STUDY ON METAL HEAT-PROTECTIVE STRUCTURES OF REUSABLE AEROSPACE VEHICLES

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This study presents some results on solving the problem of creating heat-protective elements for reusable aerospace vehicles, which includes developing materials with necessary physical and mechanical characteristics, approbation of design solutions and models of heat-protective structures, testing them under conditions simulating operational ones. Using the powder metallurgy technology, an innovative heat-resistant high-temperature alloy (Ni20Cr5.95Al)-Y2O3 was developed. According to the results of comprehensive microstructural and XRD analyses, the developed alloy had the required physical and mechanical characteristics and heat resistance in the operating temperature required physical and mechanical characteristics and heat resistance in the operating temperature
range of 20–1200°C, which made it applicable for manufacturing heat-protective structures of the *orbital spacecraft windward part. A prototype of a three-layer heat-protective structure was designed and manufactured from the developed alloy, which consisted of four three-layer panels (165 165 mm in size) with an internal honeycomb filling, which edges were joined by U-shaped elements. The prototype design validation was carried out by constructing finite element models and analyzing their stress-strain state under maximum mechanical and thermal loads, which occurred during the launch and re-entry of the reusable spacecraft. Using a gas-dynamic test bench, the prototype was tested in a high-temperature gas flow under conditions simulating operational ones. In these tests, simulation of the external factors acting on the test element and the equivalence of material damage processes under simulated and operational conditions were provided. The test results obtained corroborated the underlying concepts used in the elaboration and design of the innovative heat-resistant alloy, as well as the stress-strain state assessment of a three-layer heat-protective structure.*

Keywords: powder-metallurgy heat-resistant alloy, aerodynamic heating, reusable space systems, thermostressed state, heat-protective structure.

Introduction. Global developers of space-rocket launcher systems pay much attention to heat-protective structures (HPS) of reusable orbital vehicles, which multiple re-usage has a number of advantages including higher cost efficiency of launching useful loads into orbits and avoiding the accumulation of "space debris".

One of the main problems faced in developing reusable HPS is to ensure their required strength under strict weight limitations. The most challenging problem is thermal protection of the orbital spacecraft windward surface, which permissible surface density is limited to 10 kg/m² and which total area (about 43% of the spacecraft surface) is exposed to temperatures of 1000–1100°C. is exposed to temperatures of $1000-1100$ °C.

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Fig. 1. (Ni20Cr5.95Al)-Y₂O₃ alloy microstructure upon rolling to thickness of 24 (a), 5 (b), 2 (c), and 1 mm (d).

At present, tiled HPS, which combine the spacecraft hull thermal protection with the load-bearing function, are considered to be the most promising and reliable design option. Their reliability and multiple reusability are provided by the application of external panels made of high-temperature heat-resistant alloys capable of withstanding up to 100 flights with controlled re-entry into the Earth's atmosphere. Satisfactory performance characteristics at up to 100 flights with controlled re-entry into the Earth's atmosphere. Satisfactory performance characteristics at
temperatures up to 1000–1100°C are exhibited by alloys based on nichrome with dispersed oxide-based harden phase, which are produced by the powder technology [1]. These include the PM-1000 superalloy of European production and the MA754 alloy (US). However, the application of the above alloys in HPS is restricted by their significant specific weight ($\sim 8300 \text{ kg/m}^2$). Given this, it is expedient to develop a high-strength metal material with a lower specific weight that can be used in a hi-tech external load-bearing panel of a reusable HPS.

The density reduction with a simultaneous high-temperature strength improvement can be ensured by the addition of aluminum into existing heat-resistant nichrome-based alloys, which would result in the strengthening (Ni₃Al) γ' -phase formation [2]. An innovative (Ni20Cr5.95Al)-Y₂O₃ powder-metallurgy alloy with a de 7500 kg/m³ was developed in joint efforts of the Frantsevich Institute of Materials Science of the National Academy of Sciences of Ukraine and the Yangel Yuzhnoye State Design Office. The alloy strengthening was provided by of Sciences of Ukraine and the Yangel Yuzhnoye State Design Office. The alloy strengthening was provided by adding 1% yttrium dioxide (Y_2O_3) with melting temperature of 2430°C, which improved its high-temperature perfo performance up to 1200° C.

The aim of this study was the experimental investigation of specimens and prototypes of a load-bearing HPS made of (Ni20Cr5.95Al)-Y₂O₃ alloy under conditions simulating the operational ones.

