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## Relaxation process and luminescence of lattice defects in epitaxially grown ZnSe/GaAs layers

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### Abstract

In this work, we report on the dependence of strain relaxation in epitaxially grown ZnSe/GaAs layers on the growth process. New investigations about luminescence signals are presented, which are caused by lattice defects in these layers. The strain is due to different lattice constants of layer and substrate material and is relaxed by the nucleation of misfit dislocations during growth. This relaxation process depends on layer thickness and growth conditions. We determine the residual strain for different layer thicknesses and growth processes with X-ray diffractometry. Furthermore, the created misfit dislocations give rise to a luminescence at 2.6 eV, the so-called Y line. We found that its behaviour in ZnSe is analogous to that of Y lines in epitaxially grown ZnTe/GaAs layers. An excitation of the Y luminescence is only possible with photon energies higher than the recombination energy of free excitons. Its intensity decreases with increasing impurity concentration. It also decreases strongly for a steady state excitation at 2 K. A recovery effect of luminescence intensity is observed, if the sample is heated to room temperature. Furthermore, the strain dependence of the Y luminescence in ZnSe is compared with the maxima of bound exciton luminescence from shallow donors and acceptors.

### 1. Introduction

For the epitaxial system ZnSe/GaAs the lattice mismatch  $f(T)$

$$f(T) = \frac{a_S(T) - a_L(T)}{a_L(T)} \quad (1)$$

at a growth temperature of about 350°C is:  $f = -0.31\%$ , where  $a_S(T)$  and  $a_L(T)$  are the bulk lattice constants of substrate and layer, respectively. The lattice mismatch  $f$  causes a biaxial

strainfield in the layer material. The strain parallel to the ZnSe/GaAs interface  $\varepsilon_{\parallel}$  is given by:

$$\varepsilon_{\parallel} = \frac{a_{\parallel} - a_L}{a_L}. \quad (2)$$

When the layer growth reaches the so-called critical thickness  $h_c$  the strain starts to relax by nucleation of misfit dislocations [1]. The value of  $h_c$  depends on the growth conditions. X-ray diffraction measurements and photoluminescence (PL) measurements were carried out at room temperature (RT) and 2 K, respectively. At these temperatures, the strain of the layers is caused by the residual strain at the growth temperature,  $\varepsilon_{\text{res}}$ , and by the difference of the expansion coef-

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ficients of ZnSe and GaAs ( $\epsilon_{\Delta T} = +0.04\%$  for  $\Delta T: 350^\circ\text{C} \rightarrow 20^\circ\text{C}$ ;  $\epsilon_{\Delta T} = +0.1\%$  for  $\Delta T: 350^\circ\text{C} \rightarrow 2\text{ K}$ ):

$$\epsilon_{\parallel} = \epsilon_{\text{res}} + \epsilon_{\Delta T}. \quad (3)$$

In order to determine the strain of the layer material it is necessary to observe various asymmetric reflections as (224) and  $(\bar{2}\bar{2}4)$  in X-ray diffractometry. The strain of the layers is calculated from the angular difference between the substrate and the layer peak as described in Refs. [2,3].

For a characterisation of the crystalline quality, the FWHM values of the rocking curves of the (004) reflection are mostly used. In this case the scattered X-ray intensity is measured with a widely opened detector. For thin, fully unrelaxed layers, the rocking curve halfwidth is completely determined by the layer thickness itself, whereas for relaxed layers the FWHM values are dominated by the dislocation density [3,4]. Therefore a determination of  $h_c$  is possible by the observation of the strain as well as by the observation of the rocking curve halfwidths.

In PL measurements of ZnSe excited with interband excitation at low temperatures ( $T < 100\text{ K}$ ) extended lattice defects cause an emission at 2.6 eV (220 meV below the gap energy  $E_0$ ), called Y line. This luminescence signal was observed and described by several authors [5–8]. They found unusual optical properties for the Y line such as low phonon coupling and a small thermal activation energy of about 20 meV. A comparison of transmission electron microscopy (TEM) and cathode-luminescence measurements by Myhajlenko et al. enabled the authors to interpret the Y emission in ZnSe as electron–hole recombinations at dislocations [9].

