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APPENDICES

APPENDIX A

Analytical Equipments

A1) Possible absorption process of a photon by a molecule

Luminescence is the emission of light by a substance. It occurs when an electron returns to the electronic ground state from an excited state and loses its excess energy as a photon. The electronic states of most organic molecules can be divided into singlet states and triplet states; Singlet state: All electrons in the molecule are spin-paired Triplet state: One set of electron spins is unpaired

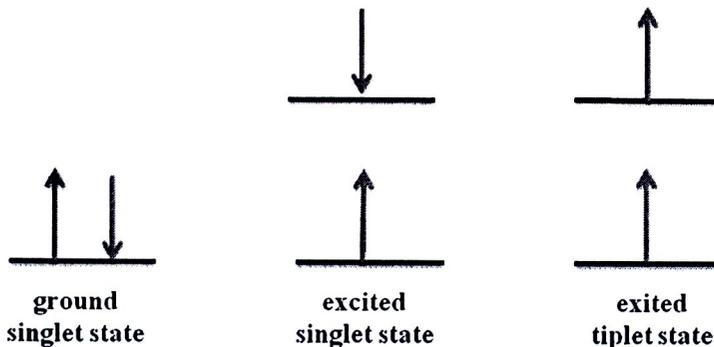


Figure.A1 The electronic states of most organic molecules

A2) Fluorescence

Absorption of UV radiation by a molecule excites it from a vibrational level in the electronic ground state to one of the many vibrational levels in the electronic excited state. This excited state is usually the first excited singlet state. A molecule in a high vibrational level of the excited state will quickly fall to the lowest vibrational

level of this state by losing energy to other molecules through collision. The molecule will also partition the excess energy to other possible modes of vibration and rotation. Fluorescence occurs when the molecule returns to the electronic ground state, from the excited singlet state, by emission of a photon. If a molecule which absorbs UV radiation does not fluoresce it means that it must have lost its energy some other way. These processes are called radiationless transfer of energy. Have a look at this diagram:

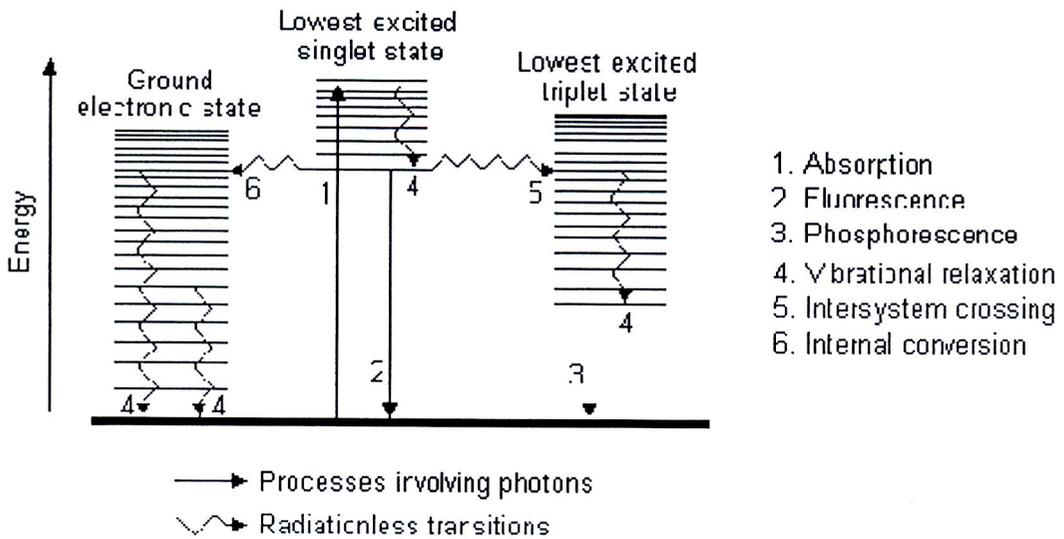


Figure.A2 Diagram show the possible physical process following absorption of a photon by a molecule

A3) Intra-molecular redistribution of energy

The molecule returns to the electronic ground state. The excess energy is converted to vibrational energy (internal conversion), and so the molecule is placed in an extremely high vibrational level of the electronic ground state. This excess vibrational energy is lost by collision with other molecules (vibrational relaxation). The conversion of electronic energy to vibrational energy is helped if the molecule is

"loose and floppy", because it can reorient itself in ways which aid the internal transfer of energy.

A4) The combination of intra- and inter-molecular energy redistribution

The spin of an excited electron can be reversed, leaving the molecule in an excited triplet state; this is called intersystem crossing. The triplet state is of a lower electronic energy than the excited singlet state. The probability of this happening is increased if the vibrational levels of these two states overlap. For example, the lowest singlet vibrational level can overlap one of the higher vibrational levels of the triplet state. A molecule in a high vibrational level of the excited triplet state can lose energy in collision with solvent molecules, leaving it at the lowest vibrational level of the triplet state. It can then undergo a second intersystem crossing to a high vibrational level of the electronic ground state. Finally, the molecule returns to the lowest vibrational level of the electronic ground state by vibrational relaxation.

