

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.

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<https://www.esrf.fr/misapps/SMISWebClient/protected/welcome.do>

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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

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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.



	Experiment title: Magnetic Interactions in FeAs type superconductors	Experiment number: HE 3535
Beamline:	Date of experiment: from: 13/7/2011 to: 18/7/2011	Date of report: 14/10/2011
Shifts: 18	Local contact(s): Dr. Flora Yakhou	<i>Received at ESRF:</i>

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Three years ago a new variety of superconducting materials such as RFeAsO and RFe₂As₂, containing iron arsenide layers was discovered (Fig. 1) [1]. As in the layered cuprates, the FeAs layer must be doped for the material to become superconducting. This experiment aimed to investigate the fundamental magnetic structure of the end member FeAs which displays a helical spin density wave. It is this end member that is the origin of the strong magnetic exchange interactions and the resulting magneto-elastic coupling. The iron pnictides display an overlap between magnetic order and superconductivity [2] and it is thought that the magnetic spin density wave plays an important role in the development of superconductivity.

We were fortunate to have high quality single crystals of FeAs grown by vapour transport by Prof. Ziq at the Department of Physics, King Fahd University, Dhahran, Saudi Arabia. The crystal and magnetic structure of FeAs was determined many years ago by powder neutron diffraction [3,4]. The structure is MnP type with a helimagnetic structure adopted below 77 K with a wavevector of (0, 0, 0.365). In an earlier attempt we failed to observe any magnetic satellites with this wavevector using either hard or soft x-rays. In this experiment we succeeded in observing magnetic satellites at a larger wavevector, confirmed by a very recent redetermination of the structure using polarised neutron diffraction [5].

A sample was mounted on the ID08 soft x-ray diffractometer with a natural (0,0,*l*) face surface normal. A scan along the *c*-axis, (coincident with the surface reflectivity) at a base temperature of 20 K found two resonant magnetic diffraction peaks at (0, 0, 0.389) and (0, 0, 0.611), see Fig. 2. These are

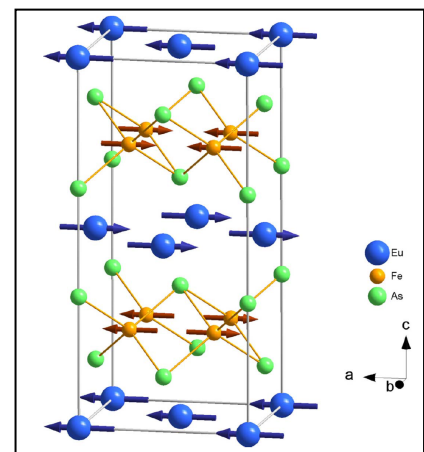


Fig. 1 The crystal structure of EuFe₂As₂ highlighting the FeAs layers found within iron pnictide materials.

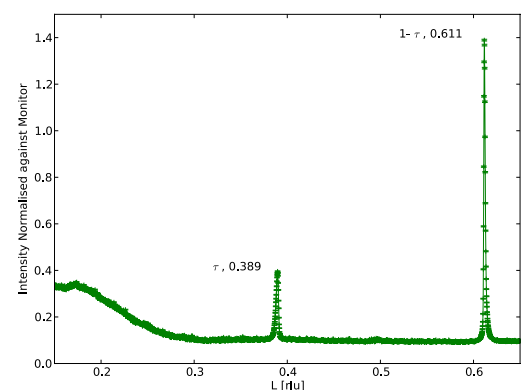


Fig. 2 A *L*-scan along (0,0,*l*) of FeAs at 20 K. The scan shows both the (0, 0, τ) and (0, 0, 1- τ) reflections with $\tau = 0.389$.

consistent with the $(0,0, \tau)$ and $(0,0, 1-\tau)$ reflections with $\tau = 0.389$. The inset of Figure 3 shows the singlet peak in a scan along $(0,0, L)$ using polarised x-rays in σ geometry. Studies of the polarisation dependence confirmed the magnetic origin of these peaks. Fig. 3 also displays an energy scan (at constant Q) through the $(0,0, \tau)$ reflection confirming the resonance at the Fe $L_{3,2}$ absorption edges. The strong resonances confirm that the magnetism originates from electrons within the 3d band of iron, as expected, whilst the complex energy splitting strongly evident at the L_3 may indicate a charge disproportionation or a strong structural distortion at inequivalent iron sites. We also collected data using both left- and right-hand circular radiation and we are hoping that fitting will enable us to determine the absolute chirality of the spin spirals in FeAs.

