

Development of Manufacturing Technology of Nozzles from Composite Materials during Radiation Cooling

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Abstract— Carbon-carbon composite materials (CCCM) are a new class of structural materials designed to create light, durable, and space-based rigid structures for aerospace, hypersonic, gas turbine engines, parts of nozzle blocks, rocket combustion chambers, and transition trusses. They have the unique ability to maintain high strength and stiffness with 2500°C. For the manufacture of thermally stressed parts of nozzles, carbon fiber fabrics are used. Antioxidant coatings based on tantalum and silicon are applied to nozzles with adhesive (made based on tantalum carbide, which provides high mechanical strength of adhesion of erosion-resistant antioxidant coating with CCCM), antioxidant and erosion-resistant layers. To deposit a layer of tantalum carbide on CCCM, we used the technology of vacuum ion-plasma deposition of tantalum film followed by carbonization with carbon. As a result, an adhesive coating of tantalum carbide is formed on the surface of the packing. Obtaining an erosion-resistant antioxidant coating on the adhesion layer of tantalum carbide is obtained by applying a slip of silicon carbide powders and soot. The study of the performance of antioxidant and erosion-resistant protective coating in high-temperature airflow was carried out on plasmatron (it is a plasma generator in which an electric current is used to form a plasma; for cooling it, channels washed by water are used). This article shows that the studied packings have a unique ability to maintain high strength and rigidity with 2500°C. There is the expediency of manufacturing such nozzles. The physical and mechanical characteristics of the material were determined.

Keywords—Carbon-carbon composite material (CCCM); model nozzle; erosion-resistant composite material; physical and mechanical characteristics; design documentation; radiation cooling nozzle (RCN).

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I. INTRODUCTION

One of the important problems of creating designs for various types of engines is the development of new materials used to manufacture the most loaded parts operating under the influence of highly enthalpy products of combustion of liquid components. The development of heat resistant material for the nozzle extension, antioxidant coating, and change in the geometry of the inner surface of the nozzle extension will increase the efficiency of the engines of the upper stages. Heat-stressed structures must be protected from high-temperature gas corrosion and erosion. Traditionally used coatings based on silicon and corundum can significantly increase the operating temperature of structures. Concerning the heat resistance of materials, structural graphite CCCM with the highest specific strength and rigidity up to 2800°C, high heat resistance, impact strength, corrosion resistance, resistance to radiation exposure, and low-temperature linear

expansion coefficients are very interesting for designers. Reducing the weight of the structures causes the use of CCCM in the hot elements of the design products of rocket and space technology.

CCCM is a new class of structural materials designed to create light, durable, space-based rigid structures for aerospace, hypersonic, gas turbine engines, parts of nozzle blocks, rocket combustion chambers, and transition trusses. They have the unique ability to maintain high strength and rigidity at temperatures up to 2500°C. For the manufacture of thermally stressed parts of the nozzles, carbon fiber fabrics are used.

The nozzles are coated with antioxidant coatings based on tantalum and silicon. The investigated nozzles are coated with adhesive, antioxidant, and erosion-resistant layers. The adhesion layer is made based on tantalum carbide, which provides high mechanical strength of the adhesion of the erosion-resistant antioxidant coating to CCCM. To deposit a layer of tantalum carbide on CCCM, the technology of

vacuum ion-plasma deposition of a tantalum film on CCCM was used, followed by carbonization with carbon. As a result of heat treatment, an adhesive coating of tantalum carbide is formed on the packing surface. Obtaining an erosion-resistant antioxidant coating on the adhesion layer of tantalum carbide is obtained by applying a slip of silicon carbide powders, soot. The technology for creating an antioxidant coating includes forming a silicon carbide coating and forming an outer coating in the form of zirconium diboride.

The study of the performance of an antioxidant, erosion-resistant protective coating in a high-temperature airflow was carried out on a plasmatron. A plasmatron is a plasma generator in which an electric current is used to form a plasma. To cool the plasma torch, channels washed with water are used.

This article shows that CCCM packing under study has the unique ability to maintain high strength and rigidity at temperatures up to 2500°C. The expediency of manufacturing a nozzle attachment from CCCM is shown. The work has been carried out to determine the physical and mechanical characteristics of the material (new augmented abstract).

