ESRF	**Experiment title:** ***Structural properties and composition of high Tc GeMn nanocolumns and their surrounding matrix studied by X-ray anomalous diffraction***	**Experiment number**: HE2341
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Report:

X-ray scattering measurements on a series of GeMn nano-columns in Ge samples have been carried out at the ID01 beamline of the ESRF. Reflectivity, Grazing Incidence Small-Angle X-Ray Scattering (GISAXS) and Grazing Incidence X-Ray Diffraction (GIXD) were carried out to study the samples. The experiment was conducted close to the Mn K-edge (6.5 keV) to enhance the contrast between Ge and Mn.

Although there were a few problems related to the low energy used – notably some beam instabilities (strong intensity oscillations) in the first two days of the experiment, and difficulties in changing the energy around the Mn K-edge while keeping the beam at exactly the same grazing incidence position – the experiment went well with interesting results for several samples.

The high Curie temperature observed for the samples is believed to be related to the structure of the GeMn columns. It was therefore the goal of this experiment to determine the crystal structure inside the columns, as well as possible interdiffusion at the interface between the columns and the Ge bulk, and the strain in the Ge matrix induced by the presence of the columns.

For this experiment we studied a series of samples grown at different temperatures and composition (see table 1)

Sample	***Composition***
GM211	6% Mn, T=140°CA, large (~6 nm) columns
GM19	6% Mn, T=125°C, 3nm columns
GM206	6% Mn, T=125°C, 2-3 nm columns
GM205	Reference sample, Ge/Ge
GM209	0.1% Mn, T=130°C
GM173	7% Mn,150°C, large (~6 nm) columns

Table 1 *: samples studied during the experiment*

For 3 different samples we first measured GISAXS images. As shown in fig. 1, these images exhibited several correlation peaks: the shortest correlation distance (~8nm) corresponds to the average inter-column distance as observed using microscopy, and features oscillations along q_z due to the finite extent of the GeMn columns (80 nm deep). These oscillations were not observed for all samples, and their absence could indicate a greater disorder in the columns positions or vertical structure. The longest correlation distance (40nm) was unexpected and could be related to defects in the Ge layer, which could play a role in the columns formation.

We also measured GIXD scans around the (220) and (400) reflections, using radial and angular scans (fig.4). These revealed the same correlation peaks as observed in GISAXS. The intensity decrease will be

used to model the strain distribution in the Ge matrix around the columns – a direct (model-free) analysis using standard analysis (Huang and Stokes-Wilson scattering) is not possible due to the presence of the correlation peaks.

In order to determine the crystal structure of the GeMn columns we then measured GIXD maps around existing Ge reflections. Close to the Ge diffraction peaks (fig.3) these have shown that the correlation peaks observed on 1D scan corresponds to a ring, indicating that there is no in-plane ordering. On large maps (fig.4) several diffuse streaks have been observed, with peaks around (*-310*) , (*-2.65 0.5 0*) and *(-1.4 1.4 0)* positions.

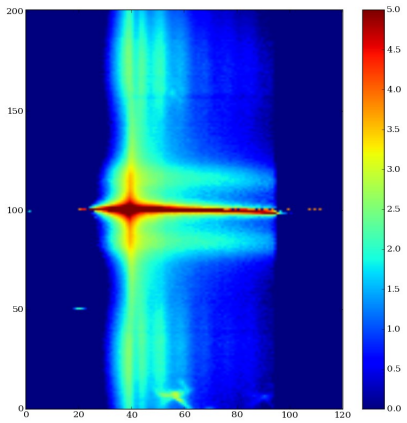

Figure 1: Grazing Incidence Small-Angle X-ray Scattering (GISAXS) image. Two correlation peaks can be observed, corresponding to 40nm and 8nm.

