Experimental Investigation of Thermo-barrier Coating Based on Zirconium Dioxide

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Abstract

One of the main problems in design and manufacture of a high-speed civil vehicles, an aerodynamic models or an experimental facility equipment, subjected to withstand a high-temperature loads, is a combination of optimal choice of construction materials and methods of their protection against the destructive influence of high temperatures. Because of a maximum melting point of most applicable materials usually remains insufficient for operation in a high temperature zones, such as a nose part, wing or tail leading edges of a vehicle or aerodynamic model, arises the necessity of usage of a thermo-barrier heat-resistant coating on the edges or other outer curvilinear surfaces of the constructions subjected to the high temperature. The thermo-barrier coating is selected in terms of its structural compatibility with used materials, required protective and mechanical characteristics. To approve the applied thermo-barrier protective coating method, a series of experimental investigation at a high temperature research facility with a samples made from same material as the reference construction and covered with the thermo-barrier coating are provided.

Keywords: *Investigation, Thermo-barrier, Coating, Zirconium Dioxide*

Nomenclature

T – temperature
P - pressure
K - kelvin
M – Mach Number
ZrO2 – zirconium dioxide

1. Introduction

In the space-rocket industry a various thermo-barrier coating systems are used to protect the vehicle critical parts against intense heating during its flight in dense layers of the atmosphere, as well as against the high-velocity gas-dynamic flow of high temperature combustion products. The coating must ensure that a significant part of the heat flow is absorbed and removed from the protected surface. The most effective and accessible coating at that moment is zirconium dioxide, applied to the protected material by a gas-plasma spraying method.

For study purposes of zirconia dioxide characteristics, its sufficient thickness of the covering layer and the possibility of application at high-speed civil vehicle production, TsAGI together with one of the leading research and production centers in the field of gas-plasma coatings Institute of Physics Problems of the RAS, carried out a series of experimental investigations of the thermo-barrier coating system based on dioxide zirconium.

Research object is a zirconium dioxide thermo-barrier coating, created by high-energy plasma powder spraying method. The coating is sufficient to provide the protection of the experimental high-speed

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vehicle against high temperatures up to 2500K, which occur when vehicle entering the atmosphere at high speed up to M=10. Because of it, one of the crucial problems of the project feasibility is the optimal choice of the materials for the vehicle construction and its protective coating method.

The typical package of thermo-barrier coating based of zirconia dioxide consists of two layers: the external one, allowing exposure to high temperatures for a specified time and the internal one, having a low thermal conductivity coefficient.

The procedure to study the zirconia dioxide coating contains the deposition technology development, as well as the combined mechanical and thermal tests of coating material samples.

2. Technology and equipment for high-energy plasma powder spraying of intermetallic and ceramic coatings

The essence of the method of high-energy plasma deposition is heating and melting of powder material by a plasma stream (Fig. 1). The deposed material enters the dispersed state in the form of small fused particles and striking towards the surface of the substrate, where fixing on it and lying one upon another, forming a protective coating.

The method of high-energy plasma spraying is a modification of the standard gas-plasma technology and characterized by increased energy characteristics of the plasma flow (V = 2400 m/s, T = 5000 - 10000K) [1], which leads to heating of the applying particles up to the melting point, compacting the deposited layers and increasing strength of their adhesion to the treated surface. Due to the high energy input to the processed material, the conditions for the formation of heat-resistant intermetallic and thermo-barrier ceramic coatings are realized [2].

