Annealing studies of high Ge composition Si/SiGe multilayers

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Abstract. For the design flexibility of SiGe based quantum cascade lasers high Ge composition Si/SiGe ($x = 80\%$) superlattices are important. We present an X-ray small angle scattering and high-resolution X-ray diffraction study on strain compensated Si/Si_{1-x}Ge_x multiple quantum well structures with Ge compositions (x up to 80%), grown on Si_{0.5}Ge_{0.5} pseudo-substrates. To test the temperature stability of such layers occurring in processing steps, in-situ annealing X-ray reflectivity measurements were performed for temperatures up to 810 °C. From the analysis of the reflectivity data, we obtain the layer thicknesses and the interface roughness of the superlattices during annealing. Using a one dimensional diffusion equation, the Ge diffusion coefficient for these conditions was obtained. Furthermore, the strain status and Ge composition in the superlattice structure and in the SiGe buffer before and after annealing were determined from the symmetrical and asymmetrical reciprocal space maps.

Introduction

Nanostructures based on SiGe technology have been intensively studied and have already been successfully used in electronic devices [1–2]. The use of Si technology has certain advantages compared to that for III–V materials. This technology could also have a strong impact on optoelectronic devices if e.g. Si-based quantum cascade lasers (QCL) would be realized. Such lasers were developed on the basis of InGaAs/InAlAs several years ago [3, 4]. However, to realize a QCL device based on Si and Ge is quite difficult, because the 4% lattice mismatch between Si and Ge produces lattice strain, which may cause interface roughness and undulations [5]. Consequently, the growth of SiGe based QCL structures needs highly stable growth conditions, low growth temperatures and a rather complicated design of the Ge contents and layer thicknesses of the quantum wells.

Previously, we have investigated the structural properties of pseudomorphic SiGe/Si electro-luminescent MBE grown cascade samples, which exhibited well-resolved electroluminescence in 10 μm wavelength range and exhibited quite a narrow line width of 22 meV [6]. The structural investigation of these samples was performed by X-ray scattering [7] and demonstrated a very good structural quality of the interfaces. The interface r.m.s. roughness had a maximum value of only 3 Å in the entire structure. Despite of the lattice mismatch between the SiGe(40%) and Si layers, the growth was pseudomorphic with respect to the Si(001) substrate.

To enhance the freedom for the design of SiGe based cascade structures, it was realized that higher Ge contents in the quantum wells are required. In this paper, we present results of the interface evolution during annealing in SiGe/Si structures with a max. Ge concentration of about 80%. We have investigated multiple quantum wells (MQW), since such structures are more appropriate for the investigation of interdiffusion than complicated cascade structures [8]. The investigated samples were measured both by X-ray diffraction (XRD) and by X-ray reflectivity (XRR). In-situ annealing experiments were performed. From the analysis of the X-ray reflectivity specular reflection curve, we determined the evolution of the interfaces during annealing, which allowed us to estimate the Ge diffusion coefficient in Si_{0.2}Ge_{0.8} MQW's.

Experimental

The pseudomorphic growth of a SiGe/Si multilayer stack on a Si substrate is limited by the critical thickness of the entire stack. If the critical layer thickness is reached, misfit dislocations are introduced. For the structure, containing Si_{0.2}Ge_{0.8} and Si layers, it is not possible to grow such MQW's directly on Si. These structures must be grown strain-symmetrized on a suitable Si_{0.5}Ge_{0.5} buffer [8, 9] to avoid the formation of misfit dislocations. In such a strain-symmetrized case, the total thickness of the multilayer can be quite large, since the excess strain is nearly zero. However, such highly strained Si/SiGe structures are metastable, and thus investigations of their properties at elevated temperatures are required.

