EFFECT OF ROTATION SPEED AND PULLING RATE ON PHYSICAL PROPERTIES OF TITANIUM-DOPED SAPPHIRE SINGLE CRYSTALS

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To my beloved parents,
Jainal Bin Sakiban & Masamah Binti Othman,
To my brother,
Mohammad Ikbal Iskandar,
And to my lovely dear,
Nurul Farhana Binti Jumaat.

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ABSTRACT

Titanium-doped sapphire single crystals with 0.20 weight percentage of dopant were successfully produced by using the Automatic Diameter Control-Crystal Growth System based on the Czochralski technique. Group A crystals were produced at a constant pulling rate of 1.50 mm h^{-1} and a rotational speed (8-24) rpm and Group B crystal were produced at a constant rotational speed of 15 rpm and pulling rate (0.75-1.75) mm h^{-1}. The presence of macroscopic defects such as gas bubbles and inclusions were found in the crystals produced at low rotation speed and at high pulling rate. XRD spectra of the crystals identified synthetic corundum as the main phase of the crystals and Al_2TiO_5 as the second phase. EDAX analysis showed the presence of Al, O and Ti element. Temperature, control power and growth rate were investigated for their correlation on the growth process and crystal diameter profile. When the rotation speed was increased, Group A crystals showed an increase in density from 3.963 g cm^{-3} to 3.999 g cm^{-3}. For group B crystals, the density decreased from 3.988 g cm^{-3} to 3.955 g cm^{-3} with increasing pulling rate. The porosity of the Group A crystals was found to decrease from 0.27% to 0.08% with increasing rotation speed. Group B crystals showed an increase in porosity from 0.09% to 0.44% with increasing pulling rate. From Vickers hardness test, the hardness of Group A crystals increased from 855 HV to 1698 HV with an increase in rotational speed. A decreased in hardness from 1439 HV to 845 HV was observed with increasing pulling rate for Group B crystals. The Young's Modulus of Group A crystals increased from 1542 MPa to 2069 MPa with increasing rotation speed. However, when the pulling rate was increased for the Group B crystals, the Young's Modulus was found to decrease from 2002 MPa to 1311 MPa.

ABSTRAK

Hablur tunggal safir yang didop dengan titanium dengan peratusan berat dopan 0.20 telah berjaya dihasilkan dengan menggunakan Sistem Pertumbuhan Hablur-Kawalan Diameter Automatik berdasarkan kepada teknik Czochralski. Hablur Kumpulan A telah dihasilkan pada kadar tarikan malar 1.50 mm per jam dan kelajuan putaran (8-24) putaran per minit dan hablur Kumpulan B telah dihasilkan dengan kelajuan putaran malar 15 putaran per minit dan kadar tarikan (0.75-1.75) mm per jam. Kehadiran kecacatan makroskopik seperti gelembung gas dan bendasing telah ditemui dalam hablur yang dihasilkan pada kelajuan putaran yang rendah dan kadar tarikan yang tinggi. Spektrum XRD daripada hablur telah mengenalpasti korundum sintetik sebagai fasa utama hablur dan $Al_2Ti_2O_5$ sebagai fasa kedua. Analisis EDAX menunjukkan kehadiran elemen Al, O dan Ti. Suhu, kuasa kawalan dan kadar pertumbuhan telah dikaji untuk hubungkaitnya pada proses pertumbuhan dan profil diameter hablur. Apabila kelajuan putaran bertambah, hablur Kumpulan A menunjukkan peningkatan dalam ketumpatan dari 3.963 g cm^{-3} ke 3.999 g cm^{-3}. Bagi hablur Kumpulan B, ketumpatannya susut dari 3.988 g cm^{-3} to 3.955 g cm^{-3} dengan meningkatnya kadar tarikan. Keporosan hablur kumpulan A didapati susut dari 0.27% ke 0.08% dengan meningkatnya kelajuan putaran. Hablur Kumpulan B menunjukkan pertambahan keporosan dari 0.09% ke 0.44% dengan peningkatan kadar tarikan. Daripada ujian kekerasan Vickers, kekerasan hablur Kumpulan A bertambah dari 855 HV ke 1698 HV dengan peningkatan kelajuan putaran. Penyusutan kekerasan dari 1439 HV ke 845 HV telah diamati dengan peningkatan kadar tarikan bagi hablur Kumpulan B. Modulus Young bagi hablur Kumpulan A meningkat dari 1542 MPa ke 2069 MPa dengan peningkatan kelajuan putaran. Bagaimanapun, apabila kadar tarikan ditingkatkan bagi hablur Kumpulan B, Modulus Young didapati susut dari 2002 MPa ke 1311 MPa.

