

**Experiment title:**Measurement of Critical Exponents and studies of Charge Stripe Dynamics in  $\text{La}_{5/3}\text{Sr}_{1/3}\text{NiO}_4$  using very High Energy X-ray Scattering**Experiment number:**

HE-665

**Beamline:**

ID15-A

**Date of experiment:**

from: 3/12/1999

to:

13/12/1999

**Date of report:**

17/02/2000

**Shifts:**

30

**Local contact(s):**

Dr. K-D. Liss

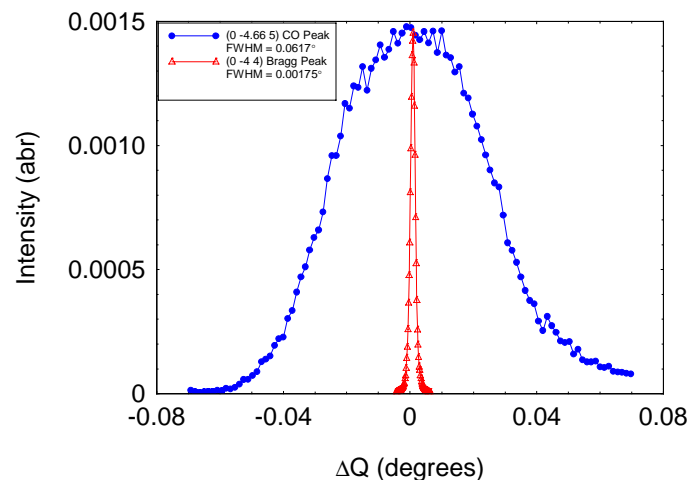
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**Report:**

Charge and spin ordering in direct space have recently attracted intensive attention due to their role in cuprate superconductivity [1] and colossal magnetoresistance (CMR) in the manganites [2]. Such charge and spin stripes were first observed in  $\text{La}_{2-x}\text{Sr}_x\text{NiO}_{4+\delta}$  by electron diffraction [3] and neutron scattering [4]. When holes are doped into the  $\text{NiO}_2$  planes, either by substitution of Sr or excess oxygen, coupled charge or spin modulations start to form. Measurements by electron diffraction and neutron scattering [5] on  $\text{La}_{5/3}\text{Sr}_{1/3}\text{NiO}_4$  have shown charge ordering at  $T_C \approx 240$  K and spin ordering at  $T_N \approx 190$  K. This composition is a model system for studying strong electron-phonon coupling, it displays no structural phase transitions at low temperatures, but three electronic phases due to charge ordering.

In recent x-ray scattering measurements on the *XMaS* beamline (BM 28) at the ESRF we have demonstrated that the charge stripes are two dimensional in nature and obtained critical exponents of the charge stripe melting transition [6]. A published study of  $\text{La}_{1.775}\text{Sr}_{0.225}\text{NiO}_4 \sim 100$  keV found intensity at both charge and spin stripe positions [7]. However, the intensity was not sufficiently high to obtain critical exponents. The purpose of this preliminary study was to see if high energy x-rays at ID15-A could be used to determine the critical exponents of the charge stripe melting occurring in the bulk of our single crystal.



The sample was placed in transmission geometry in a closed cycle He cryostat that was mounted on the Eulerian cradle of the four-circle triple-axis diffractometer at ID15-A. Measurement of the rocking curve width of Bragg peaks using 130keV x-rays was 100 times less than that of the manganite crystal or the nickelate crystal studied at Hasylab [7]. In

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addition the sample displayed no lattice strains and all the Bragg peak shapes were very sharp along the 2 $\theta$  direction (see figure 1). The sample was cooled below the charge stripe transition temperature ( $\sim 240$  K) to 100K. Satellite reflections corresponding to the charge stripe ordering with a modulation wave vector of either (0.667 0 1) or (0 0.667 1) were found surrounding the very sharp Bragg peaks. We observed weak charge stripe ordering at (4.667 0 5) (0 4.667 5) and (3.33 0 3). Scans were made as a function of temperature in both the longitudinal (analyser rocking curve) and transverse (sample rocking curve) directions. The charge stripe satellites were found to be extremely weak (much weaker than that previously observed by using 10 keV x-rays) and much broader. Figure 1 shows a comparison of the (4 0 4) Bragg peak and the neighbouring charge stripe satellite (4.66 0 5)

