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A new experimental approach to measure the refractive index of infrared optical ceramic through the transmittance

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ABSTRACT

This paper proposes a new experimental approach to measure the refractive index of infrared optical ceramic through the transmittance in some particular cases such as can not manufacture test lenses or lack of infrared goniometers. The relationships between the refractive index and transmittance as well as the thickness of infrared optical ceramic are established. The refractive index of one type of optical ceramic material can be captured through the transmittance and thickness of two measurement specimens fabricated from that ceramic. The precision of proposed method is evaluated by manufacturing three measurement specimens from the same optical ceramic material and determining their transmittances and thicknesses as the basis of this exploration. The accuracy of present approach is verified by comparing refractive indexes obtained from proposed method with those of confirmed autocollimation method and the dispersion formula. The comparative results show that the present method has good precision and accuracy. In which, the relative errors between the proposed approach and the autocollimation method are less than 1%.

1. Introduction

Generally, for optical materials, refractive index and transmittance are the two most important optical parameters [1,2]. The transmittance of optical ceramic material can be determined easily by operating on a spectrometer with a Michelson interferometer. While the determination of its refractive index is a much harder process. So far, there have been many studies on methods and techniques of determining the refractive index of optical materials [3–6]. Wei et al. [5] proposed a novel method to measure the refractive index of glass, which can be applied to measure the refractive index of the special laboratory prepared glass which is small, irregularly shaped by measuring its absorption spectrum. Rheims and his co-workers [6] used simple extensions to a standard Abbe refractometer to determine the refractive index n in the near-infrared. A technique was derived to correct for the dispersion of the glass prism and experimental results of refractive-index measurements at $\lambda = 830$ nm. Daniel et al. [7] predicted the refractive index of fully dense isotropic polycrystalline alumina (PCA) with randomly oriented grains by using the ordinary and extraordinary refractive indices (n_o and n_e) of sapphire spatially averaged over the surface of a hemisphere. Aleksey and colleagues [8] employed the measured spectral normal–hemispherical reflectance of an optically thick specimen of porous alumina ceramics produced by hydrothermal oxidation of aluminum and subsequent high-temperature treatment of boehmite (or böhmite), AlO(OH) in a combination with the published data for the absorption parameters of alumina to retrieve the near-infrared optical characteristics of the alumina ceramics at both room and elevated temperatures. Limeng et al. [9] carried out an investigation on translucent α-SiAlON ceramics, which were synthesized by hot pressing and spark plasma sintering. The in-line transmittance of the visible-infrared light through the α-SiAlON ceramics was discussed in correlation with the SiAlON compositions, rare earth dopant types, microstructures, and the refractive index anisotropy of the α-SiAlON grains. The most widely used methods are the approach of measuring the smallest deflection angle on goniometers, measurement in the pulfrich refractometer, V refractometer, and Abbe refractometer. However, the mentioned methods were applied mainly to measure the refractive index in the visible light spectrum. For the optical ceramics working on infrared environments, their refractive indexes are determined by the autocollimation method on infraredgoniometers [4]. The essence of this method is to determine the refractive angle of a prism-shaped specimen whose main cross-section is the right triangle and the orthogonal incident angle of the infrared light passing through the prism, parallel to the base and perpendicular to the reflective surface of the prism. To

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perform this method in addition to equipment requirements (collimating tubes and infrared goniometer), it is also required to manufacture a prismatic test specimen with the main cross-section of a triangle with surface size requirements must be higher than 30x50 mm. However, in many cases, the fabricated specimens, especially in research labs with limited spaces, it is difficult to manufacture test prisms. That makes determining the refractive index of these specimens very hard. Tropf et al. [10] proposed a dispersion formula to determine the refractive index of wavelength-dependent optical materials. Nevertheless, these formulas are an approximation for perfectly made materials, which need many assumptions in addition. For optical ceramic materials, the refractive index is greatly influenced by manufacturing technologies (manufacturing processes). Therefore, the dispersion formula [10] should be used as a reference only. That is the main reason to find a new experimental approach to determine the refractive index of infrared optical ceramic through the transmittance in some mentioned tough situations above.

