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- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** X-ray investigation of Ge-nanocrystals in SiC	**Experiment number**: Me 309
Beamline: BM 20	**Date of experiment**: from: 10/02/2002 to: 14/02/2002	**Date of report**: 06/11/2002
Shifts: 12	**Local contact(s)**: Andreas Bauer	*Received at ESRF:* **06/11/2002**

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Report: The aim of the experiment has been to characterize Ge-nanocrystals implanted in 4H-SiC(0001) substrates by mean of high resolution x-ray diffraction methods (HRXRD). Special interests for the optoelectronic device developer are: preferred crystallographic orientation of the formed Ge-nanocrystals in respect to the substrate lattice, the size of the grown nanocrystals as well as the chemical phase of the Ge-nanocrystals.

Samples

Ge^{+}-ions with an energy of 250keV were implanted into 4H–SiC(0001) crystal wafers produced and supplied by Cree Research Inc. The implantation temperature was kept at 20°C and 700°C, respectively. The high implantation temperature was chosen to prevent amorphization of the crystals. The ion current density was less than 1 μAcm^{-2}. Samples were tilted about 6°–8° to the ion beam to avoid channeling effects during implantation. The ion fluence of $1*10^{16}cm^{-2}$ we used causes a Ge-peak concentration of approximately 1 at.% within the projected ion range in the deepness of 105nm below the surface. Thermal annealing was carried out at temperatures up to 1600°C in Ar-atmosphere (pressure 20kPa) using a rapid thermal annealing machine working with the samples placed between two electrically heated graphite stripes. To prevent significant decomposition the annealing process was done face-to-face with an unimplanted SiC piece [1]. Figure 1 shows the corresponding cross section TEM image of a Ge-nanocrystal formed in the SiC matrix.

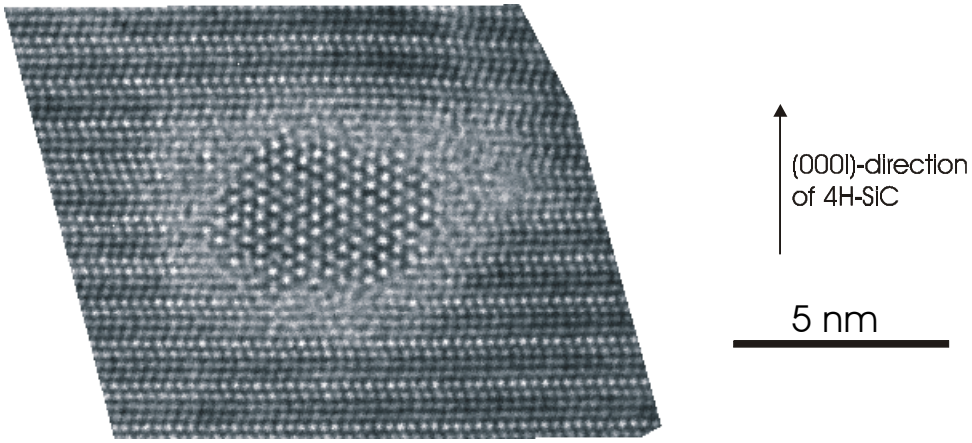

Fig. 1: High resolution TEM image of a Ge-nanocrystal formed after implanting and annealing at 1600°C in the 4H-SiC(0001) wafer

Characterization

The x-ray diffraction experiments carried out at ROBL can be separated in two parts.

- **Firstly, characterization of Ge-nanocrystals formed in SiC samples exposed to different process parameters**

Sample	*34-0*	*34-1*	*34-3*	*46-2*	*45-1*	*45-2*	*34-2*	*46-1*
Implantation at 700°C	-	X	X	X	X	X	X	X
Annealing temperature	-	-	1400°C	1500°C	1600°C	1600°C	1600°C	1600°C
Annealing time	-	-	30s	30s	1s	30s	120s	360s

