# CHARACTERISTICS OF NEODYMIUM-MODIFIED BORATE CRYSTALS AND GLASSES GROWN VIA POLYCRYSTALLINE SEED MEDIATION

WAN HAIRUL ANUAR BIN KAMARUDDIN

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> Faculty of Science Universiti Teknologi Malaysia

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### **DEDICATION**

This thesis is dedicated to my beloved mothers (Raja Maharom & Sh. Zaiton), fathers (Kamaruddin & Sy. Abd. Azam), wife (Sh. Rabi'atul Adawiyyah), kids (Wan Nur Humairah, Wan Uzair Faaris, Wan Umar Hariz and Wan Uthman Daris) and all my families, who always give support and pray for my success.

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#### ABSTRACT

A series of glass samples with composition  $(90-x)Li_2B_4O_7+10Nb_2O_5+xNd_2O_3$ , where x = 1, 5, 10, 15, and 20 mol% were prepared by the melt-quenching technique. For polycrystal seed preparation as well as crystal growth process, 1 mol% of Nd<sub>2</sub>O<sub>3</sub> was chosen.  $Nd_2O_3$  was used as a modifier to investigate the impact on the physical, structural, and optical properties of the glasses and crystals. Polycrystal seed as well as platinum wire and lithium niobate single crystal seed were used to grow crystals by Czochralski technique. The growth and growth mechanism of the crystals were studied. The glass samples and crystals were subjected to various characterisation techniques and the physical, structural, and optical properties were determined. XRD pattern confirmed the amorphous nature of the glass system while several peaks corresponded to the phases of LiNbO<sub>3</sub>, LiNb<sub>3</sub>O<sub>8</sub>, Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub>, and Nd(BO<sub>2</sub>)<sub>3</sub> were recorded for heat-treated glass (polycrystal). The microstructural of the samples (glass  $\rightarrow$  polycrystal) showed that these crystallites grew, further suggesting that the nucleation and growth process were responsible for the development of this kind of microstructure. The optimum growth conditions of pulling rates and rotation speeds were  $0.2-1.5 \text{ mmh}^{-1}$  and 5-7 rpm, respectively. To stabilise the growth rate and achieve a good growth interface, the temperature gradient was gently optimised. FTIR spectra exhibited four vibration bands around 700, 800-1200, 1200-1600, and 3200-3600 cm<sup>-1</sup> assigned to the bending of B–O–Nd, B–O stretching of tetrahedral [BO<sub>4</sub>] units, stretching relaxation of B–O in [BO<sub>3</sub>] units, and stretching of O–H group in both NdLNB crystal and glass system. The UV-Vis spectra showed ten absorption peaks and the transition at  ${}^{4}G_{5/2}$  (583 nm) was the most prominent. As the concentration of Nd<sup>3+</sup> ions for glass system increased, both optical band gap and Urbach energies decreased. Additionally, the optical band gap of NdLNB crystal was significantly lower than that of NdLNB glass which were 2.84 and 3.75 eV, respectively. The Judd-Ofelt intensity parameters of all the glass samples followed the trend of  $\Omega_6 > \Omega_4 > \Omega_2$ except for 5NdLNB glass, which followed the trend of  $\Omega_4 > \Omega_6 > \Omega_2$  which similar to NdLNB crystal. The radiative lifetime of the glass system increased with increasing Nd<sup>3+</sup> concentration. Meanwhile, radiative lifetime for NdLNB crystal was slightly higher than for NdLNB glass. Luminescence spectra showed two prominent infrared emissions at 903 and 1059 nm which were due to  ${}^{4}F_{3/2} \rightarrow {}^{4}I_{9/2}$  and  ${}^{4}F_{3/2} \rightarrow {}^{4}I_{11/2}$ transitions, respectively while NdLNB crystal exhibited four emissions at 883, 943, 1064, and 1115 nm with the same transition. SHG tests for both NdLNB crystal and NdLNB glass recorded an emission peak at 532 and 529.5 nm, respectively. The achieved results promise the use of these optimised glasses and crystals in frequency conversion, nonlinear optical application, and laser production. The new approach of using the polycrystal seed to grow a crystal with modified properties from its own glass-forming melt was also successful.

