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The Effect of Varying Furnace Angle in the Synthesis of CdS Nanomaterials using Vapor Phase Crystal Growth Technique

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Abstract—CdS nanomaterials were successfully synthesized at varying furnace angle of 0°, 45°, and 90° with a growth temperature of 800^{0}C and dwell time of 8 hours using the vapor phase crystal growth technique. The synthesized product was a yellow-orange material and was characterized using the SEM, EDX, and photoluminescence spectroscopy. The SEM photomicrographs revealed various nanostructures in a form of nanowires. Energy dispersive X-ray analyses confirmed that CdS nanomaterials showed a uniform composition of Cd and S with a minimal presence of impurities. The PL spectra of the samples revealed intense peaks in the range of 477 nm to 548 nm wavelengths where the energy band gap was obtained at 2.44 eV which is approximately that of CdS.

Keyword —CdS nanomaterials, furnace angle, vapor phase crystal growth technique

1 INTRODUCTION

Nanotechnology is a field of applied science and technology whose theme is to control materials and to fabricate devices on the atomic and molecular scale, generally between 1 and 100 nanometers[1]. This is a highly multidisciplinary field, benefiting from the efforts and developments in many fields like medicine and sciences. In the near future, nanoelectronic devices may replace microelectronics in communication and computer industries with nanostructures [2]. The emerging field of nanotechnology, materials and devices on the nanoscale, has the potential to take different fields further than ever imagined [3]. This is possible because reducing the size of a material to nanoscale proportions alters its bulk electronics, magnetic, and optical properties [4], [5].

In the past decade, the synthesis and characterization of controlled shape and structure nanometersized metal sulfide has been attracting an increasing interest because of their excellent physical and chemical properties, which are not available in their bulk material. Among these materials, CdSnanocrystalline, as semiconductors, are important because of their potential applications in photoelectronic transition devices[6], [7], [8].

Cadmium Sulfide (CdS), an important II-VI semiconductor, shows important applications in different fields especially in optical devices. The enhanced properties of CdS enable multiple new applications including the integration of nanomaterials into nanodevices such as biological labeling[9], photovoltaic (PV) solar cells[10], lasers[11], light emitting diodes (LED)[12], optoelectronic devices, and a wide array of photosensors[13].

At present, there are numerous reports on the preparation of CdS nanoparticles like nanowires[14], [15], [16],nanorods[17], [18], quantum dots[14], [19], thin films [20], [21], [22], nanobelts[23], [24], dendrites [25], nanoribbons [28], nanospheres,nanoshuttles, nanotubes[29] and other nanostructures. Several deposition methods and techniques were employed including the ultrasonical colloidchemistry deposition [20], metalorganic chemical vapor deposition [21], hydrothermal treatment [25], [26], [27], thermal evaporation [28], Solvothermal method[15], [16], chemical bath deposition[22], gas diffusion[30], microemulsions[18], reverse micellesystems[29], sol-gel route[31],in situ simultaneous copolymerization-sulfidation[32], liquid-phase deposition[33] and irradiation[34]. Some of the methods used are very time consuming and others result in a textured film. In this work we demonstrate the use of vapor phase crystal growth technique as a simple and affordable way of preparation of CdS nanomaterials.

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2 Experimental Section

2.1 Preparation of the Samples

The procedure started with the sealing of one end of the quartz tube using a high temperature blowtorch (mixture of LPG and oxygen). After which, it was cleaned using an ultrasonic cleanserfor 30 minutes, rinsed, and dried. Approximately 0.035 grams of high purity CdS powder (99.99%) was loaded into the quartz tube and was connected to the Thermionics High Vacuum System for sealing at a high vacuum pressure of ≈10^{-6}Torr as seen in Fig.1.

Fig. 1 Quartz tube connected to the high vacuum system. Inset shows the sealing of the quartz tube.

