ROBL-CRG	**Experiment title:** **The influence of the substrate temperature to the *in-situ* growth of Ge nanocrystals**	**Experiment number:** 20-02-667
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Names and affiliations of applicants (* indicates experimentalists):

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

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

Report:

The fabrication of Ge-NC's by decomposition of GeO$_x$ ($0<x<2$) out of a (GeO$_x$-SiO$_2$) superlattice structure (SL) has been studied *in-situ* sputtering chamber at ROBL. The SL of (GeO$_x$ / SiO$_2$)^{19x} were grown by dual reactive DC magnetron sputtering from elemental targets. Different Ge/O ratios in the SL structures were realized by the variation of deposition temperature.

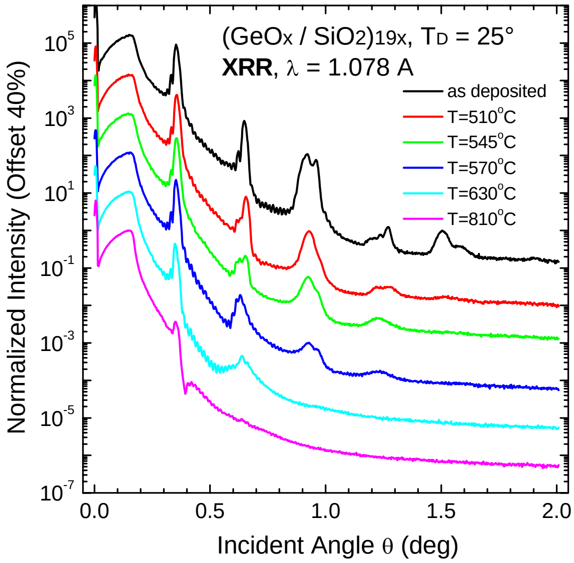

Fig. 1: *XRR scans of a SL structure deposited at RT (period thickness 9.64 nm) recorded during subsequent annealing steps.*

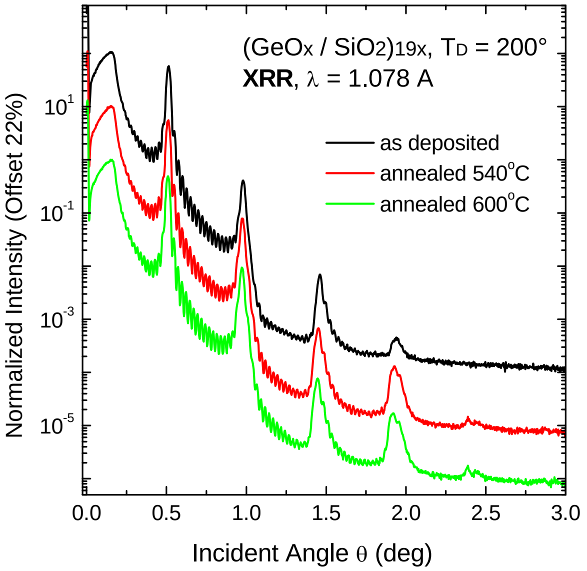

Fig. 2: *XRR scans of a SL structure deposited at 200°C (period thickness 6.22 nm) recorded during subsequent annealing steps.*

The X-ray reflectivity (XRR) curves, of the deposition at room temperature and at 200°C respectively, show clearly highly orderered SL structures. For the RT deposition one can observe a split of the superlattice peaks at the 3rd, 4th and 5th order. This indicates a slight variation off less then > 0.5nm for the period thickness of the multilayer. For samples with a deposition temperature of 200°C only a little split at the 4th order can be seen. Therefore the variarity in thickness can be taken as negligible small. The simulation of the XRR curves reveal smoother interfaces for the SL deposited at 200°C compared to RT deposition (roughness σ = 1.0 / 0.6 nm for T_D = RT / 200°C, resp.)

The diffrence of the two samples becomes more significant when the samples are post-annealed. Samples deposited at RT clearly show a degrading of the SL structure with every annealing step [Fig. 1], indicated by less and less observed SL peaks. Samples grown at T = 200^{o}C do not show this degradation [Fig. 2]. Furthermore a little improvement of the SL structure can be observed during the annealing step to 540^{o}C which is kept also at 600^{o}C.