A5) Phosphorescence

A molecule in the excited triplet state may not always use intersystem crossing to return to the ground state. It could lose energy by emission of a photon. A triplet/singlet transition is much less probable than a singlet/singlet transition. The lifetime of the excited triplet state can be up to 10 seconds, in comparison with 10^{-5} s to 10^{-8} s average lifetime of an excited singlet state. Emission from triplet/singlet transitions can continue after initial irradiation. Internal conversion and other radiationless transfers of energy compete so successfully with phosphorescence that it is usually seen only at low temperatures or in highly viscous media.

APPENDIX B

Camera constants used for the indexing of SAED pattern

Table TEM constants ($L\lambda$) at 200 kV

L (cm)	$D_{111}\text{Au}$ (mm)	$r_{111}\text{Au}$ (mm)	$D_{111}\text{Au}$ (Å)	$L\lambda$ (mm.Å)
40	8.70	4.35	2.355	10.2442
60	13.2	6.60	2.355	15.5430
80	17.2	8.60	2.355	20.2530
100	21.2	10.60	2.355	24.9630
120	25.2	12.60	2.355	29.6730
150	31.5	15.75	2.355	37.0912
200	41.5	20.75	2.355	48.8662
250	51.8	25.90	2.355	60.9945

APPENDIX C

Microwave induced plasma system

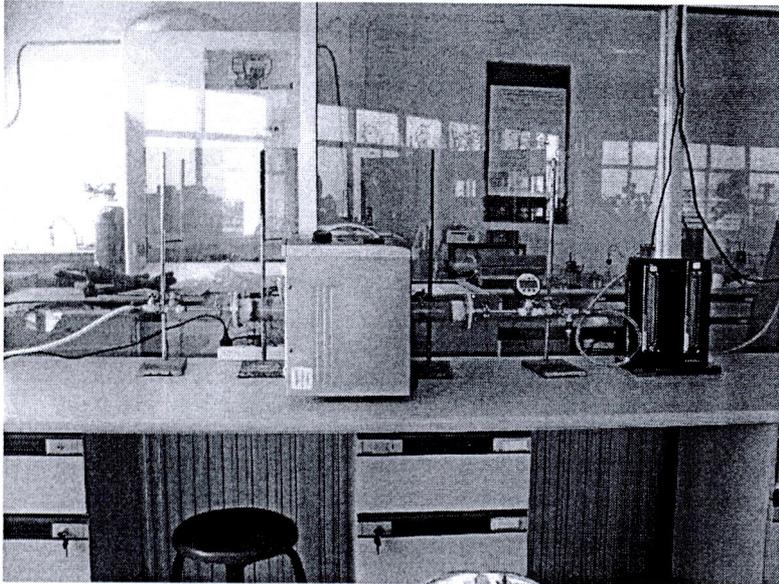


Figure C1. Microwave induced plasma system

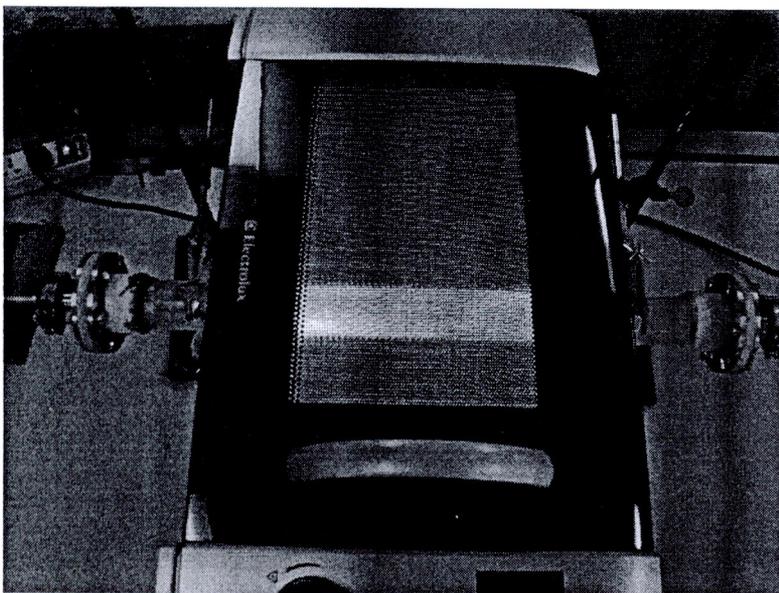


Figure C2. Microwave induced plasma system during operation

APPENDIX D

International publications

Photoemission and energy gap of CdS synthesized by solid state microwave-plasma

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Keywords: CdS; Solid state microwave-plasma; Photoemission; Energy gap

Abstract. Purified cadmium sulfide crystals were successfully synthesized from 1:2 molar ratio of Cd and S powders by a 900 W microwave plasma for 120 min and 140 min. In the present research, XRD and SAED patterns including SEM, TEM, and FIB images were used to indicate phase and morphology of the products, with their Raman peaks at 303 and 605 cm^{-1} . Photoemission was determined to be 537 nm, and direct energy gap (E_g) to be 2.48-2.51 eV.