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LIST OF SYMBOLS

A - surface area of residual deformation

A_0 - original cross-sectional area

A_c - surface area of crystal interface

d - mean diagonal

D - ratio of crystal radius to crucible radius

$d_{cryst.}$ - diameter of the crystal

dD/dt - first derivative of the diameter

d_{hkl} - interplanar spacing of the (*hkl*) spacing

dW/dt - first derivative of the crystal

E - Young's Modulus

F - peak load

F_C - force exerted on the specimen under compression

G_c - crystal growth rate

G_l - amount of liquid crystallized per unit time

H - hardness

h_c - height of crystal grown per unit time or linear growth rate

L - height of liquid crystallized per unit time

L_0 - original length of the specimen

L_m - liquid level drop

m - mass

n - integer or order of reflection

$P_{(t)}$ - control power at time

P_r - sum of the pull rate

r_c - radius of the crystal

R_{cr} - radius of the crucible

t - time

v - volume

V_b - bulk volume

V_{op} - open pore volume

W_1 - weight of sample in air

W_2 - weight of sample in liquid

W_D - weight of the dry sample

W_S - weight of saturated sample

W_{SS} - weight of the saturated sample submerged in liquid

ΔL - length displaced over original length

ε - strain

θ - diffraction angle

κ - ratio of average liquid density to solid density of the crystal

λ - wavelength of the beam

ρ - density

ρ_{air} - density of air

ρ_L - liquid density

ρ_{liq} - density of immersion liquid

ρ_S - solid density of the crystal

σ - stress

LIST OF ABBREVIATIONS

ADC	-	Automatic Diameter Control
ADC-CGS	-	Automatic Diameter Control-Crystal Growth System
CP	-	control power
Cz	-	Czochralski
DG	-	derivative gain
EDAX	-	Energy Dispersive X-ray
err	-	error
HV	-	Vickers hardness number
ICSD	-	Inorganic Crystal Diffraction Database
ID	-	inside diameter
IG	-	integral gain
LED	-	light emitting diode
OD	-	outside diameter
PDF	-	powder diffraction file
PG	-	proportional gain
PI	-	proportional-integral
PID	-	proportional-integral-derivative
RF	-	radio frequency
SEM		scanning electron microscopy
T	-	thickness
Ti:Sapphire	-	titanium-doped sapphire
WDS		wavelength dispersive X-ray spectroscopy
wt.	-	weight
XRD	-	X-ray Diffraction

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CHAPTER 1

INTRODUCTION

1.1 Problem Background

Crystals are one of the unrecognized backbones of modern technology. For the past few decades, crystal has profound yet prominence importance in electronic industry, photonic industry, communication industry, which rely on semiconductors, superconductors, polarizers, transducers, radiation detectors, ultrasonic amplifiers, ferrites, magnetic garnets, solid state lasers, non-linear optics, piezoelectric, electro-optic, acousto-optic, photosensitive, refractory of different grades, crystalline films for microelectronics and computer industries. Crystal growth is a highly-diverse subject that covers physics, chemistry, material science, chemical engineering, metallurgy, crystallography, mineralogy and many more (Caroline, 2009).

With the increasing demands to fulfil the needs of large size and high yield of crystal for commercial purposes and technological applications, the understanding on the crystal growth process was the major interest. Therefore, research on the development of crystal growth process was extensively carried out among the industrial crystal growers and scientific researchers within the past few decades. With the technological advancement of scientific instruments and analytical methods such as x-rays, electron microscopy, FTIR and NMR, crystal growth research has embarked into atomic level, which makes it possible for further understanding of the physical, chemical, optical and other properties of the structural nature of various crystals (Zhang, 1999).

Single crystal is a solid with the structure of the atoms that are perfectly arranged in periodic and repetitive manner which extends in long-range atomic order. A single crystal is formed by the growth of a crystal nucleus without secondary nucleation or impingement on other crystals. As the crystal grows, differences in chemical composition and temperature cause variations. The atomic structure determines the chemical and physical properties of the single crystal, including colour (Glazer, 1987). The occurrence of single crystals can be found in nature, but they may also be produced synthetically. They are ordinarily difficult to grow, because the environment must be carefully controlled. Nowadays, single crystals are produced synthetically in order to fulfil the demands of commercial purposes and scientific interests.

Generally, single crystal growth methods are classified into three categories which are growth from melt, growth from solution and growth from the vapour phase. The most widely employed method on production of single crystal is the growth from melt. Basically, all materials can be solidified into single crystal form from melt under these conditions; they melted congruently, they do not disintegrate into simpler chemical compounds during pre-melting and they are not subjected to a phase transformation between the melting point and room temperature (Srivastava, 2005). One of the growth methods from melt, the Czochralski is the most dominant process of single crystal growth in the industry nowadays. This technique is widely used in the large-scale production of semiconductor and optical single crystals such as silicon, GaAs, Sapphire, Ti:Sapphire, Nd:YAG, $LiNbO_3$, and many more (Brandle, 2004).

