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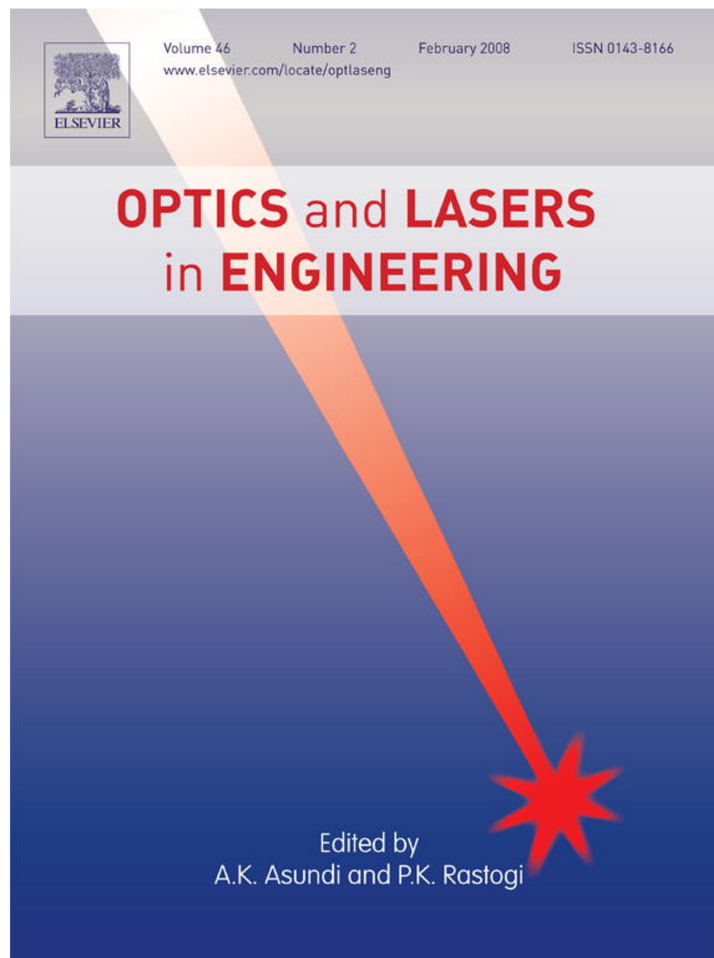

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Automated interferometric technique for express analysis of the refractive indices in isotropic and anisotropic optical materials

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Abstract

This paper deals with the technique for the refractive index measurements based on the interferometry of a rotated parallel plate (IRPP). The device consists of the Michelson interferometer, the sample rotation system and the optoelectronic registration system. A refractive index of parallel plates is determined by their rotation through measuring simultaneously a shift of interference fringes. Although the IRPP technique is known from long ago [Shumate MS. Appl Opt 1966;5:327] several considerable improvements have been done in order to improve the accuracy of the method. The measuring process is completely automated. The method has been tested on the model crystals of the lithium niobate giving the magnitudes for ordinary and extraordinary refractive indices as $n_0 = 2.2865 \pm 0.0007$ and $n_e = 2.2034 \pm 0.0007$. A considerable increase of accuracy is reached in our case by an automation of the measuring procedure, development of a new software as well as implementing the interferometric method for a precise determination of a sample zero position. The automated refractometer is offered for use in research laboratories and industry.
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Keywords: Optical refraction; Interferometry; Refractive indices; Isotropic and anisotropic optical materials