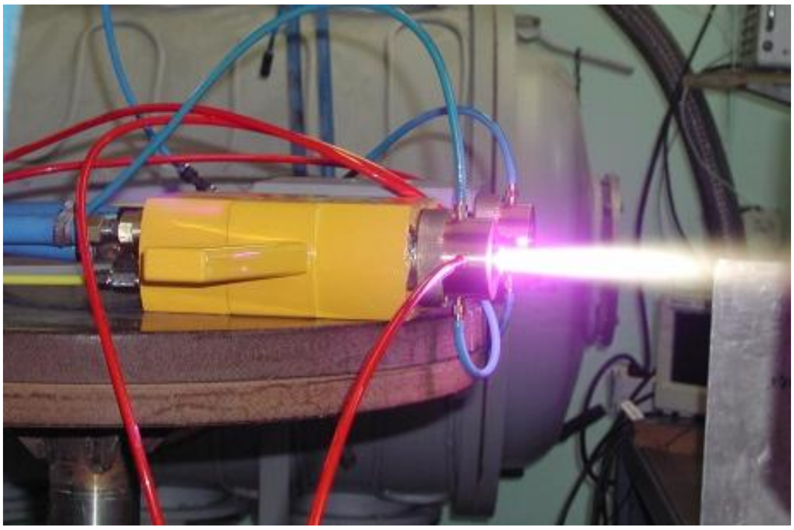

Fig. 1. Installation of plasma spraying of powder materials

Powdered mixtures of domestic production with a spherical particle shape (in the form of solid and hollow microspheres) with a different content of the yttrium oxide (Y_2O_3) additive and particle size of the main fraction of ~80 micron were tested for plasma deposition of the thermo-barrier coating. The phase composition of the powders was zirconia with tetragonal type (T-ZrO_2) of crystal lattice.

The highest hardness and lower roughness of the surface is provided by a thermo-barrier coating deposited from a powder mixture (ZrO_2 + 7% Y_2O_3) consisting of solid spherical particles (Fig. 2). Comparative sclerometric tests showed that it also has the best strength properties. This coating was chosen as optimal for application to test samples.

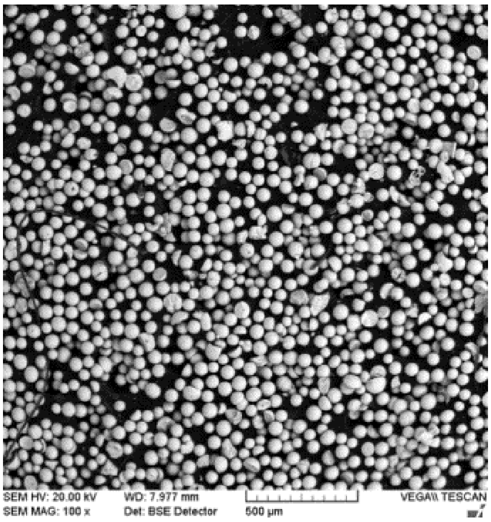

Fig.2. The granulometric composition of the mixtures ZrO2 + 7% Y2O3 with a spherical shape of whole particles (X100)

3. Test sample material and zirconium dioxide coating

For the experimental investigation, two materials for covering were selected. The first one is made from VT20 titanium alloy with thermal protection coating based on zirconium dioxide (ZrO2) on the intermetallic sublayer of the Ni-Co-Cr-Al-Y, another one is made from M1 copper alloy with the same multilayer covering.

VT20 is an alloy based on the Ti-Al-Zr-Mo-V system. Its density is 4.5 g/sm3. The alloy has high corrosion resistance under atmospheric condition and its corrosive media is high. VT20 is recommended for construction that operates under long-term temperatures up to 500°C and short-term up to 800°C.

M1 Copper alloy contains 99.9% copper. This type of metal has high values for electrical and thermal conductivity. The M1 alloy has a high compressive strength and excellent plasticity. Its density is 8.9 g/sm3.

Based on the specified materials, Institute of Physics Problems of the RAS matched and optimised the microstructure parameters of zirconium dioxide layer and its internal intermetallic sub-layer (Fig. 3). As a result, the multilayer with antioxidant and degreaser layers protective zirconium dioxide coating with following characteristics was obtained (Table 1).

Table 1. ZrO2 coating structure tests results, based on mechanical tests

Layer properties	**Value**
Common porosity,	5
Surface porosity, %	1,5
Density, g/sm3	6,8
Hardness HV, kgf/mm2	950
Roughness Ra, micron	~7

