MONOCHROMATIC REFLECTIVITY OF POWDERED INORGANIC MATERIALS

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Monochromatic reflectivity is measured on compacted specimens of powdered inorganic materials. The measured spectra are compared with literature data.

The monochromatic reflectivity of materials is one of the parameters used to calculate radiative heat transfer. However, there have been few studies of this subject [1]. The dearth of information has to do with the difficulty of determining reflectivity. Determination of the monochromatic reflectivity of powdered materials is especially challenging.

Here, we use the method of mirror reflection to determine the monochromatic reflectivity of powdered materials.

A mirror surface is created on a specimen by a specially-developed method involving compaction in a vacuum [2]. Powdered materials without a binder are used for this pressing operation. The only prerequisite is that the granular composition of the powder be sufficiently fine. The proposed method of compaction makes it possible to study the mirror reflectivity of powdered materials by established methods and to use standard spectral equipment.

Dry materials with a granular composition finer than 63 μm were used for pressing. To achieve such a composition, the material was first sifted through a screen with a 63-μm mesh and then dispersed. Only the ultrafine fractions of the powder were admitted to the die that was used for compaction. The pressing operation employed a bilateral application of pressure.

The die was made of steel quenched to a hardness HRC $\geq$ 62. The working surfaces of the die, in contact with the powder, were subjected to grinding, polishing, and lapping to a finish close to class 14 ($R_z > 0.063$ μm). Planarity was no lower than 10 Newton rings. The finished specimens had the form of parallelpipeds with the dimensions 20 × 18 × 3 mm.

The shaping of the specimen resulted from simultaneous bilateral contraction and shearing movement of the die relative to the powder. Friction caused the working surface of the die to entrain the particles of the surface layer, bringing them into motion. Here, the lateral pressure distributed the fine particles among the coarse particles, pressing them tightly into the surface layer. The side of the die smoothed out the particle layer, so to speak, and formed a solid surface having mirror reflectivity.

In adjusting the pressing regime, we simultaneously checked the mirror surface visually with a microscope. We also checked the minerological composition on an x-ray diffractometer. The pressing regime was considered to have been correct if the results of the diffraction analysis showed that there were no differences in the mineralogical composition of the particles making up the mirror surface of the specimen and the initial particles of the powder. Thus, it could then be assumed that the material of the mirror surface had the same properties as the bulk material, i.e., the optical properties of the mirror surface of the pressed specimens corresponded to the optical properties of the particles making up the specimen.

In general, the mirror surface was an adnate solid surface in which the particles of powder were jointed into a single whole. However, pores appeared between the particles located deeper in the specimen. Here, the particles simply touched one another without having grown together. The depth of the solid surface on the specimens averaged 0.6 mm.

The mirror surface of the specimens consisted of several components in the thickness direction. Thus, the surface layer had a refractive gradient, which in turn led to multiple internal reflections. Measurement of the diffuse component of the radiation reflected from

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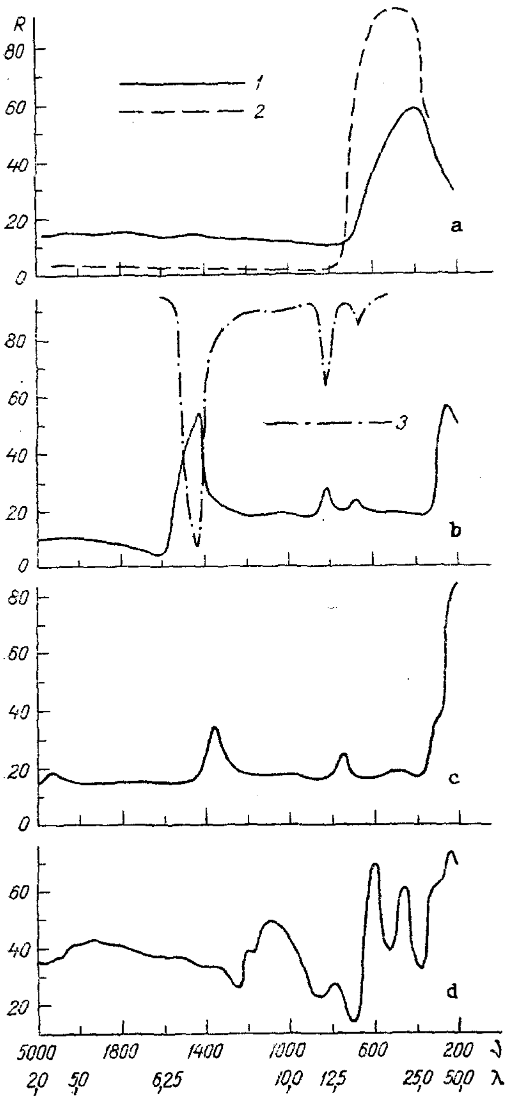