In this paper, method of determining the refractive index of infrared optical ceramic through the transmittance is proposed. The advantage of this method is that it requires the manufacture of uncomplicated test specimens. Another new point is that the device can take advantage of infrared spectrometers without the need for infrared goniometers.

The organization of this paper is as follows. Some traditional methods of measuring the refractive index of infrared optical materials and the reason to carry out this new experimental approach are introduced shortly in section 1. Methods of experimental measurement and dispersion formula are presented in section 2. Section 3 is about results and discussions. Some remarkable conclusions are summed up in section 4.

2. Method of measurement

Optical ceramics are generally made up of particles, particle boundaries, stomata, and impurities. The light passing through the optical ceramic specimen can be attenuated by the following factors (Fig. 1): reflecting at the two main surfaces, absorbed by particles and impurities, and scattering and refracting by pores, impurities, particle boundaries as well as other irregular structures. Among the factors that cause light loss, reflection depends primarily on the surface and the refractive index of the material, other factors depend on the crystal structure characteristics of the material.

For an optical specimen with the thickness d, the transmittance of the light can be determined by the Lambert-Beerfunction [11,12] as follow:

$$T = (1 - R_S)^2 e^{-\alpha d} \tag{1}$$

where T is the transmittance (%), R_S is the total light reflected at two main surfaces of ceramic material (air-material boundary surface and material-air boundary surface), α is the absorption coefficient which depends on material and scattering per unit thickness, and d is the thickness of the optical material (cm).

The total reflected light at the two main surfaces is determined by the following formula:

$$R_S = \frac{2R}{1 + R} \tag{2}$$

in which R is the reflected light at one surface, which is defined as:

$$R = \frac{(n - 1)^2}{(n + 1)^2} \tag{3}$$

herein n is the refractive index of the material.

Equations (1)–(3) present the relationship between the transmittance T and the refractive index n. Among other components of these equations, the thickness d can be determined simply. Therefore, we only need to determine the absorption coefficient.α.

To determine the absorption coefficient α, two measurement specimens with different thicknesses d_1 and d_2 are manufactured from one type of optical ceramic material. Herein, it is considered that the original ceramic material is homogeneous. In other words, the absorption coefficient α is constant. If these two measurement specimens were fabricated with equal quality surfaces, the total reflected light R_S could be considered the same. From equation (1) for two measurement specimens, we have a system of equations as follows:

$$T_1 = (1 - R_S)^2 e^{-\alpha d_1} \tag{4}$$

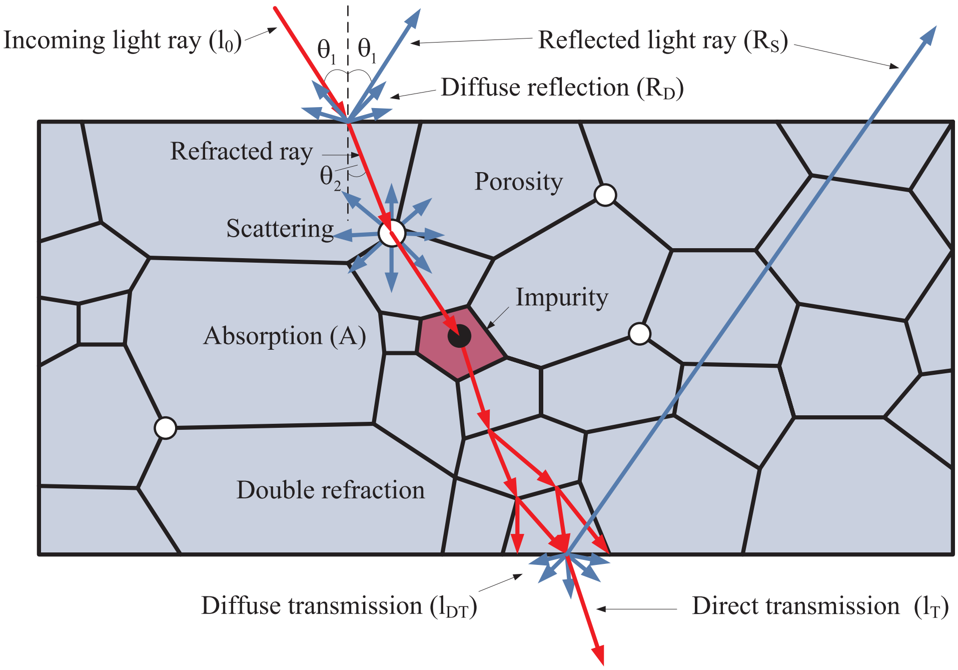

Fig. 1. The path of light rays in an optical ceramic material.