In this work we present new investigations on the Y emission of epitaxial ZnSe/GaAs layers. The maximum of the Y line in ZnSe is compared with the recombination energy of donor and acceptor bound excitons for various strain conditions. Additional measurements using excitation spectroscopy give new information about the details of the recombination process. A steady state excitation causes a strong decrease of the Y emis-

sion intensity. However, warming of the samples up to RT recovers the emission intensity.

An analogous behaviour was observed for the Y lines in epitaxially grown ZnTe/GaAs layers which appear 205 and 240 meV below the gap energy  $E_0$  [10–12]. These Y lines in ZnTe/GaAs layers are also emitted from electron–hole recombinations at extended lattice defects [12]. Therefore, in this work special attention is paid to the comparison of the Y emission in epitaxially grown ZnTe/GaAs and ZnSe/GaAs layers.

## 2. Experimental procedure

All investigated epilayers were grown in our laboratory by atmospheric pressure metalorganic vapour phase epitaxy (MOVPE) and molecular beam epitaxy (MBE). GaAs(001) was used as substrate material. The morphology of the layers was controlled by Normarski interference microscopy. The thickness was determined by interference patterns and ellipsometric measurements.

For the MOVPE process dimethylzinc-triethylamine (DMZn-TEN) and di-tertiary-butylselenide (DtBSe) were used as metalorganic precursors for Zn and Se and tertiary-butylchlorine (tBCl) and triallylamine (TAN) were used for doping experiments. A growth rate of about  $1\text{ }\mu\text{m/h}$  was obtained at a susceptor temperature of  $340^\circ\text{C}$  and an input partial pressure ratio VI/II of 3. The GaAs substrates were prepared in a standard degrease, etched for one minute in 4:1  $\text{H}_2\text{SO}_4:\text{H}_2\text{O}$  and finally preheated 15 min at  $400^\circ\text{C}$  prior to the layer growth in a  $\text{H}_2$ -atmosphere in the reactor. For further investigations of the effects of annealing, the temperature was increased to  $500\text{--}550^\circ\text{C}$ , the annealing time was also 15 min. Further details of the MOVPE growth are published elsewhere [13,14].

For the MBE growth we used an ultra high vacuum chamber with Knudsen cells at a growth temperature of approximately  $300^\circ\text{C}$ . The GaAs substrates were prepared by a similar chemical etching procedure as used for the MOVPE growth. Prior to the growth the substrates were treated with a H-plasma inside the MBE chamber, which leads to oxide free and sharp inter-

faces. The H-plasma is produced in an RF-plasma source which is normally used for p-doping of ZnSe with nitrogen [15]. For all MBE-grown samples a two-dimensional growth mode was recorded by an in-situ RHEED system.

X-ray diffraction studies were carried out with a high resolution X-ray diffractometer (Philips-MRD). The X-ray beam from a Cu-tube is monochromated by four reflections of a Bartels monochromator at the (220) and ( $\bar{2}\bar{2}0$ ) faces of two perfect channel-cut germanium single crystals [2]. The resulting beam has a divergence  $\Delta\theta$  of 12 arcsec. The samples are mounted on a computer-controlled sample holder. A xenon proportional counting system is used as a detector.

PL measurements were performed in a liquid helium bath cryostat ( $T = 2$  K) using the UV-lines of an argon ion laser for excitation ( $\sim 3.5$  eV). The luminescence was analysed with a 1 m double monochromator, which was equipped with two gratings of 1200 lines/mm<sup>2</sup>, and detected with a cooled GaAs photomultiplier cathode. Photoluminescence excitation measurements (PLE) were performed with a CW dye laser, which operated with Stilben 3. The dye was pumped with the UV-lines of a 20 watt argon-ion laser. A birefringent filter at the dye laser allowed a continuous variation of the excitation wavelength. The luminescence measurements were always carried out with small laser intensities (10 mW/mm<sup>2</sup>) in order to avoid a quenching of the Y line through optical excitation with the exception of the measurements given in Fig. 8, which were carried out as described in the text.

