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REVEALING THE STRUCTURAL DISTURBANCES IN CZOCHRALSKI SILICON BY HIGH TEMPERATURE - PRESSURE TREATMENT

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Single crystalline silicon grown by Czochralski method (Cz-Si) is typically considered to be free of extended defects; no dislocations are usually revealed in Cz-Si by X-ray (synchrotron) topography. Cz-Si contains oxygen in interstitial positions, clustering and precipitating at annealing, mostly on structural inhomogeneities. Oxygen precipitation occurs during prolonged annealing at 900 – 1000°C while short time one-step processing at ≥ 1100°C exerts practically no effect on oxygen clustering. It is known, however, that the treatment under high hydrostatic pressure (HP) promotes oxygen precipitation on structural inhomogeneities even at ~ 1130°C if done for prolonged time (*e.g.* for 5 h [1]).

The purpose of present work is to reveal the oxygen precipitation sites and so the initially existing disturbances in Cz-Si by short time treatment at 1127°C under HP.

The (001) oriented Cz-Si samples of about 0.6 mm thickness with various initial oxygen concentrations (8×10^{17} cm^{-3}, 9×10^{17} cm^{-3} and 11×10^{17} cm^{-3}) were treated for 2 h at 1127°C under 10^5 Pa, 10^7 Pa and 1.1 GPa. The defect structure of treated samples was determined using a high-resolution X-ray diffractometer in the double and triple axis configurations and X-ray synchrotron topography (this last done at ESRF at the ID19 synchrotron beamline). The defects characteristics were also estimated from X-ray diffuse scattering in the Huang region [2,3]; the intensity of this scattering arises from the long–range displacement fields associated with various defects.

The HT–HP treatment of as-grown Cz-Si under 1.1 GPa results in enhanced oxygen precipitation, mostly at the initially existing structural disturbances. The 004 reflection synchrotron topographs of the Cz-Si samples are shown in Figure 1. Annealing under atmospheric pressure does not affect the sample defect structure while the treatment under high hydrostatic pressure results in revealed structural disturbances.

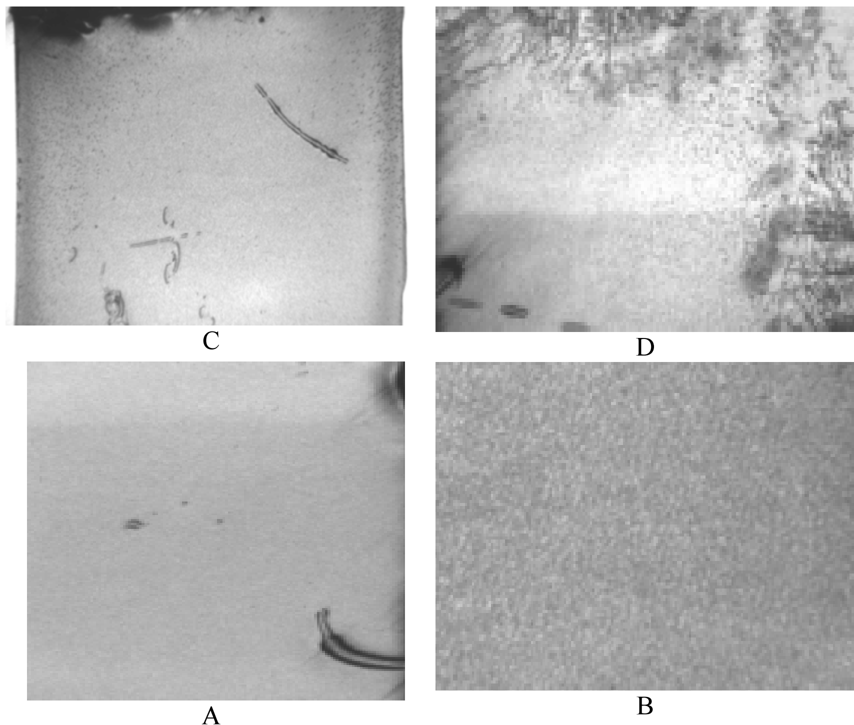

Figure 1.

Synchrotron X-ray topographs of Cz-Si with primary c_o = 11×10^{17} cm^{-3} (A and B) and 8×10^{17} cm^{-3} (C and D), treated for 2 h under 10^7 Pa (A and C) and 1.1 GPa (B and D).

These disturbances are related to the presence of primary inhomogeneities and depend on the interstitial oxygen content, c_o. The correlation between visualized defects (seen on the X-ray topographs) and reciprocal space maps is observed. For the samples treated under 10^7 Pa, no contrast coming from oxygen precipitates or dislocations has been found (Figures 1A, 1C) while the treatment under 1.1 GPa resulted in a creation of small precipitates in high density (Figures 1B, 1D). This effect is more pronounced for a sample with higher initial c_o; dislocations detected in Fig. 1D originated from the cut process. The creation of oxide clusters is accompanied with decreasing of the oxygen concentration. For example, for the sample with initial $c_o = 11\times10^{17}$ cm^{-3} the c_o value decreased to 8.58×10^{17} cm^{-3} and 7.36×10^{17} cm^{-3}, respectively, after the treatments under 10^7 Pa and 1.1 GPa. The sources of the observed effects will be discussed.

