Infrared Physics & Technology 77 (2016) 162–170

Contents lists available at ScienceDirect

Infrared Physics & Technology

journal homepage: www.elsevier.com/locate/infrared

Regular article

Near-infrared optical properties of a porous alumina ceramics produced by hydrothermal oxidation of aluminum

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HIGHLIGHTS

- The transport scattering albedo is retrieved from the normal-reflectance measurements.
- The known data for absorption coefficient of alumina in a wide temperature range are used.
- The absorption coefficient is obtained using the additive hypothesis for porous ceramics.
- The Mie theory for grains in porous ceramics is used to estimate near-infrared scattering.

ARTICLE INFO

Article history:
Received 29 April 2016
Revised 28 May 2016
Accepted 30 May 2016
Available online 31 May 2016

Keywords:
Alumina ceramics
Reflectance
Transport approximation
Mie scattering
Radiative conductivity

ABSTRACT

The measured spectral normal–hemispherical reflectance of an optically thick sample of porous alumina ceramics produced by hydrothermal oxidation of aluminum and subsequent high-temperature treatment of boehmite (or böhmite), AlO(OH), is used in a combination with the published data for absorption coefficient of alumina to retrieve the near-infrared optical properties of the alumina ceramics at both room and elevated temperatures. The spectral emissivity of porous alumina ceramics is also determined. An approximate model based on the Mie theory for single grains is suggested to estimate the transport scattering coefficient and the radiative conductivity of the material at high temperatures.

1. Introduction

Porous alumina ceramics under investigation was produced using a specific procedure based on the hydrothermal oxidation of aluminum powder [1–5]. It was shown that aluminum powder with average particle size of about 10 μm is fully oxidized during few seconds in water steam at temperature about 300 °C. The product of this oxidation is boehmite, AlO(OH), in the form of single crystals with sizes from 10 to 200 nm, and the primary crystals are then agglomerated into the particles with the size about 10 μm. The complete process including the described first stage was realized in experimental plant with the use of a continuous flow reactor [6]. The second stage is a high-temperature treatment of boehmite at first in muffle furnace at 600 °C for crystallized water removal and at then in vacuum furnace at 1600 °C for α-Al_2O_3 obtaining. This phase of pure alumina is widely used due to its advantages as compared to other phases [7,8]. The near-infrared properties of porous alumina are important for combined heat transfer analysis at the high-temperature stage of the process.

The particular objective of the present paper is twofold: (1) to obtain near-infrared radiative properties of a porous alumina ceramics in a wide range of temperatures using the suggested combined experimental and theoretical approach and (2) to develop an approximate theoretical model for both the scattering properties and radiative conductivity of the alumina ceramics.

2. Microscale morphology and average porosity of alumina ceramics

Surface morphology of a solid sample of alumina ceramics was studied with the use of JEOL JSM-7401F scanning electron microscope (SEM). A carbon substrate was used to support the samples in the microscope. The shooting was carried out at 1 kV accelerating

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http://dx.doi.org/10.1016/j.infrared.2016.05.028
1350-4495/

Nomenclature

a	grain or pore radius
d	distance
f	fraction or concentration
G	irradiation
I	radiation intensity
J	diffuse component of radiation intensity
m	complex index of refraction
M	mass
n	index of refraction
p	porosity
q	radiative flux
Q	efficiency factor
$\vec{r}$	radius-vector
R	reflectance
$\bar{S}$	relative area
T	temperature
V	volume
x	diffraction parameter

Greek symbols

α, β	absorption and extinction coefficients
γ	coefficient introduced by Eq. (9b)
δ	relative radius of cavity in particles
ε	emissivity
θ	polar angle
κ	index of absorption
λ	wavelength of radiation
μ	cosine of polar angle
$\bar{\mu}$	asymmetry factor of scattering
ξ	coefficient in Eq. (18)
σ	scattering coefficient
σ_0	Stefan–Boltzmann constant
ρ	density of alumina
τ	optical thickness
φ, ψ	functions introduced by Eq. (8)
χ	eigenvalue introduced by Eq. (9b)
Φ	scattering phase function
ω	albedo
$\vec{\Omega}$	the unit vector of direction

