EUROPEAN **S**YNCHROTRON **R**ADIATION **F**ACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report

	Experiment title: Investigation of the polar displacement patterns	**Experiment number**: MA 3817
Beamline:	**Date of experiment**: from: 08.02.2018 to: 13.02.2018	**Date of report**: 16.11.2018
Shifts:	**Local contact(s)**: Laurence Bouchenoire	*Received at ESRF:*

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Report:

The material system YMn_2O_5 undergoes a phase transition from a paramagnetic, paraelectric phase (space group *Pbam*) to an incommensurate antiferromagnetic phase at 45 K, as well as to a commensurate magnetic and ferroelectric phase at around 40 K [1]. The high temperature space group is centrosymmetric and cannot explain the observed ferroelectricity (FE) in the low temperature phase. Therefore, the lower symmetry space group $Pb2_1m$ is most frequently used to describe the low temperature phase. The atomic displacements involved in the phase transition have been repeatedly studied, but a structure refinement has not yet been obtained [2]. Other studies claim that the FE is not related to atomic displacements at all, but to orbital ordering instead [3].

To quantify and test atomic displacements during the phase transition at 40 K, and hence to study the origin of the ferroelectricity in this phase, we employed a sensitive resonant x-ray diffraction technique [4] within experiment MA3817 at BM28. The energy dependencies of different reflections have been mapped for temperatures below and above the phase transition. The large unit cell and the relatively high x-ray energy caused a very dense occurrence of interfering Renninger reflections, which made a conventional data collection of energy dependency nearly impossible. Finding a strategy to eliminate the influence of Renninger reflections is time consuming and one reason for loss of beamtime. We learned that we can compensate for Renninger effects and extract the “true” energy dependency by filtering azimuthal scans of approx. 30 points per energy. Compared to energy scans these aziscans are more time consuming and, next to problems with the sample heater, another reason

for the reduced number of reflections that has been recorded during our experiment. Data processing required a background subtraction, peak integration, filtering azimuthal scans and finally extracting the energy dependency for each reflection and temperature (Fig. 1).

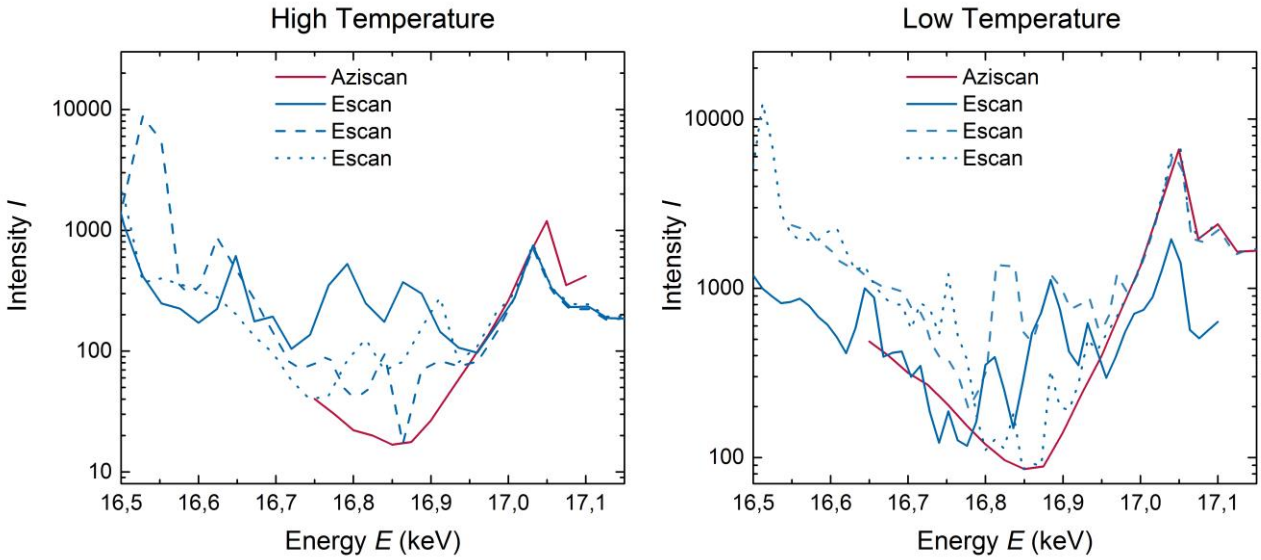

Figure 1*:* Energy scans (blue) show strong influence of Renninger reflections, which superimpose the energy curve of the two beam diffraction case. This could be filtered by means of azimuthal scans (red) to find the sensitive minimum position in the energy dependence of the reflection.

Using these results, we are fitting the isotropic and anisotropic atomic displacement parameters (ADP) for the high temperature phase. Hereby, with various ADP the energy dependency of each reflection is fit to the experimental energy scans. For the anisotropic ADP, simulation and experiment have a good agreement (Fig. 2), except the 115 and 119 reflections, which show strong Renninger dependency. The simulations with the isotropic ADP have lower comparability with the experiment, because the fit is under-determined due to a lower amount of parameters.

For a model-free determination of the FE atomic displacement patterns, the amount of recorded data may not be sufficient. For this reason, we are currently developing a theoretical displacement model describing the FE moment with a coherent motion of the different polyhedra. However, to acquire the complete set of information, further measurements will be necessary.