Much of the crystal growth process is considered as an art and technique rather than science. The process is remains an extremely delicate process due to its nature (Burgers, 1963). This difficulty arises from the complexity of relationships between the phenomena involved in the process such as heat transfer, phase transformations and fluid flow transitions (Duffar, 2000). The control of crystal-melt interface in Czochralski crystal growth has its crucial governance on the produced crystal. Thus, it is essential to take special measure of the growth parameters that modify the crystal-melt interface, especially the axial temperature gradient, the crystal rotation speed and the pulling rate (Takagi *et. al*, 1976: Dhanaraj *et al.*, 2010). Negligence to the mentioned growth parameters would probably lead to undesirable defect formations to the grown crystals such as dislocations, crystal cracking, gas bubble entrapment and much more (Buckley, 1951; Carruthers, 1967; Jackson 1967). These defects would preferably affect the quality of the crystals, regarding to their physical, structural, optical, and their mechanical properties. Thus, profound understanding on the growth parameters is essential on optimization of the crystal growth process.

Titanium-doped sapphire (Ti^{3+}:α-Al_2O_3) also known as Ti:Sapphire is a candidate for lasing medium with supreme physical and optical properties with broadest lasing range. Lasers based on Ti:Sapphire crystals were first constructed in 1982 (Moulton, 1986). It has a very large gain bandwidth, allowing the generation of very short pulses and also wide wavelength tunability. The possible tuning range is about 650 nm to 1100 nm. The absorption band of Ti:Sapphire centered at 490 nm makes it suitable for variety of laser pump sources such as argon ion, frequency doubled Nd:YAG and YLF and copper vapour lasers. Despite the huge emission bandwidth, Ti:Sapphire has relatively high laser cross sections, which reduces the tendency of Ti:Sapphire lasers for Q-switching instabilities (Xing *et al.*, 1995). With an appropriate design, Ti:Sapphire can also be used in continuous wave lasers with extremely narrow linewidths tunable over a wide range (Albers *et al.*, 1986). In addition, the host structure itself, sapphire, the single crystal form of Al_2O_3, is well-known for its excellent mechanical strength, high thermal resistance and inertness against chemical attack (Dobrovinskaya *et al.*, 2009).

In this research, an Automatic Diameter Control – Crystal Growth System (ADC-CGS) with a Czochralski (Cz) technique was utilized to grow single crystals of Titanium doped Sapphire, Ti:Sapphire. Besides that, the inter-relationship of the growth parameters to the diameter of the crystal was studied. In addition, the effect of the growth parameters (the rotation speed and the pulling rate) on the properties of the crystal such as their structure, physical and mechanical characteristics was also analysed and reported.

1.2 Problem Statement

The Ti:Sapphire single crystal have been the major interest both either in research purposes or industrial applications. It is well known for its lasing properties and its outstanding mechanical properties. However, extensive study needs to be done on the optimization of Ti:Sapphire single crystal by Czohcralski technique in order to grow high quality crystals. Rotation speed and the pull rate are the growth parameters that are directly influential to the formation of the crystal from the melt in a Czochralski crystal growth. These parameters are usually associated with the formation of growth defects such as bubbles entrapment, inclusions and crystal cracks. These defects affect the quality of the crystal in terms of their physical and mechanical properties. However, for some reasons, the exact optimization for growing the Ti:Sapphire crystal with low dislocations yet not undisclosed because it is a well-kept secret for some companies and researcher. In addition, the study of the influence of rotation speed and the pull rate on the of the Ti:Sapphire properties especially on physical and mechanical properties characterization are not splendidly mentioned in literature. Therefore, the aims of this research are to use the Czochralski technique with the aid of automatic diameter control system namely ADC-CGS for growing the Ti:Sapphire single crystal. In addition, the correlation of the growth parameters during the crystal growth process is studied. The physical properties of the crystal in reference to the growth parameters (the rotation speed and the pulling rate), are also studied.

1.3 Objectives of Research

The objectives of the research are as follows:

i. To grow Ti:Sapphire single crystals by Czochralski method using Automatic Diameter Control – Crystal Growth System
ii. To study the inter-correlation between control power, temperature, diameter and growth rate on Ti:Sapphire crystal growth process.
iii. To determine the structural phase of the grown Ti:Sapphire crystals.
iv. To ascertain the chemical composition of the grown Ti:Sapphire crystals
v. To study the effect of rotation speed and pulling rate on the physical properties of Ti:Sapphire crystals by means of density, porosity, Vickers hardness and Young's Modulus.

1.4 Scope of Research

To achieve the objectives that have been listed, the scope of research is outlined.

i. Growth of Ti:Sapphire single crystal of 0.20 wt.% Ti dopant by Czochralski Method using ADC-CGS (Automatic Diameter Control-Crystal Growth System) with implementation of changing variable of the rotation speed and the pulling rate.
ii. Finding the correlation of temperature, control power, growth rate to the diameter profile in respect to specified growth parameters (the rotation speed and the pulling rate)
iii. Determining of structural phase using X-Ray Diffraction
iv. Determining of composition of the crystal using Energy Dispersive X-Ray analysis.
v. Determining the physical properties such as density, porosity, hardness and Young's Modulus by means of Archimedes Principles test, Vickers hardness test and compressive test.

