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SEMICONDUCTOR NANOCRYSTALLINE LAYERS IN MULTILAYER SYSTEMS

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In this article, the main results on fabrication of semiconductor nanocrystalline sublayers (CdSe, Ge, Si) by various treatments of amorphous multilayer systems are summarised. The periodic structure and mechanical stability of these multilayers are considered as well as nanocrystallite size and orientation in the nanocrystalline sublayers. Some differences in the optical properties of the Ge, Si and CdSe nanocrystallites are discussed.

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1. Introduction

Along the past few years there has been a rapidly increasing interest in the preparation of semiconductor nanometer-sized crystallites. The development of various techniques for growing nanocrystallites (NCs) from a large variety of semiconductor materials (CdSe, CdS, CuBr, CuCl, Si, Ge, GaAs etc.) has opened the door for intensive studies on their linear, non-linear and electro-optical properties [1-5]. This enhanced interest is justified because NCs essentially constitute intermediate systems between solids and isolated molecules and, from a practical point of view, because of their potential applications in optoelectronic devices. Among the different methods for the preparation of nanocrystalline materials, that of thermal post-treatment of regular amorphous multilayers (AMLs) [6,7] raised recently a high interest. The main advantage of this approach is the possibility for a precise control of the sublayer thickness during the ML deposition procedure. This makes possible the fabrication of semiconductor NCs with desired size.

2. Fabrication of nanocrystalline / amorphous multilayers

It is known that the lack of long range order in the structure of amorphous materials induces a large number of defects such as bond length and bond angle fluctuations, dangling and floating bonds etc. These defects combined with the great flexibility of amorphous materials and the lack of crystalline order, eliminate the requirements for matching the lattice constants of the materials and makes possible the application of a great variety of materials in the fabrication of amorphous multilayers. Moreover, the regular AMLs have been prepared by a variety of rather simple and cheaper set-ups, as compared to those, needed for crystalline MLs. Among them are glow-discharge [8], magnetron sputtering [9] plasma and photochemically enhanced chemical vapor deposition [10,11] techniques, used for amorphous a-Si:H and a-Ge:H based AMLs, as well as physical vapor deposition [12,13] and laser ablation techniques [14], which have been applied in the fabrication of chalcogenide AMLs.

The structural modifications of AMLs, as one of the most important issue for their commercial applications, have been studied by many researchers. AMLs have been annealed for various times at temperatures around and above the crystallization temperature of the constituent material with low crystallization temperatures [6,15-22]. Laser beam crystallization under cw or pulse exposure has also been applied [23,24]. In order to study the structural changes of progressively annealed AMLs, small- and high-angle X-ray diffraction (SAXRD and HAXRD) as well as high-resolution transmission electron microscopy (HRTEM) and Raman scattering studies have been carried out. It has been established that the annealing at temperatures close but below the crystallization temperature (for example, ~ 60 min at 350 K for a-Se in a-Se/a-CdSe MLs [17-19]; 20 min at 720 K for a-Ge in a-Ge:H/a-GeN_x MLs [6] etc.), causes a decrease of disorder in AMLs. This result is illustrated by the data given in Table 1.

Table 1
Integrated Raman intensity of as-evaporated an annealed MLs of a-Se/a-CdSe in the spectral region 200-300 cm^{-1} [19].

d_{CdSe} (nm)	3.5	5.0	6.5	10
$I_{as\text{-}deposited}$ (a.u.)	4380	5200	8350	5400
$I_{annealed}$ (a.u.)	7870	7500	9650	8150

It is seen that the intensity of both Raman scattering bands at 210 and 250 cm^{-1} (the first band corresponds to the 1LO phonons in a-CdSe, the second one corresponds to vibrations in a-Se), increases after annealing a-Se/a-CdSe MLs at 360 K for 30 min in Ar atmosphere [19]. Given that the less disorder in the AMLs, the weaker the damping of vibrational modes, the observed integrated intensity increase could be due to an order in the 'bulk' part of the sublayers but, also, to an interface quality improvement. Indeed, some authors have observed a thermally induced reduction in the effective interface width, which has been related to negative sublayer interdiffusion [16-18]. At temperatures, higher than the crystallization temperature of the 'low-temperature' sublayers, microcrystallites formation takes place (for example, in a-Ge sublayers of a-Ge:H/a-GeN_x [6], a-Ge:H/a-SiN_x:H [22], a-Si:H/a-Ge:H [21] MLs and a-Se in a-Se/a-CdSe [19,20] MLs).