Table 1
The values of experimental plan.

Order	1	2	3
Thickness (μm)	1.082	2.950	7.981

$$T_2 = (1 - R_S)^2 e^{-\alpha d_2} \tag{5}$$

From equations (4) and (5) we obtain:

$$\frac{T_1}{T_2} = e^{-\alpha(d_1 - d_2)} \tag{6}$$

Then, the absorption coefficient α can be determined by the following formula:

$$\alpha = \frac{ln\frac{T_1}{T_2}}{d_2 - d_1} \tag{7}$$

Substituting R_S and α from equations (2), (3) and (7) into equation (4) we have:

$$T_1 = \left(1 - \frac{2\frac{(n-1)^2}{(n+1)^2}}{1 + \frac{(n-1)^2}{(n+1)^2}}\right)^2 \frac{ln\frac{T_1}{T_2}}{d_2 - d_1} \tag{8}$$

in which, if we have the values of T_1, T_2, d_1, and d_2 equation (8) will be a quadratic equation of n variable. Solving this equation we will capture four roots, where one root greater than 1 is the refractive index value n, which needs to be found.

The magnesium fluoride MgF_2 optical specimens are manufactured under the hot-pressing technique based on three methods to evaluate the validity of the present approach.

- Firstly, using the method of autocollimationon infrared goniometers to measure the standard refractive index.
- Next, the dispersion formula for crystal is used to calculate the refractive index according to the infrared wavelength of magnesium fluoride MgF_2 crystal, which is expressed as follows [10]:

$$n^2 - 1 = \frac{0.48755108\lambda^2}{\lambda^2 - (0.04338408)^2} + \frac{0.39875031\lambda^2}{\lambda^2 - (0.09461442)^2} + \frac{2.3120353\lambda^2}{\lambda^2 - (23.793604)^2} \tag{9}$$

in which λ is the wavelength (μm).

- Thirdly, using the proposed approach to determine the refractive index of specimens. As mentioned above, two specimens with different thicknesses are manufactured from the same optical ceramic. Nevertheless, to evaluate the precision (also called the concentration) of the proposed method, three small cylindrical specimens with 15 mm in diameter and different thicknesses are manufactured. Two out of three specimens are taken in turn to measure the transmittance and thickness in order to determine the three refractive index values according to equation (8). Then, the deviations of these three refractive index values are analyzed to evaluate the precision of the present method. Finally, refractive index values captured from the three methods are compared to one another to evaluate the validity and accuracy of present approach.

In this work, some important notes must be considered. Cylindrical specimens need to be ground and polished the working surfaces (two main surfaces) to meet the following requirements: the parallelism of two working surfaces does not exceed 30’, the surface roughness R_a does not exceed 0.050 μm, the surface cleanliness must be from grade IV or higher according to GOST 11141. The thicknesses of specimens are determined by a micrometer with the accuracy can be up to 1/1000. The transmittance in the infrared spectral range of the MgF_2 optical ceramic (2.5 ÷ 6 μm) is measured by a single-beam infrared spectrometer with a Michelson interferometer. In other words, an infrared spectrophotometer Nicolet Summit FTIR Spectrometers (measuring range of 1.28 ÷ 28 μm) is used to measure the transmittance. Transmittance could be measured parallelly to the hot-pressing direction.

3. Results and discussions

The thicknesses of three specimens that are manufactured from the same magnesium fluoride MgF_2 optical ceramic are presented in Table 1.

