EVALUATION OF THE THERMAL STABILITY OF COMPOSITE POWDER MATERIALS IN A PLASMA JET

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The paper presents the results of studying thermal stability of materials operating in a plasma jet under high temperature and rapid thermal load variation conditions. It has been shown that the use of plasma generators along with the introduction of various powder materials make it possible to simulate the operating conditions of the parts exposed to high-temperature gas flows containing heated particles. The effect of the introduced powder materials on the thermal stability of samples prepared by plasma spraying from molybdenum, as well as tungsten and tungsten-based compositions was studied. An experimental setup was proposed for evaluating the thermal stability of the composites and protective hardening coatings operating under high-temperature gas flows, which also allows determining erosion resistance.

Keywords: thermal stability, gas flow, plasma jet, thermophysical properties, thermal loading, erosion resistance.

Strength and thermal stability are the most important characteristics when it comes to the performance of various coatings under the high temperature and increased loading conditions. Therefore, selecting or creating product coatings having required heat resistance for specific operating conditions is a rather challenging task $[1 - 5]$. Thermal stability is strongly affected by such factors as size and shape of a part, elasticity parameters, thermal conductivity, and thermal expansion of the material the part is made of $[6 - 10]$.

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The appearance of thermal stresses in a product can be caused by the following situations $[11 - 13]$:

– steady-state heating under uneven temperature distribution conditions;

– occurrence of thermal shock during transient heating;

– different TCLE values of each phase in a multiphase structure of the heated product.

Besides the properties of the material, its thermal stability is strongly affected by the surface heat transfer conditions, thermal loading, and a product temperature variation rate. Therefore, when studying thermal stability, material characterization is performed using a criterial form [14, 15].

Thermal stability of the structural elements is determined by the mechanical and thermophysical properties of corresponding materials. Studying the effect of each individual characteristic and summarizing all the results is a challenging task, since it requires conducting a significant number of experiments. Therefore, the recent trend has been to study thermal stability of the parts under the conditions, closely mimicking the actual operating conditions $[16 - 21]$.

When testing materials for thermal stability under cycling thermal loads, the following two situations are mainly considered: I — cycling variations of thermal and mechanical loads, and $II -$ cycling variations of thermal loads only.

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Fig. 1. Schematic diagram of the experimental setup for thermal stability testing of the materials: (*a*) position I; (*b*) position II; *1*) control panel; *2*) IPN 160/600 power supply; *3*) plasma generator; *4*) assembly; *5*) test sample; *6*, *7*) automatic KSP-4 recorders (potentiometers); *8*) cooling liquid (gas) inlet.

Based on the above, the objective of this work is to study and evaluate the thermal stability of the powder composites operating within a plasma jet under the high temperature and rapid thermal load variation conditions without mechanical loading.

STUDY OBJECT AND PROCEDURE

A schematic diagram of the setup for testing thermal stability of the materials is shown in Fig. 1. The setup consists of a DC power supply (IPN 160/600) equipped with a control panel for operating a plasma generator. The purpose of IPN 160/600 is to feed plasma-generating gases, such as nitrogen, argon, and a nitrogen-hydrogen mixture, into the plasma spraying nozzles [22].

A cylindrical sample subject to testing is snug fit into a special water-cooled assembly and heated by a high-temperature gas flow produced by the plasma generator [23, 24]. An assembly for testing samples within a plasma jet is shown in Fig. 2. Housing (*1*) of the assembly and covers (*2*) were made of brass. To prevent oxidation of the sample during heating and cooling, inert gas (nitrogen) is supplied into the gap between quartz glass (*4*) and sample (*5*). In addition, quartz glass makes it possible to observe the sample behavior during testing [25].

To measure the temperature gradient, two thermocouples having a base of 10 mm (selected based on design consider-

Fig. 2. Assembly for thermal stability testing of the samples: *1*) housing; *2*) cover; *3*) rubber gaskets; *4*) quartz glass; *5*) test sample; *6*) thermocouples.

ations) are installed along the length of the sample. The lead wires of the thermocouples are passing through the holes in the quartz glass (see Fig. 2).

The temperature was measured using tungsten-molybdenum thermocouples [26, 27] according to GOST R 50342–92 and GOST R 8.585–2001. In this case, the EMF is passed from the thermocouples (thermoelectric transducers) to an automated recording device (KSP-4 potentiometer) having a measurement range from 0 to 10 mV. The maximum chart transport speed was 5,400 mm/h.

After a stable operation of the plasma generator was achieved, the assembly containing the test sample was quickly inserted into the plasma jet (see Fig. 1*a*). The distance between the sample and plasma generator in all experiments was 15 mm. The sample was heated within the plasma jet while in position I (see Fig. 1*a*) until the temperature on its outer surface reached 2,200°C. The sample remained exposed to this temperature for $10 - 20$ sec and then was quickly moved to position II (see Fig. 1*b*) and cooled down to 20°C by a stream of cold water (or gas). The testing continued until the sample developed cracks.