2.2 Growth of CdS Nanomaterials

The sealed quartz tube was placed in a tube furnace and was baked at a constant ramp rate of 80 minutes. The furnace was programmed at growth temperature of 800 ^{0}C, dwell time of 8 hours, ramp rate and was set at varying angles (0°, 45°, 90°) as shown in Fig. 2. To maintain a gradient necessary for the growth of nanomaterials, the quartz tube was inserted halfway through the furnace with zone 1 completely inside as presented in Fig. 3. Zone 2 is at the opening and zone 3 is outside the furnace. This caused the CdS powder at the hotter end (zone 1) to evaporate and condense at the colder part (zone 2 and 3) of the quartz tube. The quartz tubes were then allowed to cool down to room temperature.

Fig. 2Furnace set at different angle a) 0^0, b) 45^0, and c) 90^0

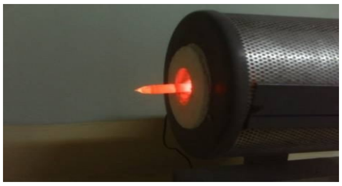

Fig. 3 Set-up for growing the CdS nanomaterials at furnace set at 0^0

2.3 Grown Nanomaterial Retrieval

Once the quartz tube has already cooled down to room temperature, it was slowly cracked in a fumehood environment. Fragments of the cracked quartz tube with deposits of CdS nanomaterials (Fig. 4) were subjected for characterization.

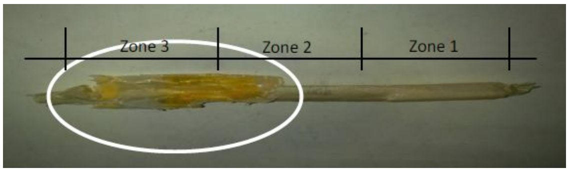

Fig. 4 Deposit CdS nanomaterials in a cracked quarts tube

2.4 Characterization

The deposited CdS nanomaterials were characterized using the SEM, EDX and PL equipment as seen in Fig. 5.The effect of the varying furnace angle will be analyzed by comparing the SEM images, EDX analyses and PL spectra of the grown nanomaterials.

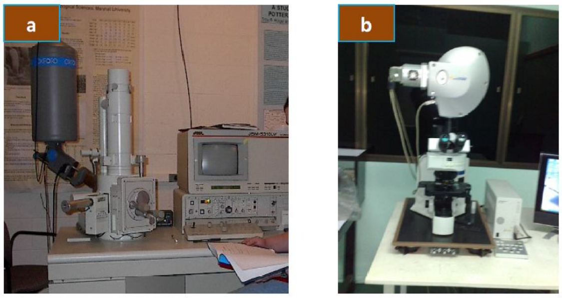

Fig.5. Instruments used in CdS nanomaterials characterization, a) Scanning Electron Microscope, and b) Spectral Microscope

3 Results and Discussion

3.1 SEM Results of the CdS Nanomaterials Grown at Different Furnace Angle

Fig.6revealed the SEM images of various nanostructures prepared at different furnace angle (0^0, 45^0, and 90^0),with constant growth temperature of 800^0C and dwell time of 8 hours. Cotton like nanowires are observed at a furnace angle of 0^0 with a diameter ranging from 181 nm to 403 nm. Fewer nanowires but smaller in diameter with dimensions of around 30-68 nm were synthesized at a furnace angle of 45^0. A prominent image of the nanowires is formed when the furnace angle is in the vertical position with diameter range of 14-50 nm.

An increase in furnace angle leads to a greater amount of dissociated CdS circulating within the tube involving the upward and downward movement in response to gravity, resulting to subsequent condensation and formation of nanostructures. This explains why eminent nanostructures such as nanowires are observed at higher furnace angle.

The deposition of CdS nanomaterials in lower temperature zone can be attributed to the angle between gravitational field and orientation of tube as affected by temperature gradient. The value of acceleration due gravity inside the quartz tube with respect to temperature gradient is proportional to furnace angle as shown in Fig. 7.

