transmitting powers of a plane layer with thickness ℓ ; $R_{\lambda\infty}$, reflecting power of optically infinitely thick layer; ε_{λ} , extinction factor; Θ , angle of incidence; E_{λ} , density of diffuse and E_{λ} ', density of monochromatic radiant flux falling on layer at angle Θ , W/m^2 ; w'* = w'/ E_{λ} ' and w* = w/ E_{λ} , relative absorbed flux densities for directed irradiation w'* and diffuse w*.

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INTEGRAL HEMISPHERICAL ABSORPTIVITIES OF METALS AT 4.2-293°K

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UDC 536.3

On the basis of experimental data the authors obtained an empirical formula enabling them to determine the integral absorptivity of metal with an optically smooth surface from the magnitude of its electrical resistivity.

The most effective types of protection of cryogenic equipment operating in the temperature range 4.2-293°K against heat influx from the environment are high-vacuum and vacuummultilayer heat insulation. The former is an evacuated space between heat-exchanging surfaces which reflects well thermal (infrared) radiation, the latter is a set of reflecting screens placed in such a space and divided by heat-insulating distance packings. The screens in vacuum-multilayer insulation are various foils and metallized polymer films with high reflectivity of the surface.

For the calculation of radiative heat exchange in such systems it is indispensable to know the integral hemispherical absorptivities (relative to heat radiation) of the metallic surfaces. The dependence of the above parameter, especially at lower temperatures, on a number of factors including technological ones makes it difficult to find reliably the theoretical values of the absorptivity of real surfaces. The experimental study of the optical properties of metals is therefore of great practical interest.

The published experimental data on the optical properties of metals at low temperatures [1-4] are very limited, often contradictory, and it is difficult to compare them with each other because of lack of information on the technology of preparing the specimens.

The present work involved the experimental investigation of the absorptivities of metals including those used as screen materials and for coatings reflecting heat radiation, in the temperature range 4.2-293°K.

At 293°K the measurements were carried out by the radiation method with the use of a thermoradiometer marque TIS. The operating principle of the instrument is based on the use of the method of comparison where the absorptivity of the investigated surface is compared with the known absorptivity of a standard. The maximal error of measurement was 25% with an absorptivity A = 0.02-0.025, and it decreased with increasing A.

Measurements at low temperatures were carried out by the calorimetric method in an experimental vessel whose diagram is shown in Fig. 1. The vessel has an inner spherical cavity 1 with a capacity of 10 liters, and on its outer surface the investigated specimen is applied. Cavity 1 is surrounded by copper screen 2 which is cooled by a cryogenic liquid

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Fig. 1. Diagram of the experimental installation for measuring the absorptivity of metals at low temperatures: 1) inner vessel with the investigated coating; 2) blackened screen; 3) protective chamber; 4) jacket; 5) heater; 6) shields in the orifice of the vessel; 7) counter GSB-400; 8) pressure regulator; I) to the potentiometric circuit; II) to the vacuum system; III) discharge of the cryogenic liquid from the inner vessel.

Fig. 2. Dependence of the absorptivity A of metals on the electrical resistivity (ρ , Ω ·cm) in the temperature range 4.2-293°K; light dots) 293°K, dark on top) 90, on the right) 77, dark dots) 4.2°K; specimens numbers 2-14 correspond to Table 1; 15) copper [12]; 16) manganin [12]: 17) copper vaporized on in a helium atmosphere [10]; curves I, II, III) calculation by formula (6), according to the classical theory, according to the theory of the anomalous skin effect, respectively.

poured into the protective chamber 3, and for convenient replacement of the specimen the screen is made dismountable. The orifice of cavity 1 is made of stainless steel pipe 14-mm diameter \times 0.3 mm, and at a distance of 220 mm from the cavity it is brought by soft solder into reliable thermal contact with the protective chamber. The inner surface of the screen facing the specimen is blackened, and its emissivity is $\varepsilon_e = 0.95$. The orifice of the inner cavity for pouring is closed by a plug with small screens of aluminum foil PÉT-DA, metallized on both sides, suspended from the plug for intercepting radiant heat influx along the pipe to the liquid.

