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MECHANICALLY-INDUCED DEFECTS IN MILLED CdZnSe NANOCRYSTALLINE POWDER

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By

IBRAHIM MUHAMMAD BAGUDO

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the degree of Doctor of Philosophy

November 2016

DEDICATION

Dedicated to the entire People of Bagudo Local Government, My family and Parent

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

MECHANICALLY INDUCED DEFECTS IN MILLED CdZnSe NANOCRYSTALLINE POWDER

By

IBRAHIM MUHAMMAD BAGUDO

November 2016

Chairman : Professor Zainal Abidin Talib, PhD
Faculty : Science

High-quality CdZnSe nanocrystals with diameters ranging from 2.6 nm to 4.3 nm were synthesized via high and low energy mechanical milling for 20 h and 100 h respectively. The XRD diffractograms of the milled powders consist of three major diffraction peaks indexed to the lattice planes (111), (002), (220) and (311) of the compound CdZnSe in the cubic phase structure. The optical spectra of the nanoparticles exhibited an onset absorption peak at 349 nm, with maximum absorption from 250-290 nm. The photoluminescence (PL) spectra exhibit broad emission bands in the wavelength range 350-900 nm. Band emissions of 1.74 eV, 1.54 eV and 1.4 eV at longer wavelengths were associated with the surface state defects. Time-resolved photoluminescence (TRPL) and photoluminescence (PL) spectroscopy measurements were carried out on mechanochemically alloyed CdZnSe nanocrystals. The TRPL emissions exhibit bi-exponential decay dynamics consisting of an initial fast component over a range of 0.05-0.87ns and a slower component (0.76-1.60 ns). The chemical nature of the mechanically induced paramagnetic defect centers induced during mechanical alloying was investigated using electron spin resonance (ESR). The ESR spectra display a faint signal at g~3.9 with a spin density of the order of ~3.34 X 10^4 spins/g attributed to Fe^{3+} introduced from the grinding medium. The intensity, linewidth and g-value of the dominant signal increase linearly with increasing milling time. The paramagnetic defect increased linearly from ~1 X 10^{20} spins/g to 7 X 10^{20} spins/g similarly, g-values increases from 1.9993(3) to 2.0026(4) and linewidth (ΔB_{PP}) from (10.89 to 40.27) mT. The center is believed to consist of several overlapping signals arising from different paramagnetic centers present in the milled sample with Zn_i-V_{Zn} predominant. The low temperature PL analyses indicate that the optical transition that gave rise to the peak at high photon energy involves an electron trapped at donor defect and hole trapped at an acceptor in association with residual impurities from the grinding medium (V_{Zn}-X), where X represents residual impurities. The large blue shift in the energy band gap of the milled samples involved Burstein-Moss effects and transitions due to

trapped charge carriers. The high-resolution transmission electron microscopy (HRTEM) histogram reveals that the centre of size distribution was 3.50, 2.66, and 2.00 nm for samples milled for 5 h, 10 h and 20 h respectively. High-transmission electron microscopy (HRTEM) revealed the successful annihilation of such defects with continuous milling. Stoichiometric defect due to slow diffusion of zinc has been identified as the major source of defect. The most probable induced centers during mechanical milling were (V_{Zn}–Zn_i), V_{Zn}, and V_{Cd}.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