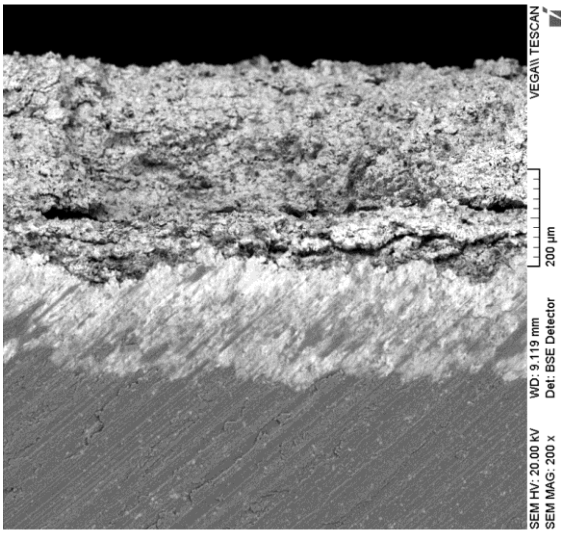

Fig. 3. ZrO2 coating microstructure

Following the local temperature and mechanical studies of the ZrO2 protection method it is shown that this thermo-barrier coating has high density and hardness values with a low porosity. Obtained complex of physic and mechanical properties is necessary to provide such important operational characteristics as heat resistance, erosion resistance and thermal protection efficiency.

4. Test samples and experimentation work chamber

To determine the sufficient thickness and investigate temperature characteristics of the multilayer that operates under conditions similar to the natural ones, it was decided to manufacture ZrO2 coated samples from VT20 titanium alloy and M1 copper alloy and test them in the TsAGI T-131 aerodynamic high temperature facility (Fig.4).

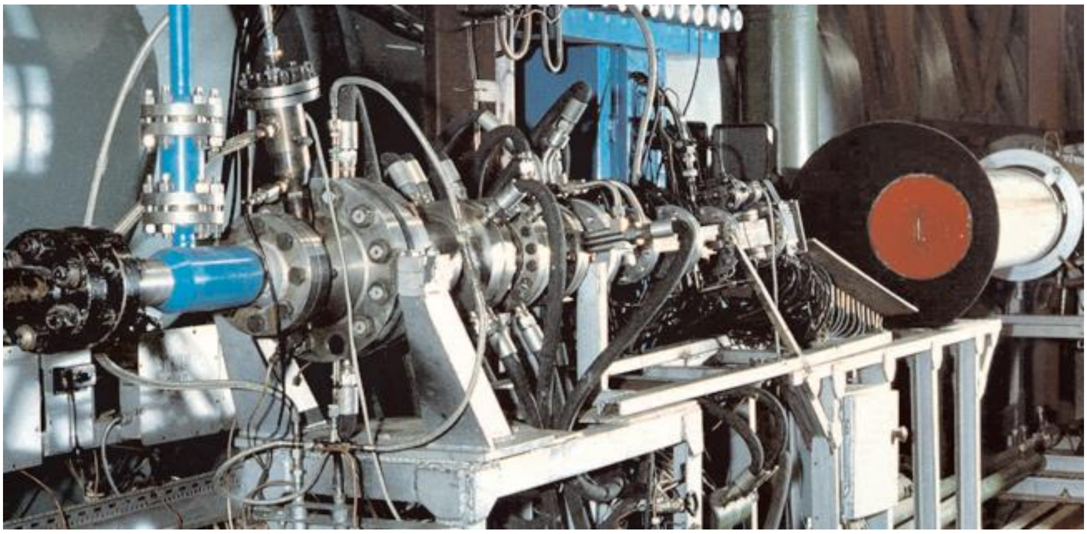

Fig. 4. TsAGI T-131 aerodynamic high temperature facility

Tests of thermo-barrier coated samples were carried out at the T-131 aerodynamic facility, designed for following research:

- working processes in models of ramjet air-jet engines;
- processes of mixture formation and combustion of various fuels in subsonic and supersonic flows;
- thermal conversion of hydrocarbon fuel;
- heat-shielding and structural materials;
- models of high-speed WFDs in free flow;
- burning on the external surfaces of aircraft;
- air intakes WFD.

The installation is provided by the following systems:

- air supply system of high (up to 20 MPa) and low (up to 1.2 MPa) pressure;
- high pressure oxygen and nitrogen supply systems (up to 20 MPa);
- hydrogen supply system (up to 15 MPa);
- fuel system (up to 12 MPa);
- water cooling system (up to 2.0 MPa);
- measuring system.

The key element of the T-131 stand is the gas-flame type kerosene air heater.