The transmittances of three specimens are shown in Fig. 2. It can be seen that all three specimens have the same shape. The smaller the thickness is, the higher value the transmittance obtains. This phenomenon demonstrates that three specimens are very homogeneous. The reason is that all three specimens are manufactured from the same hot-pressed MgF_2 optical ceramic.

The following Table 2 presents the transmittances of three specimens at different wavelengths.

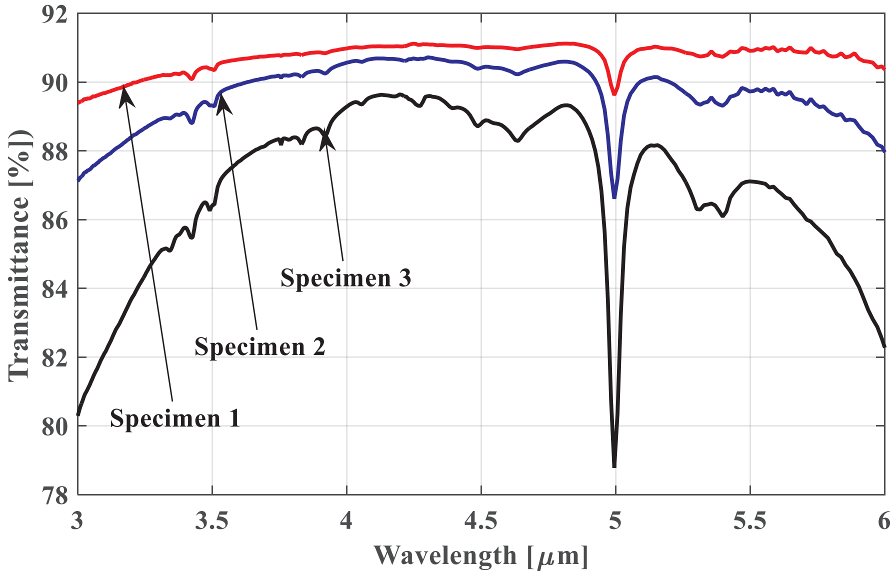

Fig. 2. The transmittances of three specimens.

Table 2
The transmittances of three specimens change as a function of wavelength.

Wavelength λ (μm)		3	4	5
Transmittance (%)	Specimen 1	89.4	91.0	89.9
	Specimen 2	87.1	90.6	87.2
	Specimen 3	80.3	89.3	80.4

Table 3
The refractive indexes obtained from the present approach.

Wavelength λ (μm)		3	4	5
Refractive index	n_{12}	1.3695	1.3573	1.3518
	n_{13}	1.3651	1.3566	1.3517
	n_{23}	1.3529	1.3546	1.3514

Note that n_{ij} is determined through refractive indexes of specimens i and j (i, j = 1–3).

By combining the values of the thickness (Table 1) and the transmittance (Table 2) of three specimens and solving equation (8), we will obtain refractive indexes n_{12}, n_{13}, and n_{23} as shown in Table 3.

The refractive indexes determined by the present approach according to the wavelength are plotted in Fig. 3. It can be seen that the refractive indexes of all three specimens are similar to one another.

Now, in order to evaluate the precision of the present approach, the average refractive index value $\bar{n}$,the standard deviation S, and relative standard deviation RSD (also called repeatability) are calculated as follows:

$$\bar{n} = \frac{n_{12} + n_{13} + n_{23}}{3} \tag{11}$$

$$S = \sqrt{\frac{(n_{12} - \bar{n})^2 + (n_{13} - \bar{n})^2 + (n_{23} - \bar{n})^2}{2}} \tag{12}$$

$$RSD = 100\frac{S}{\bar{n}}\ (\%) \tag{13}$$

From the refractive indexes listed in Table 3, by considering equations (11)–(13), the average refractive index value $\bar{n}$, the standard deviation S, and relative standard deviation RSD are obtained as shown in Table 4.

The refractive indexes obtained from the autocollimation method on infrared goniometers and dispersion formula (9) are presented in Table 5.

Table 4
The precision of the proposed approach.

