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06

Ceramic composite on the basis of silicon carbide reinforced with molybdenum wire

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Silicon carbide and ceramic materials on its basis have unparalleled combination of physical and chemical properties. However, they are inherently prone to brittle fracture, which restricts the scope of application in structures subjected to tensile, bending and impact loads. A new approach is proposed to obtain composites with ceramic SiC matrix reinforced with molybdenum wire via siliconization of porous metal-carbon base. Interaction between silicon melt and a porous carbon blank produces a bound silicon carbide frame with residual carbon and silicon, and a contact between molten silicon and molybdenum wire surface forms a layer consisting of $MoSi_2$ and Mo_5Si_3. Quasiplastic fracture was detected during three-point bending testing of composite samples. Molybdenum wire serves as a barrier preventing crack propagation in case of matrix damage.

Keywords: silicon carbide, molybdenum wire, ceramic matrix, thermal stability.

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Introduction

Silicon carbide (SiC) has one of the most remarkable combination of physical and chemical properties among all existing materials. Silicon carbide ceramics features extremely high strength, low thermal expansion coefficient, thermal stability and outstanding fracture resistance under the action of stress at high temperatures. Density of SiC ceramics depending on the composition varies from 2.3 to 3.1 g/cm^3, which is lower by approximately 30 % than the density of aluminum oxide ceramics. SiC ceramics also has high radiation resistance and chemical inertness and, thus, can be used in oxidizing and corrosive media even at high temperatures [1–4]. Due to their high near-record-breaking hardness, thermal conductivity, wear resistance, chemical and thermal resistance, structural SiC ceramics is of great interest to a wide range of applications in various industries (armor protection systems, journal bearings, end sealings of pumps for oil and gas industry, high-temperature furnace parts, magnetron sputtering targets, shell-and-tube heat exchangers for chemical industry, nozzles, high-temperature thermocouple wells for steel and iron industry, gas turbine engine parts and components, etc.).

However, high brittleness of SiC ceramics seriously restricts the scope of application in structures subjected to tensile, bending and impact loads because cracking usually causes failure of SiC ceramic parts. To extend the scopes of application of SiC ceramic materials and to improve reliable operation of ceramic products in high loading conditions, it is necessary to increase impact strength and fracture resistance, which are defined as material's capability to absorb impact energy and withstand crack growth [5–7].

Besides strength enhancement, introduction of dispersed oxide [8,9], carbide [10], boride [9,11,12], metal [13], silicide [14], nitride [12,15] particles, carbon nanotubes [16], etc., into SiC ceramics in various ways makes it possible to partially increase fracture resistance by preventing crack propagation.

The most effective way to solve the fracture resistance problem and improve performance is the development of structural ceramic materials where the properties of silicon carbide, serving as a matrix, are combined with the engineering advantages of composites reinforced with miscellaneous fibers. Either high-modulus carbon fibers or core-free SiC fibers are used as the reinforcement component material.

Composites with ceramic SiC matrix reinforced with carbon fibers (C_f/SiC composites) have good tribological properties and are used as rolling bearings and deployment systems in aerospace craft and in aircraft and train braking systems. Due to good temperature resistance of fibers, especially in inert media, C_f/SiC composites have an excellent track record as thermal protection of high-temperature aerospace structures.

Core-free SiC fibers used in SiC_f/SiC composites have high strength and Young's modulus, radiation resistance, high oxidation resistance, and the same thermal expansion coefficient as that of the SiC matrix [17–19]. Owing to the unparalleled combination of physical and chemical properties, composites with SiC matrix reinforced with SiC fibers have high fracture resistance at temperatures up to 1400 °C. Stability of properties of such composites at high temperatures makes it possible to use them in high-loaded structures operated in corrosive media during a long period of time. In particular, SiC_f/SiC composites used as materials for uncooled blades and other components of gas turbine engines (GTE) provide a considerable increase in the operating temperature and, consequently, efficiency of GTE.

Due to high radiation and heat resistance, SiC_f/SiC composites provide considerable increase in the efficiency of nuclear reactors. Combination of properties of such composites also makes it possible to regard this material as a promising one for use in thermonuclear power plants with a magnetic system designed to obtain and hold hot plasma [20–22].

