The application of NLC for detecting solid crystals surface homogeneity

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Abstract. The goal of the paper is to demonstrate wide fields of new polarizing microscope application based on nematic liquid crystals (LCs) for detecting different solid crystals surface inhomogeneities. Optical polarizing microscope (OPM) based on LCs makes possible to observe the invisible physical fields’ distribution on the objects’ surfaces. The OPM novelty consists in LC spatial light modulator (SLM) introduction in optical scheme to detect local deformations in real time. LC SLM applied as recording media has to be in direct contact with the surface under investigation. It gives the possibility to detect the invisible physical fields on the surface: intermolecular interactions, electrical, magnetic fields, etc.

The theory of LC layer deformations was developed to find the relation between real size of structural defect D and the size of its image D’ visualized with NLC layer. OPM method was used for detecting different aspects of surface inhomogeneities for different types of solid crystals. The new results were obtained for twinning boundaries in piezo quartz resonator having industrial application. LC SLM demonstrates non destructive method and better accuracy in comparison with etching, and the simplicity in comparison with x-radiation detecting. LC SLM may be combined with interference, phase-contrast and even near-field microscopes.

1. Introduction
The optical microscope operations are limited with illumination distribution detecting on the object surface in reflective regime and absorption object parameters detecting in transparent regime. The functions are increased by observation the objects in polarizing light. The inside tension and optical activity in transparent materials may be additionally observed. Optical polarizing microscopy is very powerful tool for investigation in many fields of science and technology. But it is helpless in detecting invisible physical fields’ distribution on the object surface. The combination of optical polarizing microscope (OPM) with liquid crystal spatial light modulator (LC SLM) in contact with objects’ surface makes possible to increase the microscopes’ power and functions. The propagation of LC SLM application to other microscopes types with super high resolution is also discussed.

2. The optical polarizing microscope scheme and principle of operation
The novelty of OPM consists in LC SLM introduction in optical scheme to observe LC layer local deformations in real time [1]. LC SLM applied as recording media has to be in contact with the surface under investigation (figure 1). In this case LC SLM gives the possibility to detect the invisible physical fields on the object’s surface: intermolecular interactions, electrical, magnetic fields, etc.
As it is shown on figure 1 B, C and D the possibility arises to visualize microrelief and structural defects. It also opens the horizons for bio analytic applications using LC flow in micro fluidic channels for detecting macromolecules local defects.

Nematic LCs are more sensitive to physical fields detecting in comparison with sensitivity of cholesteric and smectic LCs having super molecular structure. The principle of operation is shown on figure 2. In polarized light we observe not the invisible structural defect D, but only the deformations in LC layer D′ induced by defect (left) (figure 2). In non polarized light we observe the invisible structural defect D through transparent LC layer and see nothing (right). It is the reason to study the deformations of NLC layer, induced by initial fields.

### 3. The theory of NLC layer deformations

The light intensity over NLC layer $I(x, y)$ modulated by deformed NLC structure is described by equation:

$$I(x, y) = I_0 \sin^2 \left( \frac{\delta(x, y)}{2} \right)$$

Figure 1. Left – optical scheme: 1-light source, 2-collector, 3-aperture, 4-mirror, 5-polarizer, 6-condenser lens, 7-object, 8-LC SLM, 9-microscope lens, 10-analyzer, 11-prism-cube, 12-ocular 1, 13-CCD-sensor, 14-computer, 15-prism, 16-aperture, 17-ocular. Right – B, C, D – defects types. B – Microrelief defect; C – structural inhomogeneities; D – the distribution of non uniform electrical or magnetic field. D - Defect’s size. D’ - defect’s image size in NLC.
The phase delay $\delta(x, y)$ caused by the NLC birefringence:

$$\delta(x, y) = \frac{2\pi}{\lambda} \left[ -n_0 \cdot H + \int_0^h n(x, y, z) dz \right]$$

(2)

$H$ is the thickness of NLC; $n(x, y)$ is film reflective index of deformed zone; $n_0$ is non-deformed layer the refractive index. If the orientation has no twist deformation then only orientation bending occurs, hence:

$$n(x,y,z) = \left[ n_e^2 \sin^2 \varphi(x,y,z) + n_0^2 \cos^2 \varphi(x, y, z) \right]^{-1/2}$$

(3)

$\varphi(x,y,z)$ is the deflection angle of the long axis of the molecules; $n_o$, $n_e$ are the refractive indices of NLC layer for ordinary and extraordinary polarization. The high value of NLC optical anisotropy permits to use very thin layers to obtain sufficient value of phase delay.