Introduction

Recently, a number of II-VI semiconducting materials have been intensively studied. One of them is CdS which has been received attention, due to their wide applications in photoelectric converter for solar cells, optoelectronics, light-emitting diodes and biological labeling. It has a direct band gap of 2.5 eV for the bulk hexagonal wurtzite structure, and 3.53 eV for bulk cubic zinc blende structure [1-3]. Both physical and chemical processes have been used to synthesize the material, including solvothermal [3-6], spray pyrolysis [7], chemical precipitation method [8], and chemical bath deposition [9]. In the present research, CdS crystals were synthesized by solid state microwave plasma - the clean and fast process.

Experiment

To synthesize CdS, Cd and S powders with 1:1, 1:2, and 1:3 molar ratios were loaded into 11 mm I.D. x 100 mm long silica tubes. Each was placed in a horizontal quartz tube (Fig. 1), which was tightly closed and evacuated to 4.3 ± 1 kPa absolute pressure. Argon was gradually fed into this quartz tube. Evacuation was repeated three times. Finally, each solid mixture was heated by 900 W microwave plasma for 20, 40, 60, 80, 100, 120, and 140 min at 4.3 ± 1 kPa absolute pressure, and left to cool down to room temperature.

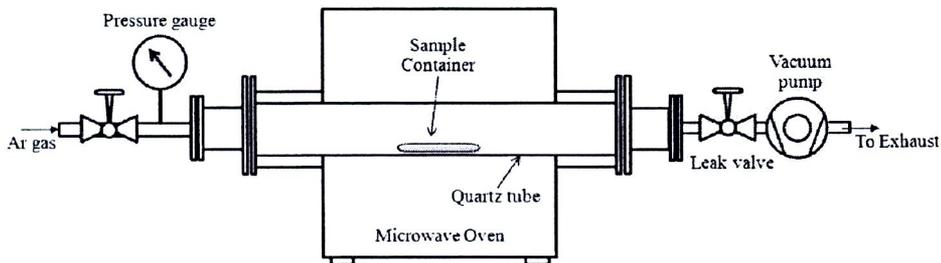


Fig. 1. Schematic diagram for the synthesis of CdS by the solid state microwave-plasma process.

The final products were characterized using X-ray powder diffractometer (XRD) with a 1.5406 Å wavelength of Cu-K α line, scanning electron microscope (SEM) operated at 15 kV, focused ion beam (FIB) technique operated at 30 kV, and selected area electron diffractometer (SAED) equipped with a transmission electron microscope (TEM) operated at 200 kV, Raman spectrometer using 30 mW He-Ne laser with 632.8 nm (red) wavelength, photoluminescence (PL) spectrometer using 450 W Xe-lamp with the 0.2 nm resolution and 325 nm excitation wavelength at room temperature, and UV-visible spectrometer operated at the 300-850 nm wavelength range with the 0.5 nm.sec⁻¹ scanning rate.

Results and discussion

The XRD peaks (Fig. 2) were indexed, and found to correspond with hexagonal CdS with the JCPDS no. 06-0314 ($a = b = 4.1360$ Å and $c = 6.7130$ Å) [10]. At 20 min and 1:1 molar ratio of Cd:S, the product contains some Cd impurity. It showed that some S evaporated, leaving Cd as residue. Upon increasing S to be at 1:3 molar ratio, the product was CdS with significant Cd and S as impurities. Thus 1:2 molar ratio of Cd:S was used. At this stage, only little impurities were still left in the product. By increasing the lengths of time in a series of steps, the impurities became lessened, and the CdS peaks were higher. Until at 120 min and 140 min, pure CdS crystals were synthesized. Lattice parameters of CdS were calculated using plane-spacing equation for hexagonal structure and Bragg's law for diffraction [11]. They are $a = b = 4.1304$ Å and $c = 6.6968$ Å, which are very close to the standard values.

