ФІЗИКА І ХІМІЯ ТВЕРДОГО ТІЛА
Т. 9, № 2 (2008) С. 368-374

PHYSICS AND CHEMISTRY OF SOLID STATE
V. 9, № 2 (2008) P. 368-374

PASC 81.15.FG, 82.45.MP

ISSN 1729-4428

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New Progress in Laser Technologies for Designing Semiconductor Nanostructures

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The overcoming some principal limitations of existent technologies in the framework of manufacturing nanostructures and the developing of novel technological methods of their manufacturing are questions of vital importance now. Because, the ratio "cost/efficiency" is the main market impediment to progress in the development of nanotechnological methods and the creation of future generations of the elemental base for nanoelectronics, we made an attempt to develop effective, low-cost and competitive technology, which can enable to produce nanostructures on the basis of II-VI semiconductor thin films. We present main results and problems related to growth of nanostructures on the basis of II-VI semiconductor thin films that were grown by Pulsed Laser Deposition method.

Key words: Pulsed Laser Deposition, Molecular Beam Epitaxy, Metalorganic Chemical Vapour Deposition

Стаття поступила до редакції 14.05.2007; прийнята до друку15.02.2008.

Introduction

Nowaday trend to smaller and smaller structures, that is, miniaturization, is standard practice in manufacturing microelectronic devices. With the increasing degree of miniaturization in microelectronics, devices designers unquestionably become confronted with the structures of matter on a scale below 100 nm. The transition from microtechnology (0.1-100 μm) to nanotechnology (0.1-100 nm) requires the ability to fabricate smaller structures as well as the exploration and application of new physical phenomena occurring on the refined scale which becomes comparable to the characteristics lengths associated with the elementary processes in Physics. That's why nanotechnology will play a dominant role in the fabrication of futuristic devices and components in the next few decades. The essential achievement is the reduction of the effective dimension of semiconductor structures from three-dimensional (bulk crystals) systems to two-dimensional (quantum wells, QW), one-dimensional (quantum wires, QWires), and even zero-dimensional (quantum dots, QD) structures [1, 2]. In this case, the quantum properties of current carriers in such semiconductor heterostructures play key role for determination of main parameters of nanodevices, which are manufactured from such structures. The above-named structures can be created on the base of low-dimensional semiconductor heterostructures designed from semiconductor thin films with submicron thickness. Therefore, motivation both for designing new types of low-dimensional semiconductor heterostructures and for overcoming some principal limitations of existent technologies in the framework of their manufacturing or for developing novel technological methods of their manufacturing are questions of vital importance now. The ratio "cost/efficiency" is the main market impediment to progress in the development of nanodevices.

I. Molecular Beam Epitaxy for growing nanodimensional semiconductor heterostructures

One of the most wide-spread technologies for growing semiconductor heterostructures and particularly quantum dots is the Molecular Beam Epitaxy (MBE) [3 - 6] (Fig.1). MBE takes place in high vacuum or ultra high vacuum (10^{-8} Pa). The most important aspect of MBE is the slow deposition rate (0.001 to 0.3 μm/minute), which allows the films to grow epitaxially. However, the slow deposition rates require proportionally better vacuum in order to achieve the same impurity levels as other deposition techniques. Under using of this technique, the growth on heated substrate of submicron or more complicated structures has place in the high vacuum installation by way the deposition of atoms (or molecules) of evaporated material that is contained in a special source. Searches of optimal deposition parameters (substrate temperature, matter flow from

evaporation source, evaporated material flow adjustment, properties of deposited materials, substrate with crystal perfection etc.) are very difficult and complicated. That is why it is hard to foresee the result in each separate case. At using of the MBE technique, arrays of nanoscaled islands (QD) that were formed in semiconductor heterostructures during the growth process do not constitute a homogeneous collection of entirely identical islands. A considerable size scattering of such islands usually is observed, and this fact leads to a significant difference in the energetic spectra of single islands. This phenomenon has crucial influence on the practical use of such structures. For example, the resultant irradiation spectra of structures with the QD array, that is a collection of single QD spectra lines, is a sufficiently wide band of several tens of meV. Therefore, production engineers have a task to minimize the size scattering of obtained QD. However, in spite of all efforts, the MBE technique makes possible to obtain QD arrays only with the size scattering of islands approximately 10% [7].

