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The Growth of CdTe Layer on GaAs Substrate by MBE

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Abstract

We present the results of growth of CdTe layer on (013)GaAs substrate. The sequence processes include the preparation of GaAs surface by chemical etching and annealing in ultra-high vacuum, the growth of ZnTe layer on atomically clean GaAs surface and then the growth of CdTe layer on ZnTe/GaAs. All processes were carried out without removing GaAs substrate from MBE set. The processes were controlled by RHEED and single wavelength ellipsometry. We found that the evaporation of arsenic oxides and gallium oxides from the (001)GaAs surface were observed at over 400 and 500°C, respectively. The growth of CdTe on (001) GaAs leads to appearance of mixture orientations because of large mismatch of lattice parameters. We study the growth of ZnTe on (001)GaAs and (013)GaAs substrates to prevent the growth of mixture orientations. We study the influence of cadmium and tellurium ratio in molecular fluxes and temperature on the growth mechanism of ZnTe and CdTe, crystal perfection, surface roughness and defects density. The optimal condition for growth of high quality thick CdTe on GaAs substrate were found.

Keywords: epitaxy, growth, MBE, ellipsometry, RHEED CdTe, ZnTe, GaAs, surface defects, roughness, ellipsometric parameters Δ and Ψ

1. Introduction

The wide gap II–VI telluride compounds (CdTe, ZnTe, CdZnTe) are widely used as materials for different applications such as in solar and high power energetic detectors. The development of epitaxial technologies allows realizing the growth of high quality II–VI compound layers on different substrates. This gives the possibility to create multilayer structures on large in diameter widely used GaAs and Si substrate. The one of the important application of CdTe (CdZnTe) layers on GaAs substrate is as alternative substrate for sequence growth of photosensitive material mercury cadmium telluride (MCT) solid solutions [1–3]. This material is very attractive for development and production of thermal imagers that used in civilian life for medical, agricultural, chemical, metallurgical fuel industries, cosmic, etc. The MCT structures on large in diameter GaAs substrates are markedly reduce the cost of the MCT epitaxial photosensitive material and applied to creation large (up to megapixel size) focal plane arrays.

Molecular beam epitaxy (MBE) is the most appropriate epitaxial method of growing MCT layers due to its low growth temperatures (~180°C), which prevents the diffusion of impurities from the substrate and reduces the background doping [4]. But the dissociation and re-evaporation of diatomic Te molecules on singular surface at such growth temperatures is very low [5]. To increase the dissociation of diatomic Te molecules it is necessary to provide growth on vicinal surfaces. The development of multi-chamber MBE set allows carrying out the growth of MCT structures on GaAs with preliminary deposited CdTe layer in one process without uploading at air atmosphere. At the growth of CdTe on GaAs substrate t is necessary to solve the physical-chemical and technological problems. The physical-chemical problems are determined by the great differences in mismatch and nature of chemical bonding of conjugation CdTe and GaAs. In first problem the relaxation of induced stress lead to equilibrium state with introducing dislocation. In second ones the appearance of crystalline defects determined by the interaction of molecular beam Cd and Te2 with atomically clean GaAs surface. Actually the investigation of the initial stage of the CdTe growth on GaAs surface showed the formation of different mixture of orientation that seems connected with large mismatch and different surface superstructures which formed at Te interaction with GaAs [6]. The nucleation of one orientation reached by growing of ZnTe layer on GaAs surface with the sequence growth of CdTe layer.

The purpose of the chapter is to present the study of processes for high quality growth of CdTe on (013)GaAs substrate.

2. The experimental study of growth processes of CdTe layers on GaAs substrates

2.1 Pre-epitaxial preparation of substrates

The critical stage to obtain reproducible epitaxial growth of films with high structural perfection is the pre-epitaxial preparation of the substrate surface. The substrate surface must be atomically clean before epitaxial growth without any chemical contamination. We studied pre-epitaxial preparation of (001)GaAs surface substrate. There are two stages: chemical etching and thermal annealing in ultra-high vacuum (UHV) at heating in the temperature over 500°C in pre-epitaxial chamber of the MBE set "Katun" type equipped by RHHED and ultra-high velocity single wavelength ellipsometer [7].

2.1.1 The study of the chemical etching of GaAs substrate

It had been clear that the most stable surface contaminations on GaAs surface substrate is carbon that cannot be removed by desorption in UHV ~10^{-8} Pa at temperatures up to 600°C [8, 9]. It had been found that the presence of carbon contamination over 6% of the monolayer is the reason of disturbed 2D growth by MBE [8]. To prevent each contamination of GaAs substrate surface it is necessary to create protective films with the following requirements: have a low carbon adhesion coefficient; to be thick and uniform enough to encapsulate the GaAs surface; have high vapor pressure and completely desorbed from substrate temperatures below the decomposition temperature for GaAs which is equal over 600°C.

Such requirements are satisfied by creation oxides film formed on the surface of (001)GaAs during chemical etching which was developed by Cho [10] and used by other researchers.

This technique was used for study *in-situ* the mechanism of removal of the oxides film by heating of (001)GaAs surface [9]. High-resolution X-ray photoelectron spectroscopy (XPS) reveals that the original oxide film contains oxidized As^{+5} and As^{+3}, Ga_2O_3 and As^0 (elementary arsenic). The heating up to 350°C leads to remove As^{+5} oxide. At temperature over 500°C it was observed desorption of As^{+3} oxide and As^0. The evaporation of Ga^{+3} oxides occurs at temperature 570–600°C.

