Detection technique of the optical and thermoradiative characteristics with compensation effect of reflection and transmittance indicatrices for the semitransparent materials with high subsurface scattering

Vladimir G. Merzlikin¹ ², Andrey V. Bystrov³ and Sergey V. Smirnov³

¹Department of Technology and Equipment of Mechanical Engineering, Moscow Polytechnic Un, Bol’shaya Semenovskaya, 38, Moscow 107023, Russia
²Department of Industrial Economics, Plekhanov Russian Un of Economics, Stremyanny, 36, Moscow 117997, Russia
³Department of Mechanical and Instrumental Engineering, Peoples’ Friendship Un of Russia (RUDN University), Miklukho-Maklaya, 6, Moscow, 117198, Russia

E-mail: MerzlikinV@mail.ru, bistrov-sun@mail.ru and smirnoff61@mail.ru

Abstract. The experimental set-up is developed for detection of reflection and transparence coefficients of semitransparent materials within visible and IR radiation diapasons. The peculiarity of the set-up is using the spectral photometric integrating spheres for effect compensation of reflection indicatrices of tested materials with fixed wavelengths 0.63 µm 1.15 µm (near IR), 2.79 µm and 3.39 µm (middle infrared) of probing laser radiation. In the first case the integrating sphere is a photometric ball which made from porous fluoroplastic with high reflectivity up to 97-99%. For the second range an aluminum sphere (sand blasting) was used. The application of the photometric sphere reduced the measurement error, connected with different sensitivity of photosensors to beam slope angle of detected radiation reflected (transmitted) for flat material samples with different indicatrices of reflection (transmission). The proposed method of experimental measurements of spectral photometric characteristics of reflection and transparent improve the accuracy of theoretical estimations of the optical (thermoradiation) parameters (absorption and scattering indexes), as well as the functions of a thermal radiation heat source for the calculation of temperature fields in semitransparent materials. For the first time, a computational and experimental estimation of scattering and absorption indexes (with an accuracy not less than 1%) were carried out for semitransparent materials with high scattering (porous ceramics based on stabilized zirconium dioxide) in the middle infrared range of wavelengths which is most problematic for optical measurements.

1. Introduction

In traditional studies of complex heat transfer and radiant heat transfer for the heat protection elements of the combustion chambers walls of diesel engines and turbines, blades of aerospace turbine, traps of overheated coolants of nuclear reactor, usually it is necessary to consider the effect of the integral heat flux using the contribution of the penetrating radiant component in the short wavelength range ~ 1-3 µm in the heating process of heat-insulating (heat-shielding) coatings.
Applicate ceramic insulating coatings (based on oxides of silicon, aluminum, zirconium) are partially transparent to the specified radiant exposure. But, as a rule, this effect is not taken into account [1, 2], although the use of these coatings becomes relevant, based the trends of the conceptual development of similar heat protection in mentioned above fields of science and technology [3, 4].

Due to the lack of optical heat exchange models the study of radiation-convective action was limited by analysis of effective heat transfer using only the exposed surface without analysis of subsurface overheating caused by penetrating radiation [1, 2].

Thus, the correct evaluation of the radiation and hence temperature fields was absent. Instead, there is the use of numerous empirical formulas or incorrect physical models (for example, for non-stationary heat transfer coefficient according to the Woschni formulas in combustion engines of ICE [1, 2, 4]), obtaining of which requires time-consuming laboratory measurements and costly full-scale tests of model and industry heat-resistant samples.

Therefore, a preliminary experimental evaluation of high-precision optical characteristics of materials and coatings will reduce the number of bench tests and predict the most appropriate selection of materials for elements of heat protection [3, 4].

2. Optical set up for experimental measurements of reflection and transparency

To evaluate the optical characteristics of a semitransparent material and coating optical set up for experimental measurements of reflection and transparency coefficients (using a photometric ball, additionally introduced into the optical layout) has been developed [5]. These data allow us to estimate the absorption and scattering indices that determine the function of the heat source in solving different problems of radiative heat transfer and produced temperature fields [3, 4].

Measurements of the reflection coefficients of experimental model samples of semitransparent heat-insulating materials were carried out on the setup shown in Figure 1.

![Figure 1. The optical layout of the test set up for measuring the reflection coefficients (a) and transparency (b) of samples of semitransparent materials respectively: in the 5a and 4b positions (probe laser - 1, integrating sphere - 2, photodetector - 3, reflective hemisphere with a hole for probe radiation - 4, the opaque reference sample – 5b, nanomicro voltmeter - 6)](image_url)

The following radiation sources were used: tricolor helium-neon laser “ЛГ-126” (here and below Russian abbreviation is used) with working laser wavelength of 0.63 μm, 1.15 μm, 3.39 μm); solid-state laser “ЛТ-2” with a wavelength of 1.06 μm; solid-state laser “УЛТ-3” with a wavelength of 2.79 μm.

The probing radiation reached the test sample, after passing through the central hole of the mirror hemisphere, then this beam was diffusely reflected from the sample and was directed (by a focusing mirror) into a photometric ball, equipped with a photodiode “ФД-24К“ (3) for measurements at wavelengths of 0.63, 1.06, and 1.15 μm (visible and near infrared range) or the photoresistor “ФР-188“ for measurements at wavelengths of 2.79 μm and 3.39 μm (middle range).
The focusing mirror was a glass spherical segment \( (4a) \) with a radius of 0.2 m and a sphere section size of 0.4 m. Aluminum was sprayed onto the working surface of the mirror, having a limiting reflection coefficient \( \sim 90\% \) in the visible, near and middle IR regions of the spectrum.