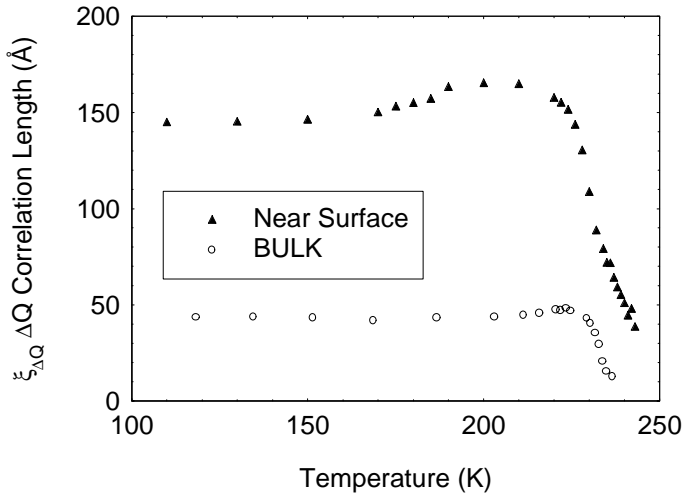


Figure 2

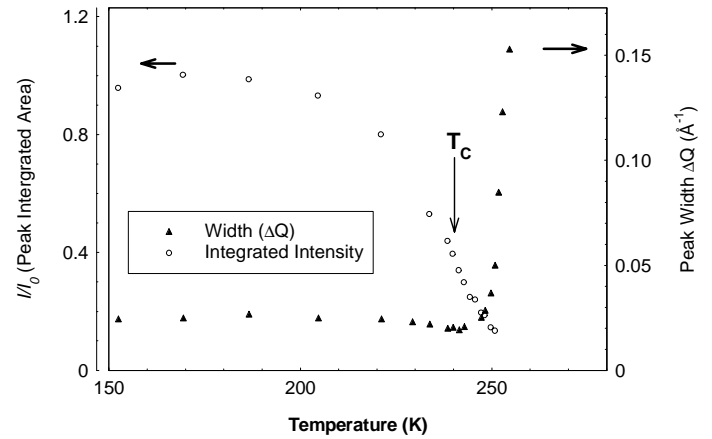


Figure 3

The charge stripe reflection is clearly very broad, which means that in the bulk of the crystal the charge stripes are correlated over only a very short distance. Fits along the longitudinal scans also demonstrate a very short correlation length ( $\sim 45$  Å), see fig 2. Such results are identical to those obtained using high energy x-rays at Hasylab [7] or neutrons [4], in spite of their crystal having a poorer rocking curve width by about 100 times. It therefore appears that the measured correlation length of the charge stripes in the bulk is an intrinsic property of the system and not affected by the crystal quality (mosaic width). However the measured correlation lengths are considerably shorter than those close to the surface (see figure 2) as measured using 10 keV x-rays [6]. Furthermore, the charge stripe satellite intensity is very much weaker than that expected on the basis of our x-ray study, or of the previous high-energy study. The intensity is related to the number of stripes within the sample. It is possible that close to the sample surface or in a high dislocation density sample that there are many more stripes than in a near perfect bulk single crystal. Measurement of the variation with temperature (fig 3) of the intensity and width of the charge stripe satellites around  $T_C$  allowed us to extract the critical exponents  $2\beta$  and  $\nu$ . These results are the first observation of critical scattering of charge stripe fluctuations above  $T_C$ .

In summary, this experiment has demonstrated the very different behaviour of charge stripe ordering in the bulk and near surface region and shown that the data is of sufficient quality to obtain quantitative critical exponents.

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