

# Experimental report

01/03/2019

**Proposal:** 4-01-1522

**Council:** 4/2016

**Title:** Nano phase separation in oxygen ordered and disordered nickelates and cuprates

**Research area:** Physics

**This proposal is a new proposal**

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**Samples:** La<sub>2</sub>NiO<sub>4.12</sub>  
La<sub>2</sub>CuO<sub>4.11</sub>

Instrument	Requested days	Allocated days	From	To
IN20	8	10	28/06/2016	08/07/2016
IN8	5	0		
IN3	2	2		

## Abstract:

The nanoscopic interplay of charge ordering (CDW) and interstitial oxygen ordering in the high temperature superconducting cuprates with excess of oxygen has attracted enormous attention very recently [1-3]. Importantly, it has been shown that CDW and oxygen ordered domains are (i) nano phase separated and (ii) anticorrelated. These results have impressively demonstrated the relevance of excess of oxygen (and how it is distributed) in these materials. Also the excess oxygen materials La<sub>2</sub>CuO<sub>4+d</sub> and La<sub>2</sub>NiO<sub>4+d</sub> exhibit oxygen ordering and charge stripe ordering reflections. Our recent microdiffraction measurements also show nano phase separation for these materials (regarding charge and oxygen ordering reflections). These La<sub>2</sub>MO<sub>4+d</sub> systems (M=Cu, Ni) will allow us to switch "in situ" (i.e. during experiment) between long-range ordered and fully disordered phases by switching between (long range) oxygen ordered and oxygen disordered phases due to different thermal treatment of the samples. The measurement and comparison of the magnetic excitation spectra in (long range) ordered and disordered phases will give us direct insight into the effects of nano phase separation.

## Nano phase separation in oxygen ordered and disordered nickelates and cuprates

The nanoscopic interplay of charge ordering (CDW) and interstitial oxygen ordering in the high temperature superconducting cuprates with excess of oxygen has attracted enormous attention very recently. Importantly, it has been shown that CDW and oxygen ordered domains are (i) nano phase separated and (ii) anticorrelated. These results have impressively demonstrated the relevance of excess of oxygen (and how it is distributed) in these materials. Also the excess oxygen materials  $\text{La}_2\text{CuO}_{4+\delta}$  and  $\text{La}_2\text{NiO}_{4+\delta}$  exhibit oxygen ordering and charge stripe ordering reflections. These  $\text{La}_2\text{MO}_{4+\delta}$  systems ( $M=\text{Cu}, \text{Ni}$ ) will allow us to switch "in situ" (i.e. during experiment) between long-range ordered and fully disordered phases by switching between (long range) oxygen ordered and oxygen disordered phases due to different thermal treatment of the samples.

We have asked for 8 days and 5 days beamtime on IN20 and IN8, respectively, in order to fulfill our purposes. However, only the beamtime on IN20 was allocated. Because of the reduced beamtime, we then decided to measure one sample only, i.e., the oxygen doped nickelate. Our experiment was not going smoothly during our beamtime. For example, the A4 motor was stuck from time to time due to some problems of the floor. More severely, the ki couldn't move due to some unknown problems and **the shutter was thus closed for 2 days.**

For the experiment we have performed on IN20, the Heusler monochromator and analyzer were used for our experiment, and two PG filters were mounted after the sample. The neutron polarization was maintained by a small field generated by a set of Helmholtz coils. The sample was first heated up to 450 K and slowly cooled down to 200 K (for ~5 hours), then cooled down to 10 K. Fig. 1 shows a neutron intensity map in the HK-plane measured in the non-spin-flip channel with neutron polarization along the x-direction. The detected signal is thus nonmagnetic. As can be seen, two superlattice reflections are observed in the reciprocal space at  $(1.2 \ 0.4 \ 0)$  and  $(1.4 \ 0.2 \ 0)$ . Polarization analysis as shown in Fig. 2 shows that the intensity is only observed in the  $\text{NSF}_{xx}$  channel, indicating that these peaks are of pure nuclear origin. A temperature dependence study shows that the peak intensity decreases above ~250 K, as shown in Fig. 3. The peak position as well as the temperature dependence of the peak intensity indicate that these peaks may originate from the oxygen ordering in the sample.