During additional experimental time at the BM28 lab source, we searched for superlattice reflections predicted for the low-temperature phase $Pb2_1m$ with the aim to use the intensity ratios of superlattice to fundamental reflections to put constraints on our structural model as well as to investigate the feasibility to perform similar resonant diffraction experiments on superlattice reflection. We also anticipated a reduced impact of the Renninger effect on these half-integer reflections. However, these reflections appeared to be too weak for detection in the lab-based measurements.

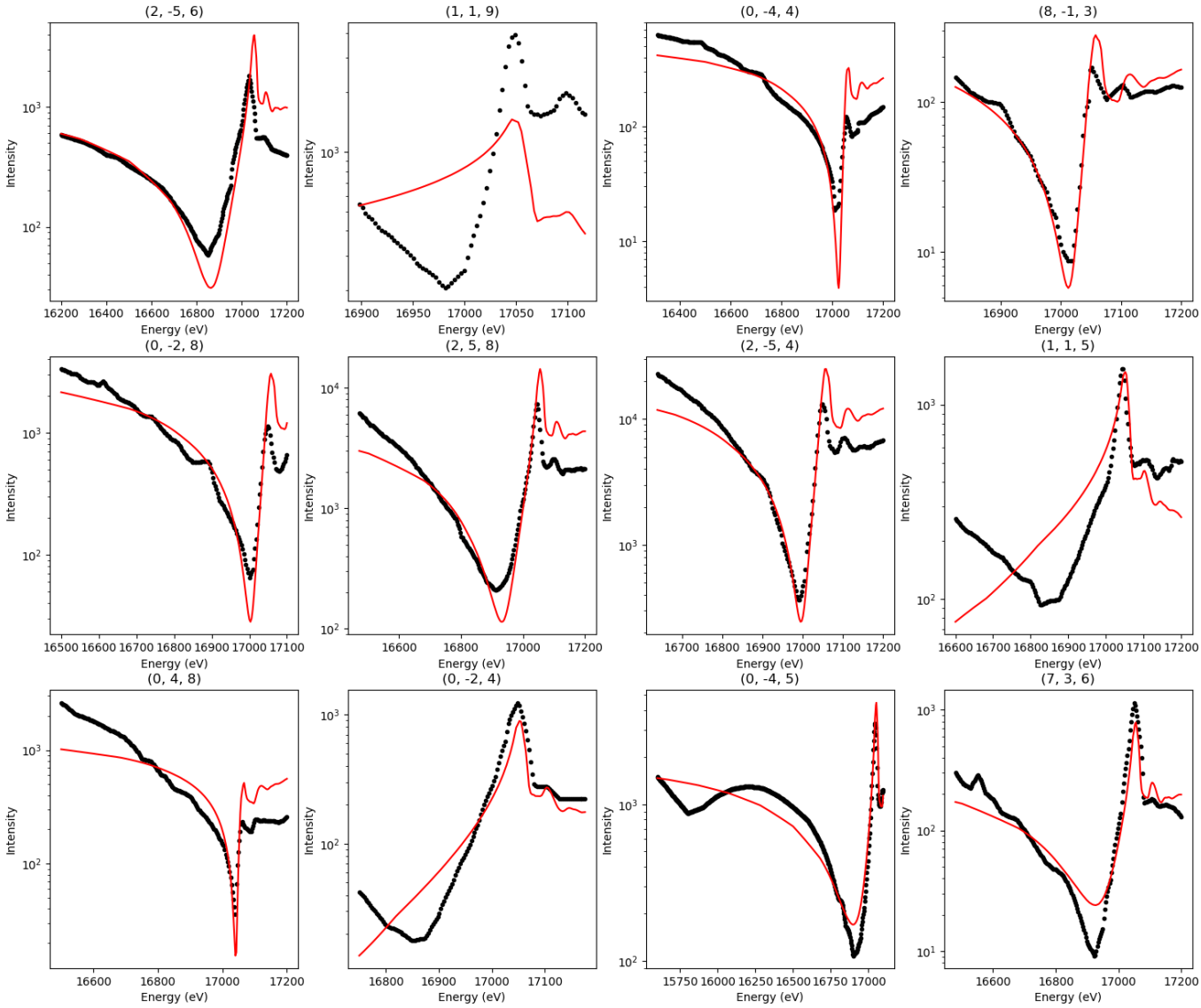

Figure 2: Best result for the simulated energy dependency with anisotropic ADP (red) in comparison to the experimental energy dependency (black) of various reflections.

Filtering out the Renninger effect using azimuthal scans resulted to be a promising approach for a more **robust collection of true integrated intensities** on any material system, which we aim to exploit in future beamtimes. With the analysis of data from this and further beamtimes, the refinement of the thermal displacements should be feasible. Already now, we can see that the thermal displacements of the structural model from literature, which we use to perform preliminary simulations and to identify interesting reflections and energies, were far from reality. Hence, as a first step, the obtained data will provide a better starting model for upcoming investigations and a full structure refinement for the FE phase.

References

[1] S. Partzsch et al. Phys. Rev. Lett. 197 057201 (2011)
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[3] S. Partzsch et al., Eur. Phys. J. Special Topics 208, 133–139 (2012)
[4] C. Richter et al., Nat. Comm. 9, No. 178 (2018)