# PHYSICAL AND STRUCTURAL PROPERTIES OF Nd:YAG CRYSTAL GROWTH BY CZOCHRALSKI METHOD

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BY CZOCHRALSKI METHOD

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Especially dedicated:

To my ever encouraging, supportive, and devoted

Family and Friends

*Thank you for being a huge inspiration for my success*

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**ABSTRACT**

Neodymium doped Yttrium Aluminium Garnet, Nd:YAG crystal with nominal dopant concentration of 1.4 at. % was successfully grown by Czochralski technique equipped with an Automatic Diameter Control – Crystal Growth System (ADC-CGS). Correlation between dopant concentrations with the length of crystal boule in relation with physical and structural properties was studied. It was found that the crystal was partially transparent with some visible macroscopic defects such as cracks, gas pores, and inclusion. Its physical appearance is strongly affected by heat zone design. From EDX analysis, it was found that the doping concentration of $Nd^{3+}$ ion increased as the crystal boule became longer than its initial pulling point. From the top to the bottom of Nd:YAG crystal boule, the concentration of $Nd^{3+}$ changed from 0.13 at. % to 0.65 at. %. The density of the samples was determined by Archimedes method which showed an increasing trend of density with the crystal length which was found to be in the range of $4.5344\pm0.0153$ $gcm^{-3}$ to $4.5628\pm0.0114$ $gcm^{-3}$. It was also discovered that Vickers hardness increased with increasing dopant concentration which was from 1590 Hv to 1776 Hv. Raman spectrum was obtained in the range of 100 $cm^{-1}$ – 1000 $cm^{-1}$. From the spectra, the intensity was found to vary and shifting occured in the band due to the $Nd^{3+}$ dopant concentration. Meanwhile, the IR spectra shows absorption occurring around 2000 $cm^{-1}$ to 4500 $cm^{-1}$. Absorption tends to be greater with increasing dopant concentration. Furthermore, the effect of melts level to the growth process has also been studied and it is suggested that the crucible should be lifted up according to the level of the melts drop during growing process to maintain the temperature gradient of the hot zone.

**ABSTRAK**

Hablur Yttrium Aluminium Garnet didop Neodimium (Nd:YAG),dengan nilai nominal kepekatan dopan sebanyak 1.4 at. % telah berjaya ditumbuhkan dengan kaedah Czochralski yang dilengkapi dengan sistem Pengawalan Diameter Automatik – Sistem Pertumbuhan Hablur (ADC-CGS). Perkaitan antara kepekatan dopan dengan kedudukan panjang tongkol hablur dari segi sifat-sifat fizikal dan struktur telah dikaji. Didapati bahawa hablur adalah separa lutsinar dengan sedikit kecacatan makroskopik yang boleh dilihat seperti retak, gelembung dan rangkuman bendasing. Bentuk fizikalnya sangat dipengaruhi oleh rekabentuk zon haba. Berasaskan analisis EDX, kepekatan ion dopan $Nd^{3+}$ meningkat apabila tongkol hablur bertambah panjang dari titik permulaan penarikannnya. Dari bahagian atas ke bawah tongkol hablur Nd:YAG, berlaku perubahan kepekatan $Nd^{3+}$ dari 0.13 at. % kepada 0.65 at. %. Ketumpatan sampel telah ditentukan melalui kaedah Archimedes dan menunjukkan arah aliran yang meningkat dengan pemanjangan tongkol hablur dan berada dalam julat $4.5344\pm0.0153$ $gcm^{-3}$ hingga $4.5628\pm0.0114$ $gcm^{-3}$. Didapati juga kekerasan Vickers hablur meningkat dengan peningkatan kepekatan dopan iaitu dari 1590 Hv ke 1776 Hv. Spektrum Raman telah diukur dalam lingkungan 100 $cm^{-1}$ – 1000 $cm^{-1}$. Dari spektra tersebut, keamatan berubah dan anjakan berlaku dalam jalur spektra akibat kepekatan ion $Nd^{3+}$. Sementara itu, spektra IR menunjukkan penyerapan berlaku sekitar 2000 $cm^{-1}$ hingga 4500 $cm^{-1}$. Penyerapan cenderung menjadi lebih besar dengan peningkatan dopan. Tambahan pula, kesan paras leburan untuk proses pertumbuhan juga turut dikaji dan disarankan bahawa krusibel patut dinaikkan mengikut aras penurunan leburan semasa proses pertumbuhan untuk mengekalkan kecerunan suhu zon panas.

