INVESTIGATION OF SPECTRAL THERMORADIATION CHARACTERISTICS OF SOLID NONTRANSPARENT MATERIALS

O. E. Khlebnikov, A. A. Khalatov, and O. V. Shikhabutinova

UDC 666.762:669.045.2

A description of an experimental plant and results of an investigation of spectral emissivity and reflectivity of ceramic materials in heating in the air as well as measurements of thermoradiation characteristics of refractory materials with a blackening coating are presented.

Improvement of the thermal protection of elements of structures operating at high temperatures is determined in the first place by the choice of materials with the required radiation properties in the infrared spectral range. Most materials, such as ceramics and alloys, are not gray emitters in the spectral range of the radiation intensity maximum at temperatures of 600-1500 K, and hence for an accurate analysis of heat transfer a knowledge of their spectral characteristics is essential. Among these characteristics are the spectral two-directional reflectivity, most completely describing the interaction of radiation with the solid surface, and the emissivity of materials. The purpose of this work is to develop the method and to measure the spectral reflection and emission coefficients for a wide class of nontransparent materials.

The experimental plant (Fig. 1) based on two independent procedures for measuring thermoradiation characteristics is designed to determine the temperature and spectral dependences of the emissivity and reflectivity of solids over the range 1-15 μm at temperatures of 300-1500 K.

The first procedure utilizes the rotating specimen method [1], suitable for an investigation of both current-conducting materials and dielectrics. For the investigation we used specimens in the shape of a cylinder with a groove over the cylinder surface generatrix or the specimen made of two halves, each being in the shape of a cylinder with a facet on one end, i.e., the black-body model (BBM) is realized in the very specimen in the form of a rectangular groove with a width-to-depth ratio equal to 1/2, or in the form of a wedge-like cavity with an apex angle of 30°.

In the body of the resistance furnace 1 the investigated specimen 2 is installed on an alundum bar, which is rotated by an electric motor at a speed higher than 180 rpm, which ensures a constant radiation intensity of the surface under study. The specimen is heated by a nichrome coil 3, packed into the frame of the heat insulation 18 of chamotte ultralightweight material. Electric power is supplied to the heater through special current suppliers, electroinsulated from the furnace body. Regulation and stabilization of the furnace temperature is performed by a VRT-3 precision temperature regulator 16 within ± 0.5 K with the aid of a chromel–alumel thermocouple located near the heater coil. The furnace operating zone temperature is measured by a second chromel–alumel thermocouple and an Shch 68003 digital voltmeter. By an EOP-66 optical pyrometer 17 the specimen temperature is measured by sighting on the slot groove through a special hole in the furnace body. Data of the sensors are recorded by an SM-4 computer.

The specimen radiation intensity and BBM are measured through the furnace window by an IKS-21 infrared spectrometer 9. To cut off the natural radiation of the furnace and to record solely the intrinsic radiation of the specimen, a water-cooled diaphragm 4 is used. A signal from bolometer 20, proportional to the radiation intensity, goes through the EPS-241 amplifier, is measured by the Shch 68003 digital voltmeter, and is recorded by the SM-4 computer.

To determine the radiation characteristics of the materials, a direct method is adopted, i.e., a method of comparing the radiation of the specimen in question with the radiation of the BBM. The measuring procedure is as follows.

After the furnace temperature has been brought up to the required stationary level, the radiation of the rotating specimen and the BBM is alternately directed using the mirrors of the spectrometer illuminator (the globar in the illuminator having been taken off) to the inlet slot of the monochromator. The alternate setting of the specimen and the BBM is performed by displacing the bar with the specimen along the axis of rotation with respect to the water-cooled diaphragm.

Institute of Technical Thermal Physics, Academy of Sciences of Ukraine, Kiev. Translated from Inzhenerno-Fizicheskii Zhurnal, Vol. 64, No. 3, pp. 318-323, March, 1993. Original article submitted March 12, 1992.