Tab. 1: The list of the measuring samples

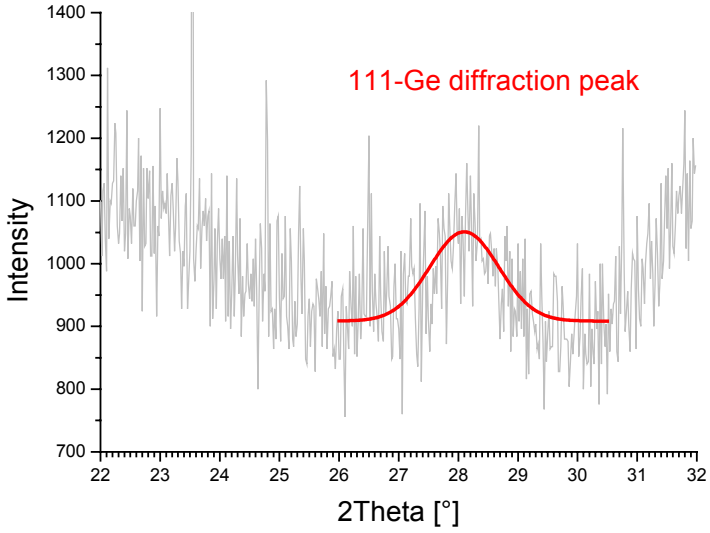

Fig. 2: θ/2θ-scan of the symmetric Ge-111-reflection, sample 34-2, λ=0,154685nm

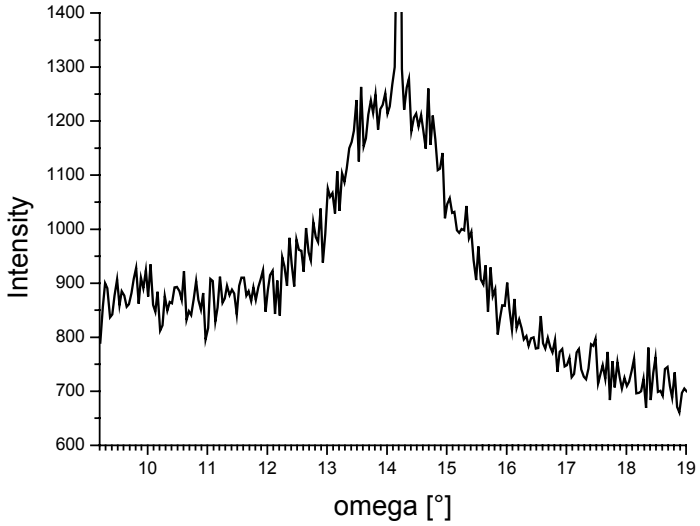

Fig. 3: ω-scan of the symmetric Ge-111-reflection, sample 46-1, λ=0,154685nm

Only samples which was annealing at 1600°C have been shown a 111-Ge diffraction peak. The occurrence of 111-Ge diffraction peaks (Fig. 2) in the XRD pattern of the annealed samples reveals the formation of crystallized Ge inside the SiC samples. The observed reflection broadening is caused by the crystallite size. Using Scherrer's formula the measured full width at half maximum $(FWHM)_{2\theta}$ results in to the crystallite size perpendicular to the wafer surface (see table 2). Reflection broadening in the ω-scans (Fig. 3), carried out at the 2θ angle position of the 111-Ge reflection, is caused by the lateral crystallite size. The measured $(FWHM)_{\omega}$ is equivalent to a lateral crystallite size (see table 2). In-plane, the lateral crystallite size is rotation symmetric. We did not find any dependence on the lateral orientation angle ϕ. It is obvious that the maximum of the 111-Ge diffraction peak is located at 2θ=28.2°. Compared to the value for crystalline Ge (2θ=27.3°) this means a significant shift of the Bragg angle toward the value for crystalline Si (2θ=28.4°). We can discuss two possible reasons for this behavior. The Ge-nanocrystallitis could be added impurities of Si atoms. On the one hand, the XRD measurements cover a sample area of nearly 1 mm^2 and for that a large volume of implanted SiC. The measurement represents an average of all crystallites in that area, including crystallites with different chemical composition. On the other hand, the shift of the Bragg angle can be caused by strain in the crystallites. The observed shift corresponds to a change of the lattice constant of $\Delta a/a=3.1*10^{-2}$.
Using Hook's formula, the hydrostatic pressure p inside the crystallites can be estimated to be $7.8 *10^8$ Nm^{-2}. In comparison with the in-plane strain of $\sigma_1=\sigma_2=2.26*10^8$ Nm^{-2} in the case of 3C–SiC deposition on 6H–SiC. This seems to be a realistic value.

