LINEAR BIREFRINGENCE OF UNIAXIAL ANISOTROPIC INORGANIC CRYSTALS MEASURED BY ELLIPSOMETRIC MEANS

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There are several optical methods (interferometric, refractometric or compensatory) for estimating the refractive indices and linear birefringence of the uniaxial layers. Now a simple ellipsometric method is applied for determining the linear birefringence of the thin anisotropic layers. This method consists in establishing the inclination of the axes of the polarization ellipse relative to the principal axes of the anisotropic uniax layer. The relation between the inclination angles of the axes of the polarization ellipse (relative to the principal axes of the anisotropic layer) at the exit and the azimuth of the incident linearly polarized light at the entrance of the layer permits to estimate (with good precision) the phase difference introduced by the uniax anisotropic layer between the ordinary and extraordinary components of light. The results of measurements on four inorganic uniaxial crystals (calcite, Iceland spar, tourmaline and quartz) are given in this communication. The results are compatible with those obtained by other methods for crystals with similar structure and symmetry.

Keywords: uniaxial crystals, linear birefringence, ellipsometric method.

1. Introduction

In this paper, a very simple method for determining the linear birefringence of uniaxial inorganic crystals is described and applied to four samples: calcite and its variety known as Iceland spar, tourmaline, and quartz.

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Calcite is a natural calcium carbonate ($CaCO_3$) belonging to the carbonates group and representing one of the most ubiquitous minerals in nature, as an important building-block in sedimentary environments. Calcium carbonate exhibits polymorphism, *i.e.* it exists in different crystal structures, depending on external conditions and thus, $CaCO_3$ crystallizes as calcite at low pressure and high temperature - having a rhombohedral symmetry, as well as aragonite at high pressure and low temperature - with an orthorhombic symmetry. Calcite crystals may sometimes grow to remarkable size (tens of centimeters), such as those in some alpine hydrothermal veins at Gonzen, Switzerland [1], or the speleal megascalenohedrons from Valea Firei - Humpleu Cave, Romania [2].

Calcite is uniaxial and optically negative, with strong birefringence (0.172 - 0.190) and it was observed that the substitution of other ions for Ca in its crystalline lattice raises the refractive indices [3].

Iceland spar (formerly known as Icelandic crystal or Latin *crystallus islandica*) is a perfectly transparent and optically clear variety of calcite, originally reported from Helgustadir Mine (Eskifjord, Iceland), but occurring in many other localities worldwide. It was on Iceland spar crystals that the birefringence effect was first observed and described by the Danish mathematician and physicist Erasmus Bartholin in 1669, later enabling Dutch physicist Christiaan Huygens to understand the nature of light as a wave and later still, Scottish geologist and physicist William Nicol to use the same type of calcite to create the first polarizing prism, known as Nicol prism.

Tourmaline is a borosilicate mineral belonging to the cyclosilicates / ring silicates group and is known to be a very complex mineral both chemically and structurally. Its general formula is:

$$XY_3Z_6\,(T_6O_{18})(BO_3)_3(O,OH)_3(OH,F)$$

where the structural positions may be occupied as follows:

X: Ca, Na, K, Pb or may be vacant

Y: Al, Fe^{2+}, Fe^{3+}, Li, Mg, Mn, Ti

Z: Al, Cr^{3+}, Fe^{3+}, V^{3+}

T: almost exclusively Si, but sometimes there can be minor Al and / or B^{3+} substitution [1].

According to the cations entering the various structural positions, the tourmaline group includes a great number of recognized and hypothetical species forming several solid-solutions with different end-members, of which the most common in nature are: *schörl* (X = Na; Y = Fe^{2+}; Z = Al), *dravite* (X = Na; Y = Mg; Z = Al) and *elbaite* (X = Na; Y = Al, Li; Z = Al). Tourmalines have rhombohedral symmetry rather than hexagonal and their crystals are mostly euhedral, ditrigonal - prismatic and of columnar habit, with lengthwise striations. The high variability in the chemical composition leads to a great number of differently colored varieties.

