

Effect of the nature of dispersion medium on the CdTe/TGA nanocrystal formation in colloidal solutions and polymeric membranes

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Abstract. In this work, physical and chemical properties of CdTe low-dimensional crystallites obtained by grinding or colloidal synthesis have been investigated. In particular, we have analyzed the photoluminescent characteristics of CdTe nanocrystals stabilized by thioglycolic acid, synthesized by using deionized water and water solutions of ethylene glycol and glycerin of different concentrations as dispersion medium. It has been shown that the stability of colloidal solutions of CdTe nanocrystals depends on the nature of dispersion medium and the stabilizer concentration.

Keywords: semiconductors, cadmium telluride, microcrystal, nanocrystal, dispersion medium, stabilizer, thioglycolic acid, photoluminescence spectrum.

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1. Introduction

Physical chemistry of nanostructures is a link between atomic physics aimed at the properties of separate atoms, and solid-state chemistry that studies substances containing the infinite number of atoms [1-4]. Basically, a nanocrystal (NC) is a very small fragment of a corresponding solid-state substance. But the properties of NCs are drastically different from those of the corresponding bulk material. One of the characteristics of bulk semiconductors is the presence of an energy gap, part of energy spectrum where optical transitions are forbidden, which separates the conduction band and the valence band. In bulk semiconductors, band gap has a fixed value for a specific material, and allowed states in the conduction and valence bands that are continuous, with quasi-free electron and holes movement through the crystal satisfactorily described by combination of “bulk-like” flat waves [5].

Behavior of charge carriers in semiconductor NCs is described by the rules of quantum mechanics, when the movement of electrons is limited in all three directions, and there are only discrete wavevectors. According to

this model, for spherical NCs with the radius R , the band gap value E_g and position of discrete energy levels of electron and holes in corresponding bands will be proportional to $1/R^2$, increasing with decreasing the NC size [5]. Therefore, while changing the size of NC, it is possible to tune positions of absorption edge and photoluminescence (PL) peaks in a broad spectral range from UV to IR, as it is confirmed by various fundamental studies and promising NC applications in light-emitting devices, biomedicine, sensors, and other fields [4].

Up to now, numerous chemical methods for obtaining low-dimensional semiconductor structures have been developed, in particular in aqueous solutions [6-10]. About ten classifications of these methods according to different physical and chemical principles can be found in the literature. A detailed overview of methods for synthesizing CdTe NC is given in [11, 12]. In this work, classification is based on the principle of changing the size of particles in the process of their preparation: “top-down” approach based on the dispersion of bulk materials and “bottom-up” approach of assembling NCs from molecular species.

Therefore, the purpose of this work was to investigate properties of CdTe particles prepared using mechanical grinding (“top-down” approach), followed by the use of the obtained dependences to optimize colloidal synthesis techniques for CdTe NCs (“bottom-up” approach).

The use of thioglycolic acid (TGA) as a stabilizer of the surface of nanosized CdTe particles in the course of their preparation has been already described in the literature [11, 13-18]. In this work, to understand the mechanisms of surface stabilization acting in CdTe micro- and nanocrystals, the influence of introducing microquantities of this stabilizer not only into the reaction medium, but also into the already formed suspensions of cadmium telluride was investigated.

2. Experimental

Highly-dispersed cadmium telluride obtained by grinding a single crystal grown using the Bridgman method was used. Particles of sizes within the range 40...150 nm were selected for investigation in this work. Thioglycolic acid (TGA) was used to stabilize the surface of CdTe particles obtained in this manner. Deionized water, methanol, ethanol, propanol, isopropanol, butanol, isobutanol, pentanol, isopentanol, heptanol, nonanol were used as the dispersion medium. The electrokinetic potential of the dispersions was determined by electrophoresis. The stability of CdTe suspensions was determined by absorptiometry.

The colloidal NC samples was obtained by deposition of cadmium and tellurium ions in an argon atmosphere in a three-necked heterogeneous semi-periodic reactor in the presence of TGA as stabilizer according to the procedure described previously in [19]. Deionized water, 10...50 % aqueous ethylene glycol solutions or 5...25% aqueous glycerin solutions were used as the dispersion medium.

Thin NC films were prepared by mixing 50 % aqueous solution of copolymer vinyl acetate-acrylate (density – 10...70 mPa·s, particle sizes close to 190...250 nm) with the colloidal solution of CdTe NCs at the ratio of 3:1, deposition on a glass substrate and drying in dark under the temperature 30 °C for 5 hours.