The crystalisation process of the Ge particles was monitored with grazing incidence X-ray diffraction as a function of annealing temperature. For both depostion temperatures the post-annealing of the SL structures is leading to the growth of Ge NC's at about 540-550^{o}C [Fig. 3a, b]. Hereby one can derive the temperature 540^{o}C as the necessary crystal formation temperature. This result corresponds with the results of M. Zacharias et al. [1] for the formation of Ge NC's out of a SiGeOx alloy films. Samples deposited at RT show no Ge (111) signal at T = 510^{o}C. A weak signal can be obtained at T = 545^{o}C. The FWHM gives, fitted and calculated with Scherrer's equation, a NC's size of 4 nm. Within further annealing up to 630^{o}C the average cystal size increases and is finally around 7.2 nm at T = 690^{o}C [Tab. 1]. The interface roughness of the SL structure increases significantly after this annealing step. Annealing at 810^{o}C finally destroys the superlattice structure. This corresponds with a huge increase of the Ge NC's size after this annealing step. The crystals are equal sized or even bigger in size then the SL period (9.64 nm).

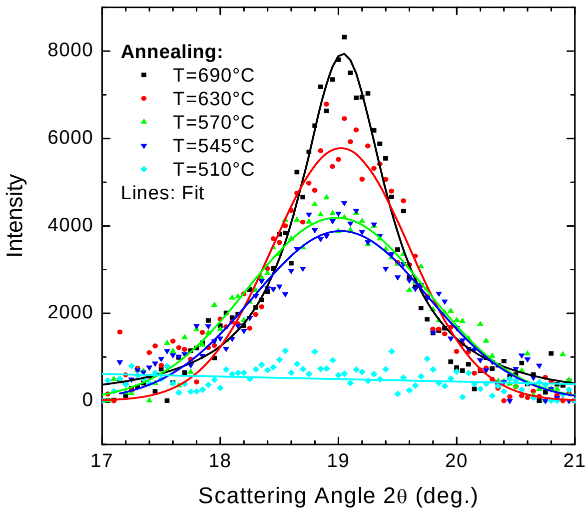

Fig. 3a: *Ge(111) signal measured with GIXRD for the SL deposited at room temperature.*

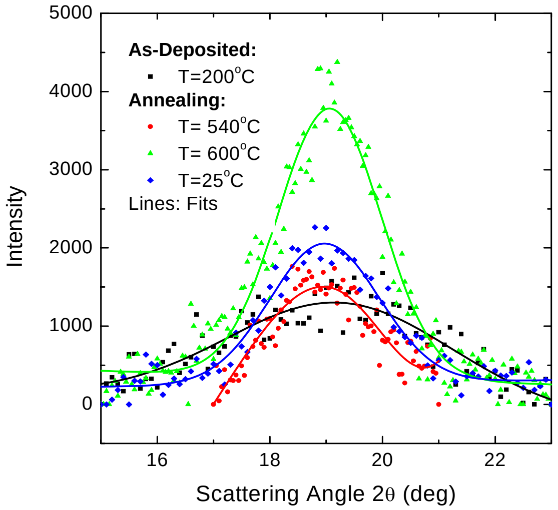

Fig. 3b: *Ge(111) signal measured with GIXRD for the SL deposited at 200^{o}C*

T_A (°C)	Ge (111), Fit 2 (Lorenzian)		
	2θ (deg)	FWHM	NC Size
545 ^{o}C	**19.032**	1.427	4.54 nm
570 ^{o}C	**18.975**	1.501	4.32 nm
630 ^{o}C	**19.031**	1.110	5.85 nm
690 ^{o}C	**19.038**	0.898	7.22 nm
810 ^{o}C	**19.021**	0.655	9.90 nm

Tab. 1: *Fit results of Ge (111) reflection*

The observed Ge (111) signal of the samples deposited at T = 200^{o}C leads to a well pronounced signal at 600^{o}C [Fig. 3b]. Here the crystal size can be determind to be 2.3 nm. It shows that Ge NC's have almost reached the maximum possible size which corresponds to the GeO_x layer size of about 2.4 nm.

From these results one can clearly conclude that the stochiometry of the GeOx layer is influenced strongly by the depostion temperature.

References:

[1] M. Zacharias et al., Thin Solid Films **278**, (1996), 32-36