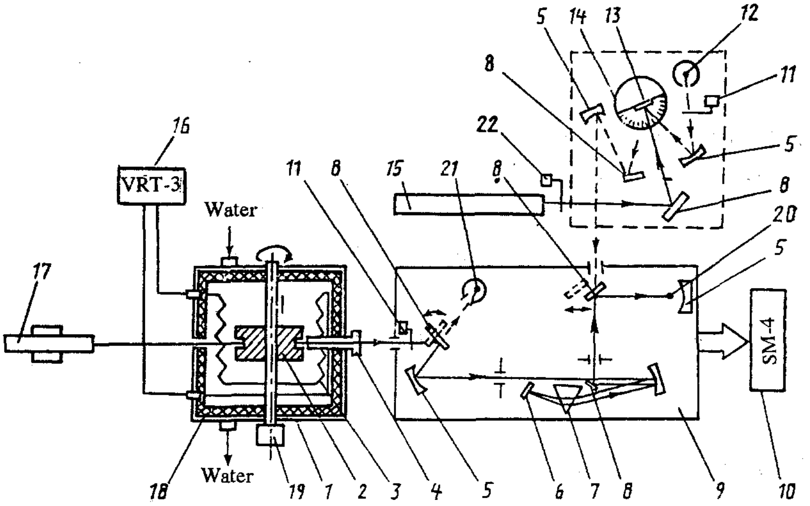

Fig. 1. Experimental plant: 1) high-temperature chamber; 2) specimen; 3) heater; 4) cooled diaphragm; 5) spherical mirror; 6) Litthrow mirror; 7) prisma NaCl; 8) flat mirror; 9) IKS-21 spectrometer; 10) data recording system with an SM-4 computer; 11) modulator; 12) emitter; 13) specimen; 14) rotating table of the IPO-12 attachment; 15) ILGN-709 CO_2-laser; 16) VRT-3 precision temperature regulator VRT-3; 17) EOP-66 optical pyrometer; 18) heat-insulation unit; 19) electric motor; 20, 21) bolometers; 22) shutter.

The spectral normal emissivity is calculated from the relation

$$\varepsilon_\lambda = \varepsilon_m \frac{I_\lambda}{I_{\lambda m}}, \tag{1}$$

where I_λ and $I_{\lambda m}$ are the radiation intensities for the specimen and the model; ϵ_m is the emissivity of the model.

The emissivity of the model was determined as a third-order polynomial from reference data [2] for the dependence of ϵ_m on the emissivity of the material of the cavity walls with the assigned geometry of the model:

$$\varepsilon_m = A_1 + \varepsilon_\lambda A_2 + \varepsilon_\lambda^2 A_3 + \varepsilon_\lambda^3 A_4, \tag{2}$$

where the coefficients of the polynomial A_1, A_2, A_3, and A_4 were determined by the least-squares method. For a cavity with depth $h = 6$ mm and width $l = 3$ mm, they have the follwing values: $A_1 = 0.0929651694$; $A_2 = 2.82229755$; $A_3 = -3.62457606$; $A_4 = 1.74583009$.

Since the spectral emissivity ϵ_λ enters in Eq. (2) as a parameter, not known in advance, the emissivity of the specimen was calculated by the iteration method in a joint solution of Eqs. (1) and (2). The error of measuring of the specimen emissivity was estimated taking into account the imperfection of the BBM, the influence of the background, the cooling of the specimen surface when passing near the diaphragm, the imperfection of the optical scheme, etc. The error of measuring the spectral emissivity does not exceed 9% at a temperature of 1500 K.

The second investigation procedure for the thermoradiation characteristics is based on measurements of spectral reflectivity.