# TABLE OF CONTENTS

# LIST OF TABLES

# LIST OF FIGURES

# LIST OF SYMBOLS

| | | |
|---|---|---|
| $\rho$ | | Density of sample |
| $\rho_L$ | - | Density of water |
| $\rho_A$ | - | Density of air |
| A | | Area |
| $W_A$ | - | Weight in air |
| $W_L$ | - | Weight in water |
| $\lambda$ | - | Wavelength of the X-ray |
| (*hkl*) | - | Crystal plane |
| $\theta$ | - | Angle of diffraction |
| *d*-spacing | - | Interatomic spacing between atom |
| *a* | - | Lattice parameter |

# CHAPTER I

# INTRODUCTION

## 1.1    Research Background

Crystal growth is a new industry but an old topic. In the early age of civilization, peoples already discover this crystal growth technique (extract salt from sea water). Evident show that it is possible to map out work back to 2500 $_{BC}$ when salt was purified by crystallization. Systematic work in the field goes back $_{AD}$ 1600 (aqueous solution growth), 1800 (high-temperature solution growth), 1850 (melt growth and vapor growth) (Buckley, 1951).

Crystal growth involves a variety of research fields ranging from surface physics, crystallography, and material sciences to condenser matter physics. Despite the fact that it has been studied broadly more than 100 years, crystal growth still plays a vital role in both theoretical and experimental research fields, plus in applications. As the improvement of scientific instruments and analytical methods, such as X-rays, electron microscopy, NMR, and scanning tunneling microscopy continues, research on crystal growth and structure characterization has entered an atomic level, which makes it promising for further understanding of the physical, chemical, and other properties of the structures nature of various crystals. In

addition, a further improvement of crystal quality also depends on the structure characterizations (Zhang, 1999).

A single crystal is defined as a long range atomic order that extends over many atomic diameters and has a repetitive structure. As the crystal growth, differences in temperature and chemical compositions cause variations. The internal arrangement of atom determines all the mineral's chemical and physical properties (Glazer, 1987). Decades ago, crystals were classified according to their morphology properties which have a similar manner for object in biological term. Definition of a crystal always comes as a homogenous space with directionally dependent properties or anisotropy. This term no longer satisfactory because other material such as glass and plastic may also possesses anisotropy properties. Hence, an ideal crystal is understood as a space containing a rigid lattice arrangement of uniform atomic cells. Crystal can be classified as real crystal if they are nature existed. Main criteria can be ranging from their rigid lattice arrangement and from uniform atomic cell structure (Kosevich, 2005).

These days, crystals are produced synthetically to assure the needs of science, technology and jewellery. The aptitude to grow high quality crystals has become an essential criterium for the competitiveness of nations (Feigelson, 2004). The Geneva rubies which appeared in about 1904 are probably the first case of commercial exploitation. They were almost certainly grown by the Verneuill process. These rubies were used primarily for jewellery but their hardness made them suitable for precise bearings (in clocks and watches), which became their main use because jewellers were hesitant to use synthetic gems. Until quite recently the production of rubies for bearings and stressed components was a big industry. Nevertheless, advances in metallic bearing materials and the latest development of electronic watches flawed this market and the current major use of rubies is jewellery (Brice, 1986).

During World War II crystals were widely used as piezo-electric transducer (for Sonar), as resonant devices to control radio frequencies, in point contact diodes (for radar and other high frequency applications) and in beginnings of the military infrared applications. While the war initiated the change from crystals for science to crystal for practical use, the invention of transistor almost certainly finished the process (Buckley, 1951). Before about 1950, the overwhelming majority or workers in the field of crystal growth were occupied in academic research. Today, the overwhelming majority of the workers in this field are employed because crystals can be made into commercially significant products.

Oxide crystals provide a lot of application for the laser industry these days. Solid state crystal is an example of oxide crystals used as a host material in the laser industry. Main factors leading for selection of this material are their attractive properties for the generation, transmission, detection and conversion of optical signal over the broad range of signal and power level (Vere, 1987).

The crystal material can be formed using a variety of techniques. The most frequently used and the most important method of producing crystal is by solidification of its own melt called crystal growth from melt or just melts growth (Brice, 1986).