The PL investigations described in the following section were carried out on MBE- and MOVPE-grown samples. For both growth methods very similar results are obtained. The impurities in undoped MOVPE ZnSe/GaAs are mainly Cl-atoms due to a pollution of the selenium source [3,14].

### 3. Results and Discussion

The strain of epitaxially grown ZnSe/GaAs layers at RT or 2 K contains the residual strain at the growth temperature,  $\epsilon_{\text{res}}$ , and the thermal

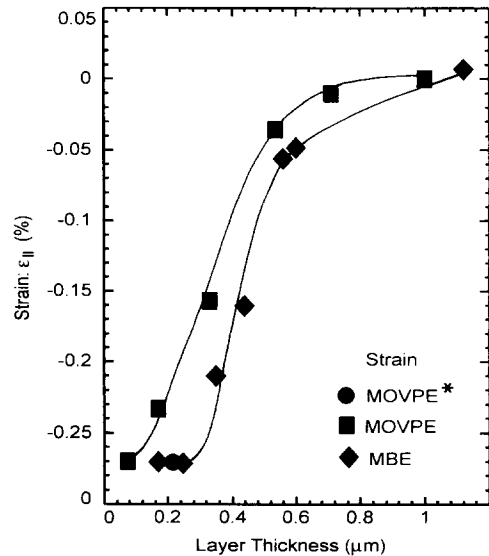


Fig. 1. Strain of ZnSe/GaAs layers for various layer thicknesses (MOVPE: annealing of the GaAs substrate at 400°C for 15 min; MOVPE\*: annealing of the GaAs substrate at 510°C for 15 min).

strain from different thermal expansion coefficients of ZnSe and GaAs ( $\epsilon_{\Delta T} = +0.04\%$  for  $\Delta T$ : 350°C  $\rightarrow$  20°C,  $\epsilon_{\Delta T} = +0.1\%$  for  $\Delta T$ : 350°C  $\rightarrow$  2 K). The strain at the growth temperature for layers with thicknesses  $d < h_c$  is determined by the lattice mismatch of substrate and layer material:  $f_{350^\circ\text{C}} = -0.31\%$ . Therefore, a constant strain is observed at RT of  $\epsilon_{||} = -0.27\%$ . For  $d > h_c$  the relaxation process starts by the formation of misfit dislocations during growth [1]. At a thickness of 0.5  $\mu\text{m}$  the strain is already largely reduced and 1  $\mu\text{m}$  thick layers are practically unstrained at RT.

Fig. 1 presents the strain of ZnSe/GaAs layers parallel to the interface ( $\epsilon_{||}$ ) at RT for various layer thicknesses (the lines represent guides to the eye). A value of  $h_c = 220 \pm 20$  nm is found for the critical thickness of MBE grown samples, whereas a value of  $h_c = 100 \pm 20$  nm is observed for MOVPE-grown layers, if the GaAs substrate is annealed at 400°C prior to growth. However, for an annealing temperature of 500–520°C the value for the critical thickness increases to  $h_c = 210 \pm 20$  nm.

Fig. 2 shows the FWHM of the (004) reflection, measured with a widely opened detector.

The full lines represent guides to the eye, the dashed line gives the halfwidths of ZnSe layers without lattice defects obtained by the formula [2]:

$$\Delta\theta = \frac{\lambda \sin \omega}{d \sin 2\theta}, \quad (4)$$

with  $\omega$  the angle of reflection and  $\theta$  the Bragg angle.

For  $d < h_c$  the FWHM values of the samples are fully determined by the layer thickness  $d$ . For  $d > h_c$  the created misfit dislocations cause a strong increase of the halfwidths. From Fig. 2 the values for the critical thickness of  $h_c = 220 \pm 20$  nm for MBE-grown samples and of  $h_c = 100 \pm 20$  nm for MOVPE-grown samples are obtained under the conditions that the GaAs substrate was annealed at 400°C prior to the growth. However, an annealing temperature of 500–520°C leads to an increase of the critical thickness which is similar to the value observed for MBE-grown samples (Fig. 3).

