INVESTIGATION OF PHOTOVOLTAIC DEVICES CRYSTALLIZATION IN MOCVD WITH IN-SITU MONITORING

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ABSTRACT: the opportunity for successful applying a combination of reflectance (R) and reflectance anisotropy spectroscopy (RAS) in-situ methods for comprehensive analysis of multijunction solar cell (MJ SC) structures grown by MOCVD has been demonstrated. It was shown that in-situ RAS measurements can be used for determining the ordering degree and doping level in the $GaInP_2$ layers grown on both GaAs and Ge substrates. The influence of different growth conditions (temperature, V-III ratio, doping, substrate' type) on $GaInP_2$ ordering has been studied combined by in-situ RAS and ex-situ photoluminescence (PL) and a good agreement between results obtained by both methods was shown. Linear electro-optical (LEO) effect in Si and Zn doped $GaInP_2$ layers grown on both GaAs and Ge wafers has been studied and calibration reflectance anisotropy (RA) signal dependencies have been obtained. In-situ research of doped Ga(In)As layers has been carried out and main features in RA spectra behavior at critical point of dielectric function has been reveled at growth temperatures. $GaInP_2$/Ge interface roughness has been estimated by the RA transients during MJ SC structures growing. Correlation between structure surface morphology and RA signal value on the heterointerface has been found.
Keywords: multijunction solar cell, epitaxy, reflectance anisotropy spectroscopy, in-situ.

1 INTRODUCTION

The in-situ measurements are an important part in investigating the semiconductor epitaxial structures and creating different semiconductor devices by epitaxial method [1]. In-situ investigations by reflectance (R) means are widely applied. However, a combination of this method with the reflectance anisotropy spectroscopy (RAS) is the most effective one.

Investigations by the RAS means proved long ago itself as a useful instrument for determining a number of key parameters of epitaxial layers, semiconductor surfaces and interfaces [2-5]. The RAS is applied effectively for comprehensive investigation of different semiconductor devices grown by the MOCVD technique, such as heterojunction bipolar transistors [6], laser structures [7], and vertical cavity surface-emitting lasers [8]. However, there is the quote small number of papers on RAS application for investigating the multijunction solar cell (MJ SC).

In spite of modern tendencies in increasing the number of p-n junctions [9], the most widely spread type of multijunction structures are triple-junction solar cells (3J SC) based on the GaInP/GaAs tandem and narrowband junction (Ge or GaSb). At this moment these SCs have demonstrated high competitive efficiencies in converting both terrestrial [10, 11] and space applications [12, 13]. However, in spite of high sunlight conversion efficiency values achieved by these structures, they are far from the theoretical ones [14]. Using the in-situ methods allows spreading up the investigation process. This paper is devoted to application of the in-situ methods for investigating the semiconductors materials composing monolithic MJ SCs structures grown by the MOCVD on Ge substrates

In general GaInP/GaInAs/Ge structures consists of a wide band gap GaInP top cell and middle cell based on GaInAs grown on Ge substrate, in which a narrow band gap p-n junction is formed. Thus, in the case of monolithic lattice matched triple-junction SCs, in-situ methods might be applied to the following fields of investigation:

- investigation of the nucleation on a Ge substrate;
- investigation of Ga(In)As solid solutions with In concentration of the order of several percent for compensating a mismatch with a substrate by the lattice parameter;
- investigation of $GaInP_2$ solid solutions lattice matched with a substrate.

The first two problems have been considered in our previous papers [15, 16]. The present work is a continuation of these investigations and focused on studying the $GaInP_2$ solid solutions necessary for both creating the top subcell and growing a nucleation layer.