The air heater combustion chamber is supplied with air, oxygen and kerosene in quantities necessary to create a flow with predetermined braking parameters T and P. It should be noted that oxygen is fed into the combustion chamber of the air heater to replenish the burnt oxygen of the air. The latter requirement is important for modeling atmospheric air in tests where combustion processes are studied. Such a method of compensation also ensures the high completeness of kerosene burning.

As fuel in the air heater, kerosene is used with a relative weight composition: 13.4% hydrogen and 86.6% carbon.

The air heater works steadily in the temperature range T0 = 850 - 2350 K and pressures up to 10 MPa. The upper limit of the working area on the pressure and the gas temperature in the air heater is currently limited to the maximum available pressure in the fuel system Pt = 10 MPa. The lower limit is determined by the minimum possible differential pressure on the fuel injectors.

The characteristics of the air heater determine the flow parameters in which the sample is tested. Therefore, it is necessary to pay special attention to the correct determination of the flow parameters at the outlet of the air heater.

For the first test runs, four plates of material VT20 and two plates of the copper alloy M1 were made (Fig.5).

To place the samples in harsh conditions of high temperature environment made by hot air stream, an experimental module was designed and manufactured for integration within the TsAGI T-131 facility system.

The module consists of two chambers – a working chamber, where samples are placed, and an air-cooling one, where the hot air is cooled and blown out into atmosphere (Fig.6).

Fig. 5. VT20 and M1 copper test plates

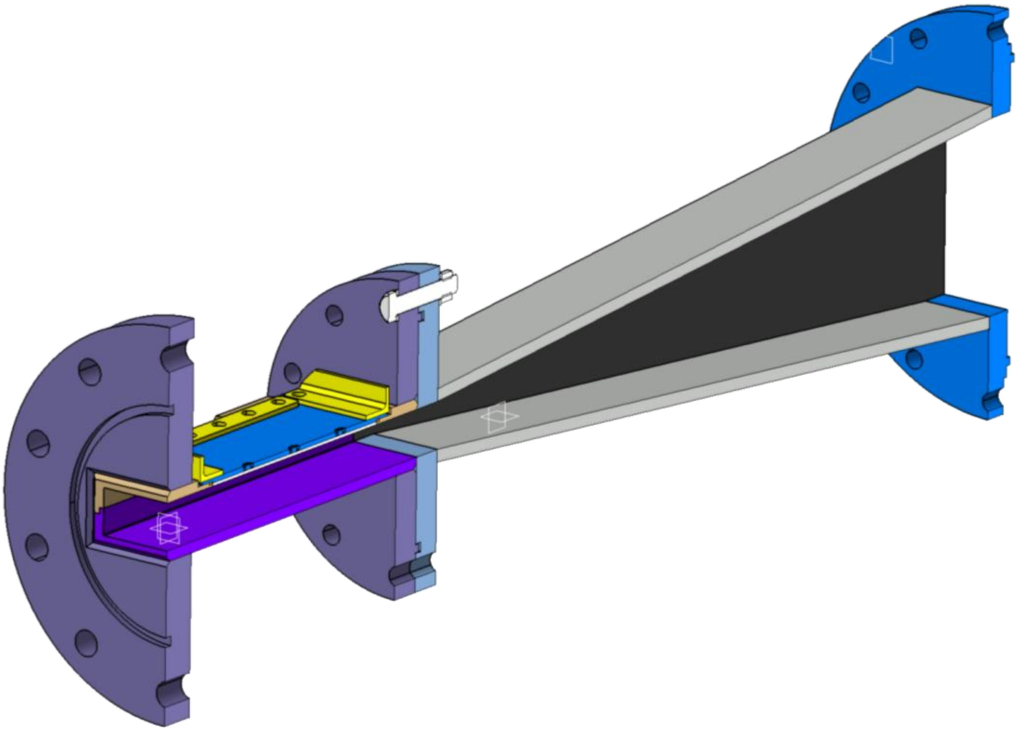

Fig. 6. Experimental module with working and air-cooling chambers

The working chamber is a welded lid made of heat-resistant steel forming a rectangular section of 100x30 mm with a cut-out on the upper lid, where the test samples are mount. The samples are pressed by bolts to the lid using corners of stainless steel, providing rigidity.