1.5 Significance of Research

It is hoped that this study could give researchers and crystal growers some profound insight about the inter-relationship of the temperature, control power, diameter and growth rate on the growth process of Ti:Sapphire and also the effect of both rotation speed and the pull rate on the physical properties of Ti: Sapphire grown by Czochralski technique. Thus, give more understanding for the Czochralski crystal growers on the optimization of rotation speed and pulling rate for Ti: Sapphire crystal growth process by this technique.

1.6 Thesis Plan

The thesis describes the growth process of titanium doped sapphire crystal prepared by Czochralski technique using Automatic Diameter Control – Crystal Growth System. This thesis is categorized into five chapters. Chapter 1 is the introduction of the research, which emphasizes the research problem statement, objective and scope of the study. Chapter 2 briefly reviews the works done by previous researcher on related crystal material, the rudimentary principles of Czochralski growth technique, fundamental theories on the characterization assessments and some basic knowledge regarding the grown materials. For Chapter 3, the details of the crystal growth experiments and measurement techniques are explained. In Chapter 4, the experimental results accompanied with discussions are represented. Within this chapter, the correlation of control power-temperature, control power-diameter, and diameter-growth rate on the crystal growth process are explained. The structural phase and elemental composition of the grown crystals is discussed. The effect of the growth parameters (the rotation speed and the pulling rate) on the physical attributes such like density and porosity, hardness and Young’s Modulus are also reported. As the epilogue of the thesis, Chapter 5 presents the major conclusions of the research and suggestions for future studies.

1.7 Research Flow Chart

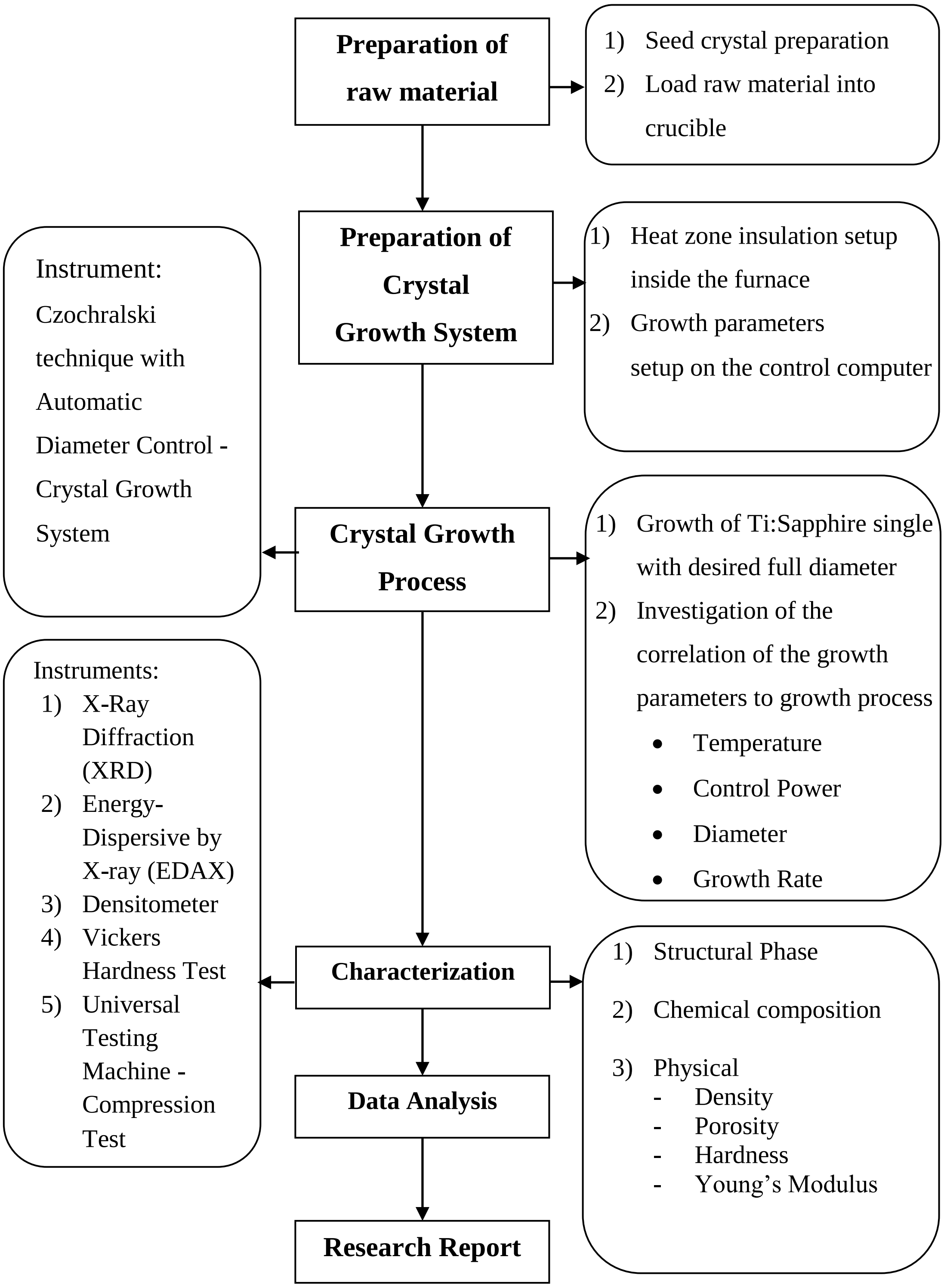

Figure 1.1: Research flow chart.

