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CARACTERISATION OF THE DISLOCATION PEFECTION OF A-LIIO$_3$ SINGLE CRYSTALS BEFORE OPTICAL-MECHANICAL TREATMENT

LIUDMIL LYUTOV

Department of General and Inorganic Chemistry, Faculty of Chemistry,
St. Kliment Ohridski University of Sofia

Людмил Лютов. ОХАРАКТЕРИЗИРАНЕ НА ДИСЛОКАЦИОННОТО СЪВЪРШЕНСТВО НА МОНОКРИСТАЛИ ОТ α-LiIO$_3$ ПРЕДИ ОПТИКО-МЕХАНИЧНА ОБРАБОТКА

Относително бездефектни области от кристалите на α-LiIO$_3$ се използват за изработване на качествени устройства. Проявяването и наблюдението на дислокациите е неизбежна стъпка при охарактеризирането на кристалите. Общо-приетото окачествяване на изкуствено създадена стена [0001] води до загуба на време и скъпи обработки. Предлаганото в тази работа охарактеризиране на призматичната стена преди обработките повишава ефективността на изработване на устройства на база на този материал.

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A relatively defect-free region of the crystal α-LiIO$_3$ should be used for the preparation of high-quality devices. The revealing of dislocations and their observation are necessary steps in the crystals characterization. The widely accepted characterization of artificially created facet [0001] brings about lose of time and expensive processing. The characterization of prismatic facet, proposed in the present work, can be executed prior to the crystal treatment thus making more efficient the technologies for production of active optical elements.

Keywords: α-LiIO$_3$, crystal, dislocation density, selective dissolution
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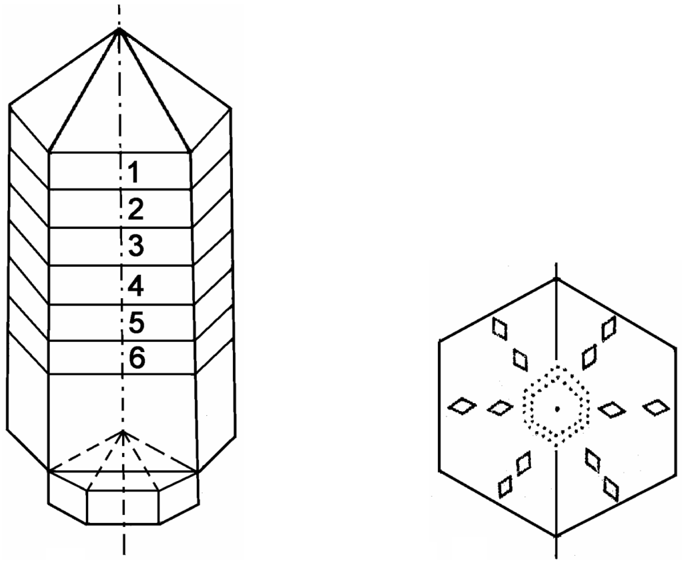
For contact: Lyudmil Lyutov, Department of General and Inorganic Chemistry, Faculty of Chemistry, St. Kliment Ohridski University of Sofia, 1 J. Bourchier Blvd., 1126 Sofia, Bulgaria, tel.:00359-02-8161378, E-mail: ll@lcpe.uni-sofia.bg

1. INTRODUCTION

Crystals of α-$LiIO_3$ find applications in laser optics as generator of a second harmonic frequency and as deflector. The inconvenience that this material is soluble in water is compensated by numerous advantages, such as a high threshold of destruction, relatively high non-linear coefficients, and a formation of large regions with high homogeneity of the refractive index (*n*). The last property allows the construction of devices with large usable apertures (up to several centimetres), which is difficult to attain with the crystals grown from melt.

For the preparation of high-quality devices, relatively defect-free region of the crystal should be utilized. The importance of dislocation density and some others defects for the applicability of the crystals is discussed in [1, 2, 3], where the methods of revealing the dislocations and of their observation [1, 3] are described as necessary steps in the characterization of the crystals. However, the information concerning $LiIO_3$, critically reviewed in [3], is either scarce [4] or contradictory, or inconvenient and inappropriate [5–7].

