Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- 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:** Precise diffraction pattern measurements of decagonal Zn-Mg-Dy and Al-Ir-Os quasicrystals.	**Experiment number:** HS-4421
Beamline: BM01A	**Date of experiment:** from: 28/09/2011 to: 01/10/2011	**Date of report:** 25/08/2012
Shifts: 9	**Local contact(s):** Dmitry Chernyshov	*Received at ESRF:*

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Report:

Experiment

The aim of the project was to collect large, high quality datasets for decagonal quasicrystalline Zn-Mg-Dy (d-ZnMgDy) and Al-Ir-Os (d-AlIrOs) samples for a detailed structural analysis. The measurements were carried out at Swiss Norwegian Beam Line (BM01A). KUMA KM6-CH single crystal diffractometer equipped with a CCD detector was used. The wavelength, the oscillation angle and the resolution was chosen as 0.6980 Å, 0.1° and 0.6 Å respectively. Diffraction experiments were done at room temperature.

Before the experiments a standard periodic crystal with a large unit cell was measured to calibrate the instrumental parameters. Then, two datasets were collected for each crystal (decagonal quasicrystalline Zn-Mg-Dy and Al-Ir-Os samples) one with a heavily attenuated beam (not to oversaturate the strong reflections) and one with low attenuation (in order to measure as many weak reflections as possible). In both datasets a full coverage of the Ewald's sphere was necessary for absorption correction. These datasets were then combined to obtain one dataset with very strong and very weak reflections together, with an intensity difference more than the dynamical range of the CCD detector would normally allow.

Results

For both crystals, the datasets were combined and merged in Laue group 10/*mmm*. For d-ZnMgDy, 620 independent reflections were observed after merging with an R-value of 4%. The pattern could be indexed by five unit vectors with $a_{1\text{-}4}$=3.19 Å and a_5=5.21 Å. For d-AlIrOs, 7160 unique reflections were measured and the internal R-value was found to be 8%. This high value seems to originate from the enormous number of weak reflections measured (around 3600 reflections with I/sigma<1). The lattice parameters were $a_{1\text{-}4}$=4.05 Å and a_5=16.86 Å. In both cases significant diffuse scattering intensities were observed.

For d-ZnMgDy, the structure solution was carried out by the SUPERFLIP software package (Palatinus & Chapuis, 2007) which utilizes the charge flipping algorithm (Oszlanyi & Suto, 2004). The resulting electron density (ED) revealed 4 layers along the periodic direction with 3 of them being symmetry independent. That indicates a 1.3 Å distance between the layers which is smaller than the Al-*TM* (transition metal) decagonal quasicrystals (2 Å, see Steurer, 2004).

The different layers in the structure exhibit different tiling types. The ED peaks at layers at x_5=0.25&0.75 decorate the vertices of a rhomb Penrose tiling whereas for the x_5=0.5 a pentagon tiling explain the structure. The layer at x_5=0 however cannot be explained by vertices of any tiling in the same local isomorphism class but the peaks are rather situated on the edge centers of both rhomb and pentagon tiling.

This unique behaviour is explained by the help of the higher dimensional crystallography. The higher dimensional atoms, or occupation domains (ODs), of the x_5=0.25&0.75 layers are as expected from an ideal Penrose rhomb tiling: There are 4 ODs located on the body diagonal of the higher dimensional unit cell. For the x_5=0.5 there is only one OD centered on the origin, confirmed by the pentagonal tiling fit for this layer. One of the ODs on the x_5=0 layer however is located on the center of the face formed by x_2 and x_3 (and symmetry equivalent positions). This is the first time such an OD is found in a decagonal quasicrystal and points out to a very different arrangement of atoms compared to the d-Al-*TM* structures.

Based on these results, a cluster of 24 Å in diameter was specified as the main building block. The cluster contains around 120 atoms with Dy atoms being in the center. The decoration of the clusters according to a tiling reported by Masakova *et al.* (2005) was used to cover the ED map. Embedding of this covering in higher dimensions with the 5D Yamamoto basis (Yamamoto, 2008) was then carried out to construct the initial model. This model is used as a starting point for the refinements done by a software package released by Yamamoto with the same paper. The refinement procedure is currently in progress, early results indicate that the initial cluster model is valid with some variations.

The d-AlIrOs phase was first discovered and solved by Katrych *et al.* (2007). The structure exhibits eight-layer periodicity with the quasiperiodic packing of the 20 Å clusters, typical for d-Al-*TM* structures. The analysis of the data is currently on going.

In summary, high quality data for two decagonal phases were collected with a satisfying amount of weak reflections. Such datasets are crucial for QC structure analysis, which can only be realized satisfactorily in a synchrotron resource, equipped with a state of the art detector technology.

Following these results, in-situ high temperature single crystal X-ray measurements for d-ZnMgDy is planned and a proposal will be sent to the SNBL. The main aim for these experiments will be to investigate high temperature stability and the phase transition behaviour of the decagonal phase. Our earlier work (Oers & Steurer, 2011) on powder X-ray measurements suggest that a phase transition from the quasicrystalline phase to a periodic phase takes place upon heating a single phase d-ZnMgDy sample to 400°C.

References

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Masakova, Z., Patera, J. & Zich, J. (2005). *Journal of Physics a-Mathematical and General* **38**, 1947-1960.

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