The inner cavity was filled with liquid helium or nitrogen next to the screen cooled by liquid nitrogen or oxygen, respectively. The experiments were carried out under steady-state conditions, the stabilization time was 2-3 h.

The absorptivity of the surface of the inner vessel was determined by Christiansen's formula

$$\frac{1}{A_{\rm c}} = \frac{1}{\varepsilon_{\rm rd}} - \frac{F_{\rm c}}{F_{\rm s}} \left(\frac{1}{\varepsilon_{\rm s}} - 1\right),\tag{1}$$

or

$$\varepsilon_{\rm rd} = \frac{Q_{\rm p}}{\sigma_0 F_{\rm c} \left(T_{\rm s}^4 - T_{\rm c}^4\right)} ; \qquad (2)$$

$$Q_{\rm p} = Q_{\rm m} - Q_{\rm t} - Q_{\rm r.g} \,. \tag{3}$$

The total heat influx to the inner cavity was determined from the amount of cryogenic liquid evaporating from it, measured by the counter GSB-400. The effect of fluctuations of the atmospheric pressure on the results of measurements was eliminated by the use of a pressure regulator.

The heat influx due to the thermal conduction of the residual gas was calculated by the formula [5]

$$Q_{\mathbf{r},\mathbf{g}} = 18.2\alpha \, \frac{\gamma+1}{\gamma-1} \, \frac{p}{\sqrt{MT_0}} \, F_c \left(T_{\mathbf{s}} - T_c\right). \tag{4}$$

			A			
№	Investigated surface	<i>T</i> , °K			$A(293'^{\circ}K)$	p(293 K)
		293	77	4,2	A(4,2°K)	ρ(4,2°K)
1	Aluminum ADI, foil annealed,	0.095				10 7
2	$Ra = 0.08 - 0.05 \mu m$ Aluminum A5, foil annealed,	0,025	0.010	0.0060	4.2	22.0
3	Aluminum A99, foil annealed, Ra = $1.25 - 0.8 \mu m$	0,035	0,012	0,0036	9,7	325,0
4	Aluminum A999, vaporized on steel Kh18N10T in a medium of pure he- lium with Ra = 0.32-0.16 um	0.035		0.0060	6.0	
5	Film PET-DA aluminized in va- cuum (10 ⁻¹ Pa), $\delta = 0.05$ -0.04 µm	0.025	0.028	0,0010	1.5	1.0
6	$Ra = 0.04 - 0.02 \ \mu m$ Copper M1, foil annealed, Ra = $0.16 - 0.10 \ \mu m$	0.018	0,020	0,0240	3,0	1,9
7	Copper MO vaporized on steel Kh18N10T in a medium of pure he-	0.005		0.0055	4.5	
8	Fium with Ra = $(0.32-0.16)$ µm Film PET-OM coppered in vacuum $(10^{-2}$ Pa), $\delta = 0.2$ µm, Ra = 0.04	0,025		0,0055	4,0	
9	- 0,02 μm Silver Sr 999.9 foil annealed,	0,025	0,015	0,0110	2,2	4,5
10	$Ra = 0.32 - 0.16 \ \mu m$ Silver Sr 999.9 vaporized in va-	0,020	-	0,0042	4,8	103,0
	cuum (10^{-2} Pa) on steel Kh18N10T with Ra = 0.32-0.16 μ m	0,020	0,016	0,0080	4,0	_
11	Film PET-05 silvered in vacuum $(10^{-2}-10^{-3} \text{ Pa}), \delta = 0.04 \ \mu\text{m},$ Ra = 0.04 - 0.02 \ \mm m	0,015	-	0,0120	1,3	_
12	Steel Kh18N10T, foil, Ra=0,1- -0,08 µm	0,120	-	0,0800	1,5	1,3
13	Lead vaporized in vacuum (10^{-2}) Pa) on steel Kh18N10T with Ra = 0.32-0.16 µm	0,050	-	0,0090	5,5	
14	High-purity niobium obtained by electron-beam melting, foil unannealed, Ra = 0.05 μ m	0,060	_	0,0320	1,9	2,4