#### ABSTRAK

Satu siri sampel kaca dengan komposisi (90-x)  $Li_2B_4O_7 + 10Nb_2O_5 + xNd_2O_3$ , dengan x = 1, 5, 10, 15 dan 20 mol% telah disediakan menggunakan kaedah pelindapan leburan. Bagi penyediaan benih polihablur dan juga proses pertumbuhan hablur, 1 mol% Nd<sub>2</sub>O<sub>3</sub> telah dipilih. Nd<sub>2</sub>O<sub>3</sub> telah digunakan sebagai pengubah untuk mengkaji kesan terhadap sifat fizikal, struktur dan optik kaca dan hablur. Benih polihablur serta wayar platinum dan hablur tunggal litium niobate digunakan untuk menumbuhkan hablur dengan kaedah Czochralski. Mekanisma pertumbuhan hablur telah dikaji. Hablur dan kaca NdLNB telah melalui pelbagai kaedah pencirian dan sifat fizikal, struktur dan optik telah diperolehi. Corak XRD telah mengesahkan sifat amorfus sistem kaca manakala beberapa puncak yang merujuk kepada fasa LiNbO<sub>3</sub>, LiNb<sub>3</sub>O<sub>8</sub>,  $Li_2B_4O_7$  dan Nd(BO<sub>2</sub>)<sub>3</sub> telah direkodkan untuk kaca terawat haba (polihablur). Struktur mikro sampel (kaca  $\rightarrow$  kaca terawat haba) menunjukkan habluran telah membesar, yang mana proses nukleasi dan pertumbuhan berperanan dalam pengembangan struktur mikro ini. Tetapan pertumbuhan optimum bagi kadar tarikan ialah 0.2–1.5 mmj<sup>-1</sup> dan kelajuan putaran ialah 5–7 rpm. Bagi menstabilkan kadar pertumbuhan dan mendapatkan sempadan permukaan yang baik, kecerunan suhu perlu dikawal dengan baik. Spektrum FTIR telah mempamerkan tiga jalur getaran di sekitar 700, 800-1200, 1200-1600, and 3200-3600 cm<sup>-1</sup> yang merujuk kepada lenturan ikatan B-O-Nd, regangan ikatan B-O unit tetrahedron [BO<sub>4</sub>] dan kelonggaran regangan ikatan B-O unit [BO3] untuk kedua-dua NdLNB hablur dan sistem kaca. Spektrum UV-Vis-NIR telah menunjukkan sepuluh puncak penyerapan dan transisi pada  ${}^{4}G_{5/2}$  (583 nm) adalah vang paling ketara. Apabila kepekatan ion Nd<sup>3+</sup> untuk sistem kaca meningkat, keduadua jurang tenaga optik dan tenaga Urbach menurun. Selain itu, didapati jurang tenaga optik hablur NdLNB jauh lebih rendah daripada kaca NdLNB yang masing-masing ialah 2.84 dan 3.75 eV. Telah diperhatikan bahawa parameter keamatan Judd-Ofelt bagi semua sampel kaca mengikuti trend  $\Omega_6 > \Omega_4 > \Omega_2$  kecuali kaca bagi 5NdLNB yang mengikuti trend  $\Omega_4 > \Omega_6 > \Omega_2$  sama seperti hablur NdLNB. Jangka hayat pendarcahaya untuk sistem kaca meningkat apabila kepekatan ion Nd<sup>3+</sup> meningkat. Manakala, jangka hayat pendarcahaya untuk hablur NdLNB adalah sedikit tinggi berbanding kaca NdLNB. Spektrum fotopendarcahaya menunjukkan dua pancaran inframerah yang ketara pada 903 dan 1059 nm yang disebabkan oleh transisi  ${}^{4}F_{3/2} \rightarrow {}^{4}I_{9/2}$  dan  ${}^{4}F_{3/2} \rightarrow$ <sup>4</sup>I<sub>11/2</sub>, manakala hablur NdLNB menunjukkan empat pancaran pada 883, 943, 1064, dan 1115 nm dengan transisi yang sama. Ujian SHG bagi kedua-dua hablur dan kaca NdLNB telah mencatatkan puncak pancaran masing-masing pada 532 dan 529.5 nm, yang mana ianya sesuai digunakan untuk penggandaan frekuensi, aplikasi optik tidak linear dan penghasilan laser. Pendekatan baru untuk menggunakan benih polihablur bagi menumbuhkan hablur dari pembentukan leburan kacanya sendiri telah berjaya.

## TABLE OF CONTENTS

## TITLE

DI	ECLARATION	ii
DI	EDICATION	iii
AC	CKNOWLEDGEMENT	iv
AI	BSTRACT	V
AI	BSTRAK	vi
TA	ABLE OF CONTENTS	vii
LI	ST OF TABLES	xii
LI	ST OF FIGURES	XV
LI	ST OF ABBREVIATIONS	xix
LI	ST OF SYMBOLS	xxi
LI	ST OF APPENDICES	xxiii
CHAPTER 1	INTRODUCTION	1
1.1	Background of the Study	1
1.2	2 Problem Statement	4
1.3	3 Research Objectives	4
1.4	4 Scope of the Study	5
1.5	5 Significance of the Study	6
1.6	5 Thesis Organisation	6
CHAPTER 2	LITERATURE REVIEW	9
2.1	Introduction	9
2.2	2 Glass	9
	2.2.1 Borate Glasses	10
	2.2.2 Lithium Borate Glasses	11
2.3	3 Glass to Polycrystal	13
	2.3.1 Crystallisation of Borate Glasses	16

		2.4.1	Classific	ation of Crystal Growth	19
			2.4.1.1	Solid Growth Technique	20
			2.4.1.2	Growth from Vapour	20
			2.4.1.3	Growth from Liquid/Solution	20
		2.4.2	Borate C	rystal	30
		2.4.3	Lithium	Borate Crystal	34
	2.5	Optica	al Properti	es and Material Characterisations	36
		2.5.1	Amorph	ous and Crystalline Phase	36
		2.5.2	Differen	tial Thermal Analysis	39
		2.5.3	Density	and Molar Volume Measurement	42
		2.5.4	Infrared	Analysis	44
		2.5.5	UV-Visi	ble Spectrum	48
			2.5.5.1	Nephelauxetic Ratio and Bonding Parameter	53
			2.5.5.2	Judd-Ofelt Analysis	54
		2.5.6	Photolur	ninescence Measurement	58
		2.5.7	Sample	Morphology and Elemental Analysis	62
		2.5.8	TEM Ar	alysis	64
		2.5.9	Nonlinea	ar Characterisation	67
	2.6	Resea	rch Gap		68
CHAPTER	R 3	RESE	ARCH M	ETHODOLOGY	73
	3.1	Introd	uction		73
	3.2	Glass	Preparatio	on	73
	3.3	Polyc	rystal Seed	d Preparation	77
	3.4	Crysta Grow	al Growth th System	by Automatic Diameter Control – Crystal (ADC-CGS)	77
		3.4.1	Crystal (	Growth Process	80
			3.4.1.1	Preparation and Heating Phase	80
			3.4.1.2	Seeding Phase	82
			3.4.1.3	Necking Phase	82
			3.4.1.4	Main Growth Phase	82
			3.4.1.5	Separation Phase	83