Summarising the facts, the above-mentioned methods of characterization follow the unique procedure: (i) cutting of the crystal perpendicular to the Z-axis to obtain basic pinacoids [0001] $[0001^{-}]$ (Fig. 1*a*); (ii) selective dissolution of the basic pinacoids, and (iii) revealing of the dislocations (Fig. 1*b*).



a *b*

Fig. 1. (*a*) Scheme of orientation and cutting of crystal of α-$LiIO_3$;
(*b*) Scheme of etching of the basic pinacoids

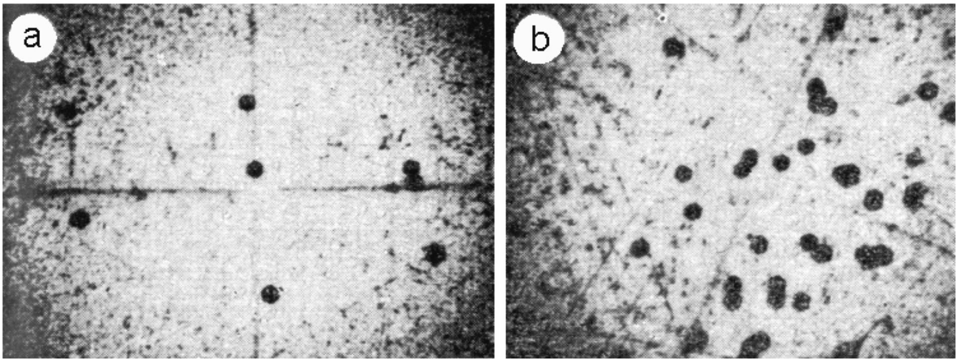


Fig. 2. Dislocations in a high quality (a) and in a low-quality (b) specimen of α-$LiIO_3$

Fig. 2 shows the actual distribution of dislocations in a high quality (a) and in a low-quality (b) specimen. In addition to the critical comments reported in [3] on the ambiguity and irreproducibility of the obtained results, the procedures cited have an important drawback, i.e., the characterization of separate regions is performed at a stage when the crystal treatment is almost half-done (orientation, cutting, grinding, polishing). Therefore, we report here a non-destructive procedure of crystal characterization prior the crucial steps of mastering.

2. EXPERIMENTAL PROCEDURE

The effects of selective dissolution of the $[101^{-}0]$ facet were studied using the solvents described in literature [3, 5–7] and ours, given at table 1.

Table 1. Solvents used in the selective dissolution of facets

HNO_3-H_2O	30-65% (mass)
CH_3COOH-H_2O	50-80% (mass)
HNO_3-CH_3COOH-H_2O	(30%, 30%, 40%, mass)
$LiIO_3$-HIO_3-H_2O	In concentrations close to the saturated solution

The dissolution patterns were observed by means of an Epival-Interphako microscope (Carl Zeiss – Jena) using the bright field, incident light, and interference contrast methods.

3. RESULTS AND DISCUSSION

We succeeded to establish an appropriate concentration and to prepare the respective solution by taking advantage of a peculiarity of α-LiIO$_3$. The latter has a low and negative temperature coefficient of solubility. Due to this fact a 2% HIO$_3$ aqueous solution was saturated with LiIO$_3$ at 42°C until the dissolution of added solid phase ceased. As far as before the last added amount of solid phase dissolves, its crystallites became rounded. The saturation was finished, if the crystallites of last-added amount solid phase remained unchanged and well faceted. The end of dissolution could accelerate the process accelerated by evaporation. The saturated solution was separated from the solid phase and stored in a closed vessel.

The solvents described in the experimental procedure did not lead to the expected results except the selective solvent (LiIO$_3$-HIO$_3$-H$_2$O), proposed by us. The results obtained with this selective solvent are presented in Fig. 3 as the respective dissolution patterns. The patterns are observed as pits on the [101¯0] facet, having the shape of tetragonal pyramids. The basis of a pyramid is on the [101¯0] facet with its apex directed toward the inner portion of crystal. The basic edge pairs of the pyramid are parallel and are perpendicular to the Z-axis.