Investigation of optical properties inherent to these NC solutions was performed in silica and polystyrene cuvettes by using a corresponding dispersion medium or bare substrates and copolymer film without CdTe NCs as a reference. PL spectra were excited with 10-mW He-Cd laser line 325.0 nm and recorded at room temperature by using MDR-23 spectrometer equipped with a non-cooled photomultiplier FEU-100.

3. Results and discussion

3.1. Stability of dispersed systems based on CdTe microcrystals

Owing to the fact that physical properties of monohydric alcohols change naturally with the increase in the size of radical, studying the properties of cadmium telluride suspensions based on them allowed us to ascertain the relationship between the physical properties of dispersion medium and characteristics of cadmium telluride suspensions. The analysis of dependences obtained during the electrophoresis studies allowed us to determine the relationship between the nature of dispersion medium and electrokinetic properties of CdTe particles. We found that when TGA is used to stabilize crystals in water, methanol, ethanol, propanol, isopropanol, butanol, isobutanol, pentanol, isopentanol, heptanol and nonanol, there is a slight but systematic increase in the electrokinetic potential of CdTe NCs. In the given solvent sequence from water to nonanol (see Table), its value decreases between the lowest (0) and highest (1.0) TGA concentrations (CTGA) used in this work along the row: 3, 3.6, 3.3, 3.4, 2.7, 2.3, 1.9, 2.6, 2.5, 1, 1.4 mW.

Table. Dependence of CdTe electrokinetic potential on type of dispersion medium and TGA concentration.

Medium	ξ , mW at C_{TGA} , kg/m ³						
	0	0.1	0.2	0.4	0.6	0.8	1.0
water	23.2	24.1	24.5	25.1	25.5	26	26.2
methanol	20.5	22.1	22.5	23.2	23.6	23.8	24.1
ethanol	19.3	21.1	21.6	22.0	22.1	22.4	22.6
propanol	18.1	19.6	20.1	20.5	20.7	21.1	21.5
isopropanol	16.5	18.4	18.4	18.4	18.7	18.8	19.2
butanol	15.2	16.5	16.7	16.9	17.2	17.5	17.5
isobutanol	14.1	15.6	15.6	15.7	15.8	15.9	16.0
pentanol	13.2	14.6	14.8	15.2	15.5	15.6	15.8
isopentanol	12.1	14.0	14.1	14.5	14.6	14.6	14.6
heptanol	11.2	11.8	12.0	12.0	12.1	12.1	12.2
nonanol	10.5	11.4	11.6	11.8	11.8	11.8	11.9

This effect may be caused by both dispersing action of TGA and formation of the adsorption layer of TGA molecules on the surface of CdTe microcrystals, because TGA contains functional groups characterized by a high level of dissociation. Therefore, it can be concluded that action of TGA as a stabilizer is related with increasing electrostatic repulsion between the CdTe particles. However, the efficiency of TGA in this series of solvents decreases with increase of the size of hydrophobic radicals in the solvent molecules. This may be caused by a corresponding decrease in the dielectric constant of these solvents, which leads to a decrease in the degree of dissociation of the polar groups. It should be also noted that the electrokinetic potential of particles suspended in alcohols with branched radicals has always lower value than in the case of alcohols with a linear structure of radicals. This is a direct confirmation that the electrokinetic properties of the studied systems are influenced not only by the size of the hydrophobic radical of the dispersion medium molecule, but also by its structure, *i.e.*, the steric factor plays a significant role in the processes of interaction between the solvent molecules and the highly dispersed CdTe.

We have ascertained that the use of TGA in the entire range of concentrations (0.01...0.1 kg/m³) leads to increasing the sedimentation stability of dispersed cadmium telluride systems for all the dispersion media studied here. The coefficient of sedimentation resistance increases in proportion to TGA concentration and achieves its maximum value at CTGA = 0.1 kg/m³.

The analysis of obtained data leads to the conclusion that TGA has capability to stabilize CdTe particles regardless of their formation method. Furthermore, stabilization of CdTe particles with TGA is the most efficient in alcohols with low molecular weight and linear structure, such as methanol, ethanol, and propanol. These solvents are sufficiently wetting the crystallite surface, ensuring the contact of stabilizer molecules with the surface of CdTe particles. Due to the low boiling point and viscosity, these alcohols can be removed by washing with deionized water, if necessary for subsequent further investigations or applications. The obtained results can be used in the development and optimization of colloidal synthesis and other wet chemistry methods for preparation of CdTe NCs.