It was found that the level of carbon pollution (less 0.5% of the monolayer) decreases with the final etching in HCl in a high purity nitrogen atmosphere [9]. In this case, both arsenic and gallium oxides are removed from the surface and only a small amount of elemental arsenic remains on the surface. The advantage of this modification method is the removal oxides at room temperature, prevents violation of the stoichiometry of the GaAs surface and reduces the possible formation of defects at high temperatures.

In accordance with the above, we had developed a method of chemical cleaning of substrates for epitaxial growth of CdTe layers on GaAs substrate by MBE.

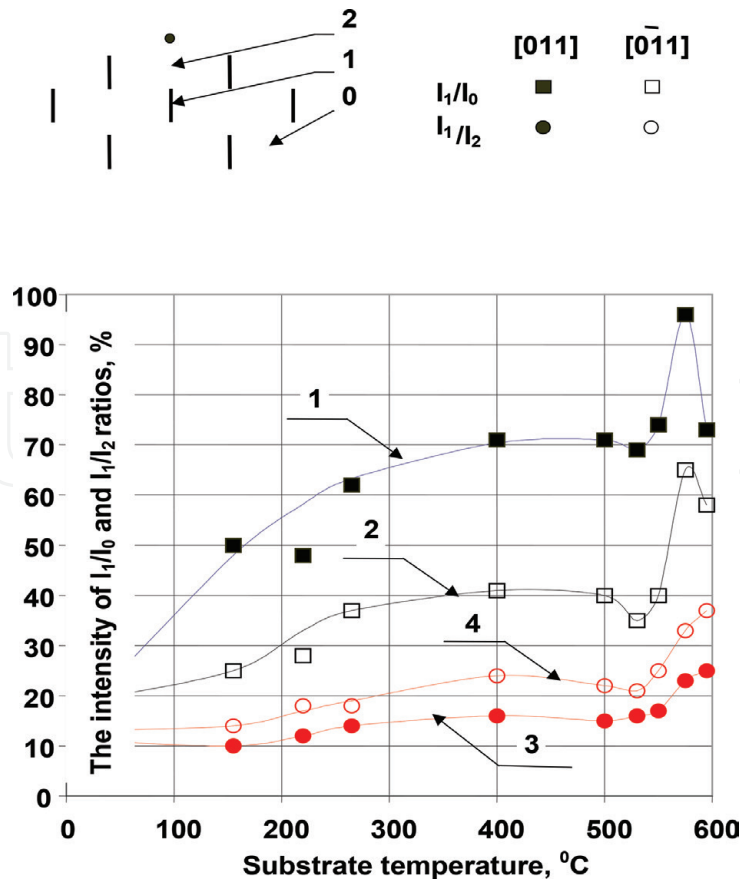The chemical preparation technique of the GaAs surface substrates includes:

- cleaning by freshly prepared organic solvents such as ethanol, boiling toluene;

- etching in mixture $H_2SO_4/H_2O_2/H_2O$ (3:1:1);

- rinsing with demonized water flow;

- drying under the IR lamp; and

- finish etching in boiling isopropanol and then in a boiling solution of HCl in isopropanol.

Further, the samples are placed on the substrate holder and moved to loading chamber where after pumping created vacuum ~5×10^{-6} Pa.

2.1.2 The study of the thermal annealing of GaAs substrate

The study of desorption protective film in UHV after chemical cleaning of (001) GaAs surface substrate was carried out in preparation chamber. The reflection high energy diffraction (beam voltage 12 keV) and single wavelength ellipsometry (wavelength λ = 632.8 nm) were used for monitoring process *in-situ* [11, 12]. The diffraction pattern during substrate heating was consistently photographed in [011] and [0−11] azimuths. The substrate temperature was controlled by a thermocouple. The vacuum was less than 3×10^{-8} Pa. After loading the GaAs substrate to preparation chamber the diffraction patterns in [011] and [0−11] azimuths at room temperature were observed as weakly elongated strips slightly visible on the background phone. At temperature increasing up to 250–300°C the diffraction reflexes brightness were increase on the background phone. In the temperature interval 300–540°C there is no noticeable change in diffraction reflexes in two azimuths. Since 540°C, there is observed sharp decreasing of the background phone with simultaneous elongating strips. Above 570°C the reconstruction of the surface and formation superstructures 2 × 1 and 3 × 1 was observed.