2 EXPERIMENTAL PROCEDURE.

The experiments have been performed in a horizontal low pressure R&D MOCVD reactor. Hydrogen was used as a carrier gas. The following metalorganic compounds were used as the third group element sources: trimethylgallium (TMGa), trimethylaluminium (TMAl) and trimethylindium (TMIn). Arsine (AsH3) and phosphine (PH3) were used as the fifth group element sources. Doping precursors for investigated epitaxial layers were disilane (SiH4) and diethilzinc (DEZn). The experimental layers for in-situ investigations as well as photovoltaic structures were grown on 2 inch in diameter substrates of two types: [100] GaAs 2^0 off to [110] and [100] Ge 6^0 off to [111] wafers. Additional information on the growth procedure and growth conditions is described elsewhere [17].

The MOCVD installation is equipped with a UV-transparent quartz window for optical in-situ measurements. A commercially available spectrometer has been used for performing R as well as reflectance anisotropy (RA) signal on rotating samples. Due to rotation in MOCVD RAS measurements are more complicated especially when it is accompanied by some degree of wobble [6]. Direct measurements during MOCVD growth processes give the absolute value of RA signal that should be taken into account in analyzing

experimental results.

According to Aspnes [18], the reflectance anisotropy for cubic crystals is the difference in reflectance between the two surface symmetry axes along [110] and [-110] directions. For (100) surface the RA signal is defined by the equation

$$\frac{\Delta R}{R} = 2\frac{R_{110} - R_{-110}}{R_{110} + R_{-110}} \qquad (1)$$

,where R_{110} and R_{-110} are the complex reflectances for the polarizations parallel to the [110] and [-110] directions.

The difference in the reflection coefficients for light polarized along the main crystalline axes results mainly from the surface anisotropy [19] (taking into account the bulk symmetry of cubic crystals). This anisotropy is characterized by a spatial configuration of dimmers formed due to dangling surface bonds. RAS allows picking out weak signals of the surface optical transitions using polarization properties.

There is a number of factors decreasing the surface isotropy, such as: surface reconstruction, morphology, atomic ordering and a surface electric field. The RA signal is a superposition of all contribution. Comparison of the spectra of experimental samples with, for example, similar reconstruction allows obtaining a contribution of ordering and/or doping to their surface anisotropy.

3 RESULTS AND DISCUSSION

3.1 GaInP2 system.

The bulk properties of the GaInp solid solutions have been rather thoroughly studied including the in-situ reflectometry, which is a useful tool for determining growth rates and compositions from the Fabry-Perot (FP) oscillations analyzing [20].

The RAS may be also applied effectively for in-situ determination of a number GaInP layer key parameters, in particular, for analyzing the doping level in GaInP layers. It was possible due to the RAS is sensitive to the electrically active impurity atoms in the layer. Such sensitivity results from the linear electro-optical (LEO) effect caused by the field of the near-surface space charge region [21]. Sensitivity of the RA spectra to both n- and p-type dopings has been demonstrated for GaInP, and the linear dependence of the LEO effect on the logarithm of the free carrier concentration has been established [22].

Nevertheless, even at alive reconstructions the GaInP anisotropy is determined not only by the near-surface static field, but also by bulk properties associated with the ordering effect. A characteristic peculiarity of the GaInP solid solutions is CuPtB-type ordering on the group-III sublattice [23]. Bulk-ordering effect results in narrowing the semiconductor band gap [24] and lower down the material isotropy. For this reason, the bulk-ordering effect contribution can be determined by the RAS means. In [25], the bulk-ordering contribution into the RA signal was separated from the surface reconstruction at different growth conditions: in varying temperature, the ratio of V and III group elements in gas phase (V/III-ratio), substrate mis-orientation, and also at a high doping level.

There are new publications devoted to investigating GaInP solid solutions by means of the in-situ RAS during MOCVD [22, 26], but epitaxial layers grown only on GaAs substrates are considered. However, GaInP in MJ SC can be used not only the top subcell, but also be a wide-band gap window for Ge bottom subcell. For this reason, to apply the in-situ monitoring for analyzing MJ SC structures, experiments were carried out on growing differently doped GaInP epitaxial layers on both GaAs and Ge substrates.