Interest for the development of $Y_3Al_5O_{12}$ (YAG) crystal growth technology still continuing at the present time because Nd-doped YAG is one of the most important laser hosts for the generation of 1.06 μ infra-red radiation (Chani *et al*., 1999). This cubic crystal which is garnet host belongs to *Ia3d* space group. It has high mechanical strength, good chemical stability, and the ability to be synthesized in large sizes with high optical quality (Powell, 1998). Nd:YAG crystals are usually grown by the conventional Czochralski (CZ) technique (Belouet, 1972, Brandle and Fratello, 1993, Galazka and Wilke, 2000).

The Nd:YAG laser is the most common member of a family of lasers that are commonly grouped together as solid-state lasers. It was invented in 1964 and has remained in a continuous development and improvement process to the present day. Current market demand for high power continuous wave Nd:YAG lasers is driving the development of innovative and efficient approaches to the manufacturing and testing processes for these lasers.

Nd:YAG lasers has been in market with over 30 years and have become the most versatile laser systems in use today. They have received widespread acceptance by the military serving as a range finders and target designators, by the medical community as surgical tools, and by the manufacturing sector where they serve a wide variety of roles, including welding, cutting, and drilling. Efficient and reliable operation of a lamp-pumped Nd:YAG laser is highly dependent on the crystal from which the beam is derived. One of the most important characteristics is the maximum laser power that can be extracted from the crystal. So, the single crystal growth processes become an extremely delicate and sensitive process. Any disturbances during the growth of the boule may affect the laser operation in the cavity.

Until now, Nd:YAG laser rods have been the highest volume product for most leading manufacturers. In the future, the optical-crystal industry will deliver tens of millions of crystal parts annually (Chani *et. al*, 1999). Nd:YAG crystals is ordinarily produced with concentrations from 0.18% to 1.8% for applications in all types of solid-state lasers systems-frequency-doubled continuous wave, high-energy Q-switched, military, industrial, medical and scientific markets (Powell, 1998). The Czochralski 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 (Golubovic *et al.*, 2002).

**1.2     Problem Statement**

The study of yttrium aluminium garnet doped neodymium crystal (Nd:YAG) which emphasized on the physical and structural properties has previously been done by many researchers. It becomes the most famous crystal especially in solid state laser material because it lasing properties are a good compromise between the strengths and weaknesses of its competitions. However, for some reasons, the exact technique for growing the Nd:YAG crystal with high quality and low imperfection was not fully revealed because it still a secret for some company and researcher. Doped percentage of $Nd^{3+}$, physical, and structural properties of the crystal have also not splendidly mention in the literature. Therefore, the aims for this research are to use the Czochralski technique enhanced with automatic diameter controller crystal growth system (ADC-CGS) for growing the Nd:YAG crystal. Moreover, physical properties and structural of the crystal are also studied in this research.

**1.3     Objectives**

The objectives for this research are:

I.      To grow Nd:YAG crystal using Czochralski technique enhanced with Automatic Diameter Control - Crystal Growth System (ADC-CGS).

II.     To observe the effect $Nd^{3+}$ concentration with the length of crystal boule from its initial point of pulling direction.

III.    To investigate the physical properties of the grown crystal.

IV.     To characterize the structural properties of Nd:YAG crystal.

## 1.4    Scope

Research that have been conduct consisted the growth of yttrium aluminium garnet doped neodymium (Nd:YAG) crystal with nominal dopant concentration of 1.4% at. Nd by using Czochralski technique enhance with Automatic Diameter Controller Growth System (ADC-CGS). Elemental analysis (EDX) is used to verify the composition of the sample.  Physical properties of the crystal are focused on density of the crystal which is measured using Archimedes principle and the hardness is determined by Vickers hardness measurement. Structure of the grown crystal is examined using Raman spectroscopy whereas for the internal properties, IR spectroscopy is being used.

## 1.5    Significant of Research

Hopefully, this research will contribute knowledge and skills about Czochralski technique for growing a crystal in order to improve our manufacturing technology to the next level. Furthermore, findings from this research can be used to enhanced the crystal properties and for a better laser beam generation in the future.

All of the growth and characterization techniques in form of this research can be referred in Figure 1.1.