3.2. Colloidal synthesis

The method of colloidal synthesis allows obtaining CdTe NCs with hydrodynamic radius within the range 1...1000 nm and pronounced PL properties. However, the environment and the chemical reaction conditions have a significant effect on the kinetics of nanocrystal growth [12, 17, 18]. The analysis of data obtained in the previous section suggests that methanol, ethanol, and propanol are the best used as the dispersion medium during colloidal synthesis of CdTe nanocrystals. In the previous work [20], the possibility to obtain CdTe NCs in anhydrous methanol was shown, when Na₂Te is used as a source of Te²⁻ ions.

However, the synthesis of CdTe NCs in methylalcohols or other monohydric alcohols by using the method described in this work is not possible. Because of the insolubility of H₂Te in all the above mentioned monohydric alcohols, it passes through the reaction medium without any reaction with cadmium ions and gets neutralized with 0.1 M sodium hydroxide solution at the exhaust of reactor, forming a black precipitate of tellurium. Therefore, we study here the effect of the dispersion medium on the properties of the obtained colloidal NC solutions for the case of synthesizing CdTe NCs in aqueous solutions with addition of different amounts of two- or three-atom alcohols, particularly ethylene glycol and glycerin.

The growth of CdTe NCs during their colloidal synthesis in aqueous solutions can be described as a three-step process. At the first stage, nucleation of CdTe clusters from Cd²⁺ and Te²⁻ ions occurs in the supersaturated solution. In this case, the chemical reaction takes place only at the highest concentration of Cd²⁺ ions. The latter take part in the form of adsorption layers on the surface of the "impurity phase" (stabilizer), *i.e.*, on the already existing surfaces, and their spontaneous nucleation in the system practically does not occur. This assumption is confirmed by that in presence of the stabilizer the appearance of CdTe NCs in the reaction medium occurs only after some induction period, which is necessary to form the nuclei of emerging phase (the appearance of CdTe NCs can be revealed due to changing the color of solution). It can be assumed that duration of the induction period is significantly affected by the time required to make contact between the reactants. This hypothesis is supported by the fact that duration of the induction period increases with decreasing the concentrations of starting reagents and with increasing the viscosity of reaction medium (during the synthesis process in ethylene glycol and glycerin solutions of various concentrations). This is probably related with the fact that reacting Cd²⁺ and Te²⁻ ions are surrounded by solvate shells, which in its turn leads to the need to replace the molecules of the dispersion medium in the solvate layer by the ions of the reagents and, obviously, requires overcoming the certain energy barrier. In addition, apparently, the induction period ends after the appearance of nuclei of a pronounced physical surface (interface), which causes the possibility to adsorb Cd²⁺ and Te²⁻ ions on this surface.

At the second stage, the size of clusters increases with decreasing the degree of supersaturation of solution. These two processes are related to the ejection of water and stabilizer molecules and are determined by the free energy of supersaturated solution. During these steps, CdTe clusters of subcritical sizes with strongly developed surface are formed.

At the third stage, after reaching the critical size, there is an increase in the size of clusters caused by the diffusion mass transfer from small clusters to the large ones, which is stimulated by the decrease in the free surface energy of NC, with the finite dimensions and stability of NC in the disperse environment. The role of dispersion medium in the course of colloidal CdTe NC

synthesis should be related to the nature of the solvation processes of all the components involved in interaction (initial, intermediate and final reaction products). In the processes of desolvation, the electron density is redistributed, which in turn exerts a marked influence on the processes of intercontact interaction in the reaction medium. The solvation of the dissolved particle (CdI_2 , stabilizer) is accompanied by a change in the electronic structure of both this particle and the molecules of the dispersion medium (deionized water, glycerin, ethylene glycol), and causes a decrease in the enthalpy and entropy of the system. As the molecule goes into solution and undergoes subsequent solvation, its Gibbs energy decreases. The higher the heat (enthalpy) of the interaction of the solute with the solvent, the higher the solubility of the substance under other identical conditions. Some solvates, such as hydrates and alcoholates of inorganic substrates, are characterized by a sufficiently high strength (enthalpy of solvation may be similar to the binding energy of 100...400 kJ/mole). Not only the starting materials are solvated, but the intermediate products of interaction (for example, cadmium complexes with stabilizer) and the final reaction products (CdTe NC) as well. Thus, the nature of the dispersion medium plays an important role in formation of CdTe NCs during their synthesis. This was partially considered in [21]. The proof of this is that at the synthesis of NC in deionized water, introduction of an appropriate amount of ethylene glycol and glycerin into the solution causes no noticeable change in the optical properties of solutions. By contrast, when ethylene glycol and glycerin are present in the reaction medium during synthesis, it changes the optical properties of obtained CdTe dispersions and provides their long-term stability. Moreover, the dependence of changes in the properties of obtained CdTe nanocrystalline solutions on the concentration of glycerin and ethylene glycol in the reaction medium is clearly observed. NCs synthesized in solutions of glycerin and ethylene glycol as a dispersion medium have their PL significantly blue-shifted, as compared with the corresponding NCs synthesized in aqueous solutions.