Tourmaline is uniaxial and optically negative, with variable pleochroism, which is particularly strong for the iron-bearing species. Generally, the refractive indices, birefringence and specific gravity are higher for increasing amounts of (Fe^{2+}+ Fe^{3+} + Mn + Ti), as shown in our previous study of some tourmalines from Lotru - Cibin Mountains (Romania) [4,5]. Tourmaline is typically present in granitic pegmatites [4], veins and in some metamorphic environments. Clear transparent colored tourmaline varieties provide quality gemstone material highly valued in jewelry. Crystals with polar symmetry showing strong piezoelectric properties are used in industrial pressure devices, such as depth-sounding equipment and other apparatus that detect and measure pressure variations; crystals that are strongly dichroic are used in optical devices for polarizing light.

Quartz is a silicon dioxide (SiO_2), but because of its framework structure applied to a silicon mineral it is classified as belonging to the tectosilicates group rather than to the oxides group. Along with feldspars, quartz is the most abundant mineral in the Earth's crust and occurs as an essential constituent of many acid igneous, sedimentary and metamorphic rocks. Generally, the silicon dioxide occurs as a number of polymorphs, each of them having specific temperature and pressure ranges, as well as distinct crystal structures: quartz, tridymite, cristobalite, coesite and stishovite.

Quartz crystals have a very pure composition, normally situated very close to 100% SiO_2, but also small amounts of other elements may be present, generally due either to minute inclusions of other minerals, or to liquid infillings in cavities within the crystals - as shown in our previous study of some quartz samples from Southern Carpathians (Romania) [6]. The quartz is uniaxial positive, and its enantiomorphism makes it „optically active": the polarization plane of the light passing along the optic axis is rotated either clockwise or anticlockwise depending on the crystal orientation, right- or left-handed. Quartz has rhombohedral symmetry, prismatic habit and present a large number of differently colored varieties. Quartz is one of the most stable minerals at the surface of the Earth. The main uses of quartz are in glassmaking, ceramic and foundry and also as an abrasive in sandpaper, sandblasting, millstones, grind stones etc.; gems and decorative materials. High-tech uses include devices for frequency control in radio, TV and other electronic communications equipment, for crystal-controlled clocks and watches and for optical devices; last but not least, quartz is the prime source of silicon, which is used in computer industry and solar cells.

2. Theoretical bases

The studied here inorganic crystals are anisotropic uniaxial, being characterized by two values of the light velocity when the propagation direction differs on the optical axis. The optical axis of the anisotropic media is the

direction for which the light propagation velocity does not depend on the orientation of the light electric field intensity (on its polarization state).

In the principal coordinate system *a*O*bc*, the uniaxial layer is characterized by two refractive indices: n_o and n_e. Generally, one supposes that the axis O*c* is the optical axis of the layer, and one defines the plane of the principal section – the plane determined by the optical axis and the propagation direction O*b* (see Fig. 1). The electric field intensity perpendicular to this plane belongs to the ordinary light component and that one parallel to the principal section is the extraordinary ray.

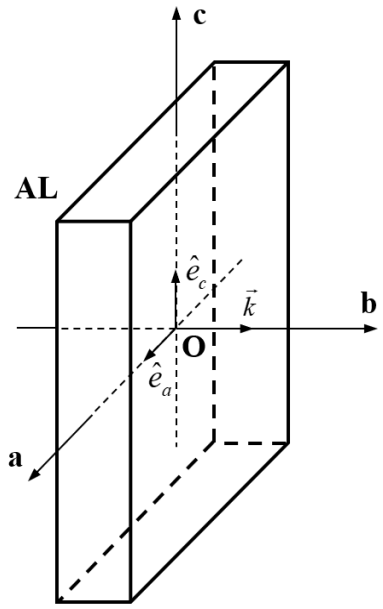

Fig. 1. Ordinary and extraordinary components of light in the anisotropic layer (AL): O*c* – optical axis, O*b* – light propagation direction, $\vec{k}$ - wave vector.

