ESRF	**Experiment title:** Correlation between structural and magnetic defects in Dy islands	**Experiment number**: HC-4054
Beamline: ID01	**Date of experiment**: from: 24.09.2020 8:00 to: 27.09.2020 8:00	**Date of report**: 16.10.2020
Shifts: 9	**Local contact(s)**: Steven Leake	*Received at ESRF:*

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Report:

The prupose of the proposal was to measure correlation of structural and magnetic defects in pure Dysprosium by using coherent X-ray diffraction. This requires measuring magnetic speckle patterns at low temperature, at the Dy L3 edge (7.797 keV), with good statistics and sufficient sample/beam stability. Such measurement is possible only thanks to the EBS.

Samples

For this experiment, a 150-nm thick film of Dy was grown by molecular beam epitaxy at Institut Jean Lamour, following a well established process. The film was split in 3 samples. 2 of them were patterned by optical lithography to obtain arrays of cylindrical dots of diameter 4 µm and 2 µm respectively. These micron size objects were designed to facilitate the positioning of the focused X-ray beam, in order to measure the structural and magnetic reflections at the same position. The 3rd sample was not patterned. The magnetic properties of the patterned samples were found to be identical to those of the continuous film.
It turned out that we did not have time to measure the 4-µm diameter dots during the beamtime. Hence only the continuous thin film and the 2-µm diameter dots were measured.

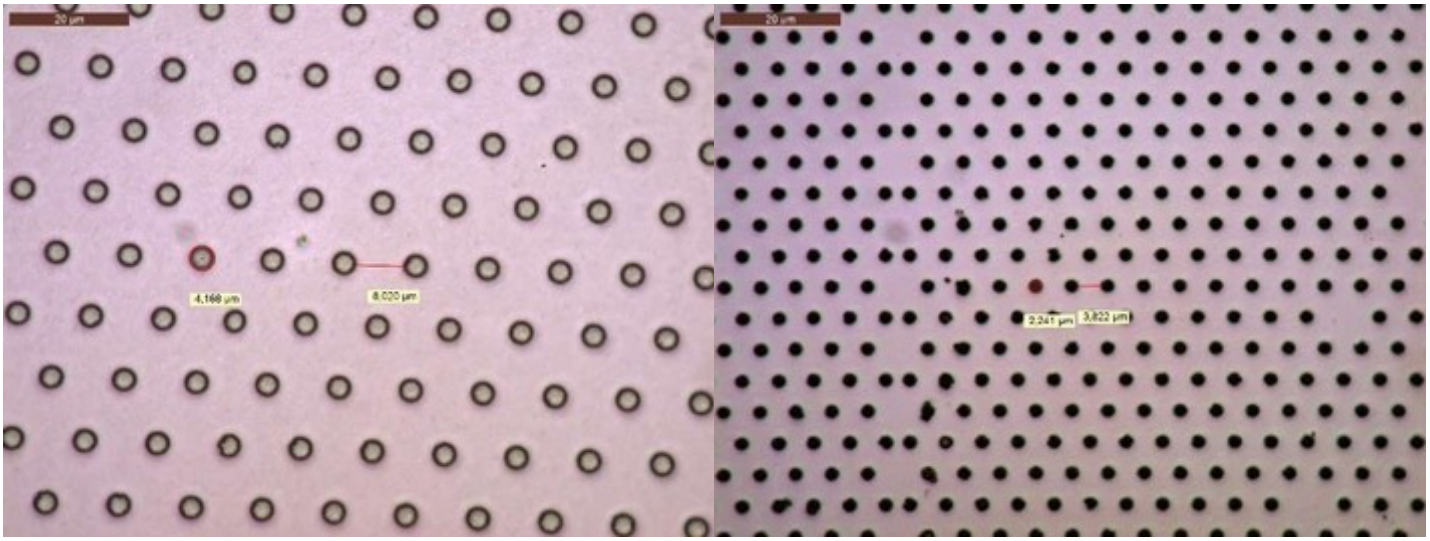

Figure 1: Optical microscopy images of the patterned samples (left: 4-µm diameter, right: 2-µm diameter)

Experimental set-up

The samples were glued (one at the time) at the top of a quartz capilary, itself mounted on the piezo stage of the diffractometer.

A Nitrogen stream was used to cool the samples (the magnetic phase of interest exists in the 89 K – 179 K range for bulk Dysprosium). The controler of the cryostream was not interfaced with the beamline control software, preventing from scanning the temperature. Measurements at several temperatures were done by manual adjustments.

Figure 2: View of the sample on its capillary, with the nose of the cryostream. The incident beam aperture is visible behind.