Figure 1 shows the changing the intensity of I1/I0 and I1/I2 ratios (curves 1, 3 and 2, 4 for azimuths in [011] and [0−11]) of diffraction patterns in central (I1) and edge (I2) parts of strip and diffused background (I0) of the (001)GaAs surface on the temperature. The intensities normalized to the background ones. It should be noted that in the temperature range of 20–300°C there is a sharp increase in I1,
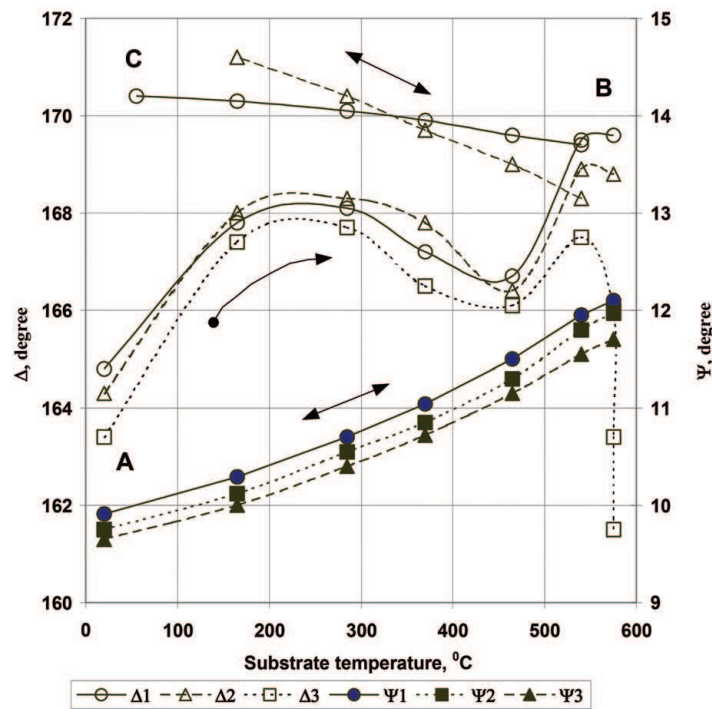
Figure 1.
The temperature dependences of RHEED intensity reflexes ratios (001)GaAs surface. Curves 1 and 3 for azimuth [011], curves 2 and 4 for azimuth [0−10].

which corresponds to the evaporation of volatile arsenic oxides. Then the noticeable change in the diffraction pattern intensities is not observed at temperature increasing up to 540°C. The sharp increase I_1/I_0 ratio and I_1/I_2 is observed at temperature above 540°C. At these temperatures gallium oxides are desorbed that lead to smooth (001)GaAs surface [8]. The exposition at temperature 570–580°C intensity reflexes decrease and strips pass into spots that associated with roughness and Ga enrichment of the surface.

The ellipsometric parameters Δ and Ψ were measured in the process of heating the GaAs substrate up to temperature 580°C and its subsequent cooling to room temperature. **Figure 2** shows the typical changes of Δ and Ψ for three (001)GaAs samples. The dependence of Ψ strictly increases at heating GaAs substrate and decrease at cooling. The dependence of Δ on the substrate temperature of N-shaped type curves (section AB) up to 150°C is due to removal of adsorbed arsenic oxides from the GaAs surface because of weaker chemical bonds compared to gallium oxides ones. Moreover, the value of the maximum in this area weakly varies from the conditions of chemical preparation of surface substrate. Further increase in temperature leads to Δ saturation in the range 250–280°C and slightly decreasing at 450°C. This dependence Δ is well correlated with the following evaporation of arsenic oxides and temperature changes of the optical constants of GaAs. In the range of 450–580°C a sharp increase Δ is due to the removal of gallium oxides [4, 8]. The ellipsometric data correlated with RHEED ones which at 580°C show the strikes with Kikuchi lines that indicated the absence of any oxide films and the atomically clean and smooth (001)GaAs surface.

At temperature cooling from 580°C to room temperature Δ gradually increases due to the change of the optical constants of GaAs (section BC). The same picture

Figure 2.
The dependences of the ellipsometric parameters Δ and Ψ on temperature for three different samples of (001) GaAs. The arrows show the direction of changing Δ and Ψ along the curves.

of changing Δ repeated at thermal cycling. At temperatures above 580°C there was a sharp Δ decreasing (Δ3, **Figure 2**) due to decomposition of GaAs and evaporation As atoms from the surface leading to roughness surface and Ga droplet formation. At heating GaAs substrates in As molecular flux parameter Δ stabilized at temperature range 170–600°C.

Thus, it was shown that *in-situ* ellipsometric control can be effectively used for thermal cleaning in UHV of the surface of GaAs substrate. It should be noted that the procedure of pre-epitaxial preparation of (013)GaAs does not differ from (001) GaAs. So the evaporation of oxides from the (013)GaAs surface during thermal heating in UHV is also not significantly different from (001)GaAs ones. The ellipsometric technique of surface control is not sensitive to the surface orientation. So we observed the same behavior of ellipsometric parameters in the case of heating (013) GaAs as for (100)GaAs.

2.2 The epitaxial growth of ZnTe and CdTe layers on GaAs substrate

The ZnTe layer 20–300 nm in thickness from elemental Zn and Te molecular sources preliminary must be grown on atomically clean GaAs substrate surface to prevent the formation mixture orientation [6]. This procedure helps us to conserve the original substrate orientation.

2.2.1 The epitaxial growth of ZnTe on GaAs substrates

The study of the growth process of ZnTe on (013)GaAs substrate for determination of optimal condition to realized high-quality ZnTe layers were carried out using ellipsometric technique [11, 13, 14]. ZnTe layer is transparent for using wavelength λ = 632.8 nm (He-Ne laser) of ellipsometer. In this case we observed periodic changing of ellipsometric parameters Δ and Ψ with ZnTe thickness associated with interference oscillations. The period of oscillations is equal to $\lambda/2(n^2-\sin\varphi)^{1/2}$ where

n and φ is the refractive index of ZnTe and the angle of incidence of the ellipsometer beam on the substrate surface, respectively. The changing Δ and Ψ in Δ-Ψ plane has closed bell-shaped curve.