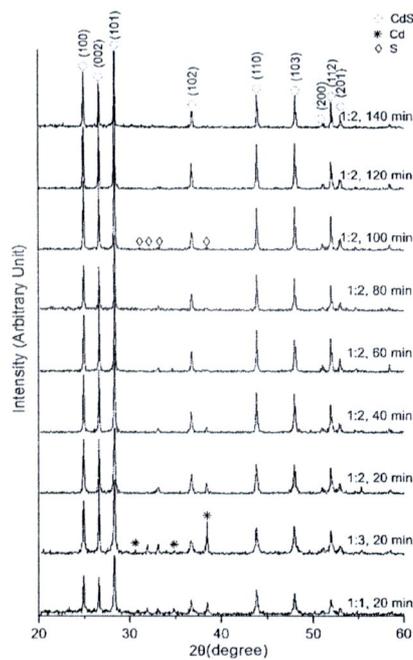


Fig. 2. XRD patterns of CdS synthesized using different Cd:S molar ratios and lengths of time.

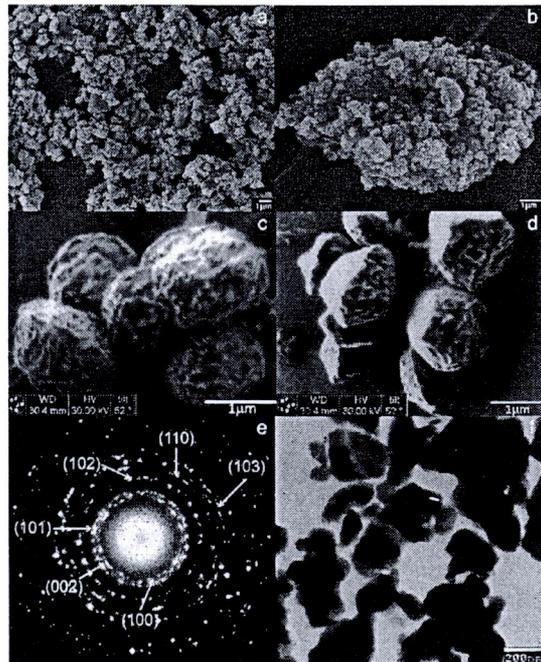


Fig. 3. (a, b) SEM images for 120 min and 140 min, (c, d) FIB images for 120 min and 140 min, and (e, f) SAED pattern and TEM image for 120 min, respectively.

Using SEM, FIB and TEM, the morphology (Fig. 3a-3d, 3f) of the products was composed of facet particles with different orientations. The SAED pattern (Fig. 3e) was interpreted as the (100), (002), (101), (102), (110) and (103) planes, corresponding to those of CdS standard [10]. This analysis was in accordance with those of the XRD results as well.

Raman spectra of CdS (Fig. 4) were expected to show the first (1LO) and second (2LO) harmonic modes at 303 and 605 cm^{-1} . It should be noted that intensity of the first harmonic is stronger than that of the second. These results are in accordance with the previous reports [8,12].

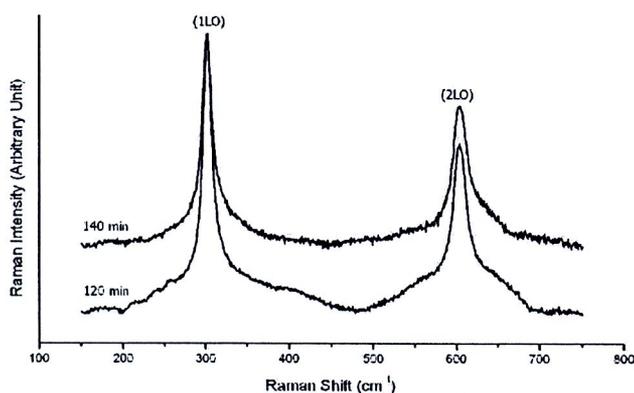


Fig. 4. Raman spectra of CdS synthesized for 120 min and 140 min.

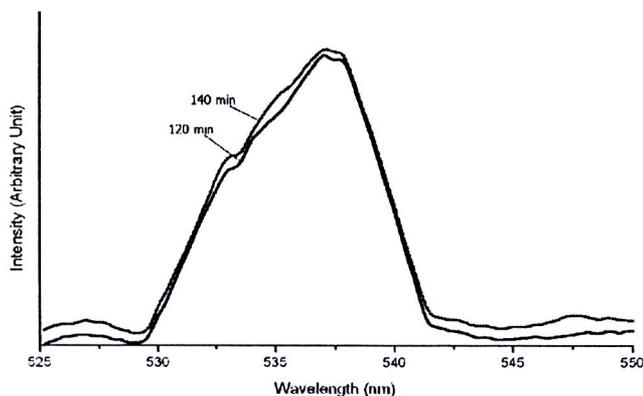


Fig. 5. Photoemissions of CdS crystals synthesized for 120 min and 140 min.