The incident X-ray beam was tuned to the Dy L3 edge (7.79 keV), which enhances the magnetic scattering of Dy atoms. The beam was focused to a 300 nm x 300 nm spot with Compound Refractive Lenses (CRLs), delivering 8.10^{10} ph/s.

In collaboration with ID01 staff, we installed a polarisation analyser on the detector arm, with the Maxipix mounted at the exit of the analyser, in order to measure 2D diffraction patterns. The incident beam had the natural linear horizontal polarisation of the undulator, the sample was oriented to scatter in vertical geometry and the polarisation analyser in horizontal geometry, such that we rejected most of the photons with unrotated polarisation. The polarisation analysis was performed with the (333) reflection of a germanium single crystal borrowed from BM28 (XMaS, UK CRG beamline). At the Dy L3 edge, the bragg angle of the Ge (333) reflection is 47°, hence transmitting only 7% of the intensity with unrotated polarisation. Ge was preferred over graphite, whose (006) reflection provides a better rejection at the Dy L3 edge, because the high mosaicity of the graphite would distort the transmitted diffraction pattern, while the high crystalline quality of Ge minimizes such distortion.

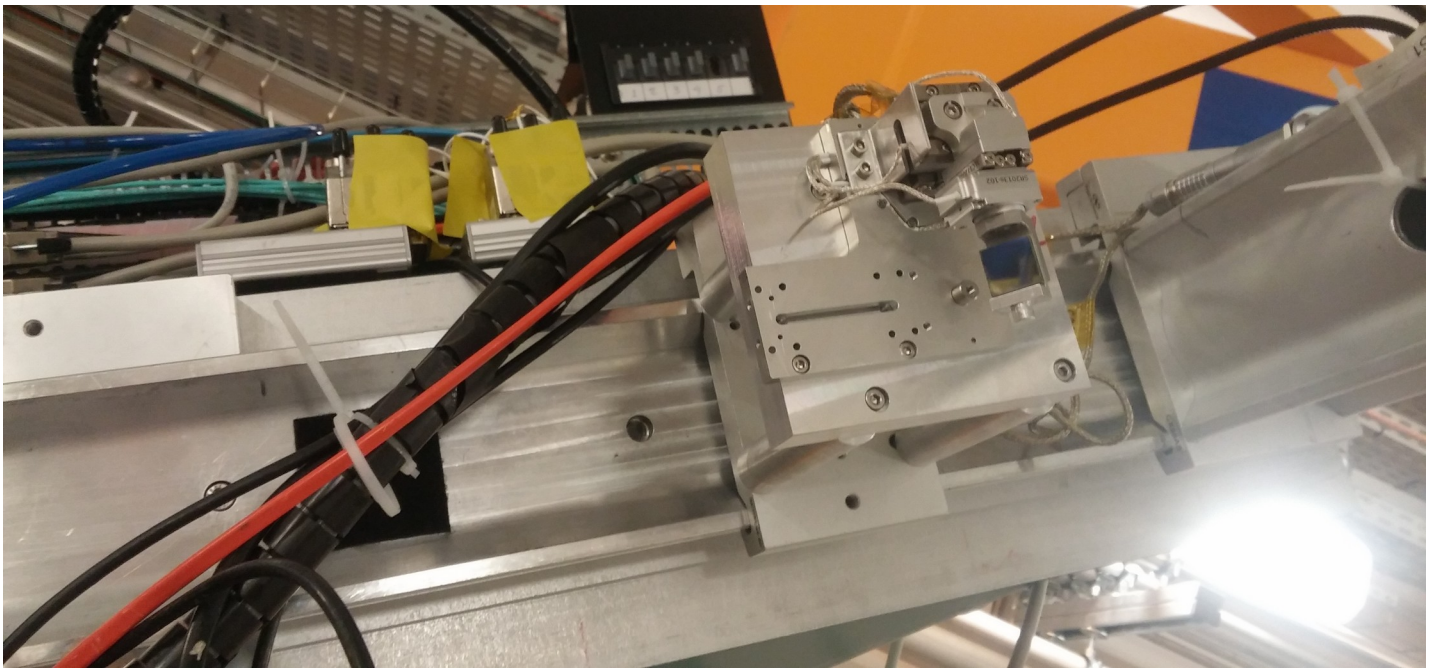

Figure 3: Photo of the crystal analyser mounted on the detector arm, at 45°. The detector, mounted at 90° from the arm as close as possible to the crystal, has been removed for the photo.

