

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 (1TO) and longitudinal optical (1LO) 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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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 \AA 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 $0.5 \text{ nm}\cdot\text{sec}^{-1}$ 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.

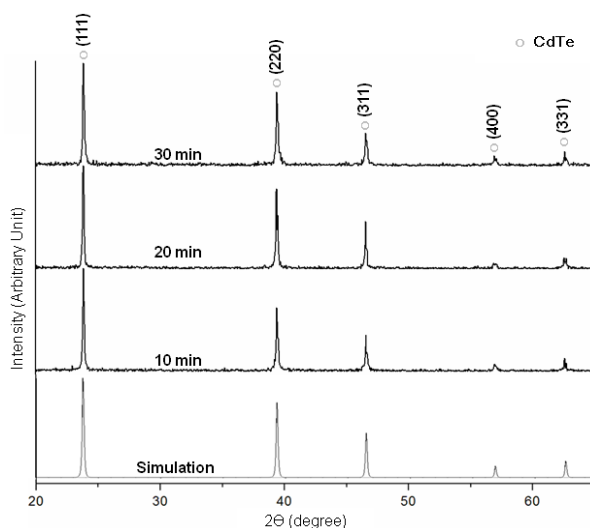


Fig. 1. XRD spectra of cubic structured CdTe synthesized by the solid state microwave-plasma process for 10 min, 20 min, and 30 min, including its simulation.

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.

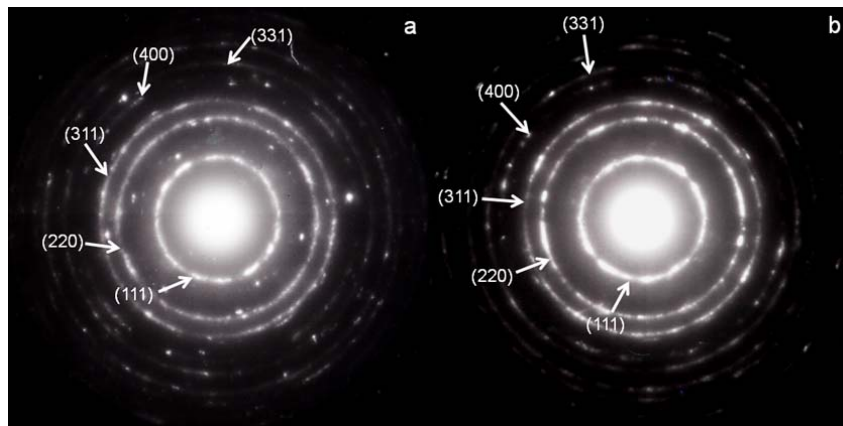


Fig. 2. SAED patterns of nanostructured CdTe, synthesized by 900 W microwave plasma for (a) 10 min, and (b) 30 min.

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.