Photoemission of CdS crystals (Fig. 5) recorded using 325 nm excitation wavelength at room temperature. The emission was represented as broad peaks with the maximum emission at 537 nm in the green-yellow region, and is in accordance with the other reports [6,12]. This emission may have different values, controlled by structural disorder and defects. It was blue-shift upon doping with Zn^{2+} . This behavior was the opposite, when the material was doped with Cu^{2+} [12]. Sometimes trap levels may reside in the band gap during processing, which result to the red-shift behavior.

UV-vis absorbance of CdS was recorded. The direct energy gap (E_g) was determined by plotting $(\alpha h\nu)^2$ versus $h\nu$ (Fig. 6), where α , h , and ν are the absorbance, Planck constant, and photon frequency [7,9,13]. The E_g was determined by extrapolation the linear portion of the curves to $(\alpha h\nu)^2 = 0$, corresponding to 2.51 eV and 2.48 eV for 120 min and 140 min, respectively. It should be noted that the change of absorption was controlled by two photon energy ranges – the high and

low energies. When the photon energy is greater than E_g , absorption was linearly increased with the increasing of photon energy. But for the photon energy less than E_g , the absorption became different from the linearity, due to the dominant photonic absorption relating to defect levels between the valence and conduction bands of the products.

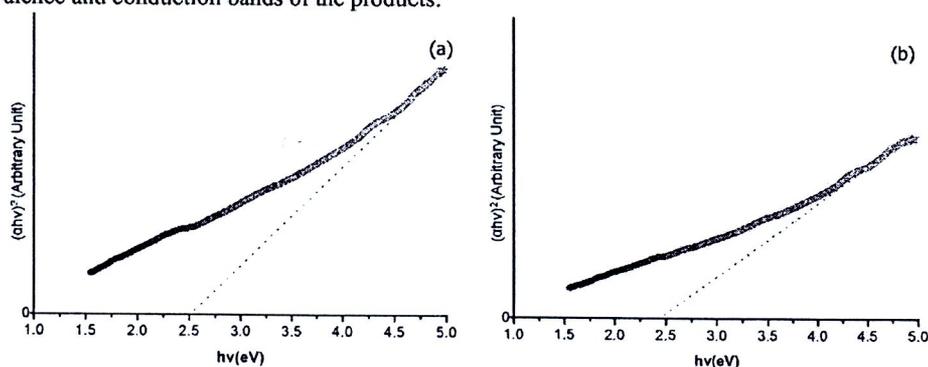


Fig. 6. The $(\alpha hv)^2$ versus $h\nu$ plots of CdS synthesized by the solid state microwave-plasma for (a) 120 min, and (b) 140 min.

Summary

Cadmium sulfide crystals were synthesized from 1:1, 1:2, and 1:3 molar ratios of Cd and S powders by a 900 W microwave plasma for 20-140 min. By varying the molar ratios of Cd:S and lengths of time, the products became pure crystals at 1:2 molar ratio of Cd:S for 120 min and 140 min. Photoemission was determined to be 537 nm, and direct energy gap to be 2.51 eV for 120 min, and 2.48 eV for 140 min.

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Eco-Materials Processing and Design XII

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Photoemission and Energy Gap of CdS Synthesized by Solid State Microwave-Plasma

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CHARACTERIZATION OF NANOSTRUCTURED CdTe SYNTHESIZED BY SOLID STATE MICROWAVE-PLASMA PROCESS

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Purified cubic CdTe nanocrystals were synthesized from 1:1 molar ratio of Cd:Te using 900 W microwave plasma. The phase was detected using X-ray diffraction (XRD), which are in accordance with those of the simulation, and selected area electron diffraction (SAED). Raman spectroscopic, scanning electron microscopic (SEM) and focused ion beam (FIB) techniques showed that the products were nanocrystals with fundamental transverse optical (TO) and longitudinal optical (LO) vibrations at 140 cm^{-1} and 169 cm^{-1} , respectively. Its direct energy gap (E_g) was determined to be 1.6 eV.

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Keywords: Nanostructured CdTe; X-ray diffraction; Raman spectroscopy; Focused ion beam technique; UV-visible absorption

1. Introduction

CdTe is one of the II–VI semiconducting materials, which has a direct band gap of 1.5 eV at room temperature [1]. It is widely used as photovoltaic detectors, photo-electrochemical cells [1], space charge limited diodes, infrared (IR) detectors [2], and high efficiency solar cells [3]. There are a number of processes used to synthesize this material – tetrapod nanocrystals by a facile one-pot synthetic method [4], nanocrystals in tri-octylphosphine-dodecylamine mixture [5], nanocrystals in aqueous solution [6], nanoparticles in aqueous solution by colloidal method [7], thin film by thermal evaporation and close spaced sublimation [1,2,8,9], thin film by spray deposition in the presence of electric field [10], thin film by sintering [3], thin film by pulsed laser deposition [11], thin film by chemical solution method [12], thin film by electrochemical method [13], and epilayer by vapor phase epitaxy [14].