Recently, new materials, which can be used as alternative to C_f/SiC and SiC_f/SiC composites, have been extensively studied. In particular, an interest is displayed in the development of composites where metal wire is used for reinforcement of ceramic matrix. Such materials can change the fracture behavior of a composite by making it more ductile [23]. Moreover, they have high strength and can withstand loads, which are inaccessible to traditional ceramics.

In particular, molybdenum wire has a high melting temperature, corrosion resistance, strength and ductility. The wire has a relatively low cost, is commercially produced and is available in Russia unlike silicon-carbide and high-modulus carbon fibers, which makes it a promising and attractive product for the development of high-temperature composite materials.

Also, production of high-temperature complex-geometry composites with SiC-based ceramic matrix faces a problem of formation because the ceramics machining process using diamond tools is not only complex, but also cost-intensive [24]. Therefore, one of the important tasks involves the development of techniques reducing the cost of composite products with hard ceramic matrix.

The authors have earlier reported [25] a possibility to produce a metal ceramic composite material via liquid-phase siliconization of porous blanks with embedded molybdenum elements in the form of wire. The obtained high-temperature metal ceramic composite samples had pseudoplastic fracture behavior. Concept demonstration used SiC−Si−C ceramics with SiC concentration of about 49 vol.% and ultimate bending strength of 105 MPa. When ceramics was reinforced with 2 mm molybdenum wire, increase in its mechanical strength was observed. This study provides the results of efforts taken to improve the mechanical strength by increasing the SiC concentration on the matrix to more than 60 vol.%. with simultaneous reduction of the diameter of reinforcing elements to 1−1.5 mm.

This paper describes the production process and properties of a high-temperature metal ceramic composite with pseudoplastic fracture behavior. Composites are obtained by siliconization of porous carbon blanks with embedded Mo wire.

1. Production of composites with SiC-based ceramic matrix reinforced with Mo wire

The metal ceramic composite process is based on a SiC ceramics production process previously developed by ISSP RAS [26]. Flow chart of the original Sic ceramics production process is shown in Figure 1.

Powder made by breaking graphite (manufacturer - Technikarb, Chelyabinsk, Russia) on a jaw crusher followed by grinding on a hammer mill was used as a basis for producing porous carbon blanks. Then the multi-fraction mixture of powders was screened on a jigging screen with a screw feeder to separate it into narrower fractional ranges.

The resulting narrow fractions were mixed at certain ratios with the SFP-011L thermally-reactive coking polymer binder with its proportion of at least 20 mass %. Then the mixture was compacted and baked to 900 °C. This resulted in a porous carbon base material, which could be subjected to machining to render a shape close to that of a finished product.

Then siliconization was performed where the optimum ratio of impregnation rate and carbide formation [27] is defined by many factors: temperature, holding time, heating rate and rate of silicon flow over carbon, rates of silicon carbide nucleation and crystal growth on carbon surface.

At the initial siliconization stage, exothermic reaction between carbon and silicon during several seconds produced a shell on the surface of graphite particles, consisting of the first thin barrier silicon carbide layer, preventing further fast penetration of silicon to carbon trapped in the particles. Then, before the silicon melt−carbon interface, preceding diffusion of silicon atoms took place in solid carbon phase behind the crystallization front of the first carbide layer [28] and a 10−15 μm layer was being formed during 10−15 min, and then the growth almost stopped. Formation of the shell consisting of silicon carbide leads to obstruction of pores in graphite particles followed by stop of silicon melt penetration into the particles. With further increase in the holding time, SiC is formed with a very slow rate and fully stops after achieving equilibrium concentration of silicon in silicon melt.

Carbide formation reaction behavior on the graphite surface is greatly affected by selection of a graphite grade, which is used to produce powders with various grain sizes for carbon base formation. Depending on the fraction ratio, composition and amount of organic binder, as well as on carbon blank compaction and heat treatment conditions, the technique allows variation of the SiC−Si−C phase composition in a wide range depending on the user requirements.