The theory of LC layer deformations near the structural defects or obtained by magnetic or electrical fields is developed and described in [2, 3].

The most interesting and practically important case is the detecting structural inhomogeneities in different materials (for example, on the solid crystals surface), shown on fig. 1C. The theory gives the distribution of LC molecules orientation near the structural defect in the case of strong anchoring conditions (figure 3).

The NLC molecules orientation induced by structural inhomogeneities is described by equation:

$$\pi \left( \frac{\varphi(x, y) - \varphi_o}{\varphi_y - \varphi_o} \right) = \arctg \left( \frac{sh(D - X) \cdot ctg \left( \frac{Y}{2} \right)}{ch(D - X) + 1} \right) + \arctg \left( \frac{sh(D + X) \cdot ctg \left( \frac{Y}{2} \right)}{ch(D - X) + 1} \right)$$

(4)

$$X = \frac{x}{H}, \quad Y = \frac{y}{H}, \quad D = \frac{n l}{H}$$
The discussed theory was used for explanation the results of experimental examination the structural defects on the surface of solid crystals and other materials. It gives possibilities to calculate parameters of external fields.

4. The application of OPM with LC SLM to detecting solid crystals surface homogeneity
OPM with LC SLM may be effectively used for detecting the NLC orientation on the surface of solid crystals, twinning boundaries detecting, phase transition application for enlarging the contrast of twinning boundary image, structural blocks and defects detecting in crystals and semiconductors, crystalline phase detecting in vitreous materials and domains detecting in ferroelectric crystals.

Figure 3. Solitary structural defect and local orientation of LC molecules induced by it.

Figure 4. Plans of symmetry detecting in guanidine aluminum sulfate gexagidrate with application of E-field to tolans mixture (left) and the scheme of molecules orientation on mono crystalline surface (right).
4.1. NLC orientation on the surface of solid crystals

At the beginning of LC materials study solid crystals were used by Ch. Mogen (1913), F. Grandjean (1916), P. Gobert (1938) and other scientists to study the behavior of LCs on solid crystals [4]. The goal was to achieve the stable and repeatable orientation of LC layers. It was demonstrated that easy LC molecules orientation has the relation with properties of crystal symmetry. At 1916 Grandjean showed that LC molecules on the surface of solid crystals are spontaneously oriented along crystallographic directions. At 1970 P.G. de Gennes established that the number of easy orientation directions is related to the symmetry of the substrate. At 1980 N. Tikhomirova & A. Ginsberg investigated surface symmetry properties of crystals NaCl, KBr, LiF, GASH, etc. [5]. At now days LC mostly used to detect the planes of crystal symmetry and solid crystals structural defects (figures 4, 5a, b). LCs gives the possibility to detect the ideal symmetry changes in crystals induced by inclusions (figures 5c and d).

![Image](a)

![Image](b)

![Image](c)

![Image](d)

**Figure 5.** Planes of symmetry on the surface of crystals KCl (a) and alums (b) visualized with NLC (MBBA+EBBA). Inclusions disturb the ideal symmetry of alums (c, d). Magnification 50x.

4.2. Twinning boundary detecting

The optical application of crystals with twinning defects is forbidden almost in all cases. Twinning defects usually are visualized by etching. Unfortunately it dramatically changes the crystal surface quality. It is possible to submit etching by non destructive technique based on LCs. The example of LCs application to twinning boundary detecting in Island spar is shown on figure 6.

