Calculation and experiment approach to predicting the properties of C/SiC and SiC/SiC composites

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Abstract. One of the current trends in aviation and space technology is higher flight speed. An increase in flight speed means an increase in the temperature of the aircraft components and tougher requirements for the physical and mechanical characteristics of the materials used. As metals are reaching their limits, heat-resistant non-metallic materials are attracting more and more attention. Of all heat-resistant non-metallic materials, the most appropriate for this purpose are monolithic ceramic materials, carbon-carbon and ceramic-matrix composite materials. This paper proposes an approach to predicting the mechanical characteristics of ceramic composite materials based on experiment and numerical simulation of the material microstructure. Multi-stage impregnation and pyrolysis (PIP) method was used to manufacture C/SiC and SiC/SiC ceramic samples, which were later subjected to tensile testing and 4-point bend testing. The results of numerical modeling and the results of experiments were compared, and the mechanical properties of the materials were determined.

1. Introduction.

Ceramic composite materials (CCM) based on carbon fiber reinforcement and silicon carbide matrices, or C/SiC composites possess a unique combination of high thermal and mechanical properties and low density. C/SiC composite structures can retain their working efficiency at up to 2700°C. However, the lifespan of such structures is limited by the oxidation of carbon fibers at temperatures above 450 °C and the oxidation of SiC matrix at temperatures above 1000-1900 °C, depending on the properties of the environment. C/SiC composites have found wide application in rocket and space technology: thermal protection elements and liquid rocket engines [1, 2]. NASA was planning to use C/SiC composite in the nose cone of the X-38 reusable spacecraft project (Figure 1 [3]). C/SiC composite is also used in the advanced liquid-propellant rocket engines: Vulcain main engine nozzle (Ariane 5 launch vehicle), EAM space platform correction engine nozzle and combustion chamber [4] (Figure 2). SNECMA used C/SiC composite in the nozzle flaps of the M88-2 gas turbine engine for Rafale fighter jet. As part of the SIMPLEX Turbo pump Blisk program, NASA is developing a disk wheel from C/SiC composite for a turbo-pumping unit of a liquid rocket engine [5]. When reinforcing the ceramic matrix with carbon fibers, both the fiber oxidation and the carbide formation along the boundaries of the fibers must be prevented. Therefore, for structures that will operate in the Earth’s atmosphere for a prolonged period of time, the CCMs reinforced with coreless SiC fibers appear the most promising. However, the strength of the modern SiC fibers starts declining at temperatures above 1250°C [6-15]. NASA is conducting CCM research under the NASA Fundamental Aeronautics Program [16]. General Electric Company is developing SiC/SiC turbine blades as part of the Adaptive Engine Technology Development program. The combustion chamber demonstrator from SiC/SiC for the CFM56 engine by
Snecma and Herakles has been tested successfully. Pratt & Whitney’s thrust vectoring nozzle flaps have successfully completed over 11,000 test cycles /5,000 hours.

Fig. 1. C/SiC composites in the X-38 spacecraft

Fig. 2. C/SiC composites in the EAM engine

2. **C/SiC and SiC/SiC manufacturing technology.**

The main methods for producing silicon carbide matrix for reinforced ceramic composite materials are gas vapor deposition (CVI), silicon melt impregnation (LSI) and polymer solution impregnation followed by multi-stage pyrolysis (PIP).

Each of the methods has a different effect on the properties and cost of the final composite. LSI causes the greatest degradation of the physical mechanical properties of the material, and CVI means the highest labor intensity. PIP allows obtaining silicon carbide matrix without expensive equipment, and there is no significant degradation of properties, which makes this method the most popular. However, it should be noted that in the case of PIP, the porosity of the material is the highest and decreases only with an increase in the impregnation and pyrolysis stages.

The main components used in this research were a polycarbosilane precursor (a carbide structure after pyrolysis), 2D carbon fabric with equal strength, fabric, and 3rd-generation coreless SiC fibers of the Hi-Nicalon Type S (OX) type. Mechanical properties of the components are in Table 1.

**Table 1.** Mechanical properties of the components of ceramic composites.

| Characteristic                  | Ceramic composite component |
|--------------------------------|-----------------------------|
|                                | SiC matrix | C fabric | SiC fiber |
| Density, g/sm³                 | 1.9        | 1.81     | 2.85      |
| Young’s modulus, GPa           | 7/7*       | 230      | 200*      |
| Poison’s ratio                 | 0.2        | 0.2      | 0.22      |
| Ultimate stress, MPa           | 75-100     | 5490     | 2300      |
| Fiber content, %               | –          | 43       | 30        |
| Filament diameter, μm          | –          | 5        | 10        |
| Warp/ weft cross-section, mm   | –          | 2 x 0.15 | –         |

* – characteristic at 1000°C

3. **Calculation and experiment investigation of the strength of C/SiC and SiC/SiC samples.**

