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PROBE DIAGNOSIS OF A FLOW OF PARTICLES DESORBED FROM THE SURFACE OF A SOLID BY A LOW-DENSITY PLASMA JET

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The dynamic interaction of bodies with a flow of low-density gas is characterized by a variety of processes and phenomena occurring at the phase boundary. If the energy of the incident particles is greater than approximately 5 eV, processes involving energy and momentum transfer are accompanied by the dispersal of surface contaminants and layers of adsorbed gases, the desorption of particles from the surface, etc. Monitoring and study of these processes are very important from phenomenological and practical viewpoints to establish a balance between transfers of momentum, mass, and energy at the phase boundary. The parameters of mass flows dispersed by inflowing particles are usually measured by the gravimetric method [1, 2]. However, this method does not distinguish between the fraction of particles desorbed from the surface due to the dispersal of adsorbed layers and coatings by the incoming flow and the fraction of the loss due to erosion of the material of the surface [3]. Such a distinction is important for describing mass transfer in gas-surface systems and momentum and energy transfer at phase boundaries.

In the present article, we describe the methodology and results of an experimental study of the parameters of particles desorbed from a surface as a result from its bombardment by an inflowing low-density plasma. It is shown that the proposed method makes it possible to determine the fraction of particles adsorbed on the surface of the solid and evaluate their parameters.

1. A body placed in a high-velocity flow of a low-density plasma is subjected not only to incoming neutral and charged particles accelerated in the near-electrode layer, but also to particles desorbed from the surface by its intensive bombardment. Within a certain range of surface potentials, the particle desorption stimulated by the incident flow results in an increase in the total momentum transferred to the surface of the body. In a detailed examination of the balance of forces acting on a body in a low-density plasma flow, the momentum imparted to the body can be determined in the form

$$F_{\Sigma}^{(1)}(V) = F_i(V) + F_e(V) + \Delta F$$

for a surface free of adsorbent and

$$F_{\Sigma}^{(2)}(V) = F_i(V) + F_e(V) + \Delta F + F_d$$

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for a surface covered by an adsorbed layer. Here, F_e is the mechanical pressure due to electron bombardment; F_i is the mechanical pressure exerted by the ions of the incident flow; $\Delta F = F_n + F_m + F_0$; F_0 is the contribution of the residual gases; F_n is the contribution of fast neutral particles; F_m is the force exerted by the metastable particles; and F_d is the mechanical pressure of the particles desorbed from the surface.

The value of F_d can be determined from the force characteristic of the conducting body:

$$F_d = F_\Sigma^{(2)} - F_\Sigma^{(1)}.$$

Here, the effect of electrostatic forces can be ignored [4] for a model of the body in the form of a flat plate or disk with the characteristic dimension $R \gg \lambda_d$ (where λ_d is the Debye radius in the undisturbed plasma). The pressure F_d is due to desorbed neutral particles and slow ions. In the region of high negative potentials, F_d is determined only by the desorbed neutral particles:

$$F_{d_1} = F_{d_n} = 0{,}5AN_{d_n}kT_w. \tag{1.1}$$

Here, N_{d_n} is the concentration of neutral particles desorbed from the surface; T_w is the temperature of the surface of the body; A is the area of the surface; and k is the Boltzmann constant. A decrease in the potential of a surface covered by an adsorbed layer is accompanied by an increase in the flow of electrons to the body along with the flow of slow ions. Their presence is manifest in an increase in the ion current on the surface and shifting of the "floating" potential of the volt-ampere characteristic (VAC) of the body to the region of positive values. In essence, the effect the presence of slow ions has on the VAC of a body covered by an adsorbed layer is similar to the change seen in the VAC of a heated electric probe due to thermionic emission [5]. Figure 1 shows the ionic branches of the VAC of clean and coated (with an adsorption layer) (curves 1 and 2) flat electric probes in a flow of a low-density plasma. In the region of the maximum of the slow-ion current – the region of transitional potentials – the surface is subjected to the action of desorbed neutral particles and slow ions. The presence of slow ions is confirmed by the change in the force characteristic – the dependence of the momentum imparted to the surface of the body by the particles of the flow on the potential of the body:

$$F_{d_2} = F_{d_n} + F_{d_i} = 0{,}5AkT_w\,(N_{d_n} + N_{d_i}^+). \tag{1.2}$$

Here, $N_{d_i}^+$ is the concentration of slow secondary ions. In the given case, it is assumed that the particles desorbed from the surface are characterized by a Maxwell distribution with a temperature equal to the temperature of the surface T_w [6].