STUDYING THERMAL STABILITY OF MOLYBDENUM COATINGS

Molybdenum samples, prepared using a plasma spraying method, were tested according to the test procedure de-

	Thickness, mm		Number of cycles before failure
Molybdenum coating	sublayer coating		
Using $5 \mu m$ powder		0.3	θ
Using powder with Ni sublayer, heat treatment in vacuum at 900° C (soaking — 1 h)	0.25	0.3	$\mathbf{0}$
	0.05	0.3	$\mathbf{0}$
Using powder with PT-10N-01 sublayer, heat treatment at 900° C (soaking - 1.5 h)	0.10	0.5	0
	0.20	0.5	θ
Using powder with molybdenum wire sublayer	0.10	0.15	10
Using 1 mm diameter wire		0.4	$10 - 15$

TABLE 1. Thermal stability of molybdenum coatings on steel 12Kh18N9T samples.

scribed in Ref. [28, 29]. The test results showed that the molybdenum coating obtained using 5 μ m powder had quite low adhesion strength and peeled off during the first cycle of thermal stability tests (Table 1). An attempt to use various sublayers to increase the strength and thermal stability of molybdenum coatings did not result in any significant improvement of their properties. The highest adhesion strength of the molybdenum powder was achieved by using a Ni–B sublayer with subsequent heat treatment in vacuum at $900 - 1,100$ °C. It should be noted that due to the formation of brittle intermetallic layers, thermal stability of such coatings turned out to be very low.

The highest thermal stability on steel was demonstrated by using a powder coating with a molybdenum wire sublayer. During thermal cycling, this coating showed no visible damage, however, it did peel off along the sublayer interface during machining. Heat treatment of this powder coating in vacuum did not eliminate this drawback. On the other hand, deterioration of the wire coating occurred due to gradual spalling of individual particles, which made it possible for such coating to survive rough machining without peeling-off, although this did cause a significant wear of the tooling.

To increase the strength and thermal stability, as well as improve technological properties of the coatings, further tests were conducted using samples made of tungsten and its compositions with molybdenum, zirconium oxide, and zirconium carbide $[30 - 32]$.

TABLE 2. Results of sample testing for thermal stability.

Material	Heating rate, $\rm ^{\circ}C/sec$	Cooling rate. $\rm ^{\circ}C/sec$	Temperature gradient along the sample length, $\rm ^{\circ}$ C/mm	Number of cycles before failure
Tungsten	227	550	470	18
$W + Mo$	465	400	430	18
$W + ZrO2$	300	290	310	29
$W + ZrC$	450	245		12

THERMAL STABILITY OF COATINGS MADE OF TUNGSTEN AND ITS COMPOSITIONS WITH MOLYBDENUM, ZIRCONIUM OXIDE, AND ZIRCONIUM CARBIDE

As can be seen from the thermal loading vs. time relationship shown in Fig. 3, the use of a plasma jet allows obtaining quite high thermal loading rates when testing materials for thermal stability. This is especially important for those materials which operate at high temperatures and high heating and/or cooling rates.

The results of thermal stability testing of the samples have shown that the addition of $ZrO₂$ significantly increases the heat resistance of tungsten, while molybdenum does not affect it, and zirconium carbide (ZrC) reduces it. According to the results of analyzing the microstructure of the samples, the addition of $ZrO₂$ results in a decreased grain size of tungsten during exposure to high temperatures, which improves its mechanical properties. On the other hand, ZrC stimulates grain growth, which worsens the properties of spray-deposited tungsten. Molybdenum does not affect the structure of tungsten, but does improve other mechanical characteristics of the material.

The analysis of the obtained data shows that by conducting tests using the proposed experimental setup, it becomes possible to achieve quite high temperature gradients along the length of the sample. For example, after testing the samples by exposing them to cycling heat loads, a fracture pat-

Fig. 3. Thermal loading of the samples during thermal stability testing.

tern becomes apparent. In this case, the samples mainly have radial cracks. This is because the temperature gradients in the axial direction exceed those in the radial direction. In addition, during plasma jet testing materials undergo significant erosion.

CONCLUSION

Testing samples for thermal stability using a plasma jet makes it possible to simulate operating conditions of the parts exposed to high-temperature gas flows.

The use of plasma generators along with the introduction of various powder materials enable simulation of the operating conditions of the parts exposed to high-temperature gas flows containing heated particles.

The proposed experimental setup utilizes a plasma jet to evaluate the thermal stability of the powder composites operating under high-temperature gas flow conditions. In addition, this setup allows conducting comprehensive studies of erosion resistance of various structural materials and protective hardening coatings.

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