Problem Formulation. The research objects were Ni20Cr5.95Al)-Y₂O₃ alloy specimens of three different thicknesses (5, 2, and 1 mm) produced by cold rolling of sintered 24 mm-thick billets of a 7500 kg/m³ density, with subsequent annealing at 1200°C and cooling in air. The effect of plastic deformation on the material st subsequent annealing at 1200°C and cooling in air. The effect of plastic deformation on the material structure was assessed via an ML-2 luminescent microscope. Microstructural studies proved the composite structure of the material under study, as well as the presence of reinforcing yttrium oxide particles (from 1.5 to $40 \mu m$ in size), which were introduced into the charge (Fig. 1).

The specimen porosity in the sintered state was assessed as 4.6%, while the size of pores did not exceed ¹⁵ m (Fig. 1a). After cold rolling of billets to the thickness of 5 mm, their porosity remained practically unchanged (Fig. 1b). Rolling up to a 2 mm thickness (and especially to 1 mm) resulted in a pore size reduction and their partial

Fig. 2. The temperature dependence of the tensile ultimate strength in air (a) and vacuum (b) for Ni20Cr5.95Al)-Y₂O₃ alloy specimens with different thickness values: (1) 1 mm and (2–4) 2 mm.

Fig. 3. Cyclic heat resistance of (Ni20Cr5.95Al)- Y_2O_3 alloy foil.

closure. Upon rolling, multiple partially closed pores can be observed in the form of chains or separate small voids. The porosity of specimens after rolling to 1 mm dropped to 3.1%, which is quite low for powder-metallurgy materials and implies quite high strength characteristics.

Results and Discussion. Physicomechanical characteristics and heat resistance of the material under study were tested on its specimens. Experimental studies of the alloy strength were carried out in the vacuum in the were tested on its specimens. Experimental studies of the alloy strength were carried out in the vacuum in the temperature range of $20-1200^\circ\text{C}$ and in air at temperature up to 800°C (Fig. 2). The analysis of e temperature range of 20–1200°C and in air at temperature up to 800°C (Fig. 2). The analysis of experimental results revealed that at temperatures of 20–400°C, the ultimate strength σ_u reached 950–1000 MPa. When heated the value of σ_u slightly dropped to 600–800 MPa, which implied the alloy hardening by γ' -phase dispersed particles in the latter temperature range. At higher temperatures, the ultimate strength dropped to $56-70$ MPa at 1200° C. Noteworthy is that the hardening phase, which ensured high enough strength characteristics of the material at Noteworthy is that the hardening phas $900-1200^{\circ}C$, is yttrium dioxide (Y_2O_3) .

The results of alloy tensile tests in air and vacuum were practically the same, which proved its high heat resistance at elevated temperatures. This finding was corroborated by heat resistance values determined via the weight variation of the material upon repeated heating and cooling of foil specimens with dimensions of weight variation of the material upon repeated heating and cooling of foil specimens with dimensions of $27 \times 15 \times 0.05$ mm. Heating was carried out in the electric furnace at 1200°C for 20 min, which simulated the HPC material operating conditions.

The alloy weight variation with the number of cycles is shown in Fig. 3. During the first cycles, no weight variation was observed, which implies a low oxidation rate and formation of an oxide layer in the alloy. Starting from the third cycle, the foil weight is changing, with the oxide layer growth and loss/entrainment. The weight variation has a general decreasing trend. The average value of the alloy specific weight variation with the number of thermal cycles is stabilized after five or six cycles and amounts to $6.82216 \cdot 10^{-6}$ g/cm².

To study the formation mechanism of a protective oxide layer on the alloy surface, its phase structure was investigated by the X-ray diffraction (XRD) analysis (Fig. 4).

Fig. 4. XRD profile of the aluminum oxide layer on the alloy surface.

Fig. 5. HPC model consisting of four tiles connected with U-shaped elements. Fig. 6. Cross-shaped plug element.

The formation of a sufficiently thin layer of the aluminum oxide is observed on the alloy surface, as evidenced by the low intensity of XRD peaks, as well as the nickel aluminide line in Fig.4. No diffusion through alumina, which is thermally and thermodynamically stable, is usually observed, which fact may explain the mechanism of the material heat resistance improvement.

High heat resistance and improved physical and mechanical characteristics of the above alloy ensured its application in HPS in the windward part of orbital spacecraft. The respective structures containing four tiles with an external three-layer panel with a 165×165 mm area and honeycomb filling were designed, manufactured under loads, which simulated the operational loading conditions. The edges of external panels were joined by U-shaped elements, which prevented the penetration of hot gases into HPS but allowed a free movement of panel
edges due to thermal expansion cycles of heating to 1100°C and cooling. edges due to thermal expansion cycles of heating to 1100° and cooling.