TABLE 1. Optical and Electrical Properties of Metals in the Temperature Range 4.2-293°K

In measurements of this kind, the greatest difficulty is caused by taking into account the heat influx to the inner vessel through the orifice. When the orifice was cooled by vapors of liquid nitrogen, Q_t was determined by a method explained in [6] and based on the simultaneous solution of the equations of heat conduction for the walls of the orifice with a unidimensional temperature field in it and energy for the gas stream. Here the physical properties of the material of the orifice and of the gas are assumed to be constant, and they are taken at the mean integral temperature of the orifice. When the orifice is cooled by helium vapors, the thermal conductivities of the material of the orifice and of the gas change abruptly near the cold end of the orifice, and that makes the calculation more difficult.

We worked out an experimental method enabling us to prevent heat influx through the orifice. For that purpose we placed a heater with a resistance of about 100 Ω into the vessel filled with liquid helium; this enabled us to increase evaporability to a magnitude at which the above-mentioned heat influx was practically completely offset by the emerging vapor. Control and measurement of the heater power were effected with the aid of a four-way potentiometric circuit. In that case

$$Q_{\rm p} = Q_{\rm m} - Q_{\rm h} - Q_{\rm r.g}.$$
 (5)

With residual pressure of the order of $1\cdot 10^{-5}$ Pa the heat influx $Q_{{\bf r}\,,\,{\bf g}}\,amounts\,to$ about 2% of $Q_{m}.$

We investigated reflecting coatings in the form of layers vaporized directly on the surface of the inner cavity of the experimental vessel, and also in the form of metal foils or metallized polyethylene terephthalate (PET) films glued to it.

The results of measurements of the absorptivities of these coatings with an error not exceeding 15% and the characteristics of the corresponding surfaces are presented in Table 1.

A comparison of the data for aluminum foils shows that absorptivity depends substantially on the chemical purity of the metal, and in transition from commerically pure aluminum A5 (99.5% A1) to highly pure aluminum A99 (99.99% A1) it decreases to one half even if the latter has greater surface roughness than A5. This result is different from the one obtained in [4] for especially pure aluminum A999 with relatively high absorptivity A (5K) = 0.014 which is practically not dependent on the smoothness of surface machining in the range of roughnesses Ra = 1.25-0.025 μ m. Strictly speaking, Rastorgueva [4] measured the integral hemispherical emissivities of metals including aluminum. However, numerous experimental data [7-9] show that in the infrared spectral range with $\lambda > 10 \ \mu$ m metals may be regarded as gray bodies since their absorptivity ceases to depend on the wavelength of the incident radiation. For gray bodies absorptivity and emissivity coincide.

The absorptivities were measured of aluminum and copper films vaporized in a stream of highly pure helium [10] on substrates of steel Kh18N10T with a surface roughness Ra = 0.37-0.16 μ m. It can be seen from Table 1 that in magnitude A (4.2°K) these coatings are close to the foils A5 and M1, respectively.

Vacuum silvering of steel Kh18N10T yielded a value A (4.2°K) almost twice as large as for silver foil, with the same marques of silver and smoothness of surface machining in both cases. This is apparently due to the insufficiently high vacuum in vaporizing (10^{-2} Pa) causing oxidation of the silver.

It can be seen from Table 1 that PÉT foils aluminized, coppered, and silvered in vacuum are characterized by the largest values of A $(4.2^{\circ}K)$ and gently sloping temperature dependence of this parameter, determined by the ratio A $(293^{\circ}K)/A$ $(4.2^{\circ}K)$. The causes of this may be the loose structure of the metal layer which to a considerable extent is a copy of the structure of the substrate; the fine grain of the coating associated with condensation of the metal on the relatively cool substrate (PÉT film does not permit heating to more than 400°K) and leading to a more important role of the grain boundaries, and also oxidation of these boundaries when the vacuum in the evaporation process is not high enough (10^{-2} Pa) .

