NEW METHODS OF DETERMINING THE RADIATION CHARACTERISTICS OF MATERIALS AT LOW TEMPERATURES

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The authors describe two new methods of measuring the spectral hemispherical reflectance of substances in the condensed state at low temperatures.

In recent years there has been increasing interest in the question of determining the radiative characteristics of materials at low temperatures [1, 2].

One of the basic methods of obtaining information on the radiative properties of solid materials at low temperatures is the method based on the use of an integrating sphere with a centrally located specimen [3, 4]. It consists of placing the cooled specimen at the center of an evacuated spherical volume, the inner surface of which is coated with material with high diffuse reflectance. The modulated emission of an external source is introduced into the volume, and by comparing the signals from the specimen and the internal coating of the sphere we determine the hemispherical directional reflectance R_{sp} of the specimen. In the case considered here this method has a number of serious shortcomings, arising from: a) the need to investigate a special coating for the inside surface of the sphere, having a high reflectance and an angular distribution of reflected flux that is close to Lambert type; b) the substantial reduction of the useful signal at wavelengths greater than 4 μm; c) the increase of systematic error, beginning at $\lambda = 3$ μm and beyond, for measurements by the two-beam method, because of the temperature difference between the cooled specimen and the "room" temperature of the radiative detector [4].

Nevertheless the integrating sphere method remained essentially the only method until recently for measuring the radiative properties at low temperatures [5].

One of the developed and approved methods is based on using the principle of a negative radiative flux [6]. We note that the negative light fluxes were first used for spectroscopic studies in [7, 8]. We now examine the essentials of this method of measuring the radiative properties of materials at cryogenic temperatures with the aid of a block diagram for implementing the method (Fig. 1).

The test specimen 1 and the radiative standard 2 are located in separate identical evacuated cavities 3 and 4 which have apertures for radiative exchange with the detector (the inside walls of the cavities are blackened), and are in essence models of an absolute black body (ABB).

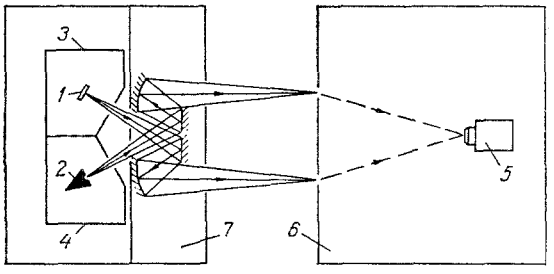

Fig. 1. Block diagram of the method of measuring the directional emittance of materials at cryogenic temperatures.

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The radiative standard in turn is also a model of an ABB. The specimen and the radiative standard are cooled to a given temperature (e.g., by liquid nitrogen) and subsequently their temperatures are kept the same. Here the temperature of the walls of cavities 3 and 4 is maintained equal to that of the thermal detector 5 of the two-beam infrared spectrophotometric system 6, which also records the ratio of the negative radiative fluxes arising from specimen 1 and standard 2.

This method can be accomplished using any commercial two-beam infrared spectrophotometer with special devices, including the optical attachment, the cavities, and systems for evacuating the cavities, cooling the specimen and the standard, and controlling the temperatures.

With the two-beam method of recording radiative fluxes we can write down the ratio of the intensities of the resultant fluxes in the optical channels of the specimen I and the standard II. The optical attachment 7 creates images of the specimen and the standard at the entrance slits of the spectrometer.

We shall now show that with this scheme one can measure the directional emittance of an object that is opaque at thermal wavelengths. To do this we consider the balance of the radiative fluxes in channels I and II.