The air-cooling chamber is made of weld stainless steel plates.

Chambers connection between themselves and the T-131 facility system is made of stainless steel flanges.

After sample are made, the Institute of Physics Problems of the RAS covered the test plates with zirconium dioxide multilayer thermo-barrier coating of 2mm layer (Fig.7).

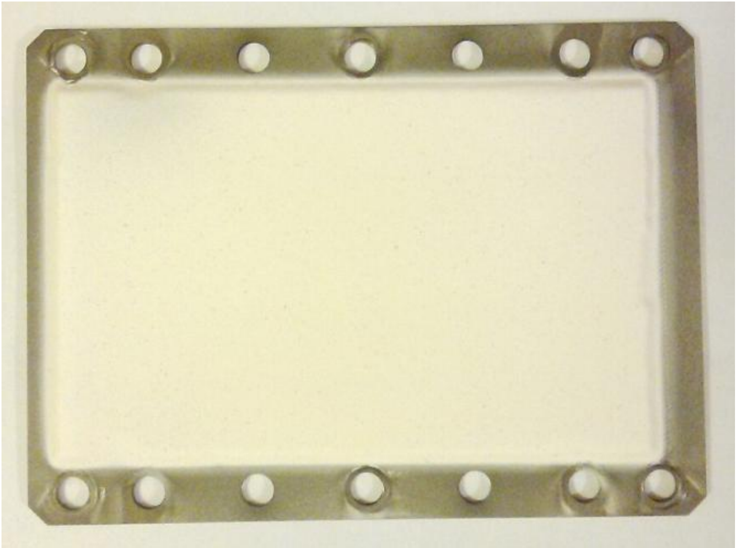

Fig. 7. VT20 test plate with ZrO2 multilayer coating

For the second test runs, it was decided to make samples of wing leading edge imitation with 1 mm radius from VT20 titanium alloy and also cover it with 2mm zirconium dioxide multilayer thermo-barrier coating (Fig.8).

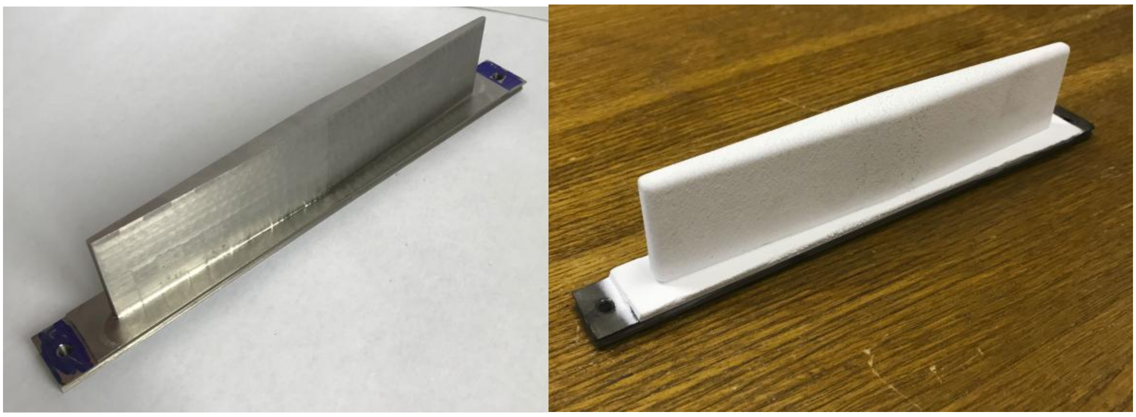

Fig. 8. VT20 test sample with leading edge imitation without and with ZrO2 multilayer coating

5. High temperature tests in T-131 TsAGI facility

To obtain a pattern of pressure distribution inside the working chamber with installed samples before main runs, a special measuring stainless steel dummies was made and tested in T-131 facility (Fig.9). In parallel to that, the technology of installing thermocouples on test samples was also tested. According to the obtained pressure distribution scheme and the developed installation technology, 9 chromel-alumel thermocouples with a diameter of 0.3 mm were installed on the plate samples on different depth and 3 chromel-alumel thermocouples were installed on sample that imitates the wing leading edge at half of the leading edge height along its length (Fig.10). Four rhenium-tungsten thermocouples of 0.35 mm diameter were also installed on experimental module walls.