Figure 1 presents the RA spectra of undoped GaInP layers grown at different conditions (temperature, V/III-ratio) and on different substrates (GaAs, Ge).

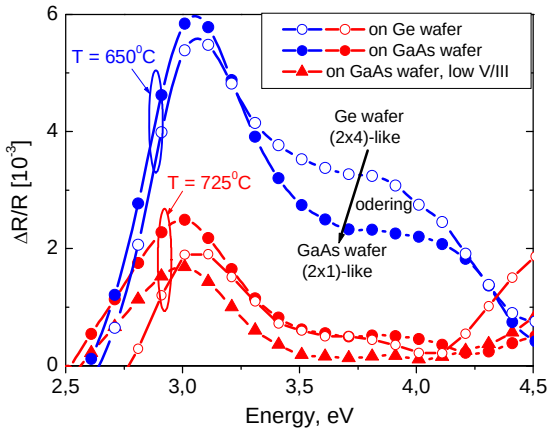

Figure 1: Effect of growth parameters on the bulk odering in $GaInP_2$ grown on Ge (○) and GaAs at high (●) and low (▲) V/III-ratio, for growth temperatures of 650^0C (—) and 725^0C (—). The surface reconstruction changes from c (2×1)-like to (2×4)-like, and bulk ordering decreases with temperature increasing, reducing the V/III-ratio and in growing on Ge substrates.

For the undoped GaInP layers grown on GaAs substrates at low temperatures (650^0C) the (2×1)-like reconstruction is observed, which is characterized by a sharp peak at 3eV [25]. Such reconstruction corresponds to the phosphorus-rich surface condition and a maximal bulk-ordering in GaInP is observed in this case. In growing GaInP on Ge at the same growth temperature (figure 1**)** the reconstruction changes from (2×1)-like to (2×4)-like. This indicates the ordering degree decreasing.

This fact has been checked by investigating photoluminescence (PL) spectra of GaInP epitaxial layers grown at similar conditions on GaAs and Ge substrates. It is seen from figure 2, that the PL peak energy of GaInP on Ge shifts towards shorter wavelength region, which indicates widening of the semiconductor band gap and, hence, reducing the bulk-ordering. Such a behavior may be partly explained by the effect of auto-doping of the epitaxial layer with Ge atoms [16], since the high concentration of the impurity atoms in the semiconductor also facilitates disordering (figure 3c).

In increasing the growth temperature, decreasing the RA signal intensity is observed and (2×1)-like reconstruction changes to (2×4)-like. This is, probably, associated with the increase of the degree of the phosphorus desorption from the surface at the higher growth temperature. The results of investigation of the PL peak energy shift for GaInP layers grown at different temperatures (figure 3a) have good agreement with the in-situ RAS data.

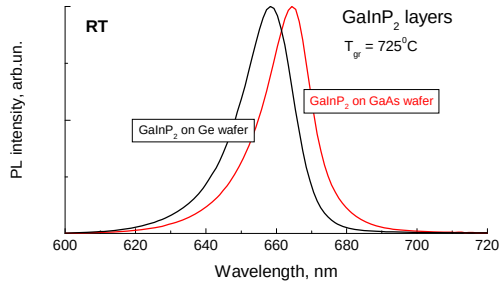

Figure 2: Photoluminescence of RT for $GaInP_2$ grown on GaAs and Ge substrates at similar conditions.

Another parameters affecting the variation of the GaInP band gap, i.e. ordering of the solid solution, is the V/III-ratio [24, 27]. Decrease of V/III-ratio leads to reduction of phosphorus atom partial pressure over a growing surface that results in forming (2×4)-III group rich reconstruction. Figure 1 shows that, in reducing the V/III-ratio in three times, disordering rises and the (2×4)-like reconstruction becomes even more obvious. The ordering decreasing in reducing the V/III-ratio is also confirmed by investigation of GaInP layer PL peak energy shift (Figure 3b).

