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:** Seeing x-ray natural circular dichroism in a charge-ordered prototype cuprate superconductor La1.875Ba0.125CuO4	**Experiment number**: 35302
Beamline: ID12	**Date of experiment**: from: Jan. 20, 2015 to: Jan. 27, 2015	**Date of report**: Feb. 27, 2015
Shifts: 21	**Local contact(s)**: WILHELM Fabrice	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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(All coauthors are experimentalists.)

Report:

For this beamtime, we initially proposed a temperature- and angle-dependent hard x-ray circular dichroism (XCD) study on a high-temperature cuprate superconductor, $La_{2-x}Ba_xCuO_4$ (x=1/8), as a continuation of the former experiment at the same beamline. Our objective is to reproduce the results we obtained last time regarding the observation of the XNCD signal and its temperature dependence and obtain direct experimental supports for its XNCD nature by the proposed integrated study.

The first 3 shifts were budgeted exclusively for beamline recommissioning and optimization after the holiday shutdown. We first loaded in our samples on Feb. 21st. We soon realized that the signal detection of the experimental setup scheme has been changed significantly since our last experiment, which led to an increase of the noise level to ~2 times from last time given similar experimental settings. This necessitated 12 hrs for each scan in order to obtain a similar level of statistics as last time. Because of the limited amount of beamtime, we had to adjust our original only focus on some key temperature and angle points.

During the discussion with Dr. Rogalev, we realized an alternative interpretation of the dichroic signal that we found last time emerge below 42 K and had (prematurely) ascribed to CD. Because there is a structural phase transition around the same temperature, the appearance of this feature could well be due to a structural linear dichroism that has different manifestations in different structural phases. Fortunately, a similar structural phase transition occurs around 240 K, which provides a way for us to assess the relevance of linear dichroism to the structural phase transition in the absence of expected CD associated with the charge ordering at low temperature. After this first step, we planned to start the angle dependence study. The sample tilt angle (θ) dependence can allow us to conclude on the CD nature of this signal because for uniaxial crystals any CD signal is expected to vary as $3\cos 2\theta - 1$ different from the linear dichroism. Nevertheless, because of the increased overall noise level of the detection as mentioned previously, this angle dependence is expected to the bulk part of our beamtime.

Related to the first step, we started a measurement first at room temperature (RT) in order to obtain dichroic spectra in the tetragonal structural phase above 240 K, and wished to compare with the one later measured in another tetragonal structural phase below 42 K (for which linear dichroism is expected to be similar). Before we had time to finish the long scan at RT with acceptable statistics, however, a catastrophic failure occurred to the synchrotron which completely changed the fate of our beamtime.

A major beam dump occurred at 5:45am on 23rd, approximately 38 hrs after beginning our experiment. It was followed by ~20 successive beam dumps within the later ~30 hrs. After this horrible period of time, user operation resumed, but with no fast feedback on the electron beam. The lack of this fast feedback led to an erratic motion of the x-ray beam, which turned out to be fatal for our experiment on a sample with macroscopic inhomogeneity on the scale of 100 microns. Fig. 1 shows typical data obtained at two different types of "bad" time in this period, in comparison to the "good" data obtained before the incident as well as on the last day of our beamtime (see the next paragraph). As marked in Figs. 1c & 1d, there were periods in which the electron beam was less unstable (c) and extremely unstable (d). The noise level seen in the measured absorption (black and yellow) and dichroism (red) curves, in neither case, is anywhere near the "good" results (green), while the level of badness clearly was correlated with the beam instability (cf. Figs. 1a & b). With no doubt, measurements were not meaningful under such a ring operation condition (although it might still be ok for other measurements).