Microwave radiation (2.450 MHz) is coherent and polarized [15]. When this radiation is supplied to chemicals, one or more of the components is capable of coupling with it. This can lead to higher heating rate than that achieved by conventional method. The entire volume of materials is internally heated up. Microwave radiation can solve the problems of temperature and concentration gradients. By focusing large amount of microwave radiation into the system, the vibrating electric field applied a force on charged particles which vibrated accordingly. Vibrations of the reactants have the influence on the reaction to proceed with efficiency. Subsequently, pure products were synthesized [15,16].

In the present research, nanostructured CdTe was synthesized by solid state microwave-plasma process. This process is rapid, done in closed system, and safe for lives. The synthesized

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product is also pure, although it was synthesized by solid state reaction process. The product was then characterized using different techniques.

2. Experimental

To synthesize CdTe nanocrystals, Cd and Te powders (purum. analytical grade, Fluka) were used without further purification. Solid mixture of 1:1 molar ratio Cd:Te was thoroughly mixed, loaded into silica tubes (11 mm I.D. x 100 mm long), and placed in a horizontal (H) quartz tube. The tube was tightly closed and evacuated to 3.9 ± 0.2 kPa absolute pressure for removal of air. Subsequently, argon was gradually fed into this H tube. The procedure was repeated three times. Finally, argon in the H tube was evacuated to a constant value of 3.9 ± 0.2 kPa absolute pressure. The solid mixture was heated in a manner of batch run by a 2,450 MHz microwave with 900 W output power to produce plasma. Each batch, the solids were heated for 10 min, left to cool down in vacuum to room temperature, and thoroughly mixed. The process was repeated under the same condition until at the completion of 10, 20 and 30 min. The products were then analyzed using X-ray powder diffractometer (XRD) with a 1.5418 Å wavelength of Cu-K α line, scanning electron microscope (SEM) operated at 15 kV, focused ion beam (FIB) technique operated at 30 kV, and selected area electron diffractometer (SAED) operated at 200 kV, Raman spectrometer using 30 mW He-Ne laser with 632.8 nm (red) wavelength, and UV-visible spectrometer operated at the 300-850 nm wavelength range with the 0.5 nm.sec⁻¹ scanning rate.

3. Results and discussion

XRD spectra of the products synthesized for different lengths of time are shown in Fig. 1. The XRD spectra were interpreted as CdTe cubic structure with F-43m space group, comparing to the JCPDS database no 15-0770 [17]. Their diffraction peaks corresponding to the (111), (220), (311), (400), and (331) planes are at $2\theta = 23.84, 39.36, 46.48, 56.84,$ and 62.48 deg, respectively. By increasing the length of time to 30 min, the product's crystalline degree was the highest. The reactants have more chance to form new product (CdTe), and the atoms have more chance to arrange themselves in their crystal lattices. Its lattice parameter (a) was calculated using Bragg's law for diffraction and the plane-spacing equation for cubic structure [18], and is summarized in Table 1. This parameter is 0.6496 ± 0.0020 nm, which is very close to that of the JCPDS database no 15-0770 ($a = 0.6481$ nm) - confirming the detection of cubic lattice structured CdTe.

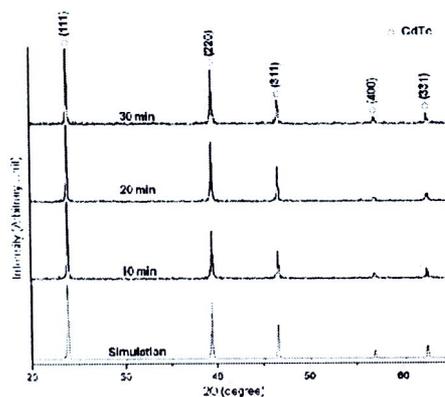


Fig. 1. XRD spectra of cubic structured CdTe synthesized by the solid state microwave-plasma process for 10 min, 20 min, and 30 min, including its simulation.

An XRD diffraction pattern of CdTe was also simulated using the CaRIne program [19] in combination with the calculated lattice parameter, and is shown in Fig. 1. The 2θ Bragg's angles and diffraction intensities of different peaks of the experiment, simulation and JCPDS database no 15-0770 [17] are shown in Table 1. Both 2θ Bragg's angles and diffraction intensities of different peaks obtained from the experiment, simulation and JCPDS database are in good accordance.

Table 1. The 2θ Bragg's angles (deg) and diffraction intensities (%) of nanostructured CdTe obtained from the 30 min experiment, simulation and JCPDS database no 15-0770, including its calculated lattice parameter (a, nm) for different crystallographic planes.