REFERENCES

Akselrod, M. S. and Bruni, F. J. (2012). Modern Trends in Crystal Growth and New Applications of Sapphire. *J. Crystal Growth*, 360: 134-145.

Albers, P., Stark, E. and Huber, G. (1986). Continuous-Wave Laser Operation and Quantum Efficiency of Titanium-Doped Sapphire. *J. Opt. Soc. Am. B.*, 3(1): 134-139.

Armou, A. and Christofides, P. D. (2001). Crystal Temperature Control in the Czochralski Growh Process. *AlChE Journal*, 47(1): 79-106.

Ashby, M. F. and Sammis, C. G. (1990). The Damage Mechanics of Brittle Solids in Compression. *Pure Appl. Geophys.*, 133(3): 489-521.

Baldochi, S. L., Mazzocchi, V. L., Parente, C. B. R. and Morato, S. P. (1994). Study of the Crystalline Quality of Czochralski Grown Barium Lithium Fluoride Single Crystals. *Mat. Res. Bull.*, 29(12): 1321-1331.

Basu, B. Enger, S., Breuer, M. and Durst, F. (2001). Effect of Crystal Rotation on the Three-Dimensional Mixed Convection in the Oxide Melt for Czochralski Growth. *J. Crystal Growth*, 230(1-2): 148-154.

Brandle, C. D. (1980). *Crystal Pulling*. In Pamplin, B. R. (Ed.) *Crystal Growth*. 2nd edition. Oxford: Pergamon. 275-300.

Brandle, C. D. (2004). Czochralski Growth of Oxides. *J. Crystal Growth*, 264 (4): 593-604.

Brandle, C. D. and Valentino A. J. (1972). Czochralski Growth of Rare Earth Gallium Garnets. *J. Crystal Growth*, 12(1): 3-8.

Brandle, C. D. and Miller, D. C. (1974). Czochralski Growth of Large Diameter $LiTaO_3$ Crystals. *J. Crystal Growth*, 24-25: 432-436.

Brice, J. C. (1986). *Crystal Growth Processes*. New York: John Wiley & Sons.

Bruni, F. J. (2004). *ADC Software Instruction Manual: Power Adjusting Type.* Release 3.0.8. Santa Rosa: Bruni Consultant.

Bruni, F. J. (2005). Automatic Diameter Control & Interface Shape Stability. Third International Workshop on Crystal Growth Technology, Beatenberg, Switzerland. 10-18 September 2005.

Brown, Robert A. (1988). Theory of Transport Process in Single Crystal Growth from Melt. *AlChE Journal.* 34(6): 811-911.

Buckley, H. E. (1951). *Crystal Growth*. New York: Wiley.

Burgers, W. G. (1963). *Principles of Recrystallization*. In Gilman, J. J. (Ed.) *The Art and Science of Growing Crystals*. New York: John Wiley & Sons, Inc. 416-450.

Burns, R. G. (1993). *Mineralogical Application of Crystal Field Theory.* 2nd edition. Cambridge: Cambridge University Press.

Callister, Jr., W. D. and Rethwisch, D. G. (2011). *Fundamentals of Materials Science Engineering: An Integrated Approach*. 4th edition. New York: John Wiley & Sons. Inc.

Cardarelli, F. (2008). *Materials Handbook: A Concise Desktop Reference*. 2nd edition. London: Springer.

Caroline, M. L. (2009). *Growth and Characterization of Some Organic and Semi Organic Amino Acid Based Nonlinear Optical Single Crystals*. Bharath University: PhD Thesis.

Carruthers, J. R. (1967). Origin of Convective Temperature Oscillations in Crystal Growth Melts. *J. Crystal Growth*, 32(1): 13-26.

Carruthers, J. R. and Nassau, K. (1968). Nonmixing Cells Due to Crucible Rotation during Czochralski Crystal Growth. *J. Appl. Phys.*, 39(11): 5205-5214.

Chauhan, A. K. (2004). Development of a PC-Based Automatic Diameter Controlled Czochralski Crystal Growth System Using aVersatile Algorithm. *Cryst. Growth Des.*, 4(1): 135-139.

Choe, K. S., Stefani, J. A., Dettling, T. B., Tien, J. K. and Wallace, J. P. (1991). Effects of Growth Conditions on Thermal Profiles During Czochralski Silicon Crystal Growth. *J. Crystal Growth*, 108(1-2): 262-276.