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THE EVOLUTION OF GaMnAs DEFECT STRUCTURE AFTER HIGH PRESSURE TREATMENT

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A lot of effort has been concentrated in recent years on investigations of ferromagnetic semiconductors, due to their potential application for spintronic devices [1]. Ferromagnetic GaMnAs is one of the materials interesting from this point of view. Since it's discovery in 1996 by Ohno *et al.* [2] many groups began to investigate the GaMnAs properties, however, some very basic problems concerning GaMnAs still remain to be unsolved. One of these is the GaMnAs lattice constant (a_{GaMnAs}) dependence on the Mn content. It is more or less accepted that a_{GaMnAs} follows the Vegard's law and increases proportionally to the Mn content, up to the maximal concentration of about 10% of Mn [3], but even this statement is still in doubts [4, 5]. Different investigators report different rates of a_{GaMnAs} increase with the Mn content (see, *e.g*., Ref. [6]). By careful X-ray diffraction measurements of several hundreds of GaMnAs samples grown by Molecular Beam Epitaxy (MBE) at different conditions we have found that these differences are due to the presence of defects, namely of arsenic antisites (As[Ga]) and manganese interstitials (Mn_I), in GaMnAs. The aim of present paper is to establish the influence of the annealing under high pressure on defects structure in GaMnAs/GaAs system.

The 0.8-2 μm thick GaMnAs layers were grown by the MBE method at 212–270ºC. The concentration of Mn was kept between 2.5% to 6%. Next the samples were treated at 275ºC (T) under high Ar hydrostatic pressure (HP, equal to 1.1 GPa) for 3 h.

The total Mn concentrations for the untreated and HP-T treated GaMnAs samples were determined by Secondary Ions Mass Spectrometry (SIMS). The in-plane and out-of plane lattice parameters of GaMnAs were measured using high resolution X-Ray diffractometer. Reciprocal space maps were made for all samples. For thin layers ($t < 1$ μm) the layer thickness was calculated from the interference fringes. Bragg reflection topographs were made for all samples at the ID19 synchrotron beamline (ESRF, Grenoble).

The GaMnAs layer remained fully strained in relation to the substrate, both before and after the treatment; the in-plane lattice parameter remained the same as that of GaAs. The FWHM (rocking curve width at half maximum) value remained the same before and after the treatment. No misfit dislocation before and after treatment was detected on X-ray topography. The thickness of layer as well as the lattice constant of GaAs substrate remained unchanged before and after the treatment. Decrease of the out–of-plane lattice parameter of layers was detected for all samples; it was dependent on the growth conditions. For example, for the $Ga_{0.9}Mn_{0.1}As$ layer, the lattice parameter was even equal to that of the substrate. Contraction of the lattice parameter can be related to the decreased concentration of interstitial Mn atoms and/or of arsenic antisites. The contribution of arsenic antisites and of Mn atoms, both substitutional and interstitial, to the a_{GaMnAs} value [5], is given by the formula:

$$a_{GaMnAs}(x,y,z) = a_0 + 0.02x + 0.69y + 1.05z \qquad (1)$$

where: a_0 - lattice constant of defects-free GaAs, x – concentration of Mn in the Ga position, y - concentration of As antisites, z – concentration of Mn in the interstitial positions.

Due to low annealing temperature, as well, we can assume that the concentration of antisites in the layer remained constant [7]. It means that the treatment under high pressure removes of Mn interstitial from the lattice, but still it is not clear what happens to the Mn atoms removed from the interstitial positions. Though it has been shown before by several groups (see, *e.g.*, Refs [8-10]) that the post growth annealing under atmospheric pressure reduced the concentration of Mn interstitials, never the effect of their reduction was such effectively. It is very important problem because interstitial Mn atoms reduce the hole concentration in the layers what, in turn, can results in lowered Curie temperature [11].

Conclusions

Post growth annealing of GaMnAs under high pressure leads to the lattice constant contraction, more effectively than annealing under atmospheric pressure. The decreased lattice constants evidence the removal of interstitial Mn atoms from the GaMnAs lattice.

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X-RAY DIFFRACTION STUDIES OF MULTILAYERED SELF-ASSEMBLED ULTRA-SMALL Ge QUANTUM DOTS

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Silicon-based heterostructures like Si/Ge/Si have attracted a great of attention in the last years, since they offer very interesting possibilities for microelectronics and optoelectronics devices. Especially, the realization of quantum wires and quantum dots Si-based structures promises achieving of superior electronic and optoelectronic properties.