1. Introduction

Refractive indices are one of the most important characteristics of optical materials and represent a considerable interest for both fundamental and applied research in many fields of science and technology. The techniques for refractive indices measurements are usually based on ellipsometry [1,2], immersion [3], minimum deviation [4] or interferometric-turning [5–7] methods. Each of those methods has its advantages and a corresponding field of applications. The ellipsometry can be used only for determination of refractive indices of thin film materials deposited on substrates. The immersion method consists in comparison of the refractive indices of crystal materials and immersion liquids. A set of immersion liquids [3] is limited by the range of refractive indices ($1.4 \leqslant n \leqslant 1.8$); thus, the immersion method cannot be applied at all for many materials. The method of minimum deviation requires a sample in the form of wedge or prism, therefore it is frequently also called as the method of prism. Such method indeed provides a rather high precision of the refractive index measurements. However, the procedure is in fact a time consuming and requires a substantial amount of a crystal or amorphous material from which the prism must be prepared. Moreover, this method is evidently not very applicable to expensive materials since a considerable part of these is lost during the preparation procedure whereas the remaining prisms are frequently not suitable for further use. Also the method of prism is not quite suitable for materials obtained by different technologies [8] or materials with spatially inhomogeneous refractive indices, i.e. the magnitude of which may vary, e.g. along the grown ingot [9].

In favor of many practical needs, a precise non-destructive interferometric technique for the optical refraction measurements represents a considerable interest. It is expected that such technique would be applicable to a

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rectangular type of thick samples, i.e. the ones most frequently used in many applications. For this purpose the interferometry of a rotated parallel plate (called hereafter as IRPP method) looks a promising one. The IRPP method has been originally developed by Michael Shumate [5] and consequently used in the experiments with several solid materials [10–12] as well as with liquids [13]. A low precision and complicated measuring procedure remained through a long time as hard problems of the IRPP technique. With some modification [6,14] the method was considerably simplified leading also to an increase of the precision. In this paper, we develop the technical aspects for realization of the ideas proposed in [14] i.e. our attention is concentrated on a designed automated interferometric device for the optical refraction measurements.

2. Method description

We start with a brief description of the automated device for the refractive index express measurements. A typical setup is shown in Fig. 1. The IRPP method is based on the Michelson interferometer technique and is suitable for parallel plate samples. The sample is set into the one arm of the interferometer noted in Fig. 1 as the Reference arm. The incident light from the He–Ne laser splits into two beams by means of a beam splitter. The first beam (the reference arm) is reflected back by the mirror 1; the second one (the measuring arm) passes the polarizer and the sample, reflects back on the mirror 2 and passes again the sample. Two beams are overlapped in the beam splitter creating thus the interference fringes, which are focused by the lens on the photodetector. A slit diaphragm before the photodetector enables to register the shift of the interference fringes caused by a sample rotation. It is realized by means of the rotating mechanism and controlled by PC through the control module device.

In order to calculate the refractive index of the sample, it is necessary to measure the change of the optical path in the sample during its rotation starting from a position of incident light normal to the sample (called hereafter as a sample zero position) to a certain angle φ. For this reason, the sample is initially placed normally to the incident light. In this case, the optical path $\Delta = nd$ (see Fig. 2), where d is the thickness of the sample and n is the refractive index. After the sample is turned out by the angle ϕ, the changes of the optical path $\delta\Delta$ reads as [6,14]

$$\delta\Delta = 2(d\cos\varphi' + d - d\cos\varphi'\cos(\varphi - \varphi') - dn). \quad (1)$$

The factor 2 in Eq. (1) appears as the beam passes the sample twice in the measuring arm of the interferometer. By taking into account the Snellius low ($\sin\varphi/\sin\varphi' = n$), the measured difference of the optical path expressed through a number of the interference fringes k ($\delta\Delta = k\lambda$, λ is the wavelength of laser generation) can be written as

$$k = \frac{2d}{\lambda}(1 - \cos\varphi - n + \sqrt{n^2 - \sin^2\varphi}), \quad (2)$$

from which the refractive index n can be easily extracted:

$$n = \frac{\sin^2\varphi + (1 - \cos\varphi - k\lambda/2d)^2}{2(1 - \cos\varphi - k\lambda/2d)}. \quad (3)$$

Amazingly, that IRPP method can be used for the measurement of the refractive indices of both uniaxial and biaxial crystals. For this purpose the following geometries of experiment can be used.