The design prototype was verified by elaborating finite element models and analyzing the stress-strain state (SSS) under maximum mechanical and thermal loads acting on HPS during the launch and re-entry of the reusable space vehicle (Fig. 5). The results of the SSS analysis of the structure with continuous U-shaped elements by the analytic and visual methods revealed that at the preset pressure of 0.013 MPa and temperature of 1100°C, the analytic and visual methods revealed that at the preset pressure of 0.013 MPa and temperature of 1100° C, the thermal expansion of panels induced stresses in most structural elements exceeding permissible ones by 10–20 times. Maximum stresses occur at the intersection of the U-shaped element and the joint of four tiles. The introduction of holes in this place allowed a free deformation of load-bearing elements and reduced stresses to an acceptable level. To seal the holes, a cross-shaped plug element, which allowed a free motion of edges during heating and cooling, was inserted into the U-shaped elements (Fig. 6).

The SSS analysis revealed that, in order to ensure the permissible stress level during deformation of an U-shaped element, it is expedient to realize a two-layer structure with a 0.15 mm thickness of each layer. Whereas at pressure of 0.013 MPa and temperature of 1100°C, the maximal stresses of 302 MPa (which exceed the permi pressure of 0.013 MPa and temperature of 1100° C, the maximal stresses of 302 MPa (which exceed the permissible

Fig. 7. A two-layer (a) and a single-layer (b) elements after testing.

Fig. 8. HPS model (a) and design prototype No. 3 (b) consisting of four tiles.

ones) arise in a single-layer structure of a 0.3 mm thickness, those in a two-layer one $(0.15 \text{ mm} + 0.15 \text{ mm})$ do not exceed 204 MPa and fall into the permissible range.

For the experimental verification of the above theoretical and numerical results, the operational performance of a single- (0.3 mm-thick) and two-layer $(2 \times 0.15 \text{ mm-thick})$ elements was tested by the repeated thermal loading which corresponded to operating conditions. Samples of the elements were installed in a test rig, providing imitation
of the joint deformation in a standard design, and heated in a muffle furnace to 1100°C with hold time o of the joint deformation in a standard design, and heated in a muffle furnace to 1100° C with hold time of 20 min and subsequent cooling in air.

According to the results of 50 thermal cycles, the two-layer element kept its integrity without visible defects; therefore, the tests were terminated, while the single-layer element cracked after the ninth thermal cycle (Fig. 7).

Thus, experimental studies of both types of HPS elements corroborated the calculation results. Accordingly, a tiled structure with an external metal panel and internal thermal insulation from quartz fibers was designed. Its surface density, calculated with an account of specific weight and thickness of used material, was 9.96 kg/m^2 , which complied with the weight limitation requirements.

To verify the feasibility of material choice and design solutions proposed for the development of reusable spacecraft HPS, comparative tests of three design prototypes, namely a single tile (No. 1), a two-tile assembly (No. 2), and four-tile assembly (No. 3), were conducted (Fig. 8).

During experimental testing, the HPS strength was evaluated under the set of loads simulating the standard ones at all stages of the product operation, namely, transportation, launch, and flight. The integration of the

Fig. 9. Regular thermal loading of the design prototype (a) and gas flow temperature variation and reference points for fixing thermocouples on the metal three-layer structure (b): (*1*) under the external plate, (*2*) on the inner plate surface, (*3*) in the gap below the cross-shaped plug element, (*4*) in the honeycomb cell, and (5) at the external (cold) side of the insulation.

three-layer structural elements into the panel was carried out by diffusion welding in vacuum. To assemble such a panel with supports and U-shaped elements, soldering in vacuum was used.

Prototypes No. 1 (a single tile of 145×145 mm in size) and No. 2 (two single tiles of 310×150 mm in size fixed by supports and connected by an U-shaped element) were subjected to a series of transportation, launch and flight loadings. During transportation tests via a SU-1 impact test bench, under the action of linear overloads via a Ts5/300 centrifuge and vibrations via an UVÉ-100/5-3000 vibrating stand, the loads corresponding to the standard ones were applied sequentially in three directions. In the initial state and after each type of testing, a visual inspection of prototypes was carried out, in order to check their integrity, while the quality of welded joints of honeycombs with linings was assessed by the thermography.

Test results revealed no loss of integrity the prototypes Nos. 1 and 2. Thermograms of three-layer panels were identical for all loading stages. The tile appearance remained unchanged transportation loading, linear overloads, and vibration tests. Upon successful testing of these prototypes, prototype No. 3 was tested, in order to verify the HPS operability at the stage of the reusable space vehicle re-entry into the Earth's atmosphere. The prototype No. 3 had the total area of 165×165 and consisted of four tiles (75 \times 75 mm each) connected by elements, supports, a cross-shaped plug element, and a cover plate (Fig. 8). Under the external three-layer panel, there was an internal thermal insulation of a 28 mm thickness made of superthin quartz fibers.

