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THE COMBINED ELLIPSOMETRIC METHOD OF COMPLETE OPTICAL CHARACTERIZATION OF CRYSTALS.
I. DETERMINATION OF THE ORIENTATION OF THE OPTICAL INDICATRIX

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The first part of the article describes in detail the method of determining the orientation of the optical indicatrix in crystals. The experimental basis of the method was the careful ellipsometric measurements of the dependence of the effective refractive index on the angle of rotation of the crystal around the normal to the investigated surface, $n_{eff} = f(\alpha)$. The measurements were carried out on crystals cadmium tungstate ($CdWO_4$) and lithium niobate ($LiNbO_3$). Note that n_{eff} is the real part of the complex refractive index of the crystal, $N_{eff} = n_{eff} - i \cdot k_{eff}$, calculated on the basis of the model "optically isotropic homogeneous medium – optically isotropic homogeneous substrate". It is experimentally proved that the extrema of the dependence $n_{eff} = f(\alpha)$ determine the orientation of the optical indicatrix. It is especially important that knowledge of the crystallographic orientation of the investigated samples is completely unnecessary for the practical implementation of the method. The proposed method is applicable to crystals of any crystallographic system. This method of determining the orientation of the optical indicatrix became the basis for the combined ellipsometric method of the complete optical characterization of crystals and is its first stage.

Key words: ellipsometry, optical indicatrix, principal refractive indexes, uniaxial and biaxial crystals.

1. Introduction

The principled possibility of the application of ellipsometry for the optical characterization of anisotropic structures was analyzed already in early papers which concerning this theme (see, for example, Ref. [1-7]). In particular, using ellipsometry D. J. De Smet [1] studied the behavior of uniaxial anisotropic surface with optical axis in plane of the surface. In another article [2], D. J. De Smet compared two different methods for calculating changes of polarization of light reflected from the surface of a biaxial material. Using any of the calculation methods, the reflection properties can be summarized in terms of a reflection matrix suitable for use in Jones calculus. R. M. A. Azzam and N. M. Bashara [3] have proposed an improved criterion for computing the normalized 2×2 complex reflection matrix of an anisotropic surface from multiple-null ellipsometer measurements. As an example, they have considered the case of absorbing uniaxial crystals with the optical axis parallel to the

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investigated surface. Later, R. M. A. Azzam [4] researched optical properties of absorbing uniaxial crystals by reflection perpendicular-incidence ellipsometry (PIE). He has derived equations for determining of the ordinary (N_o) and extraordinary (N_e) complex refractive indices. J. Lekner paid much attention to the use of ellipsometry for the study of anisotropic structures [5, 6]. In particular, J. Lekner [6] proposed a simple scheme for determining the dielectric constants and the orientation of the optical axis of a uniaxial crystal by reflection ellipsometry. This method can be applied to both nonabsorbing and absorbing crystals. However, biaxial crystals are not covered by the proposed scheme. But especially we would like to highlight the work of R. H. W. Graves [7]. In this article, the author described an iterative method for determining the optical constants of crystals if the orientation of the principal axes of the optical indicatrix is known.

It is well known [8-10] that the following parameters must be measured for the complete optical characterization of crystals:

a) the refractive index $\boldsymbol{n}$ – for the crystals of a cubic system;

b) the principal refractive indexes $\boldsymbol{n_o}$ (ordinary), $\boldsymbol{n_e}$ (extraordinary), the optical sign of the crystal (or birefringence, $\Delta\boldsymbol{n}= \boldsymbol{n_e} - \boldsymbol{n_o}$) – for the uniaxial crystals (crystals of tetragonal, trigonal and hexagonal systems);