Considering the present financial position, not every academic institution in Ukraine has a possibility to produce or purchase the MBE installation for the growth of nanoscaled structures, to carry out further research of grown structures and to use obtained results in practice. The cost of the MBE installation is more than $1.5 million on the world market. Thus, not only in Ukraine, but also in other countries, the low-cost and effective technologies for producing low-dimensional structures on the basis of submicron thin films of semiconductor multicomponent compounds are entirely absent.

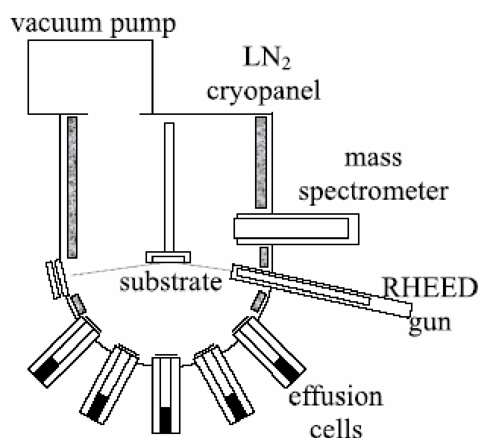

Fig 1. Scheme of a typical growth chamber in MBE system.

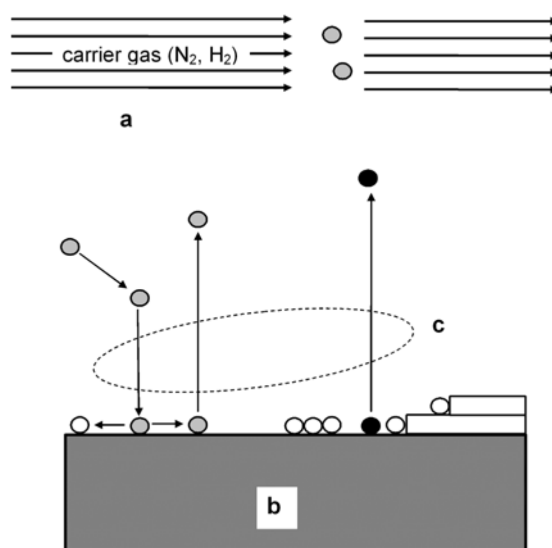

Fig.2. Principle of a MOCVD process

II. Metal Organic Chemical Vapor Deposition

Metal Organic Chemical Vapor Deposition (MOCVD) or Metal-Organic Vapor Phase Epitaxy (MOVPE) is a widely used method for preparing epitaxial structures by depositing atoms on a wafer substrate [8 - 12]. The principle of MOCVD is quite simple. But MOCVD system is technological complicated. Atoms are deposited by decomposing organic molecules (precursors) while they are passing over the hot substrate. The undesired remnants are removed or deposited on the walls of the reactor. III-V semiconductors of high purity and structural order are prepared via MOCVD. MOCVD occurs in following manner: laminar flux of carrier gas (H2 or N2) and of the precursor molecules (metal-organic compounds) (a) over the substrate (wafer) placed on a graphite susceptor inside a reaction vessel (reactor) (b) supply of thermal energy (usually between 400°C and 700°C) for decomposing the molecules, deposition of the material, evaporation of the molecular fragments into the gas phase (c) (Fig. 2). MOCVD is a chemical vapour deposition method of epitaxial growth of materials, especially compound semiconductors from the surface reaction of organic compounds or metalorganics and metal hydrides containing the required chemical elements. For example, indium phosphide could be grown in a reactor on a substrate by introducing Trimethylindium ($(CH_3)_3In$) and phosphine (PH_3). Alternative names for this process include organometallic vapour phase epitaxy (OMVPE), metalorganic vapour phase epitaxy (MOVPE) and organometallic chemical vapour deposition (OMCVD). The mostly disadvantages of MOCVD are as follows: most involve safety and contamination, hydrides and carbonyls are poisonous (especially arsine), metalorganics are pyrophoric (ignite in contact with air), high cost for compounds with sufficient purity. Formation of the epitaxial layer occurs by final pyrolisis of the constituent chemicals at the substrate surface. In contrast to MBE the growth of crystals is by chemical reaction and not physical deposition. This takes place not in a vacuum, but from the gas phase at moderate pressures (2 to 100 kPa). As such this technique is preferred for the formation of devices incorporating thermo-dynamically metastable alloys. It has become the dominant process for the manufacture of laser diodes, solar cells, and LEDs.

