

Deposition Time Effect on LN Films Properties Using Chemical Bath Deposition Method without Post Heat Treatment

Rawan Bashar Fadhil¹, Evan T. Salim^{1,*}, Wafaa. K. Khalef¹, and Forat H. Alsultany²

¹ Applied Science Department, University of Technology-Iraq, 10066 Baghdad, Iraq ² Al-Mustaqbal University College, Department of Medical Physics, Iraq

ABSTRACT

Lithium niobate (LiNbO₃) is a non-naturally-occurring salt consisting of niobium, lithium, and oxygen. Lithium niobate (LiNbO₃) is one of the most widely studied photonic crystal materials. In this work the chemical bath deposition method was employed for preparing lithium niobate (LiNbO₃; LN) nanostructures. LiNbO₃ nanostructures were deposited on quartz substrates. Stirring was continued for 12 h and deposition was performed at various times (5, 10, 15, 20 and 25 min). The Optimum condition was at 15 minute. The optical properties have been investigated using UV-Vis spectrophotometer and structural properties using X-ray diffractometer. Structural properties reveals that all the samples have a hexagonal structure with a maximum diffraction peak at the (012) diffraction plane and at $2\theta = 23.75$. Optical absorption reveals the absorbance of a sample with the energy gap shifting with decreasing in deposition time from 3.8 eV to 4 eV with the peak position of absorption in UV region.

Keywords: Lithium niobate; deposition time; CBD methods; structural properties; optical properties

1. INTRODUCTION

LN is a notable optical substance that has been widely employed in photonic industry as it exhibits unique acoustic-optical, piezo electrical, electro-optics, and photo-refractive properties. owing to its large second-order nonlinearities, LN is one of the effective materials for electro-optic applications [improve, break into 2 sentences] [1-6]. LN is used in manufacturing optical waveguides, nonlinear optics for telecommunication [7-11], photonic fibre crystals as well as in electro-optic modulation [12–14]. Regarding its significance in a wide range of applications, LN has been investigated throughout several decades. Different techniques have been used to prepare LN nanocrystals including soft chemistry, sol–gel [15-17], pulsed laser deposition, and pulse laser ablation [18, 19], magnetron sputtering [20, 21] and chemical hydrothermal techniques [22-24]. All previous preparation processes including different deposition times should be improved [25-27]. Explain the problem or knowledge gap that to be solved/contribute by this work.

The CBD method typically forms films using heterogeneous nucleation (deposition or adsorption of aqueous ions onto a solid substrate) to form homogeneous thin films of metal chalcogenides (mostly oxides, sulphides and selenides) and many less common ionic compounds [28, 29]. This method is useful in industrial applications, because it is extremely cheap, simple and reliable compared with other methods of thin- film deposition [30, 31]. It requires only an aqueous solution at (relatively) low temperatures and minimal infrastructure [32, 33]. Small crystals could be form using CBD method, which are less useful for semiconductors than the larger crystals created by other methods of thin-film deposition, but are more useful for nano materials [34, 35]. However, films formed by CBD often have better photovoltaic properties (band electron gap) than

^{*}evan_tarq@yahoo.com, & evan.t.salim@uotechnology.edu.iq.

films of the same substance formed by other methods [36, 37]. Many researchers studied the effect of interaction time on physical properties of different materials, including $LiNbO_3$ or spray pyrolysis [38-41]. In this work, lithium niobate $LiNbO_3$ (LN) nanostructures were investigated via CBD on a quartz substrate at different chemical deposition times to study the effect on the structural and optical properties of the grown films [42, 43]. The optimum conditions for different applications were determined.

2. EXPERIMENTAL METHODS

The experimental steps used in the current work are shown in Figure 1a. Citric acid (CA) and a high purity (99.99%) of niobium pentoxide were employed without any purification. Mixing Ethylene glycol (EG) with CA in a glass beaker to create a solution (for 2 h) was the next step. Then, continuous stirring with Li_2Co_3 and Nb_2O_5 was conducted for 9 h at 90 °C. The following material weights were utilised: $Li_2Co_3 = 3.7$ g, $Nb_2O_5 = 13.30$ g, CA = 10.5 g and EG = 20 g; the molar ratio between Nb_2O_5 and Li_2Co_3 was 1:1 in accordance with a previous published article [42, 44]. Samples were produced after 12 h by adding quartz substrates to a chemical bath at various deposition times (5, 10, 15, 20 and 25 min). As illustrated in Figure 1b, the quartz substrate was inserted vertically into the solution beaker. Optical interference method was utilized to measure the samples' thicknesses. X-ray diffraction (XRD) analysis was performed using a Schimadzu 6000 instrument to investigate the structural properties of LN, whilst optical properties were investigated by utilising a double-beam ultraviolet–visible (UV–Vis) spectrophotometer (Schimadzu 1800). Figure 1 should be improved. Make the appearance more professional.



Figure 1. (a) Lithium-niobate nanostructure preparation flow chart. (b) Chemical bath deposition technique.