The simulation of the changing of Δ and Ψ during the growth of ZnTe was carried out for different cases: the growth with roughness surface or/and appearance of radiation adsorption (k ≠ 0). The first reason is connected with changing of molecular fluxes Zn and Te2 (J_{Zn}/J_{Te2}) or/and growth temperature. The second reason is connected with changing in crystal perfection or inclusion of gallium or tellurium at high or low growth temperatures, respectively [15]. **Figure 3** shows the simulation results of changing Δ and Ψ for above mentioned cases. At the ZnTe growth with developed roughness surface there is a gradual Δ decreasing at the constant Ψ (see **Figure 3a**). The curves Δ and Ψ in Δ-Ψ plane are similar one another. In the case of growth of a weakly absorbing ZnTe layer (k = 0.05) the curves Δ and Ψ in Δ-Ψ plane are spiral that convergent more quickly with increasing of absorption in ZnTe layer (see **Figure 3b**). At growth of roughness and appearance of absorbed ZnTe layer the curves Δ and Ψ in Δ-Ψ plane summarized the phenomena observed in figures (see **Figure 3c**).
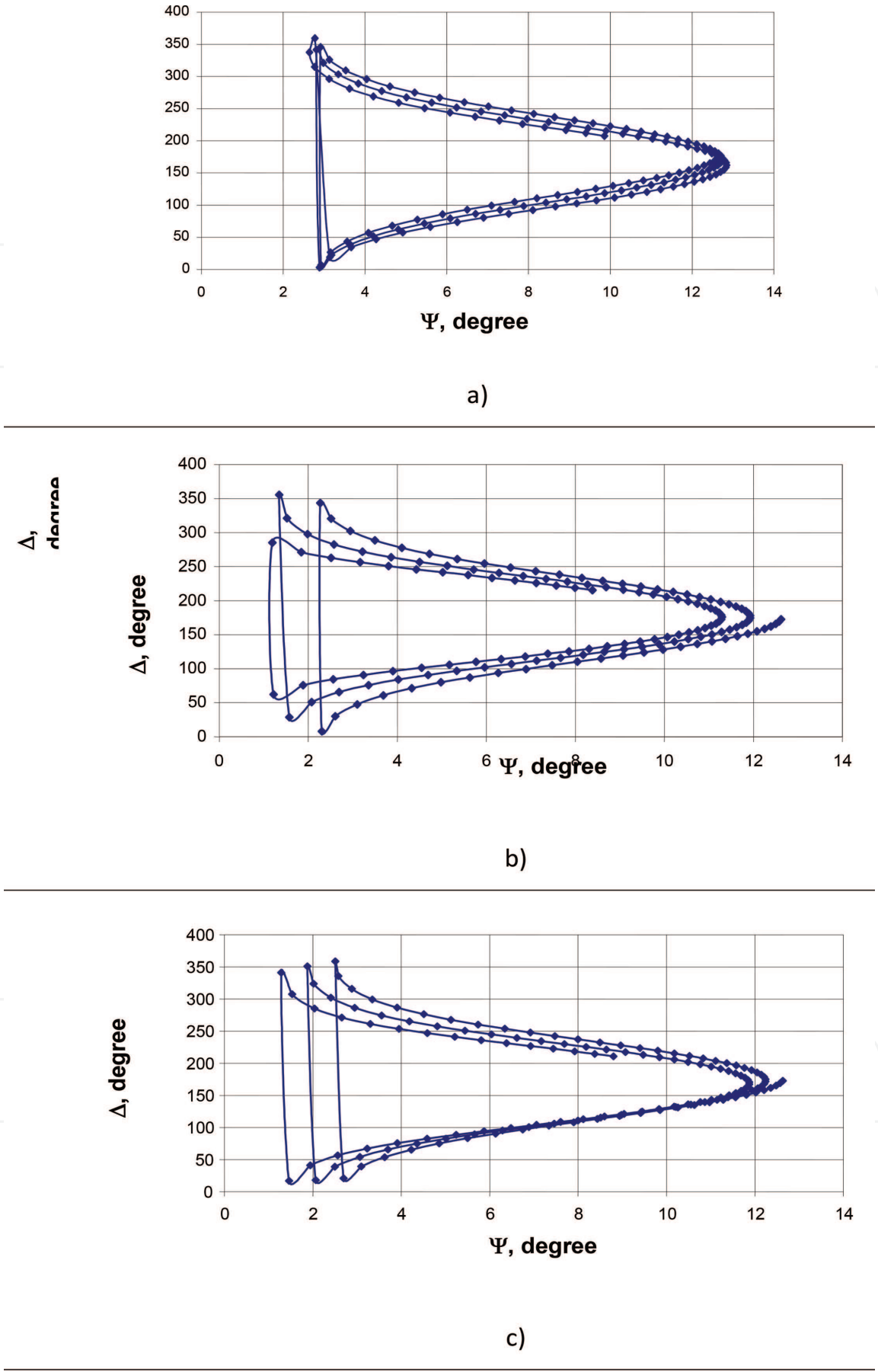
These data were used in determination of growth mechanism of ZnTe on GaAs at different J_{Zn}/J_{Te2} ratio and temperature. The growth was carried out at constant substrate temperature and different J_{Zn}/J_{Te2} ratios. In a series of sequence experiments the J_{Zn}/J_{Te2} ratio was reduced that allows determining the transition from 2D to 3D growth mechanism for a given temperature. Similar the experiments were carried out at different temperatures. **Figure 4** shows the results of experiments. The experimental results are presented by dots. The area of 3D ZnTe growth is represented by gray color. As a result there is found the optimal growth condition: the J_{Zn}/J_{Te2} = 2.75–3 and the growth temperatures 280–300°C.