The experimental studies were carried out in a high-temperature gas flow at the gas dynamic stand of the Pisarenko Institute of Problems of Strength of the National Academy of Sciences of Ukraine [3], according to the procedures described elsewhere [4]. Prior to tests, the thermographic method was used to check the quality of welded joints of linings with honeycombs in three-layer panels. The approaches, which simulated the action of external factors on the structural element and the equivalence of material damage processes in model and field conditions, were applied [5].

The results of HPS temperature measurements during testing of the prototype on a gas-dynamic stand under conditions modeling operational ones are shown in Fig. 9. To control the temperature, five chromel-alumel thermocouples were installed on the HPS: under the external plate/lining, in honeycombs between the outer and inner linings, on the inner lining surface, under the cross-shaped plug element, and inside the heat insulation.

During the tests, two cycles of thermal loading were carried out. The temperature of the gas flow was During the tests, two cycles of thermal loading were carried out. The temperature of the gas flow was 1325℃. The temperature variation in the reference points is shown in Fig. 9b. The temperatures under the external plate/lining and the cross-shaped plug element were nearly the same and after 250s fell into the range of plate/lining and the cross-shaped plug element were nearly the same and after 250s fell into the range of 1050–1100°C. After 250 s, the temperatures in the honeycomb and on the inner lining surface were about 950°C. On the the inner side of the insulation, the temperature was rising until 400 s and reached 260 \degree C, and then remained unchanged during the entire test period.

Fig. 10. The HPS prototype after two cycles of thermal loading: (a) soldered joint; (b) diffusion welding joint.

b

Fig. 11. The microstructure of (Ni20Cr5.95Al)-Y₂O₃ alloy (inner lining) before (a) and after (b) testing.

The visual inspection of the prototype No. 3 after two heating and cooling cycles revealed no significant changes in the appearance of surfaces and soldered joints of the external plates and plug element. However, fracture of the inner lining/honeycomb filling weld joint was observed in the second three-layer panel containing a defect detected before the test (Fig. 10b).

Thus, the soldered joints retained their integrity, while the presence of defects in the welded joint (namely, lack of fusion) of honeycombs with plates led to the peel-off of the HPS internal panel during thermal cycling.

To study the alloy microstructure after testing on a gas-dynamic stand, a metallographic analysis of a fragment of the inner lining of the failed second panel was performed. The cross section of the sample in the non-etched form was studied via an inverted reflected light metallographic microscope. The microstructural analysis revealed longitudinal cracks formed on the inner lining surface after two heating and cooling cycles, which led to the surface layer peel-off and the integrity violation the three-layer structure (Fig. 11b). The crack initiation mechanism may be attributed to the presence of pores and their accumulation in the surface layers. To clarify this

mechanism, the microstructure of the panel lining, which was not subjected to thermal cycling (i.e., in the initial state) was examined. It was revealed that the surface layer of the initial material also contained pores induced by cold rolling (Fig. 11a). Being subjected to high-temperature corrosion, they contributed to the surface layer damage, which manifested itself in the diffusion weld peel-off.

The observed integrity of soldered joints can be attributed to lower residual stresses arising during soldering, as well as the presence of solder liquid phase, which penetrates into large surface pores and heals them.

CONCLUSIONS

1. The mechanical characteristics of an innovative heat-resistant high-temperature (Ni20Cr5.95Al)- Y_2O_3 alloy produced by the powder metallurgy technology were experimentally determined in the operating temperature range of 20–1100°C. The results obtained proved that this alloy can be used in HPS for the windward part of orb range of $20-1100$ °C. The results obtained proved that this alloy can be used in HPS for the windward part of orbital space vehicles.

2. We performed the design of a heat-protective structure consisting of individual tiles, which external plate edges were connected by two-layer U-shaped elements, which prevented the penetration of hot gases into the heat
protection and allowed a free motion of the panel edges during their heating to 1100°C and cooling. The surfac protection and allowed a free motion of the panel edges during their heating to 1100° and cooling. The surface density of HPS with load-bearing elements was 9.96 kg/m², which complied with the available weight limitation requirements.

3. To verify the feasibility of the proposed thermal protection under operating conditions, three prototypes of the load-bearing HPS with honeycomb filling and one, two, and four three-layer panels, respectively, were produced and tested in the high-temperature gas flow simulating the operation thermal loading conditions. The results obtained corroborated the correct choice of materials and design solutions proposed in this study.

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