3. RESULTS AND DISCUSSION

3.1 Structural Properties

The influence of deposition time on structural properties is illustrated in Figure 2. The material has a nearly amorphous structure, and the intensity of dispersion in X-ray increases over time, reaching the highest crystalline structure at a specific time, beyond which crystallinity starts to decrease. As shown in Figure 2.a, the LN peaks of the sample that was stirred for 12 h and deposited for 5 min are at $2\theta = 23.75$, 32.65, 34.50, 47.65 and 54.30, which correspond to the (012), (104), (110), (024) and (116) planes, respectively. This result agrees with those of [42, 45-47]. Figure 2.b indicates that the LN peaks of the sample stirred for 12 h and deposited for 10 min are at $2\theta = 23.75$, 32.62, 34.45, 47.65 and 54.25, which correspond to the (012), (104), (110), (024) and (116) planes, respectively. This result agrees with those of [42, 45-47]. Meanwhile, Figure 2.c shows that the LN peaks of the sample that was stirred for 12 h and deposited for 15 min are at $2\theta = 23.75$, 32.65, 34.65, 47.55 and 54.32, which correspond to the (012), (104), (110), (024) and (116) planes, respectively. This result agrees with those of [27-30]. Figure 2.d shows that the LN peaks of the sample that was stirred for 12 h and deposited for 20 min are at 2θ = 23.75, 32.65, 34.65, 47.65 and 54.25, which correspond to the (012), (104), (110), (024) and (116) planes, respectively. This result concurs with those of [42, 45-47]. In Figure 2.e, the LN peaks of the sample that was stirred for 12 h and deposited for 25 min are at $2\theta = 23.85$, 32.70, 34.65, 47.75 and 54.25, which correspond to the (012), (104), (110), (024) and (116) planes, respectively. This result is in agreement with those of [27-30]. Evidently, all these peaks belong to hexagonal structure of LN nanophotonics with lattice parameters a = b = 5.1566 and c = 13.858 [42, 45-47]. Table 1 lists the structural properties of LN nanophotonics that must be measured. Scherrer's formula was used to compute crystallite size (D) [48-50].

$$D = \frac{\kappa\lambda}{\beta\cos\theta} \tag{1}$$

where the constant k is assumed to be 0.94, λ is the utilised X-ray wavelength that is assumed to be 1.54 Å, θ is the Bragg's angle and β is the full width at half maximum of the X-ray diffraction patterns. The strain (ϵ) and the dislocation density (δ) of LN nanophotonics were calculated using the following equations [9, 51-54]:

$$\delta = \frac{1}{D^2} \tag{2}$$

$$\varepsilon = \frac{\beta}{4\tan\theta} \tag{3}$$

Bragg's formula was used to compute the interplanar distance (d) for all sets of LN nanophotonics [9, 55, 56]

$$d = \frac{n}{2\sin\theta} \tag{4}$$

where n is a positive integer number, and d is the value provided in Table 1. Two phases of LN with a polycrystalline structure are identified in nanophotonics, i.e. LN and LiNb₃O₈ phases. The LN phase is preferable. However, the achieved phase is oriented at (012). A small amount of secondary lithium-deficient phase (LiNb₃O₈) is clearly observed in all the samples on the XRD results [57]. This phase is produced through oxygen–LiNbO₃ interfacial interaction. The XRD peaks of LiNb₃O₈ are at $2\theta = 24.45$, 30.75, 31.10, 35.20, 38.55, 43.47 and 43.65, which correspond to the (400), (012), (202), (-601), (-403), (203) and (420) planes, respectively. These results agree with the Joint Committee on Powder Diffraction Standards (JCPDS) card no. 01-074-2239. Notably, different peaks (Nb₂O₅) occur at $2\theta = 25.55$, which corresponds to the (–212) plane. This

result concurs with JCPDS card no. 01-074-2239). Table 1 indicates that the experimental values and measured lattice constants are similar (reported values).



Figure 2. X-Ray Diffraction patterns of LiNbO₃ nanostructure at different deposition time a) 5m, b) 10 m, c) 15 m, d) 20 m, e) 25 m.

Chemical Deposition Time (minute)	Orientation (hkl)	Peaks (2θ)	Grain Size (nm)	Dislocation Density (δ)(10 ⁹) (lines/m ²)	Strain (10 ⁻³)	dhki	Lattice Constants a ,c
5 min	012	23.65	4.77	4.37E+12	0.72	3.76	a=5.1566 c=13.85
	104	32.60	5.31	3.54E+12	0.62	2.74	a=5.1566 c=13.85
	110	34.65	6.60	2.29E+12	0.49	2.58	a=5.1566 c=13.85
	024	47.54	9.49	1.10E+12	0.31	1.88	a=5.1566 c=13.85
	116	54.31	14.77	4.58E+11	0.19	1.72	a=5.1566 c=13.85
10 min	012	23.68	5.41	3.40E+12	0.64	3.74	a=5.1566 c=13.85
	104	32.60	6.12	2.66E+12	0.54	2.73	a=5.1566 c=13.85

