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Complementary application of Raman scattering and GISAXS in characterization of embedded semiconductor QDs

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Abstract

Here we report the comparative analysis of Raman scattering and Grazing incidence small angle scattering of X-rays (GISAXS) applied on the systems of Ge and II–VI compound quantum dots (QDs) embedded in dielectric matrix. In Raman scattering, the synthesis of QDs, their phase and composition, as well as crystal quality, were determined by analyzing the material-related characteristic optical phonon modes. Additionally, from the analysis of the low frequency vibrational Raman band, the size and size distribution of nanoparticles were calculated. GISAXS was applied to study the synthesis of QDs through a proper analysis and modeling of the corresponding 2D spectra. The QD sizes were determined by a Guinier-plot analysis, whereas the Local Monodisperse Approximation (LMA) was used for the analysis of the shape and the size as well as size distribution. We have demonstrated that Raman and GISAXS give complementary results indispensable for the complete characterization of such systems. The results are to be used for further improvements of semiconductor QD preparation.

Keywords: Nanoparticles; QD; Ge; II–VI; CdS; CdSe; Implantation; GISAXS; Raman scattering

1. Introduction

Systems of small dimensions (nanocrystals or quantum dots, QDs) exhibit considerably different optical and electronic properties from their bulk counterparts due to the size-induced

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quantum confinement of free carriers. Nanometric size generates tremendous changes in electronic, optical and other properties of QDs, offering a number of potential applications in semiconductor and other industries. Their most interesting peculiarity, which stimulates their applications in very distant research areas, is the possibility of a precise tuning of their absorption and emission spectra by adjusting their size [1–3].

Due to a large surface to volume ratio, QDs are chemically reactive and have to be embedded into an appropriate host material. One of the best methods to produce well-defined buried layers with a high-volume fraction of nanocrystals is a high dose implantation [4]. However, the optimization of implantation parameters and post-implant procedure, in order to achieve proper size and size distribution in implantation-produced QDs appears to be the most challenging task for this method [2]. In that sense, appropriate characterization methods have to be applied to establish the effective correlation between functionality and production parameters. Here we demonstrate how the two widely used characterization methods, scattering of X-rays under grazing incidence, GISAXS, and Raman scattering, could provide complementary information on QD characterization.

2. Experimental

Three sets of implanted samples were studied. First set was obtained from Turan [5], where 100 keV $^{74}Ge^{+}$ ions were implanted into a 250 nm thick SiO_2 amorphous layer, grown on the (100) Si substrate by wet oxidation. The maximum of the implanted Ge ions distribution was at 75 nm [6]. Samples with doses of 1×10^{17} cm^{-2} were annealed for 1 h in N_2 atmosphere, at annealing temperatures (T_a) ranging from room temperature (RT) (not annealed) to 800 °C.

Second set was provided by White [7] and it comprised samples of amorphous SiO_2 substrate, about 1 mm thick, implanted with equal dose of 10^{17} cm^{-2} Cd and S ions of 320 keV and 115 keV, respectively. This resulted in a Gaussian depth density distribution of the dopants, with a peak volume concentration of about 6.3×10^{21} cm^{-3} of each Cd and S atoms at ∼130 nm depth, as determined by Rutherford Back Scattering [7]. The samples were subsequently annealed for one hour in vacuum, at selected annealing temperatures, from RT to 800 °C.

A third set of samples was produced at Universitaet Augsburg [8] by implanting ^{112}Cd and ^{80}Se ions into a 0.5 μm thick layer of thermally grown SiO_2 on (100)-silicon at the liquid nitrogen (LN) temperature. The implantation energy was 190 keV for Cd and 134 keV for Se, selected as to obtain the overlapping implantation profile for both elements and a projected range of approximately 80 nm. The implanted dose of Se was 4.79×10^{16} cm^{-2} whereas the dose of Cd was varied so as to achieve the series of under- (from −9%) or over- (up to +33%) stoichiometric deviation to Se. Subsequent thermal annealing was done in a rapid-thermal processing furnace, in argon atmosphere. Annealing temperatures were 700 °C, 800 °C, or 1000 °C, annealing time was either 30 s or 32 min. Details are given in Ref. [8].

GISAXS experiments were done at the SAXS-beamline of the Elettra synchrotron radiation facility in Trieste, Italy. The employed X-ray wavelength was $\lambda = 0.154$ nm (energy 8 keV). GISAXS patterns were recorded with a 2D CCD-detector containing 1024×1024 pixels, placed in the y–z plane, perpendicular to the specular plane (x–z). Detector to sample distance was $L = 1150$ mm. A thin Al-stripe (beam stopper) was inserted in front of the 2D detector in order to attenuate the very intense specular beam (reflected beam, Yoneda peak, etc.) and thus avoid overflow of the detector and increase the sensitivity for scattered signal outside the specular plane. For each sample the GISAXS pattern was first recorded at the angle of incidence, α_i, equal