One of the important problems in creating designs for various types of engines is the development of new materials used to manufacture the most loaded parts operating under conditions of exposure to high-enthalpy products of combustion of liquid components. The development of a heat-resistant material of the nozzle attachment, antioxidant coating and change in the geometry of the inner surface of the nozzle attachment will increase the efficiency of the upper stage engines. Heat-stressed structures must be protected against high-temperature gas corrosion and erosion. Traditionally used coatings based on silicon and corundum can significantly increase the operating temperatures of structures. In terms of heat resistance of materials, special attention of designers is attracted by structural graphite, which have the highest specific strength and rigidity up to temperatures of 2800°C, high heat resistance, impact toughness, corrosion resistance, resistance to radiation exposure, and low-temperature coefficients of linear expansion. The reduction in the weight of structures leads to the use of CCCM in hot elements of design products in rocket and space technology.

To produce perspective engines, it is necessary to create a technology for manufacturing model RCN of a profiled shape and obtain physical and mechanical characteristics when testing nozzle material.

II. MATERIAL AND METHOD

A. Manufacturing technology of model RCN

During the process of testing the manufacturing technology of a model RCN of a profiled shape. It was found that there are delamination and folds of material passing into the body of the part in the area of the small end on some blanks. In order to reduce the likelihood of occurrence of these defects, the following technical measures have been implemented [1]-[3]:

- height of the technological allowance of the workpiece from the side of the small end has been increased with the corresponding refinement of technological agile tooling.

- shape and number of petals are adjusted, taking into account changes in the geometry of the workpiece and design of agile tooling.
- securing the position of the petals during molding and heat treatment of the workpiece nozzle.
- pre-testing of the petals placed on the mandrel with an elastic cuff with a degree of stretching in the initial position of at least 2.0 was applied.

To increase the height of the technological allowance from the side of the small end face of the nozzle, additional mandrel elements "Bush" (Figure 1) and "Washer" (Figure 2) were developed, which allow forming a cylindrical section of the technological allowance on the nozzle blank (Figure 3). An interchangeable (technological) ring is designed to ensure the fixation of the petals by the layout on the mandrel [4].

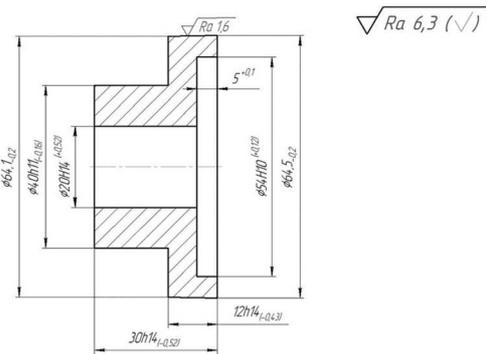


Fig. 1 Bush

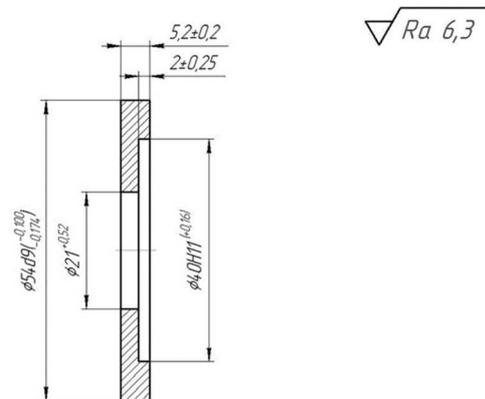


Fig. 2 Washer

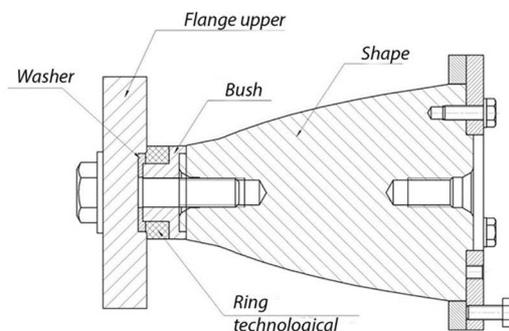


Fig. 3 Mandrel assembly

A modified mandrel for the manufacture of carbon fiber billet is presented in Figure 4.