Fig. 4 shows PL spectra of MOVPE- and MBE-grown ZnSe/GaAs layers with various thicknesses for interband excitation ( $\sim 3.5$  eV) at

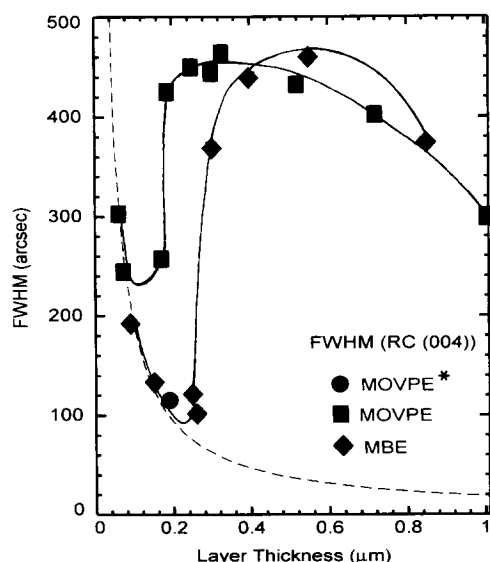


Fig. 2. FWHM values of (004) rocking curves for ZnSe/GaAs layers of various thicknesses (MOVPE: annealing of the GaAs substrate at 400°C for 15 min; MOVPE\*: annealing of the GaAs substrate at 510°C for 15 min).

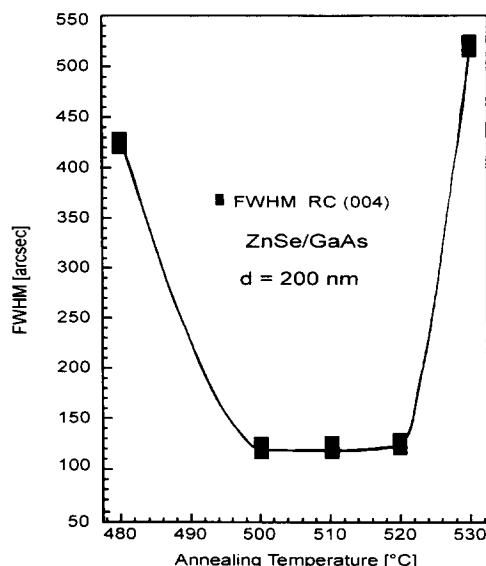


Fig. 3. MOVPE-grown ZnSe/GaAs layers (thickness: 0.2  $\mu\text{m}$ ): FWHM values of rocking curves ((004)-reflection) for various annealing temperatures of the GaAs substrates prior to the growth (annealing time: 15 min).

2 K. The spectra are dominated by strong free exciton (X) and bound exciton ( $I_2$ ,  $I_{1d}$ ) luminescence. The strain causes a splitting of the free exciton emission into a light hole ( $X_l$ ) and a heavy hole ( $X_h$ ) component and a shift of the hole spectra to higher energies for compressive strain and to lower energies for tensile strain. The luminescence signals  $I_2$  and  $I_{1d}$  are assigned to the recombination of donor and acceptor bound excitons respectively. The donors in MOVPE samples were identified as Cl-atoms introduced as impurities from the Se-source [3,14]. The deep acceptor, responsible for the  $I_{1d}$  line was attributed to a vacancy-acceptor-complex [16]. The bound excitons were identified as heavy hole excitons for compressive strain and as light hole excitons for tensile strain [3,17].

An unidentified shallow acceptor in MOVPE-grown layers with a ground-state binding-energy of about 80 meV leads to a weak DAP signal at 2.715 eV in these samples. The emission at 2.6 eV corresponds to the well known Y line in ZnSe. All investigated epilayers with  $d > h_c$  show this luminescence signal. This intensity in