KECACATAN MEKANIKAL TERARUH DALAM PENGISARAN SERBUK NANOKIRISTAL CdZnSe

Oleh

IBRAHIM MUHAMMAD BAGUDO

November 2016

Pengerusi : Profesor Zainal Abidin Talib, PhD
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Nanokristal CdZnSe berkualiti tinggi dengan garispusat di antara 2.6 nm dan 4.3 nm telah disintesis dengan cara pengisaran mekanikal (secara intensif dan tenaga rendah) selama 20 hingga dan 100 jam. Spektrum pembelauan sinar-X menunjukkan bahawa CdZnSe mempunyai tiga puncak pembelauan utama yang berindeks pada satah hkl (111), (002), (220) dan (311) dalam struktur fasa kubik. Spektrum optik nanopartikel menunjukkan penyerapan puncak mula di 349 nm dan penyerapan maksimum di 250 -290 nm. Spektrum (fotolumininesens) menunjukkan jalur pemancaran yang luas pada julat (350-900) nm panjang gelombang. Jalur pemancaran 1.74 eV, 1.54 eV dan 1.4 eV pada gelombang yang lebih panjang dikaitkan dengan kecacatan keadaan permukaan. Fotoluminesens revolusi masa (TRPL) dan fotoluminesens (PL) spektroskopi telah dijalankan ke atas nanokristal CdZnSe yang dialoi secara mekanikal. Pemancaran TRPL mempamerkan pereputan dinamik dwi-eksponen yang terdiri daripada komponen cepat dari 0.05 hingga 0.87ns dan komponen perlahan (0.76-1.60 ns). Sifat kimia kecacatan paramagnet mekanikal terjadi semasa proses pengaloian mekanikal secara tenaga tinggi dengan disiasat menggunakan resonans spin elektron (ESR). ESR spektrum terdiri daripada isyarat malap di g ~ 3.9 dengan kepadatan spin pada kuasa 3.34 X 10^4 spin/g; isyarat ini adalah disebabkan oleh Fe^{3+} daripada bahan pengisaran . Keamatan, lebar jalur dan nilai-g isyarat dominan meningkat secara linear dengan meningkatkan masa pengisaran. Kecacatan paramagnetik meningkat secara linear dari ~ 1 X 10^{20} spin/g hingga 7 X 10^{20} spin/g , nilai-g meningkat dari 1.9993 (3) ke 2.0026 (4) dan juga lebar garis (ΔB_{pp}) dari 10.89 hingga 40.27 mT . Pusat dipercayai terdiri daripada beberapa isyarat bertindih yang timbul disebabkan oleh kewujudan pusat paramagnet yang berbeza semasa proses pengisaran dengan Z_{ni}-V_{Zn} yang dominan. Analisis PL bersuhu rendah menunjukkan bahawa peralihan optik yang menimbulkan puncak pada tenaga foton tinggi yang melibatkan elektron terperangkap di kecacatan penderma dan lubang terperangkap di penerima yang bersama dengan bendasing meggunakan pengisaran sederhana (V_{Zn}-X), di mana X mewakili bendasing. Peralihan biru

yang besar dalam jurang jalur dapat dilibatkan dengan kesan Burstein Moss dan peralihan disebabkan oleh pembawa cas terperangkap. Histogram resolusi tinggi penghantaran elektron mikroskop (HRTEM) menunjukkan bahawa taburan saiz adalah 3.50, 2.66, dan 2.00 nm untuk sampel untuk masa pengisaran 5, 10 dan 20 jam. Resolusi tinggi penghantaran elektron mikroskop (HRTEM) menunjukkan bahawa kecacatan berjaya dihapuskan dengan pengisaran berterusan. Kecacatan stokiometri disebabkan oleh penyebaran zink yang lambat dan dikenalpasti sebagai sumber kecacatan utama. Kemungkinan besar pusat kecacatan teraruh semasa proses pengisaran mekanikal adalah (V_{Zn}–Zn_i), V_{Zn}, dan V_{Cd}.

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I certify that a Thesis Examination Committee has met on 9 November 2016 to conduct the final examination of Ibrahim Muhammad on his thesis entitled "Mechanically-Induced Defects in Milled CdZnSe Nanocrystalline Powder" in accordance with the 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 Doctor of Philosophy.

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LIST OF ABBREVIATIONS

A	Absorbance
A.U	Arbitrary unit
Cd_i	Cadmium interstitial
DD	Deep Defect
CdSe	Cadmium Selenide
CdS	Cadmium Sulphide
DLE	Deep Level Emissions
DL	Deep Levels
DAP	Donor Acceptor Pairs
DLTS	Deep level transient Spectroscopy
E_C	Conduction band energy
E_F	Fermi level
EM	Electromagnetic spectrum
E_t	Trap energy (eV)
EPR	Electron paramagnetic resonance
f	Frequency (Hz)
FESEM	Field Emission Scanning Electron Microscopy
IPD	Intrinsic Point Defect
HRTEM	High Resolution Transmission Electron Microscopy
LTAQ	Mechanism of luminescence anti quenching
MBE	Molecular Electron Beam
MOCVD	metal organic chemical vapor deposition
NBE	Near Band Edge emission
NB	Near Band
ODMR	Optically detected magnetic resonance
OMVPE	organometallic–vapor-phase epitaxy
PL	Photoluminescence Spectroscopy
SEM	Scanning Electron Microscopy
TRPL	Time Resolved Photoluminescence Spectroscopy
T	Time
TEM	Transmission Electron Microscope

UV-Vis	UV-Visible absorption
V_{Cd}	Cadmium Vacancy
V_{Zn}	Zinc Vacancy
V_{Se}	Selenium Vacancy
XRD	X-ray Diffraction Analysis
XRF	X-ray fluorescence spectrometer
ZnS	Zinc Selenide
Zn_i	Zinc interstitial
θ	Angle of incidence
h	Plank constant
hv	Incident photons
I	Intensity

CHAPTER 1

INTRODUCTION

1.1 Research background

The high cost of vacuum based technologies for crystal growth has triggered a renewed effort in the search for deposition methods which are cost effective. On the other hand, non-vacuum deposition methods such as mechanical attrition and hydrothermal techniques are known to induce a high density of structural defects during crystal growth. However, our basic knowledge of thermodynamics tells us even if macroscopic samples are of the highest achievable purity, foreign atoms are always present; in other words, no crystal is completely free from induced defects [1].