Fig. 4 Modified mandrel for the manufacture of carbon fiber billet

The total number of petals placed on the mandrel is 101. Petals are pre-glued to 5-point packets with a given step between the petals (Figure 5). The last package consists of 6 petals. The total number of packets is 20.

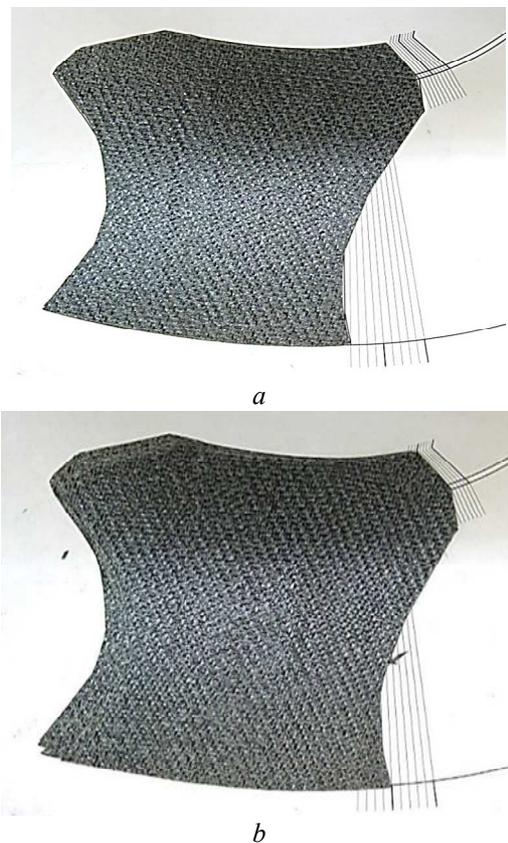


Fig. 5 Layout of the petals according to the layout in the package
a - first petal, b - package of 5 petals

Packing of petal packages is carried out following a given way for marking in the area of the large and small diameter of the mandrel with the rigid fastening of each package to the process ring with clamps (Figure 6).

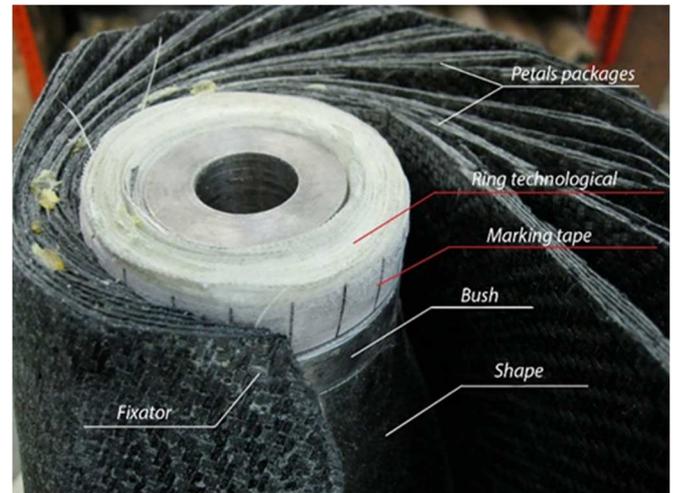


Fig. 6 Laying material packages on a mandrel

Thus, model RCN for testing as a part of model power plants were manufactured taking into account the developed proposals included in the technological process. To conduct experimental studies, 6 profiled shaped model RCN were manufactured. Antioxidant coatings based on tantalum and silicon were applied to them.

B. Manufacturing technology of model RCN

The studied coating composition consists of adhesive and erosion-resistant antioxidant layers. The adhesive layer is made on the basis of tantalum carbide, which provides high mechanical adhesion to the erosion-resistant antioxidant coating with CCCM of the nozzle and is selected based on the high melting point [5]-[7]. The technology of vacuum ion-plasma deposition of a tantalum film on CCCM with subsequent carbonization by carbon from CCCM was used to deposit a high-quality tantalum carbide layer [8], [9]. As a result of heat treatment, an adhesive coating of tantalum carbide is formed on the surface of the nozzle. The process of obtaining an erosion-resistant antioxidant coating on an adhesive layer of tantalum carbide includes the following [10]:

- preparation of a slip from a mixture of powders of Si, C, Hf, B₂, and soot;
- applying a slip on the surface of the nozzle with a soft brush in one layer and drying, as well as siliconizing the coating by capillary saturation with liquid silicon in a vacuum oven.