		3.4.1.6	Cooling Phase	83
	3.4.2	Machining	g Process	84
3.5	Mater	ial Characte	erisations	84
	3.5.1	X-Ray Di	ffraction	84
	3.5.2	Differenti	al Thermal Analyser	85
	3.5.3	Density M	leasurement	85
	3.5.4	Fourier Tr	ransform Infrared Spectroscopy	85
	3.5.5	UV-Visib	le Absorption Spectroscopy	86
	3.5.6	Photolum	inescence Spectroscopy	86
	3.5.7	Scanning	Electron Microscopy	86
	3.5.8	Transmiss	sion Electron Microscopy	87
	3.5.9	Second H	armonic Generator Test	87
CHAPTER 4	RESU	LTS AND	DISCUSSION	89
4.1	Introd	uction		89
4.2	Chara	cterisation of	of NdLNB Glass System	89
	4.2.1	Glass Con	nposition and Formation	89
	4.2.2	Physical F	Properties	91
	4.2.3	XRD Patt	ern	94
	4.2.4	Energy Di	ispersive X-Ray	95
	4.2.5	Thermal F	Properties	97
	4.2.6	FTIR Spe	ctra	101
	4.2.7	Optical Sp	pectroscopy	104
		4.2.7.1	Absorption spectra	104
		4.2.7.2	Optical Band Gap Energy and Urbach Energy	106
		4.2.7.3	Nephelauxetic Ratio and Bonding Parameter	112
		4.2.7.4	Judd–Ofelt Intensity and Radiation Parameter	115
	4.2.8	Optical Pr	operties: Luminescence Spectra	120
	4.2.9	Conclusio	n for Polycrystal Seed	126
4.3	Chara	cterisation of	of NdLNB Polycrystal Seed	126

	4.3.1	DTA for	NdLNB Polycrystal	127
	4.3.2	XRD for	NdLNB Polycrystal	128
	4.3.3	SEM for	NdLNB Polycrystal	130
	4.3.4	Conclusio	on for Crystal Growth	131
4.4	Growt	h and Grov	wth Mechanism of NdLNB Crystal	132
	4.4.1	Growth o	f NdLNB Crystal by Polycrystal Seed	133
	4.4.2	Growth o	f NdLNB Crystal by Platinum Wire Seed	134
	4.4.3	Growth Crystal S	of NdLNB Crystal by Lithium Niobate eed	136
	4.4.4	Growth N	Aechanism	137
		4.4.4.1	Summary of the Growth Mechanism	147
	4.4.5	Conclusio	on of Growth	148
4.5	Compa	arison of N	IdLNB Crystal and Glass Properties	148
	4.5.1	Physical	Properties	149
	4.5.2	X-Ray D	iffraction	149
	4.5.3	EDX Spe	ctrum	151
	4.5.4	Different	ial Thermal Analyses	152
	4.5.5	FTIR Spe	ectra	153
	4.5.6	Optical P	roperties	155
		4.5.6.1	Absorption spectra	155
		4.5.6.2	Optical Band Gap Energy and Urbach Energy	158
		4.5.6.3	Nephelauxetic Ratio and Bonding Parameter	160
		4.5.6.4	Judd–Ofelt Analysis	162
	4.5.7	Lumines	cence Spectra	166
	4.5.8	TEM Ana	alysis	169
	4.5.9	SEM Ana	alysis	170
	4.5.10	Nonlinea	r Optical Characterisation	171
	4.5.11	Conclusio Crystal an	on of the Comparison between NdLNB nd NdLNB Glass	172
4.6	A Con	nparative H	Evaluation Summary	173

CHAPTER 5		CONCLUSION AND RECOMMENDATIONS	177
	5.1	Introduction	177
	5.2	Conclusion	177
	5.3	Future Works	179
REFEREN	NCES		181
APPENDIX A-H		195	
LIST OF PUBLICATIONS		209	

## LIST OF TABLES

TABLE NO.	TITLE	PAGE
Table 2.1	Mechanisms of nucleation	14
Table 2.2	Development in the research field of borate glasses crystallisation	16
Table 2.3	Growth conditions of some borate crystals	32
Table 2.4	The effect of transition metal (TM) and heat treatment (HT) temperature and time on the volume and crystallite size for different phases (Kashif et al., 2013)	41
Table 2.5	The glass density and molar volume of various borate glass systems	43
Table 2.6	Classification of infrared radiation (Bokobza, 1998)	44
Table 2.7	Assignment of infrared bands in the spectra (Gautam et al., 2012)	48
Table 3.1	The composition of NdLNB glasses	74
Table 3.2	Crystal growth condition and parameters for growth crystal using ADC system	80
Table 3.3	Details of growth conditions used in the experiment	83
Table 4.1	Nominal composition of NdLNB glass system	90
Table 4.2	Density of NdLNB glass system	91
Table 4.3	Molar volume of NdLNB glass system	93
Table 4.4	Nominal weight (mol%) and actual weight (mol%) of oxide content in (90-x)Li <sub>2</sub> B <sub>4</sub> O <sub>7</sub> +10Nb <sub>2</sub> O <sub>5</sub> +xNd <sub>2</sub> O <sub>3</sub> glass system obtained from EDX spectral analyses	96
Table 4.5	Thermal parameters value of NdLNB glass system	99
Table 4.6	Infrared band assignments and peak position (in cm <sup>-1</sup> ) for $Nd^{3+}$ in the (90-x)Li <sub>2</sub> B <sub>4</sub> O <sub>7</sub> +10Nb <sub>2</sub> O <sub>5</sub> +xNd <sub>2</sub> O <sub>3</sub> glass system	102
Table 4.7	Absorption peak wavelength and corresponding wavenumber of glass system	105

