Thermometry of the system "heat-resistant sample - incident plasma stream"

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Abstract. To study the interacting system “heat-resistant sample – an incident plasma stream” a setup of synchronized measurement equipment was developed and tested that recorded the main parameters of such interaction. Heat resistance tests were carried out on the samples of MPG-6 grade isotropic graphite, and samples of pyrolytic graphite that were subjected to a long (60 … 100 s) exposure to nitrogen, argon and air plasma streams at atmospheric pressure. As plasma generators a series of plasma torches with a vortex stabilization of the stream and an expanding anode channels was used. The temperature and composition of the plasma in the jet and near the sample were determined using two AvaSpec2048 and AvaSpec3648 scanning optical spectrometers and the MS5402i spectrograph with the Andor matrix at its outlet. The surface temperature of the sample was determined in real time using three independent ways: two pyrometric systems - a high-speed micro-pyrometer FMP1001 and a two-position visualization of the heated sample by high-speed Motion Pro X3 and VS-FAST cameras, and the spectral analysis of the wide-range thermal radiation of the samples. The main method for determining the rate of material loss during the action of a plasma jet on it was to analyze a two-position synchronous visualization of the "jet-sample" system. When a crater was formed on the surface of the sample under the "dagger" effect of a plasma jet, a video recording system of the crater zone was used, backlit using the "laser knife" method.

1. Description of the synchronized measurement system

A series of plasma generators was designed and manufactured to model the conditions of the effect of the plasma stream on heat-shielding coatings. These plasma generators can provide a wide range of media (argon, helium, nitrogen, air, carbon dioxide), pressures (10 - 1000 torr), thermal loads (1-50 MW/m²) and the speed of plasma streams (10 - 2000 m/s). As a result, a research complex has been manufactured that includes a plasma generator of the required parameters, a chamber for observing the flow of a plasma stream flowing onto a heat-resistant sample, a system of measuring instruments and devices, and, with a significant (over 100 kW) system power, a water-cooled plasma stream receiver that vents out the plasma after its interaction with the sample. The plasma jet generators with an expanding channel of an output
electrode belong to the class of DC plasma torches with a thermionic cathode, that have a high efficiency of 70-80\% and are described in detail in [1, 2]. From the expanding anode channel of the plasma torch, a high enthalpy plasma jet flows either into the free air space of atmospheric pressure or into a chamber with controlled pressure. The chamber has a system of viewing windows in the lateral cylindrical surface and the upper flange surface of the chamber. To carry out the research on the interaction of plasma streams with heat-resistant materials, an automated measuring system was created, that includes the following measurement systems (see figure 1).

A system of synchronized video surveillance for the interaction of an oncoming plasma stream with an ablating heat-resistant sample consists of high-speed Motion Pro X3 and VS-FAST cameras and an ultra high-speed (with a frame rate of up to 100,000 s\(^{-1}\)) Phantom Miro M110 camera. These cameras provide the recording of the interaction region “jet-sample” at the prechosen scale during the whole interaction period (usually 60-100 s). To use these cameras as high-speed micro pyrometers an interference filters with an allowed bandwidth of $\delta_{\lambda_1/2}=10–12$ nm were used.

Figure 1. Schematics of the system of synchronized measurements using a "laser knife", 1 - plasma torch, 2 - plasma stream, 3 - sample, 4a - fiber optic spectrometer Avaspec 2048, 4b - fiber optic spectrometer Avaspec 3648, 5 - laser source, 6 - telescope, 7 - cylindrical lens, 8,9,10 - high-speed video cameras, 11 - FMP1001 pyrometer.
In order to measure the local values of the brightness temperature of the selected zone (1.5-2 mm in diameter), thermograms were recorded from the surface of the sample using a high-speed (time resolution up to 1 μs) FMP1001 three-frequency micropyrometer developed at the OIVT RAS with a temperature range of 1200-5000 K.

