Comparative Study of Laser Ablation and Chemical Synthesis of some Nano Chalcogenides

Thesis
Submitted for the degree of M.Sc in Science as a partial fulfillments for the requirements of the Master of Science

Submitted to
Physics Department - Faculty of Science
Ain Shams University

Presented by

Ayman Mohamed Mahmoud Mostafa

B.Sc. Chemistry & Physics (2007)
Cairo University

2013

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APPROVAL SHEET

Title of the M.Sc. Thesis

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Award Date: 2013

"In the name of ALLAH"

Cordial thanks and deepest appreciation to my lovely mother, father, brother, support me and fill my life with love and joy. Great appreciations to my wife, Eman, for her accompany, encouragement, and support through my thesis work. Finally I want thank everyone for giving me enough encouragement to reach for my dreams.

Acknowledgment

First, all praises and thanks to ALLAH -who is the ultimate source of knowledge to man without whose gracious help it would have been impossible to accomplish this work.

I am pleased to express my deep appreciation to my advisor ***Prof. Dr. Hassan Talaat****, Professor of Physics, Faculty of Science, Ain Shams University, for his continuous encouragement, helpful suggestions, and valuable comments throughout this work.*

It gives me the greatest pleasure to express my deep thanks to ***Prof. Dr. Ali Shabaka****, Professor of Spectroscopy and the Leader of Laser Group at* ***N****ational* ***R****esearch* ***C****enter (NRC), who always gives me good advices with my synthesis problems and the first who introduced me and others to the promising area of laser research.*

Also, I sincerely express my deepest gratitude for my dear big brother ***Dr. Wael Hossam Eldin,*** *Researcher of Spectroscopy,* ***N****ational* ***R****esearch* ***C****enter (NRC), for his assistance during my work.*

I am ultimately grateful to researchers, friends and colleagues in National Research Center (NRC) for their faithful help and their beneficial support during the research program.

Also my special thanks to my University Ain Shams University, Physics Department colleagues and Staff.

Finally, I would like to thank my parents and all my family members for their support and caring attitude which kept me going until the end of my work.

Abstract

In this thesis, cadmium sulfide (CdS) nanostructures with different sizes and shapes were prepared via two different pathways; chemical precipitation method (CPM) and pulsed laser ablation in a liquid environment (PLAL)

In the first part of this work, a simple synthesis method, using $CdCl_2$ as cadmium source and Na_2S as sulfur source in a closed reaction vessel equipped with ethylenediamine (en) solution, has successfully prepared a one-dimensional CdS nanostructure arrays. The method leads to reproducible results at low growth temperature. The study indicates that the Cd:S molar ratio, the reaction temperatures, and the amount of en are determining factors in controlling the particles size and shape. The Transmission Electron Microscope (TEM) images show that when the reaction temperature was carried out between 30-60°C, Tripods nanostructures were formed with the arms no uniform but their diameters have a relatively narrow size distribution. Then by raising the temperature from 60°C to 80°C, the resulting shapes are commonly uniform tripod product. Increasing further the reaction temperature to 150°C, the resulting CdS nanorods become

distorted. TEM images also show that more uniform CdS tripod nanostructures are formed at Cd:S molar ratio of 1:7 and 1:4. As the amount of en dissolved in distilled water increases from 25% using different molar ratio, the produce nanorods decrease their length till they become spherical shapes at 100% of en. The X-ray diffraction (XRD) results indicated that the prepared CdS nanostructures are crystalline with hexagonal lattice structure. The absence of $CdCl_2$ or Na_2S characteristic peaks reflects the completeness of the reaction and the purity of the prepared CdS.

From the UV-Visible spectrum, the average particles size calculated by varying the reaction temperatures from 30°C to 150°C change from 45.1nm to 12.74nm . The band gap energies gradually decrease from 2.88eV to 2.46eV. These changes have been attributed to the crystalline size-dependent properties of the energy band gap. And it's evident that the CdS synthesized at 30°C by precipitation of Cd^{2+} cations with S^{2-} anions shows the largest blue shift relative to the bulk material whereas that of the 150°C temperature shows the smallest shift. By varying the precursors molar ratio Cd:S, the calculated average particles size changed from 4.25 (for 1:1) to 2.95 (for1:4).

So, the band gap energies gradually increase from 2.94eV to 3.52eV.

In the second part of this work, CdS nanostructures were synthesized by PLAL method that was tightly controlled during the ablation process. There are a number of factors playing a crucial role in controlling shape and size of the produced nanostructures. We optimized these factors by controlling parameters of the PLAL system, controlling the used laser parameters, controlling the ablated materials and its medium. The controlling of PLAL system was carried out by studying the sound intensity produced by laser-matter interaction, the absorption spectra of the colloidal solutions, and the particles size and size distribution of the nanostructures as a function of the target position with respect to the lens focal plane. The adjustment of the ablated materials and its medium was carried out by studying the ablated target, the electrolyte solution, and the type of water.

The synthesis of CdS nanostructures by PLAL was carried via two ways; the first one is Top-Down technique while the other one is Bottom-Up technique which is introduced here for the first time. Samples were also characterized by using TEM, and Ultraviolet– visible

Spectrophotometer (UV-vis). The produced nanostructures have different shapes and sizes controlling by the previous factors.

TEM images of PLAL of S-target immersed in $CdCl_2$ solution by ns-laser (1064nm, 40J/cm^2) show that CdS nanostructures have an external spherical shape with a very narrow size distribution with diameters in the range of 5–10nm. When the pulse width was decreased to be ps-laser (800nm, 0.9mJ) ablation of S-target immersed in $CdCl_2$ solution, the product was ropes or hair-like in shape. The formed nanostructures have diameters in the range of 10-15nm and the formation of other nanostructures was not observed. When the pulse width was further decreased to be fs-laser (800nm, 0.7mJ), the product was self-assembly nanorods spontaneously produced without using any surfactant.

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