ROBL-CRG	**Experiment title:** **In-situ growth of Ge and Si NC´s out of a [(GeO_x or SiO_x)/SiO_2] multilayer structure**	**Experiment number**: 20-02-658
Beamline: BM20	**Date of experiment**: from: 12.02.2008 to: 19.02.2008	**Date of report**: 06.11.2008
Shifts: 18	**Local contact(s)**: Dr. Carsten Baehtz	*Received at ROBL:*

Names and affiliations of applicants (* indicates experimentalists):

N. Jeutter*, M. Zschintzsch*, J. von Borany*, C. Baehtz*

Institute of Ion Beam Physics and Materials Research, FZD, Dresden, Germany

Report:

Semiconductor nanocrystals (NCs) are of fundamental interest for new generations of light emitters or high-efficiency solar cells [1]. Within this experiment, Ge NCs have been fabricated by decomposition of GeO_x (0< x <2) within a (GeO_x-SiO_2) superlattice (SL) structure which enables a precise control of the size and the position of the NCs. Varying the oxygen partial pressure, the deposition temperature and the DC power, the GeO_x stoichiometry and the thickness ratio of the GeO_x-SiO_2 sub-layers can be tailored in a well-defined manner. The SL (2.5 nm GeO_x / 3.9 nm SiO_2)^{19x} was grown by reactive DC magnetron sputtering from elemental Si, Ge targets onto a SiO_2 / Si substrate. The deposition was performed at 250°C at 0.5 Pa working pressure in an Ar/O_2 mixture using the ROBL dual-magnetron sputter chamber [2], whereby the *in-situ* characterization using X-ray scattering methods (reflectivity, diffraction) of thin film systems during growth and and subsequent annealing is possible.

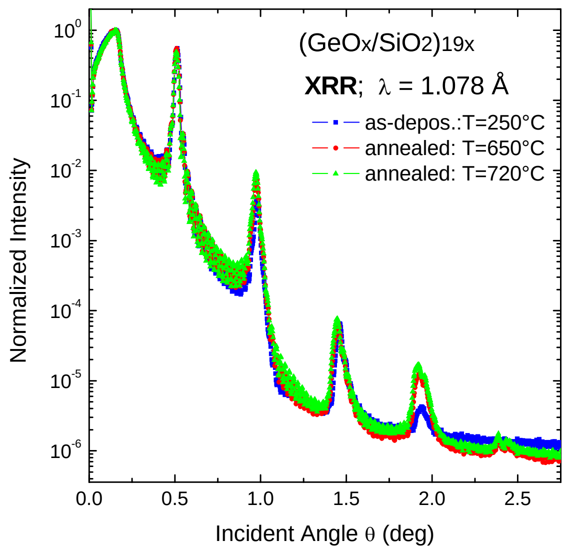

Fig. 1: *X-ray reflectivity after deposition and subsequent annealing steps*

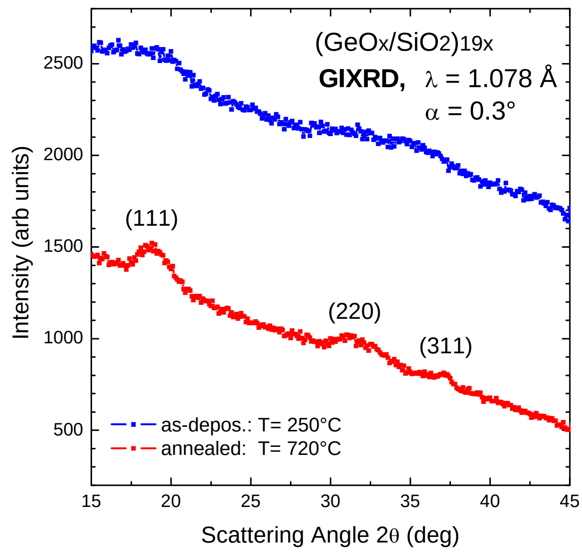

Fig. 2: *GI-XRD pattern of the Ge NCs after deposition and 720°C annealing*

Fig. 1 shows X-ray reflectivity (XRR) collected after deposition and during annealing. The intensity of the higher-order superstructure peaks increases during annealing indicating an enhancement of thickness uniformity (periodicity) and smoothing of the SL interfaces. Interface roughness (< 0.5 nm) have been derived from the simulation of the XRR curves. The formation of Ge nanocrystals by the GeO_x decomposition has been observed by grazing-incidence diffraction (Fig. 2). Although in the diffraction pattern a near-order hump is already present in the as-deposited state, a pronounced Ge(111) signal appears after 540°C annealing which did further increase after annealing at 600°C. From Scherrer's equation the crystal size was determined to 2.5 nm which corresponds perfectly to the initial thickness of the GeO_x film.