Plane	Experiment		Simulation		JCPDS database		a
	2θ	Intensity	2θ	Intensity	2θ	Intensity	
(111)	23.84	100	23.78	100	23.76	100	0.6522
(220)	39.36	71.65	39.32	75.3	39.31	60	0.6500
(311)	46.48	32.08	46.48	44.7	46.43	30	0.6478
(400)	56.84	10.16	56.83	11.4	56.82	6	0.6507
(331)	62.48	14.43	62.47	16.4	62.35	10	0.6473
							0.6496±0.0020

Fig. 2 shows SAED patterns of nanostructured CdTe synthesized by the 900 W microwave plasma for 10 and 30 min. The patterns appear as white concentric rings, characterized as polycrystals. These rings are diffuse and hollow showing that the product was composed of a number of nanosized crystals. At the present stage, the electron beam reflected and diffracted from polycrystals to form the concentric rings. Interplanar spaces (d) were calculated using their diffraction ring diameters [20], and compared with those of the JCPDS database no 15-0770 [17]. They both correspond to the (111), (220), (311), (400), and (331) crystallographic planes of the products and were specified as cubic CdTe.

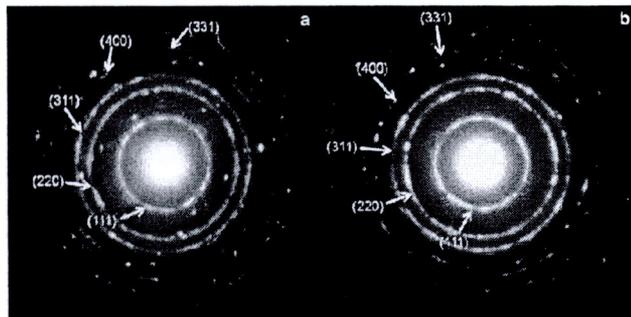


Fig. 2. SAED patterns of nanostructured CdTe, synthesized by 900 W microwave plasma for (a) 10 min, and (b) 30 min.

Morphology of the product (Fig. 3), synthesized by 900 W microwave plasma for 10 and 30 min, was investigated by SEM and FIB. Both of them show that the product was composed of a number of nanoparticles gathering together in the shape of spheres, aligning in different orientations. These spheres were larger with increasing the length of time. It should be noted that the detections of nanostructured CdTe by SEM and FIB are in good accordance with the interpretation of the SAED, explained above.

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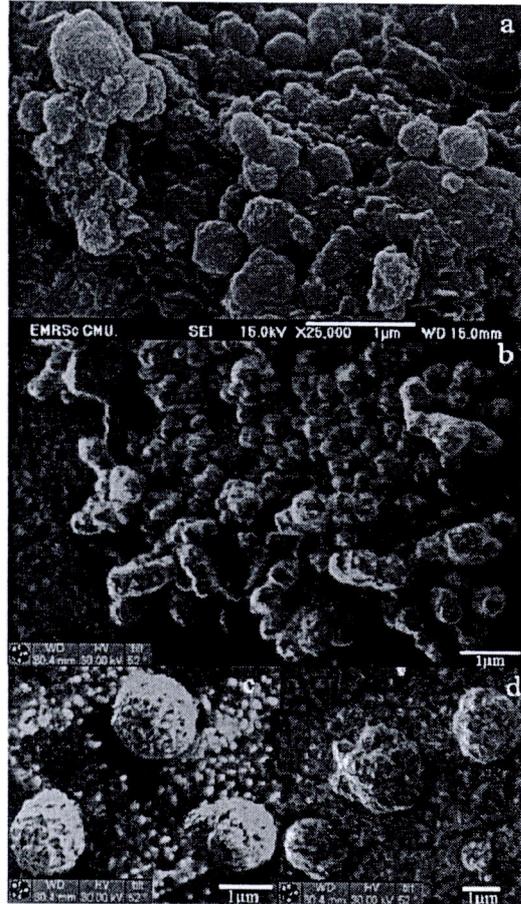


Fig. 3. SEM and FIB images of nanostructured CdTe synthesized by 900 W microwave plasma for (a, b) 10 min, and (c, d) 30 min.

The presence of cubic CdTe can be analyzed using a Raman spectrometer. For this case, test specimens are non-destructive and can be re-used for other purposes. The Raman shifts (Fig. 4) show the vibration modes of CdTe, corresponding to the fundamental transverse optical (1TO) and longitudinal optical (1LO) vibrations at 140 cm^{-1} and 169 cm^{-1} , respectively. They are in good accordance with those obtained by Dutta et al. who specified the 1TO, and 1LO Raman shifts of CdTe at 141 , and 166 cm^{-1} [21], and by Campos et al. at 141 , and 167 cm^{-1} [22], respectively. Other peaks at 120 cm^{-1} were caused by the residual Te [22], although the Te peaks were not detected by XRD measurement – its concentration could be too low to be detected by the XRD analysis. By increasing the length of time, this 120 cm^{-1} peak became lower – implying that the Te content became lessened. It should be noted that the 140 cm^{-1} peak was influenced not only by the vibration of cubic CdTe, but also the residual Te [22]. Comparing to the XRD results, the 140 cm^{-1} peak was dominantly influenced by the vibration of nanostructured CdTe.

