Optical investigation of nanophotonic lithium niobate-based optical waveguide

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Optical investigation of nanophotonic lithium niobate-based optical waveguide

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Abstract Lithium niobate (LiNbO₃) nanophotonics are prepared on quartz substrate by sol–gel method. They have been deposited with different molarity concentrations and annealed at 500 °C. These samples are characterized and analyzed by scanning electron microscope, atomic force microscopy, X-ray diffraction and ultraviolet–visible. The measured results show an importance of increasing molarity that indicates the structure starts to crystallize to become more regular. The estimated lattice constants, energy gaps and refractive index give good accordance with experimental results. Also, the calculated refractive index and optical dielectric constant are in agreement with experimental data.

1 Introduction

Lithium niobate (LiNbO₃) is a very important optical material which is widely used by the photonics industry, due to its excellent electro/acousto-optical properties [1, 2]. LiNbO₃ single crystal is an excellent material for nonlinear optics and electro-optics applications. It is one of the key materials for optic-based technologies because of its large second-order nonlinearities [3]. LiNbO₃ is a distinguished ferroelectric material due to its excellent piezoelectrical, electro-optical, pyroelectrical and photorefractive properties [4–6]. From experimental estimation, a direct and indirect energy band gap of LiNbO₃ is reported to be in the range of 3.5–4.7 eV, depending on LiNb concentrations. These changes are attributed to several parameters such as grain size, composition and defects [7–9]. There are different approaches known to synthesize undoped LiNbO₃ nanocrystals, such as sol–gel [10, 11], soft-chemistry [12], pulsed laser deposition [13, 14], RF sputtering [15] and hydrothermal methods [16, 17]. LiNbO₃ is a widely used polar material for photonic applications [18–20]. It is employed in nonlinear optics for frequency conversion, telecommunication for electro-optic modulation [21, 22] and fabrication of optical waveguide devices [23–25]. Waveguide structures are essential for many integrated-optic devices.

Recently, Miccio et al. [26] have adopted an innovative dielectrophoretic (DEP) approach based on electrode-free DEP for investigating smart but simple strategies for immobilization and orientation of bacteria. Escherichia coli DH5 alpha strain has been selected as subject of the study. The light-induced DEP is achieved through ferroelectric iron-doped lithium niobate crystals used as substrates. Due to the photorefractive (PR) property of such material, suitable light patterns allow writing spatial charges distribution inside its volume and the resultant electric fields are able to immobilize E. coli on the surface. The investigation presented here could open the way for detection or patterning applications based on a new driving mechanism.
Future perspectives also include the possibility to actively switch by light the DEP forces, through the writing/erasing characteristic of PR fields, to dynamically control biofilm spatial structure and arrangement, while Gong et al. [27] have realized electric-field-controlled two-dimensional Raman–Nath diffraction using a photorefractive diffraction grating. They have presented results for the Raman–Nath diffraction from the \( g_{44} \) grating, in which the externally applied field is perpendicular to both the grating vector and the wave vector of the incident beam. Two pairs of coherent beams were used to record the grating for two-dimensional Raman–Nath diffraction. The wave vector and the polarization of one pair lay in the \((x, z)\) plane, and those of the other pair lay in the \((y, z)\) plane. Argiolas et al. [28] have reported on the characterization of periodically poled lithium niobate structures grown by the off-center Czochralski technique with periods ranging between 2 and 10 \( \mu \)m. The domains distribution along the crystal was inspected by a profilometer scan after etching the structures and carrying a suitable data processing. The second-harmonic generation efficiency was predicted by numerically integrating the governing equations through to a recently proposed nonlinear bidirectional beam propagation method. The numerical analysis pointed out the feasibility of the backward second-harmonic generation in the sample with the shortest domain period. The predicted second-harmonic generation efficiency was finally corrected considering the phase shifts induced in the second-harmonic wave by the presence of different sized domains.

This work reports the preparation of \( \text{LiNbO}_3 \) nanophotonics by utilizing the spin-coating technique. The characterization and analysis have been elaborated as a function of molarity concentration. The refractive index is the main work on optical waveguides for \( \text{LiNbO}_3 \) because refraction coefficient between the base and deposited structures will ensure access to total internal reflection that gives us better realization of optical waveguide. The refractive index is measured and calculated to fit best application for optical waveguide.