Subscripts

a	absorption
av	average
b	blackbody
c	critical
def	defect
h	hemispherical
n	normal
open	open pores
r	radiative
R	Rosseland
s	scattering, surface
sample	sample
tr	transport
v	volume

voltage. Several typical images of high resolution are presented in Fig. 1. The sample morphology is really very complex, and one can observe the grains of quite different shape and size. There are several big and relatively dense agglomerates with size about 30–50 μm (Fig. 1a). At the same time, one can see in Fig. 1b a lot of separate small particles with typical size of 3–5 μm with numerous orifices of diameter at the level of 1 μm and also many spherical particles with diameter about 1 μm. Obviously, a contribution of various particles to the near-infrared scattering is quite different, and the main attention should be focused on homogeneous or hollow small particles.

The laboratory measurements were also made to determine both the total volumetric porosity, p, and the volume fraction of open pores, p_{open}, of the sample of alumina ceramics. The total porosity is defined as a ratio of the sample mass to the mass of imaginary bulk material:

$$p = M_{\text{sample}}/(\rho V_{\text{sample}}) \tag{1}$$

The accurate measurements showed the value the value of $p = 0.638$. The value of p_{open} was also obtained experimentally using various liquids to fill the open pores. The repeating experiments showed that $p_{\text{open}} = 0.386 = 0.605p$. This result is qualitatively clear from the SEM image presented in Fig. 1b. A lot of closed cavities in alumina grains were produced in the specific process of the hydrothermal oxidation of aluminum.

3. Measurements of the normal–hemispherical reflectance

The spectral measurements of normal–hemispherical reflectance and transmittance of flat samples is a widely-used traditional procedure to get the data for subsequent identification of the main absorption and scattering characteristics of dispersed materials [9–11]. In some cases, as discussed in [12–14], porous materials are highly scattering in the near-infrared. As a result, normal–hemispherical transmittance is low, on the order of 1% for samples of geometric thickness of about 1 mm, due to very large optical thickness. Hence, use of commercial spectrometers to measure normal–hemispherical transmittance is challenging, due to their lack of adequate signal-to-noise characteristics to measure such low values of transmittance. The measurements were performed in the wavelength range of $0.2 < \lambda < 2.5$ μm. The relative uncertainty of the measurements was no more than 0.5% at $\lambda < 2.2$ μm and reached 2% at $\lambda > 2.2$ μm.

Fortunately, there are reliable data in the literature for the spectral absorption coefficient of bulk alumina. Therefore, the measurements of normal–hemispherical reflectance, $R_{\text{n–h}}$, for optically thick samples are sufficient. The value of $R_{\text{n–h}}$ was measured using spectrophotometer Cary 500 produced by the firm Varian and equipped by integrating sphere DRA-CA-5500 with internal diameter of 150 mm. This integrating sphere was produced by the firm Labsphere. The beam diameter at the sample surface was equal to 10 mm. The schematic of the optical installation is traditional, and it is not presented in the paper. The results of measurements presented in Fig. 2 indicate that the value of $R_{\text{n–h}}$ is relatively low in the visible range and increases up to about 0.88–0.9 in the near-infrared. We also found good agreement between the spectral dependences of $R_{\text{n–h}}$ shown in Fig. 2 and similar values for the other local area of the sample surface. This result indicated that optical properties of the sample are homogeneous, and good repeatability of the $R_{\text{n–h}}$ data is confirmed.

4. Spectral optical properties of alumina in the near-infrared

There are two optical constants of every substance, and these quantities are usually treated as the complex index of refraction, $m = n - i\kappa$, where n is the index of refraction and κ is the index