If we know the values of T_w and A, we can use Eqs. (1.1)-(1.2) to determine the concentrations of desorbed neutral particles

$$N_{d_n} = 2F_{d_1}/AkT_w$$

and slow ions

$$N_{d_i}^+ = 2\,(F_{d_2} - F_{d_1})/AkT_w\,.$$

Furthermore, knowing the current of slow desorbed ions I_{d_i} in the transitional region of surface potentials (Fig. 1) [7], we can also determine the mean velocity of the desorbed particles $\bar{v}_{d_i} = 4I_{d_i}/AeN_{d_i}^+$ and their average molecular weight $\bar{M}_{d_i} = 8kT_w/\pi\bar{v}_{d_i}^2$.

The following characteristics were recorded synchronously for the clean and coated probes in the tests we conducted:

1) $I_\Sigma = I_\Sigma(V)$ – the dependence of the flow of charged particles onto the surface of the body on its potential relative to the potential of space – the VAC of the body in the plasma flow;

2) $F_\Sigma = F_\Sigma(V)$ – the dependence of the momentum imparted by the particles of the flow to the surface of the body on the potential of the latter – the force characteristic;

3) $T_w = T_w(V)$ – the dependence of the temperature of the surface of the body on its potential – the temperature characteristic of the body in the plasma.

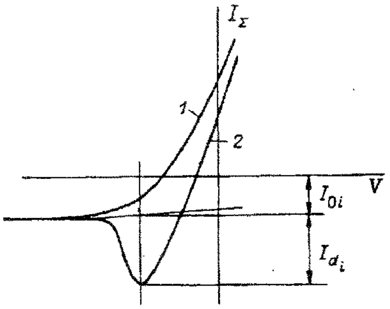

Fig. 1

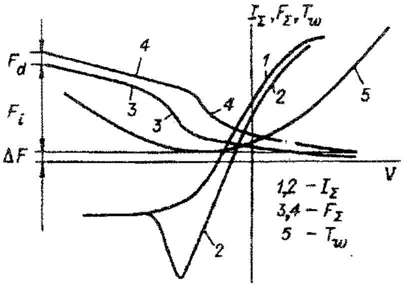

Fig. 2

Figure 2 shows these relations. Curves 1 and 2 represent the VACs for the clean and coated surfaces, curves 3 and 4 show the force characteristics corresponding to the VACs, and curve 5 shows the temperature characteristic. These relations make it possible to evaluate the parameters of particles desorbed from the surface when the process is stimulated by an incoming flow.

2. Experimental studies were conducted on a gasdynamic plasma unit with a high-velocity flow of ionized gas generated by a gas-discharge accelerator in which the working substance was ionized by electron collision with a "self-sustaining" plasma. The working gases were nitrogen, oxygen, hydrogen, and inert gases: high-purity neon, argon, krypton, and xenon. The accelerated flow, with an intensity $j_\infty^+ \approx 10^{14}$-$10^{17}$ cm$^{-2}\cdot$sec^{-1} and an ion velocity $U_\infty \approx$ 10 km/sec, entered a working chamber with a vacuum of at least 10^{-5} Pa. The vacuuum system consisted of two independent subsystems, the first being comprised of an AVÉD-40/800M electrodischarge unit and a series of 010V-3500-006 turbine molecular pumps [8]. The second subsystem consisted of nine AVP-8-4 oil-vapor pumps. Each subsystem had a productivity (in terms of air) of at least $\sim$50 m^3/sec.