At normal incidence, the light ordinary and extraordinary components propagate in the same direction in the anisotropic layer.

The difference:

$$\Delta n = n_e - n_o \tag{1}$$

is known as linear birefringence because it characterizes the propagation of the linear polarized ordinary and extraordinary radiations through anisotropic layers.

The uniax anisotropic crystals are classified in positive and negative, function of the sign of the linear birefringence.

In the propagation process, the uniax anisotropic layer introduces a phase difference, $\Delta\psi$, between the two components of light [7,8]:

$$\Delta\psi = \frac{2\pi}{\lambda}\Delta nL\,. \tag{2}$$

In relation (2), L is the thickness of the anisotropic layer and λ is the wavelength (in vacuum) of the linearly polarized light crossing the layer.

When a linearly polarized light propagates through a uniaxial crystal (in a direction different from optical axis), the phase difference $\Delta\psi$ appears between the ordinary and extraordinary components of light. At the exit from the anisotropic layer, the radiation can have different polarization states as it follows [8]:

- linearly polarized with the same azimuth, α, when the phase difference is an even number of π, or with azimuth $-\alpha$ when the phase difference is an odd number of π;
- elliptical polarized with ellipse axes parallel to the principal axes of the uniax layer, or circular polarized (for azimuth angles an odd number of $\frac{\pi}{4}$) if the phase difference is an integer number of $\frac{\pi}{2}$;
- elliptical polarized for the phase difference between the values $2m\pi < \Delta\psi < (2m+1)\frac{\pi}{2}$. In this case, the ellipse semiaxes are rotated relative to the principal axes of the anisotropic layer.

In the third case, the angle of rotation, θ, of the ellipse's axes relative to the principal axes of the anisotropic layer depends on the azimuth angle, α, of the incident linearly polarized light and on the phase difference, $\Delta\psi$, introduced by the anisotropic layer between the ordinary and extraordinary components of light. This dependence is given by relation (3) (see [9] for its demonstration):

$$\mathrm{tg}2\theta = \mathrm{tg}2\alpha\cos\Delta\psi\,. \tag{3}$$

When the angles θ and α are known, the phase difference $\Delta\psi$ can be estimated based on relation (3) and the linear birefringence can be computed using the relation [9]:

$$\Delta n = \frac{\lambda}{2\pi L}\arccos\frac{\mathrm{tg}2\theta}{\mathrm{tg}2\alpha}\,. \tag{4}$$

To decide if the inorganic crystal is positive or negative (from the sign of the difference (1)), one must establish the sense of the angle measurement in the principal section plane (sign + when the angle increases in trigonometric sense, and sign – in the inverse trigonometric sign).

The ellipsometric method for determining the angle θ was previously used to estimate the linear birefringence of the stretched polymer foils [9].

The aim of this research was to estimate, by the described ellipsometric method, [9] the linear birefringence of some uniaxial anisotropic crystal samples and to compare the results with those obtained by other optical methods.

3. Materials and Methods

For this study, naturally occurring mineral samples were used, as follows:

- Calcite - samples from Eastern Carpathians (Romania) (Fig. 2a);
- Calcite (var. Iceland spar) - sample from Guizhou (China) - already cut and mounted (Fig. 2b);
- Tourmaline (var. elbaite - "watermelon") - sample from Minas Gerais (Brazil) (Fig. 2c);
- Quartz (var. rock crystal) - samples from Maramureș (Romania) (Fig. 2d).