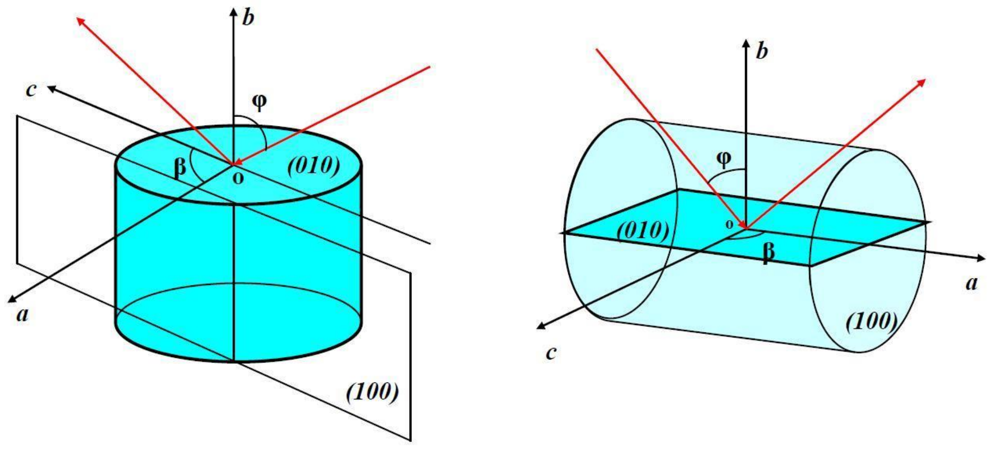
c) the principal refractive indexes $\boldsymbol{n_g}$ (grand), $\boldsymbol{n_m}$ (moem), $\boldsymbol{n_p}$ (petit), the angle between optical axes $\boldsymbol{2V}$, the optical sign of the crystal – for the biaxial crystals (crystals of orthorhombic, monoclinic and triclinic systems);

In addition, for all crystals, except for cubic ones, it is necessary to know the orientation of principal axes of the optical indicatrix (or Fletcher's indicatrix) relative to the crystallographic coordinate system.

Such an optical characterization of a crystal can be performed using ellipsometry by making measurements in the principal sections of the optical indicatrix and determining the principal refractive indices of the crystal using the relations given in the work of R. H. W. Graves [7]. Thus, it is precisely the determination of the orientation of the optical indicatrix in the most general case that comes to the fore. R. H. W. Graves [7] did not have such a problem, since in the crystals of the rhombic system, which he studied, the direction of the principal axes of the optical indicatrix coincides with the direction of the principal crystallographic axes. Thus, knowing the orientation of the crystallographic coordinate system, he automatically knew the orientation of the principal axes of the optical indicatrix. For crystals of the middle category of symmetry (trigonal, hexagonal, and tetragonal systems), the situation is even simpler, since the axis of rotation of the optical indicatrix (and for these crystals it is an ellipsoid of rotation) coincides with the principal axis of symmetry of the crystal. In other words, in the aforementioned cases, knowledge of the orientation of the crystallographic coordinate system automatically determines the orientation of the optical indicatrix. The situation is more complicated in crystals of monoclinic and triclinic systems. In monoclinic crystals of classes 2 and $2/m$, one of the axes of the optical indicatrix runs along the axis 2 or along the normal to the plane m, but the directions of the other two axes do not depend on the crystal symmetry. In triclinic crystals, the orientation of the optical indicatrix is in no way connected with the symmetry of the crystal, and it must be determined for each substance [8-9]. It will be shown below that in our proposed method for determining the orientation of the optical indicatrix, knowledge of the orientation of the crystallographic coordinate system is completely unnecessary. However, in many cases, knowledge of the crystallographic orientation of the sample can significantly simplify the determination of the orientation of the optical indicatrix. In conclusion of this introduction, it should be emphasized that all our experimental studies were carried out using single wavelength ellipsometry (laser ellipsometer LEF-3M-1, λ=632.8 nm). Therefore, in the analysis of publications related to studies of anisotropic structures, the main attention was paid to this type of ellipsometry.

2. Experimental foundations of the combined ellipsometric method

Using results ellipsometric measurements performed on plane (010) of $CdWO_4$ crystal (monoclinic system, class $2/m$, space group of symmetry $P2/c$, $a = 0.502$ nm, $b = 0.585$ nm, $c = 0.507$ nm, $\beta = 91.5^{\circ}$ [11]), we have found that the dependence of the effective refractive index, n_{eff}, from angle of crystal rotation around an axis b (crystallographic direction [010]) (Fig.1) has clearly expressed minimum and maximum (Fig.2).



a) b)

Fig.1. Scheme of ellipsometric measurements of CdWO4 single crystals: a) the crystal growth axis is perpendicular to the plane (010); b) the crystal growth axis is parallel to the plane (010).