Fig. 1. Monochromatic reflectivity of materials: a) MgO; b) Na_2CO_3; c) KI; d) pyrite cinders; 1) our data; 2) data from [1, 4]; 3) position of carbonate absorption band, R, %; ν, cm^{-1}; λ, μm.

the mirror surface of the compacted specimens showed that this component was within the range 0.5-0.001% in the region 0.6328 μm (helium-neon laser). The exact magnitude of the component depended on the chemical and mineralogical composition of the powder and decreased with an increase in the wavelength of the probing radiation. The magnitude of the diffuse component was evaluated by the method in [3]. The transmissivity of the mirror surface of the pressed specimens was studied on specimens 0.6 mm thick. The specimens used for this determiantion were prepared by layer-by-layer removal of particles from the specimen surface until a layer of 0.6 mm thickness was obtained. Measurement of the transmissivity of the specimens in a cuvette between fluorite plates on an IKS-22 spectrophotometer showed that the mirror surface of the pressed material was opaque in the fluorite transmission region.

The monochromatic reflectivity of the pressed powdered materials was measured on IKS-14A and IKS-22V spectrophotometers with an IPO-22 adapter at an angle of 16°30' in the wavelength range 2-50 μm.

The reflectivity of the specimens was studied in natural light, since natural light is used in practical calculations of radiative heat transfer.

According to normative data, the systematic error of the measurements of monochromatic reflectivity consisted of the following components: ±2.5% due to light-scattering in the spectral instrument; ±2% due to polarization; 0.2% due to imprecision in the establishment of the wavelength.

The random error of measurement of monochromatic reflectivity was due mainly to the finish (roughness) of the working surface, instability of the chemical and mineralogical composition of the test specimen, and the imperfect reproducibility of the position of the reflection attachments for the standard and the specimen. As the standard, we used an aluminized mirror and a polished plate of germanium in the form of a wedge.

We used only pressed specimens to determine monochromatic reflectivity. Their long holding in air caused some changes in the mirror surface of the specimen.

Calculations showed that the random error was negligible compared to the systematic error. With a confidence level of 0.95, the maximum total systematic error of the determination of monochromatic reflectivity was ±3.6%.

As an example, Fig. 1 shows the monochromatic reflectivity of pressed specimens of an oxide, salts, and the product of the firing of iron pyrites in fluidized bed furnaces—pyrite cinders.

The monochromatic reflectivity of MgO determined here differs from the literature data [1, 4] (Fig. 1a). In [1, 4], however, reflectivity was measured from the freshly-cut surface of a crystalline specimen of MgO. We used powdered MgO consisting of several fractions and did not take into account the positions of the crystals. This approach effectively averaged out the reflectivity of MgO.

There is no detailed information on the monochromatic reflectivity of the salts we studied [5]. Only the position of the main bands of the absorptivity spectrum of acid residues is known [6]. It is evident from Fig. 1b that the bands of the reflectivity spectrum of Na_2CO_3 are in full agreement with the bands of the absorptivity spectrum of the carbonates.

The pyrite cinders (Fig. 1d) were taken from the dust-dized combustion products of a sulfuric acid production line.

Reflectivity in the infrared region is determined by vibrations of the crystalline lattice and characteristic vibrations. The lattice vibrations depend on the masses of the atoms comprising the crystal and the character of the chemical bond. The greater the atomic mass, the farther into the longwave region the lattice will absorb radiation. Absorption of radiation connected with lattice vibrations is also related to selective reflection (residual radiation). An increase in the degree of ionic bonding is accompanied by an increase in the intensity of absorption and selective reflection by crystalline lattices.