Spectral measurements are performed using a three-channel and a single-channel optical spectrometers AvaSpec2048 and AvaSpec3648. The three-channel optical spectrometer AvaSpec 2048 (spectral range 220-850 nm) with a spectral resolution of 0.2-0.5 nm registers plasma radiation along the longitudinal coordinate of the plasma stream incident on the sample. The measurements provide control over the changing (with a periodicity of 2-4 spectra/s) plasma chemical composition and plasma parameters in the interaction zone ($\Delta Z \approx 0$-8 mm above the sample surface). Single-channel optical spectrometer AvaSpec 3648 with a spectral range of 220-1100 nm and a spectral resolution of about 1 nm monitors the radial distribution of plasma jet radiation from the near-surface plasma zone with continuous transverse displacement of the optical fiber. Monitoring of spatial-temporal changes in plasma emission spectra is carried out by scanning the plane of an intermediate sharp image of a plasma jet formed by quartz condensers using the spectrometers' light guides.

The experimental estimation of the mass loss rate of the sample material in real time is performed by weighing methods, two-position high-speed video recording and laser profilometry. The main method for determining the rate of material loss during the action of a plasma jet on it, usually, is to analyze the two-position synchronous visualization of the "jet-sample" system. For this purpose, two high-speed cameras Phantom and VS-FAST (see figure 1) are recording the "profile" of the destroyed sample from two mutually perpendicular directions with a frequency of 10-30 fps, an exposure of 10-100 microseconds and in a scale of 1:5 during the whole plasma exposure period. The two-positioned synchronized visualization of the destroyed sample allows to accurately control the change in the sample volume in time and, knowing its density and area of the ablating surface, determine the ablation rate.

To provide a detailed study of the mass loss rate from the top plane surface of the sample that the plasma is incident on, the "laser knife" method was used (see figure 1). The source of the laser radiation was a pulsed-periodic LCM-DTL-319QT laser with a pulse frequency of up to 10 kHz, a pulse duration of 7 ns, a wavelength of 527 nm, and an energy in the single pulse of up to 100 μJ. The focusing system with a telescope and a cylindrical lens formed a "laser knife" with a width of about 1 mm and a height of 35-40 mm at the samples top surface. The trace of this "knife" on the sample's surface was recorded by a Motion Pro X3 video camera.

2. Results of the combined measurements

Using the experimental setup described in previous section, tests were performed on the MPG-6 grade isotropic graphite with a density of $\rho=1.7$–1.8 g/cm$^3$, the samples had a cylindrical shape with a diameter $d_0=20$ mm and a height of 15 mm, with a flat surface facing the incoming plasma stream. The second type of graphite that was tested was the pyrolytic graphite produced by NIIGRAFIT with a size of 20x12x4 mm. In the interaction region that was chosen to be 10-30 mm from the outlet of the plasma torches nozzle the visible diameter of the plasma jet is about 10 mm. To minimize the thermal losses of the samples, they were placed on 2-3 tungsten rods with a diameter of 1 mm and a height of 8-10 mm. The parameters of the plasma generator are as follows: the arc current could vary from 150 to 400 A, with a combustion voltage of 80-90V and a plasma-generating gas flow rate of 1.5 g/s.

Calorimetric measurements of the plasma heat fluxes at a distance of 10-30 mm from the outlet of an output electrode of the plasma torch removed by water while cooling the copper sectioned cylindrical end of the calorimeter with a total diameter of 30 mm showed that a high enthalpy nitrogen plasma stream 10-15 mm in diameter with enthalpy $H \geq 20$ kJ/g, with a weight average temperature of 7000-12000 K,
provided a specific heat flux on the calorimeter surface from 0.2 to 3.0 kW/cm².

2.1. Emission spectra and plasma stream parameters

The light guide of the AvaSpec 2048 spectrometer scans a sharp image formed by the quartz condenser of the region of interaction with the longitudinal coordinates Z from +7 mm above the surface of the sample to -3 mm below it. Having a step of 1 mm and moving with a speed of 1 mm/s the spectrometer registers 6-10 spectra of the plasma stream or samples surface in each point of the above named range during the total plasma exposure time (60-100 s). The effect of the ablating sample on the plasma composition is already noticeable at 6-8 mm above the sample's surface and it undergoes significant changes as the sample is heated and ablated further. Examples of such changes are shown in figure 2. The quantitative analysis of the spectra recorded at a frequency of 2-4 spectra per second gives a detailed idea of the variation in the spectral composition of the plasma and the intensity of radiation of its atomic and molecular components throughout the entire interaction cycle.