It is known that the optical properties of a metal are correlated with its electrical resistivity [11]. In the literature there are practically no data on absorptivities and electrical resistivities of metals measured on the same specimens.

In the present work we carried out such measurements for foils and metalized PET films. Electrical resistivity was determined at 293, 77, and 4.2°K with an error not exceeding 10% from the results of measurements of resistivity by the potentiometric method with the aid of a four-way potentiometric circuit.

The results of the investigation of the optical and electrical properties of metals are presented in Fig. 2 in the form of the dependence of the absorptivity of metal on its electrical resistivity. The experimental data lie near a straight line in logarithmic coordinates and are approximated by the expression.

$$A = 6\rho^{0.44} \,. \tag{6}$$

The obtained results show that metallized PÉT films, which have higher absorptivity than the corresponding massive metals, are also characterized by higher electrical resistivity.

Measurements with aluminum foil confirm that the chemical purity of the metal has a substantial effect. Reducing the content of impurities leads to an increase of the reduced resistivity ρ (293°K)/ ρ (4.2°K) which for foil A99 is 14.5 times greater than for foil A5, and more than 17 times greater than for foil AD1 (99.3% A1). The deviation of the points for A99 from the approximating dependence is due to the effect of the surface roughness of the foil which does not manifest itself in measurements of electrical resistance but increases the absorptivity of the surface. The deviation from dependence (6) obtained for foil of silver Sr999.9 (99.99% Ag) at 4.2°K is connected with the increased absorptivity of the surface of the vessel to which this foil is glued. In the experiments narrow foil was used which; when glued to the vessel, forms a large number of joints through which glue may emerge; this impairs the reflective properties of the surface.

In Fig. 2 we also plotted the experimental data on electrical resistivities and absorptivities of annealed pure copper and manganin at 293 and 90°K obtained in [12]. It can be seen in the figure that these results lie on the approximating dependence.

For a copper coating applied to mechanically and chemically polished copper by the method of vaporization in a pure helium atmosphere, Larin [10] obtained the value A $(4.2^{\circ}K) = 0.0006$.

Dubinina et al. [14] carried out an electron microscope analysis of the structure of similar films, and this showed that they have grains that are single crystal formations 1-3 μ m large. Taking the residual resistance in this case as $1 \cdot 10^{-9} \ \Omega \cdot \text{cm}$ [1], we obtain good agreement with dependence (6).

A comparison of the values of reduced resistivity from Table 1, viz. ρ (293°K)/ ρ (4.2°K), of the investigated materials (this resistivity characterizing the effect of chemical purity of the metal and of defects of its structure) with the values of A (4.2°K) of the corresponding metals shows that the metallic surfaces that have the smallest values of A (4.2°K) are characterized by the largest values of the ratio ρ (293°K)/ ρ (4.2°K).

Thus reduced electrical resistivity may serve as criterion in the selection of material reflecting thermal radiation of a coating for low-temperature installations.

In addition to the experimental data, Fig. 2 also contains the curves calculated for copper by the Davisson-Wicks (the classical electromagnetic theory) [14] and the Domoto formulas (theory of the anomalous skin effect) [15]. The values of ρ (T) necessary for plotting them were taken from [1].