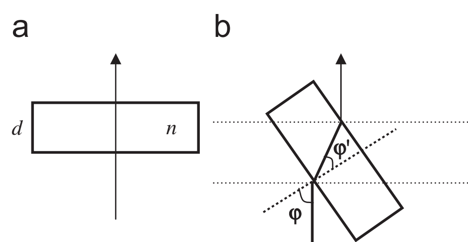

Fig. 2. Sample zero position (a) and the change of the optical path of the sample turned out by the angle φ (b).

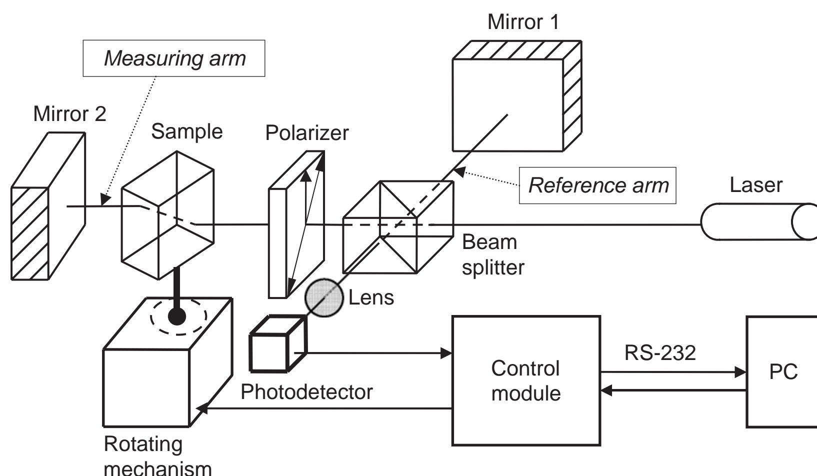

Fig. 1. The setup of the automated refractometer based on the interferometry of a rotating parallel plate (IRPP method).

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Uniaxial crystal: The optical axis of the sample must be set parallel (‖) or perpendicular (⊥) with respect to the axis of rotation **R** depending on whether an extraordinary index n_e or an ordinary index n_o is measured, respectively.

Biaxial crystal: There are two cases possible as follows:

(1) *By using two samples representing different principal crystallophysics cuts*. We introduce the notation $\mathbf{X}_1$, $\mathbf{X}_2$ and $\mathbf{X}_3$ for three orthogonal principal axes of the optical indicatrix also known as the principal crystallophysic directions. The principal crystallophysic cut would then mean a section of the optical indicatrix by (X_1-X_2)-, (X_1-X_3)- or (X_2-X_3)-plane. With two samples [e.g. of principal (X_1-X_2)- and $(X_1$-$X_3)$-cuts] three measurements should be done, i.e. by rotating the sample around one of the principal crystallographic directions ($\mathbf{R}\|\mathbf{X}_1$, $\mathbf{X}_2$ or $\mathbf{X}_3$) if one measures the refractive index n_1, n_2 or n_3, respectively. In each case the light polarization **P** is *parallel* to the rotation axis ($\mathbf{P}\|\mathbf{R}$).

(2) *By using one sample only which represents any of the principal crystallophysics cuts*. Assuming we deal with e.g. (X_1-X_2)-cut the principal refractive indices n_1 or n_2 can be measured as described above, i.e. by setting both rotation axis **R** and the light polarization **P** parallel either to $\mathbf{X}_1-$ or $\mathbf{X}_2$-direction, respectively. The remaining principal index n_3 can be measured by choosing the rotation axis $\mathbf{R}\|\mathbf{X}_1-$ or $\mathbf{X}_2$-direction, whereas the light polarization **P** is set *perpendicular* to it ($\mathbf{P}\perp\mathbf{R}$). Assuming e.g. $\mathbf{R}\|\mathbf{X}_1$ and $\mathbf{P}\perp\mathbf{R}$ the measured refractive index in Eq. (3) must be substituted by its effective magnitude n_{ef} defined as

