OPTICAL PROPERTIES OF NEODYMIUM DOPED LITHIUM NIOBATE SINGLE CRYSTALS GROWN BY CZOCHRALSKI METHOD

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To my beloved mother and father

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ABSTRACT

$LiNbO_3$ and Nd^{3+} doped $LiNbO_3$ crystals have successfully been grown by using Czochralski (CZ) method. Their crystallinity has been confirmed using X-ray diffraction (XRD) technique and the result has been used to determine the lattice parameters. The density of the crystals is measured using Archimedes principle while the transmission spectra are obtained using Fourier Transform Infrared Spectroscopy (FTIR). Their absorption characteristic is determined using Ultra Violet-Visible (UV-Vis) Spectroscopy. From the X-ray diffraction spectra, it is found that the samples exhibit single phase congruent composition and the lattice parameter of *a* and *c* are in the range of 5.1423Å – 5.1711Å and 13.8482Å – 13.9258Å, respectively. The crystals density is found to be ranging from 4.637 g cm^{-3} – 4.649 g cm^{-3} and it is in the increasing trend with respect to Nd^{3+} content. The crystals present a broad OH^- absorption band located at approximately 3486 cm^{-1}. It is observed that the addition small amount of Nd^{3+} ion into the system has small effect on the absorption band position and does not change the crystal structure. It is also found that the increasing amount of Nd^{3+} content caused the optical band gap E_g to increase from 3.72 eV to 3.80 eV and 3.48 eV to 3.60 eV for direct transition allowed and forbidden, respectively. Meanwhile, the E_g for indirect transition allowed and forbidden are about 3.36 eV – 3.52 eV and 3.15 eV – 3.36 eV, respectively. The Urbach energy, ΔE is found to be in the range of 0.30 eV – 0.45 eV and increases with Nd^{3+} content. Meanwhile, the addition of Nd^{3+} ion caused the refractive index to decrease from 2.480 to 2.008 and increase the Judd-Ofelt intensity parameters, from 0.3006×10^{-19} to 1.8490×10^{-19} for Ω_2, 0.9213×10^{-19} to 4.4042×10^{-19} for Ω_4 and 0.9511×10^{-19} to 6.2420×10^{-19} for Ω_6.

ABSTRAK

Hablur $LiNbO_3$ tulen dan $LiNbO_3$ didop ion Nd^{3+} telah ditumbuhkan menggunakan kaedah Czochralski (CZ). Kehablurannya telah ditentusahkan menggunakan teknik pembelauan sinar-X (XRD) dan hasil tersebut digunakan untuk menentukan parameter kekisi. Ketumpatan hablur telah ditentukan menggunakan prinsip Archimedes manakala spektra penghantaran telah diperolehi menggunakan spektroskopi inframerah Transformasi Fourier (FTIR). Ciri penyerapan telah ditentukan menggunakan spektroskopi ultra ungu – boleh nampak (UV-Vis). Daripada spektra pembelauan sinar-X, didapati bahawa sampel-sampel tersebut menunjukkan fasa tunggal komposisi kongruen dan nilai-nilai parameter kekisi *a* dan *c* didapati masing-masing berada dalam julat 5.1423Å – 5.1711Å dan 13.8482Å – 13.9258Å. Ketumpatan hablur pula telah didapati berada pada julat 4.637 g cm^{-3} – 4.649 g cm^{-3} dan ia meningkat dengan peningkatan kandungan ion Nd^{3+}. Hablur-hablur tersebut mempamerkan satu jalur penyerapan OH^- yang lebar yang terletak pada sekitar 3486 cm^{-1}. Pertambahan kecil Nd^{3+} ke dalam sistem telah memberi sedikit kesan ke atas posisi jalur penyerapan tetapi tidak mengubah struktur hablur tersebut. Didapati juga, peningkatan jumlah kandungan Nd^{3+} menyebabkan jurang tenaga optik E_g bertambah dari 3.72 eV kepada 3.80 eV untuk peralihan terus dibenarkan dan 3.48 eV kepada 3.60 eV untuk peralihan terus terlarang. Demikian juga untuk peralihan tak terus dibenarkan dan terlarang, E_g masing-masing berada dalam julat 3.36 eV – 3.52 eV dan 3.15 eV – 3.36 eV. Tenaga Urbach, ΔE didapati berada pada julat 0.30 eV – 0.45 eV, meningkat dengan kandungan Nd^{3+}. Penambahan kandungan ion dopan Nd^{3+} menyebabkan indeks biasan berkurang dari 2.480 kepada 2.008 dan juga meningkatkan parameter keamatan Judd-Ofelt dari 0.3006×10^{-19} kepada 1.8490×10^{-19} bagi Ω_2, 0.9213×10^{-19} kepada 4.4042×10^{-19} bagi Ω_4 dan 0.9511×10^{-19} kepada 6.2420×10^{-19} bagi Ω_6.