Wavelength λ (μm)	3	4	5
Average refractive index $\bar{n}$	1.3625	1.3562	1.3516
Standard deviation S	0.0086	0.0014	0.0002
Relative standard deviation RSD(%)	0.63	0.10	0.02

It can be observed clearly that the precision of the proposed approach is very good when the relative standard deviation is less than 1%.

Table 5
The comparative refractive indexes.

Wavelength λ (μm)	3	4	5
Autocollimation method	1.3633	1.3531	1.3468
Dispersion formula(9)	1.3599	1.3488	1.3340

The refractive index obtained from the autocollimation method on infrared goniometers is the most accurate and common confirmed value. It is used as a good reference to evaluate the results determined from two other methods. The absolute and relative errors of refractive indexes determined by the proposed method and dispersion formula (9) are shown in Table 6.

Now, from Table 6 it can be seen that the results obtained from the proposed approach are more accurate than those from the dispersion formula. For the proposed approach, the relative errors are less than 1%. Theoretically, the more accurate the refractive index is, the more specimens are made from the same optical ceramic material. However, when manufacturing a large number of specimens will face many difficulties due to their size and other technical conditions. The errors of the refractive indexes determined by the proposed method shows that there is not much difference in the accuracy of using only two specimens instead of using three specimens (n_{12}, n_{13}, and n_{23} are respectively compared to $\bar{n}$). Therefore, it is only necessary to make two specimens from the same optical ceramic material to determine refractive indexes using the proposed method with the acceptable accuracy.

4. Conclusions

This paper carried out a new experimental approach to determine the refractive index of optical ceramic through the transmittance and thickness of two specimens made from that ceramic. The proposed approach has good precision and accuracy, the relative errors are less

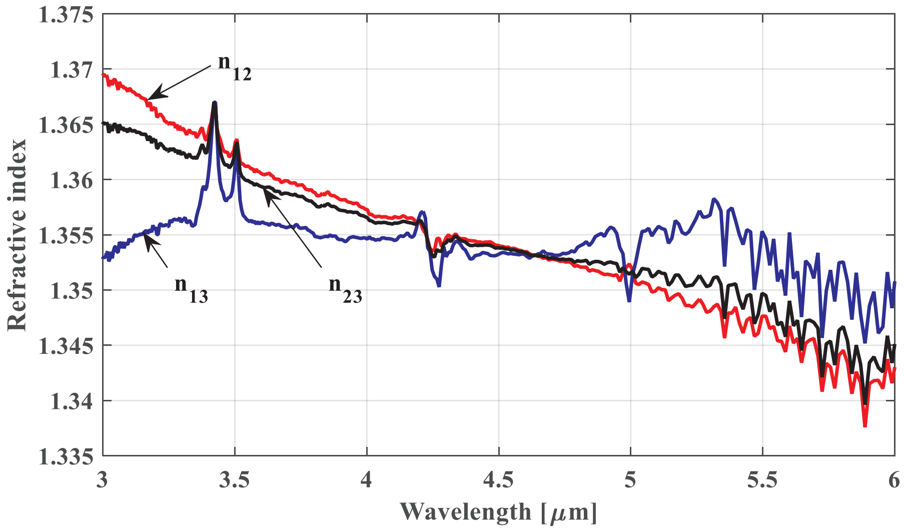

Fig. 3. The refractive indexes obtained from the present approach.

Table 6
The absolute and relative errors of refractive indexes.

Wavelength λ (μm)		3	4	5
The absolute error	Dispersion formula (9)	0.0034	0.0043	0.0128
	n_{12}	0.0062	0.0042	0.0050
	n_{13}	0,0018	0.0035	0.0049
	n_{23}	0.0104	0.0015	0.0046
	$\bar{n}$	0.0008	0.0031	0.0048
The relative error (%)	Dispersion formula (9)	0.2494	0.3178	0.9504
	n_{12}	0.4548	0.3104	0.3713
	n_{13}	0.1320	0.2587	0.3638
	n_{23}	0.7629	0.1109	0.3416
	$\bar{n}$	0.0587	0.2266	0.3589

than 1%. In this work, only two specimens are manufactured from the same optical ceramic material in order to determine exactly the refractive index with acceptable accuracy.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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