Table 4.8	Calculated optical band gap energy of the NdLNB glass system	108
Table 4.9	Refractive index for the NdLNB glass system	109
Table 4.10	Calculated Urbach energy of the NdLNB glass system	111
Table 4.11	Observed band positions (cm <sup>-1</sup> ) and bonding parameters, $\beta$ and $\delta$ of Nd <sup>3+</sup> in the NdLNB glass system	114
Table 4.12	Oscillator strength experimental $(f_{exp})$ and calculated $(f_{cal})$ (×10 <sup>-3</sup> ) of Nd <sup>3+</sup> in the NdLNB glass system	117
Table 4.13	Judd-Ofelt parameters ( $\Omega_{\lambda} \times 10^{-18} \text{ cm}^2$ ) and spectroscopic quality factor, $Q$ of Nd <sup>3+</sup> in the NdLNB glass system	118
Table 4.14	Average electric dipole, $A_{ed}$ (×10 <sup>-6</sup> s <sup>-1</sup> ) and radiative lifetime, $\tau_{rad}$ (×10 <sup>-6</sup> ms <sup>-1</sup> ) of Nd <sup>3+</sup> in NdLNB glass system	121
Table 4.15	Peak wavelength $\lambda p$ (nm), effective emission band width $\Delta \lambda eff$ (nm), stimulated emission cross-section ( $\sigma_P^E$ ) (×10 <sup>-15</sup> cm <sup>2</sup> ) and gain bandwidth ( $\Delta G$ ) (×10 <sup>-20</sup> cm <sup>3</sup> ) of Nd <sup>3+</sup> in NdLNB glass system for near infrared emission.	125
Table 4.16	A summary of NdLNB crystal growth run with different types of seed	132
Table 4.17	Parameters for Czochralski growth of NdLNB crystal using polycrystal seed	133
Table 4.18	Different experiments on crystal growth of NdLNB crystal using polycrystal seed	134
Table 4.19	Parameters for Czochralski growth of NdLNB crystal using platinum wire seed	135
Table 4.20	Different experiments on crystal growth of NdLNB crystal using platinum wire seed	135
Table 4.21	Parameters for Czochralski growth of NdLNB crystal using LN crystal seed	136
Table 4.22	Different experiments on crystal growth of NdLNB crystal using Lithium Niobate crystal seed	137
Table 4.23	A summary of the maximum control power and start pull power for NdLNB-poly, NdLNB-pt, and NdLNB-LN crystals during the seeding process	143
Table 4.24	Density, molar volume and oxygen packing density of NdLNB glass and crystal	149
Table 4.25	Nominal weight (mol%) and actual weight (mol%) of oxide content in NdLNB glass and crystal obtained from EDX spectral analyses	152

Table 4.26	Infrared band assignments and peak position (in cm <sup>-1</sup> ) of the NdLNB glass and NdLNB crystal	154
Table 4.27	Absorption peak wavelength and corresponding wavenumber of NdLNB glass and NdLNB crystal	157
Table 4.28	Calculated indirect and direct optical band gap energy, Urbach energy and refractive index of NdLNB glass and NdLNB crystal	160
Table 4.29	Observed band positions (cm <sup>-1</sup> ) and bonding parameters, $\beta$ and $\delta$ of NdLNB glass and NdLNB crystal	161
Table 4.30	Oscillator strength experimental $(f_{exp})$ and calculated $(f_{cal})$ (×10 <sup>-3</sup> ) of NdLNB glass and NdLNB crystal	163
Table 4.31	Judd-Ofelt parameters ( $\Omega_{\lambda} \times 10^{-18} \text{ cm}^2$ ) and spectroscopic quality factor, $Q$ of NdLNB glass and NdLNB crystal	163
Table 4.32	Average electric dipole, $A_{ed}$ (×10 <sup>-6</sup> s <sup>-1</sup> ) and radiative lifetime, $\tau_{rad}$ (×10 <sup>-6</sup> ms <sup>-1</sup> ) of NdLNB glass and NdLNB crystal	165
Table 4.33	Peak wavelength $\lambda_P$ (nm), effective emission bandwidth $\Delta\lambda_{eff}$ (nm), stimulated emission cross-section ( $\sigma_P^E$ ) (×10 <sup>-15</sup> cm <sup>2</sup> ), and gain bandwidth ( $\Delta G$ ) (×10 <sup>-20</sup> cm <sup>3</sup> ) of NdLNB crystal and NdLNB glass for near infrared emission.	168
Table 4.34	Comparative evaluation of NdLNB crystal and NdLNB glass system properties	175

## LIST OF FIGURES

FIGURE NO.	TITLE	PAGE
Figure 2.1	Schematic representation of glass crystallisation (McMillan, 1964)	14
Figure 2.2	Bragg law for the periodic arrangement atoms (Ladd and Palmer, 1977)	37
Figure 2.3	XRD pattern for the as-quenched (a,b) and the sample heat- treated at 500 °C for 3 h (c-f) (Prasad and Varma, 2005a)	38
Figure 2.4	Typical DTA curves of tellurite glasses (Kim et al., 1996)	40
Figure 2.5	The vibrational stretching and bending modes (Bokobza, 1998)	46
Figure 2.6	IR spectra of MgO+Li <sub>2</sub> O+B <sub>2</sub> O <sub>3</sub> glasses doped with Nd <sub>2</sub> O <sub>3</sub> (Mhareb et al., 2014)	47
Figure 2.7	Three regions of absorption coefficient vs photon energy (Tauc, 1974)	50
Figure 2.8	Tauc plot of indirect optical band gap $(ahv)^{1/2}$ and direct optical band gap $(ahv)^2$ versus photon energy, $hv$ of Eu <sup>3+</sup> doped sodium-lead-zinc-lithium-borate glasses (Rajagukguk et al., 2016)	51
Figure 2.9	Direct $(E_g^D)$ and indirect optical energy band gap $(E_g^I)$ . Note that, direct band gap occurs at k = 0, while indirect band gap occurs at k $\neq$ 0 (Zelewski and Kudrawiec, 2017)	52
Figure 2.10	Urbach tails of localised states in the band gap (Choudhury et al., 2013)	53
Figure 2.11	Schematic of typical photoluminescence process in optically excited semiconductor a) excitation, b) interband relaxation and c) recombination (Ameruddin, 2015)	59
Figure 2.12	Basic schematic of excitation and emission process in a down conversion process (Lumb, 1978)	60
Figure 2.13	Luminescence spectra of $Nd^{3+}$ in $67B_2O_3+xLi_2O+(32-x)Na_2O$ (x=8, 12, 16, 20 and 24) glasses (Ratnakaram et al., 2004)	61
Figure 2.14	(a) SEM image and (b) EDX spectra of LFB glass (Balakrishna et al., 2013)	64
Figure 2.15	Transmission electron micrographs along with the SAED patterns recorded for the (a) as-quenched, (b) 460 °C for 6 h	) L