III. Pulsed Laser Deposition

In general, the method of Pulsed Laser Deposition (PLD) is simple (Fig.3) [13 - 17]. Only few parameters need to be controlled during the process. Targets used in PLD are small compared with other targets used in other sputtering techniques. It is quite easy to produce multi-layer film composed of two or more materials. Besides, by controlling the number of pulses, a fine control of film thickness can be achieved. Thus a fast response in exploiting new material system is a unique feature of PLD among other deposition methods. The most important feature of PLD is that the stoichiometry of the target can be retained in the deposited films. This is the result of an extremely high heating rate of the target surface (10^8 K/s) due to pulsed laser irradiation. It leads to the congruent evaporation of the target irrespective to the evaporating point of the constituent elements or compounds of the target. And because of the high heating rate of the ablated materials, laser deposition of crystalline film demands a much lower temperature than other mentioned film growth techniques. For this reason the semiconductor and the underlying integrated circuit can refrain from thermal degradation.

The PLD is a highly flexible thin-film growth technique which has been successfully applied to a wide range of materials including semiconductor compounds. The energetic nature of the depositing species enhances the growth process [14], potentially enabling the deposition of high quality films on low-temperature substrates. In particular, low-temperature growth of high quality semiconductor thin films is required for applications in the micro- and nanoelectronic devices. Additionally, sequential ablation of multiple target materials allows accurate control of the film stoichiometry, enabling the growth of various heterostructures and the deposition of films with well-defined boundaries profiles. Likewise, the fabrication of semiconductor heterostructures for future nanodimensional integrated circuits will be enabled by the growth of high quality semiconductor thin films with independently controlled well-defined boundaries between of two, or more, separate films.

IV. Growing II-VI semiconductor thin films by PLD

Thin films of CdTe and ZnTe were prepared by variety of techniques, such as, PLD, two source vacuum evaporation (TSVE), MBE, MOCVD [18, 19]. Also various types of substrate materials, such as, glass, SnO2 coated glass, ITO coated glass, GaAs, silica and MCT were used for the deposition of both CdTe and ZnTe films. However, the principal limitations of the traditional semiconductor technologies do not enable the growing of semiconductor heterostructures with: a detailed control of the stochiometrical composition; high crystalline quality at a significantly lowered substrate temperature; narrow and well-defined profiles between separate films. Therefore, the basic motivations for

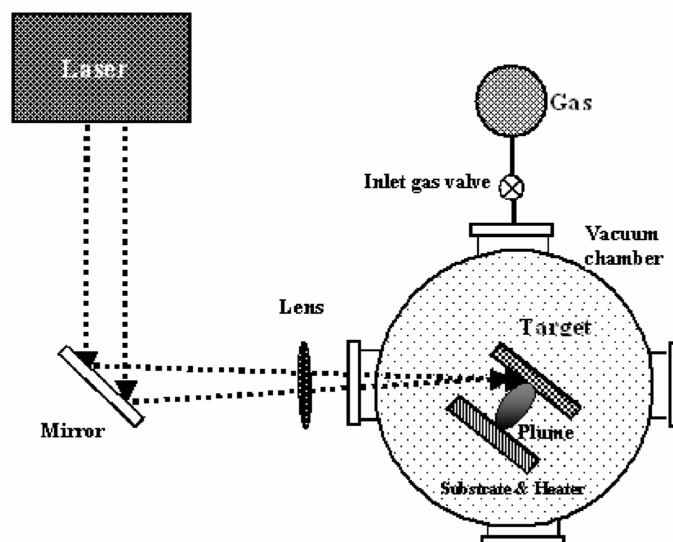

Fig.3. Scheme of PLD process

researcher efforts are directed to overcoming some principal limitations of existent technologies in the framework of manufacturing nanodimensional semiconductor heterostructures; developing novel technological methods for manufacturing semiconductor nanodimensional heterostructures; designing new types of semiconductor nanodimensional heterostructures; reduction of the main market impediment (the ratio “cost/efficiency”) in progress of nanoelectronic devices.