One such characteristic, most completely describing the interaction of radiation with the surface of a solid is the spectral two-directional reflectivity [1]:

$$R_\lambda(\theta', \varphi', \theta, \varphi) = \frac{dI_\lambda^{\mathrm{refl}}(\theta', \varphi', \theta, \varphi)}{I_\lambda^{\mathrm{inc}}(\theta', \varphi')\cos\theta' a\omega}, \tag{3}$$

where $dI^{\mathrm{refl}}_\lambda(\theta', \varphi', \theta, \varphi)$ is the intensity of the reflected radiation in the direction of the polar θ and azimuthal φ angles of reflection; $I^{\mathrm{inc}}_\lambda(\theta', \varphi')$ is the intensity of the incident radiation in the direction of the polar θ' and azimuthal φ' angles of

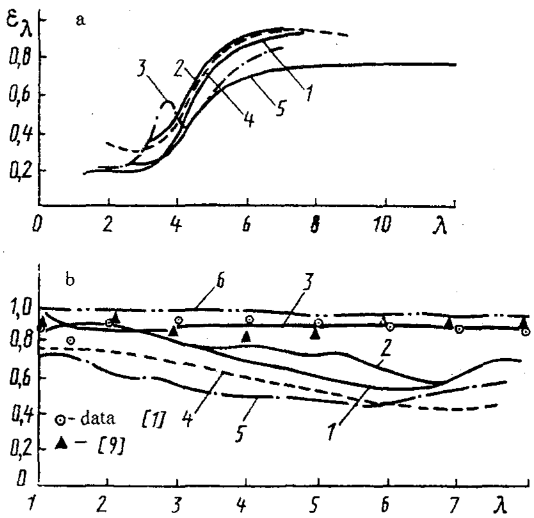

Fig. 2. Spectral emissivity of materials: a) chamotte ceramics: 1) ultralightweight chamotte (T = 1400 K); 2) lightweight chamotte (T = 1500 k); 3) chamotte (T = 1300 K); 4) chamotte (T = 573 K [5]); 5) foam-chamotte (T = 573 K) [5]; b) sintered powdered chromium (1-4) at T = 1500-1600 K, ferro-aluminum high-resistance alloy at T = 1500 K and carbon-carbon material (6) at T = 1400 K: 1) oxidation 0.5 h; 2) 1.5 h; 3) 6 h; 4) calculation from R_λ at room temperature; 5) oxidized surface.

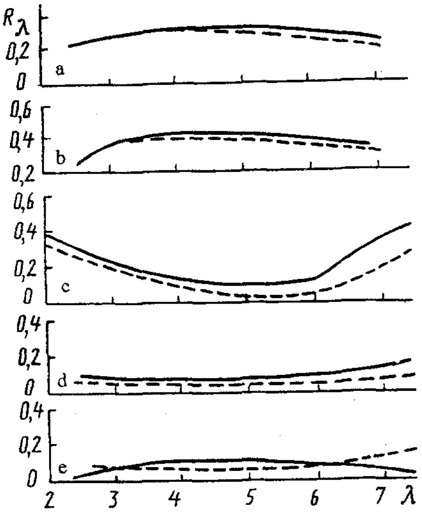

Fig. 3

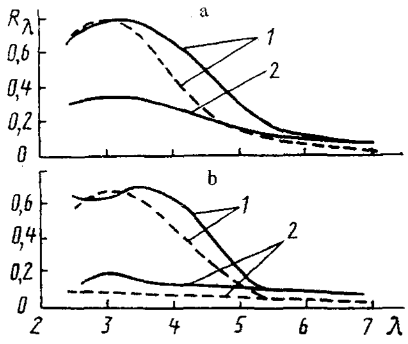

Fig. 4

Fig. 3. Spectral reflection coefficient of specimens: a) alloyed aluminum oxide; b) industrial aluminum oxide; c) alumobornitride ceramics; d) lanthanum chromite; e) silicon carbide; solid curves correspond to T = 300 K, dashed ones to 1500 K.