Chopra, D. R. and Chourasia, A. R. (1997). *X-Ray Photoelectron Spectroscopy*. In Settle, F. A. (Ed) *Handbook of Instrumental Techniques for Analytical Chemistry*. New Jersey: Prentice-Hall PTR. 809-828.

Cockayne, B. (1968). Developments in Melt-Grown Oxide Crystals. *J. Crystal Growth*, 3-4: 60-70.

Dash, W. C. (1959). Growth of Silicon Single Crystal Free from Dislocations. *J. Appl. Phys.*, 30(40): 459-474.

Derby, J. J., Atherton, L. J. and Gresho, P. M. (1989). An Integrated Process Model for the Growth of Oxide Crystals by the Czochralski Method. *J. Crystal Growth*, 97(3-4): 792-826.

Dhanaraj, G., Byrappa, K., Prasad, V. and Dudley, M. (Eds.) (2010). *Springer Handbook of Crystal Growth*. Berlin, Heidelberg: Springer.

Dobrovinskaya, E. R., Lytvynov, L. A. and Pishchik, V. (2009). *Sapphire: Material, Manufacturing, Applications*. New York: Springer.

Duffar, T. (2000). Crystal Growth Process Engineering. *J. Optoelectron. Adv. M.*, 2(5): 432-440.

Fang, H., Tian, J., Zhang, Q., Pan, Y. and Wang, S. (2012). Study of the Melt and Interface Shape During Sapphire Crystal Growth by Czochralski Method. *Int. J. Heat Mass Transfer*, 55(25-26): 8003-8009.

Fang, H.S, Pan, Y. Y., Zheng, L. L., Zhang, Q. J., Wang, S. and Jin, Z. L. (2013). To Investigate Interface Shape and Thermal Stress During Sapphire Single Crystal Growth by the Cz Method. *J. Crystal Growth*, 363: 25-32.

Garrat-Reed, A. J. and Bell, D.C. (2003). *Energy Dispersive X-ray Analysis in the Electron Microscope*. Oxford: BIOS Scientific Publishers Limited.

Giordano, L., Viviani, M., Bottino, C., Buscaglia, M. T. and Nanni, P. (2002). Microstructure and Thermal Expansion of Al_2TiO_5-$MgTi_2O_5$ Solid Solution Obtained by Reaction Sintering. *J. Eur. Cer. Soc.*, 22(11): 1811-1822.

Glazer, A. M. (1987). *The Structure of Crystals*. Bristol: Adam Hilger.

Globus, M. E. and Grinyov, V. B. (2000). *Inorganic Scintillators: New and Traditional Materials*. Kharkov: Akta.

Grabmaier, J. G., Plattner, R. D. and Schieber, M. (1973). Suppression of Constitutional Supercooling in Czochralski-Growth Paratellurite. *J. Crystal Growth*, 20(2): 82-88.

Green, D. J. (1998) *An Introduction to the Mechanical Properties of Ceramics*. Cambridge: Cambridge University Press.

Havrilla, G. J. (1997). *X-Ray Fluorescence Spectrometry*. In Settle, F. A. (Ed.) *Handbook of Instrumental Techniques for Analytical Chemistry*. Upper Saddle River, New Jersey: Prentice Hall PTR. 459-479.

He, B. B. (2009). *Two Dimensional X-Ray Diffraction*. New Jersey: John Wiley & Sons, Inc.

Henderson, B. and Bartram, R. H. (2000). *Crystal-Field Engineering of Solid State Materials*. Cambridge: Cambridge University Press.

Hurle, D. T J. (1973). *Melt Growth*. In Hartman, P. (Ed.) *Crystal Growth: An Introduction*. Amsterdam: North-Holland. 233.

Hurle, D. T. J. (1977). Control of Diameter in Czochralski and Related Crystal Growth Techniques. *J. Crystal Growth*, 42: 473-482.

Jackson, K. A. (1967). Current Concepts in Crystal Growth from the Melt. *Prog. Solis State Ch.* 4: 53-56.

Jia, L. S., Yan, X. L., Zhou, J. F. and Chen, X. L. (2003). Effects of Pulling Rates on Quality of $La_{2-x}Ba_xCuO4$ Single Crystal. *Physica C.*, 385(4): 483-487.

Jones, J. T. and Berard, M. F. (1972). *Ceramics: Industrial Processing and Testing*. Ames: Iowa State University Press.

Joyce, D. B. and Schmid, F. (2010). Progress in The Growth of Large Scale Ti:Saphhire Crystals by The Heat Exchanger Method (HEM) for Petawatt Class Lasers. *J. Crystal Growth*, 312(8): 1138-1141.

Kamaruddin, W. H. A. (2010). *Growth of Nd:YAG Crystals by using Automatic Diameter Control-Crystal by Czochralski Melt-Growth Method*. Universiti Teknologi Malaysia: Master's Thesis.

Katsurayama, M., Anzai, Y., Sugiyama, A., Koike, M. and Kato, Y. (2001). Growth of Neodymium Doped $Y_3Al_5O_{12}$ Single Crystals by Double Crucible Method. *J. Crystal Growth*, 229(1-4): 193-198.

