UNIVERSITI PUTRA MALAYSIA

STRUCTURAL, ELECTRICAL, OPTICAL AND THERMAL PROPERTIES OF NICKEL SELENIDE

CHIN PIK YEE

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By

CHIN PIK YEE

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

September 2015

DEDICATION

To my beloved parents
for their love and concern

To my supervisor Prof Dr. Zainal Abidin Talib
for his guidance, advice, understanding and endless support

To Dr. Josephine Liew Ying Chyi
for her guidance, advice and fully support

To my friends
for their wonderful encouragement and support

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

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September 2015

Chairman : Professor Zainal Abidin Talib, PhD
Faculty: Science

Fundamental studies are very crucial to generate new knowledge. In this respect, bulk material is useful to understand the characteristic of the material. Nickel selenide (NiSe) has been chosen because of its impressive photovoltaic properties and a wide range of industrial applications. To improve the solar conversion to electric efficiency, NiSe synthesized via solid state reaction method was annealed in the temperature range of 423 to 823 K.

The NiSe was in 1:1 stoichiometric form with single phase hexagonal crystal structure. FESEM micrographs showed the NiSe particle was in nanometer size. As the annealing temperature increased, the crystalline size increased from 52.7 nm to 142.8 nm while the dislocation density decreased from 36.01 x 10^{13} lines/m^2 to 4.90 x 10^{13} lines/m^2. The NiSe was a p-type semiconductor. As the NiSe annealed temperature increased, the electrical resistivity decreased from 0.0037 Ωcm to 0.00015 Ωcm. In addition, the Hall mobility increased from 1.21 cm^2/Vs to 15.06 cm^2/Vs. However, the sheet Hall coefficient and the sheet carrier density of the sample remained constant at 0.017 cm^2/C and 3.19 x 10^{20} cm^{-2} respectively. This indicates that at higher anneal temperatures, crystallite size will increase causing the electrical resistivity to decrease.

Optical analysis showed that the annealed NiSe has direct transition band gap energy in the range of 2.13 – 2.47 eV which is consistent with literature values. The decreased in the band gap of the sample with increasing annealing temperature can be interpreted as the increased in the crystallinity of the synthesized material. Photoacoustics analysis showed that as the annealed temperature increased, the surface recombination velocity decreased from 428.39 cm/s to 389.62 cm/s, while the thermal diffusivity, the diffusion coefficient and the band to band recombination lifetime of the NiSe increased from the 13.42 x 10^{-2} cm^2/s to 20.81 x 10^{-2} cm^2/s, from 4.56 cm^2/s to 5.10 cm^2/s, from 1.86 μs to 2.46 μs, respectively. This indicates that the diffused excess carrier through the NiSe surface has increased. The increasing of the surface recombination process is due to the increasing of homogeneity and crystallinity of the samples.

In conclusion, increasing annealing temperature on NiSe has increased the crystallite size and decreased the dislocation density. The electrical resistivity and optical band gap also decreased, while the Hall mobility and thermal diffusivity increased. This work has shown that optimizing the annealing temperature is an important process to improve the quality of the bulk NiSe.

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

SIFAT-SIFAT STRUKTUR, ELEKTRIK, OPTIK DAN TERMA UNTUK NIKEL SELENIDE

By

CHIN PIK YEE

September 2015

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Kajian asas adalah sangat penting untuk menghasilkan ilmu pengetahuan baru. Dalam hal ini, bahan pukal adalah berguna untuk memahami sifat-sifat bahan. Nikel Selenida (NiSe) telah dipilih kerana sifat fotovoltannya yang mengagumkan dan mempunyai aplikasi industri yang luas. Untuk meningkatkan penukaran solar kepada elektrik berkesan, NiSe disintesis melalui kaedah tindak balas keadaan pepejal telah disepuhlindap dalam suhu 423 hingga 823 K.