On the last day of our beamtime (26th), the fast feedback was turned back on and the experimental noise level recovered close to (although still worse than) the level before the incident. We managed to continue the 1st step of our experimental plan by measuring dichroic spectra in another tetragonal structural phase below 42 K. As we can see in Fig. 2, there is a clear difference between two dichroic spectra beyond the error bars, and this difference spectrum looks grossly similar to the spectrum we measured last time (after taking the difference of the dichroic spectra measured across 42 K). This result, if substantiated, would provide a good indication that the dichroic feature appearing below 42 K is not related to the structural linear dichroism. Unfortunately, given the current statistics, the data is not sufficient (albeit suggestive) to support this conclusion.

In summary, our planned experiment was severely impacted by the occurrence of the "once-per-decade" incident, to such an extent that no conclusive results have been obtained regarding the confirmation of our initial observation and further evidence for its CD origin--the main objective of this proposed beamtime. Although this is not achieved, a further understanding and consistent indication have been obtained that made it promising. Other than measuring the temperature dependence (as we have attempted to the best we could this time), a more definitive conclusion as to the CD origin of the dichroic feature has to be built upon a systematic θ dependence study (which we had intended but had no chance to start). All these should be able to be accomplished within 18 shifts, given a normal user operation with fast feedback on and the S/N level attainable from the current signal detection scheme. It is in the interest of both ESRF and us to conclude this project with this additional amount of beamtime, in light of the promise for major scientific impact held by our finding as well as the resources that have been devoted to this project by both parties.