In this present work thin CdTe and ZnTe films were deposited onto well cleaned salt, glasse and sapphire substrates by PLD method. The CdTe and ZnTe films with various thicknesses were prepared from its own constitutional compounds in a vacuum of 10^{-5} Torr at different substrate temperatures. The pulses frequency were 50 Hz and its duration were 100 ns. Properties films are characterized by several techniques like X-ray Diffraction (XRD), Selected Area Electron Diffraction (SAED) and Atomic Force Microscopy (AFM). Also the effect of deposition conditions on the structural and optical properties of the CdTe and ZnTe films are discussed.

The most significant advantage of the PLD method over other film deposition methods is preserving of stoichiometry of the thin films [13]. The extension of PLD method to multicomponent semiconductor compounds, however, has fueled a thrust to characterize the film growth environment with fast, in situ diagnostics in order to efficiently correlate gas-phase conditions with films properties such as stoichiometry, morphology, uniformity and crystalline perfection. Practical experience gained with one material can hopefully be exploited with similar materials and the results and technology methods will be used during deposition for “in situ” process control. Much of the early studies of the PLD method were concentrated on the empirical optimization of deposition parameters for individual materials, without attempting to understand all processes that occur during a generation and a transportation of ablated material from the target to the surface substrate. Moreover, ablation species generated by high power

laser pulses have strongly dependence on following irradiations conditions: density of power laser radiation, laser pulse wavelength, pulse duration, spot size of laser beam on a surface target, pressure of background residual gaseous. The ablation species was generated from of the CdTe targets by using of the Q-switched Nd:YAG laser with a pulse duration of 15 ns and a repetition rate up to 56 Hz. The laser pulses were focused on the CdTe target with a resulting density power $q=10^8$ W cm^{-2}. The targets in the form of a disk were mounted into a vacuum chamber that was evacuated to a residual pressure of 5×10^{-6} Torr. Quadrupole mass spectrometer (QMS) was used in our experiments [20]. We used QMS with a quadrupole mass filter, which was used to select atoms and ions of different mass. The abundance distributions of neutral and charged particles in the ablation species were studied. When the laser pulses with near-threshold power density were used to achieve the evaporation temperature on the target surface, we observed the completely dissociation of target compound by QMS method. Our researches shown that, in contrast to MBE method, usage of the PLD method for evaporation of CdTe target allows to obtain the Cd and Te elements in the ablation species only in atomic and ionic forms. Obtained results testify that the ablation species produced by laser pulses with the threshold power density are completely different from those produced by conventional thermal heating. It takes place because a laser pulse produces the extremely higher velocity increasing of temperature on surface target in irradiated zone [21]. At given conditions ($\tau=15$ ns, $W\geq5\times10^8$ W/cm^2) process of target material evaporation have congruent nature. We found that decreasing of the laser pulses power density causes significant decreasing of mass spectral peaks intensities of ablated compound [22]. In the case of PLD, the thermal and kinetic energies of the ablated species are in the range up to few hundred eV and have significantly influence on the substrate temperature. Irradiation of first monolayers of matter target on substrate surface by ions and atoms of this energy produces in a thin surface layer (~30 A) a network of radiative defects (vacancies), which play a role of additional crystallization centers. In process of growing, such crystallization centers diffuses into the film substance and recombines over a time $\sim10^{-8}$ s with almost no worsening of the crystal properties. The number of plasma-produced vacancies exceeds the equilibrium value for a given temperature (275 ^{0}C) by many orders of magnitude and these results in an increase of the diffusion coefficient to the same extent [14]. As is well known, diffusion of adatoms in solids occurs mainly in the unoccupied nodes of the crystal lattice (vacancies), and the high temperature at which this diffusion takes place is needed to ensure that a maximum number of atoms escape from the lattice nodes. The PLD method allows to produce the additional crystallization centers without increasing temperature of heater, where are placed the substrates. These effects are equivalence to increasing the substrate temperature.