The technology for creating an antioxidant coating includes two stages: forming a silicon carbide coating in a vapor-gas medium of silicon monoxide and forming an external coating in the form of predominantly zirconium diboride. The formation of silicon carbide coatings is carried out in a vacuum induction furnace with a residual argon pressure of 0.5 atm at a temperature of 1750 °C. Heat treatment of the outer coating is carried out in a nitrogen atmosphere [11]-[13].

III. RESULTS AND DISCUSSION

A. Manufacturing technology of model RCN

The study of the performance of the antioxidant protective coating under conditions of high-temperature airflow was carried out on a plasmatron on samples of $\text{O}40 \times \text{O}30 \times 10$ mm. At the same time, the density, open porosity and thermal conductivity of the material of the nozzle with a protective antioxidant coating were determined. In addition, the thermal stability of the coating was evaluated under the influence of a high-temperature gas flow.

Samples were made of composite material, corresponding in composition to the material of the nozzle, in the form of a plate $150 \times 150 \times 15$ mm. Upon receipt of the material and the coating on the samples, the heat treatment of the plate was carried out jointly in the manufacture of model RCN. To study the density, porosity and thermal conductivity, the coating on the samples was formed from the end surfaces. For gas-dynamic tests, the coating was formed on all surfaces of the samples [14], [15]. The test results of samples with antioxidant coatings are presented in Table 1. Both types of coatings can be perspective for nozzles with a surface temperature during operation up to 1800°C .

TABLE I
TEST RESULTS OF SAMPLES WITH COATING ON THE PLASMATRON

Sample coating	Tantalum-based	Silicon-based
Exposure time, s	1 000	1 000
Crater depth, mm	+0.13	+0.1
The initial mass of the sample, g	18.88	16.49
Loss of mass of the sample, g	0.00	+0.05
Maximum sample surface temperature, $^\circ\text{C}$	1 820	1 800

B. Study of PMC of CCCM

To study PMC of a material based on carbon fabric, two $400 \times 330 \times 4$ mm plates were manufactured according to the manufacturing mode of CCCM based on carbon fibres-900 (CF-900). It was impregnated with a phenolic binder. Carbon fibers were manufactured by hot pressing. The number of tissue layers in the plate is 20 (5 layers per 1 mm). The scheme of cutting plates into samples is shown in Figure 7.