NiSe adalah dalam keadaan stoikiometri 1:1, fasa tunggal yang berstruktur kristal heksagon. Mikrograf FESEM menunjukkan zarah NiSe adalah dalam saiz nanometer. Apabila suhu penyepuhlindapan meningkat, saiz kristalit meningkat dari 52.7 nm hingga 142.8 nm, manakala ketumpatan kehelan menurun dari 36.01×10^{13} garis/m^2 hingga 4.90×10^{13} garis/m^2. NiSe adalah semikonduktor jenis p. Apabila suhu penyepuhlindapan meningkat, kerintangan elektrik bagi sampel telah berkurang dari 0.0037 Ωcm hingga 0.00015 Ωcm. Tambahan pula, kebolehgerakan Hall meningkat dari 1.21 cm^2/Vs hingga 15.06 cm^2/Vs. Walau bagaimanapun pemalar Hall dan ketumpatan pengangkutan adalah tetap masing-masing pada 0.017 cm^2/C dan 3.19×10^{20} cm^{-2}. Ini menunjukkan pada suhu penyepuhlindapan yang tinggi, saiz kristal akan meningkat menyebabkan kerintangan elektrik berkurang.

Analisis optik menunjukkan NiSe yang telah disepuhlindap mempunyai tenaga jurang jalur peralihan terus dalam julat 2.13 – 2.47 eV dimana bersetuju dengan nilai kesusasteraan. Tenaga jurang jalur bagi sampel berkurang apabila suhu penyepuhlindapan meningkat boleh ditafsir sebagai peningkatan penghabluran. Apabila suhu penyepuhlindapan meningkat, analisis fotoakustik menunjukkan halaju penggabungan semula permukaan berkurang dari 428.39 cm/s hingga 389.62 cm/s, manakala peresapan haba, pemalar resapan dan masa penggabungan semula jalur ke jalur masing-masing meningkat dari 13.42×10^{-2} cm^2/s hingga 20.81×10^{-2} cm^2/s, dari 4.56 cm^2/s hingga 5.10 cm^2/s, dari 1.86 μs hingga 2.46 μs. Ini menunjukkan lebihan pembawa yang meresap melalui permukaan NiSe telah bertambah. Proses penggabungan semula permukaan meningkat disebabkan oleh kehomogenan dan penghabluran sampel meningkat.

Kesimpulannya, peningkatan suhu penyepuhlindapan bagi NiSe telah meningkatkan saiz kristalit, dan mengurangkan ketumpatan kehelan. Kerintangan elektrik dan jurang jalur optik juga berkurang, manakala kebolehgerakan Hall dan peresapan haba meningkat. Kajian ini telah menunjukkan suhu penyepuhlindapan yang optimum adalah proses yang penting untuk meningkatkan kualiti NiSe.

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I certify that an Examination Committee has met on 05.09.2015 to conduct the final examination of Chin Pik Yee on her degree of Master of Science thesis entitled "Structural, Electrical, Optical, and Thermal Properties of Nickel Selenide" in accordance with Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The committee recommends that the student be awarded the Master of Science.

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LIST OF ABBREVIATIONS

α_s	thermal diffusivity
α	absorption coefficient
A	proportional constant
B	magnetic field
β	full-width half maximum (FWHM)
Ø	phase angle
D	carrier diffusion coefficient
D_δ	dislocation density
E	energy
E_g	band gap energy
F	Kubelka-Munk function
f	modulation frequency
f_c	characteristic frequency
h	Planck's constant
hv	energy of photons
I	current
l_s	thickness of the sample
L_g	mean crystallite size
l_β	optical length
λ	wavelength
μ_H	Hall mobility
n	index
N	carrier density
n	natural of the transition

Q	voltage ratios
q	electronic charge
R_∞	diffuse reflectance of the sample
R_H	Hall coefficient
ρ	resistivity
S	scattering coefficient
τ	surface band to band recombination lifetime
τ_{eff}	effective recombination lifetime
V	voltages
v	surface recombination velocity
μ_s	thermal diffusion length
θ_0	initial phase angle
θ	Bragg's angle
ω	light chopping angular frequency

CHAPTER 1

INTRODUCTION

1.1 Introduction

Non-renewable energy fuel has been used for more than 150 years, and the explosion growth of the world population has ever increased our demand on the production of non-renewable energy fuel such as fossil fuel. However, our dependence on the fossil fuel will lead to global energy crisis wherein the decrease of the fuel reservoir will reduce fuel production. The decreasing of fuel production will lead to rising fuel price. This limitation of non-renewable energy and reducing production of non-renewable energy have been the motivation that have driven scientists to explore various approaches for clean, cheap and renewable energy.