At the investigation in a darkened room (in the absence of extraneous light sources) the ratio of recorded measuring signals and noise one did not exceed 1%.

The use of a photometric ball (integrating sphere) made it possible to eliminate the measurement error, connected with different sensitivity of photo detectors for beam slope angle of the incident radiation (i.e. various indicatrices) [4, 6], that allows to increase accuracy of measurements of optical and thermo-radiation characteristics and improve the evaluation of the function of thermal radiation heat source to calculate temperature fields in the exposed materials and coatings.

In addition, a simplified layout (shown in Figure 1b), allowing to measure transparence was used. In this case, when measuring the reflection coefficients in the 5a position, the reference standard sample and the tested one were alternately placed.

When measuring the transmittance of the researched sample, it was located in position 4b, but the reference standard was located in the 5b position.

Radiation attenuation was carried out using light filters or rotating disk attenuators. The measurement accuracy at using a photodiode “ФД-24К” and photoresistance “ФР-188” accordingly was not worse than 2% and 0.5%.

As reference samples (standards) quartz ceramics (made from high purity synthetic powder with Russian brand “ОСУ-14”) and aluminum with brand “АМт” (sandblasted).

### 3. Numerical evaluation of the absorption and scattering parameters

Absorption and scattering rates were calculated according to the methodology (developed by the authors), in the two-flux approximation of the solution of the radiative transfer equation based on the measurement of the reflection coefficients \( r(H) \) and transmittance ones \( \tau(H) \) for samples of different thickness \( H \) in the form of compressed powder tablets of stabilized zirconia \( \text{ZrO}_2 + 8\% \text{Y}_2\text{O}_3 \) (Table 1) [4].

The values of unknown spectral absorption \( \kappa \) and scattering \( \sigma \) indices may be calculated from experimental spectrophotometry measurements of the reflectance and the transparence of flat sample with different thickness \( H \):

\[
\kappa = \frac{b_{rad} \cdot (1 - A)}{1 + A}, \quad \sigma = \frac{b_{rad}^2 - \kappa^2}{2\kappa}, \quad b_{rad} = \frac{1}{H} \ln \frac{2U}{V - \sqrt{V^2 - 4U^2}}, \quad r(H \rightarrow \infty) = \frac{Y - U}{UY - 1},
\]

where \( U = r(H) + \tau(H); \quad V = [r(H)/\tau(H)][1 - U^2] + U^2 + 1; \quad Y = \exp(-b_{rad}H); \quad b_{rad} \) - extinction index; \( A = r(H \rightarrow \infty) \) - albedo (reflection coefficient for semi-infinite substance layer).

#### Table 1. Spectral values of experimentally measured reflection coefficients \( r(H) \) and transparence \( \tau(H) \) and calculated parameters: \( r(H \rightarrow \infty) \) - albedo; absorption \( \kappa \) and scattering \( \sigma \) indices for samples of pressed powders (\( \text{ZrO}_2 + 8\% \text{Y}_2\text{O}_3 \)) with thickness \( H = 7 \text{ mm} \)

| \( \lambda \), \( \mu m \) | \( r(H) \), % | \( \tau(H) \), % | \( r(H \rightarrow \infty) \), % | \( \kappa \), l/m | \( \sigma \), l/m |
|-----------------|-------------|-------------|-----------------|-------|-------|
| 0.63            | 97.0        | 2.6         | 98.5            | 0.6   | 5080  |
| 1.15            | 92.0        | 4.3         | 93.2            | 6     | 2430  |
| 2.79            | 91.0        | 3.7         | 92.5            | 9     | 2470  |
| 3.39            | 89.0        | 3.3         | 89.5            | 14    | 2400  |

There is a significant value of the reflection coefficient of model ceramic samples up to a wavelength of 3.39 \( \mu m \).
Low absorption materials (for example, samples of corundum or quartz) have high reflectivity values too. But only zirconia has a transparency band up to ~3.39 μm unlike samples of other oxides whose transparency bands are limited by 2-3 μm.

4. Conclusion
Experimental set up for measuring reflection and transparence coefficients of materials and coatings in the visible and infrared ranges has been developed. Using a photometric ball in an optical set up for recording reflection and transparent coefficients allowed to eliminate the measurement error, connected with different sensitivity of photo detectors with respect to the incident (on its surface) radiation, whose diffuse-directed indicatrix is indefinite. This technique improves measurement accuracy optical (thermoradiation) characteristics and estimates of the function of thermal radiation heat source for solving different problems of radiation - conductive heat transfer.

First, the optical parameters are estimated for technological samples of semitransparent coatings based on zirconium dioxide in the middle infrared: absorption ~ 14 l/m and scattering ~ 2400 l/m indexes with a reflection coefficient of a semi-infinite layer (albedo) ~ 89.5%.

Performed experimental measurements in the visible and near IR diapasons corresponds to the data reported by other authors [3, 7].

Presented method of high-precision optical recording is important for selection the type of semitransparent Thermal Barrier Coatings [1-3, 7] with correctly certified parameters to study specific radiant heat transfer in heat-stressed elements in the IR range for heat and power plants, engines (combustion chambers, furnaces) and vehicles.

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