Thus PLD method allows growing ZnTe crystalline films at significantly reduced substrate temperatures. Another fundamental difference between the films that were grown by MBE and films that were grown by PLD method is the instantaneous deposition rate. At using the MBE method, the film growth is continuous, so that the deposition flux is random in time and space. As a result, morphology of the surface of grown films is an integration that is created by various kinetic competitions from a variety of growth modes. Analysis of XRD spectra for ZnTe films grown on glass, quartz and sapphire substrates testifies that the substrate temperature is main factor, which determines the crystalline structure of films [23]. Fig. 4 shows the XRD spectra of the ZnTe thin films grown on sapphire at various temperature

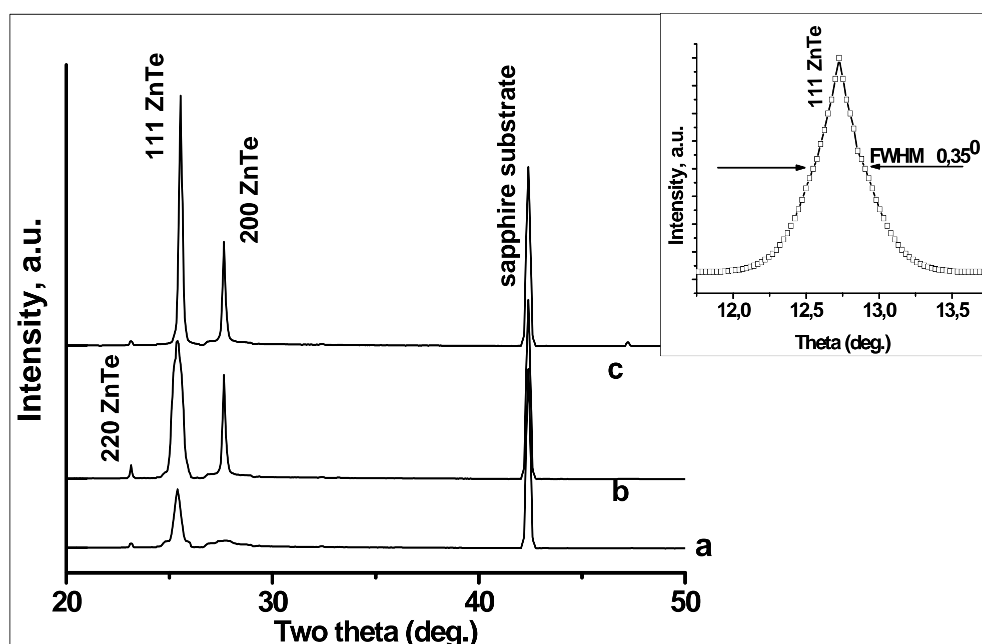

Fig.4 XRD spectra of ZnTe thin films grown on sapphire at various temperature substrates: 100 °C; (b) 220 °C; (c) 275 °C. The insert pattern shows the typical X-ray rocking curve for the (111) reflection of ZnTe film grown at a temperature of 275 °C.

substrates. The XRD spectra for ZnTe films grown on glass and quartz substrates were similar to XRD spectra for ZnTe films grown on sapphire substrate. The ZnTe films were amorphous, when the substrate temperature was ranged from the room temperature to 100 ^{0}C. The XRD spectra of the film grown at 100 ^{0}C are shown in Fig.4a. This diffractogramme is typical for ZnTe amorphous films. The increase of substrate temperature causes formation of crystalline structure in the grown films. The films grown at T_s=200 ^{0}C have the polycrystalline structure. Fig.4b represents the XRD spectra that correspond to the ZnTe polycrystalline film. These diffraction peaks at around 2θ = 25,4^0, 27,6^0 and 41,9^0 are corresponding to the (111), (200) and (220) planes of the ZnTe film and testifies about its polycrystalline structure. Increase of the substrate temperature up to 220 ^{0}C does not affect the polycrystalline nature of the films. The temperature range from T_s=220 ^{0}C to T_s=250 ^{0}C corresponds to increased ordering of the film orientation. Only the ZnTe films that were deposited at the elevated temperatures were textured and possessed the cubic structure. Highly oriented ZnTe films were grown at the substrate temperature of 275 ^{0}C. XRD spectra for the film grown at 275 ^{0}C are shown on Fig.4c. Except of one peak of sapphire substrate (0006), only two diffraction peaks of ZnTe (111) and (200) with maximum intensity appear in the XRD spectra. The narrow and high-intensity diffraction peaks indicate that grown films possesses a good crystalline orientation. The FWHM of the (111) peak is 0,35^0 indicates that ZnTe films grown at the substrate temperature of 275 ^{0}C have high quality crystalline structure and the (111) direction is the prevalence crystalline orientation of all crystallites. The sharpening of the peak with increasing of the substrate temperature indicates that the crystallites size increases, also. In this case, the substrate temperature is the defining factor for formation of the ZnTe films with a higher degree of crystalline perfection. The crystalline ZnTe thin films can be grow by MBE [24] and MOCVD [25] methods, when growth temperatures are ranging between 300 – 350 ^{0}C and 325 - 400°C, accordingly. Hence, it can be asserted that the ZnTe films with perfect crystalline structure can be prepared by PLD at much lower substrate temperature than those for MBE and MOVPE. This effect can be explained on the base of heightened energy of the PLD process, which is a far-from-equilibrium.