On the other hand, an increase of glycerin concentration leads to broadening the PL band and its gradual shift towards lower energies, with appearance of a long-wave wing at the concentration 25%. It allows us to make a conclusion about the change in both mean size and size distribution of the formed CdTe NCs. The use of ethylene glycol as a dispersion medium does not lead to similar consequences and the PL band width remains practically the same for the entire concentration range, but shows a gradual shift towards longer wavelengths with increasing the concentration of ethylene glycol from 10 up to 50% [21]. It takes place likely due to that the increase in the molecular weight of the solvent and the viscosity of the medium, as compared to water, lead to an increase in the inertia of the growth process of the nanocrystal nuclei, resulting in particles with a more

narrow size dispersion. The hydroxyl groups of ethylene glycol and glycerin can also compete with TGA molecules in the process of modifying the growing CdTe NC surface, thereby creating a denser stabilizing layer around the particle and increasing the degree of stabilization due to electrical and steric factors. However, an excessive concentration of ethylene glycol and glycerin can reduce the rate of nucleus formation, and larger NCs will consequently form in the system, with a lower colloidal stability of such systems over time.

We found that in terms of long-term stability of optical properties of CdTe NCs studied, the optimal concentration of ethylene glycol and glycerin in the reaction medium is close to 10%. Thus, storage of the CdTe solution obtained under these conditions for 6 months did not result in significant changes of their optical properties, with only a slight spectral diffusion taking place. In solutions with a content of glycerin 5%, white precipitates of unknown nature were formed over time. CdTe NCs coagulate in all solutions with ethylene glycol and glycerin concentrations exceeding 10%. In the course of a few months, the colloidal solution becomes more transparent, with formation of precipitates, the color of which is more intense when the concentration of cadmium telluride in the solution is higher. This is a direct evidence of formation of larger CdTe NCs.

It is also noteworthy that, according to the literature, colloidal synthesis is one of the simplest and most affordable methods of producing CdTe NCs. However, their widespread use for fabrication of optoelectronic devices and some other important applications is not possible in the form of solution. Therefore, the part of our work was devoted to developing effective techniques for transferring CdTe NCs from colloidal solutions to polymer matrices. Therefore, we investigated the change in the optical properties of NCs synthesized in deionized water after their incorporation into the matrix of polyvinyl acetate-acrylate copolymer. Being based on the results of our previous work [19], where we ascertained that the optimal $[\text{Cd}^{2+}]:[\text{Te}^{2-}]$ ratio for obtaining fluorescent CdTe NCs is 8:1, NCs with the same nominal composition were also synthesized in this work for investigation of NC/polymer composites.

Fig. 1 shows the normalized PL spectra of CdTe NC synthesized at the nominal ratio of $[\text{Cd}^{2+}]:[\text{Te}^{2-}] = 8:1$ and stabilized only with TGA (Fig. 1a) and with TGA after addition of EG as a promoter of stabilization (Fig. 1b). The PL spectrum of CdTe/TGA NCs (Fig. 1a) is a superposition of two channels of radiative recombination caused by annihilation of free excitons (~ 2.4 eV) and recombination through surface levels created by defects (~ 2.1 eV) [15]. The PL spectrum of CdTe/TGA NCs obtained using ethylene glycol as a TGA promoter contains only a relatively narrow band of excitonic luminescence ~ 2.3 eV. Thus, the analysis of obtained PL spectra allows us to conclude that the use of EG as a TGA promoter allows significant reduction of defect concentration and improves NC size dispersion.