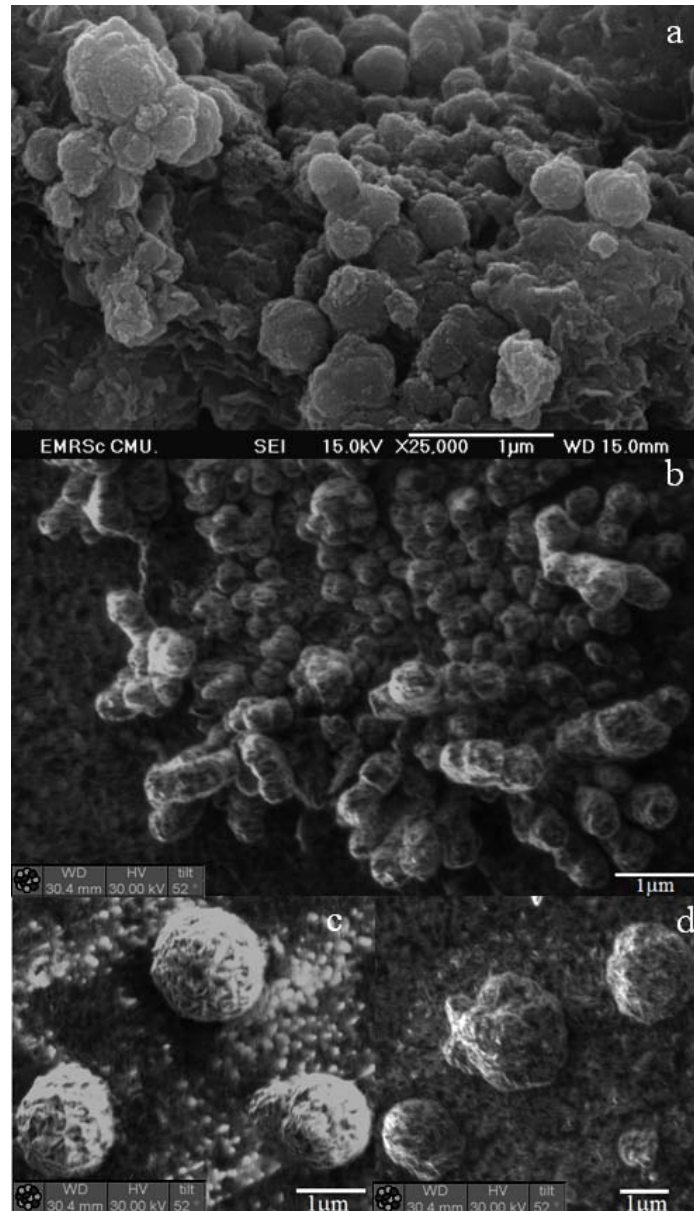


Fig. 3. SEM and FIB images of nanostructured CdTe synthesized by 900 W microwave plasma for (a, b) 10 min, and (c, d) 30 min.

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.

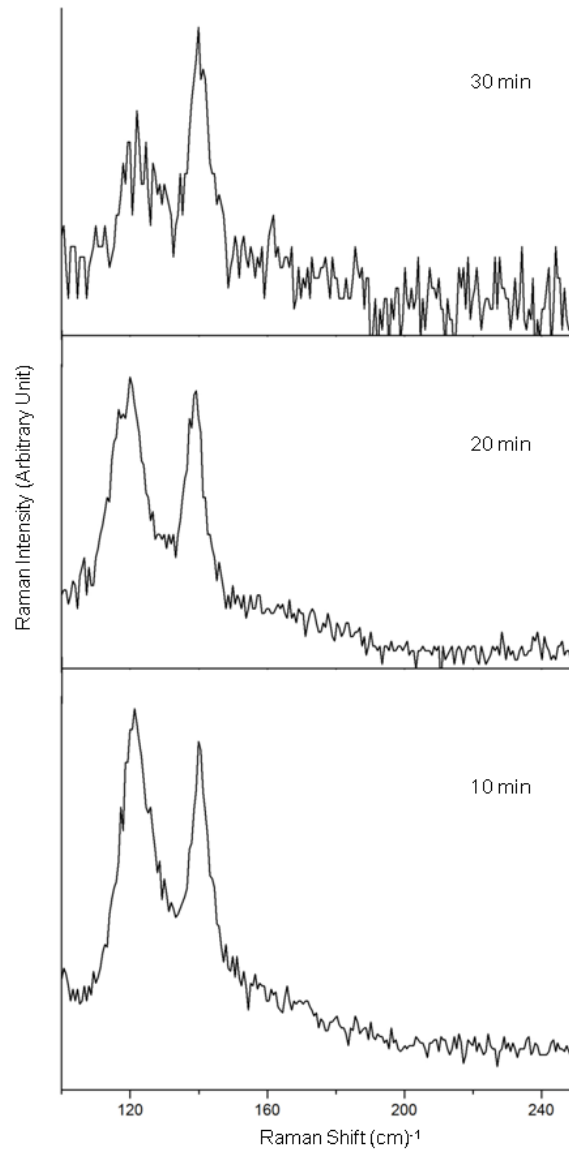


Fig. 4. Raman shifts of nanostructured CdTe, synthesized by 900 W microwave plasma for 10 min, 20 min, and 30 min.

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].

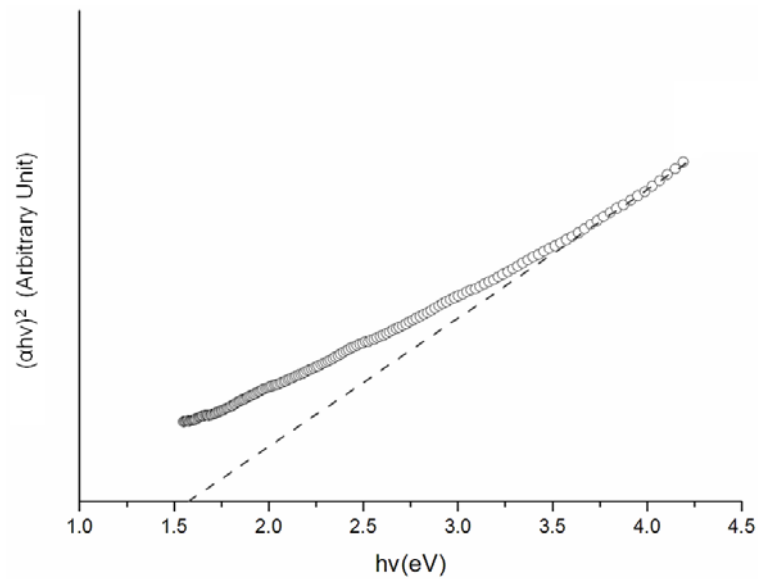


Fig. 5. The $(\alpha hv)^2$ and hv plot of nanostructured CdTe, synthesized by 900 W microwave plasma for 30 min.

4. Conclusions

Purified cubic CdTe nanocrystals were successfully synthesized by the solid state microwave plasma process. Its 1TO and 1LO 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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