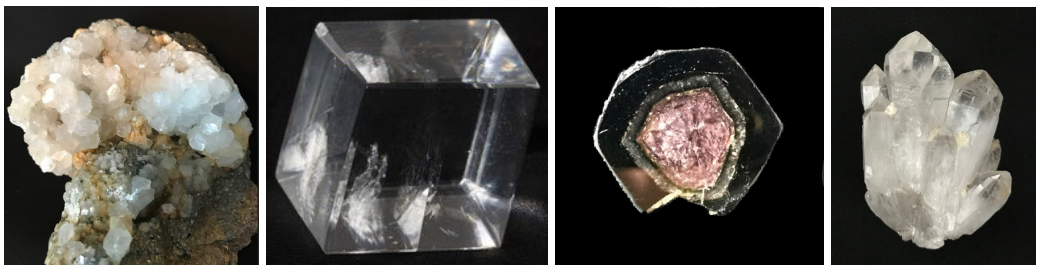

Fig. 2. Samples studied in this paper: Calcite crystals from hydrothermal deposits (Eastern Carpathians, Romania) (a); Calcite (var. Iceland spar) from Guizhou (China) (b); Tourmaline crystal, elbaite "watermelon" variety (cut perpendicularly to the long axis) (Minas Gerais, Brazil) (c); Quartz crystals aggregate (Maramureș, Romania) (d).

To characterize the uniaxial layers from the anisotropy point of view, there are several known methods such as interferometric [10-14], microscopic [15] with compensators [16,17] and so on. The interferometric method based on channeled spectra is also applicable to optical active crystals [18].

Our measurements are based on new ellipsometric method, applied for the first time to polymer foils [9], which uses a very simple device for estimating the angle of rotation of the axes of the polarization ellipse (in the emerging beam) relative to the principal axes of the anisotropic layer. The device used for linear birefringence determination is illustrated schematically in Fig. 3.

As luminous source a sodium source emitting yellow light (λ = 589.3 nm), or an incandescent source with interferential filter for λ = 656.3 nm, corresponding to Hα line of hydrogen were used. The lens L was placed with the principal object focal plane in the discharge zone of the lamp to obtain a parallel beam incident to the polarizer P. The polarizing filter P transforms natural light emitted by lamp S in linearly polarized light. The anisotropic layer, AL, under the study was crossed by light, at normal incidence, parallel to one of the principal axes differing by optical axis.

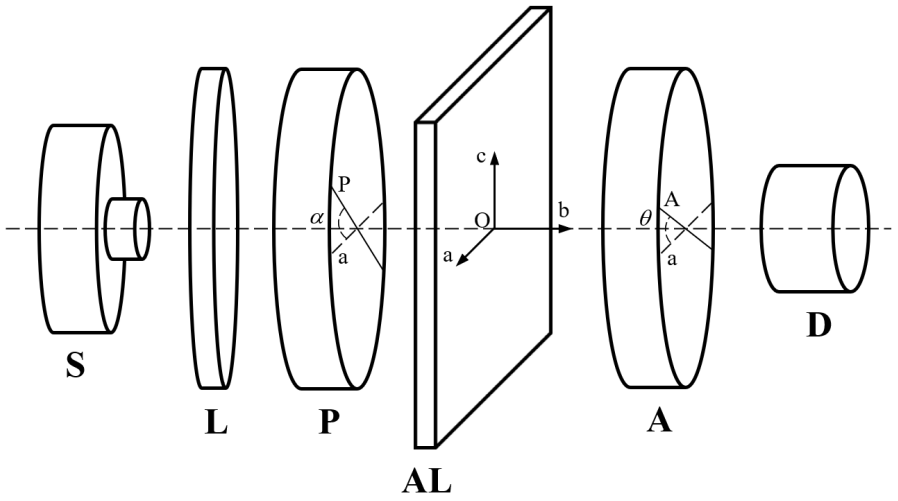

Fig. 3. Device for ellipsometric method: S - monochromatic source; L - collimating lens; P and A - identical polarizing filters; AL - uniaxial layer; D – detector (The polarization directions P and A were respectively indicated on the polarizing filters).

One polarizing filter A, identical with P, is used to analyze the polarization state of emerging light from AL. The polarizing filters have known transmission directions marked on their settings.

The luminous flux was measured by the light detector D (a Si based photodetector with amplifier, manufactured by PHYWE and having the spectral range 300-1150 nm). The intensity of electric current generated by a photodiode (with high sensitivity in the measurement spectral range) is proportional to the emergent luminous flux from the analyzer A.