More accurate prediction of phase ratio, density and, consequently, strength of SiC−Si−C ceramics used a previously developed phase composition calculation method for materials obtained via carbon matrix siliconization [29].

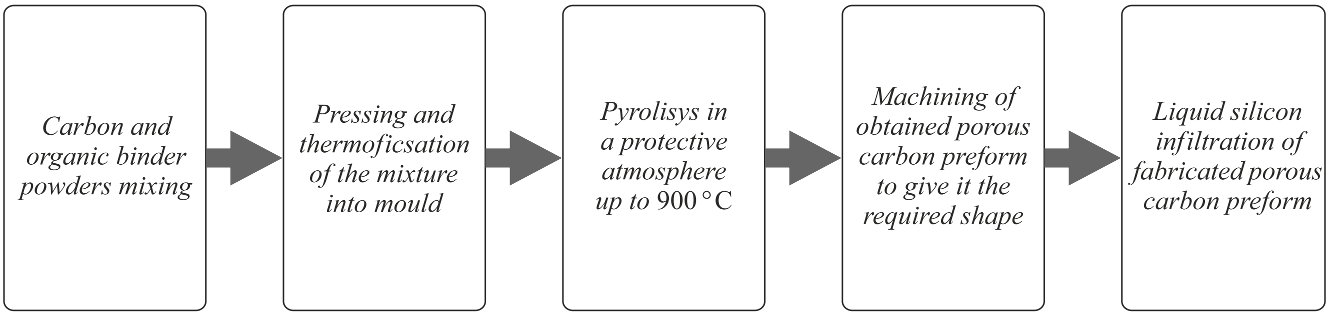


Figure 1. Sequence of the SiC$-$Si$-$C ceramic production process using siliconization of porous carbon matrix.

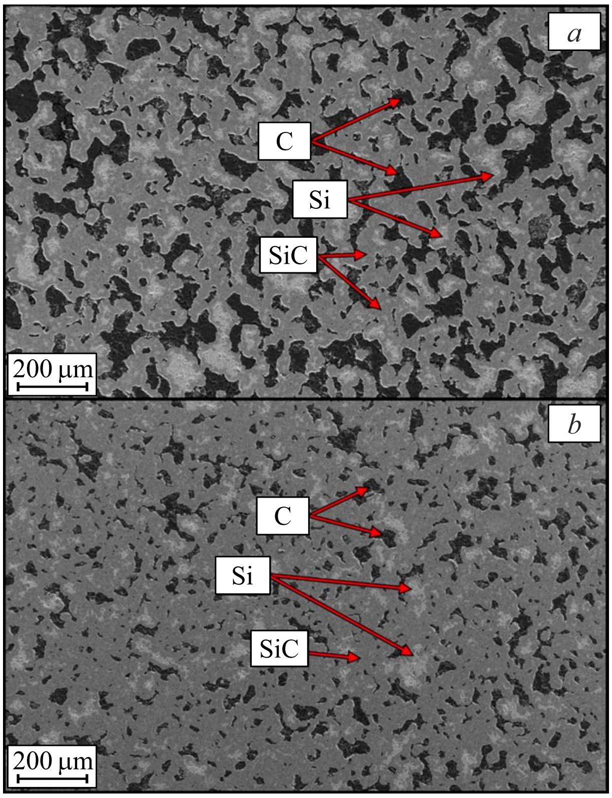


Figure 2. Microstructure of SiC$-$Si$-$C ceramics consisting of: *a* — SiC — 60 vol.%, C — 30 vol.%, balance Si; *b* — SiC — 75 vol.%, balance C — 15 vol.%, Si.

Samples of high-strength SiC composite material reinforced with Mo wire were produced using fine graphite powders ($50-100\,\mu$m and smaller than $50\,\mu$m), thus, providing a SiC concentration in the ceramic matrix more than 60 vol.%. This is due to the fact that fine graphite particles almost completely pass into SiC, while the fraction of residual encapsulated graphite reduces considerably. Microstructures of two SiC ceramics options achieved using the above-mentioned powders are shown in Figure 2. A compound using carbon filler powders finer than $50\,\mu$m was chosen as a base for the high-strength composite SiC matrix to provide a SiC concentration after siliconization of about 75 vol.% and an ultimate bending strength of 273 MPa.