The working part of the chamber was shielded by panels cooled with liquid nitrogen. To increase the degree of ionization and dissociation, the molecular gas fed into the ionization chamber of the accelerator was directed onto the surface of an incandescent cathode. It was established in [9] that doubly charged ions may appear in the flow when the energy of the electron beam is greater than 28 eV. Although the number of such ions is no greater than 0.5% of the total at a pressure $\sim 1.5 \cdot 10^{-2}$ Pa, we conducted our studies in flows of molecular gases at electron-beam energies up to 28 eV. This allowed us to avoid having doubly charged ions appear in the flows of low-density plasma.

The degree of dissociation of the ion flow and the composition of the residual gases in the working part of the chamber were monitored using an MKh-7303 mass spectrometer with an energy analyzer. To measure and monitor the parameters of the plasma flow, we used a system consisting of movable electric probes, a multi-electrode probe-analyzer and an SHF interferometer with a range of 3 cm.

The VAC and derivatives of the probe current with respect to voltage were measured in the automatic regime. The error of an individual VAC was no greater than ±2%. The potential of the plasma was determined by the second-derivative method and from the electronic branch of the probe characteristic plotted in semi-logarithmic coordinates. The scheme employed to measure the derivatives of probe current also allowed us to record plasma noises in the probe circuit, which in turn made it possible to check the accuracy of the determination of plasma potential: the maximum of plasma noise corresponded to the potential of space. The potential of the plasma was also determined on the basis of the point at which the VACs of the cold and heated thermoprobes diverged. The scatter of plasma potential was no greater than ±4%, which illustrates the relatively high accuracy of the measurements of ion energy W_i transferred by the particles to the plasma—layer interface. When W_i was calculated with the assumption that the accelerating potential was equal to the difference between the potential of the anode of the source and the local potential of the plasma, the resulting values were found to agree satisfactorily with the data from the multi-electrode probe-analyzer. The scatter of W_i was no greater than ±4.5%.

We checked for the presence of negative ions in the plasma flow by examining the VAC of a single probe and the readings of the SHF interferometer [10]. The condition of quasineutrality was used to perform this check. The use of such a procedure is justified by the fact that the SHF-diagnostic methods are based on the scattering of electromagnetic radiation on free electrons in a medium. By comparing values of charged-particle concentration found from the electronic and ionic branches of the VAC of the electric probe and measurements of N_e made using an SHF-interferometer [10, 11], it is possible to evaluate the fraction of negative ions in the flow: $N_e + N_i^- = N_i^+$. No negative ions were detected in plasma flows of molecular gases generated by the gas-discharge accelerator (with ionization of the working substance by electron collision).

In studying the interaction of the plasma flow and the surface of a solid, we used targets in the form of disks with a diameter ~38 mm and a thickness of ~1.2 mm. Current leads and a miniature thermocouple were attached to the back side of each disk. The lateral and rear surfaces of the disk and the elements of the thermocouple were coated with a heat-resistant dielectric (ceramic). Before the study, we calibrated the transducer in a thermostat and determined the relation $T_w = T_w(E)$, where E is the emf of the thermocouple. The targets were made of polycrystals of chemically pure materials with a polished working surface (Al, Ti, Ta), a chemically polished disk-shaped single crystal of Si(111), aluminum alloys AMg6-M and D16AT (rolled), and vacuum-shielding heat insulation (VSHI). The targets were placed on a compensation-type microbalance based on the standard magnetoelectric system of dc milliammeter N359. The target holder was placed in a dielectric tube (glass) with an inside diameter of about 18 mm in order to increase the sensitivity of the microbalance and minimize the contribution of uncharged particles in the flow to the force balance. To additionally shield the target from interactions with particles in the incoming flow, we placed screens having a potential equal to the potential of the target at the side and rear of the latter. The range of forces that could be measured over the distance $L \approx 450$ mm was approximately $5 \cdot 10^{-3}$-250 dyn. The measurements were made in the automatic regime with synchronous recording of the force $F_\Sigma = F_\Sigma(V)$, temperature $T_w = T_w(V)$, and volt-ampere $I_\Sigma = I_\Sigma(V)$ characteristics. To determine the orientation of the target relative to the velocity vector of the incident flow, we used a cylindrical probe made of molybdenum wire 0.04 mm in diameter and 2.3 mm in length. The peak of the ion current recorded by this probe with rotation about the vertical and horizontal axes corresponded to the orientation of the probe along the flow and made it possible to evaluate the degree to which the flow was nonisothermal [12]. The error of the angular orientation of the targets in the flow as no greater than ±20'

