| <u>ESRF</u>        | <b>Experiment title:</b><br>CDW transition in $(Er,Lu)_5Ir_4Si_{10}$ compounds | Experiment<br>number:<br>HS 1732    |
|--------------------|--|-------------------------------------|
| Beamline:<br>ID10a | Date of experiment:from: 26. June 2002to: 2. July 2002                         | Date of report:<br>22. January 2003 |
| Shifts:<br>18      | Local contact(s):<br>Dr. Federico Zontone                                      | Received at ESRF:                   |

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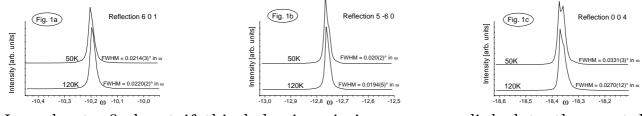
## Report:

The group of compounds with the composition  $(\text{Er},\text{Lu})_5\text{Ir}_4\text{Si}_{10}$  is known to possess a rich phase diagram with a number of temperature and composition dependent phase transitions [1,2,3]. These structural phase transitions are accompanied by unusual changes in electrical behaviour. For our experiment we selected  $(\text{Er}_{0.66}\text{Lu}_{0.34})_5\text{Ir}_4\text{Si}_{10}$  for which a single phase transition from a normal tetragonal (P4/mbm, a = 12.51Å, c = 4.20Å) structure into a commensurately modulated structure at 102K was known beforehand [3]. Similar compounds had been studied before, but the refinement of the modulated structure has failed so far. The primary purpose of our experiment was to investigate any possible changes in symmetry during the phase transformation, which may show as a change in lattice parameters and hence in alterations of reflections. For our experiments the beamline ID10a had to be equipped with a standard four-circle diffractometer with a point detector attached. The selected wavelength was 0.5Å. For cooling we used our own closed-cycle He-cryostat because in contrast to

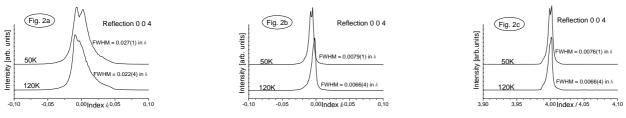
crystal movements. It took two days for the setup to reach an operable state. Upon cooling down to 50K we recorded several reflection profiles in different directions to obtain as much spatial information as possible. The profiles were primarily focussed on the main reflections and their possible splitting but the satellites were also checked to verify the q-vector. After the work at 50K the temperature was raised to 120K to allow comparisons between observations above and below the transformation.

the cryostat available at beamline ID10a it remains mechanically stable during the

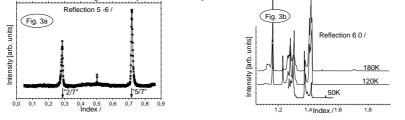
The comparison of  $\omega$ -scans for a few reflections (Fig. 1) shows that the reflections widths at 50K are generally slightly larger than those as 120K. For the 004 reflection even a splitting of the reflections becomes visible (Fig. 1c).



In order to find out if this behaviour is in some way linked to the crystal lattice additional q-scans in prominent directions were recorded. The only reflections for which a splitting could be found were the 004 and the 006. All other reflections scanned show the behaviour known from their  $\omega$ -scans. Scans of the 004 reflections along a<sup>\*</sup>, b<sup>\*</sup>, and c<sup>\*</sup> reveal the anisotropic nature of the splitting.



Though visible in all three directions (Fig. 2) it is most pronounced along a<sup>\*</sup> (Fig. 2a) but in this direction the reflection is also significantly broader. Above the transformation temperature the splitting is at least reduced if not gone, but the quality of the crystal does not allow a definite conclusion. In particular the scan along a<sup>\*</sup> (Fig. 2a) seems to suggest the presence of some remaining splitting at 120K. If the splitting at 50K was due to a lowering of symmetry the deviation from 90° would be  $0.02^{\circ}$ .



Scanning satellites revealed two different properties. One kind of them is normal (Fig. 3a) with sharp profiles, the other produces a diffuse structure (Fig. 3b). The first kind disappears above the phase transition, the second kind remains (Fig. 3b). It is not clear whether this intensity is due to a property of the crystal or of the experimental setup.

The experiments have provided an upper bound of  $0.02^{\circ}$  for a possible monoclinic distortion. Diffuse scattering features were observed for the first time. Crystals of better quality are required for a more detailed analysis of these phenomena.

[1] Galli F., Ramakrishnan S., Taniguchi T., Nieuwenhuys G.J., Mydosh J.A., Geupel S., Lüdecke J., van Smaalen S. (2000) Phys. Rev. Letters 85, 158-161

<sup>[2]</sup> Becker B., Patil N.G., Ramakrishnan S., Menovsky A.A., Nieuwenhuys G.J., Mydosh J.A. (1999) Phys. Rev. B59, 7266-7269

<sup>[3]</sup> van Smaalen S., Daniels P., Galli F., Feyerherm R., Dudzik E., Nieuwenhuys G.J., Mydosh J.A. (2002) J.Phys. IV France 12, Pr9-347-350