Here n_{eff} is the real part of complex refractive index, $N_{eff} = n_{eff} - i \cdot k_{eff}$, calculated on basis of model “optically isotropic homogeneous medium - optically isotropic homogeneous substrate”. For this case, the fundamental ellipsometric equation is of the form [12]:

$$\rho = tg\Psi \cdot e^{i\Delta} = \frac{\sin\varphi \cdot tg\varphi - \sqrt{(N_{eff}/n_0)^2 - \sin^2\varphi}}{\sin\varphi \cdot tg\varphi + \sqrt{(N_{eff}/n_0)^2 - \sin^2\varphi}}. \tag{1}$$

In this equation ρ is the relative reflection coefficient; Ψ and Δ are the ellipsometric parameters of the investigated surface of crystal; φ is the angle of incidence, and n_0 is the refractive index of the medium. If the medium is atmospheric air, then $n_0 \approx 1$ can be taken. Thus, solving equation (1) relative to N_{eff}, we shall receive

$$N_{eff} = n_{eff} - i \cdot k_{eff} = \sin\varphi \cdot \sqrt{\left(\frac{1-\rho}{1+\rho} \cdot tg\varphi\right)^2 + 1}, \tag{2}$$

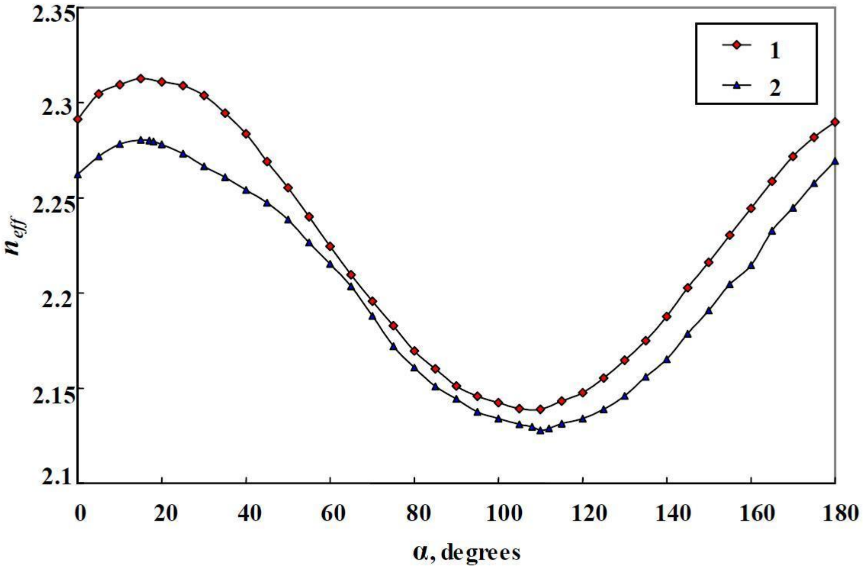


Fig.2. Dependence of the “effective” refractive index n_{eff} on the angle of rotation α of the plane (100) of the $CdWO_4$ crystal relative to the plane of incidence of the laser beam (measurements were performed on the plane (100), angle of incidence φ = 50 °, wavelength λ = 632.8 nm). 1 – sample No. 2-1 (pure $CdWO_4$, the crystal growth axis is perpendicular to the plane (010)), 2 – sample No. 3-1 ($CdWO_4$ doped by Fe, the crystal growth axis is perpendicular to the plane (010)).

The name “effective refractive index” is conditional and chosen to emphasize that this quantity is auxiliary parameter and is not one of the principal refractive indexes. Such character of dependence $n_{eff} = f(\alpha)$ was experimentally confirmed by ellipsometric measurements on various $CdWO_4$ crystals, made at the different angles of incidence. In particular, we have investigated $CdWO_4$ crystals with growth axis both in plane (010) and perpendicular to this plane (Fig.1). Besides pure crystals, we have also researched $CdWO_4$ crystals doped by Fe and PbO. For example, Fig. 2 shows the dependences $n_{eff} = f(\alpha)$ measured on two different samples at the same angle of incidence of the laser beam. As we can see, despite the differences in the values of n_{eff} (which is quite natural for different samples), the positions of the extrema in these dependences are the same. The angle of rotation of the crystal around the normal to the plane of the sample was measured relative to the plane of incidence, with which a certain plane of the crystal was aligned in the initial position. It was found that in all the investigated $CdWO_4$ crystals, the plane (100) was turned relative to the plane of incidence of the laser beam by about 18.5-19° in the case of fixing the maximum in the dependence $n_{eff} = f(\alpha)$ (if you rotate the crystal clockwise, Fig.1). Correspondingly in the case of fixing the minimum in the dependence $n_{eff} = f(\alpha)$ the plane (100) was turned relative to plane of incidence by about 109°

