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Grazing-incidence X-ray reflectometry for structural characterization of samples containing Ge/Si quantum dots

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Abstract. High-resolution grazing-angle X-ray reflectometry measurements have yielded experimental and theoretical intensities of specular and diffuse reflection from MBE grown structures with single-layer unburied and multi-layer buried Ge/Si quantum dots (QD). The face slopes measured with a high precision (±0.1°) from the position of diffuse scattering peaks in direct space have supported the validity of the known model of QD formation within wells with a structure of inverted pyramids with {11n}, $n = 7\dots 11$ faces.

Introduction

While transmission electron microscopy, atomic force microscopy (AFM) and near-field scanning optical microscopy do enjoy wide use in analysis of nanosized objects, their application in comprehensive structural studies of quantum dot (QD) systems meets with difficulties. X radiation finds increasing recognition as an integral nondestructive probe for studying nanosized multilayered structures, including atomic-scale roughness of interfaces and interdiffusion. The methods of X-ray diffractometry and reflectometry based on analysis of the specular and diffuse X-ray scattering components in both reciprocal and direct spaces provide rich information for analysis of QD heterostructures.

A recent high-resolution grazing-angle X-ray reflectometry (HRXRR) study has analyzed X-ray scattering from samples with multiple QD ensembles MBE-grown in the In(Ga)As/GaAs system [1]. The position of the experimentally observed diffuse-scattering intensity peaks was found to be totally determined by the slope angle α of the QD pyramid faces (the so-called diffraction grating blaze condition), which had been theoretically predicted earlier [2]. A comparison with the results of numerical modelling of scattering based on the boundary integral equation method suggests that a straightforward geometric condition $2\alpha = \theta_{inc} \pm \theta_{dif}$ permits one to accurately derive α from the position of the intensity peak whose shape depends on many parameters [3]. Besides, the position and amplitude of the Bragg peaks can be employed to deduce the roughness/interdiffusion of an interface and the QD height.

We are reporting here on characterization of MBE-grown structures with one-layer unburied and multilayered buried Ge/Si QDs with the use of experimental and theoretical data on the specular and diffuse scattering intensity.

1. Experimental and numerical

Samples with Ge quantum dots were MBE grown on the vicinal Si(001) surface on a BALZERS UMS 500P setup [4]. Single-layer #1 and #2 samples with Ge QDs without capping layer were produced by Ge deposition at 700°C on a stressed SiGe layer with 10 and 20% Ge content which was grown on a 100-nm thick Si buffer. A multilayered #3 sample with dome QDs and 20 Ge/Si superlattice layers with a period of 30 nm was grown at 650°C on a 50-nm thick Si buffer layer and had the same capping layer. A multilayered sample #4 with pyramid and hut QDs and 20 layers of Ge/Si superlattice with a period of 11.7 nm was grown at 550°C on a 50-nm thick Si buffer layer and had the same capping layer.

Surface morphology of the substrates and grown structures was studied with AFM. This method was used to investigate the size, shape and surface density of the self-organizing nanoislands, as well as to quantify surface roughness of the substrates and Si buffer layers. The AFM measurements were performed *ex situ* in air, in semi-contact mode on Solver PRO and Nanoscope III microscopes.

The specular and non-specular scattering measurements by HRXRR were conducted on a Philips Expert Pro reflectometer with a four-crystal Ge monochromator in $\theta/2\theta$ rocking curve scanning mode, and in θ and 2θ modes when studying diffuse scattering indicatrix. The measurements were performed at a wavelength $\lambda(\mathrm{CuK}_{\alpha 1}) = 0.154$ nm. The detector was a gas-discharge counter with an extremely low intrinsic noise, of the order of 0.1 quantum/s, with an entrance slit adjustable in width [1]. The distance to the sample was 20 mm, the slit width at the crystal monochromator was $100\mu m$ to obtain a strong signal in measurements of scattered light, slit height was 1-5 mm, and the scan step was chosen in the 0.001-0.005° interval, depending on the desired resolution for an angular beam divergence of 0.003°. The detector slit could be varied from 0.1 to 3 mm.