Figure 5 shows the experimental change of Δ and Ψ in Δ-Ψ plane at optimal condition growth of ZnTe on (013)GaAs substrate (rhombs). The solid curve is calculated data at constant values of optical constants and roughness of growing ZnTe layer. The arrows show the direction of changing of Δ and Ψ with increasing ZnTe thickness. Such behavior of Δ and Ψ does not change with the growth of thick ZnTe layer. The diffraction patent shows the extended clear strips.

2.2.2 The epitaxial growth of CdTe on GaAs substrates

At the initial stage of CdTe direct growth on atomically clean (001)GaAs substrate surface the convergent spiral curve of ellipsometric parameters in Δ and Ψ in Δ-Ψ plane was observed for three experiments (see **Figure 6**).

The starting point O corresponds to Δ and Ψ of (001)GaAs substrate. The final point A corresponds to the Δ and Ψ for the bulk CdTe. The calculation curves Δ and Ψ was compared with experimental data basis on model of homogeneous crystalline perfect system CdTe/GaAs. It is apparently clear that the direct growth of CdTe on (001)GaAs gives poor crystalline perfection. The reason of such behavior is the formation of mixture orientations (001)CdTe and (111)CdTe [6]. The analogous data were suggested by the systematic deviation of the measured ellipsometric parameters from the calculated curve at growth up to 100 nm (noticeable see for curve 2 with triangular markers in **Figure 6**). The observed deviation of calculated Δ and Ψ at growth from experimental was explained by suppose the difference of optical constant values of growth CdTe layer from analogous for CdTe bulk or layers in thicknesses 2–3 μm. The good agreement between experimental results and calculation was observed at practically constant refractive index n = 3 which is typical for thick films and higher k which approached to CdTe volume value (see inset

a)

b)

c)

Figure 3.
The results of calculations of ellipsometric parameters changing for the growth of the ZnTe film on the GaAs substrate: (a) the roughness develops from 0 to 5 nm; (b) the absorption with k = 0.05; (c) the roughness develops from 0 to 2.5 nm and the absorption with k = 0.03.
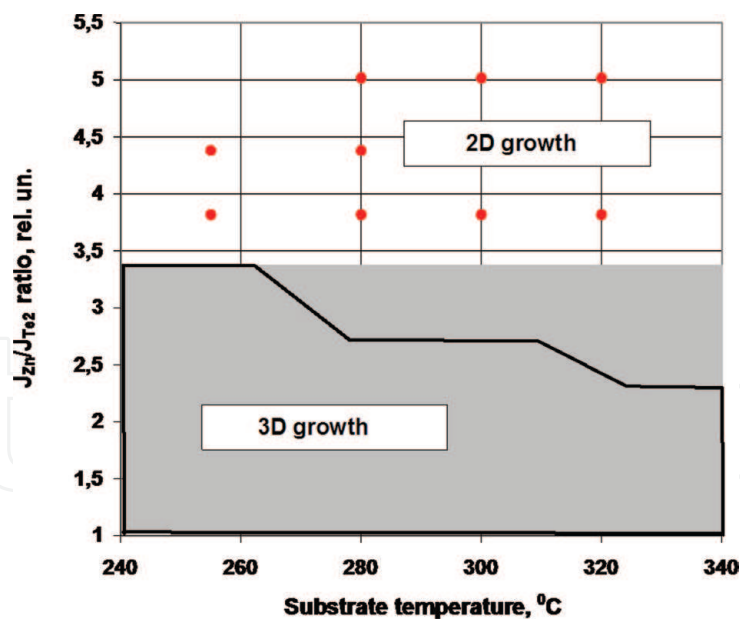
Figure 4.
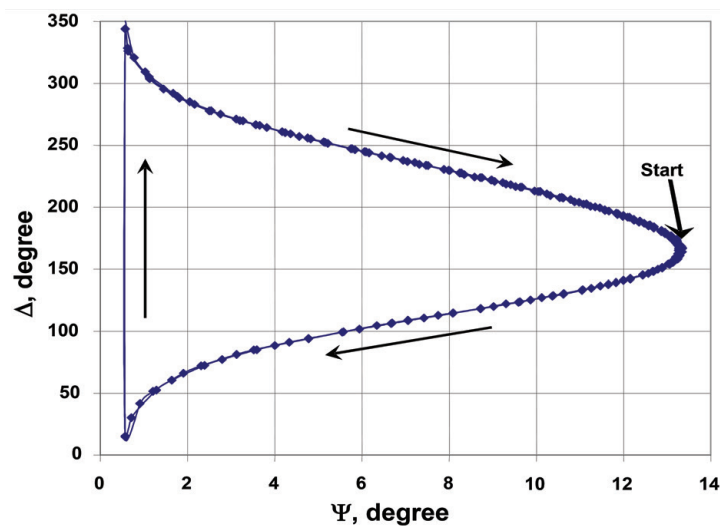*Transition from 2D to 3D growth mechanism ZnTe growth.*