### 2 Experimental methods

The preparation procedure of \( \text{LiNbO}_3 \) nanophotonics is shown in Fig. 1. \( \text{Nb}_2\text{O}_5 \) (ultra-purity, 99.99 %) and oxalic acid (A.P.) are used without further purification. The solution is prepared by mixing \( \text{Li}_2\text{CO}_3 \), \( \text{Nb}_2\text{O}_5 \), citric acid (CA) and ethylene glycol (EG). The molar ratio between \( \text{Li}_2\text{CO}_3 \) and \( \text{Nb}_2\text{O}_5 \) was 1:1 in order to maximize the formation of \( \text{LiNbO}_3 \) stoichiometry phase as follows (\( \text{Li}_2\text{CO}_3 = 0.925, 1.85, 2.775 \) and 3.7 g, \( \text{Nb}_2\text{O}_5 = 3.325, 6.650, 9.975 \) and 13.30 g, CA = 2.625, 5.25, 7.875 and 10.5 g and EG = 20 mm\(^3\)). Firstly, the \( \text{Li}_2\text{CO}_3 \), \( \text{Nb}_2\text{O}_5 \) and citric acid were dissolved in ethylene glycol with heating and stirring at 90 °C for 8 h, and then mixed all together with continue heating and stirring at 90 °C for 8 h. To obtain homogeneous and crack-free films of \( \text{LiNbO}_3 \), the precursor was deposited by spin-coating technique on quartz substrates at a spinning speed of 3000 rpm for 30 s. Seven layers were prepared, and the film was dried at 120 °C for 10 min and was annealed (calcined) at 500 °C for 2 h in static air and oxygen atmosphere to remove the organics. The structural evolution of the as-prepared nanophotonics was examined using high-resolution X-ray diffraction (HR-XRD), (X’Pert Pro MRD PW3040 system diffractometer, PANalytical Company, Netherlands) equipped with Cu-K\( \alpha \) radiation of wavelength \( \lambda = 0.15418 \) nm at 40 kV and 30 mA. The thickness of the annealed samples was studied using scanning optical reflectometer model (Filmetrics F20, China). The scanning electron microscopy (SEM) (JOEL JSM-6460LV, Oxford instruments Analytical Ltd., Japan) was used to investigate the surface morphology of \( \text{LiNbO}_3 \), and atomic force microscopy (AFM) (SPM-9600, Scanning Probe Microscope, Shimadzu, Japan) was utilized for investigating the roughness of \( \text{LiNbO}_3 \). The optical
properties were investigated using the double-beam ultraviolet–visible (UV–Vis) spectrophotometer (Shimadzu UV–Vis 1800, Japan).

3 Results and discussion

3.1 Structural properties

The XRD of LiNbO₃ nanophotonics deposited on quartz substrates grown by sol–gel method is shown in Fig. 2. It is observed from Fig. 2 that the peaks at 2θ = 23.634, 32.637, 48.355, 53.106 and 55.879 correspond to (012), (104), (110), (024), (116) and (122) planes. All the peaks could be indexed to the hexagonal structure with lattice parameters a = b = 5.1566, c = 13.858, which were very close to the reported data in Simoes et al. [5]. The crystalline structure becomes more crystalline with increasing molarity concentration as shown in Fig. 2. The measured structural properties of LiNbO₃ nanophotonics are listed in Table 1. Crystallite size (D) was calculated using Scherrer’s formula [29].

\[ D = \frac{K \lambda}{\beta \cos \theta} \]  

(1)

where \( K \) is a constant taken to be 0.94, \( \lambda \) is the wavelength of X-ray used (\( \lambda = 1.54 \) Å), \( \beta \) is the full width at half maximum of XRD pattern, and \( \theta \) is Bragg’s angle.

In addition, the dislocation density (δ) and strain (ε) of LiNbO₃ nanophotonics were determined using the following relations [29].

\[ \delta = \frac{1}{D^2} \]  

(2)

\[ \varepsilon = \frac{\beta}{4 \tan \theta} \]  

(3)

The interplanar distance (d) is calculated for all set of LiNbO₃ nanophotonics using Bragg’s formula [29].

\[ d = \frac{n \lambda}{2 \sin \theta} \]  

(4)

where \( n \) is positive integer number, and \( d \) is given in Table 1.