Figure 2. Spatial-temporal variations in the emission spectra of plasma in the interaction zone with MPG-6 sample. Current 300 A, \(q_0\) \(\approx\) 0.8 kW/cm²: a) \(z=2\) mm above the sample, b) \(z=6\) mm. Short wavelength region in window – 30:1 scale.

The spatial-temporal variation in the emission spectra of an incident nitrogen plasma jet of atmospheric pressure in the region of the graphite sample location at a 15-30 mm distance from the nozzle makes it possible to determine the axial values of the electron temperature of plasma that is 6000-9000 K. The spectra presented in figure 2 show that the line spectrum contains a large number of nitrogen and copper (anode nozzle material) spectrum lines. In the short-wave part of the spectrum, molecular spectra of \(N_2\) (2⁺ system, bands of bands 337, 316 and 297 nm), \(N_2^+\) (1⁻ system, bands of bands 356, 391 and 428 nm) and violet bands CN (transition \(B_2^\Sigma^+\rightarrow X_2^\Sigma^+\)) are present. The presence of a large number of atomic N I lines in the investigated plasma spectra with excitation energies of the emitting levels from 11 to 15 eV and the fulfillment of the Boltzmann law with an electron temperature \(T_e\) for the relative distribution of radiating atoms over the excitation states in the near-axis region of the plasma allow us to use the “Boltzmann exponential” method to determine \(T_e\) [2]. The plasma in this region is isothermal (the temperature of the heavy particles is close to the electron temperature), the electron concentration corresponds to its equilibrium values at the electron temperature and amounts to \((1-10)\times10^{15}\) cm⁻³. The
information about the change in electron temperature in each axial region during the heating of the sample and the assumption that the Boltzmann law with $T_e$ is valid for coupling of the populations of the ground and excited states of the radiating molecules makes it possible to determine the characteristics of the spatial-temporal variations in the concentration of CN and C$_2$ molecules observed in the spectra. A combined analysis of these changes with the data on the loss rate of the samples material will help to clarify the relative role of the graphite surface nitration and its sublimation rate as a function of its surface temperature.

2.2 Measurement of mass loss rate of the sample material in real time

We present an example of an estimate of the MPG-6 graphite samples mass loss rate when exposed to a nitrogen plasma jet (arc current 300A, gas flow rate 1.5 g/s). An estimate of the mass loss rate from the heated samples surface can be performed as follows:

$$\dot{m} = \frac{\Delta M}{S \cdot \tau}$$

here $\Delta M$ – the samples weight loss, $\bar{S}$ - the arithmetic average surface area of the sample before and after the test, $\tau$ – the duration of the mass loss process.

Figure 3. MPG-6 (left) and pyrolytic (right) graphite before and after the tests

Figure 3 shows a graphite sample with a starting diameter of 20 mm that was subjected to a thermal interaction with a total duration of 80 s and lost about half of its original mass as a result. The main method for determining the rate of material loss during the interaction of a sample with a plasma jet was the analysis of a two-positioned synchronized visualization of the “jet-sample” system. Two high-speed cameras Phantom and VS-FAST with a frequency of 30-50 fps, an exposure of 20-100 μs, and with scale of 1:5 have recorded the "profile" of the destroyed sample from two mutually perpendicular directions during the whole plasma interaction process. Interference filters that were introduced periodically into the cameras optical paths provided the possibility of measuring temperature fields on the sample’s surface. Registration of the samples dimensions makes it possible to accurately control the change in the volume of the sample over time, and knowing its density and the area of the ablating surface, determine the rate of decrease of its material:

$$\dot{m}(\tau) = \frac{dV}{d\tau} \cdot \frac{\rho}{\bar{S}(\tau)}$$

The loss rate calculated in this way during the stage of quasi-stationary heating varies in the range 5-8 mg/cm$^2$s at an arc current of 300A in the period from 30 to 100 s after the start of the plasma torch.
A detailed study of the samples mass loss rate from the zone of direct interaction with plasma jet was performed by laser knife method developed by us. Its essence consists in recording the trace of the "laser knife" on the surface of the sample, which deepens as the matter ablates from the crater. The system formed a "laser trace" about 1 mm wide on the upper surface of the sample, passing through the epicenter (the "brake" point) of the plasma jet. The trace of this "knife" (see the diagram in figure 1) on the surface of the sample was recorded by Motion Pro X3 camera (item 8) with 30 frames per second mode and 1 μs exposure. To improve the quality (contrast) of the laser trace image, an interference filter with an allowed bandwidth of $\lambda_0 \approx 525$ nm, close to the wavelength of the laser, was periodically placed in front of the Motion Pro camera lens. Examples of obtained video frames with a current of 300 A with a filter of 525 nm and without it are shown in figure 4a, 4b. The procedure for processing the crater profiles described in [3] consisted in analyzing the profile of the crater and determining the volume and area of the profile rotation body at each instant of time. As a result, the results of estimating both components of the material loss rate - the loss from the crater under the direct action of the plasma jet and the decrease from the heated surface of the sample that is not in direct contact with the plasma stream - were obtained.

Values were estimated by the following expression:

$$\dot{m}_{cr} = \frac{\Delta m_{cr}}{\tau \cdot S_{cr}}$$

Here $\Delta m_{cr} = V_{cr} \cdot \rho$ – the final mass loss from the crater during test time $\tau$, $V_{cr}$ - volume of the crater at the end of the test, $S_{cr}$ – the average value of the crater surface area taken equal to half of its final value. The mass loss rate averaged over the entire outer surface of the sample can be estimated as:

$$\dot{m}_{surf} = \frac{\Delta M}{\tau \cdot \tilde{S}}$$

where $\Delta M$ is the total mass loss of the sample minus $\Delta m_{cr}$, $\tilde{S}$ - is the average arithmetic surface area of the sample before and after the test (the surface area occupied by the crater at the end of the test is less than 1 cm², which is about 15% of the total surface area $\tilde{S}$).

A nonmonotonic behavior of the dependence is observed, characterized by rapid growth and a high maximum ($\dot{m}_{\text{MAX}} = 15$ mg/cm²s) of the ablation rate in the period $t \approx 10\div15$ s. By this time, as pyrometric measurements show, the heating of the sample is completed, accompanied by the plastic deformation of graphite in the zone of plasma interaction and its possible structural changes [4]. According to [4], with rapid heating of artificial graphite, accompanied by an increase in internal stresses, the appearance and multiplication of dislocations associated with the presence of defects in the packing of...
layers occurs. Then (20÷80 s) a quasistationary plasma-sample interaction occurs, when the thermal energy of a plasma jet with specific density \( q_0 \) absorbed in the crater zone is expended on radiant losses from the sample surface, maintaining the reached temperature level, and its nitrization and sublimation. The energy balance when neglecting the slightly exothermic process of surface nitrization is as follows:

\[
W_{\text{abs}} \approx q_0 \cdot S_{\text{abs}} = \varepsilon \sigma \left( T_{\text{surf}}^4 - T_{\text{cr}}^4 \right) \cdot S_{\text{surf}} + T_{\text{surf}}^4 \cdot S_{\text{surf}} \cdot \left[ \dot{m}_{\text{cr}} (t) \cdot S_{\text{cr}} + \dot{m}_{\text{surf}} (t) \cdot S_{\text{surf}} (t) \right],
\]

where \( W_{\text{abs}} \) – part of the plasma jet energy absorbed by the sample, \( q_0 \) – the specific absorbed power \( S_{\text{abs}} \) – the crater surface area affected by the stream \( q_0 \), \( \sigma \) – Stefan-Boltzmann constant, \( T_{\text{surf}} \) – the surface temperature of the sample outside the crater, \( S_{\text{surf}} \) – the surface area of the sample minus the surface area of the crater, \( T_{\text{cr}} \) – the surface temperature of the sample inside the crater, \( S_{\text{cr}} \) – the surface area of the crater, \( S_{\text{cr}} \approx S_{\text{abs}}, H_{\text{subl}} \) – the enthalpy of sublimation of the sample material. Using the experimental data for 300A current one can estimate all the terms on the right-hand side of Eq. (1). The enthalpy of graphite sublimation, according to [5], is assumed to be 60 kJ/g. The surface temperature in the crater is about 3500 K, the surface temperature outside the crater is \( T_{\text{surf}} \approx 2500 \) – 3000 K, the rate of ablation from the crater is \( \dot{m}_{\text{cr}} (t = 70 \text{ s}) \approx 8 \text{ mg/cm}^2\text{s} \), on average the ablation rate from the surface excluding the crater is 5 mg/cm²s, from the top surface – about 6 mg/cm²s. Numerical estimates of the components of equation (5) indicate a significant predominance of radiation and ablation losses from the surface of the sample that excludes the crater when compared to both of these losses from the crater area. At the stage of quasistationary heating (40÷80 s), the contributions of radiative and sublimation losses are commensurable, \( W_{\text{rad}} \approx W_{\text{subl}} \) and correspond to the specific absorbed power \( q_0 \approx 800 \text{ W/cm}^2 \).

