UNIVERSITI PUTRA MALAYSIA

THERMAL DIFFUSIVITY MEASUREMENT OF COPPER SELENIDE USING PHOTOFLASH TECHNIQUE

NORNI HIDAYAWATI MAT DAUD

FS 2012 43

THERMAL DIFFUSIVITY MEASUREMENT OF COPPER SELENIDE USING PHOTOFLASH TECHNIQUE

By

NORNI HIDAYAWATI MAT DAUD

Thesis Submitted to the School of Graduate Studies Universiti Putra Malaysia in Fulfilment of the Requirements for the Degree of Master of Science

June 2012

DEDICATION

To my beloved family, supervisor and all my dearest friends.

Thanks for their guidance, supports, understanding, caring, love and encouragements.
May ALLAH bless us always.

Abstract of thesis presented to Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

THERMAL DIFFUSIVITY MEASUREMENT OF COPPER SELENIDE USING PHOTOFLASH TECHNIQUE

By

NORNI HIDAYAWATI BT MAT DAUD

February 2012

Chairman: Prof. Mohd Maarof H.A. Moksin, PhD

Faculty: Science

For several past decades, the studies on the thermal properties of copper selenides (CuSe) have excluded thermal diffusivity even though thermal diffusivity directly reflects the combined effects of thermal conductivity, specific heat and density of materials put together. In this report the research findings of the thermal diffusivity of CuSe as measured by using photoflash technique at the temperature range from 80 K to room temperature are presented. The samples of CuSe which were based on the formula of Cu_xSe_{1-x} were prepared using solid state method. The first set of the samples was prepared with similar x=0.5 composition, and each of the pellets was sintered at different sintering temperature from 150 to 275 ^oC for four days. The second and third sets of the samples were prepared by varying Cu composition with x = 0.3, 0.4, 0.5, 0.6, 0.7, and each set was sintered at 250 and 275 ^oC respectively for four days. XRD micrograph indicates that all the diffraction peaks were indexed to hexagonal CuSe phase for the samples with x = 0.5 and 0.6, and sintered at 250 ^oC upwards. However, the samples with x = 0.7 and were sintered at 250 ^oC and 275 ^oC had cubic Cu_2Se

single-phase while the sample with x = 0.8 and sintered at 250 °C had Cu_2Se phase with two different crystallographic forms of monoclinic and cubic crystal structure. The sample with the most symmetrical crystal structure, which was cubic crystal structure was found to have highest thermal diffusivity while a distorted and the least symmetrical crystal structure was found to have lowest thermal diffusivity. SEM micrograph and density measurement showed that the grain size as well as density increased with the increase of sintering temperature, and with the increase of x up to 0.7. On the other hand the specific heat data showed a decreasing trend with increasing sintering temperature and increasing x. However the specific heat only slightly increased as the sample's thermal diffusivity measuring temperature increased in the range of 80-300 K, showing that the specific heat was more or less independent of temperature. The grain size that increased with the sintering temperature affected the increased in the measured thermal diffusivity. In the bigger grain size samples, phonons would encounter lesser number of grain boundaries, experiencing a longer apparent mean free path and lower phonon-phonon scattering; the thermal diffusivity inevitably becomes higher. The similar increasing trend of thermal diffusivity was also observed for the samples with the increasing x.

The thermal diffusivity was consistently decreased with increasing measuring temperature from 80 to 300 K, for all samples. At the very low temperature, the phonon-phonon relaxation time is very long due to the phonon occupation probability which is very low. When the temperature increases, a higher population of thermally excited phonons is expected causing the number of phonon collisions to increase. Thus lattice scattering lowers the thermal carrier mobility more and more at higher

temperature due to the smaller mean free path. Therefore, their ability to transport heat away from the source is less, thus decreasing the thermal diffusivity value of this sample. The thermal diffusivity of the CuSe for all set of samples was in the range of (0.225-5.268) mm^2/s at the temperature of 80 K to 300 K.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

PENGUKURAN RESAPAN TERMA KUPRUM SELENIDA MENGGUNAKAN TEKNIK FOTOKILAT

Oleh

NORNI HIDAYAWATI BT MAT DAUD

Jun 2012

Pengerusi: Prof. Mohd Maarof H.A. Moksin, PhD

Fakulti: Sains

Beberapa dekad yang lepas, kajian ke atas kuprum selenida (CuSe) hanya tertumpu pada sifat-sifat termanya sahaja. Di sini kami melaporkan beberapa hasil kajian berkenaan resapan termanya menggunakan teknik fotokilat pada suhu rendah, 80 K, sehingga suhu bilik. Kajian resapan terma ini adalah penting untuk mendapatkan pengurusan terma yang memuaskan untuk teknologi peranti ini. CuSe berdasarkan pada rumus Cu_xSe_{1-x} telah disediakan dengan menggunakan kaedah keadaan pepejal. Set pertama sampel telah disediakan dengan komposisi yang sama iaitu x=0.5, dan setiap pelet telah disinter pada suhu pensinteran berbeza dari 150 hingga 275 ^oC selama empat hari. Set kedua dan ketiga sampel telah disediakan dengan mengubah Cu atau x = 0.3, 0.4, 0.5, 0.6, 0.7, dan setiap set telah disinter pada suhu 250 ^oC dan 275 ^oC selama empat hari. Mikrograf XRD menunjukkan bahawa semua puncak belauan diindekskan kepada fasa CuSe bagi sampel yang disinter pada 250 ^oC keatas dengan x = 0.5 dan 0.6. Walau bagaimanapun, sampel dengan kandungan x = 0.7 disinter pada suhu 250 ^oC dan 275 ^oC menunjukkan fasa tunggal Cu_2Se kubik telah diperolehi, sementara sampel dengan x = 0.8 disinter pada 250 ^oC mengandungi fasa Cu_2Se dengan dua bentuk kristalografi yang