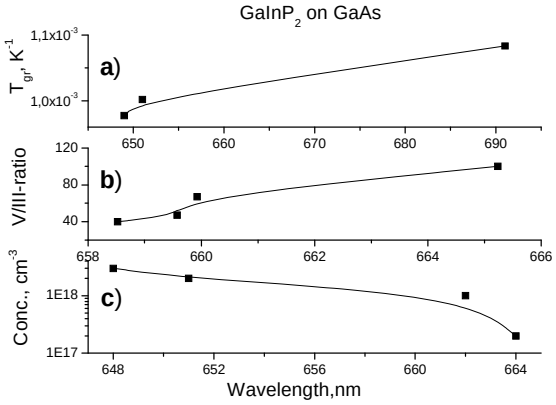

Figure 3: Dependence of the $GaInP_2$ absorption edge on growth conditions: **a)** growth temperature, **b)** V/III-ratio, **c)** free charge carriers concentration (curve is showed for Zn doped $GaInP_2$) with all other conditions being the same. (Growth temperature for b) и c) was 725^0C).

Thus, the in-situ measurements can be used as a tool for analyzing the ordering degree in the GaInP solid solutions, which is important from viewpoint of creation of GaInP/GaInAs/Ge MJ SCs, with the increased U_{xx} of the top subcell to widen the spectral range of converted radiation.

The LOE effect in n- and p-GaInP grown at different conditions has been also studied.

Figure 4 shows that, at a definite GaInP surface reconstruction, a correlate variation of the RA spectrum in the short-wavelength region in dependence on a type and a doping level is observed. The effect of a near-surface field induced by impurity is clearly indicated for a low ordering degree in increasing temperature and decreasing the V/III-ratio (see insert in figure 4), and also for GaInP on Ge in spite of the surface reconstruction close to (2×4)-like one (figure 5).

Besides, it is clear from figure 4 that, in increasing the doping, a gradual transition of the (2×1)-like reconstruction to the (2×4)-like one take place. This indicates that the spectrum shape can not be explained only by the influence of the near-surface field induced by doping. The high impurity atoms concentration may effect the surface morphology and, at very high doping (as, for example, for GaInP-p(Zn) with 1×10^{19} cm^{-3} free carrier concentration) the surface reconstruction. It was found that impurity atoms affect strongly PL peak position and 1.92 eV was achieved (figure 3c).

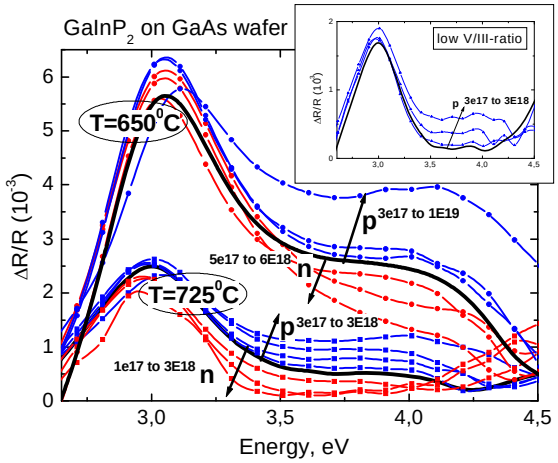

Figure 4: RA spectra for undoped (——), p-type: Zn (——) and n-type: Si (——) doped GaInP layers grown on GaAs wafers at 650^0C and 725^0C growth temperatures; in the insert – the RA spectra are GaInP grown at low V/III-ratio.