$$n_{ef} = \left[n_1^{-2}\cos^2\varphi + n_3^{-2}\sin^2\varphi'\right]^{-1/2}, \tag{4}$$

therefore the refractive index n_3 then reads as [14]

$$n_3 = \frac{n_1 \sin\varphi}{\left[n_1^2 - (n_1 - 1 + \cos\varphi + k\lambda/2d)^2\right]^{1/2}}, \tag{5}$$

where the magnitude of n_1 may be measured within the geometry $\mathbf{R}\|\mathbf{P}\|\mathbf{X}_1$. By using light sources with different wavelengths one may also measure the dispersion of the refractive indices n_i (λ).

Let us estimate the precision of the IRPP method. Using Eq. (3) and the experimental errors for the turning angle $\delta\varphi$, the interference order δk, the sample thickness δd and the laser wavelength $\delta\lambda$, one obtains the error for the refractive index measurement δn as

$$\delta n = \left\{\left(\frac{\partial n}{\partial\varphi}\delta\varphi\right)^2 + \left(\frac{\partial n}{\partial k}\delta k\right)^2 + \left(\frac{\partial n}{\partial d}\delta d\right)^2 + \left(\frac{\partial n}{\partial\lambda}\delta\lambda\right)^2\right\}^{1/2}, \tag{6}$$

which after inserting of the corresponding components takes the form

$$\delta n = \left\{\left[\frac{(1-\chi-2n)\sin\varphi}{2(1-\cos\varphi-\chi)}\delta\varphi\right]^2 + \left[\frac{\sin^2\varphi}{2(1-\cos\varphi-\chi)^2} - \frac{1}{2}\right]^2 (\delta\chi)^2\right\}^{1/2}, \tag{7}$$

where

$$\chi = k\lambda/2d, \quad \delta\chi = \left\{\left(\frac{\lambda}{2d}\delta k\right)^2 + \left(\frac{k\lambda}{2d^2}\delta d\right)^2 + \left(\frac{k}{2d}\delta\lambda\right)^2\right\}^{1/2}. \tag{8}$$

Considering e.g. $\delta d = 0.01\,\mu\text{m}$ as typical for samples with $d\sim 10\,\text{mm}$, $\delta\lambda = 3\times10^{-8}\,\mu\text{m}$ as for modern He–Ne lasers ($\lambda = 0.6328\,\mu\text{m}$), $\delta k = 0.007$ and $\delta\varphi = 1\times10^{-6}$ one obtains $\delta n = 4.4\times10^{-6}$. We see that the estimated error of IRPP technique is indeed small compared to other known methods so its further development seems to be quite actual.

3. Experimental device description

The designed refractometer (see Fig. 1) is based on the Michelson interferometer (the laser, two mirrors, beam splitter, polarizer, lens), the sample rotation system (the rotation mechanism with high gearing coefficient, step-motor and the attachment for a sample holding) and opto-electronic registration system (the photodetector, control device and computer). Automation of measuring process consists in design of a precise sample rotation mechanism and the control module. In addition, new software for experimental data processing has been elaborated, which enables to carry out the experiment and perform the data processing.

3.1. The precise sample rotation mechanism and the control module

The rotation system consists of the crystal holder, rotation mechanism with the gearing coefficient of 2250 and the step-motor with a step of 1.8°. With some modifications of the microcontroller program the step may be reduced up to 0.9° thus ra sample can be rotated with the precision of about 1.4′≈0.0004°. The control module operates the rotation mechanism, measures a light intensity and transfers the data by means of D/A converter into the digital form suitable for further computer analysis, whereas PC stores the data into a file. The control module allows also to turn a sample clockwise or counter-clockwise if needed, to set a step of the rotation with angular resolution of 2.9″ or 1.4″, to perform the conversion of a light intensity with a resolution of 12 bit, to regulate a photodetector sensitivity and several other functions providing an optimal operation regime of the device.