2.3. Methods and results of measuring the surface temperature of a test sample with time resolution

The temperature of the sample surface in the epicenter of the interaction zone with the plasma jet was measured by an FMP1001 optical micropyrometer. It is equipped with a long-focus lens that provides a working distance of 300-500 mm and registers the temperature in an area of 2-3 mm. The measurements were carried out on a brightness channel of 650 nm with a speed of 1 ms. Thermograms were obtained for the heating of graphite samples in a nitrogen plasma at an arc current of 300 A. Their form is shown in figure 5.

The videograms of the samples glow obtained with the help of a three-color matrix make it possible, to use a video camera as a high-speed micro-pyrometer with a spatial resolution [6], and to obtain temperature fields on the sample surface. Figure 6 shows examples of recorded types of luminescence of the samples and the results of their programming conversion to a temperature field using a brightness calibration by a reference radiation source.
Figure 5. Thermogram of the graphite MPG-6 sample, at 300A current.

An interference filter with an allowed bandwidth of 12 nm (used also for recording the emission of a reference source) cuts out a spectral region of this width, free of intense plasma spectral lines, to avoid the interference of plasma radiation on the results of pyrometry. To ensure a minimum error in determining the temperature of the samples which varies in the range of 1500-3800 K, the radiation of the reference source ($T = 2400$ K) was recorded using the same working optical configuration as was used when heating the samples, with a varying exposure time and using standardized neutral filters.
Figure 6. Temperature fields on the samples surface.

Analysis of the dependence of the samples surface spectral radiation intensity on the wavelength in a wide range $\Delta \lambda = 400$-$1000$ nm with the Planck's emissivity shows that, to within $1\%$, the Wien law holds true in our region of variation of $\lambda$ and $T$. Therefore, the best method to determine the temperature of the samples lateral surface $T_w$ by spectroscopic method is to construct the dependence of the relative spectral intensity of the sample radiation from the wavelength in the Wien coordinates [7], shown in figure 7. This approach allows us not only to determine $T_w$ with an error of not more than $10\%$, but also to obtain confirmation of a weak change in the emissivity of the surface of graphite samples with the frequency and temperature [8]. Evidence of this is the linear dependence presented in figure 7 ($\lambda^3 \cdot I_\lambda$), which correspond to the Wien law with $\epsilon_\lambda = \text{const.}$
Figure 7. Determination of the temperature of the samples side surface using spectroscopic method, arc current of 150A (250 W/cm²) and 300 A (500 W/cm²). Below - a fragment of the "Planck" spectra of the radiating surface.

3. Conclusion

A measuring system with a synchronized control system was created ensuring the execution of a high-speed two-position visualization of the plasma jet and the interaction zone of the "jet-sample" with a frame rate of up to 100,000 s⁻¹, a time resolution of up to 1 μs and a spatial resolution of 20-30 μm, recording of plasma and sample radiation spectra in real time, micropyrometric measurements in the range of \( T = 1200-5000 \) K with a time resolution of up to 1 μs.

It should be emphasized that the simultaneous use of several methods for determining such important parameters as the temperature and velocity of the plasma movement, the surface temperature of the sample, the rate of decrease of its material, provides an increase in the accuracy and reliability of the combined measurements.

The performed research shows the high suitability of the experimental setup created for solving the problems of applied gas and plasmodynamics, primarily research in the field of creation and testing of thermal protection and the study of complex heat and mass transfer in "high-speed plasma flow-solid" systems.

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