An approach is proposed for determining the mechanical properties of ceramic composites based on the comparison of experimental results and numerical simulations. First, the strain in characteristic zones must be determined experimentally. Then a finite element calculation of the loads must be conducted.
3.1 Strength analysis of C/SiC samples. For experimental studies, we used two samples, with the working area of 250 mm x 4 mm x 14 mm. Fiberglass pads were glued to the either side of each sample to ensure grip during testing (Figure 3). The samples were made from polycarbosilane and carbon fibers (2D isotropic fabric) using PIP technology. The tests were carried out using the Instron 5985 as per the GOST 25.601-80 standard. The sample was clamped into wedge-shaped hydraulic grips and stretched until it fractured. The longitudinal strain in the center of the working area of the sample was measured using the Epsilon extensometer (Figure 4).

The tests demonstrated the linear increase in the load up to the initial failure of the samples. The fracture point for both samples corresponded to the stress level in the section of the working area of ~ 60 MPa and longitudinal strain of 0.11%. The fracture area is the junction of the working area and the grip (Figure 5).

To determine the mechanical properties of the C/SiC composite, a 2D woven structure model was created in the Digimat software package (Figure 6). The input data were the component characteristics (Table 1). The properties of the obtained material are in Table 2. A finite element model of the sample was created in ANSYS and progressive loading was simulated numerically. The boundary conditions in the model are shown in Figure 7: in the lower end part, the sample is fixed in the X and Y directions, a force distributed between the nodes of the top line was applied to the upper end part. Elements simulating a multilayer composite were used.
The comparison of the numerical and experimental results of longitudinal strain at point 1 (Figure 7) is shown in Figure 8. The following points are marked: A - the moment of initial fracture and B -- the moment of final fracture. Up to A, the deformation is close to elastic. In section A-B, progressive destruction of the sample occurs, but this section is negligible. The stress distribution at the moment of the initial fracture (point A, Figure 8) in one layer is shown in Figure 9.

There is good agreement between the results of finite element modeling and experiment up to the initial fracture moment for the C/SiC composite. The maximum stress zone along the X axis (A, Figure 9) qualitatively coincides with the fracture zone of the sample in the experiment.

3.2 Strength analysis of SiC/SiC samples. Three test pieces with dimensions of 4.00 x 2.5 x 48 mm were used for the experiment (Figure 10). These samples were manufactured using PIP technology. The manufacturing process involved four stages of polymer impregnation and pyrolysis.
Four-point bend test for SiC / SiC samples was conducted at 1000°C on the Instron 5985 testing machine with a EuroTherm high-temperature furnace. The lower support span was \( L_1 = 40 \) mm, and the upper support span was \( L_2 = 20 \) mm. Analysis of the loading diagrams showed that after a certain threshold value, a load drop was observed due to the fiber failure, with the load instantly transferred to other fibers. The testing terminated automatically when the load fell by 50% of the maximum. Thus, the failure is quasiplastic. Upon visual inspection of the tested samples, no damage was observed.

The mean value of the ultimate flexural strength from four-point bend test was \( \sim 157 \) MPa and the ultimate strain was \( \sim 0.22\% \).

To determine the mechanical characteristics of the SiC/SiC samples, a 3D structure model was created in the Digimat software package (Figure 11). The input data were the component characteristics (Table 1). Properties of the resulting material are in Table 2.

A finite element model of the sample was created in ANSYS to simulate progressive loading. The boundary conditions are shown in Figure 12: at the point of contact with the lower supports, the sample was fixed in the Y direction, the central part of the sample in the YZ plane was fixed along the X axis, and in the YX plane, along the Z axis; the force was applied at the point of contact with the upper supports. 3D elements with specified properties in the local coordinate system were used.

The comparison of the numerical and experimental results of longitudinal strain at point 1 (Figure 12) is shown in Figure 13. The following points are marked: A - the moment of initial fracture and B -- the moment of 50% decrease in the load. Up to A, the deformation is close to elastic. In section A-B, progressive fracture is clearly visible.

There is good agreement between the results of finite element modeling and experiment up to the initial fracture moment of the SiC/SiC composite.
4. Conclusion.
The C/SiC and SiC/SiC composite samples were fabricated using PIP technology. Ultimate tensile strain for C/SiC at room temperature was ~0.11%. Ultimate flexural strain from the four-point bend test for
SiC/SiC at 1000°C was ~ 0.22%. It was demonstrated that up to the initial failure moment, the C/SiC and SiC/SiC are subjected to linear strain, and the progressive fracture is characteristic only of SiC/ SiC. Numerical modelling of the microstructure and loading compared to the strain gauge readings allowed obtaining the mechanical properties of the ceramic composites. With the proposed models, the sample strain can be predicted with a high degree of accuracy up to the initial failure moment.

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