Basic research on the defects in semiconductors is of vital importance, since their electrical and optical properties are strongly dependent on the density and the distribution of localized states in the band gap [2]. A knowledge of the rate, mechanism and the control of formation of defects in semiconductors is a key to developing the technology for realizing the ultimate potential of modern electronics. It is obvious that defects are detrimental to device performance and effort should be made to reduce their concentration below certain threshold during crystal growth [3]. Recently, a new trend has appeared in which mechanically induced defects generated via plastic deformation by high energy ball milling had been utilized to enhance magnetic properties, induce paramagnetic and spin glass behavior in nanocrystalline materials [4],[5],[6].

Mechanical milling is associated with the following fundamental processes during grain refinement [7],[8],[9]

I. atomic- level disordering
II. formation of vacancies, vacancy clusters, interstitials, grain boundaries, dislocations
III. atomic level strain
IV. contamination by residual metal impurities from the grinding media
V. change in morphology and size reduction.

1.1.1 Atomic-scale disorder

Atomic-scale chemical disorder, occurs when large fractions of atoms are forced into common interface from which they undergo mixing and alloying. The energy required for interface formation and atomic motion comes from the collision of the balls with grinding medium.

1.1.2 Defect generation by mechanical milling

In mechanical milling, lattice defects are generated continuously. In fact, during the mechanical milling process, a considerable amount of mechanical energy is generally stored in the of form lattice defects such as vacancies, interstitials, dislocations and grain boundaries [10],[11]. A relatively high concentration of lattice defects is required to stabilize metastable states and enhance reactivity of the elemental constituent during milling process. A crystal defect can be regarded as any permanent perturbation of the crystal structure. Perturbations in the crystal lattice extending for a particular atomic distance in any direction are related to intrinsic point defects (IPD). These consist of vacancies, interstitial atoms and Frankel pairs and are of particular importance in this work [12]. There is much interest in the identification of IPD which come into existence when lattice atoms are displaced from the normal site to an interstitial site. Intrinsic point defects are generally present in group II-VI compounds in significant concentration due to the conditions of thermodynamic equilibrium under which the crystal was grown [13]. These conditions result in departure from stoichiometry and depend on atomic interactions among the elemental constituents of the mixture.

The determination of formation mechanism and evolution of IPD present a considerable challenge as does the identification and quantification of the associated energy level in the forbidden gap. The introduction of IPD induces the displacement of the lattice atoms that surround it. The atoms involved are first, second, etc. neighbors in terms of their arrangements and depending on the extent of the perturbation. If a large distortion of the lattice surrounding an isolated vacancy occurs, the Jahn-Teller effect stems in. An isolated vacancy thus formed would constitute a deep level in the forbidden energy region [14].

Depending on the charge state, the IPD are capable of introducing localized state in the semiconductor prohibited region of both donor and acceptor levels that could be located at different distances from the conduction and valence band.

1.1.3 Contamination by residual metal impurities from the grinding media

An extrinsic atomic defect involves foreign atoms which have been introduced unintentionally during crystal growth. Residual metal impurities could substitute some atoms in the host material due to wear and tear of the grinding media. The residual impurities consists mainly of (Fe, Ni and Cr) if the grinding media is made up steel. Extrinsic defects arise due to the introduction of atomic impurities into the lattice. Since foreign atoms may interact strongly with intrinsic defects and the host material, the effect of this interaction should also be considered in milled powders. It has been established that over a wide temperature range, foreign interstitials may migrate towards intrinsic defects and interact with them [12].

1.1.4 Particle size reduction

In general, particle size reduction is accompanied by an increase in defect concentration and micro strain during mechanical milling. As the particle size decreases to nanometer scale, the surface-to-volume ratio increases. This leads to an increasing percentage of atoms on the surface of the particles compared to those at the interior. The surface atoms are chemically more active due to their higher adjacent coordinate atoms and increased number of dangling bonds. As the result of these imperfections at the surface of the particles, additional electron states are introduced in the band gap of the material . This also influences the system Hamiltonian [15].