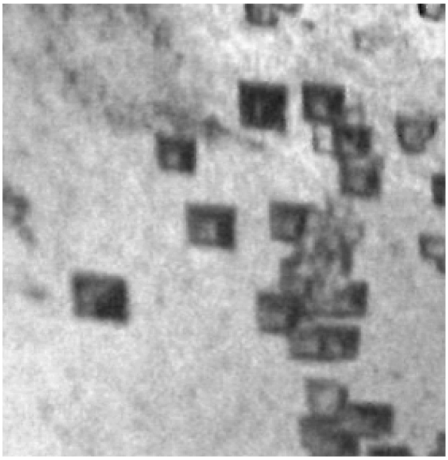


Fig. 3. Etching pits on the [101^{-}0] facet (incident light, bright field, interference contrast)

A reasonable explanation of the good results obtained with our solvent (LiIO$_3$-HIO$_3$-H$_2$O) could be the fact that the defect regions in the crystal have a higher thermodynamic potential than those with a lower number of defects (or defect-free regions). When such a crystal is brought into a contact with a solvent of appropriate concentration, the solvent turns out to be unsaturated with respect to the defects and saturated for the defect-free regions. Therefore, dissolution of the defect regions will take place, with the others regions remaining unaffected.

We recommend the following conditions of selective dissolution of the $[101^{-}0]$ facet: temperature of 20ºC, duration of 5 to 48 h, and a ratio of the selectively dissolved surface (S_{etch}) to the solvent volume (V_{ss}) $S_{etch} / V_{ss} = 0.4\ cm^{-1}$.

It is possible to change these conditions in order to induce changes in the selective dissolution.

The actual distribution of dislocation density (Fig. 4*a*) corresponds quite well to the theoretical expectations [1,3], with this density being the highest at the basis and decreasing toward the crystal apex (Fig. 4*b*).

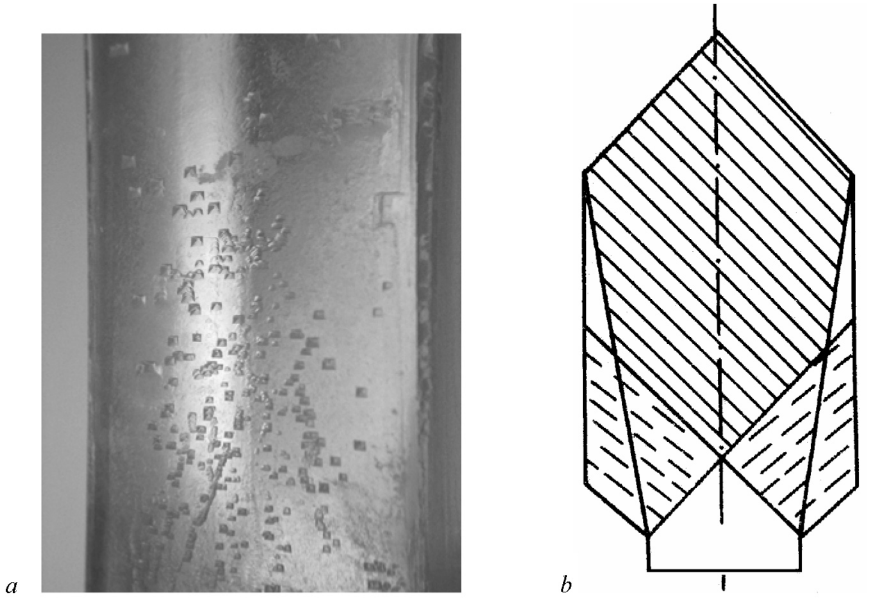


Fig. 4. (*a*) Real distribution of the dislocation density on the [101—0] facet; (*b*) Theoretical distribution of the dislocation density

4. CONCLUSIONS

The fair agreement between the theoretically predicted and the experimentally observed distribution of defects proves that the observed dissolution patterns are actually dislocation sites. The distribution of defects on different crystal specimens can be used for the characterization of perfection in the crystal regions. The distribution pattern of defects in crystal can be used in practice for selection of the most suitable regions for appropriate purpose. Moreover, this characterization can be executed prior to the crystal treatment, thus making more convenient the technologies for production of acousto-optical and other elements. By that means utilisation of unprocessed crystal became completely, efficient and rationally.

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