3. Experiments were performed in the cross section of a jet with a uniform distribution of the following parameters: concentration of charged particles $N_\infty^+ \approx 10^8$-10^{11} cm^{-3}; electron temperature $T_e \approx 4$-5 eV; ion-flow velocity $U_\infty \approx 10$ km/sec; pressure in the working part of the vacuum chamber $\sim 10^{-4}$-10^{-3} Pa.

In the first series of measurements, no special treatment was administered to the working surfaces of the transducers in the plasma flow. The targets were introduced into the incident flow behind the screens — outside the shadow of the large flat plate. Thus, conditions on the target corresponded to the conditions on the surfaces shielded from the plasma in the evacuated volume. After the required set of characteristics was recorded, the surfaces of the targets were cleaned by bombarding them for about 15-20 min with ions in a low-density plasma flow. Here, the potential of the surface $V \approx -250$ V. Approximately 15-20 min of further electron bombardment heated the specimens to temperatures below the point where the materials of the working surface of the target and thermocouple would have been destroyed. A high negative potential $V \approx -250$ V was then supplied again to the target and the working surfaces were aged by bombardment with the plasma flow for the 10-min period immediately prior to the measurements. The working surfaces were cleaned as a result of the intensive bombardment by the ion flow. The volt-ampere, temperature, and force characteristics were recorded beginning with $V \approx -250$ V.

Information on the condition of the working surfaces during measurements such as those described above can be obtained from determinations of integral hemispherical emissivity ε_{th}, a coefficient γ_i characterizing secondary ionic-electronic emission, and the work function κ. These quantities can be determined independently from the volt—ampere and temperature characteristics [13, 14]. With the targets having been administered the above-described preliminary treatment in the low-density plasma flow, the values we obtained for ε_{th}, γ_i, and κ allowed us to conclude that surface impurities had no significant effect on the measurements or the structure of the force, temperature, and volt-ampere characteristics.

TABLE 1

Target material	$j^+_{d_i}/j^+_\infty$
Al	1,37
AMg6-M	2,23
D16AT	2,41
Ti	0,95
Si(111)	1,17
Ta	2,16
VSHI	1,82

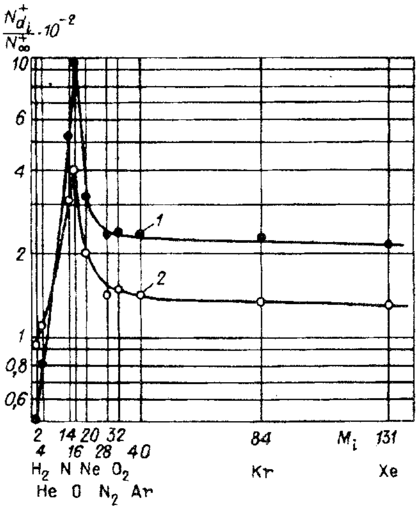

Fig. 3

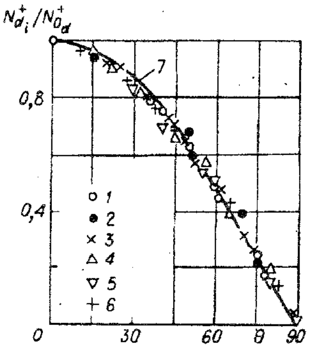

Fig. 4

Figure 3 shows ratios of the concentration of ions N^+_{di} desorbed from the surface of targets of D16AT and Al (curves 1 and 2) to the concentration of ions in the incident flow N^+_∞ within a broad range of bombarding-particle molecular weights, working pressures in the vacuum chamber, and surface temperatures $T_w \approx 300$-330 K. The quantity N^+_{di}/N^+_∞ is proportional to the ratio of the densities of the respective ion flows j^+_{di}/j^+_∞ The concentration (or intensity) peak corresponding to particles with the mass number m = 16 can probably be attributed to the fact that reactive cleaning of the surface takes place along with desorption by ion collision in the given case.

