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# The evolution of the morphology of Ge nanocrystals formed by ion implantation in SiO<sub>2</sub>

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#### Abstract

Grazing incidence small angle X-ray scattering was applied to study the synthesis and growth of Ge quantum dots in Ge-implanted SiO<sub>2</sub>. Ge ion doses were up to  $10^{17}$ /cm<sup>2</sup>, and subsequent annealing temperatures up to  $T_a = 1000$  °C. Results suggest that ordered and correlated Ge QDs can be achieved by high-dose implantation followed by medium-T annealing.

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#### 1. Introduction

Physical properties of Ge nanocrystals or quantum dots (QDs), like tunable absorption, intense photo- and electroluminescence and third-order optical nonlinearities, are strongly dependent on QDs size. This makes them suitable for electronic, optoelectronic and photonic applications, like in sensor technology, for integrated opto-couplers in microsystems in biotechnology, for electronic nonvolatile memories, etc. [1]. Ion implantation offers great flexibility in the QDs formation by control of the process parameters, considerable freedom from thermodynamical limitations and extreme chemical purity [2,3]. Additionally, it enables dense packing of nanocrystals, and is compatible with the conventional silicon-based integrated circuit technology.

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In this paper the formation and growth of Ge QDs in the implanted  $SiO_2$  was investigated by means of grazing incidence small angle X-ray scattering (GISAXS), as a function of Ge ion dose and post-implantation annealing temperature.

### 2. Experimental details

100 keV <sup>74</sup>Ge<sup>+</sup> ions were implanted into a 250 nm thick SiO<sub>2</sub> amorphous layer, that was grown on (100) Si substrate by wet oxidation [4]. Samples with doses of  $1 \times 10^{17}$  cm<sup>-2</sup> and  $6 \times 10^{16}$  cm<sup>-2</sup> were annealed at temperatures,  $T_a$ , ranging from  $T_a = RT$  (not annealed) to 1000 °C, for 1 h in N<sub>2</sub> atmosphere. GISAXS experiments were carried out using X-ray photons of energy E = 8 keV (wavelength,  $\lambda = 0.154$  nm) at the Austrian SAXS beamline of the synchrotron radiation facility ELETTRA, Trieste, Italy. The two-dimensional GISAXS patterns were recorded with a 2D CCD detector containing 1024 × 1024 pixels, placed in the *y*-*z* plane, perpendicularly to the specular *x*-*z* plane [3].

#### 3. Results and discussion

The majority of 2D GISAXS patterns comprised of quasi-isotropic, half-rings, example of which is shown in the inset of Fig. 1. This (as well as other, not shown) GISAXS pattern showed quite a symmetric intensity distribution in all directions. These rings are interpreted as scattering from (spherical) Ge QDs; the interference maximum being related to the spatial correlation between isolated Ge QDs embedded in amorphous matrix. The formation of nanoparticles in  $SiO_2$ substrate was confirmed by Raman spectroscopy (appearance of the frequency mode in Raman spectra), while the spherical shape of QDs was established with transmission electron microscopy (TEM) in a few analogously implanted + annealed samples (not shown). They are chemically identified as Ge QDs through the appearance of the characteristic TO mode in Raman spectra and with grazing incidence X-Ray Diffraction in some of these samples (not shown).

Fig. 1. Vertical scans of 2D GISAXS pattern of SiO<sub>2</sub> samples implanted with Ge ion dose  $D_1 = 1 \times 10^{17}$ /cm<sup>2</sup>, and annealed at various annealing temperatures for 1 h in N<sub>2</sub>. Spectrum of the unimplanted SiO<sub>2</sub> substrate is added for comparison. Annealing temperatures (in °C) are indicated in the figure. Inset (upper): 2D GISAXS pattern of as-implanted sample. Inset (lower): Fits of spectrum of the sample annealed at  $T_a = 500$  °C using one size distribution (dashed line) and two size distributions (full line).

Fig. 1. shows one-dimensional (1D) GISAXS plots obtained by cross-sectioning 2D pattern parallel to the z-axis close to the beam-stopper, for samples implanted with the same Ge ion dose  $D_1 = 10^{17}/\text{cm}^2$ , but annealed at different annealing temperatures. By applying traditional analysis, the radius of gyration,  $R_g$ , of QDs was estimated from the so called Guinier plot, and the average interparticle distance, L from the curve maximum positions (Table 1). A strong half ring was present in the 2D GISAXS spectrum of as-implanted sample (inset of Fig. 1) showing that QDs were formed



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