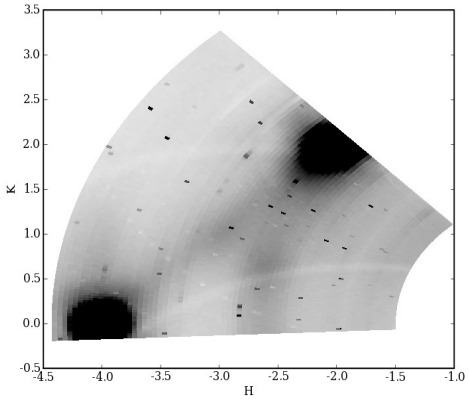

Figure 2: Grazing Incidence X-ray diffraction (GIXD) map in the vicinity of the (-220) and (-400) reflections. Diffuse streaks can be seen with maxima around (-310) and (-2.65 0.5 0), and around (-1.4 1.4 0)

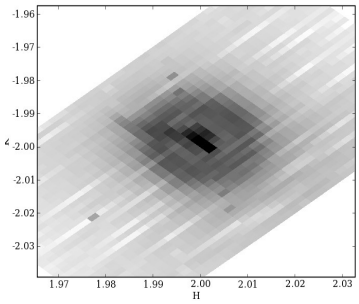

Figure 3: GIXD scattering map around the (2-20) Ge reflection, exhibiting a correlation ring around the Bragg peak. The distance corresponds to ~40nm.

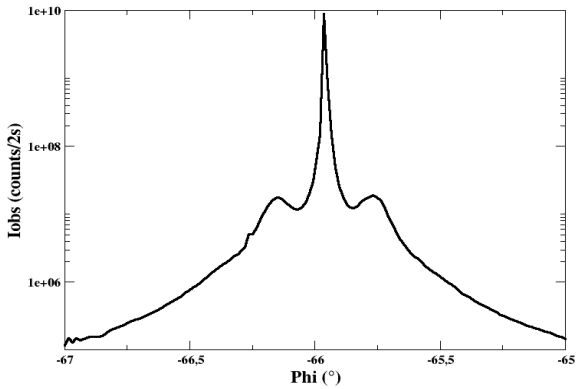

Figure 4: angular scan around the (-220) reflection, exhibiting to correlation peaks corresponding to a distance of ~40nm.

Preliminary analysis

While the analysis is going on, some results can already be pointed out:

- the “8 nm” correlation peak, corresponding to the average distance between columns as observed in microscopy, is not present on all samples. This could indicate that the regularity (vertically) of the columns can be strongly affected by the growth conditions.
- the “40 nm” correlation peak/ring, observed both in GISAXS and GIXD was unexpected, and is present on all samples. It could be related to defects in the Ge bulk, and may play a role in the genesis of the columns
- the presence of diffuse peaks in the GIXD map (fig. 2) indicate that the GeMn phase must correspond to a crystallographic phase which is more complex than a simple substitution of Mn in the Ge lattice, as was expected due to the average composition in the columns. The fact that diffuse streaks link the Ge diffraction peaks to the GeMn peaks indicate that the two lattices are probably geometrically related.

Future work

Due to the beam instabilities when changing the energy around the Mn K-edge (it was not possible to compare intensity measured at a given position in reciprocal space for two different energies, due to the beam shifting), *it was not possible to conduct anomalous scattering* (multi-wavelength) measurements. This will be crucial in determining the structure of the GeMn phase as it will allow to determine the relative contribution of the Ge and Mn atoms on the non-Ge peaks. To achieve this we will work around the Ge K-edge for the next experiment – it is less ideal in terms of anomalous sensitivity but will provide a more stable beam, and will also allow to probe more reflections.

In order to obtain more information on the GeMn phase, *we will also look at out-of-plane reflections*. As the GeMn structure is extended perpendicularly to the surface of the sample, we expect peaks with a sharper extent along the q_z direction. This was not possible during this experiment due to the long wavelength (~2Å) and due to the geometrical restrictions of the “Huber Tower” goniometer. Using the “Chi Circle” goniometer combined with a 11keV energy beam will allow us to completely measure truncation rods starting from in-plane diffuse peaks.