Figure 5.
The change of ellipsometric parameters Δ and Ψ in Δ-Ψ plane in case of growth of ZnTe on (013)GaAs in optimal conditions. The arrows show the start of growth ZnTe and direction of changing parameter with increasing thickness. The rhombs is experimental data. The solid curve id calculated data.
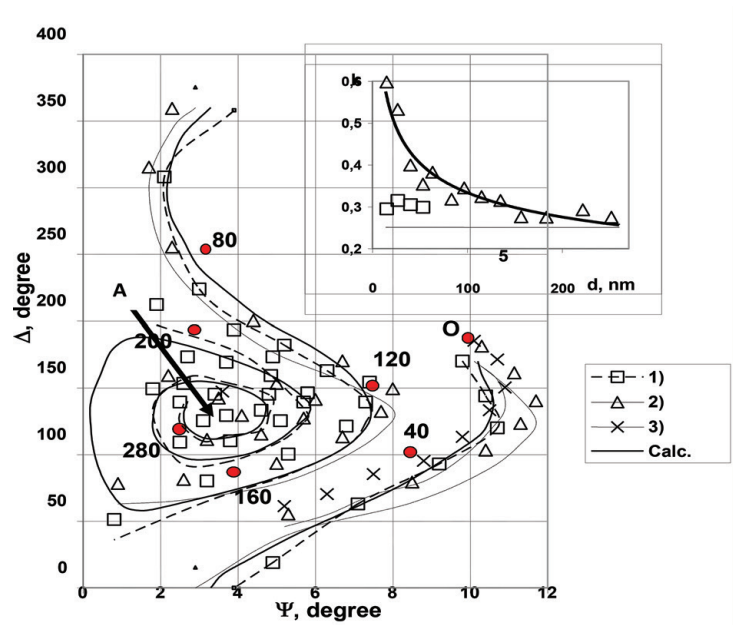
to **Figure 6**). This means that the adsorption at the interface CdTe/GaAs is higher at the beginning of growth and decrease from k = 0.6 up to k = 0.25 at thickness 200 nm characterized for bulk CdTe values. Such behavior of k can be explained by formation poor crystalline structure formation of CdTe at initial growth stage of because of great mismatch 13.6% at conjugation of CdTe and GaAs.

So for growing of high quality CdTe layer on GaAs it is necessary to introduce the additional layer with appropriate mismatch to GaAs and with the same crystalline structure as CdTe.
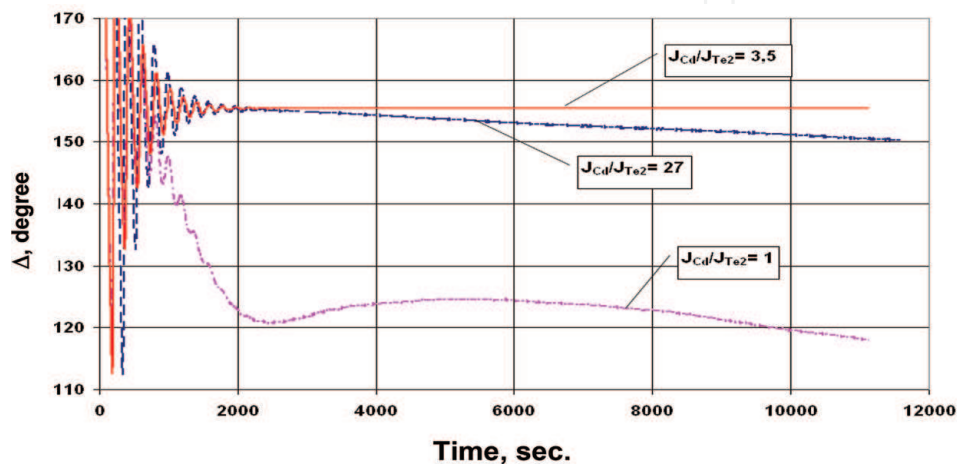
At growth of CdTe on (013)ZnTe/GaAs the changing of Δ and Ψ in Δ-Ψ plane represents by convergent spiral curve. For high quality crystalline CdTe layer on (013)ZnTe/GaAs there is the optimal growth condition which depends on J_{Cd}/J_{Te2} ratio and temperature. For this purpose we used measurement of Δ and Ψ during the growth *in-situ* at changing of J_{Cd}/J_{Te2} ratio and temperature. The first requirement for high quality CdTe layer is the 2D growth that determines flatness