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3.2. *Software for control of measurements and data processing*

The software consists of two programs providing the control over the measurement and data processing. The control program operates completely the measuring process and saves the data into a file. In addition, the data may be reviewed during the measurement—the typical results are presented in Fig. 3. The program of the data processing allows to import the data from a file and represent them in real time. The data represent sinusoidal oscillations with a variable frequency. Noises and vibrations can distort substantially the interference picture, which considerably influences a counted number of interference maximums. For this reason the algorithm of digital band-transmission filter with variable transmission band and variable average transmission frequency has been elaborated. The software filter also cut the constant component of received signal, which then substantially simplify the algorithm of interference maximums counting. The effect of the filtration is evident from the example presented in Fig. 4. The software was elaborated in Win32 environment and works within the operation systems Windows95/98/ME/NT/2000/XP.

4. Testing of the equipment

The equipment has been tested on the uniaxial crystal of $LiNbO_3$. Preparation of the sample for the optical refraction measurements includes a precise X-ray crystal orientation and an optical polishing of the crystal cuts. The plate thickness has been controlled within an accuracy of 1 μm only. The laser beam ($\lambda = 0.6328\,\mu m$) with polarization parallel to the sample rotation axis was used for the measurements of the extraordinary refractive index n_e, whereas with the orthogonal polarization for the ordinary refractive index n_0. The optical axis was oriented parallel to the sample rotation axis. The measurement by IRPP method gives $n_0 = 2.2865 \pm 0.0007$ and $n_e = 2.2034 \pm 0.0007$. The theoretically estimated error of the measurements ($\delta n = 4.4 \times 10^{-6}$) is substantially lower comparing the experimental one. This fact is caused by several reasons, in particular, the sample thickness, interference order and turning angle were determined with lower precisions comparing with that mentioned in Section 2. In our case $\delta d = 1\,\mu m$ and $\delta k = 0.01$. An error related with the turning angle $\delta\varphi$ could be defined as a step size, i.e. 0.0004°. However, the most serious problem arises with a fixing of a sample zero position, which indeed plays a key role in the IRPP method. It is rather a complicated problem to fix precisely an incident laser beam orientated normally to a sample surface. The maximum of this peak is broad and usually hard to verify with a high precision. Taking into account this problem, the authors [12] have determined a zero position via the numerical estimations. We propose here an alternative method, which is based

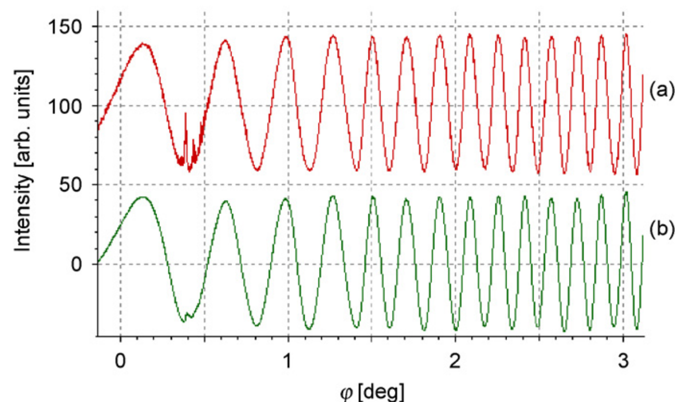

Fig. 4. The angular dependence of the light intensity as obtained for the parallel plate of lithium niobate crystal: (a) the row data; (b) the filtered data obtained as sequence data-processing procedure. The software filter cuts off the constant onset intensity as well as reduces the noise.