	and 500 °C for 3 h and (c) 500 °C for 3 h heat treated of	
	85LiBO <sub>2</sub> +15Nb <sub>2</sub> O <sub>5</sub> glasses (Prasad and Varma, 2005b)	66
Figure 2.16	Schematic of the experimental setup for determining nonlinear coefficients of materials in powder form by SHG measurement: (1) Nd:YAG laser, (2) 90 ° prism, (3) focusing lens, (4) sample of powder on the substrate, (5) a table with fiber mount, (6) quartz optical fiber, (7) spectrometer (Morozov et al., 2015)	68
Figure 2.17	Schematic representations for the research gap of the current study	71
Figure 3.1	The schematic diagram of glass sample preparation	75
Figure 3.2	Flow of glass preparation	76
Figure 3.3	Flow chart of polycrystal seed preparation	78
Figure 3.4	The first 13 control parameters of the Automatic Diameter Control (ADC) system	79
Figure 4.1	Density versus Nd <sub>2</sub> O <sub>3</sub> concentration	92
Figure 4.2	Molar volume versus Nd <sub>2</sub> O <sub>3</sub> concentration	93
Figure 4.3	XRD patterns of NdLNB glass system	95
Figure 4.4	EDX spectrum of NdLNB glass system	96
Figure 4.5	DTA curves of NdLNB glass system	97
Figure 4.6	Thermal stability against Nd <sub>2</sub> O <sub>3</sub> concentration	100
Figure 4.7	Glass forming tendency against Nd <sub>2</sub> O <sub>3</sub> concentration	101
Figure 4.8	FTIR spectra of the NdLNB glass system	102
Figure 4.9	Absorption spectra of NdLNB glass system	105
Figure 4.10	Plot of $(\alpha hv)^{1/2}$ vs $hv$ for indirect band gap of the NdLNB glass system	107
Figure 4.11	Plot of $(\alpha hv)^2$ vs $hv$ for direct band gap of the NdLNB glass system	107
Figure 4.12	Indirect band gap energy vs Nd concentration	108
Figure 4.13	Direct, $E_g^D$ optical band gap energy vs Nd concentration	109
Figure 4.14	Graph of $ln \alpha$ against $hv$ of the NdLNB glass system	110

Figure 4.15	Urbach energy vs Nd concentration	112
Figure 4.16	Bonding parameter vs Nd concentration	115
Figure 4.17	Luminescence spectra of NdLNB glass system	122
Figure 4.18	Energy level diagram of NdLNB glass system	124
Figure 4.19	DTA thermogram for NdLNB glass	127
Figure 4.20	Picture of (a) NdLNB glass seed and (b) NdLNB polycrystal seed	128
Figure 4.21	XRD patterns for NdLNB (a) as-quenched, (b) 580 °C, and (c) 600 °C heat-treated samples	128
Figure 4.22	SEM image for NdLNB as-quenched glass (a and c) and NdLNB 600 °C heat-treated glass (b and d) at different magnifications	131
Figure 4.23	The variation of control power and growth rate against elapsed time for (a) NdLNB-poly, (b) NdLNB-pt, and (c) NdLNB-LN crystals during the seeding process	140
Figure 4.24	The variation of control power and diameter against elapsed time for (a) NdLNB-poly, (b) NdLNB-pt, and (c) NdLNB-LN crystals during the seeding process	141
Figure 4.25	The variation of control power and diameter against elapsed time for (a) NdLNB-poly, (b) NdLNB-pt, and (c) NdLNB-LN crystals during the crystal body growth	144
Figure 4.26	The variation of diameter and growth rate against elapsed time for (a) NdLNB-poly, (b) NdLNB-pt, and NdLNB-LN (c) crystal during the crystal body growth	146
Figure 4.27	XRD patterns for (a) NdLNB glass, and (b) NdLNB crystal	150
Figure 4.28	EDX spectrum of NdLNB glass	151
Figure 4.29	EDX spectrum of NdLNB crystal	152
Figure 4.30	DTA curves for NdLNB glass and NdLNB crystal	153
Figure 4.31	FTIR spectra of the NdLNB glass and NdLNB crystal	154
Figure 4.32	Absorption spectra of NdLNB glass and NdLNB crystal	156
Figure 4.33	Plot of $(\alpha h v)^{1/2}$ vs $h v$ for indirect band gap of the NdLNB glass and NdLNB crystal	158
Figure 4.34	Plot of $(\alpha hv)^2$ vs $hv$ for direct band gap of the NdLNB glass and NdLNB crystal	159
Figure 4.35	Graph of $ln \alpha$ against $hv$ of the NdLNB glass and NdLNB crystal	160

Figure 4.36	Luminescence spectra of NdLNB glass and NdLNB crystal under excitation at 355 nm. Inset shows the comparison energy level of the Nd <sup>3+</sup> ions transitions for NdLNB crystal and NdLNB glass	167
Figure 4.37	(a) TEM image, (b) and (d) d-spacing of $Li_2B_4O_7$ at (112) plane, and (c) SAED pattern of NdLNB crystal	169
Figure 4.38	SEM image of NdLNB as-quenched glass (a and c) and NdLNB crystal (b and d) at different magnifications	170
Figure 4.39	Emission spectrum of SHG of the NdLNB glass	171
Figure 4.40	Emission spectrum of SHG of the NdLNB crystal	172