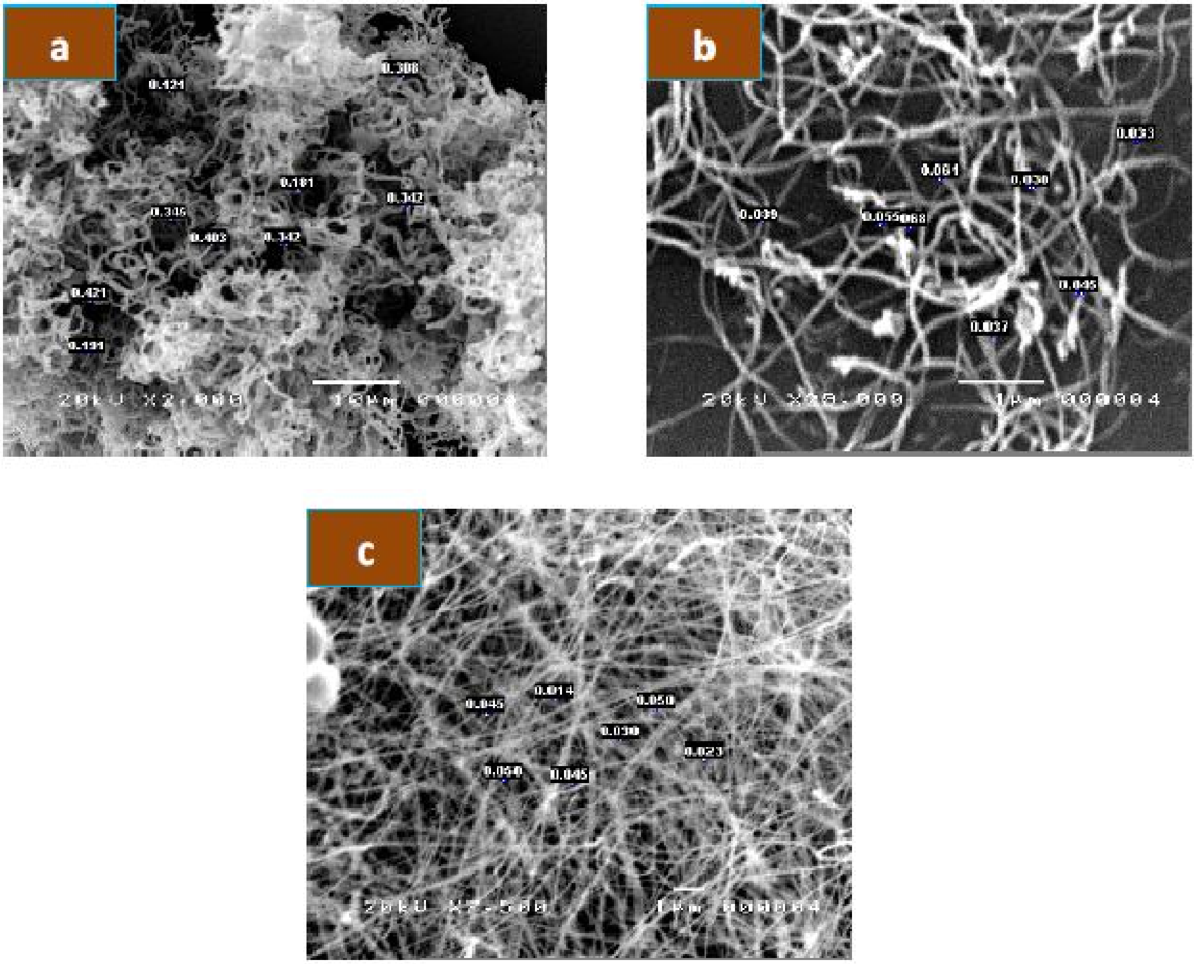

Fig. 6 SEM images of various nanostructures prepared at 800 ^{0}C temperatures with dwell time of 8 hours and different furnace angle a) 0^0,b) 45^0, and c) 90^0.