The calculations based on rigorous electromagnetic theory were performed using a modified method of boundary integral equations (MIM) [5], which turned out to provide high accuracy and fast convergence at large ratios of the characteristic period D and height h of QDs to wavelength λ [6]. The error of the calculations estimated from the energy balance was $\sim 1.E{-}6$ for 400-1600 collocation points at each boundary of the modeled structures. The time taken up by calculations of a scattering intensity curve with one statistical set of parameters on a workstation with two Quad-Core Intel® Xeon® 2.66 GHz processors, 8 MB L2 Cache, 1333 MHz Bus Clock and 16 GB RAM is ∼ 2 min when operating on Windows Vista® Ultimate 64-bit and employing eightfold paralleling.

2. Results and discussions

The QD face slopes measured with a high precision (±0.1°), primarily along the [110] and [1-10] directions from the position of the diffuse scattering peaks in direct space have yielded supportive evidence for the validity of the known model of QD formation inside wells having an inverted pyramid structure with {11n}, $n = 7\ldots11$ faces, as well as proved the possible existence of similar faces in dome QDs [7, 8]. We observed these reflections reliably both in single-layer samples and in multilayered samples with a capping layer, structures which can be identified with those of the pyramid and hut and also the dome QDs. Inverted pyramids in the base of the QDs of the samples under study were observed also by AFM.

We have observed and studied a small deviation (by a few degrees) of central normals to the reflecting faces from the [110] and [1-10] directions, which affects noticeably the peak scattering intensity variation. The experimental HRXRR studies of the wells and QD faces have revealed that (1) long-range order in the distribution of self-organizing QDs in different samples grown on vicinal (001) substrates with a Si buffer layer is practically absent in the [100] or [010] directions, and (2) the corresponding faces may be not flat [9]. The AFM data were used to refine the MIM-based model to include generation of reasonable boundary profiles and to optimize the number of points to be arranged.

Typical experimental and theoretical curves of diffuse scattering intensity obtained with the use of HRXRR are shown graphically in Figs. 1 and 2 for the #2 structure. The position and shape of the main peaks in both graphs correlate well; to compare their amplitudes, however, one should reduce the three-dimensional scattering problem to the two-dimensional one [1]. Thus, the traditional use of HRXRR in determination of the layer parameters and of boundary imperfection has been extended in the method considered to include the geometry of QDs epitaxially grown in different systems. To eliminate the effect of factor (1), one should grow QDs with periodic masks, and to reduce the impact of (2), the temperature of deposition of the capping layer should be reduced.

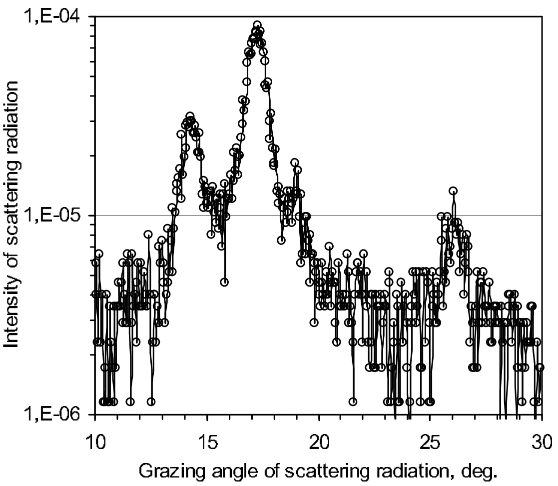

Fig. 1. Measured non-specular reflectance of #2 for 0.154-nm wavelength and 0.304° grazing incidence vs. grazing diffraction angle with +5° deviation from [110] or [1-10].

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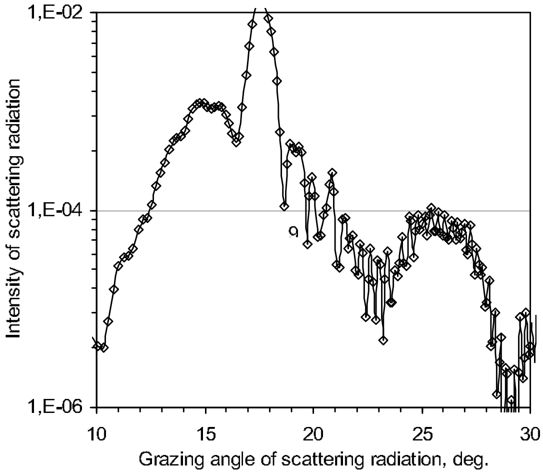

Fig. 2. Calculated diffuse reflectance of #2 for 0.154-nm wavelength and 0.304° grazing incidence vs. grazing diffraction angle.

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