(Fig.2). The assumption arose that the optical indicatrix orientation can be defined by measuring dependence $n_{eff} = f(\alpha)$. Namely, extrema of dependence $n_{eff} = f(\alpha)$ define orientation of ellipsoid of the optical indicatrix. For verification of this assumption we have used the fact that in monoclinic crystals in classes 2 and 2/*m* one of principal axes of the optic indicatrix direct along axes 2 or is perpendicular to plane *m*. If our assumption is correct that in dependence $n_{eff} = f(\alpha)$, measured on the plane (100) of $CdWO_4$ crystal (or any other plane, which is perpendicular to plane (010)), the maximum or minimum must be observed in direction of crystallographic axis *b*. The results of ellipsometric measurements, performed on the plane (100) of the $CdWO_4$ crystal, convincingly confirmed the correctness of this assumption: the dependence $n_{eff} = f(\alpha)$ has a distinct minimum in the direction of the crystallographic axis *b*.

For full confidence in made conclusions we performed also research of $LiNbO_3$ crystals (trigonal system, class 3*m*, space group of symmetry *R*3*c*, rhombohedral cell parameters: $a = 0.549$ nm, $\alpha = 55^{\circ}53'$, hexagonal cell parameters: $a = 0.515$ nm, $c = 1.386$ nm [13, 14]). Since $LiNbO_3$ crystal belongs to the trigonal system, its optical indicatrix is a uniaxial ellipsoid (ellipsoid of rotation). The axis of rotation of this ellipsoid coincides with the optical axis of crystal. Performing the ellipsometric measurements on planes (100) and (010) $LiNbO_3$ crystal we have received results which in a qualitative sense are identical to the results for $CdWO_4$ crystal. Namely, the clear minimum was recorded in dependence $n_{eff} = f(\alpha)$ in direction of crystallographic axis *c* (crystallographic direction [001]). Besides, in studies of the $LiNbO_3$ crystal we attached much importance to measurements on plane (001). In the $LiNbO_3$ crystal the plane (001) corresponds to circular cross section of the optical indicatrix. Therefore the n_{eff} value should not depend on the angle of crystal rotation around an axis *c*. The results of ellipsometric measurements fully confirmed this conclusion: at measurements on plane (001) of well oriented $LiNbO_3$ crystals the spread of n_{eff} values was accidental and was not more than $\pm 0{,}001$ at angle of incidence $\varphi = 45^{\circ}$.

The results of ellipsometric measurements can be presented as an analogue of an elliptical cross section of an optical indicatrix. Let us set aside on a certain scale the value of n_{eff}, measured at a certain angle of rotation of the crystal around the vertical axis, on a straight line rotated by the same angle α relative to the reference plane. We measured the angle α clockwise relative to the plane of incidence of the laser beam. With this plane in the initial position, we combined a certain plane of the crystal. Performing this procedure for the entire range of angles (0÷360°, e.g. with step 5°), we shall obtain a curve, which is ellipse (Fig. 3). The resulting ellipse is an analogue of the elliptical cross section of the optical indicatrix. This elliptical cross section corresponds to the plane of the crystal on which these ellipsometric measurements were performed. For clarity, the ellipses in Fig. 3 are slightly lengthened along the abscissa axis by the appropriate choice of scale. It should be emphasized that this is only an analogue of the elliptical cross section, since the quantitative values of the refractive indices that correspond to the actual cross section of the optical indicatrix will be somewhat different. This becomes quite obvious if we take into account that the parameters of this ellipse depend on the angle of incidence of the beam on the surface of the investigated sample (Fig.3). However, the orientation of such an analogue of an elliptical cross section exactly corresponds to the orientation of the cross section itself. Precisely this form of presentation of the measurement results of the dependence $n_{eff} = f(\alpha)$ gives the opportunity to understand how is determined the optic indicatrix orientation.