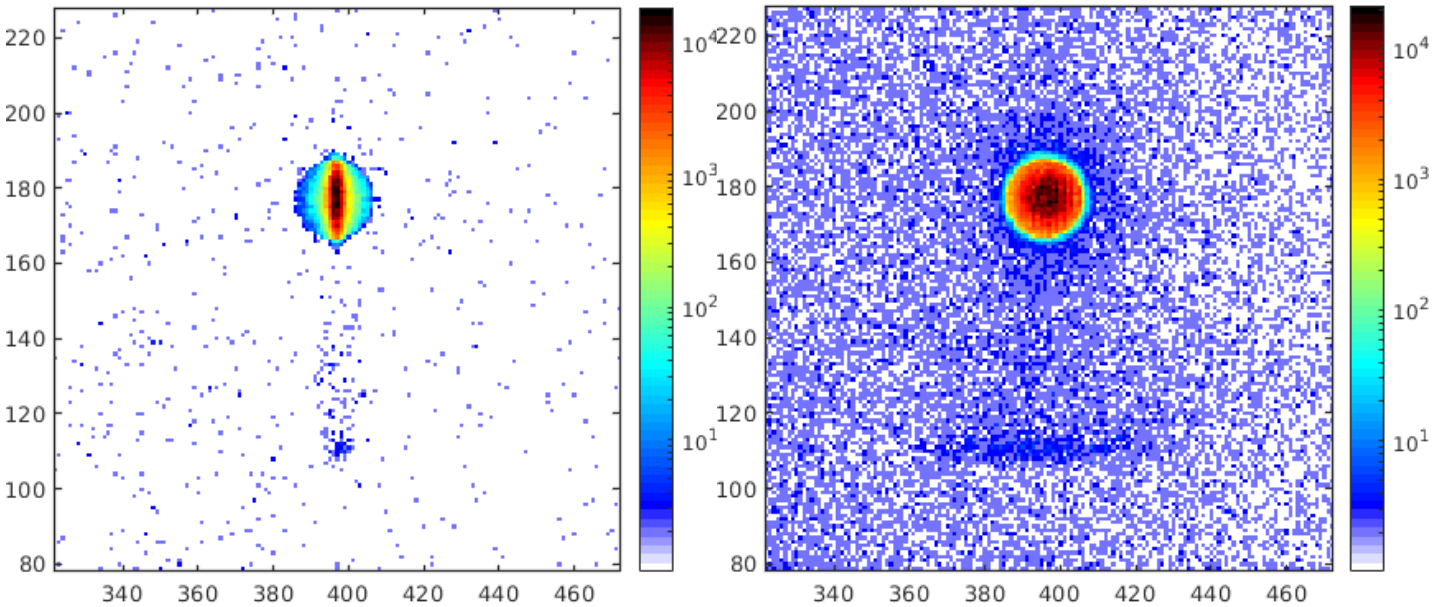

Figure 4: Left: Image of the direct beam through the crystal analyser. The analyser selects a vertical slice of the beam of FWHM~3 pixels=0.01°, while the incident beam is otherwise isotropic (as seen from the diffuse shape around the central slice). Right: the full incident beam is recovered by integrating over the analyser angle. The crystal analyser creates a weak artefact below the main signal, which is not yet understood.

Experimental results: continuous Dy film

The rocking curve of the (002) reflection of the Dy film yields 1.10^8 cts/s at the peak (without the analyser, integrated on the Maxipix), with FWHM 0.23°. The speckles denote the structural defects of the Dy film.