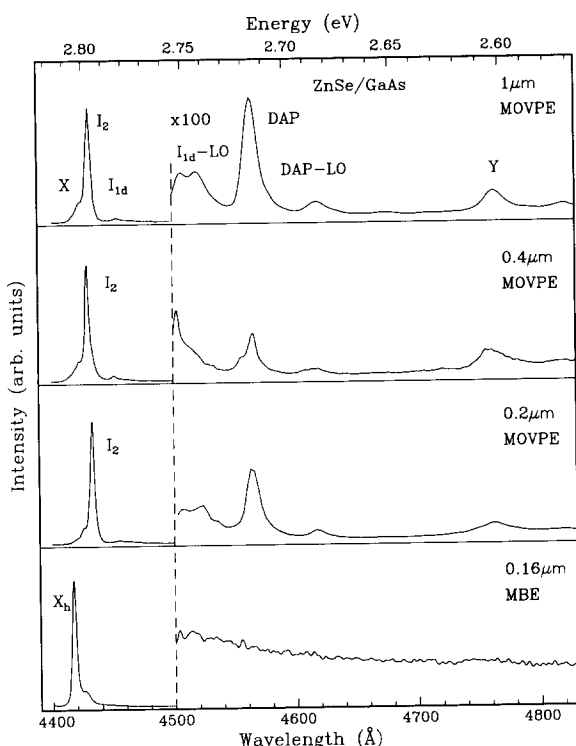


Fig. 4. Luminescence spectra of ZnSe/GaAs layers at 2 K with various thicknesses (MOVPE: annealing of the GaAs substrate at 400°C for 15 min).

MOVPE samples is limited by the impurity concentration in these layers as described in Section 3.3. However, for layer thicknesses  $d < h_c$ , as for the MBE ZnSe/GaAs sample with  $d = 160$  nm, the Y emission almost vanishes (Fig. 4), corresponding to a very small dislocation density (Fig. 2). This observation confirms the results of Myhajlenko et al. [9] that the luminescence signal is caused by dislocations created during epitaxial growth when plastic relaxation starts. The deep luminescence below 2.5 eV is very weak in these samples and confirms their high quality.

### 3.1. Investigations of the recombination process corresponding the Y line

Fig. 5 presents data obtained with photoluminescence excitation spectroscopy (PLE) from a 1  $\mu\text{m}$  thick ZnSe/GaAs layer grown by MOVPE. The monochromator was set on the maximum of the Y band (at 2.6 eV). The excitation energy of

the dye laser was varied between 2.85 and about 2.61 eV. An effective excitation only takes place at energies above the recombination energy of free excitons. The strain splitting of  $X_h$  and  $X_l$  excitons at 2 K for a 1  $\mu\text{m}$  thick sample is only 2 meV and therefore too small to be resolved in this measurement. The minimum at 2.800 eV corresponds to the exciton ground state and is due to a strong light absorption at free exciton resonance in a thin surface layer. Furthermore, the dislocation density is rather low in ZnSe/GaAs even in thick, relaxed layers ( $< 10^6 \text{ cm}^{-2}$ ). Therefore, most free excitons are trapped at impurities particularly at Cl-donors in MOVPE-grown samples as in Fig. 5, before they reach a dislocation line. An excitation of the Y line with photon energies smaller than the free exciton resonance is not possible. This result corresponds to the observation of the Y emission in ZnTe/GaAs [12] and gives further support to the interpretation as an electron–hole recombination at dislocation lines.

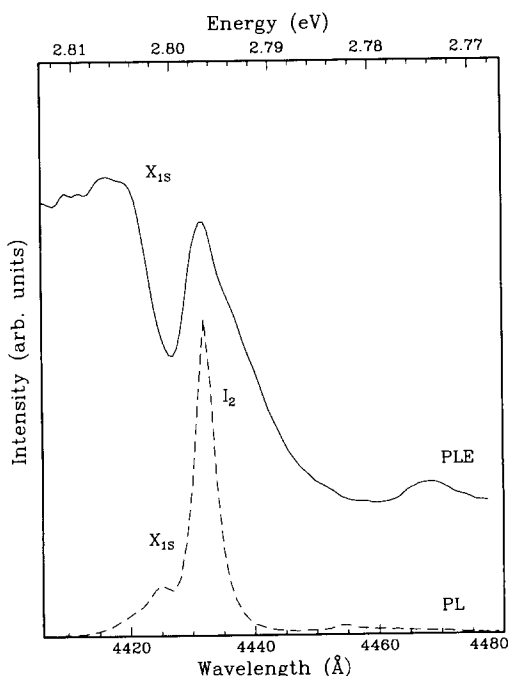


Fig. 5. Photoluminescence spectrum (PL) and photoluminescence excitation spectrum (PLE) for a ZnSe/GaAs layer of 1  $\mu\text{m}$  thickness at 2 K.