3. Periodic structure and mechanical stability

The crystallization of sublayers in AMLs by annealing or laser exposure can cause a number of structural and mechanical changes. Among them are deterioration of the interface smoothness, periodic structure and mechanical stability of AMLs. Some increase in the magnitude of the internal strains may also be expected. As these changes are quite important for the practical applications of the nanocrystalline/amorphous multilayer systems, the first three of them will be discussed in this paragraph. To the best of our knowledge, systematic studies on internal strains in annealed AMLs have not been performed up to now.

Small and high angle X-ray diffraction spectra of a-Se/a-CdSe MLs, annealed at various temperatures for 18 hours, are shown in Fig. 1 [20] (the glass transition temperature of a-Se is $T_g \approx 320$ K). The ML periodic structure vanishes after annealing at 333 K. After annealing at 363 K, a crystalline phase is evident by its main diffraction peak situated at $2\Theta = 29.6^o$ (CuK_α radiation). The SAXRD spectra a-Ge:H/a-GeN_x MLs [6] are displayed in Fig. 2 (the crystallization temperature of a-Ge is 723 K). It is seen from the figure that the repeat distance in the peak position (i.e. the ML period), as well as the integrated intensity and shape of the spectrum (i.e. the interface quality), do not change appreciably after the crystallization of a-Ge:H sublayers [6]. These results, supported by the HRTEM ones [6,9,22], illustrate that in the a-Ge based AMLs, the a-Ge sublayers crystallize without disrupting the multilayer structure, and that after crystallization the interfaces are atomically smooth and uniform. It has also been established [24,25], that in the a-Si:H based AMLs, both thermal and laser-beam crystallization of the a-Si:H sublayers do not destroy the regular periodic

structure of multilayers. Fig. 3 displays a SAXRD pattern of a microcrystalline $CdSe/a\text{-}SiO_x$ ML, annealed at 673 K for 60 min [26]. It is seen that this ML also exhibits a very good periodicity.

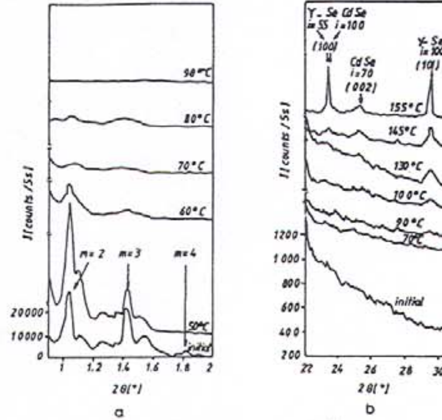

Fig. 1 Small-angle (a) and high-angle (b) X-ray diffraction patterns of a-Se/a-CdSe ML with a repeat distance of 22.4 nm and 14 periods at different annealing temperatures [20].

The above short review of the results on the periodicity of various nanocrystalline/amorphous multilayers shows that the periodic structure of amorphous multilayers is resistant to annealing or laser beam illumination when the constituent material with high crystallization temperature has a rigid structure. In such MLs, the growth of semiconductor microcrystals in the 'low-temperature' sublayers is restricted along the ML axes and, thus, crystallization does not destroy the ML periodic structure.

The crystallization of sublayers from the 'low-temperature' material in a-Ge and a-Si based AMLs, requires high annealing temperatures (> 770 K for a-Ge:H [6] and > 970 K for a-Si:H [16]). At these temperatures undesired interdiffusion and alloying of the constituent materials may take place at ML interfaces, which will destroy significantly the AML periodicity. It has been reported, that in a-Si:H/a-Ge:H MLs [21,27,28], the annealing at temperatures > 670 K leads to strong material intermixing, followed by the crystallization of the resulting silicon germanium alloy. However, after a similar annealing of $a\text{-}Ge:H/a\text{-}GeN_x$ [6], $a\text{-}Ge:H/a\text{-}SiN_x:H$ [22] and a-Ge/a-C [9] MLs, such alloying has not been observed.

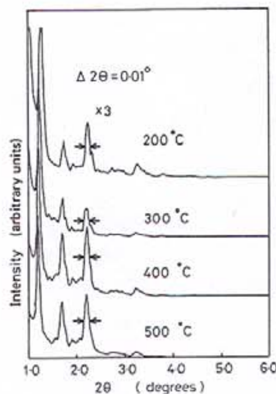

Fig. 2 Small-angle X-ray diffraction patterns of $a\text{-}Ge:H/a\text{-}GeN_x$ ML at different temperatures [6].

