

## Automatic interferometric equipment for refractive index determination of isotropic and anisotropic materials

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### ABSTRACT

Novel computer aided interferometric equipment for non-destructive refraction index express-measurements of the parallel plate samples made of isotropic and anisotropic optical materials was designed. The measurement error lies in forth sign after comma, that allows to recommend interferometric-turning method for wide using.

**Keywords** : refractive index, interferometric-turning method, isotropic and anisotropic optical materials.

### 1. INTRODUCTION

Refractive indexes belong to one of the most important parameters of isotropic and anisotropic media, which knowledge is necessary for solving of different physical tasks both fundamental and applicative. So at fundamental studies of optical material characteristics such as thermo-, piezo [1-4], electro-, magneto- [5,6] and non-linear optical coefficients [7] or acousto-optic figure of merit [8,9] previous measurement of refractive index is needed. At various materials applications, e.g. at calculation of phase synchronism conditions at the second harmonics generation [7], at calculation of polarization changes of light parameter in electro- and acousto-optical cells [10], etc., knowledge of their refractive indexes is also obligatory, moreover with high precision.

Several methods are used today for refractive index measurement. The most widespread of them are: 1) method of ellipsometry [11], immersion-interferential method [5], minimum deviation method [12], and interferometric-turning method [13,14]. Each of these methods has its advantages and shortcomings as well as correspondent field of application.

So method of ellipsometry can be used only for determination of refractive indices of thin film grown on substrates. Immersion method consists in comparison of refractive indices for crystal material and immersion liquids. Passport collection of immersion liquids [15] includes ones with refractive indices in the range of 1.4÷1.8, that substantially restricted a range of the method application for a lot of crystals.

Application of minimum deviation method demands a sample in the form of wedge or prism, therefore it is frequently called by a method of prism. The method provides rather high precision of  $n$  measurement for optical materials, however, it is uneconomic because it demands the certain part of the expensive crystal material produced in form of prism to be sacrificed. As a rule, obtained prisms are already unsuitable for further investigations. Besides, refractive index values of the prism material can differ substantially from value of  $n$  in the samples grown by other method [16]. Moreover, refractive index value can even change along one grown ingot [17].

Therefore, for practical tasks it is important to measure the refractive indices of the samples, which will be investigated later. The most of working samples for their application are obtained in the form of plates with thickness of a few millimeters, which is unsuitable for refractive index measurement by use of above mentioned methods. For this purpose the interferometric-turning method can be only used. It consists in non-destructive measurement of refractive index for parallel-sided plates from optical materials at their turning in one arm of interferometer. After the refraction measurements the samples are suitable for further practical use.

This method has recently been developed [18]. We made several improvements and patented them [19-23] and nowadays is a frequently used one [24-26]. Unfortunately its application is limited up to now by low precision and complication of a measuring process, which requires a substantial equipment and program support.

The aim of present work is a development of an automatized equipment (supplied with a corresponding program software) for non-destructive refraction index express-measurements of the parallel plate samples made of isotropic and anisotropic optical material. The tested samples are thus suitable for further usage after their measurements.

## 2. METHOD CHARACTERIZATION

The novel experimental computer aided equipment for refractive index determination of isotropic and anisotropic materials consists of the Michelson interferometer (laser, two mirrors and splitting prism), the rotation device (rotation mechanism with high gearing coefficient, step-motor and apparatus for fixing sample) and opto-electronic registration system (polarizer, lens, photodetector, control device and computer) (the equipment is described in detail in [21]). The designed software for measurement control drives step-motor that rotates a sample due to driving gear and records interference fringes shift  $k$  by means of photodetector. For calculation of absolute refractive index it is necessary to found a change of optical path in the sample at its rotation from „0” to some angle  $f$  („0” is a position of normal incidence of light beam on a sample surface). Then, absolute refractive index  $n$  can be easily written [19]:

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

where  $\lambda$  is laser wavelength,  $d$  is sample thickness. It is worthy to note, that the method can be used for measurement of refractive indices for both uniaxial and biaxial crystals. Typical results of measurements are shown in Fig.1.