Initially one obtains the conditions of experiment verifying the coincidence of the propagation direction with one of the principal axes of AL and one notes the azimuth angle between the transmission direction of P and the principal axis of the layer. The analyzer A is then rotated until one obtains the maximum of the indication at the detector (when its transmission direction

becomes parallel to the major axis of the polarization ellipse). One notes the angle θ between the transmission direction of A and the principal axis of AL (Fig. 1).

The rotation angles were measured with digital angle gauge inclinator, rigidly fixed on the settings of the polarizing filters. The accuracy of the angular measurements was 0.01 degree, which determined a relative error in the birefringence estimation up to 3%.

The precision of the estimations is assured by the identification with high precision of principal axes of the polarization ellipse. For this reason, each measurement was made three times and the average values of the measurements were used in estimating the phase difference, $\Delta\psi$.

4. Results and Discussions

For each azimuth angles α (measured relative to the principal axis O*a* of the anisotropic layer in the range 10-80 degrees, by rotating the polarizer P – see Figs. 3 and 4) of the incident linearly polarized monochromatic radiation, the angles θ between the axis of the polarization ellipse and the principal axis O*a* of the crystal were estimated by the maximum and minimum flux density at the detector level (indicated by the maximum and minimum of the intensity of the electric current in the external circuit of the diode, at the rotation of the polarizer A) (see Figs. 3 and 4).

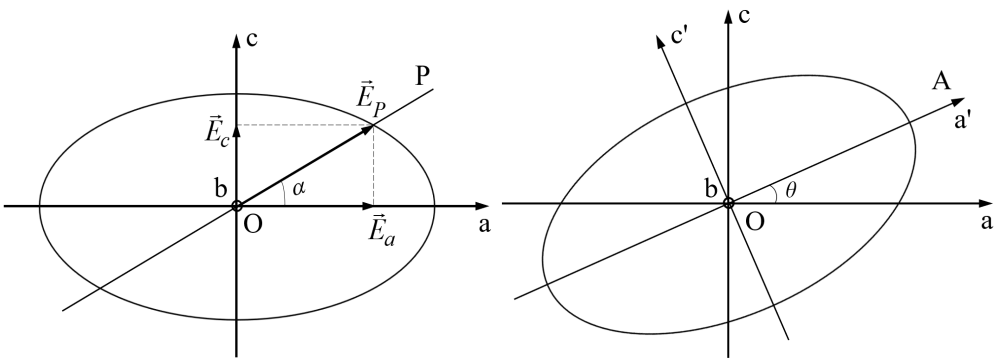

Fig. 4. Polarization ellipse at the exit from AL: (a) for $\Delta\psi = (2m+1)\frac{\pi}{2}$, (b) for $2m\pi < \Delta\psi < (2m+1)\frac{\pi}{2}$ (α – azimuth angle, θ – rotation angle of the polarization ellipse relative to O*a* axis).

In order to avoid the imprecision, bound by the periodicity of the trigonometric function cos$\Delta\psi$ for the inorganic crystals characterized by high

values of Δn, or for which the cutting of very thin layers was not possible, two identical plates cut from the same crystal and having perpendicular optical axes were used, minimizing the phase difference between the ordinary and extraordinary components of light.

The phase difference introduced by the two crystalline layers of thicknesses L_1 and L_2 is expressed by relation:

$$\Delta\psi = \frac{2\pi}{\lambda}\Delta n \Delta L\,, \tag{5}$$

with

$$\Delta L = L_1 - L_2\,. \tag{6}$$

In this way, the imprecision due to the periodicity of the trigonometric function is eliminated [9].

The obtained experimental data show a good linear dependence between the functions tg2θ and tg2α. The phase difference and the linear birefringence were estimated by the slope of the dependence (3).

The results obtained by the average data of the three measurements for each azimuth angle are given in Tables 1-4 and the corresponding Figures 5-8 for the studied inorganic crystals.

