Development verification of coatings made from porous ceramic-matrix composite materials

Sergey Reznik^{1,}, Pavel Prosuntsov¹, and Konstantin Mikhaylovskiy¹*

¹Bauman Moscow State Technical University, 105005, 5/1, 2-nd Baumanskaya str., Moscow, Russia

Abstract. The paper presents methodology and results of tests conducted for porous carbon ceramic specimens on a high-temperature gas dynamic facility to simulate real operating conditions for heat shields. The structure of the materials before and after tests is compared.

1 Introduction

Ablative heat-shield materials are traditionally used to protect the load-bearing structure of re-entry space vehicles. At present, PICA is considered to be the best heat shield material. PICA, which stands for Phenolic Impregnated Carbon Ablator, is composed of a porous carbon base and phenol-formaldehyde resin. However, studies conducted at NASA Langley research center [1] show that the use of carbon materials such as ACC-6, together with high porosity thermal insulation, makes it possible to create more weight-efficient designs. Ceramic matrix composites (CMC) reinforced with carbon fiber, despite their high oxidation-resistance and erosion-resistance and good mechanical characteristics, possess high density and thermal conductivity, which reduces their attraction as heat-shield materials. The most perspective direction in heat shield development is associated with porous CCM [2, 3] that combine the effective density in the range 1200 - 1800 kg/m³ and a substantially lower thermal conductivity coefficient [4, 5]. However, to date there is no experimental evidence that porous CCM can withstand the high-enthalpy air flux.

2 Test objects

The test objects were samples of porous ceramic matrix composite materials with the apparent density 1200-1800 kg/m³, reinforced with ceramic fibers. The samples' microstructure was investigated using the PhenomProX optical microscope. The images of CMC with the 1200 kg/m³ apparent density (Fig. 1a) clearly show carbon fibers 20 µm in diameter, covered with a 1 micron crust of silicon carbide. There are pores, 60 to 100 µm size, between the fibers. The images of the material with 1400 kg/m³ density (Fig. 1b) clearly show fiber microstructure with 2 to 3 µm silicon carbide crust. The characteristic pore size between the fibers, compared to the 1200 kg/m³ sample, decreased and became 50 to 70 µm. A sample of 1600 kg/m³ material (Fig. 1c), compared to less dense samples,

* Corresponding author: sreznik@bmstu.ru

looks much more monolithic with individual local pores. With the same diameter carbon fibers, the thickness of the silicon carbide crust increased up to $4\text{ }\mu\text{m}$. The pores are smaller, in the range of 30 to $40\text{ }\mu\text{m}$. A sample of 1800 kg/m^3 material (Fig. 1d) looks almost monolithic. Individual fibers are almost indistinguishable. The thickness of the silicon carbide crust increased up to 8 microns. The characteristic pore size in the sample does not exceed $20\text{ }\mu\text{m}$.

3 Test equipment

The CCM samples were tested at the high-temperature gas dynamic test facility with a high-frequency BTV-4 (VGU-4) plasmatron at the Ishlinsky Institute for Problems in Mechanics of the Russian Academy of Sciences (Moscow) (Fig. 2) [6-8]. The VGU-4 plasmatron in the standard subsonic mode enables the pressure range in the test chamber from 600 Pa to atmospheric pressure and the enthalpy range from 10 to 40 MJ on the jet axis.

Fig. 1. CCM surface microstructure KMKM (380x magnification optical microscope):

a – apparent density 1200 kg/m^3 ; b - 1400 kg/m^3 ; c - 1600 kg/m^3 ; d - 1800 kg/m^3

Fig. 2. High-temperature gas dynamic test facility with VGU-4 high-frequency plasmatron

4 Test methods

Thermal tests were conducted using test coupons: circular plates with 26.5 mm front face diameter, a conical side surface with 30° divergence half-angle and 4.0 mm thickness. The coupons were heated incrementally up to 1720°C and subsequent held at this temperature. The temperature of the frontal surface of the samples was measured using the "Tandem VS-415U" thermometer calibrated according to the model of an absolutely black body in the 700° to 2300°C temperature range.

Fig. 3 shows a typical thermal image of the investigated CCM sample surface. In the course of the experiment, the display showed the maximum surface temperature and the temperature distribution as functions of time. The maximum temperature was specified for a rectangle selected on the surface and the temperature distribution was taken along the scanning line in Fig. 3. Fig. 4 shows the main parameters of the plasmatron operation and the surface temperature as functions of time. The surface temperature was measured by a spectral -ratio pyrometer and a thermal camera.

Fig. 3. Coupon thermal image and areas where the maximum temperature was measured

Fig. 4. Plasmatron operating parameters and surface temperature as functions of time; measurement by a spectral-ratio pyrometer and a thermal camera: *T* – temperature, °C; *N* – energy, kW; *P* – pressure, GPa

5 Test results

To illustrate test results, let us consider a CCM sample with 1600 kg/m³ apparent density. The test coupon was incrementally heated for 360 s up to 1720°C and was held at this temperature for 420 s. The total mass ablation was 0.8091 g (19.2%), with zero linear ablation. The surface remained uniform, without any visible fiber ablation areas (Fig. 5). The front surface was coated with a silicon dioxide crust found to be intact (Fig. 6). Fibers’

shapes were visible beneath the crust, and single fibers ablated. This CCM sample has passed the tests successfully. No failure occurred, even though in the last two minutes local failure origins were observed in the form of rapid (a few seconds long) formation and disappearance of the local overheating zones, which is typical of silicon carbide materials and coatings.

Fig. 5. Front surface of the test coupon with 1600 kg/m³ apparent density: a – before test; b – after test

Fig. 6. Surface of the 1600 kg/m³ after thermochemical resistance tests (magnification 400x)

6 Conclusion

The tests of porous CCM samples conducted on the VGU-4 plasmatron demonstrated that this class materials with 1600 kg/m^3 apparent density can successfully withstand high-enthalpy air flux and, consequently, is suitable for reusable heat shields.

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