ESRF

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

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report"),*
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

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.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number 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: Investigating the influence of disorder on magnetic, phonon, CDW, and charge excitations with controlled dopant disorder in oxygen doped cuprate La2CuO4+y	**Experiment number**: HC 4168
Beamline:	**Date of experiment**: from: 02/24/2021 to: 03/05/2021	**Date of report**:
Shifts: 18	**Local contact(s)**: Kurt Kummer, Nick Brookes	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*Matteo Rossi, SLAC National Accelerator Lab

*Haiyu Lu, SLAC National Accelerator Lab

*Yonghun Lee, SLAC National Accelerator Lab

*Wei He, SLAC National Accelerator Lab

*Jiajia Wen, SLAC National Accelerator Lab

*Young Lee, SLAC National Accelerator Lab

*Wei-Sheng Lee, SLAC National Accelerator Lab

*Leonardo Martinelli, Politecnico di Milano

*Lucio Braicovich, Politecnico di Milano

*Giacomo Ghiringhelli, Politecnico di Milano

Report:

During this beamtime, we performed high-resolution resonant inelastic x-ray scattering (RIXS) measurements at the Cu L_3-edge on La-based cuprates, La_2CuO_{4+y} (LCO). The LCO were deliberately prepared to exhibit oxygen order and the superconducting transition temperature T_c is 42 K. The objective of the beamtime is to investigate phonon excitations and their interplay with the underlying charge density wave (CDW) state in both the oxygen ordered and disorder states within the same samples. The measurement was first performed on the oxygen ordered LCO sample. Subsequently, the temperature were elevated to room temperature to disrupt the oxygen ordering, followed by a cooling period for RIXS in the oxygen disordered state. During this process the oxygen order superlattice peak located at (0.165, 0.075, 1.5) was monitored directly by the in-chamber photo diode. This allowed us to track and characterize the changes of the oxygen order.

The spectrometer was set at 149.5° geometry to maximize the momentum-transfer for the momentum-dependence measurements. We have also set the energy resolution to ~ 40 meV, in order to clearly resolve the phonon excitations, with reasonable data acquisition efficiency (~ 2 hours per spectrum including the overhead). The polarization was set to vertical (*i.e.* σ-polarization) to maximize the scattering cross section from charges and phonons. We have obtained high quality and high resolution data along the (0,0) - (1,0) direction (i.e. H-direction) that allows to study the CDW and phonon.

We initially observed an unfortunate consequence during our experiments: the oxygen order was susceptible to deterioration when exposed to X-ray illumination at ID32. Figure 1 illustrates the rocking curve of the oxygen order peak at varying X-ray exposure times. It is evident that the peak weakens as the exposure duration increases. Although reducing the exit slit opening size somewhat mitigated the radiation-induced disordering of the charge order, the issue persisted. This unexpected radiation-induced disordering effect has hindered our ability to fully characterize the intrinsic properties of the charge order in the oxygen-ordered state.

It is important to note that we conducted investigations using two different sets of samples, and the identical issue persisted in both cases, ruling out any concerns related to sample quality. Notably, during our preliminary resonant soft X-ray scattering experiments at SSRL, SLAC, we did not encounter this radiation-induced damage issue. We suspect that the considerably higher X-ray fluence at ID32 (attributed to a smaller beam spot, approximately 2.5 µm x 26 µm, compared to 100 µm x 100 µm at SSRL) is likely the primary cause of the observed radiation-induced oxygen disordering.

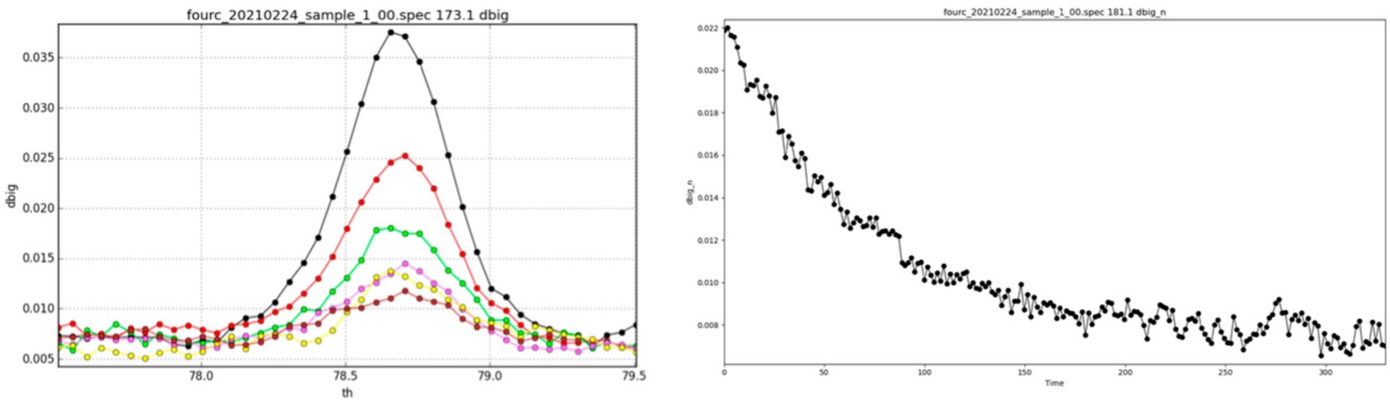

Figure 1. (left) Rocking curve of the oxygen order superlattice peak at different x-ray exposure time. (right) Oxygen order superlattice peak intensity as a function of time.