Mo wire (Ural-Metal, Yekaterinburg, Russia) was embedded into the porous carbon matrix at the formation stage.

Figure 3 shows a diagram of preparing samples reinforced with 1.5 mm Mo wire. A mixture of carbon and thermally-reactive coking polymer binder powders were put into a mold. Then the powder surface was smoothed. After that the metal wire was placed on the prepared surface in a particular order at predefined intervals. Then another portion of the carbon and polymer mixture was added into the mold and the resulting mixture was compacted at 15 MPa. Blanks reinforced with 1.0 mm Mo wire were made in the same way. Further treatment and siliconization stages coincide with those described in the flow chart.

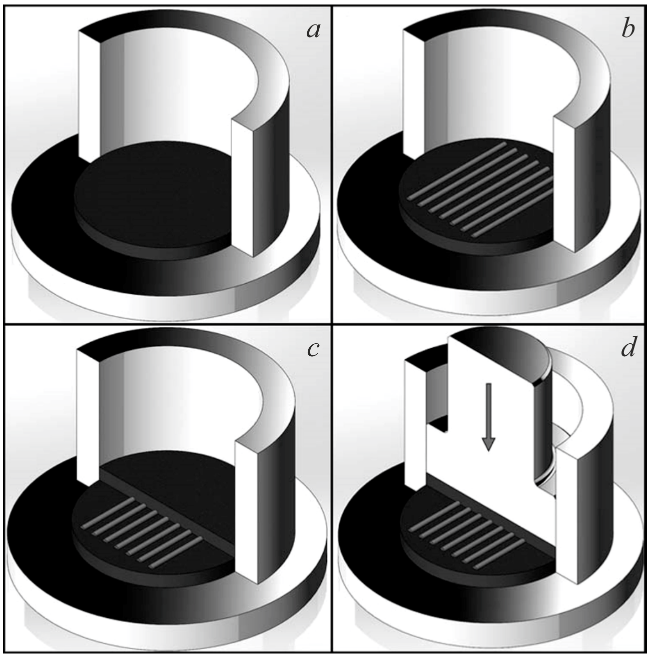
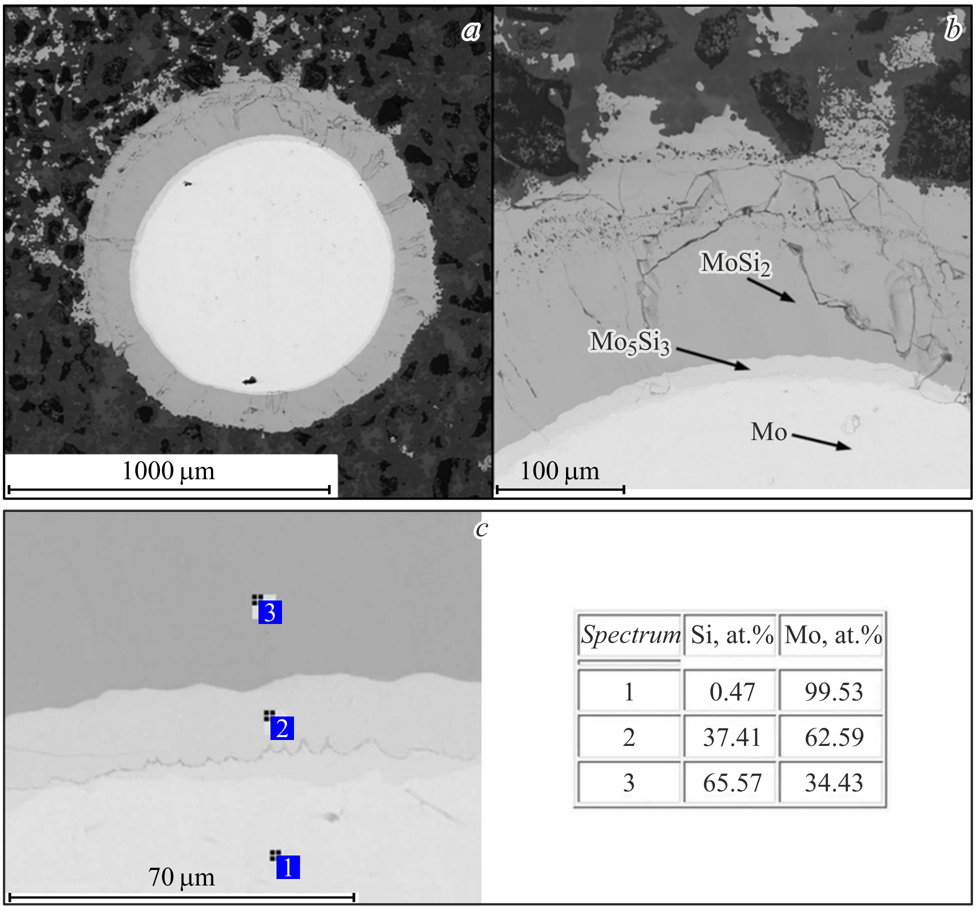