## 1.6    Thesis Plan

This thesis describes the growth process of neodymium doped yttrium aluminium garnet crystal prepared by Czochralski technique using Automatic Diameter Control – Crystal Growth system. This thesis is separated into five chapters. Chapter 1 is the introduction of the research, which specifies the research problem statement, objective and scope of study. Chapter 2 briefly explains the previous work done on related crystal material, fundamental theory for characterization process and some basic knowledge about crystal properties and materials. For Chapter 3, details about the experiment and characterization techniques are explained. In Chapter 4, all the experimental results along with discussions are given. Dopant concentration which is the main variable that changed all the result is discussed in this chapter. In addition, the effects of this dopant on physical and structural properties are also reported in this chapter. To conclude, Chapter 5 presents the major conclusion of the research and suggestions for further studies.

8

## 1.7    Research Flow Chart

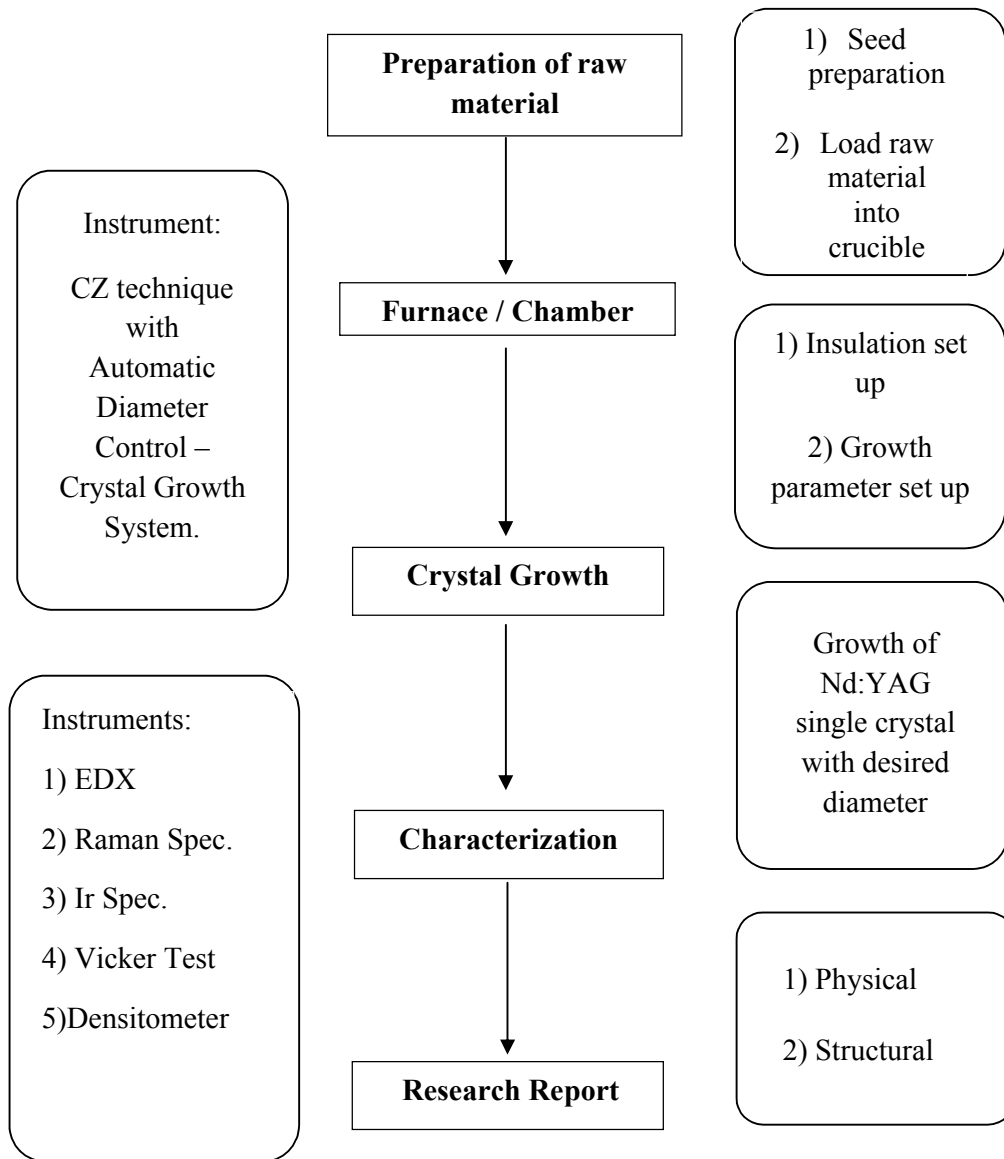The work step in this research can be referred from the flow chart shown in Figure 1.1.



**Figure 1.1:** Research flow chart

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