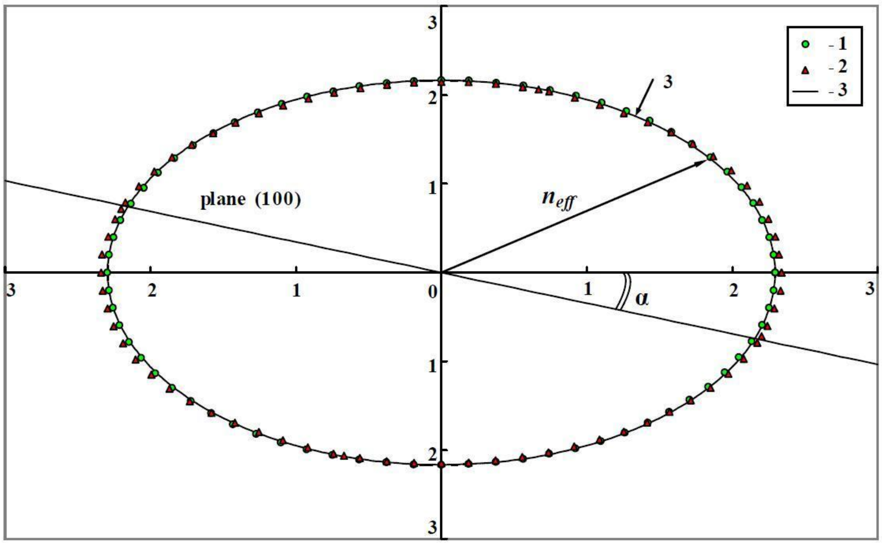


Fig.3. Presentation of the dependence $n_{eff} = f(\alpha)$ in the form of an analogue of the elliptical cross section of the optical indicatrix. The measurements were performed on the plane (010) of the $CdWO_4$ crystal (sample No. 2/1). Symbols – experiment (1– angle of incidence $\varphi = 55$ °, 2 – $\varphi = 45$ °); thin solid line (3) – theoretical ellipse obtained by fitting to experimental points.

3. The method of determination of the optical indicatrix orientation of crystals

The proposed method of determination of the optical indicatrix orientation in the most general case can be illustrated by the example of research of crystal with unknown orientation of crystallographic coordinate system. So, it is necessary to determine the orientation of the three principal axes (we denote them as N_1, N_2, N_3) and correspondingly the orientation of the three principal cross sections of the optic indicatrix. For this purpose it is necessary to make such steps:

1. An arbitrary plane of the investigated crystal qualitatively is grinded, polished, etched (if necessary). After that, the dependence $n_{eff} = f(\alpha)$ is measured on it. Next, we determine the position of the extrema of this dependence. If the crystal is high-quality and the plane is carefully prepared for elipsometric measurements, then minimum of dependence $n_{eff} = f(\alpha)$ will be shifted relative to the maximum on 90°. In other words, minor and major axes of an ellipse are mutually perpendicular. Therefore, we determine the position, for example, of the minor axis of an elliptic cross section, turn the investigated crystal on 90° around the vertical axis and make sure that this position corresponds to the major axis direction. As a result of these measurements we obtain the analogue of the elliptic cross section of the optical indicatrix, which is orientated relative to indicatrix, for example, as well as shown in Fig. 4. Let us denote the n_{eff} values, which correspond to minor axis C_1D_1 and major axis A_1B_1 of this ellipse, as $n_{effmin1}$ and $n_{effmax1}$.

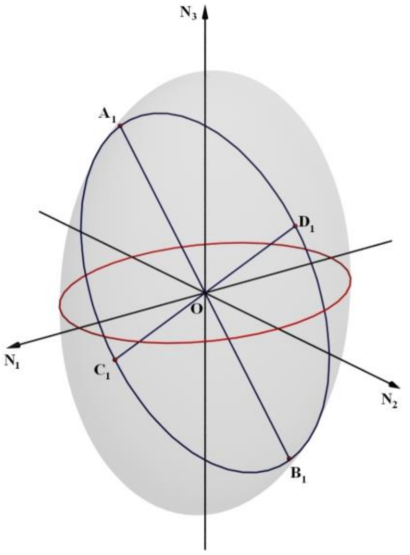


Fig.4. Mutual orientation of the optical indicatrix and the analogue of its elliptical cross section, obtained by measuring the dependence $n_{eff} = f(\alpha)$ on an arbitrary crystal plane.