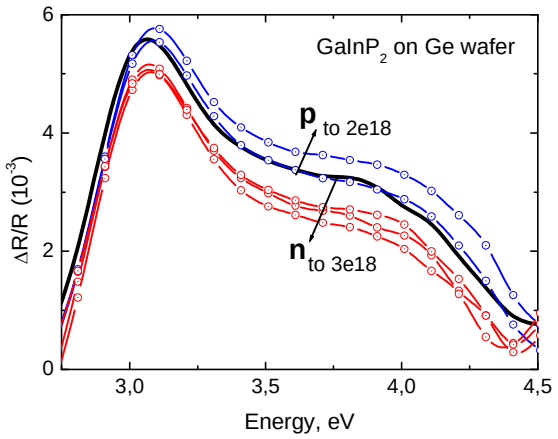

Figure 5: RA spectra for undoped (——), p-type: Zn (——) and n-type: Si (——) doped GaInP layers grown on Ge wafers at 650^0C.

Since the RA spectrum for an undoped GaInP includes a signal due to both the surface reconstruction and bulk ordering, to describe the anisotropy induced by the doping impurity, Δ RAS may be the most suitable value. Δ RAS is a difference between signals for doped and undoped layers as $\Delta RAS = \Delta R/R_{doped} - \Delta R/R_{undoped}$. The dependence of the Δ RAS value for the short-wavelength region (corresponding photon energy are

3.5÷3.8 eV) shows a direct dependence on the logarithm of the free charge carrier concentration (measured by the Hall technique) (figure 6). As would be expected, the data for the layer with the free-carrier concentration higher than 1×10^{19} cm^{-3} did not fit the corresponding curve.

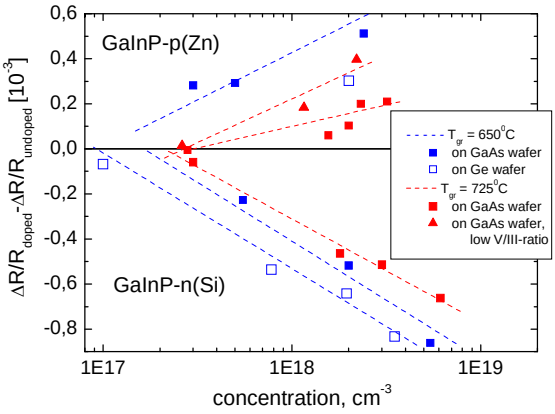

Figure 6: Dependence of the short-wavelength Δ RAS signal ($\Delta R/R_{doped} - \Delta R/R_{undoped}$) on the free charge carrier concentration in GaInP layers grown at different conditions on GaAs and Ge wafers (legend on the insert).

With obtained calibration curves the concentration of free charge carriers for n- and p-GaInP layers grown on GaAs substrates can be resolved down to ~$1\div2\times10^{17}cm^{-3}$ at 650°C and down to ~$3\times10^{17}cm^{-3}$ at 725°C. With temperature increasing the Δ RAS value is decrease that correlates with general RA signal decreasing.

3.2 Ga(In1%)As system

Gallium arsenide and their solid solutions are underlying many semiconductors devices including photovoltaic converters. For this reason there are a great number of publications related to investigations of GaAs by means of the in-situ methods. Including obvious correlation between the GaAs (100) surface reconstruction used in the MOCVD and the RA spectra shape has been established [28, 29]. Also, the LEO effect has been studied, and it has been found that this effect is directly proportional to the logarithm of free-carriers concentration of both n- and p-types [30].

For manufacturing monolithic isoperiodic GaInP/GaInAs/Ge MJ SCs, the solid solutions of GaAs with In (1% concentration) to ensure lattice matching between the middle subcell and Ge substrate are used. It has been established that introducing to the solution 1% of indium does not, practically, affect the undoped material RA spectrum and also the LEO effect in doped epitaxial layers [16].

GaAs layers and GaIn(1%)As solid solutions doping by Si and Zn impurity atoms have been studied. The main features in the spectra behaviors of differently doped layers reveal themselves at room temperature at the critical points of the dielectric function: E_0 и $E_0+\Delta_0$ (at 1.41eV and 1.75 eV correspondingly), E_1 и $E_1+\Delta_1$ (at 2.93 eV and 3.17 eV), and also at $E`_0$ (4.49 eV) and $E`_0+\Delta_0`$. [31].