A comparison of the theoretical and experimental dependences in Fig. 2 shows that the classical theory yields the largest discrepancy with experiments, and this increases with decreasing ρ and A, attaining one order of magnitude or more. In accordance with the classical theory, the absorptivity of a metal does not depend solely on its electrical resistivity but also on the spectral composition of the incident radiation (or, if integral characteristics are involved, on the temperature of the radiation source). This found expression in the corresponding formulas of the form $A_{\lambda} = f(\sqrt{\rho/\lambda})$ or $A = f(\sqrt{\rho T})$, where ρ is a function of the temperature. It was already shown above that for $\lambda > 10 \ \mu m$ metals are characterized by the magnitude $A_{\lambda} = A = \text{const.}$ This conclusion is in good agreement with the empirical formula (6) obtained in the present work in accordance with which the hemispherical integral absorptivity of metal in the longwave part ($\lambda > 10 \ \mu m$) of the infrared range of the spectrum depends solely on its electrical resistivity. The dependence of the latter on the temperature also determines the temperature dependence of the absorptivity of a metal.

Taking the anomalous skin effect (ASE) into account enables us to obtain better agreement with the experimental data; however, calculations according to the theory of ASE are fairly complex and require knowledge of the temperature dependences of characteristics of a metal such as the speed of electrons on a Fermi surface, the concentration of conduction electrons, the effective mass of an electron.

The nature of the dependence of the absorptivity of a metal on its electrical resistivity brings to mind the possibility of using superconductors as ideal reflectors of thermal radiation at helium temperatures. Such a possibility was examined theoretically by Jones [16] who obtained by calculation the values $A = 6 \cdot 10^{-6} - 1 \cdot 10^{-7}$ for niobium in the superconducting state at temperatures T = 4.2 - 2.0 °K.

Rastorgueva [4] was the first to measure the integral hemispherical emissivity of niobium at temperatures of 5-360°K. The lowest value of this parameter was obtained at T = 9.2-9.3°K (temperature of the superconduction transition); it was $\varepsilon \equiv A = 0.04$. According to the author's data, a further lowering of the temperature to 5°K led to an increase of A to 0.055.

In the present work we obtained the absorptivity of niobium A $(4.2^{\circ}K) = 0.032$. Measurements of the electrical resistivity of the same specimen showed that at $4.2^{\circ}K$ it does not pass into the superconducting state. This is due to the fact that the foil used in our experiments was not heat-treated for relieving internal stresses after production (annealing at 1473-1763°K for 10 h in a vacuum of 10^{-3} Pa with the use of oil-free means of evacuation). Unfortunately, the thesis [4] does not contain any information on the technology used in producing the niobium specimen.

We measured the absorptivity of yet another superconductor - lead - at 4.2°K (transition temperature is 7.2°K) vaporized in a vacuum of 10^{-2} Pa on the surface of steel Kh18N10T. Regardless of the fact that with such a residual pressure the lead may oxidize during the vaporizing process, we obtained a coating with the value A (4.2°K) = 0.009 which is close to silver vaporized in vacuum, with a higher value of the ratio A (293°K)/A (4.2°K) than silver has.

Thus, from the results of measurements of the optical and electrical properties of various metals we obtained the empirical dependence (6) which makes it possible reliably to

evaluate quantitatively the integral hemispherical absorptivity of a metal with an optically smooth surface with specified temperature according to the electrical resistivity of this metal measured at the same temperature, without carrying out fairly complex and lengthy calorimetric and radiational measurements (especially under conditions of vacuum and of cryogenic temperatures).

NOTATION

 Q_m , Q_r , Q_t , $Q_{r.g}$, Q_h , heat fluxes: total measured, radiational, along the orifice, by heat conduction of the residual gas, from the heater, respectively; ε_{rd} , reduced degree of blackness of the system vessel-screen; A, A_{λ} , integral and hemispherical absorptivity, respectively; σ_0 , Stefan-Boltzmann constant; F_c , F_s , surface area of the inner vessel and of the screen, respectively; T_c , T_s , T_0 , temperature of the vessel, of the screen, of the medium, respectively; M, molecular mass; γ , ratio of isobaric to isochoric heat capacity; α , accommodation coefficient; p, residual gas pressure, Pa; δ , thickness of the metal coating on PET film; ρ , electrical resistivity; λ , wavelength of the radiation.

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