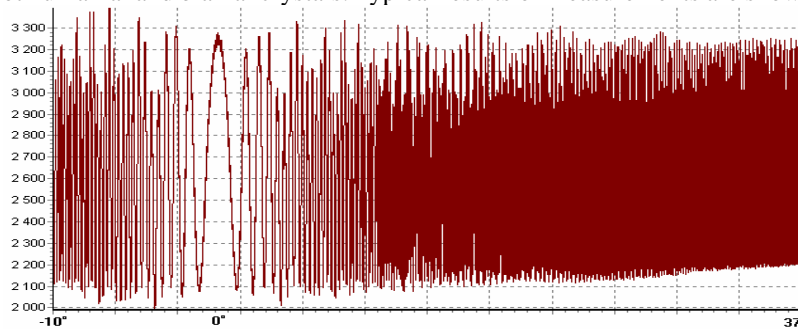


Fig. 1. The results of refractive index measurements for LiNbO<sub>3</sub> crystal in the range of rotation angle  $f$  from -10° to 37°.

For previous signal processing filtration is used to avoid a noise influence. Two filters are used: 1) filter of low frequencies and 2) filter of high frequencies. Filter of low frequencies delivers from noises which worsen calculation of maximum number  $k$  (total maximum number can reach 10 thousands. Therefore automatic calculation of maximum number is only used). Filter of high frequencies saves from constant component of input signal. This allows to simplify considerably maximum account algorithm, that in turns decreases a possibility of mistakes arising at maximum account. Digital filters of first order are used for filtration. Their equivalent is integration and differential circuit with additional modifications:

1. Since useful signal is sinusoidal signal of variable frequency, minimal and maximal frequencies can differ in a thousand times. In our case use of simple filters leads to partial or full loss of useful information. Therefore, filters with variable frequency of cut were used. Frequency cut change low for the filter is determined by software user due to indication of  $\lambda$ ,  $d$ ,  $f_0$  and  $n$ . Parameters  $f_0$  and  $n$  are assigned approximately. A criterion of correct determination of the parameters is absence of considerable change of useful signal amplitude in output signal.
2. Such filters are known to change phase of sinusoidal signal. In our case this leads to essential increase of error for refractive index determination. Therefore, filtration is conducted in two stages. Firstly reading takes place from  $X_0$  to  $X_n$ . Then in on the way back from  $X_n$  to  $X_0$ . That is filtration occurs two times. As a result of first stage signal phase changes on 45° in one side. As a result of second stage phase changes on 45° in the other side. Total phase shift is equal to zero.

We have estimated measurement error for theoretically possible conditions of experiment. Regarding our data [19,20] an absolute error of thickness determination at plate thickness  $d=10$  mm can be equal to  $\delta d = 0.01 \mu\text{m}$ , unstableness of ???-302 laser wavelength is equal to  $3 \cdot 10^{-8} \mu\text{m}$  for  $\lambda=0.6328 \mu\text{m}$ . For really possible measurable errors of interference order  $\delta k = 0.007$  and turning angle  $\delta \varphi = 1 \cdot 10^{-6}$  theoretically possible error for refractive index determination is equal to  $4.4 \cdot 10^{-6}$ . Rather high value of obtained error of measurement is conclusive proof for practical application of the described interferometric -turning method.

## 3. APPROBATION OF THE EQUIPMENT

Approbation of the equipment was realized on example of LiNbO<sub>3</sub> and langasite uniaxial crystals. Optical light beams with polarization parallel to sample rotation axis were used for measurement of extraordinary refractive index  $n_e$ ,

while beams with polarization transverse to rotation axis were used for measurement of ordinary refractive index  $n_o$ . Optical axis was established parallel to the sample rotation axis.

As a result of measurements conducted the following data were obtained:  $n_o=2.2865\pm0.0007$ ,  $n_e = 2.2034\pm0.0007$  for  $\text{LiNbO}_3$  and  $n_o=1.8988\pm0.0008$ ,  $n_e = 1.9117\pm0.0008$  for langasite. For comparison literature data of refractive index measurement for  $\text{LiNbO}_3$  crystals are provided in Table. As shown from table, the refractive index depends strongly on crystal stoichiometry. In particular, shift of stoichiometry to Nb content increase leads to considerable rise of extraordinary refractive index  $n_e$  value as well as to slight decrease of  $n_o$  value. Our results indicate that the investigated crystals contain excess of Nb.