In comparison with etching the application of LCs on the Island spar surface doesn’t change the quality of polished crystal surface. In addition NLCs visualize very small size structural defects that are not detected by etching (figure 6, right).
4.3. Phase transition application for enlarging the contrast of twinning boundary image

The image of twinning boundaries, visualized in Island spar at room temperature, has a low contrast (figure 7a). Contrast may be increased by heating LCs up to the cleaning temperature to isotropic phase at one part of the boundary with smaller anchoring energy (figure 7b).

![Figure 7](image)

**Figure 7.** Phase transition application in LCs for enlarging visual contrast of twinning boundary in Island spar: MBBA:EBBA, $t_a=20^\circ C$; $t_b=53.4^\circ C$; 50x [7].

Structural defects in crystalline quartz resonators also may have natural reason or appear in the fabrication process, changing frequency parameters of resonator (figure 8a, b). We suggested the use of NLCs in industry for detecting structural defects with more accuracy than etching (figure 8c) and much easy than X-ray technique [8].

![Figure 8](image)

**Figure 8.** LCs application to structural defects detecting in industrial crystalline quartz resonator, MBBA:EBBA, 50x.
4.4. Structural blocks detecting in Mica
NLCs may efficiently visualize structural inhomogeneities in mica that characterize the insulator properties of the material (figure 9, left and right).

![Figure 9](image)

**Figure 9.** Blocks in \{KAl2[AlSi3O10](OH,F)2\} visualized with NLCs. MBBA, \(t = 20^\circ\text{C}\). 100x.

4.5. Structural defects in laser crystals detecting
Non linear laser crystals optical characteristics depend on their structural uniformity. Structural defects dramatically decrease the efficiency of frequency transformation. It was demonstrated that NLC method gives more information and higher spatial resolution on structural uniformity in comparison with second harmonic generation. The efficiency of NLC method was confirmed by series of experiments with KTP laser crystals [9]. The example of detecting structural defects on KTP crystal surface is shown on figure 10.

![Figure 10](image)

**Figure 10.** Structural defects in KTP laser crystal decreasing the efficiency of frequency transformation visualized with MBBA:EBBA; 100x.

4.6. The crystalline phase detecting in vitreous materials
The invisible solid crystals appear in vitreous materials under the influence of UV radiation and heating. It is possible to visualize their appearance using NLCs (figure 11) [10].

4.7. Structural defects in semiconductors
NLCs may be used for detecting lattice defects and dislocations accumulation on different types of Si-surface (figures 12 and 13).
Figure 11. The distribution of light intensity in laser beam section (a), the object fragment subjected to laser radiation (b). Solid crystals in vitreous materials visualized with NLCs; 1300x (c). MBBA:EBBA [10].

Figure 12. Crystalline lattice defects and dislocations accumulation visualized on Si surface (left). Structural defects on the surface of α-Si:H layer (right). 500x. MBBA:EBBA. $t = 20^\circ$C.
Figure 13. Dislocations accumulation visualized on α-Si/Fe layer before (left) and after (right) annealing (t=800°C, 2 hours). MBBA:EBBA. 200x. t = 20°C [11].

4.8. Domains detecting in ferroelectric crystals TGS and X-radiation defects
Longitudinal domains are not visualized on the surface of ferroelectric crystal in optical microscope. The application of NLCs gives the possibility to visualize in polarizing microscope not only the longitudinal domains but also domain wall movement with change of applied electric field. X-radiation defects were easily visualized on the surface of ferroelectric crystal TGS (Cu-radiation; D = 0.2 mrad); TGS thickness – 1 mm (figure 14) [12].

The idea to make visible the distribution of physical fields (defects) on the material surface may be extended on other types of microscopes, for example near field microscope. Such idea may be used in visualizing defects at nanoscale level. The first application of LCs in super resolution microscopy was described in paper [13].

5. Summary
OPM with LC SLM opens wide possibilities in visualizing structural inhomogeneities of solid crystals in material science and high technologies. OPM with LC SLM application may be spread on interference, phase-contrast, confocal and near-field microscopy and may be considered as new

Figure 14. Longitudinal domains and X-radiation defects visualized on the surface of ferroelectric crystal TGS; MBBA:EBBA, 50x.
universal instrument to detect invisible physical fields on objects’ surface with high sensitivity and spatial resolution

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