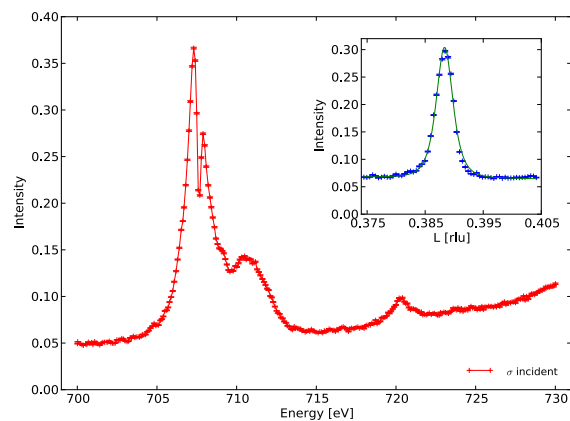


Fig.3 An energy scan through the iron L_3 and L_2 absorption edges at constant wavevector of the $(0,0, \tau)$ reflection of FeAs at 15 K. The inset shows an L scan through the resonantly enhanced reflection obtained at an energy of 707.5 eV.

Figure 4 shows the result of our studies of the temperature dependence of the $(0,0, \tau)$ reflection. The wavevector of the incommensurate spiral spin density wave (effectively τ) varies with temperature. The figure shows two separate temperature dependencies overlaid showing τ varying from 0.388 at 15 K up to 0.401 at 70 K before the collapse of the spiral spin density wave at 71 K.

These are remarkable results. This is the first time the magnetic satellites in FeAs have been observed using x-ray diffraction. The moment of only 0.5 Bohr magnetons is very small but has been observed using resonant soft x-ray scattering due to the extremely large resonant enhancement of the magnetic reflections at the iron L_3 and L_2 edges. Repeating such measurements at the arsenic L_3 and L_2 edges we failed to observe any resonant reflections. This shows that the arsenic atoms are not magnetic due to hybridisation. This probably reflects the fact that the 3d shell of As is completely full and hence there is no observable resonant enhancement at the L edges ($2p - 3d$).

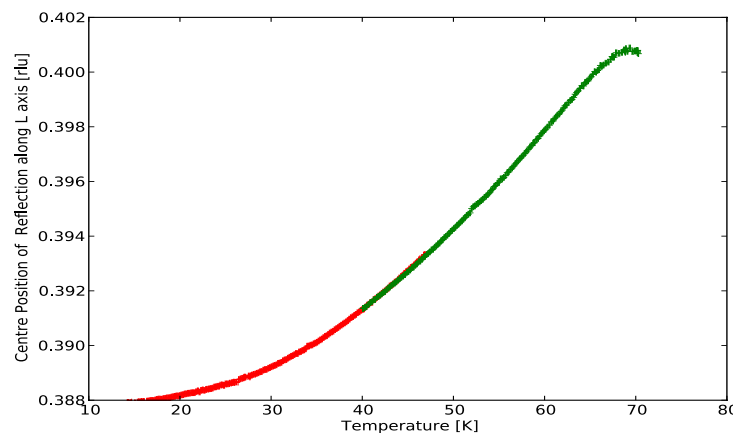


Fig. 4 Variation of the incommensurate spin spiral wavevector with temperature.

We are now hoping to combine these results with full polarisation analysis using the RASOR diffractometer at Diamond. By studying the variation of intensity with both incident and scattered x-ray polarisation we believe it may be possible to determine whether the spirals are circular or elliptical, and to determine the ellipticity. This research will form part of the doctoral thesis of Mr. Thomas Frawley and we are confident that a high quality publication will result in an internationally competitive journal.

[1] Y. Kamihara *et al.* J. Am. Chem. Soc. **130**, 3297 (2008) [2] R. H. Liu *et al.*, Nature **459**, 64 (2009) [3] K. Selte and A. Kjekshus, Acta Chem. Scand. **23**, 2047 (1969). [4] K. Selte *et al.*, Acta Chem. Scand. **26**, 3101 (1972) [5] E.E. Rodriguez *et al.*, Phys. Rev. B **83**, 134438 (2011).