The usage of fossil fuel causes the emission of greenhouse gases such as carbon dioxide, methane and nitrous oxide. This will lead to air pollution, water contamination, acid rain and global warming wherein our nature environment will be devastated, and humankind suffer various health problems due to the devastation of the environment. The utilization of nuclear energy can produce tons of energy, but it brings considerations such as proper waste management and safety issues. Thus, clean, cheap, environmentally friendly, sustainable and renewable energy resources are in serious demand. Renewable energy resources such as biomass, hydroelectric, wind, solar, hydrogen fuel, geothermal, ocean and wave are potential candidates that are growing rapidly in terms of research and small scale application.

Solar energy meets the world's energy demand wherein it does not contribute to the greenhouse gases in its energy production. Solar energy is produced directly from sunlight using photovoltaic devices. Photovoltaic device such as a semiconductor device transform photon energy from sunlight into electrical energy by utilizing photoelectric effect in a semiconductor. Photovoltaic device can be easily installed on the roof or the walls of a building. The fabrication process of a photovoltaic device can be different with various materials. Photovoltaic device's performance, cost of manufacturing, toxicity, and durability depend on the materials used and method of fabrication.

Metal chalcogenides semiconductors are formed by compounding sulfur, selenium or/and tellurium with other metal materials such as nickel and copper. Chalcogenides can form binary, ternary and quaternary compounds with other transition metal elements. Selenium based chalcogenides have excellent properties on photon absorption, electrical conduction, and it is relatively easier to control its crystallinity and a low band gap (Zhang et al. 1999, Hankere et al. 2010).

The band gap energy and the production cost are two important factors that need to be considered in producing photovoltaic device. The band gap energy must match with the solar spectrum in order for maximum absorption of solar photons (Zainal et al. 2005). This has resulted in semiconductors from Group VI-VIII to the forefront in consideration for fabrication of polycrystalline chalcogenides semiconductor. Nickel is less toxic and environmental friendly compared to lead and cadmium-based semiconductor (Shi et al. 2013). Nickel is also a cheaper material to fabricate polycrystalline chalcogenide semiconductor.

1.2 Nickel Selenide

Nickel selenide (NiSe) is a binary semiconductor compound which has received extensive attention because it exhibits desirable electronic and magnetic properties for solar energy production (Zhuang et al. 2006). NiSe is a p-type binary metal chalcogenide semiconductor with optical band gap of 620 nm (2.0 eV). Resistivity of NiSe thin film is $10^2 \Omega$cm at room temperature. (Mane et al. 2000, Anuar et al. 2004, Zainal et al. 2005, Zhuang et al. 2006, Moloto et al. 2009, 2011, Sobhani et al. 2011, 2012). It has a wide range of potential applications in the field of material science fabrication of photovoltaic, electronic and optoelectronic devices such as optical recording material, solar cell, sensor, laser materials, optical fibers, superionic conductor, photodetectors, window material, thermoelectric converter, optical filter, electro-optical devices, and storage energy material (Xue et al. 2006, Moloto et al. 2009).

This research focused on NiSe compound, which has shown impressive photovoltaic properties and a wide range of industrial applications. Fundamental studies are paramount to generate new knowledge of the underlying foundations of phenomena and understanding that may prove crucial in applied research. Therefore, the relationship between synthesis method and the material’s structural and physical properties will contribute in the development of greater fundamental science and engineering basis for the material. The fundamental investigation such as structural and physical properties (electrical, thermal, optical and transport properties) of nickel selenide compound will be carried out in this research. The research findings will help to further advancement of knowledge in basic material physics, which is important for technological application such as device substrate characterization and processing control.

In fundamental material physics studies, bulk material is useful to understand the characteristic of the material. Thus, the first objective of the present study was to synthesize the nickel selenide compound by using solid state reaction. This will be followed by characterizing the structural, optical, electrical, thermal, and transport properties of the materials via XRD, FESEM, EDX, ED-XRF, UV-Vis-NIR, Van Der Pauw technique, TGA, laser flash, and photoacoustic technique respectively. The bulk samples were heated under nitrogen gas to study the annealing effect on structural, thermal, optical, electrical and transport properties. The performance characteristic can lead us to understand the material properties. Consequently, these findings will provide the fundamental knowledge that will help to improve the efficiency, design structure and decrease the manufacturing cost for photovoltaic solar cells devices.