Khattak, C. P. and Schmid, F. (2001). Growth of the World's Largest Sapphire Crystals. *J. Crystal Growth*, 225(2-4): 572-579.

Kingery, W. D. (1960). *Introduction to Ceramics*. New York, London: John Wiley & Sons, Inc.

Kobayashi, N. (1978). Computational Simulations of the Melt Flow during Czochralski Growth. *J. Crystal Growth*, 43(3): 357-363.

Kobayashi, N. (1981a). Heat Transfer in Czochralski Crystal Growth. In Wilcox, W. R. (Ed.) *Preparation and Properties of Solid State Materials*, 6. New York: Dekker.119-253.

Kobayashi, N. (1981b). Effect of Fluid Flow on The Formation of Gas Bubbles in Oxide Crystals Grown by The Czochralski Method. *J. Cryst. Growth*, 54(3): 414-416.

Kokta, M. (2007). Growth of Oxide Laser Crystal. *Opt. Mater.*, 30(1): 1-5.

Kusuma, H. H. (2007). *Merekabentuk Dan Membina Sistem Pertumbuhan Hablur Kaedah Czochralski Dengan Menggunakan Pemanasan Rintangan*. Universiti Teknologi Malaysia: Master's Thesis.

Kusuma, H. H. (2012). *Structural and Optical Properties of Sapphire Doped Transition Metals Single Crystal by Czochralski Method*. Universiti Teknologi Malaysia: PhD Thesis.

Kvapil, J., Perner, B. and Kvapil, Jos. (1974). Liquid/Solid Interface Profile of Melt Grown Oxide Crystals: II- Crystal Quality. *Czech. Phys. B.*, 24(12): 1345-1350.

Lawn, B. R. (1993). *Fracture of Brittle Solids.* 2nd edition. Cambridge: Cambridge University Press.

Lawn, B. R. and Evans, A. G. (1977). A Model for Crack Initiation in Elastic-Plastic Indentation Fields. *J. Mater. Sci.*, 12(11): 2195-2199.

Lawn, B. R. and Wils-haw, T. R. (1975). Indentation Fracture: Principles and Applications. *J. Mater. Sci.*, 10(6): 1049-1081.

Li, H., Ghezal, E. A., Nehari, A., Alombert-Goget, G., Brenier, A. and Lebbou, K. (2013). Bubbles Defetcs Distribution in Sapphire Bulk Crystals Grown by Czochralski Technique. *Opt. Mater.*, 35:1071-1076.

Lim, L. C., Tan, L. K. And Zeng, H. C. (1996). Bubble Formation in Czochralski-Grown Lead Molybdate Crystals. *J. Crystal Growth*, 167(3-4): 686-692.

Mencik, J. (1992). *Strength and Fracture of Glass and Ceramics, Glass and Science Technology*, 12. Amsterdam: Elsevier.

Miller, D. C., Valentino, A. J. and Schick, L. K. (1978). The Effect of Melt Flow Phenomena on the Perfection of Czochralski Grown Gadolinium Gallium Garnet. *J. Crystal Growth*, 44(2): 121-134.

Miyazawa, S. (1980). Fluid-Flow Effect on Gas-Bubble Entrapment in Czochralski-Grown Oxide Crystals. *J. Crystal Growth*, 49(3): 515-521.

Moulton, P.F. (1986). Spectroscopic and laser characteristics of Ti:Al_2O_3. *J. Opt. Soc. Am. B.,* 3(1): 125-132.

Nassau, K. (1983). *The Physics and Chemistry of Color: The Fifteen Causes of Color*. New York: John Wiley & Sons, Inc.

Nehari, A. N., Brenier, A., Panzer, G., Lebbou, K., Godfroy, J.,Labor, S., Legal, H., Cheriaux, G., Chambaret, J. P., Duffar, T. And Moncorge, R. (2011). Ti-Doped Sapphire (Al_2O_3) Single Crystals Grown by the Kyropoulos Technique and Optical Characterizations. *Cryst. Growth Des*., 11(2): 445-448.

O'Kane, D. F., Sadagopan, V., Giess, E. A. and Mendel, E. (1973). Crystal Growth and Characterization of Gadolinium Gallium Garnet. *J. Electrochem. Soc*., 120: 1272.

Okano, Y., Fukuda, T., Hirata, A., Takano, N., Tsukada, T., Hozawa, M. and Imaishi, N. (1991). Numerical Study on Czochralski Growth of Oxide Single Crystals. *J. Crystal Growth*, 109(1-4): 94-98.

Oliver, B. R. E. G. (2005). *Natural Sciences: Chemistry. Crystallography. Mineralogy; Practical laboratory chemistry. Preparative and experimental chemistry*. New York: Interscience Publishers, Inc.

Paschotta, R. (2008). *Encyclopedia of Laser Physics and Technology, 1*. Weinheim: Wiley-VCH.

