<table>
<tr><td>ESRF</td><td>Experiment title:
Structural investigation of metallic nanoclusters in wide band gap semiconductors</td><td>Experiment number:
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18</td><td>Local contact(s):
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<tr><td colspan="3">Names and affiliations of applicants (* indicates experimentalists):
Bente Walz*, D.V. Novikov*,
DESY, Hamburg, Germany

J. Grenzer*, G. Talut*
Forschungszentrum Dresden-Rossendorf e.V.,
Institute of Ion Beam Physics and Materials, Germany</td></tr>
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Report:

In this experiment the investigation of secondary phase formations (bcc-Fe. $Fe_{3-x}N$) in iron-implanted GaN substrates and the analysis of resulting structures by reciprocal space mapping in wide- and small-angle scattering mode was proposed.

The experiment was carried out at the ROBL beamline on the pseudo-six circle diffractometer, in mirrored monochromatic setup with a X-ray wavelength λ=0.124 nm. Single crystalline wurtzite GaN(001) films of about 3μm thickness, epitaxially grown by metal organic vapour phase epitaxy (MOVPE) on Al_2O_3(001), were implanted with 195 keV ^{57}Fe ions to a maximum Fe concentration of 4 at.% at the projected range R_p = 85 nm. The standard technological process requires a subsequent heat treatment in N_2 atmosphere at the pressure of 1.1 bar. The equipment available at the beamline at the time of the experiment, however, could not be run at elevated gas pressures in the sample cell. Therefore, the annealing experiments were carried out at a reduced N_2 pressure of 0.5 bar and in a temperature range from 700°C to 850°C. The *in situ* reciprocal space mapping measurements were carried out using a Pilatus 100K detector with 10s acquisition time per point.

In the frame of the experiment, we have found out that the annealing behavior of ion-implanted samples at a low gas pressure differs strongly from those treated at higher pressures. Instead of the formation of Fe clusters at elevated temperatures, we observed the formation of iron nitride phases. Figure 1(a,b) shows two X-ray scattering patterns recorded under low- and high- temperature conditions. The wide diffuse spots indicated by arrows come from Bragg scattering of the nanosized particles of the secondary phase incorporated into the GaN matrix. The full width at half maximum of the observed reflections corresponds to the particle dimensions of about 5 nm. The bright vertical lines come from the Bragg reflections of the bulk single crystal environment and could be used for additional angular position and temperature control and calibration. The low-temperature phase can be determined as ε–Fe_3N and is well known from literature. The Bragg reflection of the high-temperature phase can be attributed to several iron nitride phases, including FeN, ζ-Fe_2N or disordered ε–$Fe_{3-x}N$. Remarkably, the observed phase transformation appeared to be fully reversible: by controlling the temperature, the system was transformed back and forth between the initial low-temperature and the new high-temperature states. With each annealing cycle the integral intensity reduces slightly indicating a loss of Fe due to an out diffusion towards the sample surface. Figure 1(c) shows the angular position of the iron nitride phase peak depending on the sample temperature.

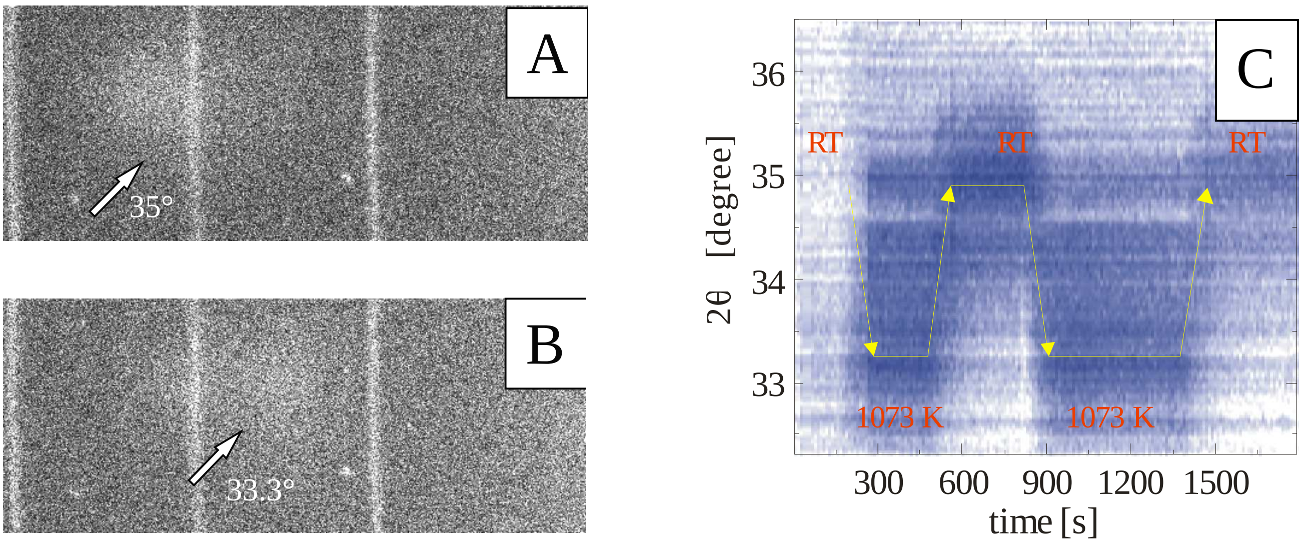

Figure 1. X-ray scattering patterns and temperature dependent phase composition behavior in Fe-implanted GaN [1].

The obtained results open a way for engineering of selected secondary phases, embedded into GaN matrix and possessing different electronic, magnetic and other properties, by variation of processing parameters. In a follow-up experiment it was also shown, that nanoparticles obtained in the current experiment display a superparamagnetic behaviour [1].

[1] G.Talut, J.Grenzer *et al* Annealing of Fe implanted GaN in reduced atmosphere, *in preparation*