The resultant radiative fluxes in the channels of the specimen and standard, respectively, can be written as

$$Q_{\lambda\,\mathrm{sp}} = K\left[\varepsilon_{\lambda \mathrm{d}} E_\lambda(T_\mathrm{d}) - \varepsilon_{\lambda\,\mathrm{sp}} E_\lambda(T\mathrm{sp}) - R_{\lambda\,\mathrm{sp}} E_\lambda(T_\mathrm{w})\right], \quad (1)$$

$$Q_{\lambda \mathrm{st}} = K\left[\varepsilon_{\lambda \mathrm{d}} E_\lambda(T_\mathrm{d}) - \varepsilon_{\lambda \mathrm{st}} E_\lambda(T_{\mathrm{st}})\right]. \quad (2)$$

The coefficient K is postulated to be the same for the two channels of the measuring system.

It is a condition of the measurements that the temperatures of the specimen and the standard and also of the walls of cavities 3 and 4 must be maintained the same in pairs. In addition, the radiative detector should be at the temperature of the cavity walls. Let us also assume that the radiative properties of the detector and the standard are close to perfect over the entire thermal region of the spectrum, i.e., we put $T_{sp} = T_{st}$, $\varepsilon_{\lambda d} = \varepsilon_{\lambda st} = 1$ in Eqs. (1) and (2).

In Eq. (1) the first term describes the emission of the sensor at the temperature of the surrounding medium, the second term describes the self-emission of the specimen, and the third term describes the emission of the cavity walls at the temperature of the surrounding medium, reflected by the specimen in the solid angle in which the optical system views the specimen.

Under the conditions considered the balance equations are simplified appreciably:

$$Q_{\lambda \mathrm{sp}} = K\left[E_\lambda(T_\mathrm{s}) - \varepsilon_{\lambda\,\mathrm{sp}} E_\lambda(T\mathrm{sp}) - R_{\lambda\,\mathrm{sp}} E_\lambda(T_\mathrm{s})\right], \quad (3)$$

$$Q_{\lambda \mathrm{st}} = K\left[E_\lambda(T_\mathrm{s}) - E_\lambda(T_{\mathrm{sp}})\right]. \quad (4)$$

The responses of the detector in the specimen and standard channels are proportional to the corresponding resultant fluxes

$$I_{\lambda \mathrm{sp}} \sim Q_{\lambda \mathrm{sp}} \text{ and } I_{\lambda \mathrm{st}} \sim Q_{\lambda \mathrm{st}}$$

The quantity γ_λ recorded by the two-beam spectrometric system is equal to the ratio of intensities in the two optical channels:

$$\gamma_\lambda = I_{\lambda \mathrm{sp}} / I_{\lambda \mathrm{st}} = \varepsilon_{\lambda \mathrm{sp}}$$

In accordance with the Kirchhoff law here we use the relation $1 - R_{\lambda sp} = \varepsilon_{\lambda sp}$. Thus, with this method we can measure the directional emittance of opaque materials at low temperatures.

To estimate the region of application of the method we used Eq. (3) to calculate the spectral curves of resultant flux for two substantially different specimen temperatures Fig. 1). As can be seen from these data, with the method one can measure the emissive properties of materials at cryogenic temperatures in the spectral region from 3 to 25 μm. Here the maximum sensitivity of the method lies in the range 6-12 μm (depending on the object temperature).

We determined the directional hemispherical reflectance of ammonia, carbon dioxide, and sulfur at liquid nitrogen temperature (∿80°K) (Fig. 3). Comparison of the data obtained with