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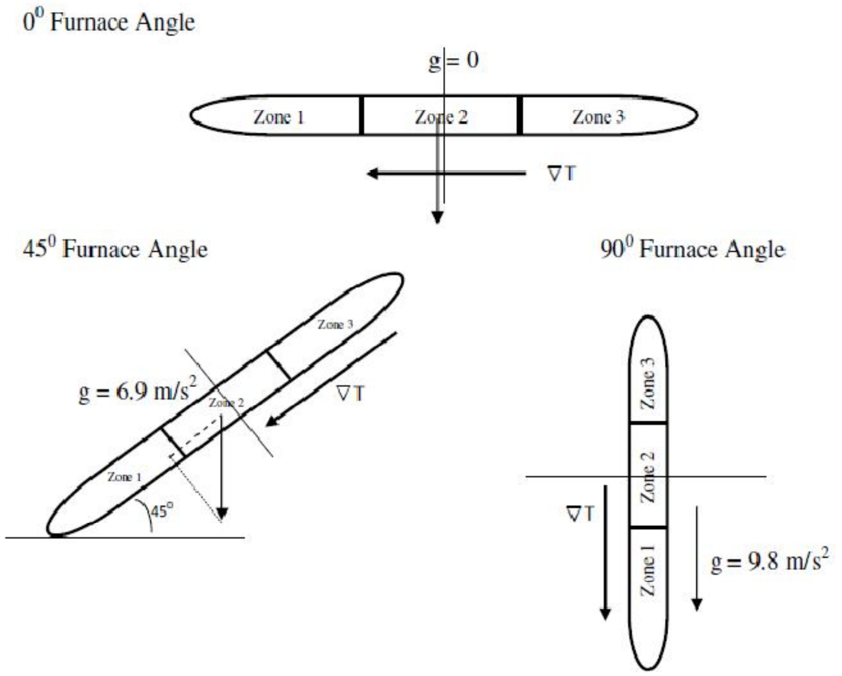

Fig. 7 Diagram of the angle between gravitational field with respect to temperature gradient and orientation of tube

3.2EDX Results of the CdS Nanomaterials Grown at Different Furnace Angle

Fig. 8 shows the stoichiometric amount of CdS sample (69.46% Cd and30.54% S) at a growth temperature of 800 ^{0}C, dwell time of 8 hours and furnace angle of 0^0. At the 45^0 furnace angle, a 61.79% Cd-38.21% S was obtained while 71.45% Cd-28.55% S atomic percentage was observed in the samples grown at 90^0. Based on the results, atomic and elemental composition of CdS nanomaterials were affected by furnace orientation.