Figure 3. Sequence of producing a porous carbon blank reinforced with Mo wire: *a* — forming the first charge layer in the mold, *b* — placing the metal wire, *c* — forming the second charge layer, *d* — blank compaction.



Spectrum	Si, at.%	Mo, at.%
1	0.47	99.53
2	37.41	62.59
3	65.57	34.43

Figure 4. Microstructure of a fragment of composite with SiC−Si−C ceramic matrix reinforced with Mo wire (*a,b*); elemental analysis of the metal−silicide−ceramics transition area (*c*).

Siliconization was performed by sprinkling where silicon was melted in a separate container placed above the metal carbon blank and then flew down on it. At the same time, the porous base was impregnated with the melt and silicon interacted with the graphite powder to form silicon carbide, and reaction of interaction with the Mo wire surface took place, resulting in formation of a silicide layer between molybdenum and ceramic matrix.

The study used samples where the ceramic matrix with a density of 2.85 g/cm^3 consisted of SiC — 75 vol.%, C — 15 vol.%, balance — Si.

It should be emphasized that after silicon melt treatment, dimensions of porous carbon blanks with embedded metal wire vary negligibly, i.e. the shape and dimensions can be considered to be the same. Due to the fact that there is no shrinkage after interaction with the silicon melt, complex parts can be produced by means of mechanical treatment of a soft carbon blank. After impregnation with silicon, a SiC ceramic part with minimum machining allowances is obtained.

This technique makes it possible to produce SiC ceramic composite materials reinforced with Mo wire. Such materials retain the advantages of SiC ceramics and simultaneously have better fracture toughness due to reinforcing elements. Moreover, Mo and Mo silicides can potentially increase the heat resistance of resulting composite materials.

2. Phase composition and microstructure of the obtained composites

Microstructure and elemental composition of the obtained composites were analyzed using the Carl Zeiss Supra 50 VP (Carl Zeiss, Dresden, Germany) high-resolution (1.3 nm with an accelerating voltage of 20 kV, working distance of 2 mm) field emission scanning electron microscope with INCA Energy+ microanalysis system.

Figure 4 shows the microstructure of a composite with SiC-based matrix reinforced with Mo wire. Analysis shows that a chemical reaction between the silicon melt and Mo wire surface produces a silicide layer, whose mean thickness is 100−150 μm. Despite the fact that Mo wire partially passed into silicides, it was still metallic and ductile internally. It is important that molybdenum disilicide constituting the major portion of this layer forms a stable bond with the ceramic matrix, ensuring the optimum composite performance. A lower