Table 1

Linear birefringence of the Carpathian calcite sample
(λ = 589.3 nm, L_1 = 3.2653 mm, L_2 = 3.2650 mm, ΔL = 0.0003 mm)

α (degrees)	θ (degrees)	tg2α	tg2θ	cos$\Delta\psi$	$\Delta\psi$ (degrees)	Δn
10	–8.65	0.36397	–0.31146	–0.85573	–31.15954	–0.17002
20	–17.85	0.83910	–0.71857	–0.85636	–31.08991	–0.16964
30	–27.85	1.73205	–1.46594	–0.84636	–32.18192	–0.17560
40	–39.17	5.67128	–4.84585	–0.85445	–31.30049	–0.17079
50	39.18	–5.67128	4.85441	–0.85596	–31.13363	–0.16988
60	27.99	–1.73205	1.48144	–0.85531	–31.20598	–0.17027
70	17.83	–0.83910	0.71751	–0.85509	–31.22980	–0.17040
80	8.77	–0.36397	0.31607	–0.86839	–29.72725	–0.16221

(The mean value of the birefringence and its standard deviation are $\Delta n = 0.16985 \pm 0.00364$.)

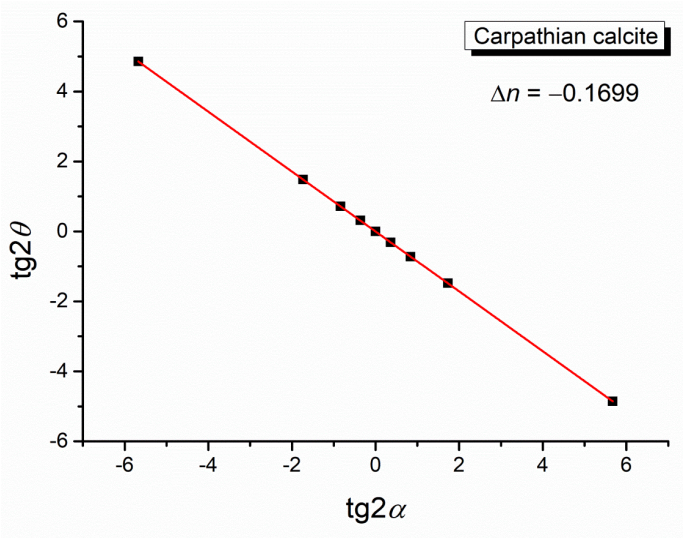

Fig. 5. tg2θ vs. tg2α for Carpathian Calcite sample (λ = 589.3 nm; L_1 = 3.2653 mm, L_2 = 3.2650 mm, ΔL = 0.0003 mm = 0.3×10^{-6} m; Δn = –0.1699 the average value of Calcite linear birefringence).

Calcite is characterized by similar structure as Iceland spar and have appropriate values for the birefringence [7,12,20].

Table 2

Linear birefringence of the Chinese Iceland Spar sample
(λ = 589.3 nm, L_1 = 3.2655 mm, L_2 = 3.2650 mm, ΔL = 0.0005 mm)

α (degrees)	θ (degrees)	tg2α	tg2θ	cos$\Delta\psi$	$\Delta\psi$ (degrees)	Δn
10	–6.27	0.36397	–0.22243	–0.61112	–52.32934	–0.17132
20	–13.53	0.83910	–0.51084	–0.60880	–52.49757	–0.17187
30	–23.26	1.73205	–1.05452	–0.60883	–52.49521	–0.17186
40	–36.90	5.67128	–3.44202	–0.60692	–52.63278	–0.17231
50	36.87	–5.67128	3.42862	–0.60456	–52.80293	–0.17287
60	23.30	–1.73205	1.05747	–0.61053	–52.37210	–0.17146
70	13.48	–0.83910	0.50865	–0.60619	–52.68582	–0.17249
80	6.25	–0.36397	0.22169	–0.60909	–52.47636	–0.17180

(The mean value of the birefringence and its standard deviation are Δn = 0.17200 ± 0.00052.)