## LIST OF ABBREVIATIONS

ADC-CGS	-	Automatic diameter control - crystal growth system
BBO	-	Beta-barium borate
BF	-	Bright field
BO	-	Bridging oxygen
BSE	-	Back-scattered electrons
CB	-	Conduction band
СВО	-	Cesium triborate
CLBO	-	Cesium lithium borate
CR	-	Cross relaxation
DF	-	Dark field
DP	-	Diffraction pattern
DTA	-	Differential thermal analysis
EDX	-	Energy dispersive X-ray
FTIR	-	Fourier transform infrared
FWHM	-	Full width half maximum
HT	-	Heat treatment
IR	-	Infrared
JCPDS	-	Joint Committee on Powder Diffraction Standards
J-O	-	Judd-Ofelt
KAB	-	Potassium aluminum borate
KDP	-	X-Ray diffraction
KTP	-	Potassium titanyl phosphate
LBO	-	Lithium triborate
LCOB	-	Lithium calcium oxoborate
LFB	-	Lithium Fluoro Borate
Li	-	Lithium
LTB	-	Lithium tetraborate
Nb	-	Niobium
NBO	-	Non-bridging oxygen
Nd	-	Neodymium

NdLNB	-	Neodymium lithium niobium borate
NLO	-	Nonlinear optical
NR	-	Non-radiative
0	-	Oxygen
PL	-	Photoluminescence
PR	-	Pulling rate
R	-	Radiative
RE	-	Rare earth
RECOB	-	Rare earth calcium oxyborate
RS	-	Rotation speed
SADP	-	Selected area diffraction pattern
SAED	-	Selected area electron diffraction
SAW	-	Surface acoustic waves
SE	-	Secondary electrons
SEM	-	Scanning electron microscopy
SFD	-	Self-frequency doubling
SHG	-	Second harmonic generation
TEM	-	Transmission electron microscopy
ТМ	-	Transition metal
TSSG	-	Top seeded solution growth
UV-Vis -IR	-	Ultraviolet visible infrared
VB	-	Valence band
XRD	-	X-Ray diffraction
YAB	-	Yttrium aluminum borate
YAG	-	Yttrium aluminium garnet
YCOB	-	Yttrium calcium oxoborate

## LIST OF SYMBOLS

A	-	Absorbance
$A_{ed}$	-	Electric-dipole
$A_{md}$	-	Magnetic-dipole
α	-	Absorption coefficient
В	-	Constant
β	-	Nephelauxetic ratio
$ar{eta}$	-	Average nephelauxetic ratio
С	-	Speed of light
d	-	Thickness of the sample
D	-	Diameter
$E_u$	-	Urbach energy
$E_{f}$	-	Energy of electron of final state
$E_i$	-	Energy of an electron at lower band
$E_g^D$	-	Direct optical energy band gap
$E_g^I$		Indirect optical energy band gap
$\varepsilon(v)$	-	Molar extinction coefficient
e	-	Electron charge
F	-	Force constant
fexp	-	Experimental oscillator strength
$f_{cal}$	-	Oscillator strength
$\Delta G$	-	Gain bandwidth
hv	-	Photon energy
$H_R$	-	Hruby parameter
λ	-	Wavelength
$\Delta \lambda_{eff}$	-	Effective gain bandwidth emission
$arOmega_\lambda$	-	Judd-Ofelt parameters
т	-	Electron mass
$m_r$	-	Atomic weights in kg of cation
$m_o$	-	Atomic weights in kg of anion
μ	-	Reduced mass

n	-	Refractive index
NA	-	Avogadro number
OPD	-	Oxygen packing density
θ	-	Angle
ρ	-	Density
$ ho_L$	-	Density of distilled water
Р	-	Pulling rate
$ ho_s$	-	Crystal density
$\rho_l$	-	Melt density
Q	-	Quality factor
rms	-	Root-mean-square
R	-	Crucible radius
$\sigma_P^E$	-	Stimulated emission cross-section
$S_{ed}$	-	Line-strength for electric
δ	-	Bonding parameter
t	-	Crystallite size
$T_c$	-	Glass crystallization temperature
$T_g$	-	Glass transition temperature
$T_m$	-	Glass melting temperature
$\Gamma_M$	-	Decay rate
$\Gamma_{NR}$	-	Non-radiative decay
$\Gamma_R$	-	Intrinsic radiative decay rate
$ au_{rad}$	-	Radiative lifetime
$\Delta T$	-	Glass thermal stability
$U_{\kappa}$	-	Values of reduced matrix elements
v	-	Wavenumber
Vg	-	Wavenumber of particular transition to an ion
$v_a$	-	Wavenumber of same transition for aquo ion
$V_m$	-	Molar volume
$W_1$	-	Weight of sample in air
$W_2$	-	Weight in distilled water
X <sub>i</sub>	-	Mole fraction

### LIST OF APPENDICES

APPENDIX	TITLE	PAGE
Appendix A	The example calculation of the glass composition according to the 1 mol%	195
Appendix B	Crystal growth machine and insulation set up	196
Appendix C	The pictures of the instruments used for material characterisation	198
Appendix D	Plot of $(\alpha \hbar v)^2$ vs $\hbar v$ for direct band gap of the NdLNB glass system	203
Appendix E	The example calculation of Urbach energy for NdLNB glass sample	204
Appendix F	Picture of as-quenched NdLNB glass with composition of 1 mol%	205
Appendix G	Pictures of NdLNB crystals grown by Czochralski technique using different types of seeds	206
Appendix H	The calculation of crystallite size for NdLNB crystal	208

#### **CHAPTER 1**

#### **INTRODUCTION**

#### **1.1 Background of the Study**

Solid state materials can be classified as crystals, polycrystals (ceramics), or amorphous depending on their arrangement of constituent molecules, atoms, or ions. Several materials can form a crystal with the same composition but in different arrangements of the atoms which create different three-dimensional structures (Myerson, 2002). Non-crystalline solid, often referred to as amorphous solid, is the opposite extreme of a crystal. This type of solids has neither reticular nor granular structure. Amorphous solids possess short-range order but are devoid of any organised structure over longer distances; in this respect they resemble liquids. However, their rigidity and cohesiveness allow them to retain a definite shape, so for most practical purposes, they can be considered solids. In fact, the only obvious distinction between amorphous materials, such as glass, and liquids is the high viscosity (resistance to flow) of the amorphous solids.