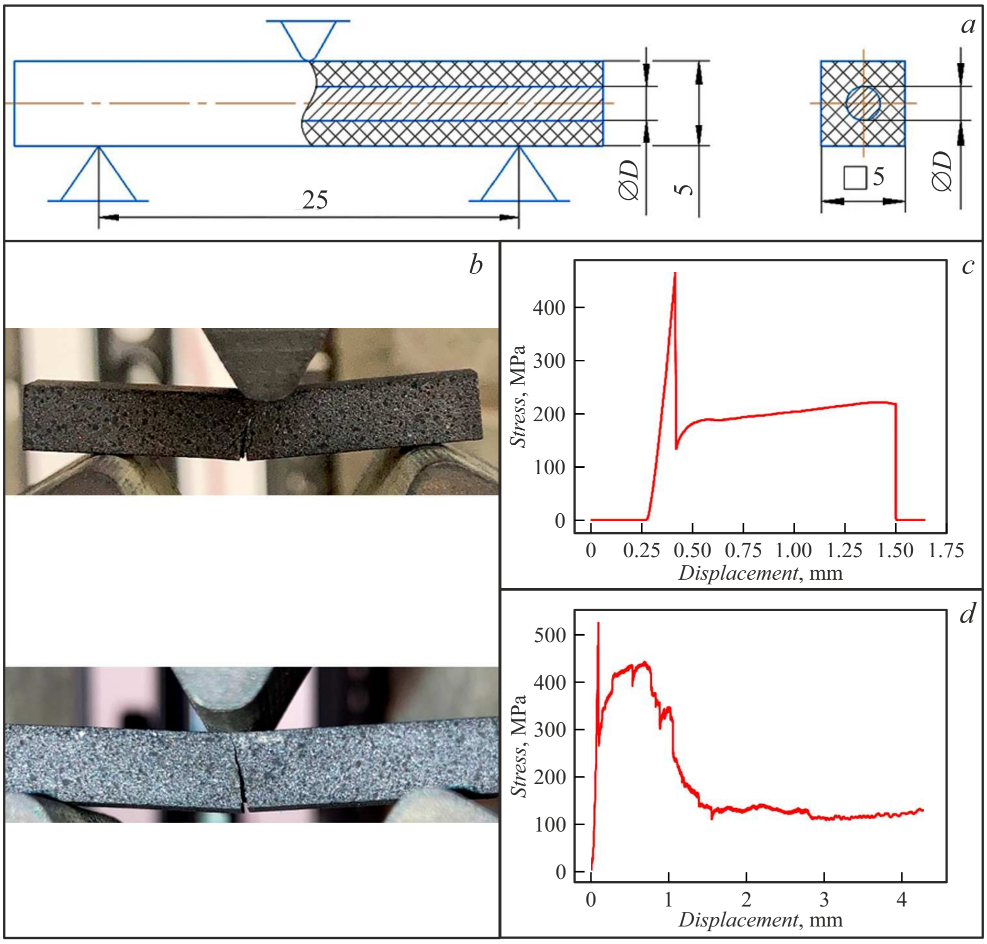


Figure 5. Loading diagram and sample dimensions, $D = 1.0$ and 1.5 mm (*a*), sample deformation during testing (*b*), sample loading diagram with 1.0 mm wire (*c*), with 1.5 mm wire (*d*).

silicide, Mo_5Si_3, layer much thinner than disilicide is formed between the residual molybdenum in the wire and $MoSi_2$ layer, which is confirmed by the elemental analysis data. Experiments show that both the total silicide thickness and ratio of $MoSi_2$ and Mo_5Si_3 layers can be varied depending on heat treatment temperature and time. The next stage of study involves the investigation of silicide layer formation reaction kinetics and analysis to predict changes in the composite microstructure and properties.

Considering that an approximate silicide layer thickness is about $100-150\,\mu$m, a reinforcing wire diameter of $\leq 250\,\mu$m provides a composite with a ceramic matrix reinforced with continuous molybdenum silicide fibers (or silicides of other heat resistant metals in case when, for example, niobium or tungsten wire is used for reinforcement). Composites of this type can ensure plastic deformation of composites at temperature above the visco-elastic transition temperature [30] of silicides and, consequently, can have high fracture resistance at temperatures above 1000 °C–1200 °C [31].

3. Mechanical testing of composite samples

For strength evaluation of composites with a SiC-based matrix reinforced with Mo wire, three-point bending tests using the I1147M (Tochpribor, Ivanovo, Russia) test machine with computer-based control system and measurements with the maximum working zone load up to 50 kN were performed. Composite samples with dimensions $5 \times 5 \times 35$ mm were used, and the distance between supports was 25 mm.