### 3.2. Dependence of recombination energy on the strain in the layer

A tensile strain of  $\varepsilon_{\parallel} = +0.15\%$  at 2 K in ZnTe/GaAs layers causes a shift of donor and acceptor bound excitons of  $4.5 \pm 0.5$  meV to lower energies compared to results from unstrained layers. A similar shift of  $4 \pm 1$  meV is observed for the  $Y_1$  and the  $Y_2$  line [3,12]. ZnSe/GaAs layers were investigated for strain values between  $\varepsilon_{\parallel} = -0.2\%$  at 2 K when  $d < h_c$  (no strain relaxation during growth) and  $\varepsilon_{\parallel} = +0.07\%$  at 2 K when  $d = 1 \mu\text{m}$  (strain relaxation during growth). The maxima of donor and acceptor bound exciton luminescence ( $I_2$  and  $I_1$ ) shift from  $2.801 \text{ eV} \pm 0.5$  meV to  $2.793 \text{ eV} \pm 0.5$  meV and from  $2.795 \text{ eV} \pm 0.5$  meV to  $2.788 \text{ eV} \pm 0.5$  meV respectively [3,17]. In comparison, the maximum of the Y line shifts from  $2.608 \text{ eV} \pm 1$  meV to  $2.601 \text{ eV} \pm 1$  meV when the strain changes from  $\varepsilon_{\parallel} = -0.2\%$  to  $\varepsilon_{\parallel} = +0.07\%$ . Thus, the strain shift of the Y emission in ZnSe/GaAs is similar to the strain shift of bound excitons, as it is observed in ZnTe/GaAs layers.

### 3.3. Dependence of the Y intensity on the impurity concentration

An increase of the impurity concentration in ZnSe/GaAs layers causes a strong decrease of the Y emission similar to that observed in ZnTe/GaAs [12]. In doped samples with a donor or acceptor concentration of more than  $10^{15} \text{ cm}^{-3}$  the Y emission vanishes completely as seen in Fig. 6.

### 3.4. Decrease of the Y intensity caused by optical excitation

An unusual property of the Y luminescence in ZnSe is the decay of the emission intensity during a steady-state laser excitation as shown in Fig. 7. This so-called photoluminescence “fatigue-effect” was already observed for the Y lines in ZnTe/GaAs [10] and for luminescence signals in materials with high defect densities as for example in the glassy semiconductors  $\text{As}_{1-x}\text{Se}_x$  [18,19], in amorphous  $\text{Ge}_x\text{Se}_{1-x}$  [20,21] and amorphous silicon [22–24].

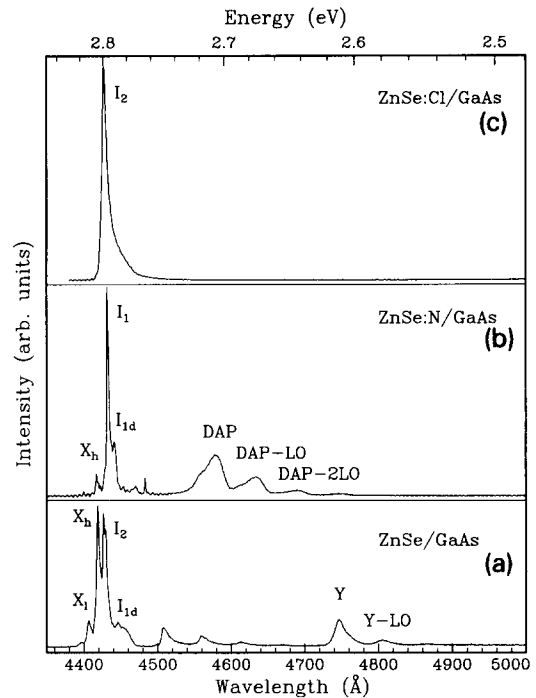


Fig. 6. Luminescence spectra for an undoped, a chlorine doped and a nitrogen doped MBE-grown ZnSe/GaAs layer with a thickness of  $0.3 \mu\text{m}$  at 2 K (acceptor concentration in (b): about  $10^{16} \text{ cm}^{-3}$ , donor concentration in (c): about  $10^{17} \text{ cm}^{-3}$ ).