Sample	***45-2***	***34-2***	***46-1***
Implantation at 700°C and annealing at 1600°C	X	X	X
Annealing time	30s	120s	360s
$(FWHM)_{2\theta}$	1,98°	1,16°	0,80°
crystallite size perpendicular to the wafer surface	4nm	7nm	10nm
$(FWHM)_{\omega}$	2,98°	2,20°	1,84°
lateral crystallite size	5nm	7nm	9nm

Tab. 2: The crystallite sizes lateral and perpendicular to the wafer surface of the Ge-nanocrystals formed after implanting and annealing at 1600°C in the 4H-SiC(0001) wafer

➢ **Secondly, characterization of the SiC substrates in the different stages of the nanocrystals-production-process**

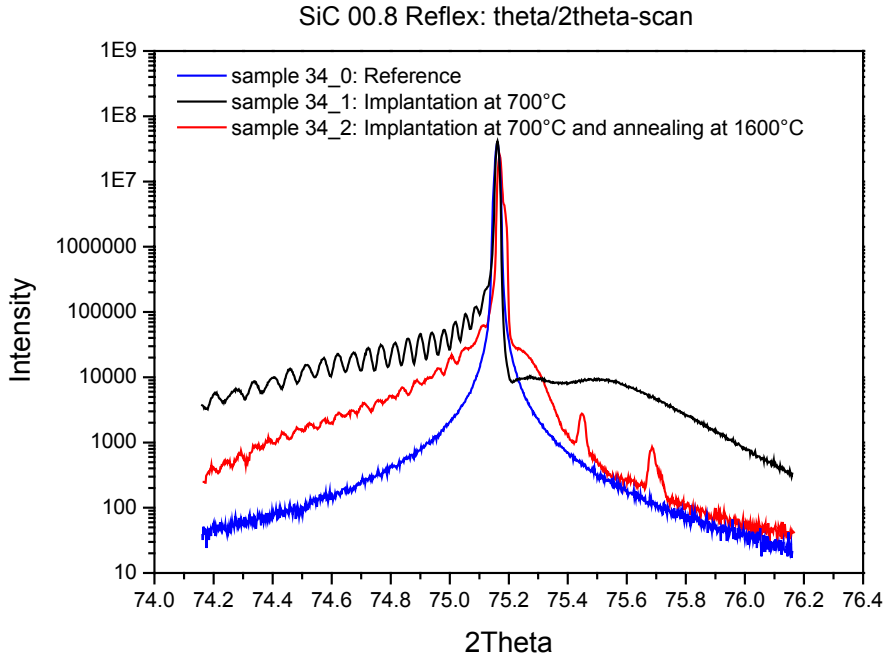

Fig. 4: θ/2θ-scan of the symmetric SiC-0008-reflection – three stages of the nanocrystals-production-process:
(1) a untreated 4H-SiC(0001) bulk single crystal wafer (sample 34-0)
(2) a sample after the Ge^{+}-ions implantation with an energy of 250keV and a ion fluence of $1*10^{16}cm^{-2}$ at 700°C (sample 34-1)
(3) a sample after the Ge^{+}-ions implantation and thermal annealing at 1600°C in Ar-atmosphere (pressure 20kPa) for 120s (sample 34-2)

The implantation of Ge-atoms generates in the SiC lattice a strong disordered interlayer in a depth of 100nm and with a thickness of approx.10nm. The crystal area above the strong disordered interlayer is structural nearly undisturbed. The left side wing oscillation of the symmetric SiC-0008-reflection, as shown in Fig. 4, is caused by the interference effects of the diffracted x-ray beam due to the layer thickness above the buried strong disturbs interlayer. Hence, the depth of the interlayer can be determined using the oscillation period. The interference effect was reduced after the thermal annealing at 1600°C. This is understandable by formation of Ge-nanocrystals (see Fig. 1) and the strong disordered interlayer heals partially up.

The x-ray diffraction experiments have been very useful in characterization of the implanted and anneals Ge:SiC wafer. Additional investigations are required to clear up the observed reflection shift of the Ge-nanocrystals. Is this caused by any impurities or hydrostatic strain of the Ge-nanocrystalls.

We thank all members of the ROBL beamline team, especially Dr. N. Schell and A. Bauer for the helpful support at the time of execution on this experiment.

[1] Ch. Schubert, U. Kaiser, A. Hedler, and W. Wesch, T. Gorelik, and U. Glatzel, J. Kräußlich, B. Wunderlich, G. Heß, and K. Goetz; Journal of Applied Physics, Vol. 91 No. 3 (2002), pp.1520-1524