The data in Table 1 shows the ratio of flow intensities j^+_{di}/j^+_∞ in the irradiation of targets of different materials by Kr^+ ions in a low-density plasma jet.

Figure 4 shows the angular distribution of ions desorbed from surfaces irradiated by a high-velocity low-density plasma flow ($U_\infty \approx 10$ km/sec). Points 1-7 respectively characterize the incident-ion–target pairs Kr^+ – Si, O^+ – AMg6-M, Kr^+ – D16AT, $N_2{}^+$ – Al, Kr^+ – Ti, and Kr^+ – AMg6-M. Curve 7, showing $N^+_{di}/N^+_{0d} = \cos\theta$ (N^+_{0d}, illustrates the orientation of the target relative to the velocity vector of the incident flow θ = 0.

The position of the maximum of current density j^+_{di} relative to the y-axis on the VAC and the magnitude of the "floating" potential depend slightly on the target material in the case of bombardment by gaseous ions of one kind. This indicates that particle desorption occurs from an adsorption layer on the target surface which has roughly the same composition as that established by the spectrum of the residual gas. Estimates made of the flux of desorbed ions from the formula ($j^+_{di} \approx j^-_e Q^+ \sigma$ (where j^-_e is the flux of electrons of the low-density plasma reaching the target, σ is the coverage of the surface by the adsorbed layer, Q^+ is the effective excitation cross section in the desorption of particles from the surface) gives a value for

Q^+ which equals the cross section for the interaction of electrons with molecules in the gas phase $Q^+ \approx 10^{-16}$ cm² [14, 15]. Strictly speaking, we are concerned here with ions N^+_{di} formed when electrons in the incident flow ionize molecules of gases desorbed by ion collision with the target surface. The degree of ionization of the desorbed particles is no greater than ~10-12%. This is consistent with the values obtained for the average molecular weight $\bar{M}_{di}$ of ions desorbed from the surface: $\bar{M}_{di} \approx 24.7$, 25.9, and 26.6 for bombarding-ion—target pairs Xe^+ — D16AT, Kr^+ — AMg6-M, and He^+ — D16AT. These values are close to the average molecular weights established by mass spectrometry for the mixture of residual gases in the working chamber: CO — 70-78%, H_2O — 12-15%, H_2 — 5-8%, CO_2 — 4-7%. The changes which occur in the composition of the residual gas in relation to the evacuation regime and vacuum system that are employed have an appreciable effect on the average molecular weight of the ions desorbed from the target surfaces. When the working chamber was evacuated by the system of oil-vapor diffusion pumps, water and hydrocarbon components with mass numbers $m \approx 17$; 27; 41; 43; 55; 56; 57; 67; 69; 70; 71 etc. predominated in the spectrum of the residual gases. The average molecular weight of this system $\bar{M} \approx 43.4$. These results agree with the measured average molecular weights of ions desorbed from the surface of targets irradiated by the low-density plasma flow: $\bar{M}_{di} \approx 41.6$, 42.1, 39.8, and 43.6 for the pairs N_2^+ — D16AT, Kr^+ — AMg6-M, Xe^+ — AMg6-M and N_2^+ — Al, i.e., these results agree satisfactorily with the spectrum of the residual gases that largely determine the composition of the adsorption layer on the surface of the bombarded targets. There is almost no change in the character of the ratios $N^+_{di}(M^+_i)/N^+_\infty$ and $N^+_{di}(\theta)/N^+_{0d}$. Here the mean velocity of the desorbed ions changes from $\bar{v}_{di} \approx 4.2\cdot10^4$ to $3.1\cdot10^4$ cm/sec.

The data reported above demonstrates the feasibility and efficacy of using the proposed method to monitor the parameters of particles desorbed from a surface irradiated by a low-density plasma flow. The method can also be used under static conditions.

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