The PL intensity of the Y emission in ZnSe and ZnTe decreases rapidly during the first seconds of laser excitation. This is followed by a slow decrease over 10 min, where the emission intensity reaches nearly a constant level. These measurements were carried out at 2 K because of the strong temperature dependence of the Y intensity [3].

A decrease of the luminescence during a steady state excitation as observed for the Y lines in ZnSe and ZnTe was already described by Redfield and Bube [24] for the PL intensity of semiconductors with a high density of lattice defects as amorphous silicon by the formula:

$$I_Y(t) = C \left[ N_S + (N_0 - N_S) e^{-Kt^\beta} \right], \quad (5)$$

$K$  and  $\beta$  are the characteristic parameters of the observed decrease of luminescence. The constants  $C$ ,  $N_S$  and  $N_0$  are determined by the intensities at the start and at the end of the

excitation ( $C$ : constant of normalisation;  $N_0$ : number of optical active defect centers at the beginning of the excitation;  $N_s$  number of optical active defect centers, after a constant level of luminescence is reached).

Fig. 7 presents the experimental results and a fit of the luminescence intensity on the excitation time with formula (5) for the  $Y_1$  and  $Y_2$  line in ZnTe and for the Y line in ZnSe. For the parameters  $K$  and  $\beta$  we found:  $K_{\text{ZnTe:Y}_1} = 0.026$ ;  $\beta_{\text{ZnTe:Y}_1} = 0.72$ ;  $K_{\text{ZnTe:Y}_2} = 0.66$ ;  $\beta_{\text{ZnTe:Y}_2} = 0.19$ ;  $K_{\text{ZnSe:Y}} = 0.36$ ;  $\beta_{\text{ZnSe:Y}} = 0.3$ .

A warming of the sample to RT leads to a recovery of the Y emission. The experiments presented in Fig. 8 were carried out as follows: First a luminescence measurement of a 1  $\mu\text{m}$  thick ZnSe/GaAs layer with a small laser intensity (10  $\text{mW}/\text{mm}^2$ ) was carried out as shown in Fig. 8a. Then the illuminated spot was excited with 100  $\text{mW}/\text{mm}^2$  for 15 min and the initial measurement with 10  $\text{mW}/\text{mm}^2$  was repeated (Fig. 8b).

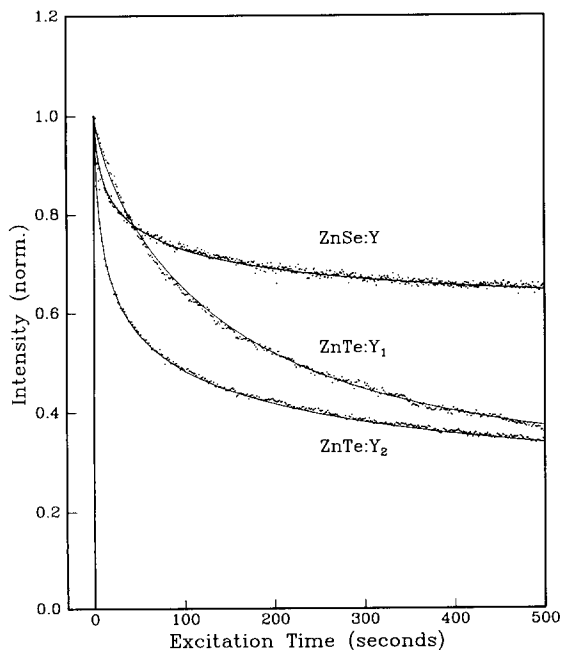


Fig. 7. Decrease ("fatigue-effect") of the  $Y_1$  and  $Y_2$  luminescence from a 1  $\mu\text{m}$  thick ZnTe/GaAs layer at 2 K and of the Y line from a 1  $\mu\text{m}$  thick ZnSe/GaAs layer at 2 K (excitation intensity of 50  $\text{mW}/\text{mm}^2$ ). The dotted lines are a fit with Eq. (5).