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The investigated samples were grown on SiGe graded relaxed pseudo-buffers, with a Ge content in the top relaxed buffer layer of $x = 0.5$ ($\text{Si}_{0.5}\text{Ge}_{0.5}$). These graded buffers were deposited by a low-pressure chemical vapour deposition (LPCVD) technique, with a continuous grading of the Ge concentration, at ST Microelectronics. The multilayer structure was grown by molecular beam epitaxy (MBE) at a temperature of about 300 °C. The top of the MQW structure was finally covered by a system of graded layers grown in such a way as to preserve the strain symmetrization. The structure of the investigated samples (J036, J039, K027) is schematically shown in Fig. 1. All samples had very small surface misorientation (miscut) with respect to (001) crystallographic direction – less than 0.15°. They were grown on the $\text{Si}_{0.5}\text{Ge}_{0.5}$ pseudo-substrate with an additional buffer layer graded up to Ge contents of 80%, and terminated by a 3 nm thick constant $\text{Si}_{0.2}\text{Ge}_{0.8}$ composition layer. The multilayer structure of 30 periods ($d_2 - \text{Si}_{0.2}\text{Ge}_{0.8}$; $d_1 - \text{Si}$) followed which was covered first by a 5 nm Si layer, followed by the same graded layer system grown in reversed order. The whole sample structure was finally capped by 5 nm Si top layer. The differences between the samples J036 and J039 were their multilayer periods and thicknesses d_1 , d_2 ; the samples J036 and K027 were nominally the same, but the sample K027 included B doped layer as shown in Fig. 1.

The interface morphology in the MQW was investigated by X-ray reflectivity and the strain status of the samples was obtained from coplanar X-ray diffraction. We have measured XRR reciprocal space maps (RSM) and XRD maps in order to get information on the replication of the interface roughnesses together with the lateral and vertical correlation properties. Furthermore, the strain status of the superlattice structures was obtained from XRD. All the scattered intensities in the RSM's were recorded

Fig. 1. Nominal structure of MQW sample K027. The sample J036 has a similar structure as K027 but without B doping and sample J039 has only $d_1 = 3$ nm thick Si barriers and $d_2 = 5$ nm wide SiGe quantum wells.

by a position sensitive detector using a rotating anode facility with CuK_α radiation. Collimation and further monochromatization of the beam were done by a parabolic graded multilayer mirror and Ge channel-cut crystal.

The experiments at elevated temperatures, including XRR measurements during in-situ annealing, were performed on the beamline KMC2, at the Berlin synchrotron BESSY. The changes of interface morphology during annealing were investigated by specular reflectivity scans using a conventional scintillation detector with a linear slit. The annealing was performed under a vacuum of ($\approx 10^{-6}$ mbar) in a Be dome shaped vacuum chamber, which allowed for a sufficiently large range of incident and exit angles not only for XRR but even for diffraction experiments. The maximum temperature was slightly less than 810 °C, which was the read-out from a thermocouple mounted close to the sample. The temperature of the sample heater was controlled by a computer using the spec diffraction software.

The experimental data of specular reflectivity scans were fitted by Marquart-Levenberg least square method [10] with a model for the roughness, which increased with increasing layer thickness and was taken to be different at the Si/SiGe and SiGe/Si interfaces. This detailed analysis of specular reflectivity allowed us to model the electron density profile of the MQW structures. Diffraction RSM's were measured only ex-situ and analyzed using the positions of the intensity maxima in reciprocal space, without further theoretical simulations.

Results and discussion

From transmission electron microscopy (TEM) and atomic force microscopy (AFM) data follows that the structural quality of the interfaces of investigated Si/Si_{0.2}Ge_{0.8} samples is very good. The r.m.s. surface roughness from AFM was (4 ± 1) Å and also TEM cross sectional data showed a comparably small interface roughness. Moreover, resonant superlattice tunnelling was observed in these samples [11]. The high structural quality was confirmed by the present XRR investigations, as shown for sample K027 in Fig. 2.

X-ray specular reflectivity allows to estimate the thicknesses and electron densities of the layer in the investigated structures. The average periods of the multilayers can be obtained very precisely and for the samples J036, J039 and K027, we obtained (13.62 ± 0.04) nm, (8.21 ± 0.04) nm and (13.05 ± 0.16) nm, respectively. The sample K027 showed moreover a small linear deviation of the real period with respect to the average period, increasing from the buffer towards the top layer with about 0.01 nm per period. The average multilayer periods were obtained from the specular reflectivity fits, as will be described later. The interface r.m.s roughness was increasing within the interval (0.4–1.5) nm from the bottom to the top.