From the survey of published work on the material under study, there is no detailed investigation especially on heat diffusion and carrier transport properties in nickel selenide bulk samples. Thus, the current work was carried out to study thermal and carrier transport properties of the nickel selenide bulk samples by using photoacoustic technique. The thermal and transport properties of nickel selenide bulk sample would provide useful information in future fabrication process of selenide based photovoltaic devices.

1.3 Photoacoustic Technique

Photothermal science is a study on phenomena based on the conversion of absorbed optical energy into heat. Alexander Graham Bell had carried out an experiment on a condensed matter sample (Favier, 1997) and found that when light beam periodically shines on the surface in an enclosed cell, it will produce a loud signal. This phenomenon known as photoacoustic which is an indirect acoustic signal produced by photothermal heating effect.

Rosencwaig and Gersho (1976) developed a thermal piston model where the pressure in the photoacoustic cell depends on the heat generated by the absorbed light. From the phenomena, a theory on the photoacoustic effect was derived which is known as R-G theory. This theory is the basis for other theories using microphone as photoacoustic detection on a solid sample. Since 1970's, the R-G theory has been widely used in subsequent photoacoustic study. The intense light sources, sensitive detection equipment and sensitive signal processing equipment are the three major factors in developing the photoacoustic research. The sensitivity of photoacoustic spectroscopy can be increased by using lasers or high-pressure arc lamps, sensitive detector such as a condenser and electrets microphones, and processed by filters and sensitive lock-in amplifiers. Photoacoustic spectroscopy is a very fast, sensitive and accurate tool for measuring the optical, thermal and carrier transport properties of solid samples. In this research, photoacoustic technique is used to characterize the optical, thermal and carrier transport properties of nickel selenide.

1.4 Problem Statement

Metal chalcogenidehas excellent properties on photon absorption, electrical conduction, and have low band gap (Zhang et al. 1999, Hankere et al. 2010). To optimize photons absorption, the band gap energy must match with the solar spectrum (Zainal et al. 2005). Fundamental investigation on the structural and physical properties (electrical, thermal, optical and transport properties) is crucial to understand these properties. In addition, studying the annealing effect on the material will provide the fundamental knowledge that helps to improve the solar conversion to electric. Work from earlier researchers show that there is a need to further investigate on areas such as heat diffusion and carrier transport especially in nickel selenide bulk samples. Thus, the current work was carried out to probe the annealing effect on the thermal and carrier transport properties of the nickel selenide bulk samples by using photoacoustic technique. The thermal and carrier transport properties of nickel selenide bulk sample would provide useful information in future fabrication process of selenide based photovoltaic devices.

1.5 Research aim

The research aim is to investigate the effect of annealing on transport properties of NiSe.

1.6 Research objectives

The objectives of the present work are as follow:

1. To synthesize the polycrystalline nickel selenide powder by using solid state reaction method.

2. To characterize and to study the annealing effect on the structural, composition, morphology, electrical, optical and thermal properties of the NiSe polycrystalline metal chalcogenides compound.

1.7 Scope of the study

This thesis is organized into five chapters. Chapter 1 is a brief introduction on motivation of the study which included the problem statement, aim and objectives of the research.

Chapter 2 is a survey of previous works on preparation method of nickel selenide. This chapter also discusses on previous research on the characteristics of the nickel selenide, and photoacoustic characterization of semiconductor.

Experimental details on the method of preparation of nickel selenide are discussed in Chapter 3. This includes the experimental setups and characterization techniques of XRD, ED-XRF, EDX, FESEM, TGA, laser flash, Van der Pauw, UV-Vis-NIR and photoacoustics spectroscopy.

All the experimental results on the structural, composition, morphology, electrical, thermal, optical and transport properties of nickel selenide are reported and discussed in detail in Chapter 4. The annealing effect on the structural, composition, morphology, electrical, thermal and optical properties of NiSe are also elaborated in detail in this chapter.

Finally, Chapter 5 summarizes all the present analysis work and provides conclusions on what valuable information has been learned beyond previously published work. Recommendations are also given for future research directions and experiments needed in this research area.

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