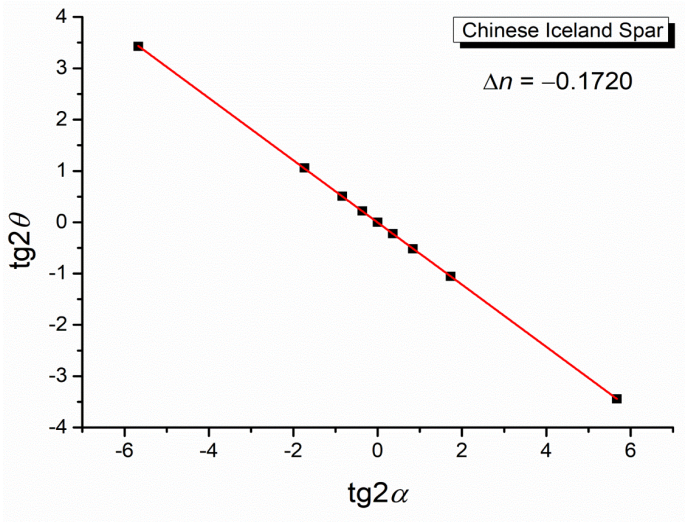

Fig. 6. tg2θ vs. tg2α for the Chinese Iceland Spar sample (λ = 589.3 nm; L_1 = 3.2655 mm, L_2 = 3.2650 mm, ΔL = 0.0005 mm = 0.5×10^{-6} m; Δn = –0.1720 the mean value of Iceland Spar linear birefringence).

Similar data with those shown in Table 2 and Fig. 6 are listed in the consulted literature [7,8,19,20].

Table 3

Linear birefringence of the Brazilian Tourmaline sample
(λ = 656.3 nm, L_1 = 3.2655 mm, L_2 = 3.2255 mm, ΔL = 0.040 mm)

α (degrees)	θ (degrees)	tg2α	tg2θ	cos$\Delta\psi$	$\Delta\psi$ (degrees)	Δn
10	–7.20	0.36397	–0.25676	–0.70544	–45.13468	–0.00206
20	–15.23	0.83910	–0.58811	–0.70088	–45.50220	–0.00207
30	–25.31	1.73205	–1.21829	–0.70338	–45.30115	–0.00206
40	–37.99	5.67128	–4.00483	–0.70616	–45.07668	–0.00205
50	37.98	–5.67128	3.99889	–0.70511	–45.16137	–0.00206
60	25.33	–1.73205	1.22002	–0.70438	–45.22059	–0.00206
70	15.25	–0.83910	0.58905	–0.70200	–45.41214	–0.00207
80	7.21	–0.36397	0.25713	–0.70646	–45.05244	–0.00205

(The mean value of the birefringence and its standard deviation are Δn = 0.00206 ± 0.00001.)

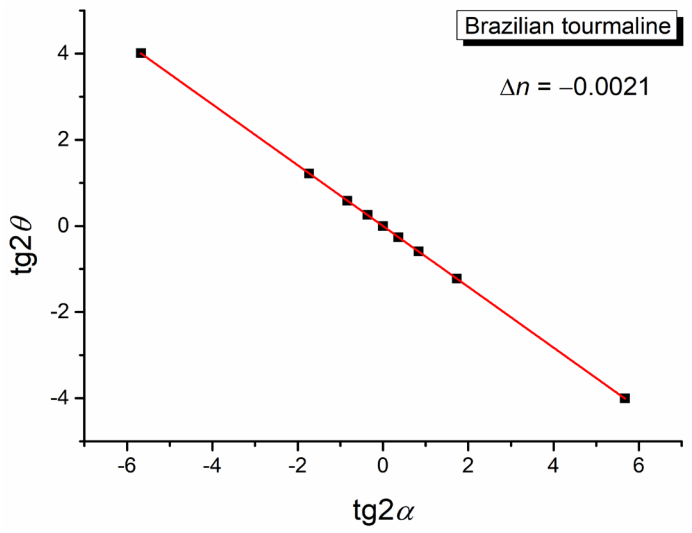

Fig. 7. tg2θ vs. tg2α for the Brazilian Tourmaline sample (λ = 656.3 nm; L_1 = 3.2655 mm, L_2 = 3.2255 mm, ΔL = 0.040 mm = 4×10^{-5} m; Δn = –0.0021 the mean value of Tourmaline linear birefringence).