 $\textbf{Table 1} \ \text{LiNbO}_3 \ \text{nanophotonic parameters at different deposition time}$

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	110	34.64	9.91	1.01E+12	0.33	2.57	a=5.1566 c=13.85
	024	47.54	15.19	4.33E+11	0.19	1.87	a=5.1566 c=13.85
	116	54.31	24.62	1.64E+11	0.11	1.71	a=5.1566 c=13.85
15 min	012	23.74	5.80	2.96E+12	0.59	3.75	a=5.1566 c=13.85
	104	32.60	6.64	2.26E+12	0.50	2.71	a=5.1566 c=13.85
	110	34.65	13.20	5.73E+11	0.24	2.57	a=5.1566 c=13.85
	024	47.54	25.32	1.55E+11	0.119	1.86	a=5.1566 c=13.85
	116	54.31	36.93	1.64E+11	0.116	1.68	a=5.1566 c=13.85
20 min	012	23.75	6.24	2.56E+12	0.55	3.74	a=5.1566 c=13.85
	104	32.61	7.24	1.90E+12	0.46	2.70	a=5.1566 c=13.85
	110	34.65	15.85	3.98E+11	0.20	2.54	a=5.1566 c=13.85
	024	47.55	37.98	1.55E+11	0.11	1.91	a=5.1566 c=13.85
	116	54.32	73.87	1.64E+11	0.11	1.68	a=5.1566 c=13.85
25 min	012	23.76	8.12	1.51E+12	0.42	3.74	a=5.1566 c=13.85
	104	32.61	9.96	1.00E+12	0.33	2.73	a=5.1566 c=13.85
	110	34.65	15.85	3.98E+11	0.20	2.57	a=5.1566 c=13.85
	024	47.57	18.99	2.77E+11	0.15	1.87	a=5.1566 c=13.85
	116	54.36	24.61	1.64E+11	0.11	1.71	a=5.1566 c=13.85

3.2 Optical Properties

Figure 3 illustrates the absorbance spectra (inset Figure 3) of the formed LN thin film after various deposition times (5, 10, 15, 20 and 25 min) via the chemical bath deposition technique. The peak position of absorption for all the samples appeared in the UV region, as shown in the absorption spectra in Figure 3. The absorption edge for all the samples was between 300–330 nm and 300–350 nm. Moreover, absorption increased when wavelength was reduced. The transition of an electron from the upper portion of the valence band to the lowest region of the conduction band is referred to as spectral absorption in the wavelength range below 350 nm. The UV–Vis absorption spectra present a broad "shoulder" centred at about 305–350 nm (Figure 3). This result agrees with that of [58, 59]. Energy gap was calculated using a plotting graph between (α hv)2 and hv (eV). Figure 3 presents the obtained energy gap values. The sample value was observed to be about 3.8–4 eV. The energy gap values at different deposition times (25, 20, 15, 10)

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and 5 min) were about 3.95, 3.96, 3.98, 3.99 and 4 eV, respectively. In accordance with Scherrer's equation, energy gap decreased as particle size increased. This is because bandgap energy is inversely proportional to the size of nanoparticles due to the 'quantum confinement effect'. That is, bandgap energy increases between the valence and conduction bands with decreasing particle size because of the confinement of electrons and holes. Bandgap decreasing or increasing with respect to particle size actually depends on the principal host in which metal nanoparticles are embedded.

In semiconductors, the relation that controls the absorption edge for transitions can be obtained according to the Tauc's model, as follows [60-64]:

$\alpha hv = A(hv - Eg)n$

(5)

In this equation, hv is the photon energy of the incident light, and Eg is the optical bandgap. The value of 'n' for an allowed direct transition is $\frac{1}{2}$, whilst that for indirect transition is 2. In the current case, the prepared material has an indirect bandgap wherein the lower energy state in the conduction band and the higher energy state in the valance band do not have the same wave vector (i.e. the momentum is not conserve). Transition occurs with phonon absorption or emission. The variation of $(\alpha hv)^2$ against (hv) yields an excellent straight-line fit to the absorption edge, and the extrapolated (hv) at $(\alpha hv)^2 = 0$ provides the Eg of the indirect transition depicted in Figure 3.

As shown in Figure 4, the energy gap decreased as chemical deposition time increased due to the increase in particle size when clear redshift could be observed [65-68].



Figure 3. Energy gap and absorption of Lithium niobate film, in set the absorbance as a function of deposition at different deposition time a) 5m, b)10m, c)15m, d)20m, e)25m.



Figure 4. Energy gap of LiNbO₃ nanophotonics film as a function of deposition time.

4. CONCLUSION

Deposition time exhibited a remarkable effect on LN nanostructures when the chemical bath deposition method was used. Structural properties ensure the transformation from a polycrystalline with a moderate hexagonal structure to an amorphous one at the longest deposition time. The optical absorption reveals lower absorbance for samples with an energy gap shifting from 3.8 eV to 4 eV with a decrease in deposition time.

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