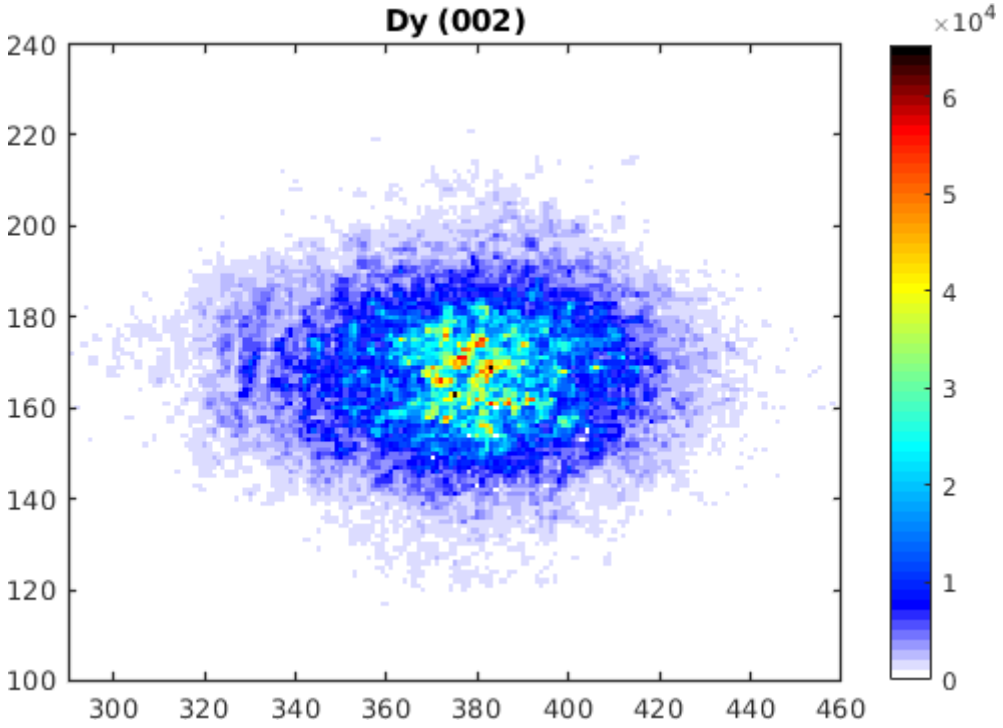

Figure 5: Speckle pattern on Dy (002) reflection at ~80 K. 3s snapshot with 1/3.6 attenuation filters. Maximal intensity is 8.10^4 cts/s after normalisation.

Similarly to the measurements on the direct beam, only a vertical slice of the speckle pattern is visible when measured with the analyser, and an integration over the analyser angle must be done. By compairing vertical slices of speckles with and without the analyser, we can estimate the efficiency of the analyser at ~20%, with no loss of speckle visibility (Figure 7).

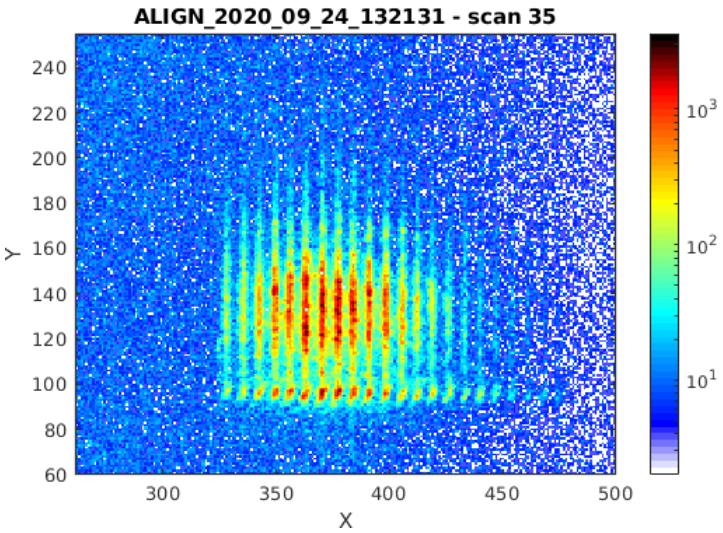

Figure 6: Speckle pattern at the Dy (002) reflection, recorded with the analyser. Intergation over the analyser angle with a too large step (0.02°). Each step of the scan captures a vertical slice of the pattern.

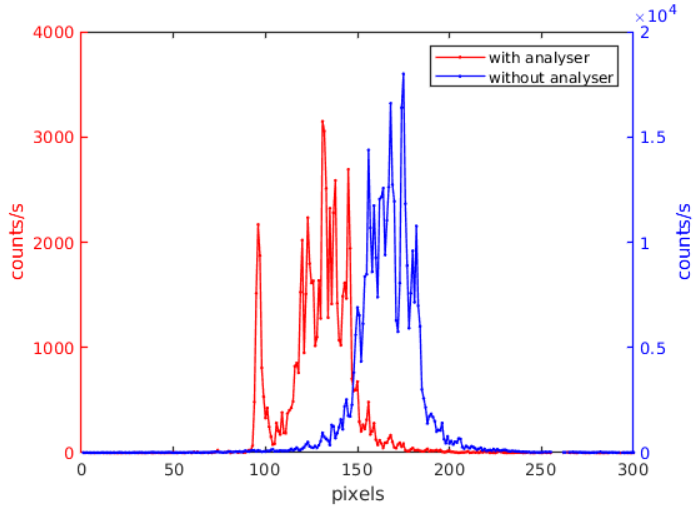

Figure 7: Slice throught the speckle patterns of the Dy (002) reflection, with and without analyser. The efficiency of the analyser is around 20%.

Bulk Dysprosium host a helical antiferromagnetic order between 89 K and 179 K, whose periodicity is incommensurate with the crystal lattice and varies with temperature. Even at the Dy L3 edge, where magnetic scattering is strongly enhanced, the magnetic satellite reflections (0,0,L+/-τ) are hidden in the tails of the Bragg reflections. They can be however measured with the polarisation analyser, which rejects most of the Thomson scattering while retaining the magnetic scattering.