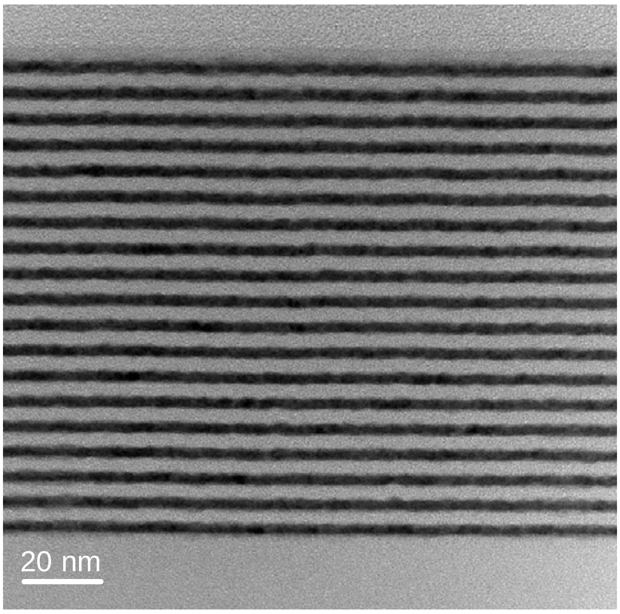

Fig. 3a: *TEM-image of the GeO_x-SiO_2 SL after 720°C post-annealing.*

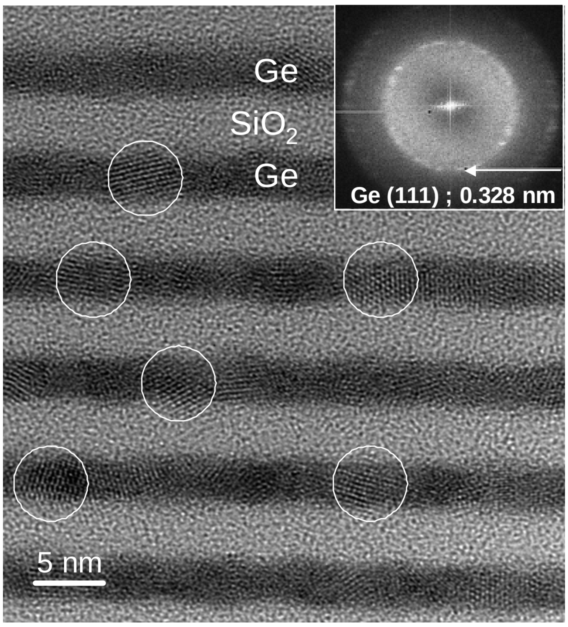

Fig. 3b: *HR-TEM-image with the Ge NCs (marked with white circles), the inset shows the corresponding FFT image.*

The NCs formation was proved *ex-situ* by transmission electron microscopy (TEM). Figure 3a shows a cross-sectional image of the superlattice after 600°C annealing, confirming the smooth interfaces and negligible interdiffusion of the SL obtained by XRR. Applying high-resolution TEM, Ge NCs were detected by imaging of Ge(111) lattice planes (Fig. 3b) or the corresponding spots at the FFT image (Fig. 3b, inset). For this sample, more a nanocrystalline Ge film have been realized which can be changed towards separated Ge nanoclusters by increasing the oxygen content in the GeO_x sub-layers during deposition.

References:

[1] S. Foss, T.G. Finstad, A. Dana, A. Aydinli, Thin Solid Films **515**, (2007), 6381-6384.

[2] W. Matz, N. Schell, W. Neumann, J. Bøttiger, J. Chevallier, Review of Scientific Instruments **72**, (2001), 3344-3348.