For tourmaline (see Table 3 and Fig. 7), a birefringence in accordance with literature was found [7,12,19, 20].

Table 4

Linear birefringence of the Carpathian Quartz sample
(λ = 589.3 nm, L_1 = 2.1636 mm, L_2 = 2.1516 mm, ΔL = 0.012 mm)

α (degrees)	θ (degrees)	tg2α	tg2θ	cos$\Delta\psi$	$\Delta\psi$ (degrees)	Δn
10	4.26	0.36397	0.14981	0.41160	65.69462	0.00896
20	9.43	0.83910	0.34160	0.40710	65.97703	0.00900
30	17.50	1.73205	0.70021	0.40427	66.15482	0.00902
40	32.80	5.67128	2.20449	0.38871	67.12567	0.00916
50	–32.79	–5.67128	–2.20244	0.38835	67.14815	0.00916
60	–17.56	–1.73205	–0.70333	0.40607	66.04193	0.00901
70	–9.45	–0.83910	–0.34238	0.40803	65.91871	0.00899
80	–4.20	–0.36397	–0.14767	0.40572	66.06373	0.00901

(The mean value of the birefringence and its standard deviation are Δn = 0.00904 ± 0.00008.)

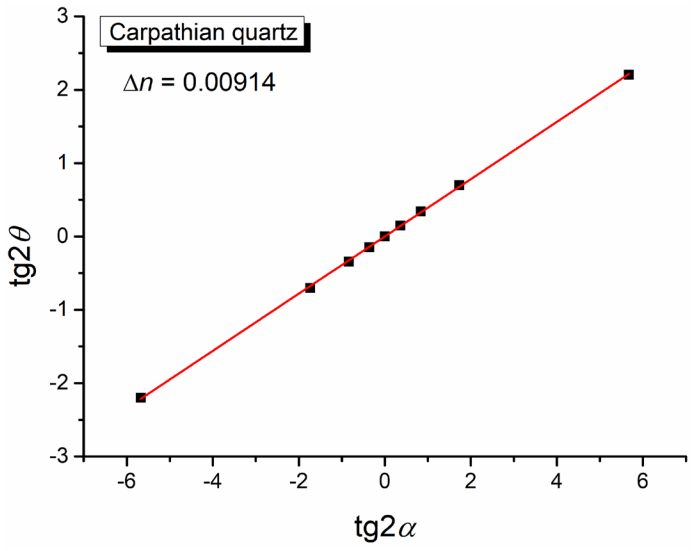

Fig. 8. tg2θ vs. tg2α for the Carpathian Quartz sample (λ = 589.3 nm; L_1 = 2.1636 mm, L_2 = 2.1516 mm, ΔL = 0.012 mm = 1.2×10^{-5} m; Δn = 0.00914 the mean value of Quartz linear birefringence).

The data in Table 4 and Fig. 8 are in accordance with the values obtained for quartz birefringence by other methods [6,7,15,20].

The obtained data reported here demonstrate the applicability of the proposed ellipsometric method in estimating the linear birefringence of uniaxial thin layers. For transparent materials with high values of linear birefringence or with large values of their thickness, one can use two layers cut from the same material and having appropriate thicknesses.

5. Conclusions

The ellipsometric method described in this paper is applicable to the transparent crystalline layers with the condition that the phase difference is not higher than 180 degrees. For crystals with high linear birefringence, or with high thicknesses, one can use two identical crystalline layers having perpendicular principal axes with different values of the principal refractive indices to minimize the phase difference between the ordinary and extraordinary components of the monochromatic radiation.

The method can be applied to characterize the anisotropy of both the transparent inorganic crystals and for other transparent anisotropic layers such as polymer foils or liquid crystalline layers.

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