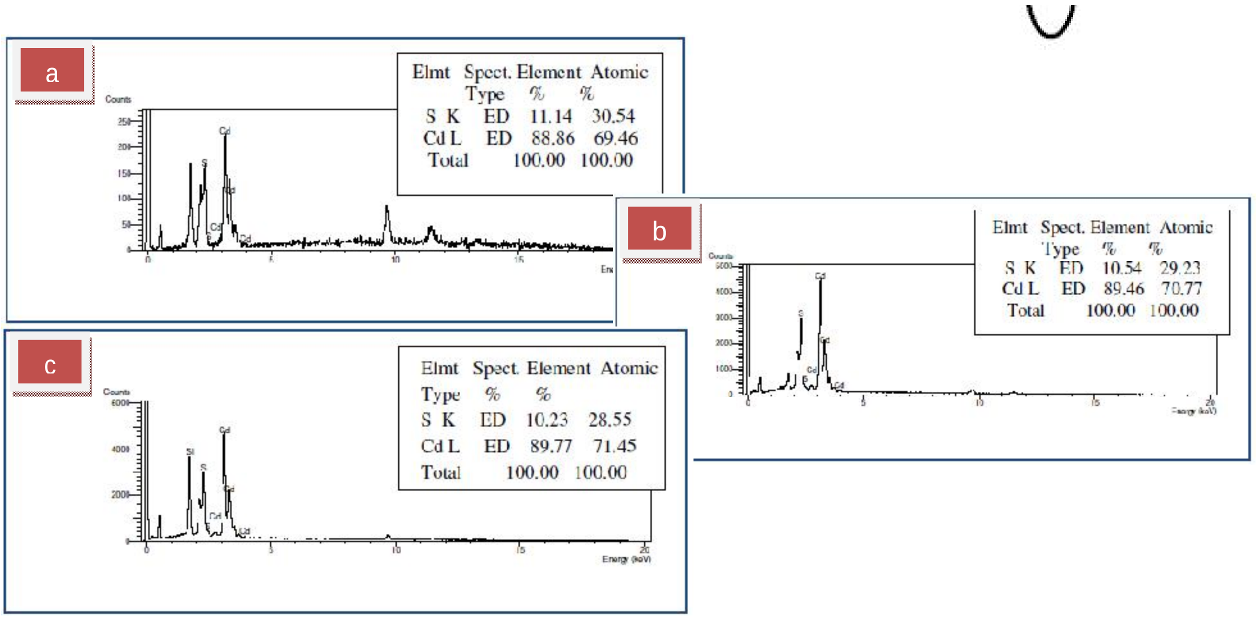

Fig.8Energy Spectrum of the sample synthesized at different furnace angle a) 0^0,b) 45^0, and c) 90^0

3.3PL Results of the CdS Nanomaterials Grown at Different Furnace Angle

Fig. 9 shows the PL spectra of the samples prepared with growth temperature of 800 ^{0}C, dwell timeof 8 hours and with varying furnace angle. The wavelength peaks recorded are 513.40 nm, 507.64 nm, and 507.57 nm for the furnace angle of 0^0, 45^0, and 90 ^{0}respectively. The average energy band gap of CdS nanostructures was calculated to be 2.44 eV. This is also very close to the theoretical value of CdS energy band gap.

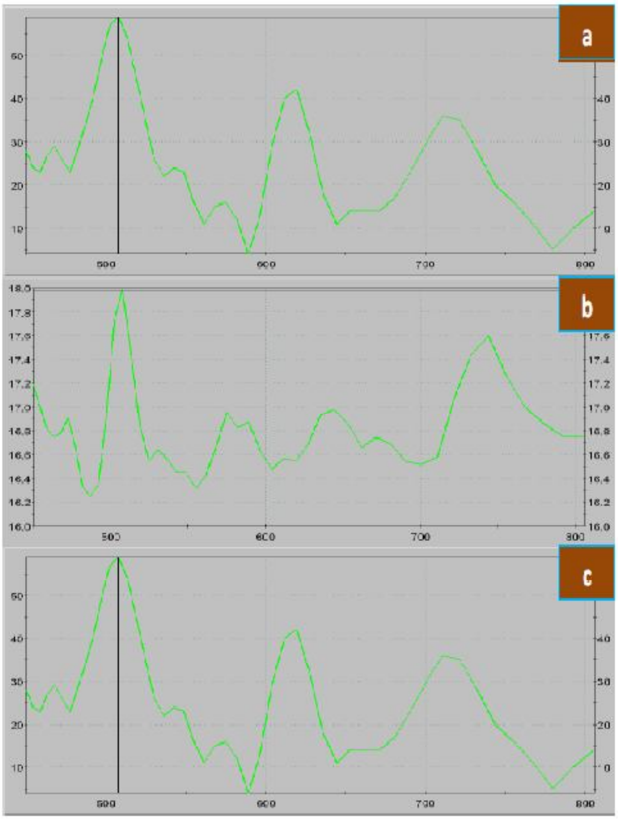

Fig. 9 Photoluminescence Spectra of the Nanomaterials different furnace angle, a) 0^0, b) 45^0, and c) 90 ^{0}

4 CONCLUSION

Cadmium Sulfide nanomaterials were successfully synthesized at varying furnace Angle using the Vapor Phase Crsytal Growth Technique. The study was able to characterize the surface morphologies and elemental and atomic composition of the nanomaterials using SEM and EDX, respectively. Photoluminescence spectra were also determined using spectral microscope.

From the results of the SEM, various nanostructures were formed when the growth conditions were varied.The deposition of CdS nanomaterials in lower temperature zone can be ascribed to the orientation of tube as affected by thermal gradient and gravitational field. Increasing the growth conditions increases the variety and amount of nanomaterials and produces lengthy but smaller in diameter nanomaterials specifically the nanowires. EDX analyses also showed that cadmium percentage increased when the diameter of nanowires is smaller. Optical properties of CdS nanomaterials were obtained by the photoluminescence spectra. Minimal deviations were observed in different furnace angles. The difference can be attributed to artifacts in the sample.

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