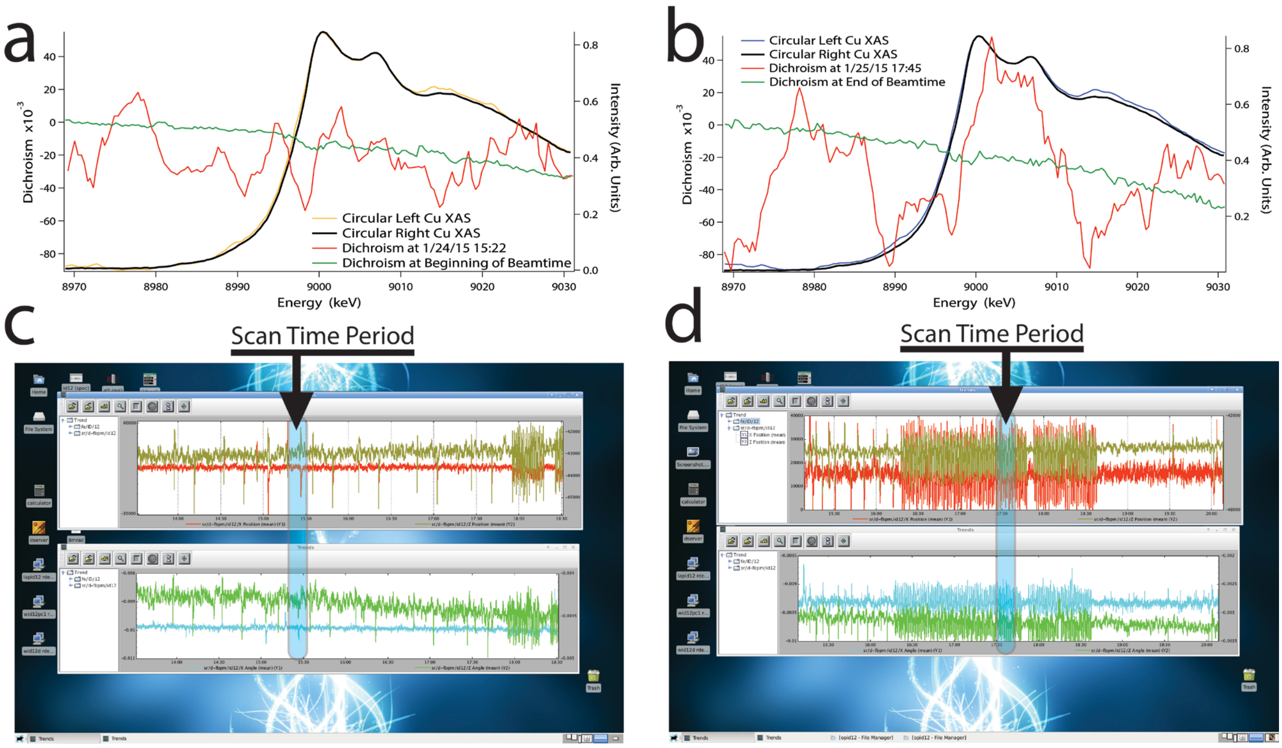

Fig. 1 a & b) XAS [black and (yellow, blue)] and corresponding dichroism (red) for specific time specified in c & d, as well as dichroic spectra from scans obtained at the beginning of the beamtime (before the incident) to illustrate relative noise level. c & d) Screenshots of ring electron beam parameters with highlighted blue regions in which scans used for a & b were obtained on 24^{th}~25^{th}.

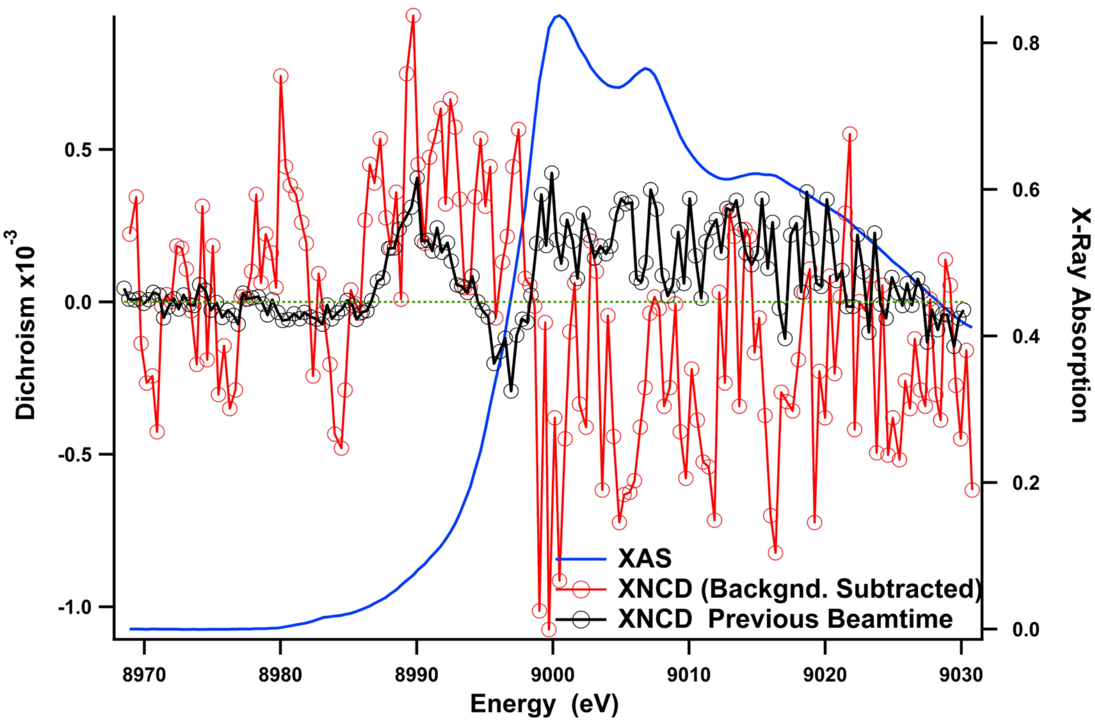

Fig. 1 Difference (red) between dichroic spectra measured in the tetragonal phase above 240 K and the one below 42 K, showing a possible CD signal in agreement with the result obtained from the previous beamtime. XAS spectrum (blue) measured above 240 K is shown as an eyeguide. Note the much increased noise level compared with previous experiment due to the limited time on the scans and change of signal detection scheme used in the experimental setup.