From the diffuse scattering in the reflectivity data, we have determined the vertical and lateral correlation properties using dynamical DWBA theory for X-ray scattering in multilayers [12]. The rather good vertical roughness corre-

Fig. 2. X-ray reflectivity map of multilayer sample K027. RDS – resonant diffuse scattering and Bragg like peaks prove the vertical correlation of the interfaces.

lation of the interfaces is evident from Fig. 2. From the width of the resonant diffuse scattering sheets (RDS) in Q_z direction we found that almost the whole multilayer stack was vertically correlated. The simulations of the diffuse scattering resulted in vertical correlation length $A_{\perp} = (230 \pm 50)$ nm for sample K027. The vertical correlation length for samples J036 and J039 was

(300 ± 50) nm and (200 ± 50) nm respectively. The direction of the oblique interface replication, obtained from the inclination of the RDS sheets was found to be only $\chi = (9 \pm 6)^\circ$, actually almost at the resolution limit and in the direction parallel to the miscut for all samples.

Due to the good quality of interfaces, we can observe also subsidiary maxima in Q_z direction (Kiesig fringes) in the diffuse scattering and also the Bragg-like peaks. The Bragg-like peaks are formed due to multiple reflections at the interfaces and can be simulated only with the fully dynamical approach in the XRR calculations. Their appearance proves that multiple scattering processes between interfaces play indeed a significant role.

From dynamical DWBA simulations [12] of the scans parallel to Q_x direction, see inset of Fig. 2, we obtained a lateral correlation length $A_{\parallel}$ of (2.5 ± 0.5) nm for sample K027 and a fractal scaling coefficient $h = 1$, see Ref. [13]. The subsidiary maxima close to the specular peak $Q_x = 0$ in the Q_x scans also showed that a long periodic waviness is likely to be present at the interfaces with a lateral period of about (10 ± 3) μm . This waviness is not included in the simulations due to the complexity of the model and thus the simulated diffuse scattering around $Q_x = 0$ has smaller values than the measured one.

We have also performed diffraction experiments on sample K027 to obtain the strain status of the multilayer structures with respect to the $\text{Si}_{0.5}\text{Ge}_{0.5}$ buffer and the Si substrate. The RSM's for symmetrical (004) diffraction and asymmetrical (224) diffraction are shown in Fig. 3.

In principle, three main maxima corresponding to certain Ge compositions in the entire SiGe layer sequence can be seen in the diffraction RSM's in Fig. 3.

1. two maxima correspond to two steps in the graded buffer

$$\circ \quad x_{\text{Ge}} = 0.33, \varepsilon_{\parallel} = 4 \cdot 10^{-4} \text{ and } R = 1.03.$$

$$\triangleright \quad x_{\text{Ge}} = 0.51, \varepsilon_{\parallel} = 12 \cdot 10^{-4} \text{ and } R = 1.06.$$

Fig. 3. Symmetrical (004) (left panel) and asymmetrical (224) diffraction maps (right panel) for sample K027. $\circ$ – 1st step in the graded buffer; $\triangleright$ – 2nd step in the graded buffer; $\diamond$ – $\text{Si}_{0.2}\text{Ge}_{0.8}$ layers in the MQW structure.

2. a maximum corresponding to the Si/SiGe MQW layers

$$\diamond x_{\text{Ge}} = 0.77, \varepsilon_{\parallel} = -112 \cdot 10^{-4} \text{ and } R = 0.64,$$

where x_{Ge} is the Ge composition, $\varepsilon_{\parallel} = \frac{a_{\parallel}^L - a_r^L}{a_r^L}$ denotes the lateral strain and $R = \frac{a_{\parallel}^L - a^S}{a_r^L - a^S}$ the degree of relaxation.