A large lattice mismatch in Ge/Si heterostructure presents an interesting approach for the fabrication of self-assembled GeSi three-dimensional (3D) islands (Stranski-Krastanow growth). Such islands (quantum dots) are formed under suitable growth conditions: elastically strained Ge layers relax via formation of strained Ge islands. This process is realized in two steps: in the first one 3D Ge islands are formed just after exceeding the critical thickness (about 4-6 monolayers of Ge) – these islands (called “hut”-clusters) are shaped as tetrahedral pyramids with side orientation of {105}-type. There are no misfit dislocations in such “hut”- clusters (the strain relaxation in them is realized via partial elastic deformation). In the next step, for thicker Ge layers, bigger relaxed Ge islands are formed with the side orientation of {113}-type (so-called “dome”, or macro-islands) [1]. The strain relaxation inside the “dome” islands is realized by creation of misfit dislocation network. From the application point of view the “hut”-clusters are preferred in the heterostructure with the self-organized quantum dots due to absence of misfit dislocation and the smaller sizes (a plane size of “hut” dot is of about 10 nm and a height of 1.5 nm) [1].

In order to achieve a high density of dots, what is very important from the technological point of view, dot multilayers were grown. In such a multilayer thin Ge layer containing Ge dots is overgrown with much more thicker Si spacer layer, and next, this sequence is repeated several times. Such structure is named *superlattice* due to appearance of periodicity in the growth direction – this period is equal to the thickness of two repeating layers.

The structure and properties of this kind of superlattices have been studied by several methods, including atomic force microscopy (AFM), transmission electron microscopy (TEM), photoluminescence (PL), Raman scattering, X-ray absorption spectroscopy (XANES and EXAFS), X-ray diffraction and reflectometry. However, the results of structural characterization of self-assembled dot structures by X-ray diffraction methods are rare and limited in the literature, especially for ultra-small Ge quantum dots.

In this work we attempted to apply the X-ray synchrotron radiation for study of self-assembled ultra-small Ge dots inside Si/Ge superlattice. This superlattice nominally consisting seven times repeated Ge(1 nm)/Si(30 nm), was grown at low temperature (250°C) on Si (001) substrate covered by Si(115 nm, 780°C)/Si(10 nm, 450°C) buffers.

To confirm the formation of quantum dots (QDs) in this sample photoluminescence measurements were performed at 8 K using an Ar^+ laser at a wavelength of 514.5 nm. The liquid nitrogen-cooled Ge detector was used for spectra collection. The dominant feature detected in the spectra (Fig. 1) is the luminescence band centered around 0.77 eV, which is commonly assigned to the Ge QDs photoluminescence [2].

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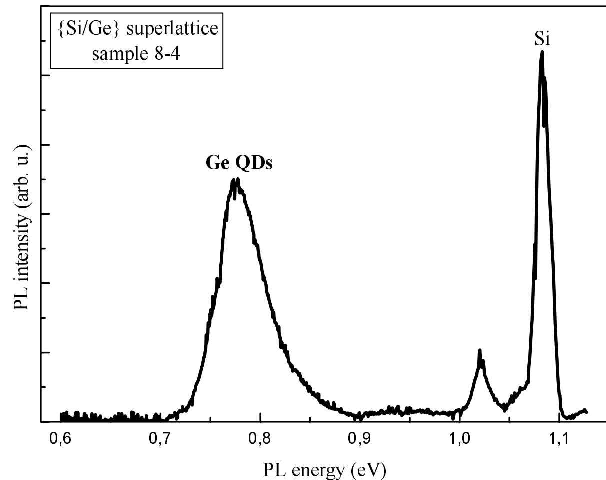

Figure 1.

The X-ray diffraction measurements were recorded at the W1.1 wiggler beamline in Hasylab (Hamburg). The monochromator was set to 8048 eV (λ = 0.154051 nm). The sample was aligned at (001) planes of Si substrate and next, a 2θ-ω scan was performed in a wide range of 2θ angles. The obtained diffraction pattern (see Fig. 2) will be discussed.

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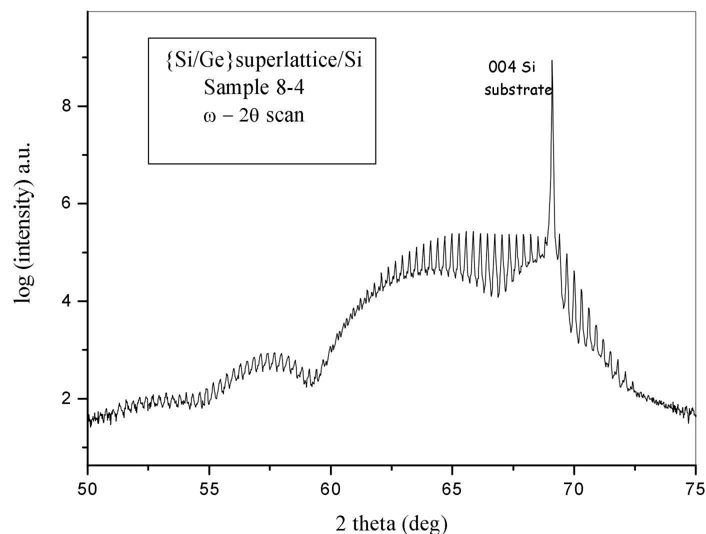

Figure 2.