of growing surface and creates minimal defects density. **Figure 7** shows the change of the ellipsometric parameter Δ for CdTe growth for different J_{Cd}/J_{Te2} ratio and constant temperature. The growth rate was 2.1 μm/h. Growth temperatures T = 290°C. In the case of J_{Cd}/J_{Te2} = 3.5 there is no changes in the ellipsometric parameters Δ. At large J_{Cd}/J_{Te2} = 27 a weak monotonic decrease in the ellipsometric parameter Δ is observed. But at low J_{Cd}/J_{Te2} = 1 there is observed sharp decreasing Δ practically from the beginning of growth. So it means that it is necessary to have the excess of Cd over Te in molecular fluxes. The observed decreasing of surface roughness is can be explained low sticking coefficient of Cd at growth temperature. This factor can influence on other CdTe characteristic such as: crystalline perfection and surface morphological defects.
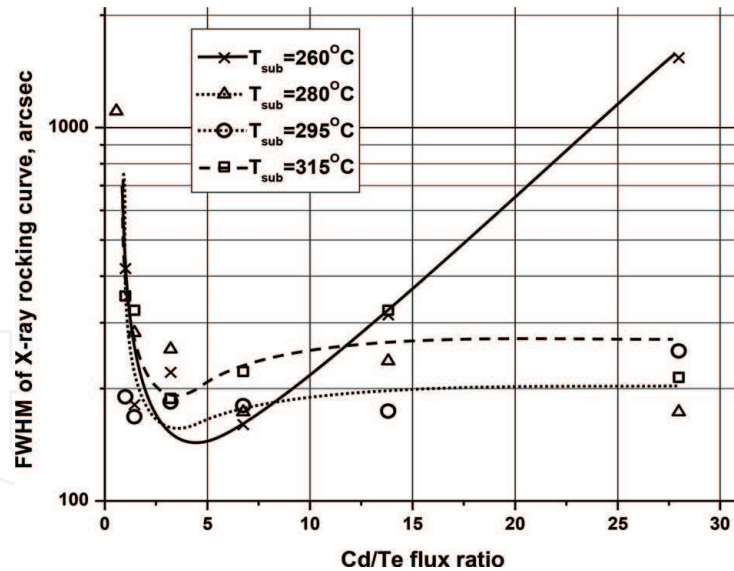
We study in detail the influence of J_{Cd}/J_{Te2} ratio on FWHM, roughness and surface defects density at different growth temperature (**Figures 8–10**) The structural perfection of the grown films was characterized by the full width at half maximum (FWHM) of the rocking curve measured on a double crystal X-ray diffractometer
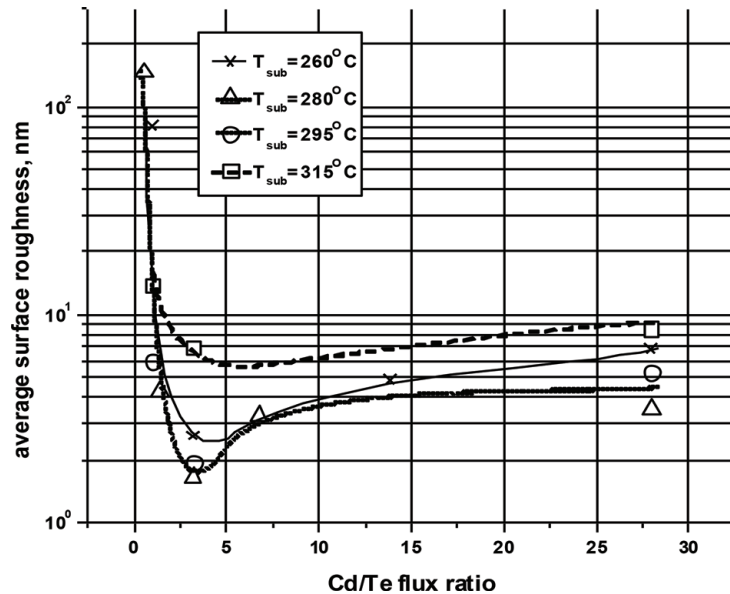

Figure 6.
The ellipsometric parameters change in Δ-Ψ plane at growth of CdTe on (001)GaAs. Markers show experimentally measured values for three experiments. The lines show the calculated changing of ellipsometric parameters. The red circles correspond to the CdTe layer thickness in nm. In insert the absorption coefficient changing with thickness used for fit calculation and experimental data sample 1 and 2. O—start of growth.

Figure 7.
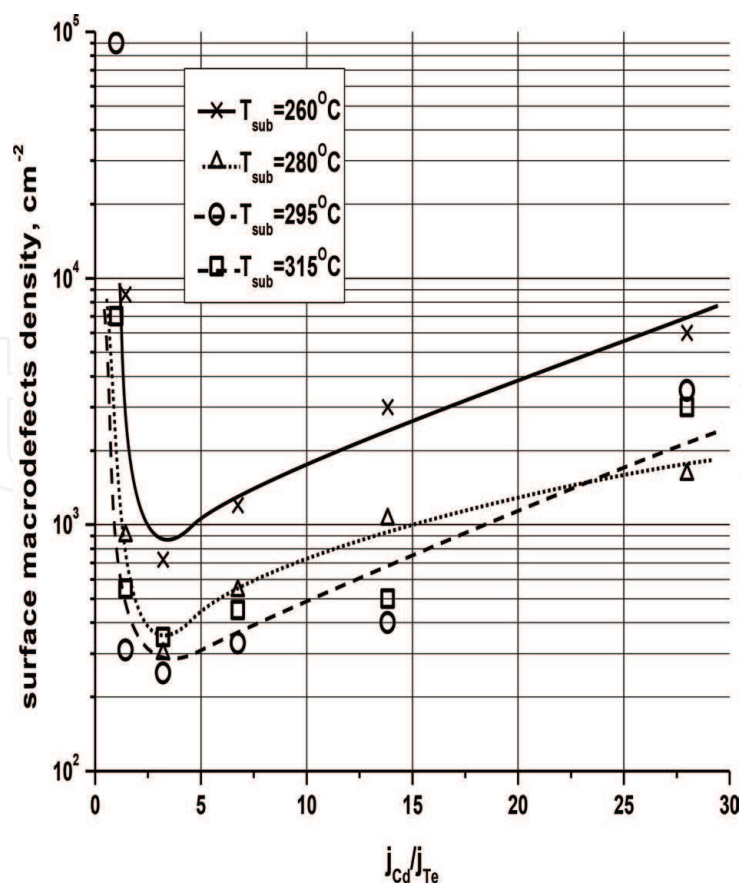*The change of ellipsometric parameter Δ with the time at the growth of CdTe on (013)ZnTe/GaAs.*

Figure 8.
The FWHM dependences on J$_{Cd}$/J$_{Te2}$ ratio at different growth substrate temperatures.