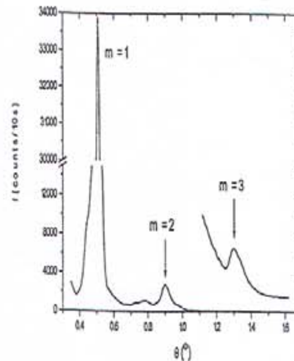

Fig. 3 Small-angle X ray diffraction patterns of $nc\text{-}CdSe/a\text{-}SiO_x$ multilayer structure (with d_{CdSe} = 4.0 nm and 20 periods, annealed at 673 K for 60 min) [26].

Also, material intermixing did not take place in a-Si:H/a-SiN_x [16], a-Si/SiO_2 [25] (annealed at temperatures >1070 K) and CdSe/SiO_x [26,29] (see Fig. 4) multilayer structures.

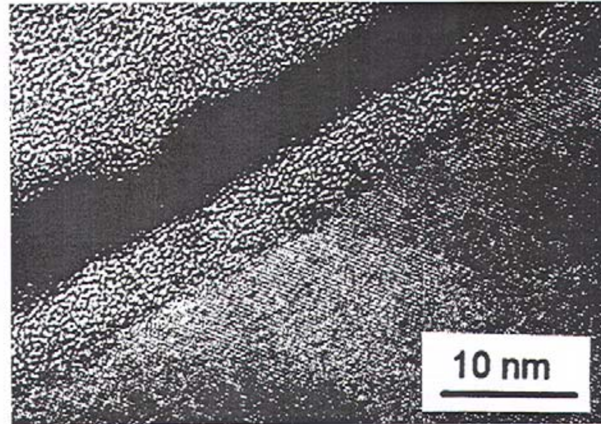

Fig. 4 Cross sectional TEM micrograph of a CdSe nanocrystalline layer sandwiched between two SiO_x ones. The structure was annealed for 60 min at 670 K [29].

Various results, obtained under annealing of different AMLs may be understood if one assumes that several competitive factors affect material intermixing and alloying at the ML interfaces. Diffusion coefficients of the atoms of each constituent material into the other one, and the possibility for alloying of both materials (i.e. the correlation between the magnitudes of the bond energies in the constituent materials and their alloy) could be among them. Based on this assumption, various approached may be used in order to avoid the sublayer intermixing. One of them is the selection of proper pairs of materials; the data reviewed in this article imply that semiconductor/semiconductor oxide (or nitride) pairs [6,9,16,22,25,26] are better than semiconductor/semiconductor ones [21,27,28]. This suggestion is supported by the results for CdSe nanocrystallites embedded in glass (no alloying) [1] and semiconductor (considerable alloying) [30] matrices. The second approach, which may be applied to avoid thermally induced sublayer intermixing, is the application of combined photo-thermal, 'pure' photoinduced or some other crystallization processes. It has been shown [22], that crystallization of a-Ge in a-Ge:H/a-SiN_x:H MLs is similar to an explosive process and is caused by the high stress of the a-Ge:H, which is triggered by laser heating. If a similar crystallization mechanism takes place in some semiconductor/ semiconductor AMLs and the necessary laser heating is not too high, it would be possible to prevent material intermixing during the crystallization procedure. It has also been observed [31,32] that a considerable diffusion of Se and Te takes place in chalcogenide materials. On the other hand, it has been shown [33,34], that 'pure' amorphous selenium and, particularly, selenium-tellurium alloys exhibit strong photo-thermally induced crystallization at room temperature. Moreover, they crystallize under laser beam exposure at fairly low temperatures (<100 K), at which the thermally induced atomic diffusion is extremely slow.

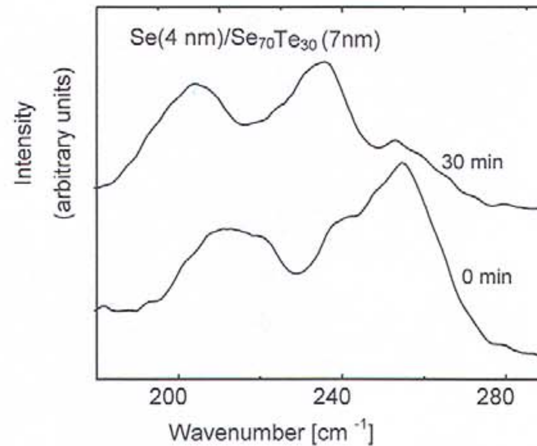

Fig. 5 Raman scattering spectra of a-Se/ a-$Se_{70}Te_{30}$ ML. The bands at about 235 cm^{-1} and 255 cm^{-1} correspond to Se-Se vibrations in crystalline and amorphous Se-Te alloys, respectively. It is seen that the exposure at 38 K, to the radiation of 647.1 nm Kr^+ laser line (55 W/cm^2) for 30 min leads to a strong increase of the 237 cm^{-1} band.