$a_{\parallel}^L$ denotes the lateral lattice parameter of the strained layer, a_r^L denotes the lattice parameter of the fully relaxed layer and a^S the lattice parameter of the Si substrate. To obtain the correct strain values of the layers, the possible lattice tilt must be taken into account. In our case, the lattice tilt of the layers in the buffer and of the layers in the MQW varied from 0° , close to the Si wafer, to 0.08° in the MQW with respect to the Si wafer. The above stated values are corrected for the lattice tilt.

We point out that in Fig. 3, the lateral width of the satellites of the multilayer peak is much larger than the width of the substrate peak. This indicates the presence of dislocations originating from the buffer. So far it is still not clear which part of the scattered intensities in the shape of broad satellites originates from misfit dislocations and which part from threading dislocations penetrating the multilayer structure. Due to the growth technique, one expects that the misfit dislocations are confined to the graded buffer and the MQW contains only the threading dislocations. Nevertheless, this matter will be the subject of further investigations.

A very important property of the investigated samples is their temperature stability. Thus, we have performed a series of in situ specular reflectivity measurements to get an estimate on the Ge interdiffusion at the $\text{Si}_{0.2}\text{Ge}_{0.8}/\text{Si}$ interfaces for an annealing temperature of about 810°C . Since the structural changes become very rapid after reaching this temperature, we had to perform short and fast specular scans (each of them not longer than 40 min).

The sample K027 used for the in-situ annealing experiment was preannealed at a temperature of 550°C for 10 min in nitrogen atmosphere ex-situ. The MQW structure evidently did not change. We have continued annealing of this sample at a temperature $T = 810^\circ\text{C}$ in vacuum. The time evolution of the specular reflectivity of the sample K027 is depicted in Fig. 4 (upper panel) for temporal periods from 40 min to 290 min.

We have used at least four annealing times for the evolution of the layer electron density and could fit the logarithmic decrease of the electron density contrast. The electron density profile was obtained from the specular scans using the procedure as outlined below.

To obtain the sample structure and the electron density profile, it is necessary to assume a proper model and fit its parameters to the experimental data. The theory used for data analysis of specular reflectivity scans is based on the matrix optical formalism [14] with modified Fresnel coefficients for ideal interfaces; multiplied by a Debey-Waller factor $r_j \exp(-2\sigma_j k_{zj} k_{zj+1})$, see Ref. [15], where σ_j is the roughness of j -th interface and k_{zj} are z components of the wave vectors in the layers. The roughness profile at each interface was assumed with a maximum replication and non-zero intrinsic roughness. We have assumed different and independent roughness profiles at the Si/SiGe and

Fig. 4. Evolution of the specular reflectivity together with its simulation (upper panel), the relative electron density obtained from the simulation of specular reflectivity in the MQW sample K027 during in-situ annealing at temperature of about 810°C (central panel), Arrhenius plot of the electron density contrast $\Delta\rho$ during annealing, from which the diffusion coefficient D is estimated for the multilayer with a given period (lower panel).

SiGe/Si interfaces in the MQW and independent roughness profiles in the graded regions, below and above the MQW. This means four different roughness profiles, each given by the equation $\sigma(z) = \sqrt{\sigma_n^2 + (\sigma_0^2 - \sigma_n^2) \frac{z}{D_0}}$, where $\sigma_{0,n}$

denote the roughnesses at the lower and upper end of the multilayer. D_0 denotes the distance between boundary interfaces and z is the distance from the lower boundary interface [16]. Such a model for the roughness profile together with layer thicknesses and their Ge contents using the nominal parameters as an input, see Fig. 1, was used for the simulation.

The results of the specular reflectivity simulations for the different annealing times are shown together with the measurements in Fig. 4 (upper panel). The corresponding electron density profiles obtained from the resulting reflectivity fits are plotted in Fig. 4 (central panel). The electron density profile is plotted relatively to the buffer, e.g. normalized to the electron density of $\text{Si}_{0.5}\text{Ge}_{0.5}$. One notices that even from the reflectivity curve taken towards the end of the annealing procedure (300 mins), it was possible to estimate roughly the residual periodic profile of the electron density and an almost total disappearance of the MQW.

