ESRF	**Experiment title:** Study of valence in heavy-fermion systems by XAS in pulsed magnetic fields up to 30 T	**Experiment number**: HC1040
Beamline:	**Date of experiment**: from: 29/01/2014 to: 04/02/2013	**Date of report**:
Shifts:	**Local contact(s)**: MATHON Olivier	*Received at ESRF:*

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Report:

The experiment HC1040 was the continuation of the experiment HE3934: it enabled us to carry on our study of valence by XAS in the heavy-fermion materials $YbRh_2Si_2$ and $YbCu_2Si_2$ in pulsed magnetic fields up to 30T and down to 2 K.
Simultaneous XAS and resistivity measurements were performed using the standart 4-point technique. Four electrical contacts (made by spot welding and reinforced by silver paint, cf. Fig. 2) were then systematically added to the samples to check the sample temperature and the field-induced transitions. Single crystals polished to thin platelets (thickness < 20 µm) were mounted directly in the He-flow cryostat, on Si substrates with a hole for the x-ray beam. Magnetic field was applied along both *a*- and *c*-axis directions to test if the valence in each of these two systems is intimately connected to the magnetic anisotropy, and thus to their magnetic properties.

Two days were first dedicated to solve some issues raised in our previous experiment (HE3934). The difficulty of making homogeneously thin single crystals leads to very small size (~ 100 µm) of the samples and makes the experiment complex. Previously to the measurements of XAS spectra, we then decided to systematically perform x-ray absorption maps of our samples to determine the best position (the most homogeneous, with thickness adjusted to one absorption length) on each sample.
Half a day was then devoted to establish the best acquisisition mode including the synchronization of the beam with the triggering of both the magnetic field pulse and the detector. The mode with one acquisition

window of 4.9 ms centered on the maximum field value was preferred to series of magnetic field pulses (5 to to 10 pulses) with shorter acquisition windows (~1 ms).
The temperature of each contacted sample was checked carefully thanks to simultaneous resistivity measurements, as performed in the previous experiment (see experiment report HE3934). To avoid any beam heating of the sample, the beam heat load was reduced by selecting a restricted range of incident energies and by adding aluminium foils before the sample in the incident beam path.

Fig. 1 shows the difference graphs of XAS spectra measured for $YbRh_2Si_2$, with the beam (and the field) parallel to the *a*-axis at zero field and 30 T between 2 and 180 K. These results are quite noisy. We first ascribed this effect to the very small size of our sample (~ 40 µm) which might be more sensitive to the field pulse induced vibrations of our device. These vibrations might have not been detected before on the other larger samples (~1-2 mm for $YbRh_2Si_2$ and $YbCu_2Si_2$, ***H*** // ***c*,** experiment HE3934).

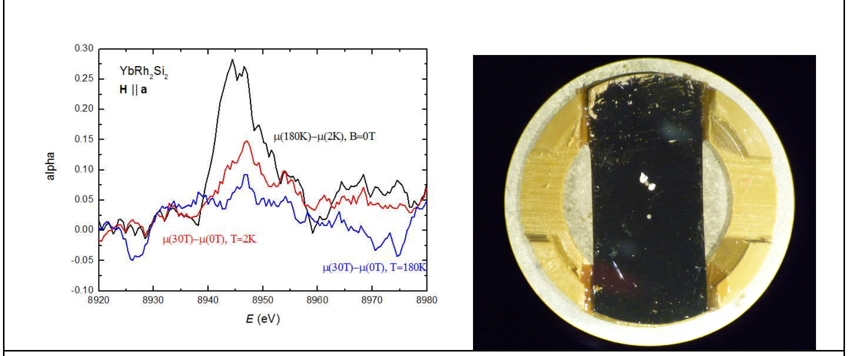

*Fig.1: (left) Difference between XAS spectra of the $YbRh_2Si_2$ sample, **H** || **a**. (right) Photo of the experimental set-up: the sample glued on a silicon support, and a hole to measure the direct beam.*

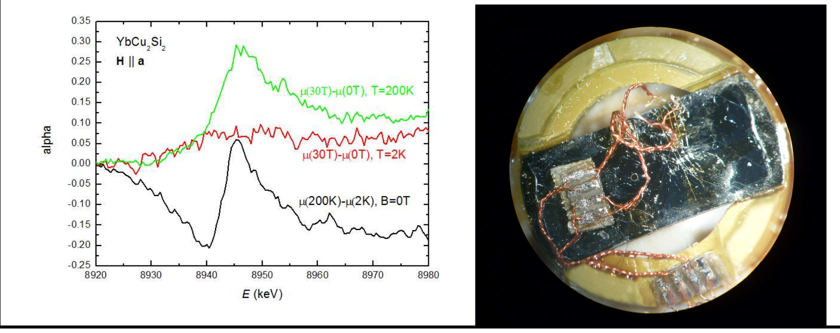

*Fig.2: (left) Difference between XAS spectra of the $YbCu_2Si_2$ sample, **H** || **a**. (right) Photo of the experimental set-up: the sample with four contacts glued on a silicon support, and a hole to measure the direct beam.*

We then focused our attention on the $YbCu_2Si_2$ compound (sample size ~ 1 mm), applying the field along the *a*-axis.
Fig. 2 shows the difference graphs of XAS spectra measured for $YbCu_2Si_2$ at zero field and 30T between 2 and 200K. Unfortunately, the results are also very noisy. The sample being large enougth to be not sensitive to pulse induced vibrations, another issue was suspected. Numerous efforts were done to improve the quality of our data, however all unsuccessful. Finally, the problem was identified: a twisted pair of copper wires supposed to be glued inside the sample stick was no more glued. It then moves and vibrates inside the sample stick along the incident beam path damaging the XAS spectra. It was however too late for the ESRF sample environment to repair the sample stick before the end of our experiment. The design of a new sample stick and new sample holder, which will avoid this problem, has already been discussed with the ESRF sample environnement and is planned to be built for the future experiment.

Despite this technical problem, this experiment was very useful to solve outstanding issues and to establish the feasibility of such a complex measurement combining ED-XAS with high pulsed magnetic field. It enabled us to plan our futures studies: next step is now to obtain high quality data on the two systems mentioned above applying the magnetic field in both *a*- and *c*-axis directions and to investigate a third system $YbPd_2Si_2$ up to 30T on ID24. Samples with larger and more homogeneous surface will be prepared using a better controlled mechanical polishing allowing to reach 10 µm thick.