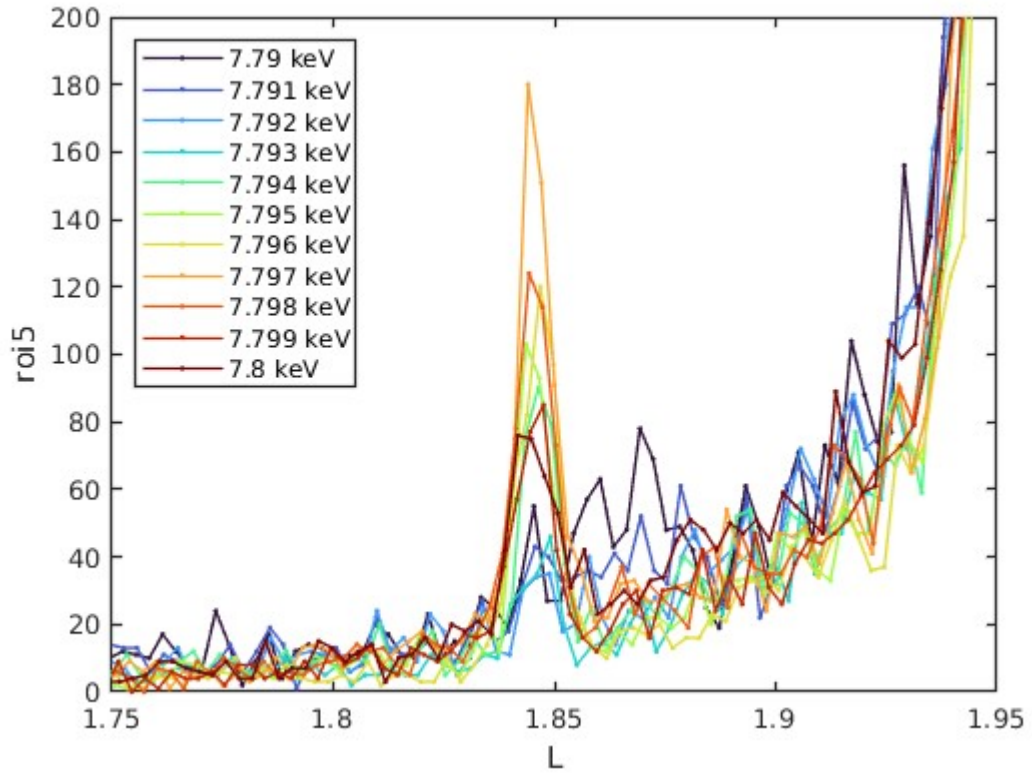

Figure 8: (0,0,L) scans at various energies. The magnetic reflection at L=1.845 is best enhanced at 7.797 keV.