The nanophotonics have polycrystalline structure, where two phases of LiNbO₃ could be recognized (Δ and δ phases). The Δ phase is found to be a preferred phase. However, the performed phase has (012) orientation. The XRD clearly indicates the presence of a small amount of secondary Li-deficient phase (LiNb₃O₈), at all molarity concentrations. This phase is originated from an interface reaction between the oxygen and LiNbO₃, that could be detected by XRD at peaks 2θ = 24.407, 30.262 and 35.981 corresponding to (400), (410) and (212) planes. There is an impurity like Nb₂O₅, where detected as shown at peaks 2θ = 24.433 and 31.623 corresponding to (−105) and (014) planes. The measured lattice constants have showed good agreement with experimental values (reported values) as given in Table 1.

3.2 Morphological studies

It is a very interesting parameter for integrated-optic and optoelectronic applications. Figure 3 shows SEM images (5 × 5 µm) of LiNbO₃ nanophotonics. Since the density
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a Ref. [47] exp.; b Ref. [30] exp
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of nucleation for the LiNbO$_3$ nanophotonics was not uninformed, it is noticed that a high proportion of pores and voids appear, as a result of impurities such as Nb$_2$O$_5$, and LiNbO$_3$ nanophotonics grow up smoothly at higher molarity concentration, which led to perfect distribution (Fig. 3). Therefore, the structure is more homogenous at higher molarity concentration. As discussed earlier, the higher molarity concentration leads to increase in regular

![Fig. 3](image-url)
distribution of LiNbO₃ nano- and micro-structures as indicated in Table 1. SEM illustrates ice layers shape at low magnification as shown in Fig. 3e.

The grain size and root mean square could be affected by molarity concentration. Figure 4 shows AFM images of the LiNbO₃ nanophotonics with a uniform dense surface and exhibits a decrease in grain size as the molarity concentration increases. The surface topography of LiNbO₃ nanophotonics as observed from the AFM micrographs proves that the grains are uniformly distributed within the scanning area (5 μm × 5 μm), with individual columnar grains extending upward. This surface characteristic is quoted from the topographic image, which is uniform, smooth and homogeneous at 1.0 mol than others. Moreover, the decrease in average grain size from 128 to 84 nm appears on the higher molar concentration (Fig. 4a; Table 2). From XRD results, we can classify the material as micro- and nano-material, but in optimum condition, 1 mol/L can be classified only as nano-material. On the other hand, we note that the surface roughness increases (ranging between 10.6 and 16.0 nm) as the molarity concentration increases, because of lack of solubility and inverse correlation with grain size (Table 1). The average surface roughness was optimum to fabricate the optical waveguide.

Fig. 4 AFM images of LiNbO₃ nanophotonics at different molarity concentrations. a 0.25, b 0.5, c 0.75 and d 1 mol/L.
The thickness was determined using an optical reflectometer. The thickness increases as the molarity concentration increases, as shown in Fig. 5. Therefore, the increase in molarity affects negatively on the grain size that guides to smoothen the surface.

**3.3 Optical properties**

The transmission spectra of LiNbO₃ nanophotonics are shown in Fig. 6a, and it is found that the transmission decreases as molarity concentration increases, due to increase in deposition rate and structure thickness. These values of transmission are about 38–93 % with molarity concentration 1.00–0.25 mol/L (Table 2). The deposited samples were yellow to brown in color and show low transmittance due to excessive LiNbO₃ atoms in the structure [30]. The lower value of transmittance is attributed to these excessive LiNbO₃ ions existing at interstitial sites that probably absorb light.