In raising temperature, the increase in the number of free-carriers occurs, which screens partially the static near-surface field, induced by doping atoms. The RA signal becomes weak due to such free-carrier screening in combination with thermal broadening of the RA spectrum peaks [32].

However, fabrication of MJ SC structures by MOCVD requires the in-situ measurements at high temperatures. In our work, epitaxial layers doped with both Si and Zn and grown at 600-700°C have been in-situ studied. They have shown c(4×4)-like reconstruction [28]. Even at the growth temperature of 600°C for both n- and p-type layers, obvious dependencies of the Δ RAS value (undoped RAS spectra subtracted) on the level and type of doping were observed, practically for all mentioned characteristic energies (figure 7). But it should be taken into account that they are shifted towards longer wavelength region.

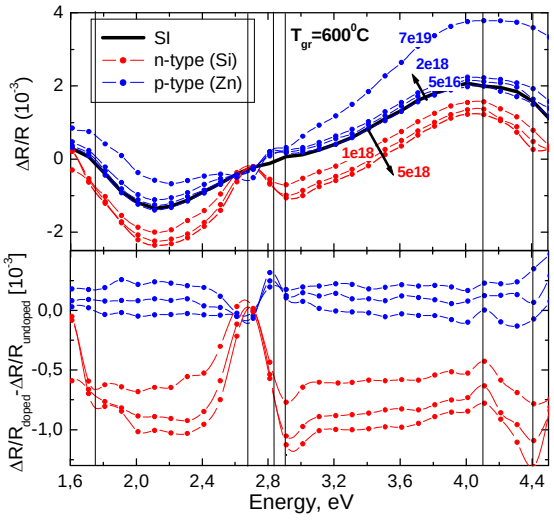

Figure 7: RA spectra (upper part) and ΔRAS spectra ($\Delta R/R_{doped} - \Delta R/R_{undoped}$) for p-type: Zn (——) and n-type: Si (——) doped Ga(In)As layers at 600°C growth temperature; energies near the critical points of dielectric function are marked by vertical black lines.

It is seen from the Figure 7 that, for the energies E_1 and $E_1+\Delta_1$, noticeable signal jumps are observed, which have a shape of oscillations with opposite behavior for p- and n-doping. Similar behavior, but more weak, is observed in the short-wavelength spectrum region for $E`_0$ and $E`_0+\Delta_0`$. For the long wavelength characteristic energies E_0 and $E_0+\Delta_0$, the LEO effect features reveal weaker. Besides, a general shift of the RA spectra is observed for different doping, which allows, from practical viewpoint, determining the layer doping level at any wavelength.

In further increasing temperature, the spectra behavior for the energies E_1 and $E_1+\Delta_1$ becomes less pronounced (figure 8). The signal intensity drop due to mentioned above screening of the near-surface field by free-carriers. In this case, a thermal widening and variation of peaks` forms take place resulting in that, even at 650°C and higher, one may clearly distinguish only one peak for energies E_1/ $E_1+\Delta_1$, instead of two ones.

Therefore, for the high growth temperatures Δ RAS in ultra-violet (UV) spectral range is most appropriate value described LOE effect. Figure 9 shows the direct

dependence of Δ RAS on the logarithm of free-carriers concentration (obtained from Hall measurements). Using the obtained calibration curves, the charge carrier concentration can be determined directly during the MOCVD process starting from $2\div3\times10^{17}$ for both n-type (Si) and p-type (Zn) layers. There were not found an obvious dependence of LEO effect on temperature in the range $600\div700^0$C.