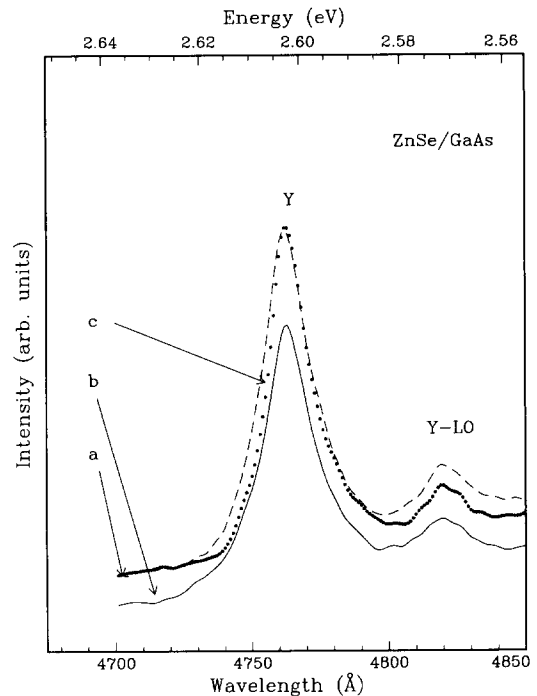


Fig. 8. Recovery of the Y emission of a 1  $\mu\text{m}$  thick ZnSe/GaAs layer after optical excitation (fatigue-effect). Spectrum (a): PI spectrum at 2 K and an excitation energy of 10  $\text{mW}/\text{mm}^2$ , spectrum (b): repetition after illumination with 100  $\text{mW}/\text{mm}^2$  for 15 min, spectrum (c): repetition after heating of the sample to RT (recovery effect).

The integral intensity of the Y emission decreased during the excitation with 100  $\text{mW}/\text{mm}^2$  compared to the initial intensity. After this procedure the sample was heated to RT and then again cooled to liquid helium temperature in order to repeat the luminescence measurement with 10  $\text{mW}/\text{mm}^2$  excitation energy (Fig. 8c). It can be seen, that the heating to RT led to a recovery of the Y emission to the original value, as it was observed for both Y lines in ZnTe [12]. Care was taken that always the same spot of the sample was illuminated in these measurements. During heating the sample was kept in darkness.

The observed effect can be attributed to the formation of meta-stable nonradiative centers as was figured out for the Y bands in ZnTe/GaAs. These centers are created by the absorbed light and lead to a disappearance of the radiative recombination. Finally the warming to room tem-

perature causes a recovery of the radiative recombination centers and therefore a recovery of the Y luminescence.

#### 4. Conclusion

The critical thickness  $h_c$  for the heteroepitaxial system ZnSe/GaAs depends strongly on the growth conditions and above all on the substrate preparation. A value of about 220 nm is found for MBE-grown samples (substrate preparation with H-plasma in the MBE chamber) and for MOVPE-grown layers, if the GaAs substrate is annealed to 500–520°C prior to the growth, whereas an annealing temperature of 400°C reduces  $h_c$  to about 100 nm. For thicknesses larger than  $h_c$ , plastic relaxation occurs by the formation of misfit dislocations.

These dislocations cause a luminescence signal at 2.6 eV, which is about 220 meV below the gap energy, called Y line. Its intensity decreases with increasing impurity concentration. A strong decrease is observed for samples which are doped with donors or acceptors as chlorine or nitrogen. The recombination process was studied with excitation spectroscopy. Excitation only occurs for photon energies higher than the recombination energy of free excitons. A strain dependent energy shift of the Y band is observed similar to the shift of bound excitons. Therefore, the Y line in ZnSe is associated with an excitonic recombination at extended lattice defects as was observed for the Y lines in ZnTe. A steady state illumination of the layers with laser light causes a decrease of the intensity of the Y emission. Annealing to RT leads to a recovery of the Y luminescence intensity similar to the effect observed for the Y lines in ZnTe/GaAs layers.

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