Fig. 5 a, b, c shows a evolution of typical cubical crystalline structure of CdTe films at increasing of substrate temperature. Fig. 5 d is the SAED pattern taken from a converted the CdTe films from cubic structure to hexagonal then substrate temperature exceeds a temperature threshold.

The surface morphology of CdTe thin films were observed by an AFM method. The image was (Fig. 6) obtained from CdTe film grown at 275 ^{0}C substrate temperature with a scanning area of 5μm X 5μm. It was established that grain size of the films becomes larger and tends towards uniformity as the substrate temperature increases up to 275 ^{0}C. Feature islands less than 100 nm have been produced, but significantly smaller dimensions should be possible. The AFM micrographs indicate that the CdTe thin films prepared by PLD are well crystallized, quite smooth and crack free. The root-mean-square (RMS) roughness for the films grown at 275 ^{0}C even reaches 10 nm, approaching atomic smoothness level.

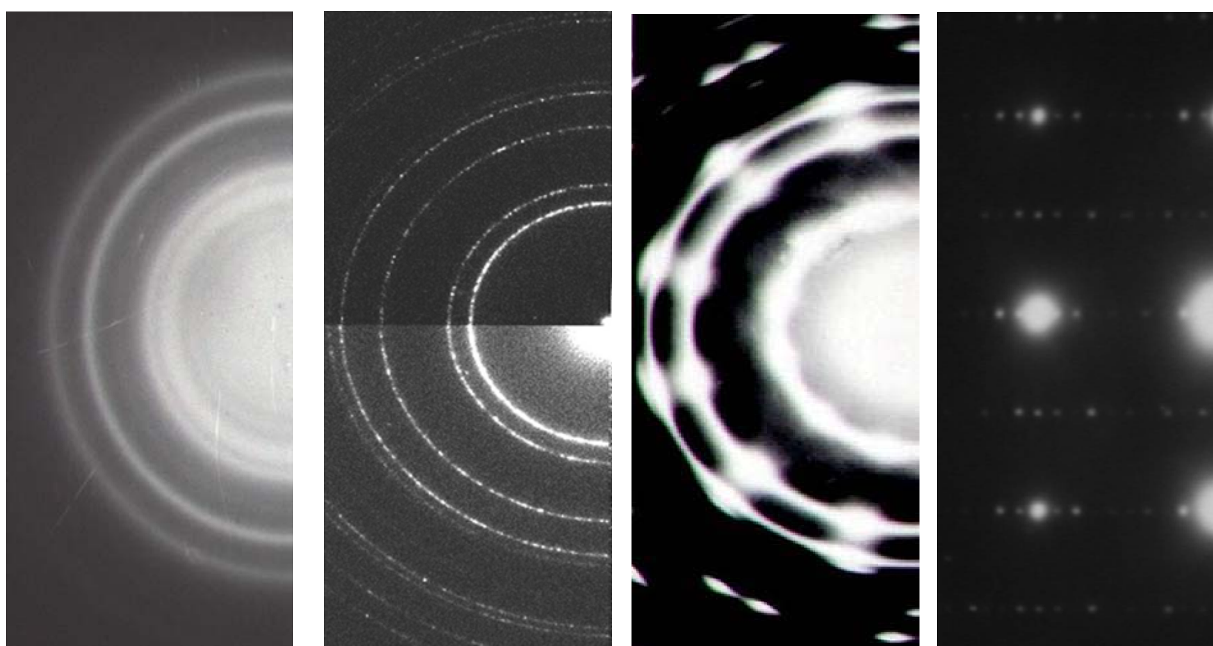

Fig.5. SAED investigation of CdTe thin films grown at different substrate temperatures (a - T_s =75 ^{0}C b- T_s = 200 ^{0}C, c- T_s = 250 ^{0}C d - T_s = 300 ^{0}C)