Such photocrystallization in a-$Se_{70}Te_{30}$ sublayers of a-Se/a-$Se_{70}Te_{30}$ MLs, was evidenced at 38 K, on the Raman scattering spectra. The results are presented in Fig. 5. The AML was exposed to the 647.1 nm Kr^+ laser line (55 W/cm^2) for 30 min. Thus, using photocrystallization at room and low temperatures, it is possible to make optical recording in amorphous chalcogenide MLs with minimum alloying at the ML interfaces.

The mechanical stability of annealed and laser-beam illuminated AMLs is very important for their practical applications, but the data concerning this problem are rather scarce. There was reported [22] that using slow thermal annealing, it is possible to crystallize the a-Ge:H sublayers of a-Ge:H/a-SiN_x:H MLs with no bubbling or pitting of the surface. However, when rapid changes in the volume of the a-Ge:H sublayers occur upon crystallization either by quick thermal annealing or by laser beam illumination, physical damage of the AML (cracking and bubbling) has been observed. On the other hand, we have not observed any mechanical damage after quick annealing of CdSe/SiO_x MLs. The strong effect of the rate of the annealing process on the mechanical stability of some annealed AMLs may result from a large difference in thermal expansion coefficients of the constituent materials and laser beam crystallization should be applied carefully on such multilayer systems.

4. Nanocrystallite size and orientation

Preparation of nearly monodispersive semiconductor nanocrystallites is very important from fundamental and practical point of view [1,4,5]. The uniform size of NCs allows for a detailed study of the effect of carrier confinement on the electronic structure and vibrational properties of semiconductors. On the other hand, high quality optical devices (quantum dot lasers, photo- and electroluminescence devices, etc.) may be fabricated using semiconductor NCs with narrow size distribution. In amorphous periodic structures the sublayer thickness may be preliminary picked out and precisely controlled during the ML preparation. Contamination effects that may complicate the optical properties of isolated NCs may be strongly reduced. For this reason, a number of scientists have carried out the crystallization of sublayers in AMLs in an attempt to prepare nearly monodispersive Ge [9,26,28,35,36], Si [24,25] and CdSe [29,37] NCs. One can see from Fig. 4 that in the CdSe nanocrystalline layer (d_{CdSe}= 4.5 nm) sandwiched between two SiO_x ones the NC size along

the ML axes is equal to the layer thickness. Similar result has been reported for nanocrystalline silicon sublayers of nc-Si:H/a-SiN:H MLs, having sublayer thickness < 10 nm [24] and nc-Si/SiO_2 MLs with d_{Si}=3.0-13.0 nm [25]. However, HRTEM studies carried out on annealed a-Ge/a-C MLs (d_{Ge}=29 nm), have shown [9] that, though most of the Ge crystallites extend through the whole thickness of the Ge sublayer, there are also crystallites smaller than the sublayer thickness. In a-Ge:H/a-GeN_x MLs (d_{Ge}=20-30 nm), annealed for 20 min at temperatures >770 K [6], it has also been found that the size of Ge NCs is smaller than the sublayer thickness. These results indicate that, generally, when the sublayer thickness is below 10 nm and the annealing is carried out for a long time and at high enough temperatures, the NC size is approximately equal to the thickness of the respective sublayers of AMLs.

The HRTEM studies on the nanocrystallite shape, performed on annealed a-Si/SiO_2 ML with d_{Si}=7.7 nm, have shown [25] that Si are almost spherical (with a small amount of residual amorphous material between the nanocrystallites). The Ge NCs in nc-Ge:H/a-GeN_x MLs [6] are also spherical and, in addition, they show strong orientation with Ge (111) planes perpendicular to the ML axes. However, in the CdSe sublayers of nc-CdSe/a-SiO_x MLs, the NCs are neither spherical nor oriented (Fig. 4). As it has been suggested in [6], the oriented nucleation can be explained on the basis of simple thermodynamical considerations. The most energetically favourable nucleation corresponds to the minimization of the interfacial energy after crystallization. The interfacial energy in the case of partially oriented crystallization of ML sublayers is lower than in the case of unoriented one. Generally speaking, the orientation of NCs in nanocrystalline/amorphous MLs and their shape are determined by the nature of both constituent materials. This means that in nanocrystalline/amorphous MLs having the same semiconductor sublayers but different second constituent materials, different NC orientation or lack of orientation may be observed.

