, capable of performing the function of reinforcing components, preserving the structure when exposed to high temperatures and aggressive environments.

2. Materials and Method
Thanks to the study [7], a method was implemented to create a reinforcing component based on silicon carbide by siliconizing carbon fabric with SiO vapour. For this, a siliconizing unit was created based on a vacuum furnace. Siliconization of carbon tissue (Figure 1) takes place in a high-temperature laboratory reactor of periodic action, placed in a vacuum furnace at temperatures up to 1400°C. The reactor consisted of two corundum crucibles nested in each other. Siliconizable material was wound around the inner crucible, and an equimolar mixture of Si and SiO\(_2\) was placed on the bottom of the outer crucible as a source of SiO gas. This method allows to obtain a textile material based on silicon carbide, which can be used as a reinforcing component in the form of fabrics for layering, fibers and particles [4].

The starting components in the research were silicon carbide powders, a sintering additive YAG, and SiC fibers obtained by siliconizing carbon fabric with SiO vapors. Nanosized powder particles of silicon carbide were obtained by the SHS method at ISMAN RAS, Chernogolovka. The synthesis of SiC powder was carried out under nitrogen pressure, and a temperature of 1900-1950°C since the thermal effect of the direct reaction of silicon with carbon is insufficient for the synthesis in self-sustaining combustion mode. The spherical shape of the silicon carbide powder SHS allows dense sintering of ceramic masses (Figure 2a).
Figure 2. SEM a - silicon carbide powder obtained by SHS method, b - silicon carbide fiber obtained by siliconizing silicon fabric with SiO vapors.

Figure 2b shows a silicon carbide fiber obtained by siliconizing a carbon fabric with SiO vapour. The resulting fabric, consisting of continuous SiC fibers, the diameter of which is 5 μm, was mechanically crushed to a size of 1-2 mm. The fiber content in the samples ranged from 3 to 7 wt.%.

Due to the presence of a high proportion of Si-C covalent bond, the compaction of silicon carbide ceramics is difficult to realize without the participation of sintering additives. The most commonly used mixture of Y₂O₃ – Al₂O₃ oxides in a ratio of 3:5, which formed yttrium-aluminium garnet during sintering, was chosen as a sintering additive.

The starting components mixture was carried out in a planetary mill in the environment of isopropyl alcohol. The samples were formed by the method of one-sided semi-dry pressing. Sintering was carried out by hot pressing in argon at 1850°C for 30 minutes.

3. Results and discussion

In this paper, the microstructure and some physicochemical properties of hot-pressed silicon carbide reinforced silicon carbide materials obtained from SHS silicon carbide powders were studied (table 1). The SEM analysis of the obtained ceramic samples showed the absence of interaction between the matrix material and the reinforcing component (Fig. 3). The absence of interaction between the matrix material and the reinforcing component indicates that the resulting ceramics is a composite material and the implementation of ceramic reinforcement is carried out successfully.

| SiC SHS, wt. % | SiC fibre, wt. % | Firing conditions | Density, g/cm³ | Strength, MPa | K₁C, MPa·m⁰.⁵/₂ |
|----------------|-----------------|------------------|----------------|---------------|-----------------|
| 100            | 0               | T=1850°C, 1 h, Ar| 3.19           | 370±24        | 4.7             |
| 97             | 3               | 1 h, Ar         | 3.20           | 455±22        | 5.2             |
| 95             | 5               | Ar              | 3.20           | 471±22        | 5.7             |
| 93             | 7               | Ar              | 3.20           | 520±26        | 5.9             |
The physicochemical properties of the resulting densely sintered composites were studied. The fibers concentration increases in SiC matrix leads to an increase in the mechanical properties of the composite. The maximum physicomechanical properties of ceramics based on silicon carbide, reinforced with SiC fibers, were the following. The samples density was obtained 3.20 g/cm$^3$, strength 520 MPa (for monolithic ceramics 390 MPa), the value of the crack resistance coefficient 5.9 MPa·m$^{1/2}$.

4. Conclusion.
The silicon carbide powder was used as the initial components of the SHS, due to the spherical shape of the particles. It made possible to obtain a denser workpiece. The use of silicon carbide fibers as a reinforcing material is a promising area. Reinforcing components tend to increase fracture toughness by redirecting the crack paths and diverting the strain energy, or minimizing its growth. Thus, reinforcing ceramics with fibers has a positive effect on the basic physicochemical properties of silicon carbide ceramics; namely, it makes it possible to increase the values of strength and fracture toughness coefficient. This is due to the level of physicomechanical properties of modern fibrous reinforcing fillers, and to the implementation of a more complex, in comparison with monolithic ceramics, mechanism of destruction of a ceramic composite, which is especially true for impact loads [1].

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