The glass composition is extremely important in the formation of glass. Generally, glass is composed of different oxides that serve various functions, particularly in research and industry. The presence of strongly bonded large networks of atoms in the liquid is a basic condition for glass formation. A superior glass must have many bonds or linkages of the types with high bond strengths like B–O–B, Si–O, Ge–O, and P–O as glass formers. Some oxides, such as borate (B<sub>2</sub>O<sub>3</sub>), silicate (SiO<sub>2</sub>), germinate (GeO<sub>2</sub>), and phosphate (P<sub>2</sub>O<sub>5</sub>) are defined as glass formers because they are capable to form glassy network by themselves under normal quenching conditions but act as glass formers when mixed with others, such as ZnO, PbO, MgO, CaO, and BaO (Gautam et al., 2012).

A material can be transformed into a crystal by slow and gradual transformation from solid, liquid/solution, or vapour phase. Several techniques adopting one of these three basic methods can be employed for growing from small to big crystals (Brice, 1986). Selection of appropriate techniques of crystal growth is highly dependent on the chemical and physical properties of the material to be grown and the suitability of the technique to grow the required crystal (Seevakan and Bharanidharan, 2018). This is because each technique to be used has certain parameters to be considered for the success of crystal growth such as growth kinetics, chemical reactivity, temperature gradient, solubility, shape, purity, decomposition, melting point, and the cost involved (Brian, 1980).

Presently, borate glass and crystal are receiving much more attention due to their applications in technologies such as solid-state lasers, nonlinear optics, and solar energy (Alajerami et al., 2012). Borate is one of the popular glass formers because it can easily combine with a variety of oxides to form a binary glass system (Kaur et al., 2014). Borate network building units can be either triangles (BO<sub>3</sub>) with non-bridging atoms or tetrahedral (BO<sub>4</sub>) with all bridging oxygen atoms, making it an interesting system. In comparison with other conventional glasses, borate glass is most recently analysed due to the superior optical and mechanical properties, chemical durability, stability against atmospheric moisture, good solubility of rare earth ions, low melting temperature, and good corrosion resistance (Wu et al., 2011). Moreover, borate glasses and crystals are suitable hosts for various transition metals and rare earth ions, making them suitable for optical materials.

Several attempts have been made to improve the values of borate glass and crystal. It is possible to change the coordination geometry of boron from BO<sub>3</sub> to BO<sub>4</sub> by adding alkali oxides (Balachander et al., 2013). As a modifier, lithium which is more electropositive, will cause essential changes in the binary lithium borate system, such as enhancing the bonding strength by forming ionic bonds with oxygen (non-bridging oxygen) and decreasing the hygroscopic nature of borate (Bhogi et al., 2015). Lithium borate is one of the most useful nonlinear optical materials for ultraviolet and visible laser applications. Laser systems with lithium borate as a key component in their design are widely utilised in applications like ophthalmology, materials

processing, marking, optical data storage, and semiconductor manufacturing. Several transition metals including Nb, V, Ti, and Cr are employed as second modifiers to change the glass structure, create non-bridging oxygen (NBO), enhance bond strength, and reduce glass stickiness (Bhagat, 2003). The chemical and physical properties of the glasses are altered when NBO is created. However, the modifications continue to rely on their connectivity to change these properties.

Rare earth (RE)-doped glasses and crystal materials have been extensively studied for fabricating lasers, semiconductors, and amplifiers (Kumar et al., 2013a). RE ions are used to investigate local structural changes in the materials due to their unique spectroscopic properties upon the optical transitions in the intra 4f-shell (Semwal and Bhatt, 2013). Since the demonstration of laser activity in neodymium single crystals made by Krupke (1971), Nd<sup>3+</sup> has been widely used as a laser-active ion in amorphous and crystalline states (Kumar et al., 2013a). The two host materials most commonly used for this laser ion are yttrium aluminium garnet (YAG) and glass. When doped in YAG, the Nd:YAG crystal produces laser output primarily at 1.064  $\mu$ m. However, when doped in glass, the Nd:glass medium lases at wavelengths ranging from 1.054 to 1.056 µm, depending upon the type of glass used. In glass, Nd can be doped to extremely high concentration, but it is limited to a maximum concentration of 1.0%–1.5% in YAG crystals (Kuhn, 1998). Furthermore, owing to the commercial importance of Nd<sup>3+</sup> doped glasses and crystal lasers, many studies on the optical properties and structural role of Nd<sup>3+</sup> ion and its interaction with other ions have been carried out (Anjaiah et al., 2015; Mhareb et al., 2014; Pal et al., 2013; Vijaya and Suresh, 2012).

Looking at the great prospect of lithium niobium borate containing neodymium in the field of material science, the physical, structural, and optical properties of these materials need to be further investigated.

### **1.2 Problem Statement**

Several studies have been reported regarding the preparation of lithium borate glasses (Prasad and Varma, 2005a; Graca et al., 2008; Kashif et al., 2012; Khalek et al., 2012; Kashif et al., 2013; Farouk et al., 2015). The physical, structural, optical, and electrical properties of the glass systems show a great potential host for nonlinear optical (NLO) and lasing materials. Looking at the great prospect of rare earth doped lithium borate crystal for NLO and laser applications, it is necessary to develop an accurate method for their growth.

Based on the previous studies, Mhareb et al. (2014) has reported the influence of Nd on lithium-magnesium-borate glasses, Kashif et al. (2013) has studied the effect of V, Mn, Co, and Cu on lithium-niobium-borate glasses, and Wu et al. (2011) has reported the influence of Nd on lithium-borate glasses. Those studies only reported the preparation of the glasses using the melt-quench technique, and not much effort has been given to grow a polycrystal seed from melt-quench. Usually, the crystal seeds are made from the Czochralski technique, which is the common practise of crystal growth. (Kashchiev, 2000). However, neodymium-doped lithium-niobium-borate via seeding technique has not been performed yet. Besides that, most of the reported work is limited to the presence of LiNbO<sub>3</sub> nanocrystals by a controlled heat treatment process but emphasis has not been laid down on the formation of crystals by using a seeding technique. Thus, it had become desirable to undertake systematic studies to fully explore the impact of seeding on the modified properties. Moreover, a well-controlled crystal growth process with appropriate growth parameters involving temperature gradient, growth rate, pulling rate, and rotating speed must be achieved in order to produce a good crystal. The physical, thermal, structural, optical, and SHG properties of the crystals and glasses will also be reported.