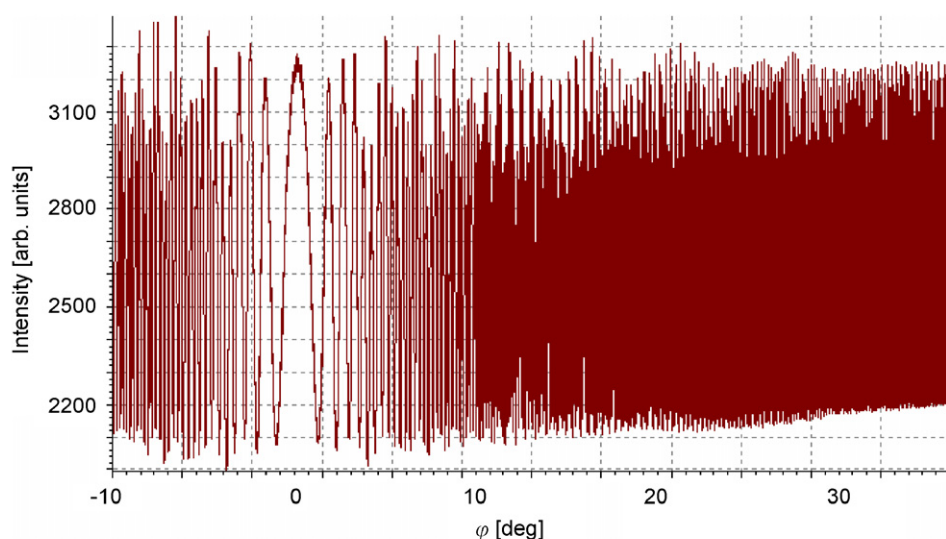

Fig. 3. The angular dependence of the light intensity as obtained for the parallel plate of lithium niobate crystal in the range of turning angles $-10^\circ < \varphi < 37^\circ$.

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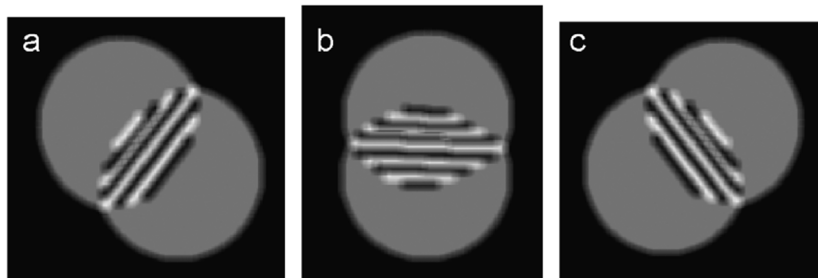

Fig. 5. The interference fringes at different orientations of the sample: (a) and (c) correspond to the sample slightly shifted out of its zero position; (b) represent the case when the sample is oriented normally to the laser beam, i.e. coincides with a sample zero position.

on an interference principle and provides thus the best precision. It does not require an additional equipment and can be applied in the following way:

- the sample is mounted into the holder;
- the mirror is set behind a sample and closed by a black paper;
- a sample is set approximately in its zero position;
- when the beam is reflected from a sample surface it can be superposed with the beam reflected from the other mirror leading thus to an appearing of interference fringes (see Fig. 5). The interference picture helps to verify a sample orientation. In the Fig. 5(a) and (c), a sample is slightly shifted out relatively to its zero position, whereas the Fig. 5(b), represent the case when a sample is oriented normally to the laser beam. By following this procedure a sample can be set into a zero position with absolute precision of about $15' \approx 0.004^\circ$.

The error of the IRPP method decreases at large turning angles as this evidently follows from Eqs. (7) and (8). Accordingly, the refractive measurements should be conducted at maximum possible turning angles (normally in the region $50^\circ < \varphi < 87^\circ$), where the error magnitude $\delta n = 0.0007$ as for the sample thickness $d = 10\,\mathrm{mm}$. Nevertheless, the precision in our measurements is almost of one order of magnitude better comparing with [12], where the same technique for refractive index measurements has been applied. A considerable increase of the precision is reached in our case by an automation of the measuring procedure, improvement of the software as well as implementing of the interferometric method for a precise determination of a sample zero position.

The accuracy of the method can be considerably better if to improve the precision of sample thickness measurements. With e.g. of $\delta d = 0.1\,\mu\mathrm{m}$ the accuracy of IRPP method may be improved even by almost of one order of magnitude.