Table. Refractive indices of  $\text{LiNbO}_3$  crystal

293	0,6328	$n_e$	<b>2,189</b>	Excess of Li	[27]
293	0,6328	$n_o$	2,2878	Excess of Li	[27]
293	0,63282	$n_e$	<b>2,2024</b>	Li/Nb=0.946	[27]
293	0,63282	$n_o$	2,28647	Li/Nb=0.946	[27]
298	0,6328	$n_e$	<b>2,2028</b>		[28]
298	0,6328	$n_o$	2,2866		[28]

It is worthy to note that theoretically estimated error of measurement  $\delta n = 4.4 \cdot 10^{-6}$  substantially exceeds experimental one. This fact is due to the measurable parameters in equation (1), i.e. sample thickness, interference order, and turning angle, were determined with lower precision, than it is mentioned in section 2. So in our case  $\delta d = 1 \mu\text{m}$  for the crystal with thickness  $d=10 \text{ mm}$ , real value of interference fringe shift was  $\delta k = 0.01$ . An error of turning angle determination  $\delta\phi$  could be defined in our case by value of sample turning step, i.e.  $0.0004^\circ$ . However, at described measurements another problem arises – complication of sample zero position founding, which plays key role at determination of real rotation angle measurement error.

The founding of “0” position, at which laser beam is normal incident at sample surface is rather complicated problem, since near zero position wide interference peaks exist, maximums of which are hardly to determine with high precision. Taking into account last problem the authors [14] determined “0” by use of approximate calculations.

We propose program method for “0” founding. Principle of program method can be understood from figure 2:

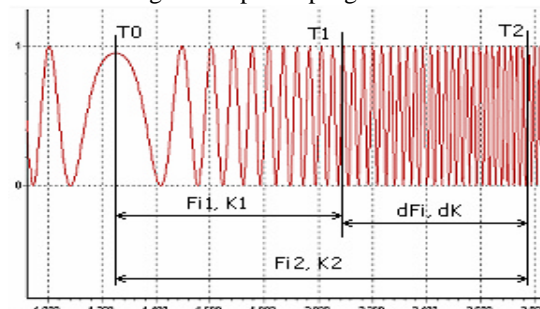


Fig.2. Illustration of sample zero position determination

Points  $\phi_0, \phi_1, \phi_2$  are chosen at will. Then knowing  $\phi, d, F_i(\phi_1)$  and  $K_1$  one can calculate  $n$  by equation (1). Value  $n$  can be also calculated from  $\phi, d, F_i+dF_i$  and  $K_1+dK$ . If point  $\phi_0$  be correct then refractive indices in two cases will be equal. But  $\phi_0$  is chosen at will and therefore these results will differ. An algorithm was used which choose  $F_i$  by such a way in order the results coincide. By means of the above described method the sample zero position can be determined with precision  $0.004$  degree, which specify error of angle determination  $\delta\phi=0.004^\circ$ .

Estimated at the above parameters precision of refractive index determination  $\delta n$  for the sample with  $d=10 \text{ mm}$  depends on turning angle, asymptotically decreasing at movement to high angles. Consequently, determination of  $n$  should be conducted at maximum turning angles as possible  $\phi = 50 \div 87^\circ$ , where error value of refractive index determination is  $\delta n = 0.0007$ .

Precision of refractive index measurement can be improved any more due to precision increasing of sample thickness determination or fixing of the turning angle. But, according to our estimations at given sample thickness determination precision of  $\delta d = 1 \mu\text{m}$  a decrease of angle error in order of magnitude ( $\delta\phi=0.0004^\circ$ ) leads only to double

rise of precision of  $n$  determination. Meanwhile at use of parallel-sided plates with  $\delta d = 0.1 \mu\text{m}$  the precision of refractive index determination can be increased in order of magnitude.

#### 4. CONCLUSIONS

Therefore, automatized equipment for non-destructive refraction index express-measurements of the parallel plate samples made of isotropic and anisotropic optical material by interferometric-turning method was designed. The equipment was shown to give high precision of refractive index measurement. So, precision in our experiments is almost in order of magnitude greater than in previous papers, where interferometric method of refractive index determination was used. Increase of measurement precision is approached due to their automatization, improvement of software as well as owing to more precise determination of sample zero position.

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