The sample was then cooled down again with the same procedure to 10 K. Figure 4 shows the scans along the diagonal direction and two peaks centered at  $H = 0.38(1)$  and  $0.62(1)$  were observed in the  $\text{SF}_{xx}$  channel, indicating that they are of magnetic origin. The single sharp intensity at the half integer

position is most probably from higher order contaminations. We next tried to study the magnetic couplings by mapping the spin wave dispersions. Figure 5 shows the inelastic neutron intensities measured in the  $SF_{xx}$  channel. As can be seen, no dispersion was observed for energies up to 22 meV. Note, that the strong peaks around  $H = 1.55$  and  $1.75$  below  $E = 12$  meV are some spurious peaks. The missing of magnons either due to large spin gap or due to the dispersion along other directions needs to be clarified in future studies.

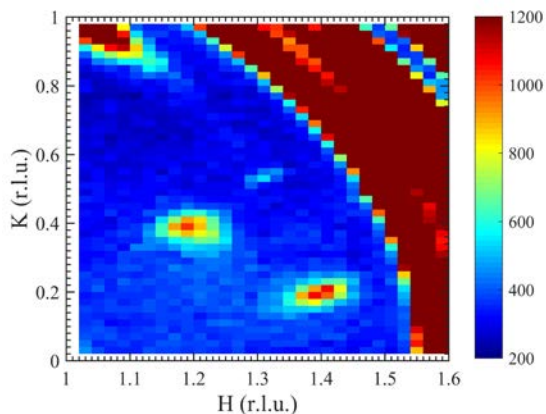


Fig. 1 Elastic neutron scattering intensities  $NSF_{xx}$  measured in the  $HK$ -plane.

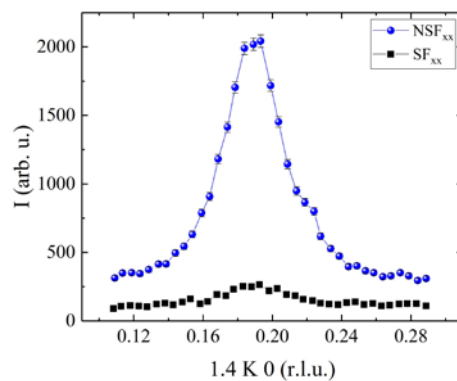


Fig. 2 Elastic neutron scattering intensities measured in the spin-flip and non-spin-flip channels.

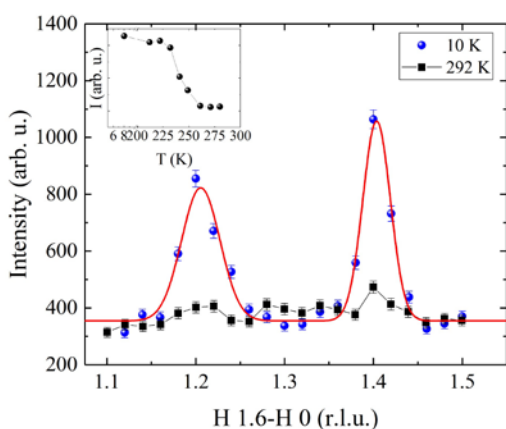


Fig. 3 Temperature dependence of the oxygen ordering peak.

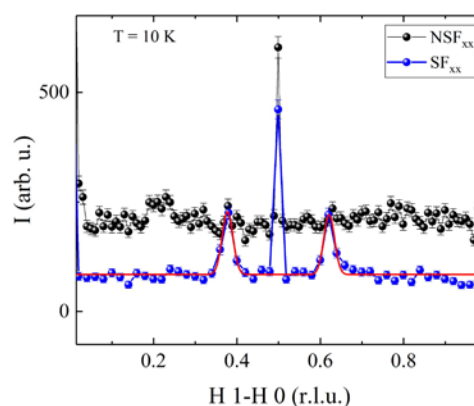


Fig. 4 Elastic neutron intensities,  $NSF_{xx}$  and  $SF_{xx}$ , measured along the diagonal direction.

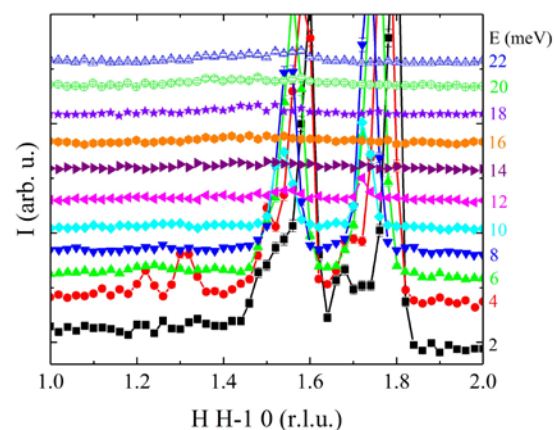


Fig. 5 Inelastic neutron intensities measured at 10 K in the  $SF_{xx}$  channel.