Peng, Y., Shu, C., Chew, Y. T. and Qiu, J. (2003). Numerical Investigations of Flows in Czochralski Crystal Growth by an Axisymmetric Lattice Boltzmann Method. *J. Comput. Phys*., 186(1): 295-307.

Philip, M. and Bolton W. (2002). *Technology of Engineering Materials*. Oxford, Massachusetts: Butterworth-Heinemann.

Pimputkar, S. M. and Ostrach, S. (1981). Convective Effects in Crystal Grown from Melt. *J. Crystal Growth*, 55(3): 614-646.

Raghavan, P. S. and Ramasamy, P. (2002). Recent Trends in Crystal Growth Technology. *PINSA*, 68A(3): 235-249.

Rapoport, W. R. and Khattak, C. P. (1988). Titanium Sapphire Laser Characteristic. *Appl. Optics*, 27(13): 2677-2684.

Rasmussen, J. J. (1969). *Effect of Dopants on the Defect Structure of Single Crystal Aluminum Oxide*. Massachusetts Institute of Technology: PhD Thesis.

Reed, T. B and Fahey, R. E. (1966). Resistance Heated Crystal Puller for Operation at 2000°C. *Rev. Sci. Instr.*, 37(1): 59.

Rice, R. W. (1998). *Porosity of Ceramics*. New York: Marcell Decker, Inc.

Robertson, D. S. (1966). A Study of the Flow Patterns in Liquids Using a Model Czochralski Crystal Growing System. *Br. J. Appl. Phys.*, 17(8): 1047.

Rojo, J. C., Dieguez, E. and Derby, J. J. (1999). A Heat Shield to Control Thermal Gradients, Melt Convection, and Interface Shape during Shouldering in Czochralski Oxide Growth. *J. Crystal Growth*, 200(1-2): 329-334.

Sangwal, K. And Benz, K. W. (1996). Impurity Striations in Crystals. *Prog. Cryst. Growth Ch.*, 32(1-3): 135-169.

Shiroki, K. (1977). Simulations of Czochralski Growth on Crystal Rotation Rate Influence in Fixed Crucibles. *J. Crystal Growth*, 40(1): 129-138.

Smith, J. D. and Fahrenholtz, W. G. (2008). *Refractory Oxides*. In Shackelford J. F. and Doremus, R. H. (Eds.) *Ceramics and Glass Materials: Structure, Properties and Processing*. New York: Springer. 87-110.

Srivastava, A. (2005). *Optical Imaging and Control of Convection around a KDP Crystal Growing From Its Aqueous Solution*. Indian Institute of Technology: PhD Thesis.

Sugii, K., Iwasaki, H., Miyazawa, S. and Niizeki, N. (1973). An X-Ray Topographic Study on Lithium Niobate Single Crystals. *J. Crystal Growth*, 18: 159.

Takagi, K., Fukuzawa,T. and Ishii, M. (1976). Inversion of the Direction of the Solid-Liquid Interface on the Czochralski Growth of GGG Crystals. *J. Crystal Growth*, 32(1): 89-94.

Tang, H., Li, H. and Xu, J. (2013*). Growth and Development of Sapphire Crystal for LED Applications*. In Ferreira, S. O. (Ed.) *Advance Topics on Crystal Growth*, 307-333. InTech.

Tavakoli, M. H. (2008). Numerical Study of Heat Transport and Fluid Flow during Different Stages of Sapphire Czochralski Crystal Growth. *J. Crystal Growth*, 310(12): 3107-3112.

Tavakoli, M. H., Omid, S. and Mohammadi-Manesh, E. (2011). Influence of Active Afterheater on the Fluid Dynamics and Heat Transfer During Czochralski Growth of Oxide Single Crystals. *Cryst. Eng. Comm.*, 13: 5088-5093.

Tavakoli, M. H., Abasi, T. A., Omid, S. and Mohammadi-Manesh, E. (2013). The Role of Inner and Internal Radiation on the Melt Growth of Sapphire Crystal. *Cryst. Res. Technol.*, 48(2): 58-68.

Teal, G. K. and Little, J. B. (1950). Growth of Germanium Single Crystals. *Phys. Rev.*, 78: 647.

Wang, C. J. and Huang, C. Y. (2008). Effect of TiO_2 Addition on the Sintering Behavior, Hardness and Fracture Toughness of an Ultrafine Alumina. *Mat. Sci. Eng. A-Struct.*, 492(1-2): 306-310.

Wang, E. Z. And Shrive, N. G. (1995). Brittle Fracture in Compression: Mechanisms, Models and Criteria. *Eng. Fract. Mech.*, 52(6): 1107-1126.

Xing, Q., Zhang, W. and Yoo, K. M. (1995). Self-Q Switched Self-Mode-Locked Ti:Sapphire Laser. *Opt. Commun.*, 119(1): 113-116.

Zhang, Z. (1999). Crystal Growth. *Proc. Natl. Acad. Sci. USA*, 96(20): 11069-11070.