The wide direct band gap makes LiNbO₃ a good material for potential applications in optoelectronic devices, such as multilayer dielectric filters and solar cells due to decreasing window absorption loses that will improve the short circuit current of the cell. The energy band gap ($E_g$) is found by plot $(\alpha h\nu)^2$ versus $h\nu$ [31] as shown in Fig. 6b, and given in Table 2 with good accordance with experimental value [32]. The optical reflectance ($R$ %) of LiNbO₃ nanophotonics was measured using double-beam UV–Vis and was calculated from the absorption and the transmittance spectrum, using the relation; $R + T + A = 1$. Figure 6c shows the reflectance of LiNbO₃ nanophotonics. The refractive index ($n$) was determined from a transmittance spectrum as a function of the wavelength in the range 300–700 nm. There is a change in the refractive index in the visible range; it was estimated to be 2.21–2.55 at 330 nm as shown in Fig. 6d and given in Table 2. The refractive index changes slightly and steadily after 330–700 nm [33, 34]. It is noticed that the refractive index increases as the molarity concentration increases. This behavior may be attributed to the increasing thickness because of the molarity concentration variation. The highest values of refractive index are suitable for optical waveguide. Figure 6e shows the direct relationship between $n$ and the molarity concentration that justifies that the molarity affects the refractive index, directly. The refractive index $n$ is an important physical parameter related to microscopic atomic interactions. Theoretically, the two different approaches in viewing this subject are the refractive index related to density, and the local polarizability of these entities [35]. On the other hand, the crystalline structure represented by a delocalized picture shows that $n$ will be closely related to the energy band structure of the material, complicated quantum mechanical analysis requirements and the obtained results. Many attempts have been made to relate the refractive index $n$ and the energy gap $E_g$ through simple relationships [36–40]. Here, the various relationships between $n$ and $E_g$ will be reviewed to validate the current work. Ravindra et al. [41] had suggested different relationships between the band
gap and the high-frequency refractive index, and presented a linear form of $n$ as a function of $E_g$:

$$n = \alpha + \beta E_g,$$

(5)

where $\alpha = 4.048$ and $\beta = -0.62 \text{ eV}^{-1}$.

To be inspired by simple physics of light refraction and dispersion, Herve and Vandamme [42] had proposed an empirical relation as:

$$n = \sqrt{1 + \left(\frac{A}{E_g + B}\right)^2},$$

(6)

where $A = 13.6 \text{ eV}$ and $B = 3.4 \text{ eV}$. Ghosh et al. [43] had taken a different approach to the problem by considering the band structural and quantum-dielectric formulations of Penn [44] and Van Vechten [45]. Introducing $A$ as the contribution from the valence electrons and $B$ as a constant additive to the lowest band gap $E_g$, the expression for the high-frequency refractive index is written as:

$$n^2 - 1 = A/(E_g + B)^2,$$

(7)
where \( A = 25E_g + 212, B = 0.21E_g + 4.25 \) and \( \left( E_g + B \right) \) refers to an appropriate average energy gap of the material. Thus, these three models of variation \( n \) with energy gap have been calculated. Also, the calculated values of the optical dielectric constant \( \left( \varepsilon_{\infty} \right) \) were obtained using the relation \( \varepsilon_{\infty} = n^2 \) [46]. The calculated refractive index and optical dielectric constant are as given in Table 2. The results indicate that the model of Ghosh et al. is an appropriate model for waveguide applications. The increase in thickness results in overall increase in refractive index. This increase is due to overall decrease in transmission, which is consistent with the established result that the crystallinity of the films improves as thickness increases.

### 4 Conclusion

The LiNbO\(_3\) nanophotonics have been chemically prepared by spin-coating technique. SEM explains the nanophotonics will be more homogenous as the molarity concentration increases. The polycrystalline nature of LiNbO\(_3\) nanophotonics is confirmed by XRD. The highest intensity is shown at orientation (012) at \( 2\theta = 23.634 \), with a significant increase as the spin-coating speed at 3000 rpm. The structure becomes more crystalline as the molarity concentration increases. Also, morphology gives homogeneity, topography shows gain size decreasing from 128 to 84 nm, and roughness is ranging between 10.6 and 16.0 nm as molarity increases. Optimal properties give values of transmission that is about 38–93 \%, and the measured energy band gaps were 3.65, 3.74, 3.85 and 3.97 eV. Refractive index determined from the transmission spectrum and specific empirical models revealed that the highest value and Ghosh et al. model are more appropriate for optical waveguide.

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