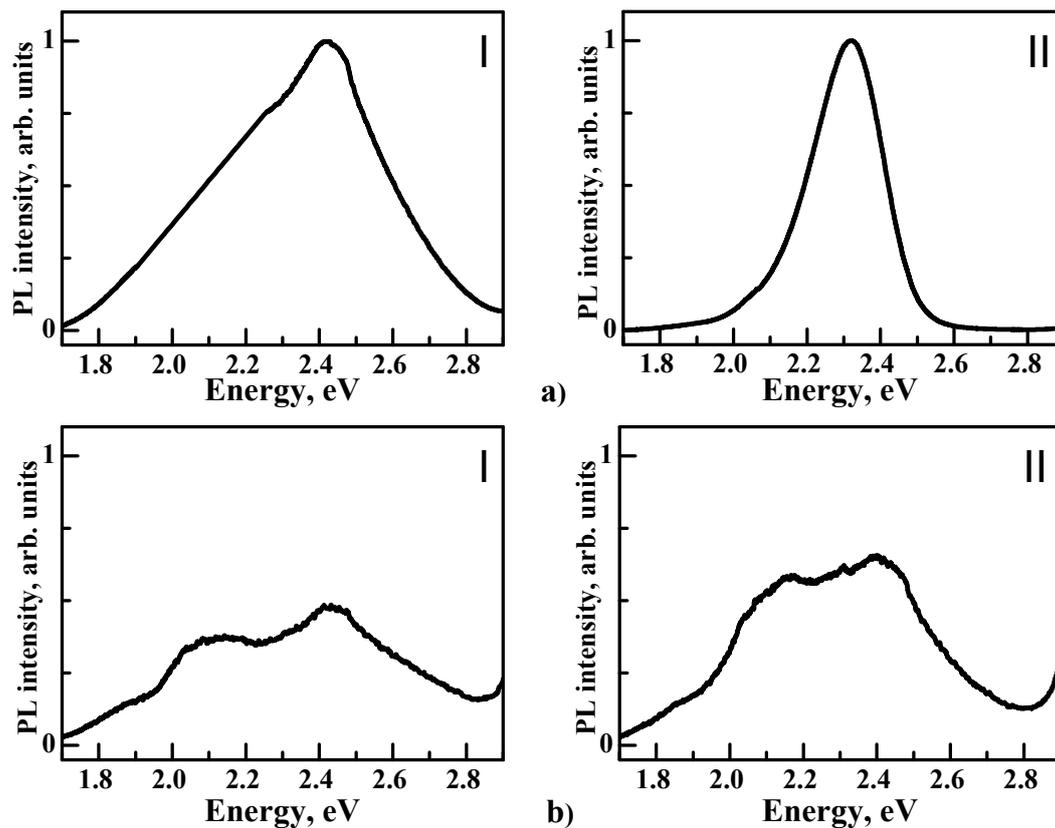


Fig. 1. PL spectra of colloidal solutions (a) and film structures (b) based on CdTe/TGA, dispersion medium – water (I) and 10% ethylene glycol solution (II).

Fig. 1 also shows the normalized PL spectra of thin films formed of CdTe/TGA NCs (Fig. 1b, I) and CdTe/TGA/copolymer vinyl chloride acrylate composite (Fig. 1b, II). Fig. 1 shows that in both cases the PL bands are simultaneously related with two channels of radiative recombination caused by annihilation of free excitons (~ 2.4 eV) and recombination through surface levels created by defects (~ 2.1 eV). The positions of the PL maxima in both cases coincide, from which one can judge the structural similarity of the NCs formed. However, with the use of EG as a TGA promoter, PL intensity increases markedly. Therefore, it can be concluded that vinyl acetate-acrylate copolymer is efficient matrix-forming material for transfer of CdTe NCs from colloidal solutions to thin films, with preserving their optical properties. Besides, this relatively cheap material, produced by tons throughout the world, makes the suggested method of NC film formation more attractable in comparison with those described in the literature [12]. We also have shown that it is advisable to use CdTe NCs, stabilized by TGA and using EG as a promoter of stabilization for transfer to vinyl acetate-acrylate copolymer films from colloidal solutions.

4. Conclusions

It has been found that using the thioglycolic acid within the broad concentration range ($0.01 \dots 0.1 \text{ kg/m}^3$) leads to an increase in the sedimentation stability of dispersed systems based on CdTe microcrystals in a wide range of alcohols used as dispersion medium. The coefficient of sedimentation stability increases in proportion to the increase in the concentration of TGA and achieves its maximum value at $C_{\text{TGA}} = 0.1 \text{ kg/m}^3$.

The nature of the dispersion medium significantly influences on the processes of formation of CdTe NCs stabilized by TGA during their colloidal synthesis. In particular, it has been found that the use of two- and three-atom alcohols as a dispersion medium can significantly increase the sedimentation stability of the obtained dispersions.

Increasing the viscosity of aqueous solutions of glycerin and ethylene glycol used as dispersion medium during colloidal synthesis of CdTe NCs allows increasing the stability of NC solutions and the intensity of NC PL. It has been ascertained that the most optimal concentration of ethylene glycol and glycerin in the reaction medium is close to 10%.

The use of vinyl acetate-acrylate copolymer as a matrix material allows transfer of CdTe NCs from colloidal solutions to obtain non-toxic nanoheterogeneous film structures characterized by satisfactory optical properties. Using ethylene glycol as a promoter of NC stabilization by TGA has been also offered for transfer to vinyl acetate-acrylate copolymer films from colloidal solutions.

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