berbeza iaitu struktur kristal monoklinik dan kubik. Sampel yang mempunyai struktur kristal yang paling simetri, iaitu struktur kristal kubik, didapati mempunyai nilai resapan terma yang paling tinggi manakala struktur kristal yang terganggu dan kurang simetri didapati mempunyai resapan terma yang paling rendah. Mikrograf SEM dan pengukuran ketumpatan menunjukkan saiz butiran serta ketumpatan meningkat dengan peningkatan suhu pensinteran, dan dengan peningkatan kandungan *x* sehingga 0.7. Haba tentu menunjukkan corak yang menurun dengan peningkatan suhu pensinteran dan peningkatan kandungan *x*, dan sedikitnya bertambah apabila suhu pengukuran bertambah menunjukkan bahawa haba tentu adalah hampir tak bergantung kepada suhu. Seperti yang telah dijelaskan, saiz butiran adalah berkadar terus dengan suhu pensinteran dan begitu juga dengan resapan terma. Dalam sampel yang mempunyai saiz butiran lebih besar, fonon akan bertemu dengan bilangan sempadan-sempadan butiran yang berkurangan. Seterusnya menjadikan lintasan bebas min untuk fonon semakin membesar sekaligus mengurangkan serakan fonon-fonon apabila melibatkan saiz butiran yang lebih besar, menjadikan nilai resapan terma semakin meningkat. Corak peningkatan resapan terma yang serupa juga diperhatikan untuk sampel yang mempunyai peningkatan kandungan *x*. Pengukuran pada suhu yang rendah menunjukkan resapan terma berkurangan secara konsisten dengan kenaikan suhu dari 80 hingga 300 K untuk semua sampel. Pada suhu yang sangat rendah, masa relaksasi fonon-fonon adalah sangat panjang kerana kebarangkalian pengisian fonon adalah sangat rendah. Apabila suhu meningkat, peningkatan populasi fonon teruja secara terma dijangka menyebabkan bilangan perlanggaran fonon meningkat. Oleh itu, serakan kekisi menurunkan mobiliti pembawa dengan lebih banyak pada suhu yang lebih tinggi disebabkan oleh lintasan bebas min yang lebih kecil. Oleh itu, keupayaan mereka untuk mengangkut haba dari

sumber menjadi kurang, dan seterusnya menurunkan nilai resapan terma sampel ini. Kajian ini menunjukkan bahawa nilai resapan terma untuk pelet CuSe adalah dalam lingkungan 0.225 kepada 5.268 mm^2/s yang diukur dari 80 K hingga suhu bilik seperti yang dilaporkan dalam kajian literatur.

ACKNOWLEDGEMENTS

At the outset, I would like to express my deepest praise to Allah SWT who has given me the patience, strength, determination and courage to produce this thesis despite all the challenges.

I would also like to extend my appreciation to my advisor, Prof. Dr. Mohd Maarof Moksin, for his enthusiastic supervision during this work. He has constantly encouraged me to remain focused to achieve my goal. His great vision and insightful comments have helped me to establish the overall goal of this research and to move the investigation forward. I would like to thank Prof. Dr. Azmi Zakaria, Assoc. Prof. Dr. Mansor Hashim, committee members, friends and to all the staff of Physics Department for their critical comments and kind help during the completion of my thesis.

I am forever indebted to my family. All this wouldn't be possible without the understanding, patience and encouragement from my parents Mat Daud Awang and Selmah Tuah who has been the driving force of my achievement.

I certify that an Examination Committee has met on **date of viva** to conduct the final examination of Miss **Norni Hidayawati Mat Daud** for her Master of Science thesis entitled **"Thermal Diffusivity of Copper Selenide Using Photoflash Technique"** in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

W. Mahmood Mat Yunus, PhD
Professor
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Jumiah Hassan, PhD
Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Internal Examiner)

Halimah Mohamed Kamari, PhD
Senior Lecturer
Faculty of Science
Universiti Putra Malaysia
(Internal Examiner)

Mohamad Deraman , PhD
Professor
Faculty of Science and Technology
Universiti Kebangsaan Malaysia
(External Examiner)

_____

SEOW HENG FONG, Phd

Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

Mohd. Maarof Moksin, PhD
Professor
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Azmi Zakaria, PhD
Professor
Faculty of Science
Universiti Putra Malaysia
(Member)

BUJANG BIN KIM HUAT, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or other institutions.

_____

NORNI HIDAYAWATI MAT DAUD

Date: 8 June 2012

TABLE OF CONTENTS