Photographs of composite samples with SiC-based matrix reinforced with Mo wire during the three-point bending test process and loading diagrams are shown in Figure 5.

Partial brittle fracture of the ceramic matrix typical of such materials occurred at the initial loading stage. Bending stress occurs at the bottom of the sample cross-section, induces ceramic component fracture and initiates a crack, which quickly propagates upwards. If there were no reinforcement, total fracture of the sample would have occurred at this time. However, the reinforcing element prevents further crack propagation, stopping the crack

Results of three-point bending testing of SiC$-$Si$-$0C composites without reinforcement and with Mo wire reinforcement

Sample No. №	Mo wire diameter, mm	Fracture stress, MPa	Fracture mode
1-1	0	273	Brittle
1-2	0	260	Brittle
1-3	0	251	Brittle
2-1	1.0	299	Quasi plastic
2-2	1.0	322	Quasi plastic
2-3	1.0	289	Quasi plastic
3-1	1.5	376	Quasi plastic
3-2	1.5	353	Quasi plastic
3-3	1.5	414	Quasi plastic

growth. Due to plastic deformation, it also preserves the sample's load bearing capacity. Consequently, the matrix starts fracturing and load is redistributed to the Mo wire with the $MoSi_2-Mo_5Si_3$ shell. Coupling between the matrix and reinforcing element was broken in the sample, the interface slid and the wire fully carried the load. Under the action of external applied load, the interphase shell ensures effective load transfer and plays a critical role in reinforcing Mo wire. As the load further increases, the wire starts extending in length at the shell/matrix interface and breaks at the maximum load. Brittle fracture of the silicide layer, after which the wire was still internally ductile, was observed on the failure surface in the molybdenum wire zone. Fracture stresses for each series of testing are listed in the table.

For unreinforced ceramics, mean strength is 261.3 MPa, range is 22 MPa, for reinforced ceramics, these are 303.3 MPa and 33 MPa with a wire diameter of 1 mm, and 381 MPa and 61 MPa with a wire diameter of 1.5 mm.

Conclusions

A process for producing composites with SiC-based ceramic matrix reinforced with Mo wire has been developed. At the initial stage, a porous carbon blank with molybdenum wire placed inside it was formed. Then, the metal carbon blank was subjected to siliconization.

Interaction between silicon melt and the porous carbon blank produced a bound silicon carbide frame with residual carbon and silicon, and a contact between the silicon melt and molybdenum wire surface formed a layer consisting of $MoSi_2$ and Mo_5Si_3 between the ceramic matrix and molybdenum. Molybdenum disilicide constituting the major portion of this layer formed a stable bond with the ceramic matrix.

Mechanical strength of the reinforced ceramics increased compared with the standard non-reinforced SiC$-$Si$-$C ceramics. Maximum increase was observed with a wire diameter of 1.5 mm, while the 1 mm wire gives a less significant effect. One of the probable causes is that the tensile stress zone contains a smaller concentration of metal. More reasonable arrangement of reinforcing elements, increasing the number of reinforcing elements with simultaneous reduction of diameter can provide even more significant improvement of mechanical strength.

Quasi plastic fracture of samples occurred during the three-point bending test. Reinforcing elements serve as barriers, preventing crack propagation in the case of matrix failure.

The developed process for producing a composite material with silicon carbide matrix reinforced with molybdenum wire (or wire made of another heat resistant alloy or metal, for example, tungsten or niobium) makes it possible not only to increase the composite fracture resistance, but also to solve the formation problem and, consequently, to reduce the cost of products by minimizing the scope of final machining.

For deeper understanding of the composite formation process and to control mechanical properties, the scope of future work includes the study of thickness and composition of the formed metal silicides depending on the holding time and siliconization or annealing temperature.

It is also planned to perform a quantitative estimate of the strength of bond between the reinforcing wire and ceramic matrix, and produce a metal-ceramic composite using wire made of hard-melting metal with a diameter smaller than 200 μm to provide a significant increase in the strength of material.

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Conflict of interest

The authors declare no conflict of interest.

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