5. Optical properties of nanocrystalline sublayers

Semiconductor nanocrystallites smaller than the exciton Bohr radius exhibit unique optical properties due to the confinement of the electron excitation. For example, the optical absorption and emission can be tuned across the visible spectrum by changing the size of nanocrystallites. Moreover, a strong increase in the intensity of radiative recombination in indirect-gap semiconductors (such as Ge and Si) my be achieved by carrier localization in nanocrystallites of these materials [35].

Visible light emission has been observed from nanocrystalline sublayers of Ge and Si in various MLs. Comparative studies have been performed on Ge nanocrystallites embedded in SiO_x matrix and nc-Ge/SiO_x multilayers [36,37]. It has been shown [36] that the photoluminescence spectrum from multilayered samples, had a narrower band than the spectrum of the Ge nanocrystallites in SiO_x matrix. This result has been attributed to the uniform size distribution in the MLs. The origin of the visible photoluminescence in both Ge and Si nanocrystalline films is still unclear. The position and the shape of the photoluminescence band, reported by different authors, are quite different [36-38]. In some multilayers [38,39], the position of the band depends on the NC size, and direct band-to-band recombination mechanism and quantum size effect on the peak position has been suggested. In other cases [22,36,37], the band position does not change with decreasing NC size, which indicates that photoluminescence cannot be attributed to effects of quantum confinement. Recombination via interface defects [38] situated either in the NCs or in the surrounding (oxide or nitride) material has been assumed [22].

Visible photoluminescence has been measured in nc-CdSe/a-SiO_x multilayer at room temperature. A gradual increase in the peak energy of the luminescence band has been observed with decreasing CdSe sublayer thickness (Fig. 6). This increase follows the 'blue' shift of the optical band gap, E_g, calculated by assuming conditions of one-dimensional carrier confinement (along the ML axes) in CdSe sublayers. For this reason, band-to-band recombination, affected by quantum confinement effect, has been suggested [40].

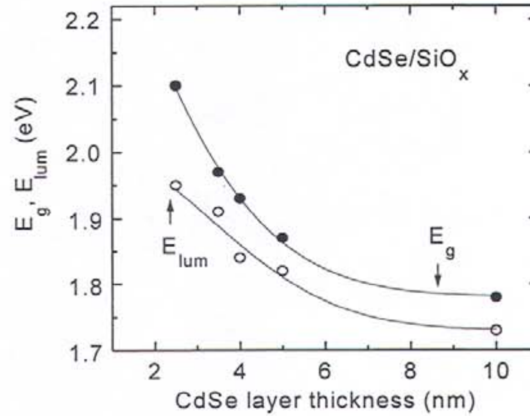

Fig. 6 Thickness dependence of the calculated band gap energy, E_g (full circles) and the measured peak position energy, E_{lum} (open circles) of the photoluminescence band of nc-CdSe/a-SiO$_x$ multilayers. The E_g values have been calculated assuming conditions of one-dimensional carrier confinement.

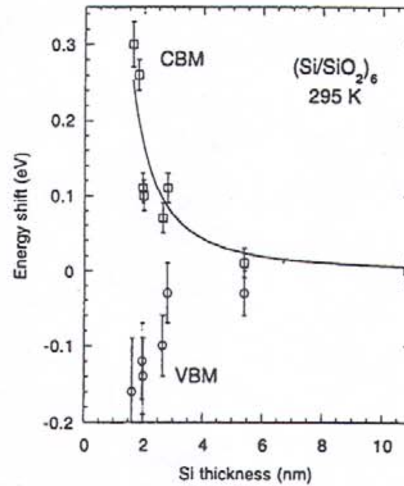

Fig. 7 The shift in conduction (CBM) and valence (VBM) band at room temperature for nc-Si/SiO$_2$ MLs as a function of Si sublayer thickness. The solid line is calculated by assuming conditions for three-dimensional carrier confinement [39].