### **1.3** Research Objectives

In order to solve the problem as stated in Section 1.2, several objectives have been outlined as follows:

- (a) To grow neodymium modified lithium-niobium-borate polycrystal seed via melt-quench assisted glass route and thermal annealing.
- (b) To determine the crystallisation behaviour and microstructural features of the neodymium-lithium-niobium-borate glass and polycrystal.
- (c) To optimise the growth mechanism of neodymium-lithium-niobium-borate crystals by employing polycrystal, platinum and lithium niobate crystal seeds.
- (d) To determine the improvement in physical, thermal, structural, optical and SHG properties of the crystals and glasses.

### **1.4** Scope of the Study

In this study, the composition of  $(90-x)Li_2B_4O_7+10Nb_2O_5+xNd_2O_3$  glass system, where x = 1, 5, 10, 15, and 20 were prepared by conventional melt-quenching technique. The borate glass is chosen as a host due to the high stability and high forming ability of glass. For the preparation of polycrystal seed and crystal growth, the primary focus was only on 1 mol% of Nd<sub>2</sub>O<sub>3</sub>. Neodymium oxide was used as a modifier in order to investigate the impact on the physical, structural and optical properties of the glasses and crystals. Consequently, the heat treatment procedures were executed to crystallise the nucleated glass to produce NdLNB polycrystal seed. X-ray diffraction (XRD) and Scanning Electron Microscopy (SEM) were then used to ascertain the crystallisation behaviour and microstructural features of the glass and polycrystal before it will be applied for the next crystal growth process. NdLNB crystals were grown by various types of seed such as polycrystal seed that was prepared in the previous stage, platinum wire and lithium niobate single crystal seed with Automatic Diameter Control - Crystal Growth System (ADC-CGS). The growth and growth mechanism were described, along with the challenges likely to be encountered during growth of the crystal. NdLNB crystals and glasses were subjected to various characterisation techniques and their properties were determined. The densities were measured by Archimedes method. X-ray diffraction (XRD), Differential thermal analyser (DTA), Fourier transformed infrared (FTIR), Ultraviolet-Visible (UV-Vis), Photoluminescence (PL) spectroscopy, Scanning Electron

Microscopy (SEM) and Transmission Electron Microscopy (TEM) characterisations were made to examine the physical, thermal, structural and optical properties. Measurements of the SHG of the crystal and glass samples were performed using the SHG powder technique test set up based on a method of Kurtz and Perry (1968).

#### **1.5** Significance of the Study

This research will give a novelty to the crystal growth process by using a polycrystal seed from an amorphous material and thus inventing a new NLO and lasing material as well. This invention also has the potential to be applied in growing other brand-new glass forming melt crystal such as phosphate, tellurite and silicate which are used for efficient harmonic generation, frequency conversion, laser production, and in other important applications related to nonlinear optics and its associated technologies. Besides, it can nurture knowledge and create new specialists in this area. Moreover, this crystal growth technology is progressively in advance in almost all developed countries. But, unfortunately not much effort has been made in Malaysia to exploit this technology specifically in Czochralski crystal growth technique. Considering its significance, this study has been initiated.

#### **1.6** Thesis Organisation

In this thesis, there are five chapters. Chapter 1 presents the background, problem statement, objectives, and significance of the study. A literature review is presented in Chapter 2. It provides the basis of general information about borate glasses, crystallisation of borate glasses and borate crystals. In addition, a brief description of the crystal growth is highlighted. Nevertheless, the reviews of measurement methods and analysis of each characterisation instrument are also reported. Chapter 3 describes the experimental and measurement techniques, which include sample preparation of glass by the melt-quenching technique, polycrystal by the heat-treatment process, and crystal by the Czochralski technique, and the apparatus used for characterisation and analysis such as DTA, XRD, FTIR, UV-VIS, PL, SEM-

EDX, TEM, and SHG tests. Chapter 4 consists of the experimental results and discussions. This chapter is divided into four sub-sections: the first part describes the synthesis and characterisation of the glass system, the second part explains the characterisation of polycrystal seeds, the third part describes the growth and growth mechanism of the crystals, and the last part presents the comparison of crystal and glass properties. Finally, Chapter 5 summarises the main findings achieved through this research, and suggests several recommendations for future studies. Additional information associated with the thesis is given in the Appendices.

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#### LIST OF PUBLICATIONS

#### Journal with Impact Factor

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### **Indexed Journal**

 Kamaruddin, W. H. A., Rohani, M. S., Sahar, M. R. and Liu, H. (2017) Effect of Nd<sup>3+</sup> on the properties of lithium niobium borate crystal and glass, *Solid State Phenomena*, 268, pp. 210-216. (Indexed by SCOPUS)

#### **Non-Indexed Conference Proceedings**

- Kamaruddin, W. H. A., Rohani, M. S., Sahar, M. R. and Liu, H. (2018) Glass formation and crystalline phase in the ternary Nd<sub>2</sub>O<sub>3</sub>-Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub>-Nb<sub>2</sub>O<sub>5</sub> system, 30th Regional Conference on Solid State Science and Technology, Melaka, Malaysia.
- Kamaruddin, W. H. A., Rohani, M. S., Sahar, M. R. and Liu, H. (2017) Polycrystalline seed-mediated growth of neodymium-lithium-niobium-borate crystal by czochralski technique, 1st International Malaysia-Indonesia-Thailand Symposium, UiTM Perlis, Malaysia. pp. 295-299.
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Modification in structural and optical properties of undoped and Ce, Nd, doped lithium niobium borate glasses, 5th International Conference on Solid State Science and Technology, Langkawi, Malaysia.