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LIST OF SYMBOLS

α - Absorption coefficient

A - Absorption intensity

A_j - Sellmeier parameter

c - Speed of light

d - Atomic spacing

d - Sample thickness

E_g - Optical energy gap

e - Electron charge

eV - Electron Volt

ΔE - Width of the band tails

ε - Permittivity

f - Vibration frequency

f_{cal} - Theoretical oscillator strengths

f_{exp} - Experimental oscillator strengths

f_{md} - Oscillator strengths contains of magnetic-dipole

hkl - Crystal plane orientation index

m - Mass

$\mathrm{\mu}$ - Mobility

μ - Permeability

n - Refractive index

n^* - Complex refractive index

$\mathrm{\rho}$ - Resistivity

ρ - Density

ρ_a - Air density

ρ_l - Liquid density

Q - Quality factor

R - Refraction intensity

v - Speed

$\bar{v}$ - Wavenumber

W_a - Weight of sample in air

W_l - Weight of sample in immersion fluid

ω - Frequency

θ - Angle

λ - Wavelength

Ω_q - Judd-Ofelt parameters

$\left\langle \left\| U^q \right\| \right\rangle$ - Double reduce matrix elements

LIST OF ABBREVIATIONS

ADC - Automatic Diameter Controller
CB - Conduction band
CGS - Crystal Growth System
CZ - Czochralski
DCCZ - Double crucible Czochralski
FTIR - Fourier transformed infrared
g - Gram
hr - Hour
IR - Infrared
JCPDS - Joint Comitee on Powder Diffraction Standard
J-O - Judd – Ofelt theory
LN - Lithium niobate
mm - Millimetre
mV - Millivolt
NIR - Near infrared
nm - Nanometer
PI - Proportional Integral
PID - Proportional Integral Derivative
RF - Radio Frequency
rpm - Rotation per minute
UV - Ultraviolet
Vis - Visible
VB - Valence band
XRD - X-ray diffraction

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Crystal growth involves a variety of research fields ranging from surface physics, crystallography and material sciences to condense material physics. Even though it has been studied extensively for more than 100 years, crystal growth still plays an important role in both theoretical and experimental research fields, as well as in applications. Nowadays, crystals are produced artificially to satisfy the needs of science, technology and jewellery. The ability to grow high quality crystals has become an essential criterium for the competitiveness of nations (Kamaruddin, 2010).

Two methods have been developed for growing lithium niobate crystal with a near-stoichiometric composition. Kitamura *et al*. (1992) reported that large stoichiometric lithium niobate crystal could be grown from Li-rich melt using the double crucible Czochralski (DCCZ) method with an automatic powder supply system. Malovichko *et al*. (1993) also reported that stoichiometric lithium niobate crystal could be grown from a congruent melt doped with 6 wt.% K_2O as flux and that the incorporation of potassium into crystal can be disregarded.

Lithium niobate, $LiNbO_3$ crystal is well known for its remarkable physical properties, such as electro-optical, acousto-optical and non-linear optical properties, which lead to wide technical applications and fundamental researches. Pure $LiNbO_3$ and doped $LiNbO_3$ crystal are great variety of application such as photorefractive devices (Kratzig and Schirmer, 1988), solid-state lasers (Johnson and Ballman, 1969), optical waveguides (Armenise *et al.*, 1983) and also widely used for holographic data storage and frequency doubling (Yang *et al.*, 2002). A $LiNbO_3$ crystalline host can also produce higher absorption and emission cross-sections than amorphous glass (Qiang *et al.*, 2002). Doping with foreign ions modifies the optical properties of the matrix and makes the system useful for a great variety of those applications. Another potential application of $LiNbO_3$ is in Nd^{3+}–based compact diode-pumped self-frequency doubled lasers which emit green radiation, useful for applications in optical data storage, undersea imaging, and excitation sources to replace ion gas lasers for science and pumping of parametric oscillators and amplifiers (Capmany *et al.*, 1999). $Nd:LiNbO_3$ crystal is certainly one of the most well known, multi-used, easy-to-grow and structurally simplest laser materials. Commercially available $LiNbO_3$ crystal is usually grown from congruent melt using conventional Czochralski (CZ) method. This technique, also known as crystal pulling, is widely known for growing single crystals from the melt and has become the method of choice for the growth and production of many bulk oxide materials (Kamaruddin, 2010).