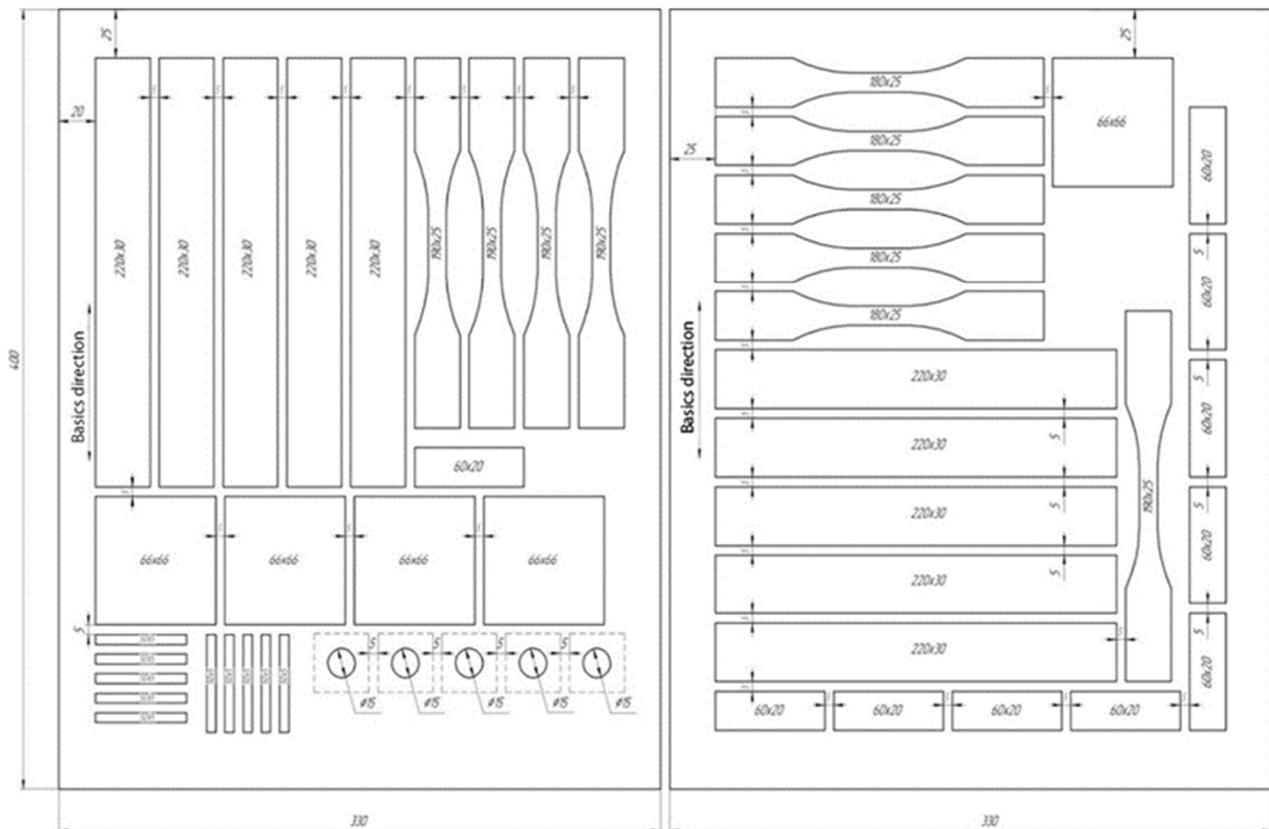


Fig. 7 Scheme of cutting plates into samples

Then carbon fiber underwent carbon redistribution. This changes the mass and thickness of the plates. PMC of CCCM was determined from experimental studies conducted based

on the universal testing machine "Instron". The location of the samples on the installation is shown in Figure 8.



Fig. 8 Sample location before testing

The level of performance achieved in comparison with existing analogs is shown in Table 2.

TABLE II
PMC OF CARBON MATERIALS

Material	Carbon fabric (90-degree filament angle)	Proposed	Carbon fabric (45-degree filament angle)
Matrix	Phenolic resin coke and pyrocarbon		
Tensile stress tensile along the base, MPa	124	153	260
Tensile modulus along the base, MPa	21	82.7	75
Breaking stress during compression along the base, MPa	145	199	140
Poisson's ratio along the base	0.155	0.051	0.1
Density, kg/m ³	1.32	1.53	1.45
Thermal conductivity coefficient at 20±5 °C in the direction perpendicular to the layers, W/(m·K)	2.5	2.0	1.4÷2.8
Coefficient of linear thermal expansion, 10 ⁻⁶ K ⁻¹ at a temperature, T °C	the direction of coefficient of linear thermal expansion measurements		
100	1.37	1.35	1.3
300	1.77	1.80	1.84
500	2.09	2.13	2.15
900	2.53	2.59	2.63

IV. CONCLUSION

The studies showed that the model RCN with a profiled shape with two types of antioxidant coatings made according to the above-mentioned technology could be considered promising engines with a nozzle at a surface temperature during operation of 1200 °C. Analysis of the physicomechanical characteristics obtained during experimental studies carried out on a tensile testing machine showed a significant improvement in stress indicators for a

tantalum sample of 124 MPa and a sample based on silicon it is twice as high, in the modulus of elasticity of 21 MPa for tantalum, a silicon base is three times higher. Times higher, in terms of the coefficient of thermal conductivity, the samples based on tantalum and silicon have the same values.

The presence on the surface of the sample of heat-resistant antioxidants made of a mixture of Si, C, Hf, B₂, and soot did not lead to a change in the material's thermal conductivity. Tests on a plasmatron showed that composite samples based on tantalum are more resistant to mass carryover. There is practically no carryover from the tantalum sample. The weight loss from the silicon-based sample is 0.05 g, although the specimens based on tantalum withstand higher temperatures of 1820°C based on silicon 1800°C.

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