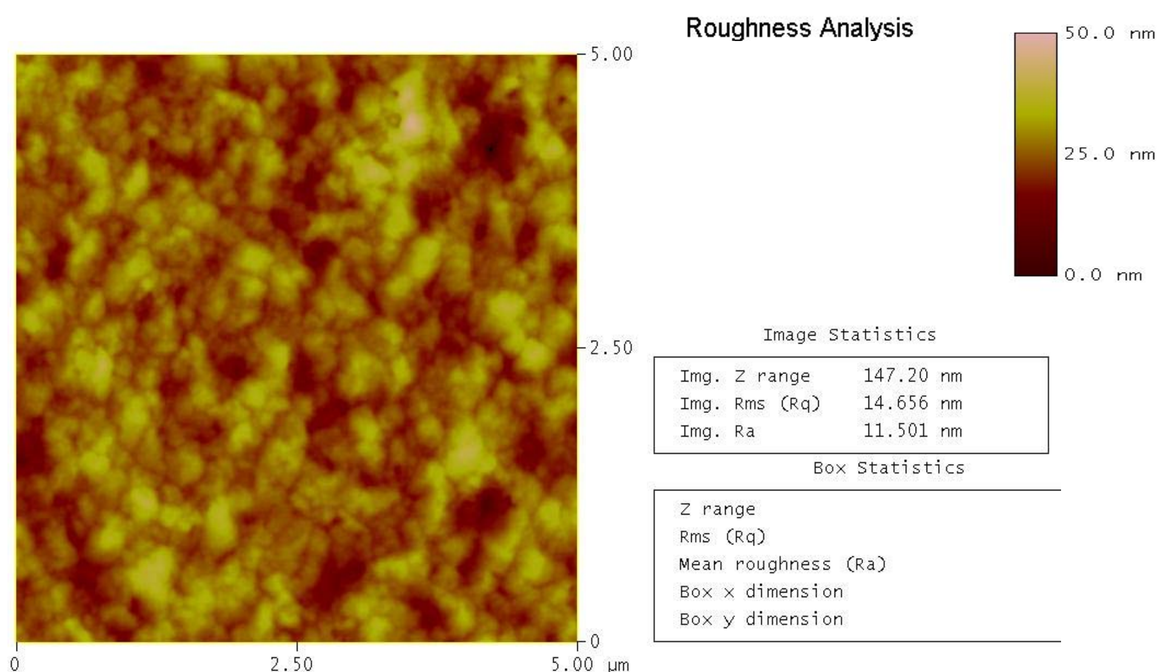

Fig.6. AFM image of CdTe films grown by PLD at substrate temperature of T_s = 275 ^{0}C

Conclusion

In comparison with other growth methods, the PLD technology has follows advantages: the PLD method provide the preservation the stoichiometry of multicompound targets in films (for example, the MBE method uses resistive heating for material evaporation from separated sources); the PLD process can be held both in the high vacuum and the background atmosphere of various gases (the MBE methods demand high vacuum, the MOCVD requires a mixture of various poisonous gases); the PLD method allows to change the wavelength of laser radiation and deposition conditions unreservedly (choosing types of substrates, substrate temperature, various background ambiences) and, thus, to control film growth modes at the significantly reduced temperature of a substrate; the high deposition speed allows to raise the “effective vacuum” during manufacturing processes; film thickness can be simply controlled by the quantity of laser impulses; the PLD technology installation is cheaper in hundreds of times than the MBE installation; the PLD technology is environmentally appropriate (after completing the MOCVD process, one must utilize toxic gases).

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Новий прогрес лазерних технологій для проектування напівпровідникових наноструктур

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При виготовлені наноструктур надзвичайно важливим питанням є подолання принципових обмежень існуючих технології при їх формування та розробка нових методів для виготовлення таких наноструктур. Оскільки, співвідношення «вартість/ефективність» є основною ринковою перепоною для поступу нанотехнологій і створення майбутньої елементної бази наноелектроніки, ми зробили спробу розробити ефективну, дешеву та конкурентноздатну технологію, що дозволить виготовляти наноструктури на основі тонких плівок напівпровідникових сполук AIIBVI. Представлено основні досягнення в формуванні наноструктур та проблеми пов'язані з виготовленням таких наноструктур на основі тонких плівок напівпровідникових сполук AIIBVI, що були вирощені методом Імпульсного Лазерного Напилення.