2. Let us fix of the position, for example, of the major axis of this elliptic cross section. By grinding we turn this plane on ≈2÷3° around the major axis A_1B_1. The new elliptic cross section of the optical indicatrix corresponds to obtained new plane. Having prepared this plane for measurements, we determine the position of the extrema of the dependence $n_{eff} = f(\alpha)$ (Fig.5, axes C_2D_2, A_2B_2). Let us compare obtained value $n_{effmin2}$ with $n_{effmin1}$. If $n_{effmin2} < n_{effmin1}$ (Fig.5), that we repeat the procedure of plane rotation around major axis A_2B_2 of this new ellipse in the same direction. If $n_{effmin2} > n_{effmin1}$, that we rotate the plane in the opposite direction. These measurements should be made at the same angle of incidence. The aim of these measurements is to find a plane with lowest n_{effmin} value. As follows from the properties of the triaxial ellipsoid the minor axis CD of the elliptic cross section, which corresponds to this plane, is one of the principal axes of the optical indicatrix. If rotate a plane in the same direction as it made on Fig.5, that the minor axis of the sought-for elliptic cross section will be axis N_1 (Fig.6a). It is clear that depending on the orientation of the initial elliptic section relative to optical indicatrix (Fig.4), we can eventually reach another elliptical cross section, whose minor axis CD is the axis N_2 (Fig.6b).

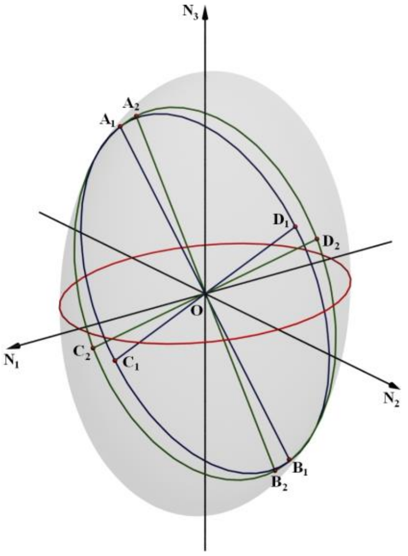
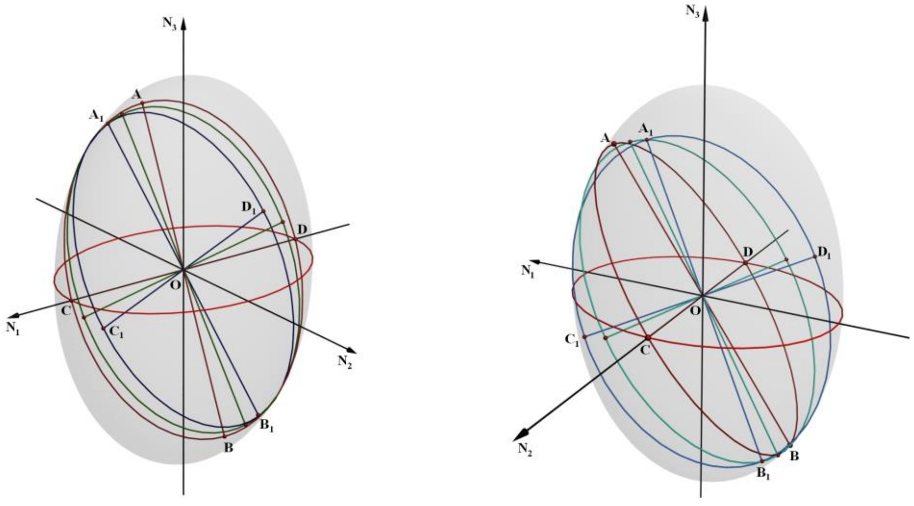


Fig.5. Mutual orientation of the optical indicatrix and several analogs of its elliptical cross sections, obtained as a result of successive measurements of the dependence $n_{eff} = f(\alpha)$ on two crystal planes, when the value of $n_{effmin2}$, which corresponds to the minor axis C_2D_2, is less than $n_{effmin1}$ (axis C_1D_1 of the initial elliptical cross section).