Fig. 9. Stainless steel dummy plate with 10 mm pressure probes

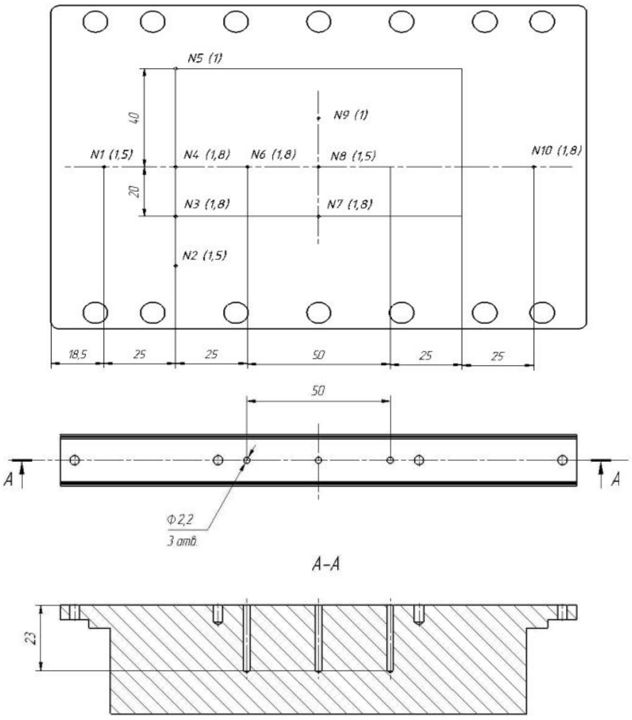

Fig. 10. Test plate and wing leading edge imitation samples thermocouples positioning

In the debugging tests it was found that the total pressure in the preheater at the stationary mode is 12 atm, and the total pressure in front of the sample is about 6 atm. The high-speed head is 280 kPa, which is much higher than expected in flight. Thus, at the stand, the conditions that are significantly worse for the thermal protection coating are simulated than in the flight.

For test runs in T-131 facility the following operating parameters were provided: Mach number of the air stream is 3, total pressure is 11 MPa and temperature is up to 2000K, operation time of one run from 40 to 60 seconds.

Totally there were made 4 runs for each test plate (Table 2) and 2 runs for sample that imitates the wing leading edge (Fig.11).

Table 2: 1st run results for VT20 test plate

1st run	Test plate middle termocouple	Chamber flank termocouple	Chamber bottom termocouple
t0C start	19,6	17,5	13,2
t0C end	558	511	680
dt0C	413,8	471,1	682,8
T start	10:23:01	10:23:00	10:23:00
T end	10:24:02	10:23:47	10:23:47
dT, sec	61	47	47