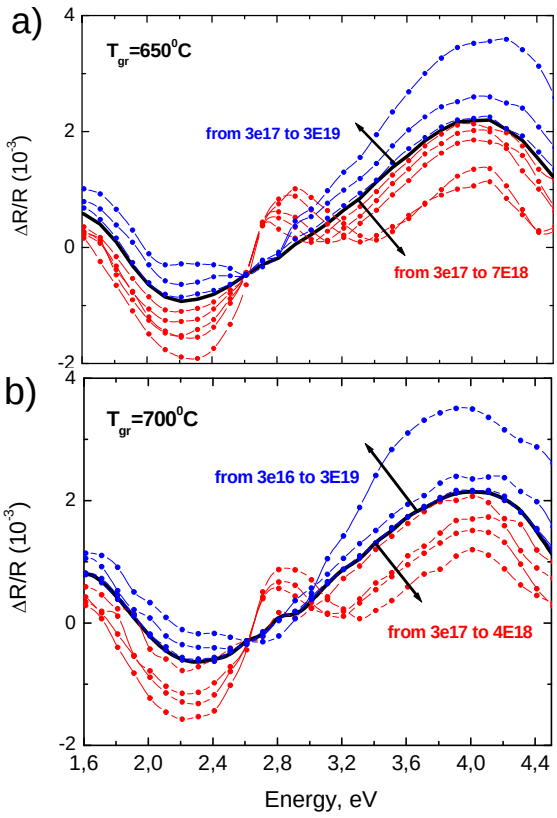

Figure 8: RAS spectra for p-type: Zn (——) and n-type: Si (——) doped Ga(In)As layers at different growth temperature a) $T_{gr} = 600^0$C, b) $T_{gr} = 700^0$C

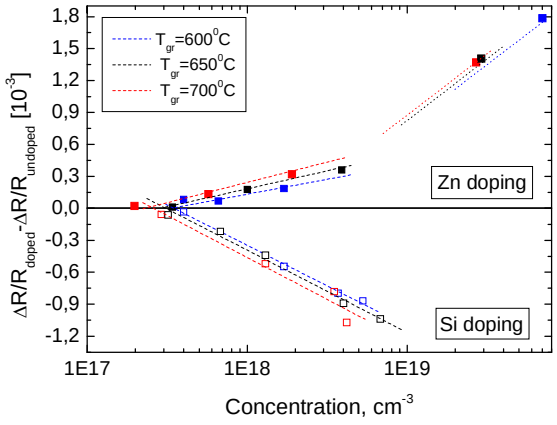

Figure 9: Dependence of short wavelength Δ RAS signal ($\Delta R/R_{doped} - \Delta R/R_{undoped}$) on free charge carrier concentration in n- and p-Ga(In)As layers at different growth parameters.

At very high impurity atom concentration, the RA signal value apparently depends on the superposition of two factors: the effect of active atoms on the near-surface electric field and the surface condition change. This reviled for Δ RAS in all spectral range including the UV one. Δ RAS values for p-type layers with high Zn concentration (approximately more than 1×10^{19} cm^{-3}) didn't fit logarithmic dependence shown on figure 9.

3.3 Photovoltaic structures grown on Ge wafer.

The experimental investigations of the Ga(In)As layers grown at temperature range of $600\div700^0$C allowed establishing a correlation between the RA spectra of both doping types in a wide concentration range as well as a direct dependence of the Δ RAS on the logarithm of the free-carriers concentration for the short wavelength range. Similar dependencies have been found for $GaInP_2$ of both doping types. This result allows analyzing homojunctions and their doping levels for the GaInP and GaAs subcells in MJ SC with single-wavelength measurements at the short-wavelength region. Figure 10 shows such a triple-junction GaInP/GaInAs/Ge structure measurements at photon energy of 3.8 eV. Simultaneously with RAS measurements the normalized reflection was measured at 2.1 eV. The short wavelength measurements characterized by the larger absorption depth allow obtaining the quite large number of FP oscillations on the reflection, which is useful for composition, growth rate and thickness of a material determination.