Fig. 4. Spectral reflection coefficient of refractories with no covering (1) and with a covering (2): a) ultralightweight chamotte; b) lightweight chamotte; solid curves correspond to T = 300 K; dashed ones to 1500 K.

incidence in the solid angle $d\omega$. With a fixed azimuthal angle $\varphi' = \varphi$ in Eq. (3) there remains only the dependence on the angles θ' and θ. From Eq. (3), other radiation characteristics can be obtained, for example, the directional-semispherical reflectivity:

$$R_\lambda(\theta') = \int_0^{2\pi} R_\lambda(\theta', \theta)\cos\theta d\theta \tag{4}$$

and in accordance with Kirchhoff's law – the directional emissivity:

$$\varepsilon_\lambda(\theta') = 1 - R_\lambda(\theta'). \tag{5}$$

The basis of the experimental plant (Fig. 1) is formed by the ILGN-709 radiation heater CO_2 laser 15 with shutter 22, the monochromator of the IKS-21 infrared spectrometer 9, the IPO-12 optical attachment, permitting measurements at various angles of incidence and reflection from 10 to 85°. This is attained by moving the rotating table 14 with specimen 13 in the plane of the angle of incidence, simultaneously turning the emitter unit. Using this attachment, diffusivity of the specimen surface reflectivity was checked. The specimens were manufactured 30 mm in diameter and 6-10 mm thick.

Radiation from the globar 12 modulated with a frequency 9 Hz was focused on the specimen surface for a fixed angle of incidence. In measuring $R_\lambda(\theta', \theta)$ a reverse path of the ray in the monochromator was used. The reflected ray was directed into the spectrometer outlet slot, was further dispersed by an NaCl prism, and arrived at the bolometer 21 through the inlet slot. A signal from the bolometer was amplified by the EPS-241 device and was recorded by the CM-4 computer.

The reflection coefficient was determined by a relative method for the fixed wavelengths from the relation [3]

$$R_\lambda(\theta', \theta) = \frac{I_\lambda(\theta', \theta) R_\lambda^{st}}{I_\lambda^{st}(\theta', \theta)}, \tag{6}$$

where R^{st}_λ is the reflection coefficient of the standard; $I^{st}_\lambda(\theta', \theta)$ and $I_\lambda(\theta', \theta)$ are the intensities of the reflected signal from the standard and the specimen. The powder NaCl, having near-diffuse reflectivity, served as a standard. A layer of the powder was applied to the end surface of a metal cylinder using vacuum lubrication and then was set by a window glass.

The investigation of the reflection coefficients on the IPO-12 optical attachment was performed at fixed wavelengths. The angles of incidence and reflection were varied and lay within the limits 10-85°. The monochromator was calibrated in terms of absorption spectra for chloroform, water vapor, and carbon dioxide in the atmosphere. The specimen temperature was measured by a chromel–alumel thermocouple, tightly pressed against the investigated surface. The error of measurements of reflectivity for the specimens does not exceed 10% with a confidence coefficient 0.95.

The results of measuring the spectral normal reflectivity (Fig. 2) suggest the selectivity of properties of chamotte refractories. A characteristic feature of chamotte materials is an increase in the emissivity in the range 3-5 μm, which is confirmed by a qualitative agreement between our measurements and the data of other authors [1, 4, 5, 8] for specimens similar in composition.

The spectral reflection coefficient of the investigated materials (Fig. 3) also suggests the selectivity of reflection of chamotte refractories in the near-infrared spectral region. The reflectivity of chamotte materials was investigated in the air at room temperature and at 1500 K. A variation of the monochromatic reflectivity (Fig. 3) and emissivity (Fig. 3) of alumobornitride ceramics is characterized by minimum values in the range 4-6 μm. For the remaining materials – ceramic specimens of aluminum oxide, silicon carbide, carbon–carbon composite [6], ceramic chromium, and lanthanum chromite – the reflection coefficient weakly depends on a the wavelength.