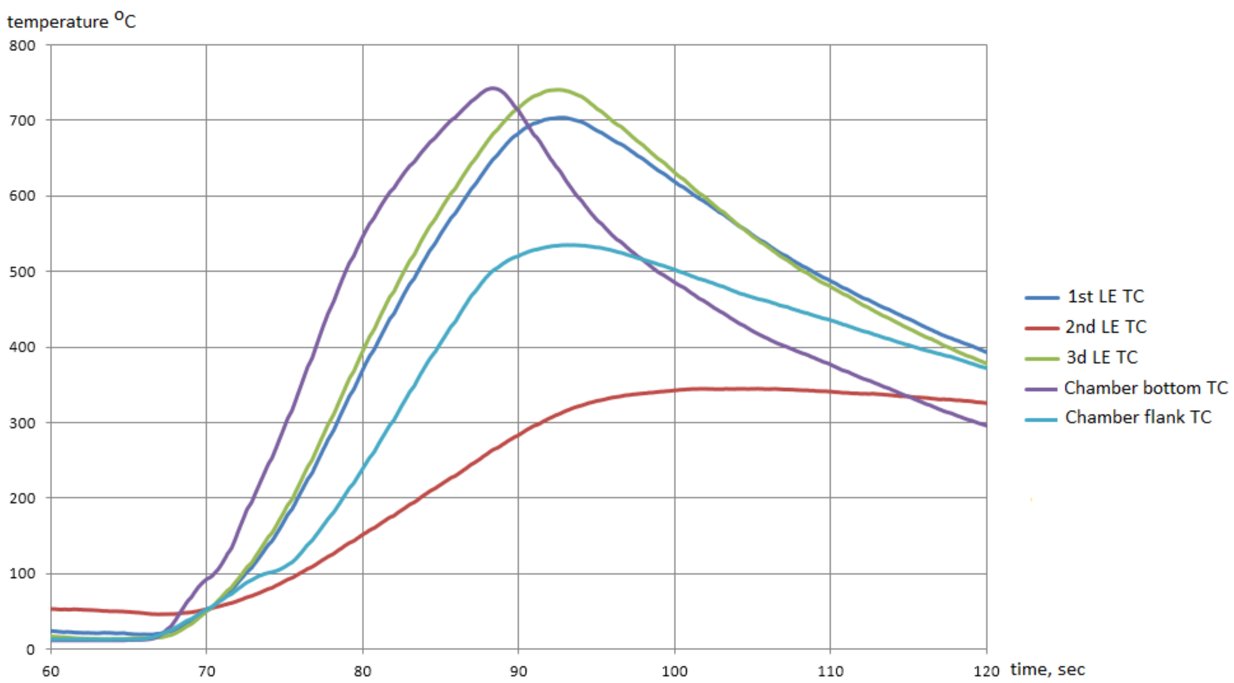

Fig. 11. Usage of thermocouples (TC) when simulating the wing leading edge (LE)

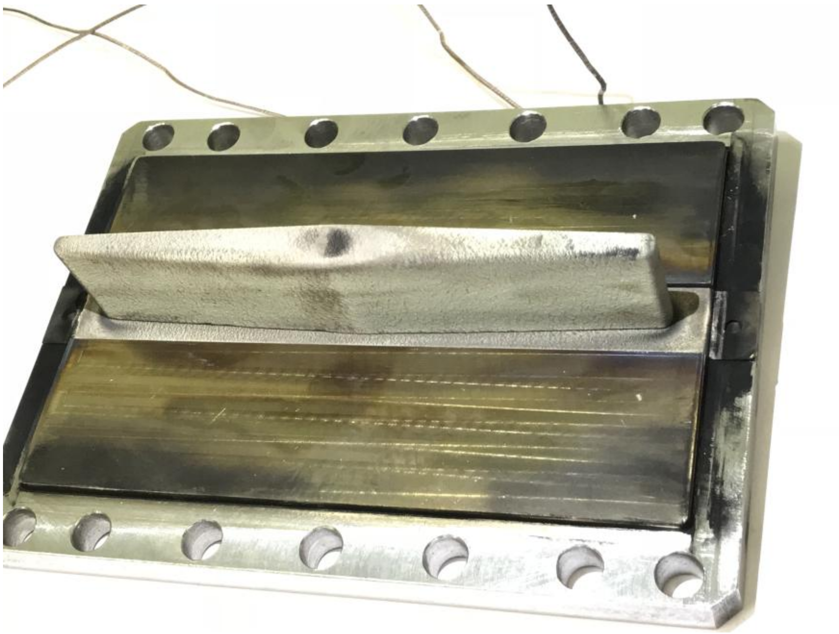

Fig. 12. Sample with leading edge imitation after test runs

6. Results

The thermo-barrier coating of zirconium dioxide formed on the intermetallic sublayer of the system "Ni-Co-Cr-Al-Y" is designed for high-temperature protection of the titanium alloy VT-20 and copper alloy M1. The zirconia coating has a spheroidal surface morphology and a layered microstructure in cross-section with a columnar grain structure in the interlayers, has low porosity and high hardness.

According to the results at the T-131 facility test, it was found that after carrying out high-temperature tests, the multilayer thermo-barrier coating of plate samples retained the stoichiometric and phase composition, as well as the surface morphology and internal columnar microstructure of the ZrO_2 grains.

The obtained data on the heat flux distribution over the thickness of the test samples material during the entire operation time make it possible to recommend the coating based on zirconium dioxide for use on high-speed vehicles and aerodynamic models as a protective coating against high temperature effects.

Acknowledgements

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