From each electron density profile, corresponding to a certain state during annealing, we obtained the electron density contrast $\Delta\rho$ between layers in the MQW, defined as the difference of the electron densities between neighboring $\text{Si}_{1-x}\text{Ge}_x$ and Si layers, normalized to the electron density of the substrate. $\Delta\rho$ was plotted in a log scale in Fig. 4 (lower panel) and we can see from the logarithmic fit that the $\Delta\rho(t)$ dependence fulfills an Arrhenius dependence.

Using the results of Fig. 4 (lower panel), we assume that during annealing interdiffusion occurs and we thus estimate an interdiffusion coefficient between Si and SiGe with 80% of Ge. For the sake of simplicity, we assume an infinite periodic structure with a sinusoidal profile of the electron density at the beginning of annealing. This assumption is only a rough approximation. Using the first

Fourier coefficient of electron density profile $\Delta\rho(z, t) \approx \Delta\rho(t) \exp(2\pi iz/\text{period})$ in the diffusion equation, we obtain for the amplitude of oscillations the following dependence $\Delta\rho(t) \sim \exp(-4D\pi^2/\text{period}^2 t)$, where D is the diffusion coefficient and “period” denotes the multilayer period. From the results of Fig. 4, we obtain a value for diffusion coefficient for Ge in Si $D = (0.15 \pm 0.05) \cdot 10^{-20} \text{ m}^2/\text{s}$ at 810°C .

From the specular reflectivity measurements in Fig. 4, it is also evident that the period of the MQW structure is preserved during the entire annealing time and only the ratio of Si and SiGe layer thicknesses is slightly changing. The SiGe layer thicknesses increase from 9.1 nm to 11.5 nm, thus Si diffuses into SiGe, as observed also in [17]. Since the interdiffusion of Si and Ge is described by Fick’s law, the diffusion coefficient must fulfill the temperature dependence $D(T) = D_0 \exp(-E_a/k_B T)$. Using the value of $D_0 = 0.2 \text{ cm}^2/\text{s}$ from Ref. [18], we obtain for the activation energy $E_a = (3.4 \pm 0.2) \text{ eV}$ for a Ge content of $x = 0.8$. This is in good agreement with the previous results, which give the value for the activation energy E_a of 3.1 eV for $x > 0.3$ for SiGe, see Ref. [18].

If we compare our value for the diffusion coefficient with results by Csik et al. [17] for amorphous SiGe ($x = 0.25$), where for $D \approx 8 \cdot 10^{-18} \text{ m}^2/\text{s}$ was found, we see that apparently the values for crystalline materials are much smaller.

From the diffraction data, we found that the periodic modulation due to the MQW structure completely disappeared after annealing for almost 5 hours at 810°C . The diffracted intensities are particularly sensitive to the strain status of the MQW. Consequently, we compare the diffraction reciprocal space maps before and after annealing for symmetrical (004) and asymmetrical (224) diffraction. The RSM’s of sample K027 are shown before annealing

Fig. 5. Symmetrical diffraction map (004) (left panel) and asymmetrical diffraction map (224) (right panel) for sample K027 after annealing. $\circ$ – 1st step in the graded buffer; $\triangleright$ – 2nd step in the graded buffer.

Fig. 3, and after annealing in Fig. 5. We find that the lattice parameters of the graded layers in the buffer did not change since the corresponding peak positions stayed constant. However, the multilayer structure is strongly affected.

Conclusion

The structural investigation of $\text{Si}_{0.2}\text{Ge}_{0.8}/\text{Si}$ multiquantum-well samples before and during thermal annealing show that close to a temperature 810°C , the multilayer experiences strong interdiffusion. Nevertheless, the period of the multilayer is still clearly observable even after about 3 hours at 810°C as a small undulation of the electron density. From the X-ray diffraction data we conclude that the strain status of the graded buffer layer is nearly preserved after this annealing step, whereas no observable strain modulation due to the MQW structure remains.

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