5. Conclusions

We present here the automated device for the refractive index measurements based on the interferometry of a rotated parallel plate (IRPP technique). The device consists of the Michelson interferometer, the sample rotation system and the optoelectronic registration system. A refractive index of parallel plates is determined by their rotation through measuring simultaneously a shift of interference fringes. Although the IRPP technique is known from long ago [5], several considerable improvements have been done in the present work in order to improve the accuracy of method. The measuring process is completely automated. The method has been tested on the model crystal of the lithium niobate giving the magnitudes for ordinary and extraordinary refractive indices as $n_0 = 2.2865 \pm 0.0007$ and $n_e = 2.2034 \pm 0.0007$, respectively. A considerable increase of the precision is reached in our case by an automation of the measuring procedure, improvement of the software as well as implementing the new interferometric method for a precise determination of a sample zero position. The designed automated interferometric device has several advantages:

- a completely automated process for express precise measurements of refractive indices of isotropic or anisotropic optical materials;
- a possibility of non-destructive control providing the samples tested to be used in further applications;
- a refractometry of low-symmetry crystal materials, including the uniaxial and biaxial crystals;
- measurements of the refractive index dispersion.

The proposed automated device thus can be offered for use in scientific research laboratories and industry.

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References

[1] Huang Z, Chu J. The refractive index dispersion of $Hg_{1-x}Cd_xTe$ by infrared spectroscopic ellipsometry. Infrared Phys 2001;42:77–80.

[2] Adamson P. Laser diagnostics of nanoscale dielectric films on absorbing substrate by differential reflectivity and ellipsometry. Opt Laser Technol 2002;34:561–8.

[3] Romanjuk MO. Crystal optics. Kyiv: IZMN; 1997. 432p. (in Ukrainian).

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[4] Daimon M, Masumura A. High-precision measurements of the refractive index and its temperature coefficient of calcium fluoride in a wide wavelength range from 138 to 2326 nm. Appl Opt 2002;41:5275–81.
[5] Shumate MS. Interferometric measurement of large indices of refraction. Appl Opt 1966;5:327–31.
[6] Andrushchak AS, Mytsyk BG. Refractive indices measurement of the isotropic and anisotropic materials by the interferometric method. Izmeritelnaya Tech 1992;5:333–46 (in Russian).
[7] Schlarb U, Betzler K. Influence of the defect structure on the refractive indices of undoped and Mg-doped lithium niobate. Phys Rev B 1994;50:751–7.
[8] Fischer DW, Ohmer MC, Schunemann PG, Pollak TM. Direct measurement of $ZnGeP_2$ birefringence from 0.66 to 12.2 μm using polarized light interference. J Appl Phys 1995;77:5942–5.
[9] Scheel HJ. Historical aspects of crystal growth technology. J Cryst Growth 2000;211:1–12.
[10] Nicholls JFH, Henderson B, Chai BHT. Accurate determination of the indices of refraction of nonlinear optical crystals. Appl Opt 1997;36:8587–94.
[11] Maruyama H, Inoue S, Mitsuyama T, Ohmi M, Haruna M. Low-coherence interferometer system for the simultaneous measurement of refractive index and thickness. Appl Opt 2002;41:1315–22.
[12] Gillen GD, Guha S. Refactive-index measurements of zinc germanium diphosphide at 300 and 77 K by use of a modified Michelson interferometer. ApplOpt 2004;43:2054–7.
[13] Musso M, Aschauer R, Asenbaum A, Vasi C, Wilhelm E. Interferometric determination of the refractive index of liquid sulphur dioxide. Meas Sci Technol 2000;11:1714–20.
[14] Andrushchak AS. Two-beam interferometer for measurement of refractive index of isotropic and anisotropic materials. Patent of Russian Federation no. 2102700 from 20.01.98. Bull. no. 2 [in Russian].