3. So we have obtained the elliptic section with one of the principal axes of the optical indicatrix, for example, N_2 (Fig.6b). Next we prepare to measurements the plane, which is perpendicular to obtained plane and to this principal axis of the optical indicatrix. One of the principal cross sections of the optical indicatrix corresponds to this plane, in this case, *KFLG* (Fig.7). Performing ellipsometric measurements on this plane, we determine the directions of the two other axes of the optical indicatrix, in this case, N_1 and N_3 (Fig.7).

4. In crystal optics the principal axes of the optic indicatrix are denoted as N_g, N_m, N_p in accordance with magnitudes of principal refractive indexes [8, 9]. Determining of n_{eff} values in directions of the principal axes, we can to change designations of axes already on this stage. Let us denote the principal axis, which correspond the greatest n_{eff} value, as N_g, middle value – N_m, least value – N_p. If in the process of ellipsometric measurements it was found that one of the principal cross sections is a circle, that the optic indicatrix is uniaxial ellipsoid (or ellipsoid of rotation). In this case the axis which perpendicular to circular cross section is optic axis of this uniaxial crystal and is denoted as N_e. The examined succession of determination of the optic indicatrix orientation in crystal is not only one. It can be fixed in initial elliptic cross section the position of the minor axis C_1D_1 (Fig.4). The next plane for measurements can be obtained by rotation of initial plane around this minor axis C_1D_1. In this case, it is necessary to find the elliptical cross section, which corresponds to the maximum value of n_{effmax}.



a) b)

Fig.6. Mutual orientation of the optical indicatrix and several analogs of its elliptical cross sections, obtained as a result of successive measurements of the dependence $n_{eff} = f(\alpha)$ in the process of finding the minimum value n_{effmin}, which corresponds to the small axis CD of the sought elliptical cross section.
a) The axis CD of this elliptical section is the main axis N_1 of the optical indicatrix.
b) In this case, the axis CD is the main axis N_2 of the optical indicatrix.

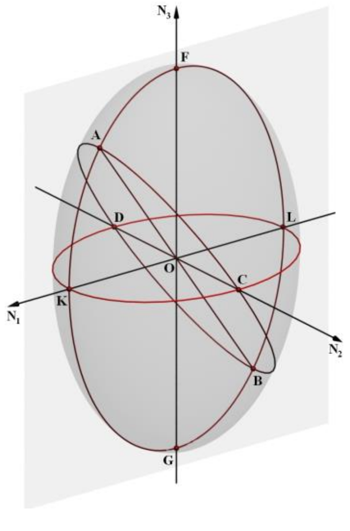


Fig.7. Mutual orientation of the optical indicatrix and analogs of its elliptical cross sections obtained as a result of measuring the dependence $n_{eff} = f(\alpha)$ on two crystal planes, which contain all the principal axes of the optical indicatrix.

In Fig.8 is shown the optic indicatrix orientation relative to crystallographic coordinate system in $CdWO_4$ crystal as an example of practical application of this method. As is true of all monoclinic crystals, so with $CdWO_4$ one of the principal axes of the optical indicatrix (in this case N_p) coincides with axis b.

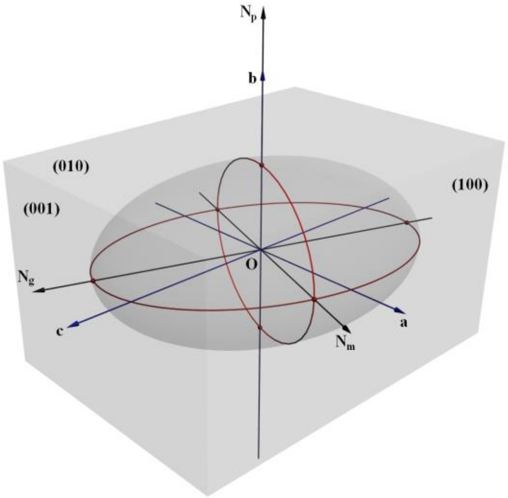


Fig.8. Orientation of the optical indicatrix relative to the crystallographic coordinate system in a $CdWO_4$ single crystal (for λ=632.8 nm).