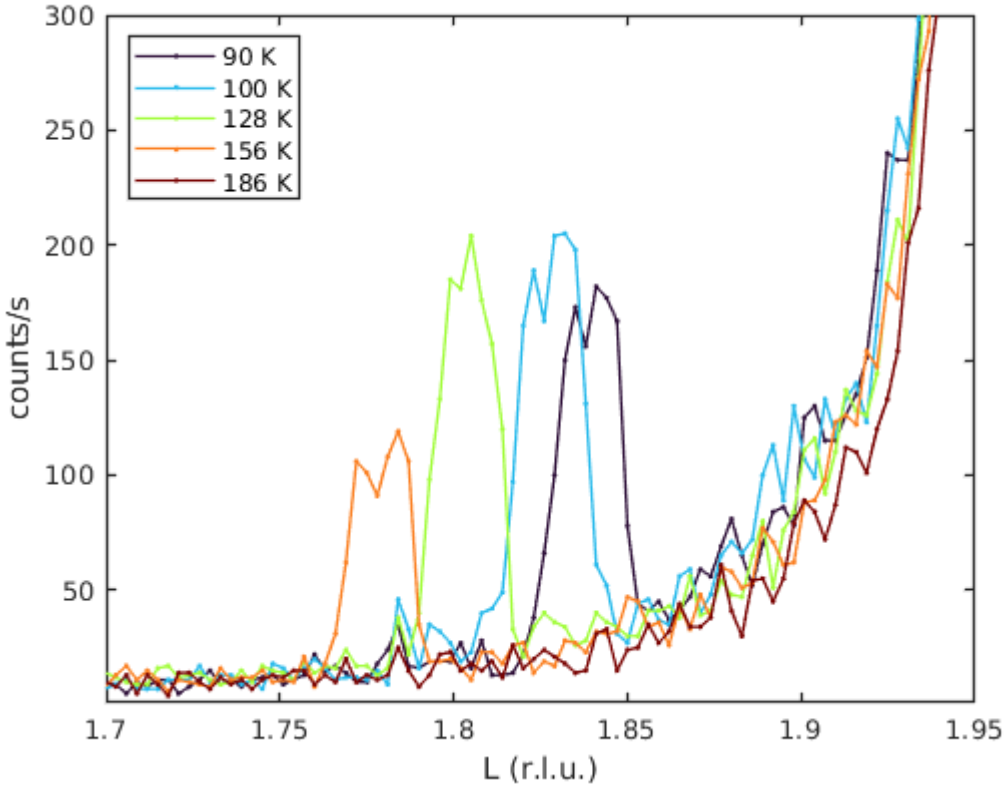

Figure 9: (0,0,L) scans at the L3 edge as a function of temperature. The magnetic propagation vector varies with the temperature. The magnetic reflection has disappeared at 186 K, in agreement with the expected Néel temperature (179 K).

We tried to record speckle patterns of the Dy(002) and Dy (0,0,2-tau) at the same position on the continuous film, but there is no way to know by how much the beam drifted between both angular positions. Nevertheless,

a general idea of the speckle pattern was obtained. For each reflection, 2 orthogonal slices of the reciprocal space were measured by scanning successively the analyser angle and the sample rocking angle.