The rare earth ions (Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb) are characterized by a closed xenon shell and usually the 4f shell is not completely filled (with 14 electrons). The optical transitions typical of rare earth spectra are attributed to intra-4f “forced” electric-dipole transitions of electrons already in the 4f shell to these unoccupied 4f levels. The intensitivity to host is a result of the lanthanide contraction, which is a consequence of imperfect screening by the 4f electrons that leads to an increase in effective nuclear charge as the atomic number increases in the lanthanide series (Dickerson *et al.*, 1970). Thus the 4f wavefunctions actually lie within the closed $5s^2$ $5p^6$ xenon shell for the lanthanide rare-earths, and the 4f electrons are shielded from the surrounding environment by the 5s and 5p electrons. This leads to energy levels that have small host-induced splitting and are only weakly mixed with higher energy states. This results in a greatly reduced vibronic interaction with the host, which leads to weak non-radiative relaxation of excited states (and thus longer excited-state lifetime), sharp spectral features, large cross sections and high quantum yields.

The optical properties of the rare-earth compounds have been widely studied for 30 years. This is due to their huge application as laser and luminescent materials. The position of the discrete energy levels of the $4f^N$ configuration in solids is, in general, well simulated using a Hamiltonian which implies the adjustment of both free atom and crystal-field parameters. The neodymium compounds have been among the most studied because of the relative simplicity of the $4f^3$ configuration and of its important potential applications (Derouet *et al.*, 2001).

1.2 Problem Statement

Lithium niobate is a widely studied optoelectronic material due to its technological applications. Doping with foreign ions modifies the optical properties of the matrix and makes the system useful for a great variety of applications such as photorefractive devices, solid state lasers or optical waveguides (Armenise *et al.*, 1983). In the previous research, some reports on spectral properties and optical properties of Nd doped lithium niobate crystals are published (Jiangou *et al.*, 1992; Jinhou *et al*, 2009). But, some properties such as crystal lattice parameter, characterization of absorption spectra and refractive index are not systematically being reported. Besides that, the preparation of the crystal is also not being reported. This research describes a Czochralski Crystal Growth System which has unique features to permit automatic adjustment of the power level to the furnace in order to maintain a specific diameter during the growth process. Using this automatic diameter control system, pure lithium niobate ($LiNb_3O$) crystal and neodymium doped lithium niobate ($Nd{:}LiNb_3O$) crystal have been reproducibly grown.

The optical and structural properties of the crystals also will also be investigated. This study is hoped useful in order to extend the understanding and application of lithium niobate crystal.

1.3 Research Objectives

In order to provide more information on the crystal properties, the objectives of this research are:

a) To prepare $LiNb_3O$ and $Nd:LiNb_3O$ crystals.
b) To determine the crystallinity of the samples using X-ray diffraction and obtain their lattice parameters.
c) To determine the density of the crystals.
d) To study the transmission spectrum of the crystal from Fourier transform infrared (FTIR) spectroscopy.
e) To study the absorption spectrum of the crystal from UV-Visible spectroscopy and determine the band gap energy, Urbach energy, refractive index and Judd-Ofelt parameter.

1.4 Scope of Study

The research study covers the growth of pure lithium niobate and neodymium doped lithium niobate crystal via Czochralski method. The structural and optical properties of the samples are characterized.

1.5 Summary of Thesis

This thesis contains five chapters. Chapter 1 gives a brief overview of previous work of crystal development and the discussion about the problem statement, the objective and the scope of this research. Chapter 2 comprises the literature review of this research. This chapter consists of the theoretical background of crystal and lithium niobate crystal, the properties of neodymium ion (Nd^{3+}) in solids and some theory of Czochralski method. Afterwards, this chapter will provides some theoretical review on the characterization method of x-ray diffraction and determination of lattice parameter using the spacing formula. Other characterization such as infrared spectroscopy, absorption, refractive index, transition mechanism and density are also provided. Chapter 3 describes the experimental equipment and techniques used in the research. This would include the sample preparation from the raw material until the growth of the crystal using Czochralski Automatic Diameter Controller. The apparatus which are being used in the growth process and the growth parameter will be described in detail. This is followed by the characterization of the samples by using X-Ray diffractometer (XRD), infrared (IR) spectrometer, UV-visible spectrometer (UV-Vis) and densitometer. Chapter 4 discusses the results of the research. These include the growth parameters, density, XRD pattern, lattice parameter, IR vibrational spetra, absorption spectra, refractive index and quality factor from Judd Ofelt analysis. The conclusion of the study is described in Chapter 5.

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