Two other principal axes, N_g i N_m, lie in plane (010). The axis N_m of the optical indicatrix turned on angle $\alpha \approx 19°$ relative to crystallographic axis a. Since the angle between axes a and c is equal $\beta = 91.5°$ [11], that characteristic angle (or principal angle of full blackout) is equal $cN_g \approx 17.5°$. It should be noted that this orientation of the optical indicatrix relative to the crystallographic coordinate system in the $CdWO_4$ crystal corresponds only to the wavelength λ =632.8 nm. Since crystals of the monoclinic system are characterized by dispersion of the indicatrix axes, the orientation of the optical indicatrix changes with a change in the wavelength.

4. Conclusions

At first glance, it may seem that the presented method for determining the orientation of the optical indicatrix is somewhat cumbersome. In fact, this is not the case. Only the first step in this method (namely, determination of the orientation of the first principal axis of the optical indicatrix) is rather laborious But in laboratories where there is modern equipment for processing the surfaces of the investigated crystals, this first step will not present a big problem. All subsequent steps to achieve this goal are much easier. From the description of the method for determining the orientation of the optical indicatrix, it is obvious that it is applicable for crystals of any crystallographic system. It is especially important that the method is applicable even if the crystallographic orientation of the samples is unknown. In conclusion, it should be said that this method became the basis of the combined ellipsometric method of the complete optical characterization of crystals. The proposed combined ellipsometric method consists of several stages. Thus, in this part of the article, we have considered only its first stage – the determination of the orientation of the optical indicatrix. In the second part of the article, we hope to consider the second stage – the determination of the optical constants of the crystal.

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КОМБІНОВАНА ЕЛІПСОМЕТРИЧНА МЕТОДИКА ПОВНОЇ ОПТИЧНОЇ ХАРАКТЕРИЗАЦІЇ КРИСТАЛІВ.
І. ВИЗНАЧЕННЯ ОРІЄНТАЦІЇ ОПТИЧНОЇ ІНДИКАТРИСИ

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Комбінована еліпсометрична методика повної оптичної характеризації кристалів складається з декількох етапів. У першій частині праці докладно описаний еліпсометричний метод визначення орієнтації оптичної індикатриси у кристалах. Знання орієнтації оптичної індикатриси потрібне для виконання необхідних еліпсометричних вимірювань у головних перерізах індикатриси з метою визначення головних показників заломлення кристала. Експериментальною основою методу стали ретельні еліпсометричні вимірювання залежності ефективного показника заломлення від кута повороту кристала навколо нормалі до досліджуваної поверхні, $n_{eff} = f(\alpha)$. Зауважимо, що n_{eff} – це дійсна частина комплексного показника заломлення кристала, $N_{eff} = n_{eff} - i\cdot k_{eff}$, розрахованого за моделлю “оптично ізотропне однорідне середовище – оптично ізотропна однорідна підкладка”. Очевидно, що за допомогою такої моделі не можна визначити точні значення головних показників заломлення оптично анізотропної структури (кристала). Тому назва “ефективний показник заломлення” є умовною і підкреслює допоміжний характер величини n_{eff}. Вимірювання залежності $n_{eff} = f(\alpha)$ були виконані на кристалах вольфрамату кадмію ($CdWO_4$) і ніобату літію ($LiNbO_3$). Зокрема, було з’ясовано, що залежність $n_{eff} = f(\alpha)$, виміряна на площині (010) кристала $CdWO_4$ в діапазоні $\alpha = 0 \div 360°$, має яскраво виражені мінімуми і максимуми. Експериментально доведено, що такий характер залежності $n_{eff} = f(\alpha)$ є загальним для оптично анізотропних кристалів і її екстремуми визначають орієнтацію оптичної індикатриси. Це стає цілком очевидним, якщо результати вимірювань залежності $n_{eff} = f(\alpha)$ представити у вигляді аналогу еліптичного перерізу оптичної індикатриси. Саме така форма представлення залежності $n_{eff} = f(\alpha)$ і дає змогу чітко зрозуміти сутність цього методу. Особливо важливим є те, що для практичної реалізації методу знання кристалографічної орієнтації досліджуваних зразків є зовсім необов’язковим. Запропонований метод застосовний для кристалів будь-якої кристалографічної сингонії. Цей метод визначення орієнтації оптичної індикатриси став основою пропонованої еліпсометричної методики повної оптичної характеризації кристалів та її першим етапом.

Ключові слова: еліпсометрія, оптична індикатриса, головні показники заломлення, одновісні і двовісні кристали.

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