Thus, the monochromatic reflectivity of powdered materials was measured on pressed specimens. Sharp and narrow bands were recorded in the reflectivity spectra. The presence of selective bands makes it possible to identify these bands with the components that make up complex inorganic compounds. For example, the reflectivity spectrum of pyrite cinder includes distinct bands identified with Fe_2O_3 and SiO_2. An x-ray diffraction analysis showed that the cinder contained mainly Fe_2O_3, considerably less SiO_2, and very small amounts of other minerals. In view of the fact that pyrite cinder consists mainly of Fe_2O_3, the principal bands in the reflectivity spectrum belong to Fe_2O_3. The bands of SiO_2 — which do not coincide with those of Fe_2O_3 — appear against a background of Fe_2O_3 bands in the form of small peaks in the region of the frequencies 1190, 800-810, and 384 cm^{-1}.

Thus, the characteristic bands in the reflectivity spectrum of a complex inorganic compound can be used to judge the presence of its simplex components, i.e., to diagnose the composition of a complex compound from its reflectivity spectrum. However, this method of diagnosis is not reliable enough for complex compounds because of the overlap and additivity of the spectral bands of the contituent elements. In light of this, a correct diagnosis will require that the spectrum of the complex substance be augmented by information on the spectra of the constituent elements and the expected composition.

The above-described method of diagnosis is a supplement to established methods for determining the chemical and mineralogical composition of minerals.

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TWO-ZONE MODEL OF HEAT TRANSFER IN A FURNACE

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A model of heat transfer in a furnace is proposed. The model is in the form of a circular channel separated lengthwise into sections. Each section is represented by two coaxial zones. A single section models a one-chamber furnace.

All of the engineering methods used to calculate heat transfer deal with single volumes. The volume is usually characterized by an effective temperature. The attempts made to calculate this temperature have generally produced results which are unreliable and, in truth, could not help but be unreliable. The formula derived by A. M. Gurvich [1] for use in the normative method does away with the notion of effective temperature but is also of limited application [2]. In the new single-volume method developed in [3, 4], the nonisothermality of the medium is accounted for through different values for the heat flux on the surface of the volume. However, these differences necessitate the solution of an internal problem. Simultaneous solutions of internal and external problems have been obtained by zone methods, but they are complicated and fail to meet several requirements. Below, we study a relatively simple two-zone model.

1. Physical Scheme. If the combustion chamber is long (such as in a rotary or continuous furnace), it is represented in the form of a channel with a flowing medium. The channel is divided into sections, but until now the practice has been to calculate heat transfer in each section in accordance with a single-volume model. The estimate obtained in [2] showed that heat flux is considerably overstated and that there is a corresponding underestimation of the temperature of the outgoing gases. In the present investigation, the volume is subdivided into two coaxial zones. In a reverberatory furnace, it is possible to distinguish a flame core or central layer (CL) and a more or less conservative layer (NL) which separates the core from the heating surface.

In our model, planarity of the flame is not required. In large furnaces, this provision leads only to a local increase in heat transfer or necessitates the installation of multiple burners that complicate the design of the furnace. Uniformity of the heat fluxes is usually the foremost criterion in determining the quality of heating, so planarity is undesirable.

2. System of Bodies. A circular channel or variable cross section is divided into sections of constant cross section. Two of them, i, and i-1, are shown in Fig. 1. The volumes of the medium are represented by two coaxial layers, CL and NL. The surface of the channel receives heat uniformly. The fields of all pertinent variables are axisymmetric. The equations describe heat transfer in the i-th section with allowance for the flow of the medium entering from section i − 1. The parameters are recalculated at the boundary. If there is just one section, then this section models a one-chamber furnace.

3. General Conditions of the Heat-Transfer Problem. In the i-th section of the channel:

1) the temperature field does not change along the axis;

2) the central layer (CL) is isothermal; its temperature and the field in the NL are found in the course of solivng the problem;

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