In the previous paper the conditions of deoxidation of Ge substrates and the suppression of auto-doping by germanium of the photoactive parts of SC structure have been found [16]. These results are necessary for growing structures on Ge with high crystalline perfection and surface morphology, as well as good photovoltaic properties.

Special attention has been given to the studies of GaInP/Ge interface (circle in middle part of figure 10). Experimental investigations allowing one to determine the most optimal conditions for growing nucleation GaInP layers on Ge substrates have been carried out. The quality of interface can be determined by analyzing of the RA signal temporal dependence. It is known, that high RA signal at the interface directly correlate with a rough interface [33].

Three RA transients from GaInP layers grown on Ge after different pre-epitaxial treatment are shown in figure 10 (lower part). The maximum RA signal rise was observed at growth on a not completely deoxidized germanium substrate. Further growth of a bulk semiconductor structure resulted in imperfect crystallization of the epitaxial layers. This has been confirmed by comparison of the R signal recorded in the single-wavelength mode with successful run. Also ex-situ investigations with SEM show bad morphology for such layers. The minimal value of the RA signal at the $GaInP_2$/Ge interface achieved was 8. In this case, the reflection signal level remained unchanged during the following growth of the bulk crystal.

Thus, in-situ measurements allow determining the key parameters for successful nucleation on germanium substrates, including ones, which are difficult obtaining by means of ex-situ methods.

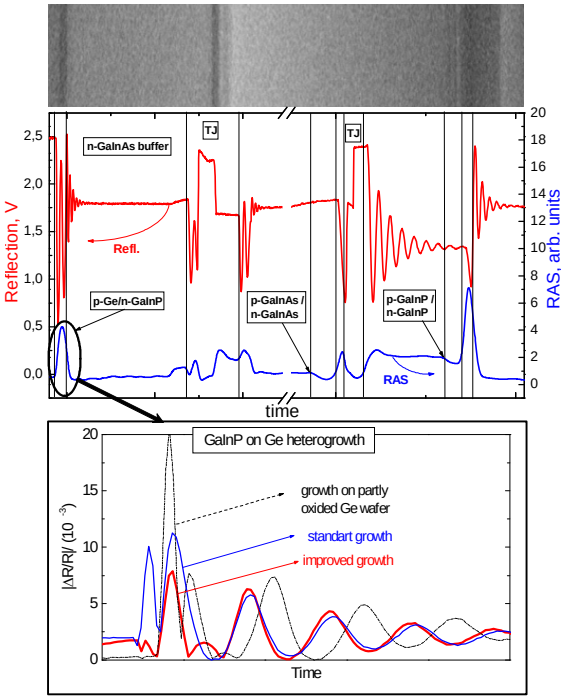

Figure 10: SEM image (upper part) and single wavelength in-situ measurements (middle part) with RAS (on 3.8 eV) and R (on 2.1 eV) of $GaInP_2$/GaInAs/Ge based 3J SC structure; transients from GaInP on Ge heterogrowth (lower part) at different pre-epitaxial treatments.

4 SUMMARY

In-situ investigations have been curried out aimed to create a complex method for analysis of growth process and control of MJ SC structures key parameters. By RAS means, the bulk-ordering effect in the $GaInP_2$ solid solutions grown on both GaAs and Ge wafers has been studied. By means of ex-situ PL spectra investigations, the growth parameters resulting in the GaInP layers E_g rise have been determined and a correlation between the bulk-ordering and RA spectra has been established. This indicates that the in-situ measurements may be used as a tool for analyzing the bulk-ordering degree in GaInP solid solutions in growing a wide-band gap subcell in a MJ SC. The LOE effect in GaInP and Ga(In)As has been investigated, and calibration dependencies of Δ RAS value on free-carriers logarithm have been obtained for the short-wavelength region at high growth temperatures, which allows carrying out an analysis of homojunctions in SC structures. By the MOCVD technique, GaInP/GaInAs/Ge SC structures have been grown, and a opportunity for successful application of combined RAS and R in-situ measurements has been demonstrated.

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