Figure 9.
The surface roughness dependences on J$_{Cd}$/J$_{Te2}$ ratio at different growth substrate temperatures.

with a germanium monochromator for reflex (004). It is seen in **Figure 8** that in region J$_{Cd}$/J$_{Te2}$ ratio <1 there is a sharp increase FWHM for all growth temperatures. In RHEED pattern point reflexes are observed that indicated 3D growth. We think that 3D growth is determined by the presence of adsorption of Te2 on the growing surface [9]. At increasing J$_{Cd}$/J$_{Te2}$ over 1 FWHM slightly varied up to J$_{Cd}$/J$_{Te2}$ = 28, except the case of growth temperature 260°C. RHEED patterns show 2D growth mechanism. Thus, FWHM reaches minimum at J$_{Cd}$/J$_{Te2}$ in range 1.5–7 for all temperatures of the substrate.

The surface roughness of the grown CdTe films was determined by scanning area 47 × 47 μm^2 with the help of AFM Solver P-47 H (NT-MDT). Yet at initial stage of growth CdTe at J$_{Cd}$/J$_{Te2}$ <1.5 the parameter Δ is dramatically decreased (tens of degrees at a thickness less than 1 μm). The RHEED reflexes change from the extended strips to spots. This is due to 3D growth of CdTe because of enrichment tellurium on the growing surface. The roughness of CdTe surface slightly developed over all growth condition at J$_{Cd}$/J$_{Te2}$ in range 2.5–28 up to 6 μm in thickness of CdTe

Figure 10.
The surface defects dependences on J_{Cd}/J_{Te2} ratio at different growth substrate temperatures.

layer with observed slightly reduction in parameter Δ. So the height of the surface roughness at J_{Cd}/J_{Te2} = 2–5 is 2–4 nm and at J_{Cd}/J_{Te2} = 28 increases approximately two times. The minimum roughness less that 1 nm reveals at J_{Cd}/J_{Te2} = 6.

The experimental data analysis of development roughness of CdTe surface at growth allows to determine the optimal growth conditions, such as growth temperatures 260–295°C and J_{Cd}/J_{Te2} = 1.5–7, for which the surface roughness is 2–3 nm.

The density of surface defects density of CdTe layer was determined by measuring the density of brilliant points on the surface with the help of optical microscope in reflected light with a built-in CCD camera. The dependences of the density of surface defects density on J_{Cd}/J_{Te2} ratio at different growth temperatures are presented in **Figure 10**. There is a sharp increase in surface defects density at J_{Cd}/J_{Te2} <1.5. The same changing in surface defects density is observed with increasing J_{Cd}/J_{Te2} in the range 5–28. It should be noted that surface defects density are minimal for grown temperature 295°C at any J_{Cd}/J_{Te2}. We found the optimal growth conditions for CdTe layer with a minimal surface defects density are as the growth temperature 280–315°C at J_{Cd}/J_{Te2} = 1.5 ÷ 7.

The analysis of total data represented in **Figures 8**–**10** allows to determine the optimal growth condition for growth of high-quality CdTe films on (013)ZnTe/ GaAs substrates. The CdTe growth at J_{Cd}/J_{Te2} = 5 ÷ 7 and temperatures 285–295°C gives the following parameters: FWHM 160–190 arcsec, surface roughness 2–4 nm and the surface defects density 200–300 cm^{-2}.

3. Conclusion

The study of preparation of atomically clean GaAs surface by sequence processes of special chemical etching and thermal annealing has been done and studied by both RHEED and ellipsometric control *in-situ*.

The changing of RHEED patents and parameter Δ reveals the changing in surface adsorbed contamination and roughness at heating up to 580°C connected with desorbing of arsenic oxides at low temperatures 400°C and then gallium oxides at high temperatures over 500°C. The comparison of the temperature dependence of the diffraction reflexes intensity with *in-situ* ellipsometric measurements showed the efficiency of ellipsometric control during thermal cleaning of the surface of GaAs substrates in ultrahigh vacuum.

The ellipsometric control *in-situ* was used in the growth of ZnTe on the (013) GaAs substrate. We found the influence of roughness and absorption on changing of ellipsometric parameters Δ and Ψ. This gives us to determine the areas of 2D and 3D ZnTe growth depending on growth temperatures and zinc to tellurium ratios in molecular fluxes. We found the optimal growth condition for epitaxy of high quality ZnTe layer on (013)GaAs substrate.

The dependences of FWHM, roughness and surface density defects on growth temperatures and cadmium to tellurium ratios in molecular fluxes reveals the growth condition for epitaxy of high quality CdTe layer on (013)ZnTe/GaAs substrate.

The high quality (013)CdTe/ZnTe/GaAs substrate is can be used as an alternative substrate and material for solar, high power energetic and IR photonics.

Acknowledgements

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