Heating of the specimens does not have a substantial effect on the thermoradiation properties. The temperature variation of the reflectivity of materials except for chamotte (Fig. 4) and alumobornitride ceramics is insignificant and lies within the limits of experimental error.

The measurements performed have shown a substantial variation in the thermoradiation characteristics of metal specimens (see Fig. 2b) in the process of oxidation in heating in the atmosphere.

Apart from measurements of the thermoradiation characteristics of refractory materials, investigations of the spectral reflectivity of a blackening coating (Fig. 4) [7] used to increase the surface reflectivity were conducted. Four components – metal oxides (wt. %): Fe_2O_3 (18-20); Cr_2O_3 (41-44); NiO (29-32); and Mn (7-9) – enter into the composition of the coating. The coating offers resistance to heat and to combustion products. From the data obtained it follows that the reflection coefficient of refractories substantially decreases after the coating has been applied; the maximum variation occurs in the short-wave spectral region for chamotte specimens, i.e., materials having a high reflectivity in the range 2-5 μm.

The blackening coating changes the character of the spatial distribution of the reflected energy depending on the angles of incidence and reflection. Without the coating, chamotte specimens do not possess the property of an ideal diffuse reflector. This is confirmed by investigations performed in [4, 5], where the angular distribution of the reflected radiation from compounds similar in chemical composition is determined. The blackening coating, as follows from the results of measurements, reduces the mirror component of the reflection for a refractory and has diffuse reflection at angles up to 50°.

In conclusion, it should be pointed out that the experimental plant developed for measuring thermoradiation characteristics of materials at temperatures of 300-1500 K makes it possible to investigate both the emissivity and reflectivity with no possibility of determining a particular characteristic by one of the procedures and with the aim of mutually checking the results of measuring ϵ_λ and calculating the emissivity from $R_\lambda(\theta')$ according to Kirchhoff's relation. Agreement of the results of measurements and calculations of ϵ_λ (Fig. 2b) for chamotte refractories indicates the correctness of the measuring procedures.

NOTATION

T, temperature; ϵ, R, emissivity and reflectivity of material; *I*, radiation intensity (brightness); λ, radiation wavelength; θ, φ, polar and azimuthal angles of reflection; θ', φ', polar and azimuthal angles of incident radiation; d_ω, elemental solid angle. Subscripts: λ, spectral value; m, model; refl, reflected; inc, incident; st, standard.

LITERATURE CITED

1. A. E. Sheidlin (ed.), Emitting Properties of Solid Materials [in Russian], Moscow (1974).
2. E. M. Sparrow and R. D. Sass, Heat Transfer by Radiation [Russian translation], Leningrad (1971).
3. L. S. Slobodkin and Yu. M. Sotnikov-Yuzhik, Methods for Determination of Thermoradiation Properties of Polymer Coatings [in Russian], Minsk (1977).
4. V. N. Zapechnikov, V. T. Pushkin, and A. G. Zen'kovskii, Izv. Vyssh. Uchebn. Zaved., Chern. Metallurg., No. 1, 121-125 (1985).
5. V. N. Zapechnikov, V. T. Pushkin, and A. G. Zen'kovskii, Izv. Vyssh. Uchebn. Zaved., Chern. Metallurg., No. 5, 129-132 (1986).
6. O. E. Khlebnikov, A. A. Khalatov, and O. V. Shikhabutinov, Prom. Teplotekh., **13**, No. 3, 84-87 (1991).
7. O. E. Khlebnikov, A. A. Khalatov, and V. S. Pikashov, Prom. Teplotekh., **10**, No. 5, 84-86 (1988).
8. V. G. Abramovich and V. L. Gol'dshtein, Intensification of Heat Transfer by Radiation Using Coatings [in Russian], Moscow (1977).
9. R. I. Hayes and W. H. Alkinson, Ceramic Bulletin, **3**, No. 7, 616-622 (1964).