The shift in the conduction band at room temperature for nc-Si/SiO$_2$ MLs (measured by soft X-ray Si $L_{2,3}$ edge absorption) with Si sublayer thickness is displayed in Fig. 7. The experimental shift is compared with the shift calculated by effective mass theory assuming infinite potential barriers [39]. This comparison indicates that, in nanocrystalline Si sublayer, the carriers undergo three-dimensional localization in each separate Si nanocrystallite, which fact is due to the presence of high barriers between NCs. On the contrary, the results displayed in Fig. 6 show that, though CdSe sublayers in CdSe/SiO$_x$ MLs are nanocrystalline (Fig. 4), the carriers are not confined in the separate CdSe NCs, but only along the ML axes. Perhaps, this is due to rather low potential barriers between CdSe NCs. The same conclusion has been derived from the resonant Raman scattering experiments, carried out on CdSe/SiO$_x$ MLs [40].

It is known that the lower is the dimensionality of the semiconductor, the greater is the quantum size increase in its optical band gap. Hence, it is important to achieve as great as possible increase in the band-gap of nanocrystalline CdSe by three-dimensional carrier localization, which would shift the peak of band-to-band radiative recombination to the green spectral region. For this purpose, the technique used for CdSe/SiO_x ML production [13], has been modified as follows. Based on the well known fact, that the surface roughness of layers deposited by vacuum evaporation increases with layer thickness, we prepared [3] samples that consist of a number alternating SiO_x and CdSe, in which d_{SiOx}=20 d_{CdSe}. In these samples island-type CdSe "sublayers" are formed. HRTEM studies [41] have shown that, in the annealed samples (for 60 min at 670 K), the semi-isolated CdSe islands are nanocrystalline with nearly spherical shape. Moreover, a large CdSe-SiO_x interface and relatively narrow size distribution of the nanocrystalline particles were observed. The nanocrystallite size is nearly twice that of the CdSe "sublayer" thickness. The changes in the optical band gap of CdSe nanocrystallites in these samples were investigated [3,42] using the constant photocurrent method. The main results are displayed in Fig. 8. A "blue" shift of the optical band gap is observed with decreasing average nanocrystallite size, which is greater than that in CdSe/SiO_x MLs and has been ascribed to three-dimensional carrier confinement in these nanocristallites.

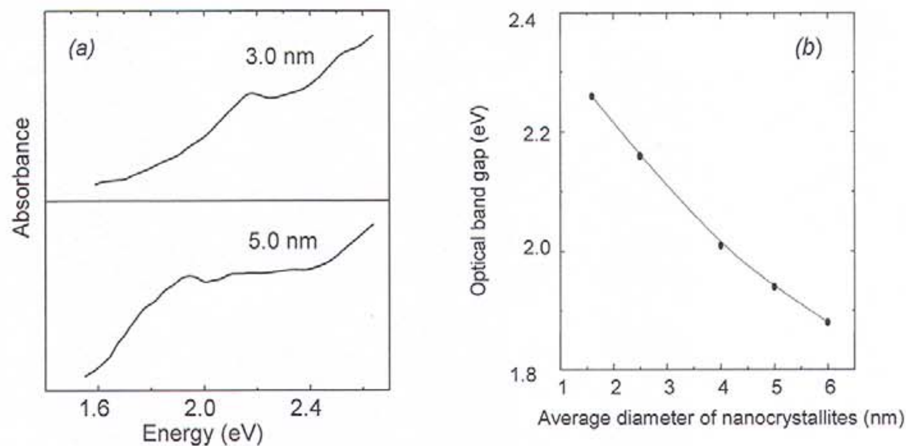

Fig. 8 a) Linear absorbance of two SiO_x-CdSe films having average nanocrystallite size of 5.0 nm and 3.0 nm and b) size dependence of the optical band gap of CdSe nanocrystallites embedded in a SiO_x matrix. It is clear that the size induced increase in the optical band gap of the nanocrystallites is steeper than in CdSe/SiO_x MLs.

6. Conclusion

There was shown that thermal or laser beam post-treatment of regular amorphous multilayers are essential steps in the fabrication of CdSe, Si and Ge nanocrystalline layers. The main advantage of this approach is the possibility to get semiconductor nanocrystallites with desired sizes and relatively narrow size distribution. Special attention should be paid to the intermixing of the constituent materials and mechanical damage in multilayers, which may take place during the crystallization process. There was also pointed out that, because of the different height of the potential barriers between nanocrystallites in the nanocrystalline sublayers, different size induced changes in the optical and vibrational properties of nanocrystalline/amorphous multilayers may be observed.

Acknowledgements

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