Nevertheless, we obtain data in the nominally oxygen ordered state. The momentum-dependent RIXS maps, both above and below Tc, are shown in Fig. 2. Notably, we observed the signature of a CDW diffraction peak centred at $Q_{CDW} \sim 0.25$ *r.l.u.* in the quasi-elastic region. This confirms the existence of the CDW state in the oxygen doped LCO. A branch of dispersive paramagnon excitations (energy from > 0.1 eV) and phonon excitations with an energy scale of ~ 0.06 eV can be clearly resolved. Upon comparing the data collected in the superconducting state (20 K) and the normal state (45 K), we observed a slight reduction in the charge order, while the phonon anomaly appeared to exhibit a slight enhancement in the superconducting state.

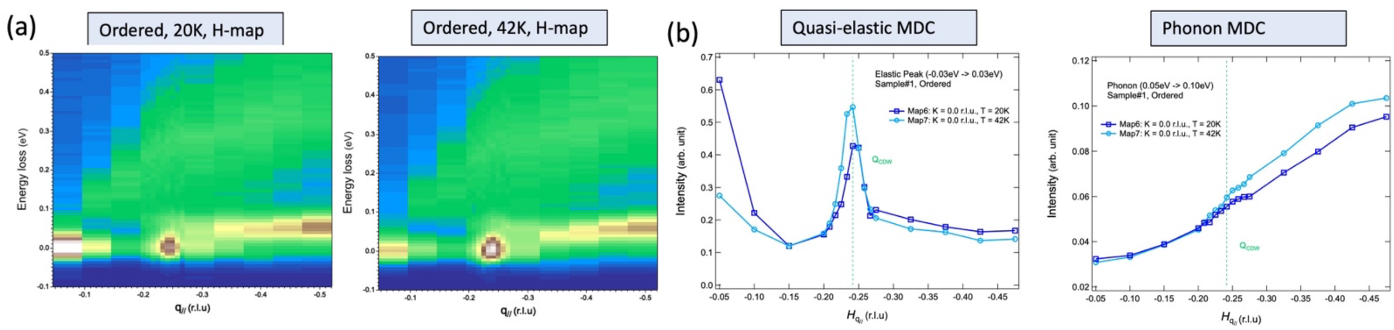

Figure 2 LCO in oxygen ordered state (a) RIXS energy-momentum map along the H-direction taken in the superconducting state (20K) and normal state (42K) (b) Momentum distribution curve (MDC) of the quasi-elastic peak intensity (left) and the phonon intensity (right) taken at the two temperatures. The quasi-elastic peak MDCs and the phonon MDCs are obtained by integrate the RIXS intensity in the energy window between -0.03 eV to 0.03 eV and 0.05 eV to 0.1 eV, respectively.

Next, we disrupted the oxygen order and repeated the measurement. Representative data are displayed in Fig. 3. In both superconducting and normal states, the CDW at 0.25 r.l.u. and the branch of phonon excitation is still clearly visible. However, we discover that the charge order intensity can be dramatically vary at different sample locations (Fig. 3b, left panel). This indicates that the sample somehow become spatially inhomogeneous after the thermal cycle. Notably, the phonon intensity also appears to be sensitive to the strength of the charge order. However, due to this issue of spatial inhomogeneity, it becomes challenging to draw definitive conclusion regarding the intrinsic behaviour of CDW and phonon in the oxygen disordered state.

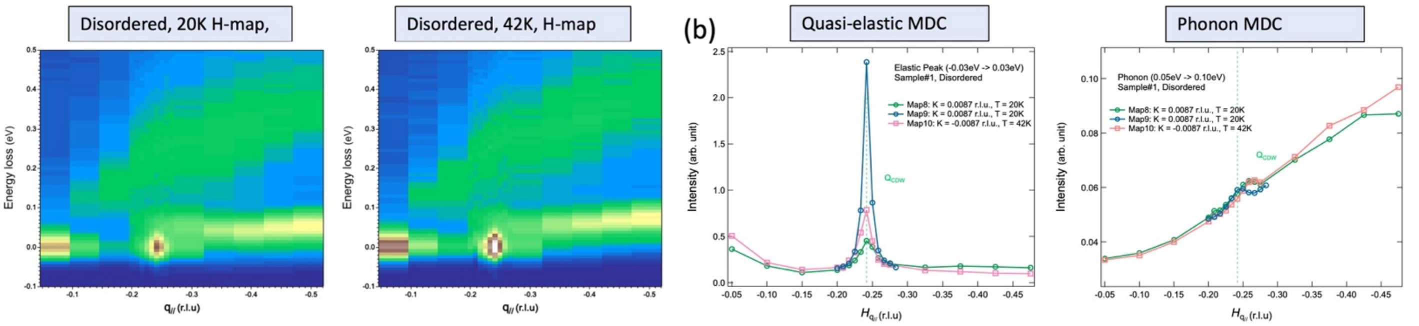

Figure 3 LCO in oxygen disordered state (a) RIXS energy-momentum map along the H-direction taken in the superconducting state (20K) and normal state (42K) (b) Momentum distribution curve (MDC) of the quasi-elastic peak intensity (left) and the phonon intensity (right) taken at the two temperatures. The quasi-elastic peak MDCs and the phonon MDCs are obtained by integrate the RIXS intensity in the energy window between -0.03 eV to 0.03 eV and 0.05 eV to 0.1 eV, respectively.

In summary, although our samples produced robust RIXS spectra of high quality, we encountered unforeseen challenges. These challenges encompassed both radiation-induced damage in the oxygen-ordered state and spatial inhomogeneity in the oxygen-disordered state. Regrettably, these technical issues have hindered our ability to arrive at a definitive conclusion regarding the impact of oxygen ordering/disordering on the behavior of charge order and phonons in LCO. We have meticulously documented this data, which may prove valuable in addressing these issues in future investigations.