The surface states act as electron or hole traps. Fast and efficient trapping of the photogenerated carriers on the surface of the particles should be expected. The resultant emission band is also expected to be broadened and highly Stokes-shifted [16].

1.2 Problem statement

The research of mechanically induced defects in solid state follows two main trends: investigation into structural changes and study of thermodynamic effects. The purpose of this research is to determine the extent of deviation from ideal lattice arrangement during crystal growth. We intend to identify and evaluate the concentration and effect of mechanically induced defects introduced during the milling process. To the best of my knowledge this is the first time in which such investigation of induced mechanical defects has been carried out on CdZnSe nanocrystals.

1.3 Hypothesis

The hypothesis of this study is that lattice distortion that occurs during severe plastic deformation is expected to result in deviation of an ideal lattice arrangement of the initial material. This will generate mechanically induced defects in the milled powder.

In addition, stoichiometric defects are expected due to the differences in melting point and hardness of the elemental constituents of CdZnSe samples used. This is due to inability of the elements to adequately diffuse during the alloy formation. As such Localized states at the band gap are expected to introduce additional electronic states or paramagnetic defects that will act as traps for photogenerated carriers.

The lifetime of the carriers is expected to decrease with increasing milling time due to increase in concentration of mechanically induced defects.

A deviation from the Curie Law for paramagnetic defects measured at low room temperature by ESR is anticipated due to residual metal contaminants in the host material.

1.4 Research questions

- **I.** What is the effect of milling parameters, i.e, milling time and energy input on the generation of mechanically induced defects?
- **II.** How does residual contamination affect the properties of the milled powders?
- **III.** What is the effect mechanically induced defects on the carrier dynamics (average life time) of the resultant material?

1.5 Research objectives

Based on the research questions outlined above, the major goal of this work is to identify and quantify various structural defects that are mechanically induced. In order to achieve this aim the following objectives have been set:

- I. To synthesize CdZnSe nanocrystals by high and low energy ball milling using variable milling time and intensity of the milling. X-ray diffraction (XRD) is employed to evaluate the rate at which elemental constituents are forced into chemical mixing.
- II. To identify and quantify mechanically induced defects present in the resultant powder using electron spin resonance (ESR), continuous wave photoluminescence (cw-PL) spectroscopy and X-ray fluorescence (XRF).

III. To determine the effect of residual metal impurity contaminants on the chemical properties of the milled powder by low temperature ESR analysis.
IV. To determine the effect of mechanically induced defects on the life time of the carriers.

1.6 Scope of the study

Chapter One: Provides an overview of the main subject of the study, which is introduction of defects semiconductor materials during crystal growth. The importance of this topic in the semiconductor industry lies in the need to reduce the production cost of opto-electronic devices in terms of eliminating vacuum deposition methods. The aim and objectives of the study have been highlighted.

Chapter Two: This chapter focuses on the defect centers found in group II-IV semiconductors. The defects were induced through different synthetic methods. The corresponding energy levels of the defects in the band gap have been provided. The origin to these defects has been discussed.

Chapter Three: This chapter discusses the theoretical foundation required to understand defect physics in semiconductor materials. Emphasis was given to electron spin and photoluminescence spectroscopy.

Chapter Four: The experimental procedure adopted to synthesize the semiconductor nanocrystal is presented. High energy ball milling was used to synthesize the milled powders under intensive and low input energies. The aim is to understand the evolution of mechanically induced defects with variable milling parameters.

Chapter Five: This chapter concentrates on understanding the mechanism of alloy formation through structural, morphological and evolution of the phases. The allowing rate of the individual elements (Cd, Zn and Se) was monitored by XRD diffractometry. The HRTEM and FESEM provide information on the structure and morphology of the milled powders.

Chapter Six: This chapter is mainly concerned with the optical properties of the milled powders. The evolution of defects with milling time was discussed both at room temperature and below room temperature. The origin of large shifts in the energy band gap of the milled powders was reconsidered to include contributions from defects and impurities present in the milled samples. UV-visible and photoluminescence spectra in the high energy region are presented and discussed.

Chapter Seven: The results from electron spin spectroscopy and photoluminescence spectroscopy in the low energy region of the spectrum are presented and discussed. The evolution of the mechanically induced defects and impurities has been accurately quantified. The different number of spin taken part in the transition was analyzed. The consequences of the introduction of impurities from the grinding media into the host materials have been confirmed to produce a dilute magnetic behavior in the milled powders.

Chapter Eight: This last chapter presents conclusions that have been deduced from the results and recommendations for the use of high energy ball milling.

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