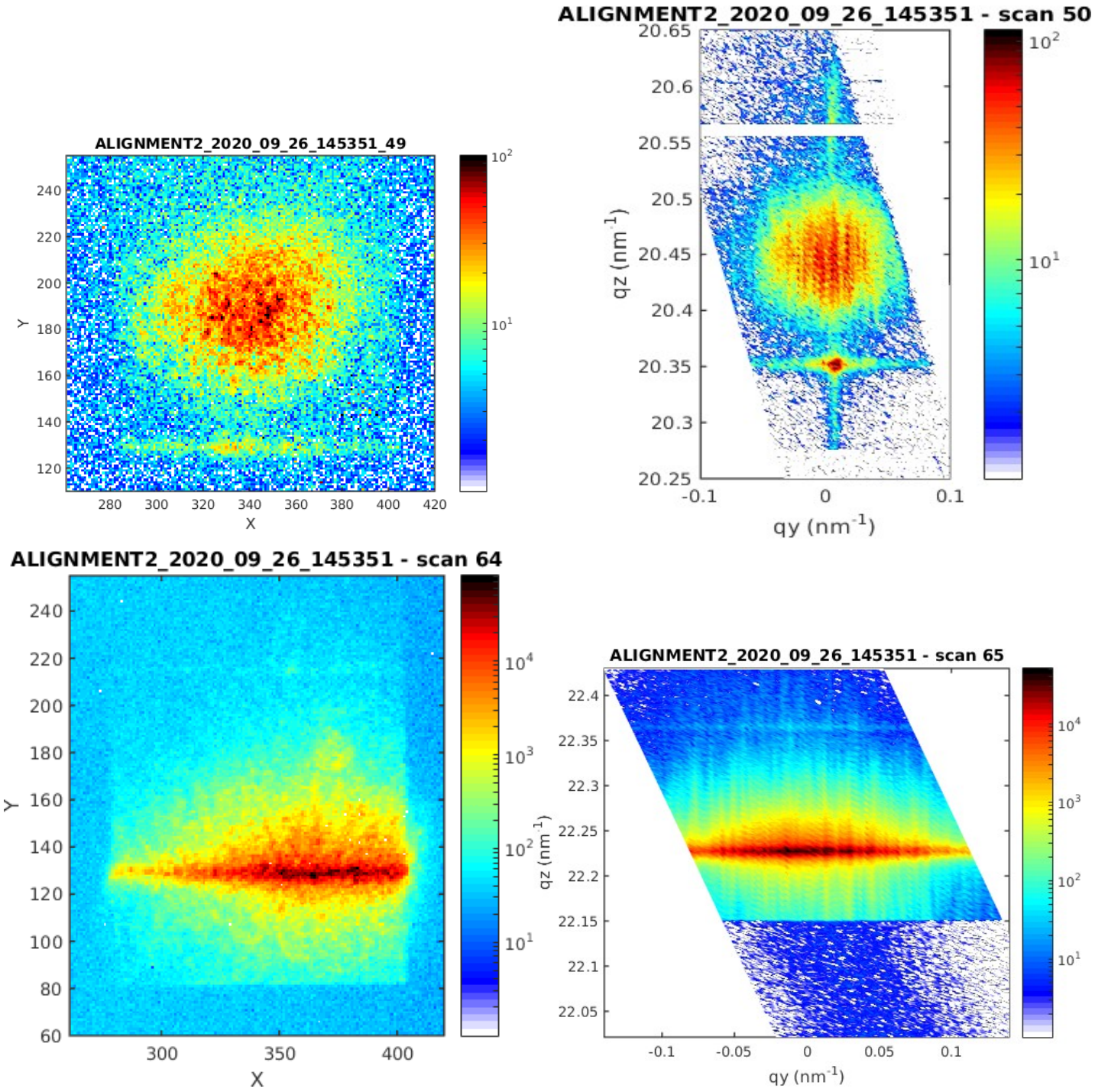

Figure 10: Orthogonal slices of the speckle patterns measured at the magnetic (002-tau) (top) and charge (002) (bottom) reflections. For both reflections, the left slice is obtained by integrating over the analyser angle while the right one is obtained by integrating over the sample angle.

Results on patterned samples

We measured the sample with patterned dots of diameter 2 µm (see optical microscopy in Figure 1). The ID01 *in situ* optical microscope, combined with fast scanning diffraction maps, allows to find a particular dot that had been chosen from SEM images. In addition, the patterned sample allows to precisely position the sample and the beam at the centre of rotation of the diffractometer.

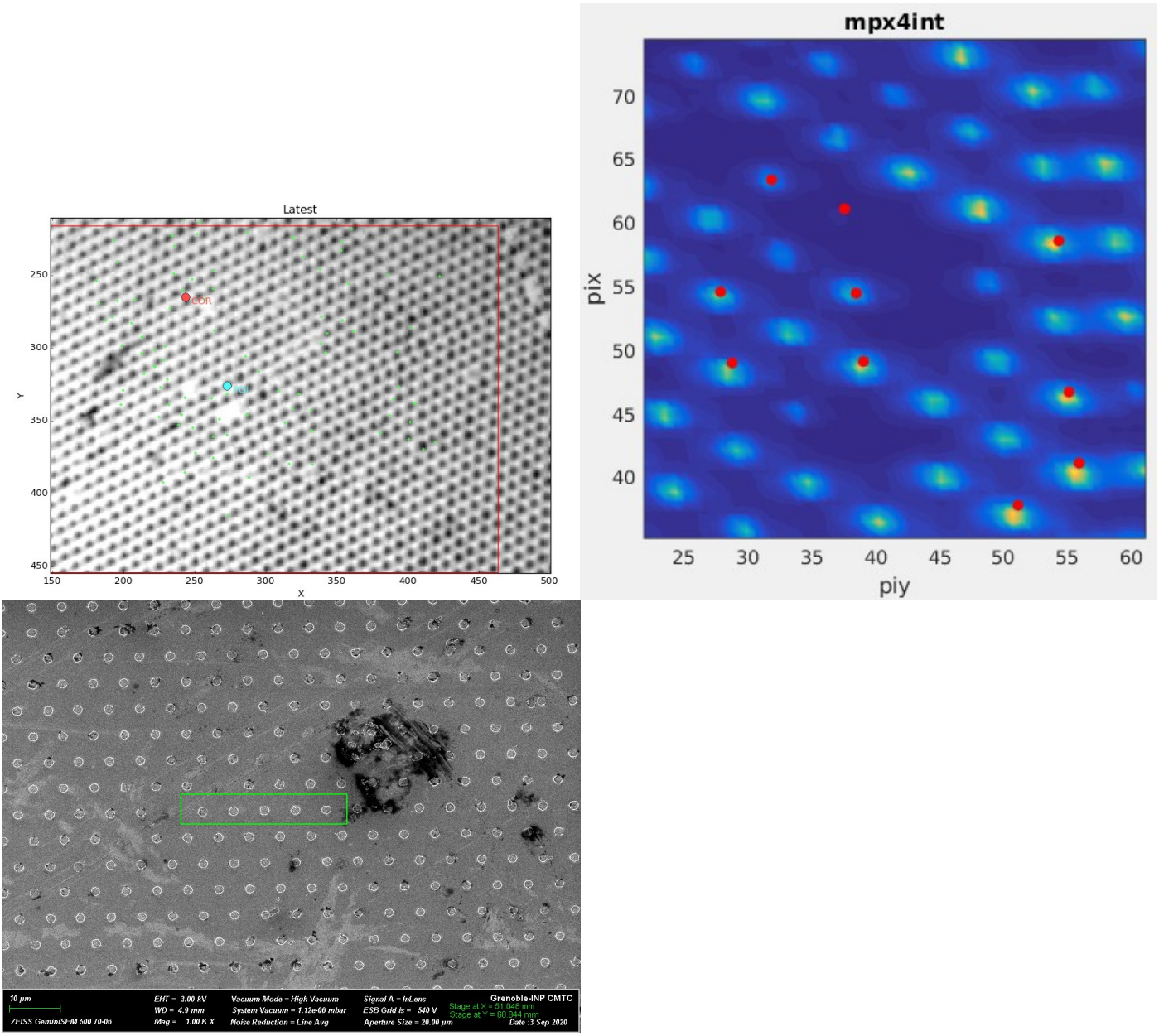

Figure 11: Several views of the same area of the array of 2µm dots. Top left: in situ optical microscope. The red dot marks the centre of rotation. Top right: Scanning diffraction microscopy on the Dy (002) reflection (the red dots mark positions of interest). Bottom left: scanning electron microscopy in the lab.