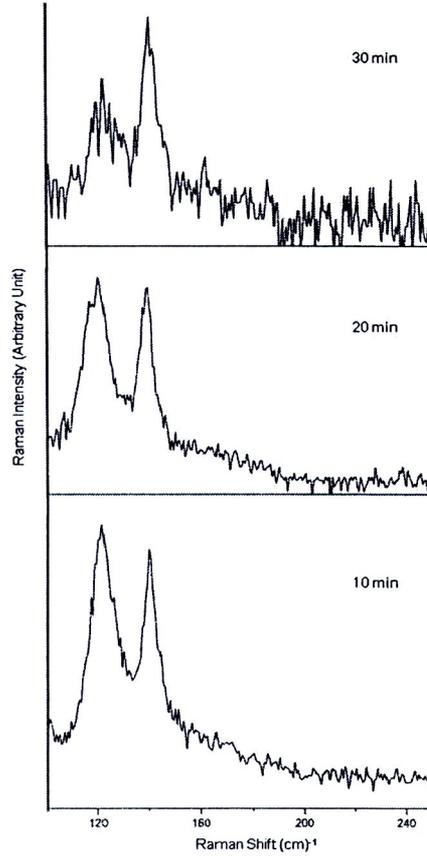


Fig. 4. Raman shifts of nanostructured CdTe, synthesized by 900 W microwave plasma for 10 min, 20 min, and 30 min.

The UV-visible spectrum of nanostructured CdTe, synthesized by 900 W microwave plasma for 30 min, was investigated using UV-visible spectrometer at room temperature. Its direct energy band gap (E_g) was calculated from the $(\alpha h\nu)^2$ vs $h\nu$ plot (Fig. 5), where α , h and ν are the absorbance, Planck constant and frequency [23], respectively. The absorbance increases with increasing photon energy ($h\nu$). The direct band gap of the semiconducting nanostructured CdTe was determined by extrapolating the broken line to the photon energy axis at $\alpha = 0$, which gives the energy band gap of CdTe to be 1.6 eV. This value is in accordance with 1.64 eV of the as-deposited CdTe film determined by Kokate et al. [23], and 1.6 eV of the cubic CdTe film by Pandey et al. [11]. These energy band gaps are also influenced by film thickness, particle-size, and degree of crystallinity [2].

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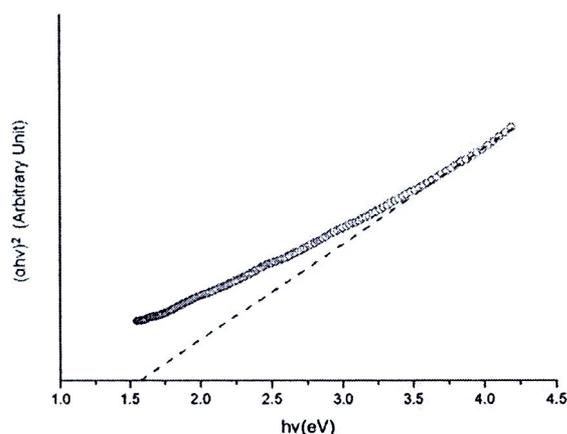


Fig. 5. The $(ahv)^2$ and $h\nu$ plot of nanostructured CdTe, synthesized by 900 W microwave plasma for 30 min.

4. Conclusions

Purified cubic CdTe nanocrystals were successfully synthesized by the solid state microwave plasma process. Its TLO and ILO modes were respectively detected at 140 cm^{-1} and 169 cm^{-1} , including the determined direct band gap (E_g) of 1.6 eV.

Acknowledgements

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- (2). The 12th International Symposium on ECO-materials Processing and Design, Chiang Mai, Thailand. “Characterization of purified nanostructured Cubic-ZnTe synthesis by solid-state microwave plasma process” on January 8-11, 2011.
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International Publications

- (1). Wonchai Promnopas, TitipunThongtem and SomchaiThongtem, “Characterization of nanostructured CdTe synthesized by solid state microwave-plasma process”, *Chalcogenide Letters, Materials Letters*, 8, (2011), 1-7.
- (2). Wonchai Promnopas, TitipunThongtem and SomchaiThongtem, “Photoemission and energy gap of CdS synthesized by solid state microwave-plasma”, *Materials Science Forum*, 695, (2011), 17-20,
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