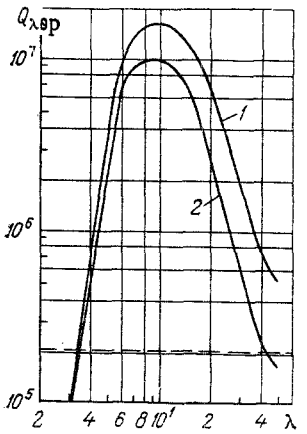

Fig. 2

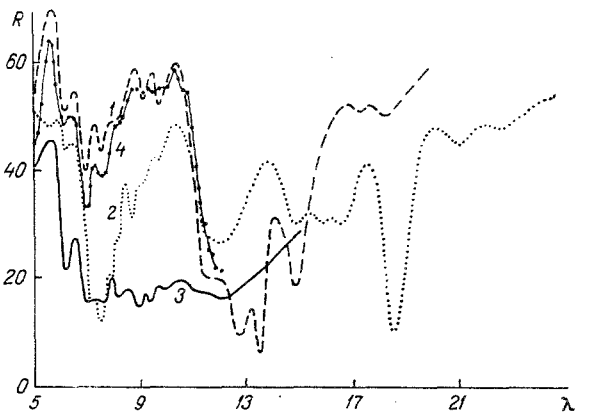

Fig. 3

Fig. 2. Calculated spectral curves of resultant flux density in the specimen channel $Q_{\lambda sp}$ (the scheme of Fig. 1) for cavity and radiative detector temperature of 300°K: 1) $T_{sp} = 80$ K; 2) 250; the broken line shows the threshold sensitivity of the commercial type BMK-60 bolometer. The quantity $Q_{\lambda sp}$ is in W/m^3, and λ is in μm.

Fig. 3. Directional hemispherical reflectance R of cryocondensates of gases: 1) CO_2; 2) SO_2; 3) NH_3; 4) CO_2 [3]. The quantity R is in percent, and λ is in μm.

literature data, e.g., the CO_2 spectrum of [3], shows good agreement.

This method is simpler to implement, compared with the integrating sphere method, because there are no requirements as to the optical properties of the inside surfaces of the evacuated cavities. In addition, the method has the maximum sensitivity in the thermal spectral region where the traditional methods have low sensitivity.

The essentials of the second possible method for measuring the emittance characteristics of materials at low temperatures [9] are as follows. The specimen is also placed in a cavity with a blackened inside surface, and the cavity is then evacuated. Then the specimen and the detector are cooled to a given temperature, and the measurements are taken by comparing signals from the internal wall of the cavity and the specimen. In this case the source of the standard radiation is the cavity itself, which has the temperature of the surrounding medium. Here the useful signal will be determined only by the reflective properties of the specimen, since the temperatures being equal means that the resultant radiative fluxes between the specimen and the detector will be zero. To convince ourselves that this is true, we obtain expressions for the resultant flux density at the detector.

When viewing the specimen and the cavity walls the response of the detector is, respectively,

$$I_{\lambda sp} \sim K[R_{\lambda sp} E_\lambda(T_w) + \varepsilon_{\lambda sp} E_\lambda(T_{sp}) - E_\lambda(T_d)], \quad (5)$$

$$I_{\lambda st} \sim K[E_\lambda(T_w) - E_\lambda(T_d)]. \quad (6)$$

It is assumed that $\varepsilon_d = \varepsilon_w = 1$. The ratio of these signals gives

$$\gamma_\lambda = I_{\lambda sp}/I_{\lambda st} = R_{\lambda sp}$$

Thus, we obtain the result that the measured quantity γ_λ is the spectral directional hemispherical reflectance of the specimen.

The spectral regions of application of the two methods described coincide, but we can accomplish the second more simply, since it does not require the temperatures between two pairs of objects to be equated, but requires the use of a cooled radiation.

Consequently, the proposed methods for determining the emittance characteristics (hemispherical reflectance) of opaque substances in the solid state at cryogenic temperatures offer the possibility of measuring these characteristics with a high signal-to-noise ratio (greater

than 10) in the thermal spectral region (4-25 μm) where traditional methods have low sensitivity.

NOTATION

Q, resultant radiative fluxes; T, absolute temperature; E(T), radiative density of an absolutely blackbody at temperature T; R, hemispherical directional reflectance; ε, directional emittance; K, optical-geometric factor; I, detector response to incident radiation; γ, measured quantity. Subscripts: λ, spectral quantity; sp, specimen; st, standard; d, radiative detector; s, surrounding medium; w, cavity walls; es, external source of radiation.

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