The diffraction patterns measured at the Dy (002) reflection of the dots were highly speckled, similarly to the ones of the continuous film (Figure 12). This speckled structure indicates the presence of many structural defects in the dots.

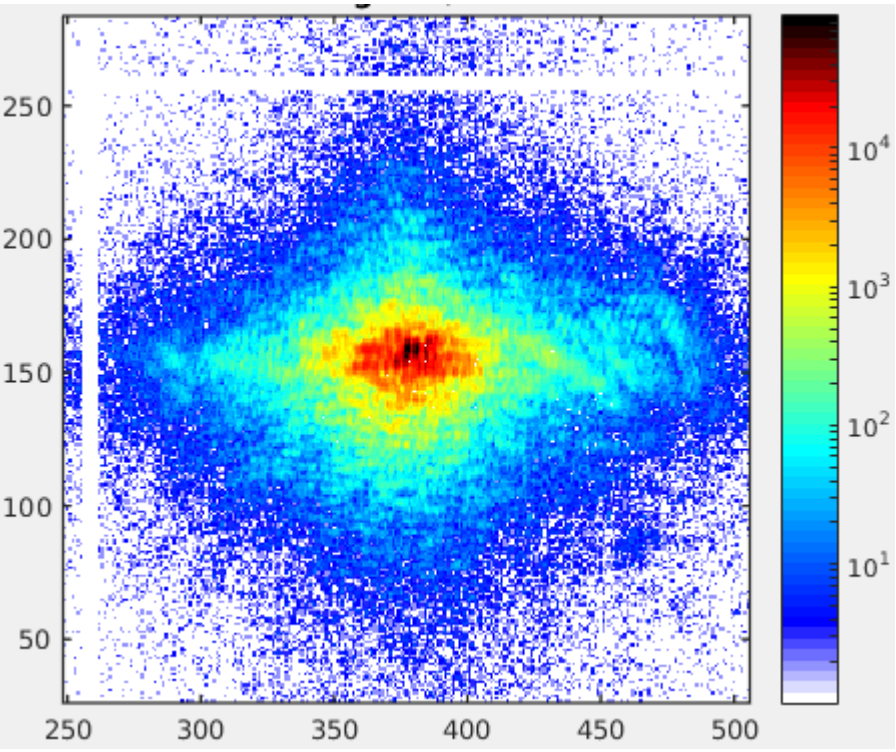

Figure 12: Typical speckle pattern at the peak of the Dy (002) reflection of a dot.

Unfortunately, there was some drift between beam and sample, possibly because of the cryostream, andwe were not able to stay reliably on one dot to align the polariser and look for a magnetic reflection.

Conclusions

On the positive side, our polarisation analyser was used for the first time to extract the rotated polarisation and allowed to measure the magnetic reflection from the continuous film, and to measure its speckle pattern by integrating a scan of its rocking angle. The Ge crystal used for polarisation analysis was of sufficiently good quality to avoid strong distortion of the speckle pattern. This is however quite inefficient, as each pixel column of the detector is measured at a different position of the analyser angle. It would be interesting to use a bent crystal instead of the flat one, in order to integrate the diffraction pattern in a single snapshot. For this, the radius of curvature would need to be D*sqrt(2), where D is the sample-to-analyser distance.

Let us note that, to our knowledge, **the speckle patterns recorded at the magnetic reflections are he highest energy measurements of pure magnetis speckles**. Previously, the higest energy reported for such measurements was the Uranium M4 edge (3.7 keV), in compounds like UO2, UAs, USb, and UGa3, by some of us. **This improvement has been made possible by the EBS**.

The magnetic reflection was found to behave like in the bulk, with similar temperature dependence (a quantitative comparison remains to be done).

We were also able to find pre-selected dots and measure their speckle patterns at the Dy (002) reflection.

On the negative side, beam/sample drift prevented us from measuring the magnetic reflection from the dots. It is probably due to the cryostream, although we did not have time to investigate the issue. For future measurements, a different cooling system could be tried.

Without measurements of the magnetic speckle pattern from individual dots, the initial goal of the proposal could not be done.