Superconductivity-Insensitive Order at $q \sim 1/4$ in Electron-Doped Cuprates

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One of the central questions in the cuprate research is the nature of the normal state that develops into high-temperature superconductivity (HTSC). In the normal state of hole-doped cuprates, the existence of a charge density wave (CDW) is expected to shed light on the mechanism of HTSC. With evidence emerging for CDW order in the electron-doped cuprates, the CDW is thought to be a universal phenomenon in high- T_c cuprates. However, the CDW phenomena in electron-doped cuprates are quite different than those in hole-doped cuprates. Here, we study the nature of the putative CDW in an electron-doped cuprate through direct comparisons between as-grown and postannealed $\text{Nd}_{1.86}\text{Ce}_{0.14}\text{CuO}_4$ (NCCO) single crystals using Cu L_3 -edge resonant soft x-ray scattering (RSXS) and angle-resolved photoemission spectroscopy (ARPES). The RSXS result reveals that the nonsuperconducting NCCO shows the same reflections at the wave vector ($\sim 1/4, 0, l$) as the reported superconducting NCCO. This superconductivity-insensitive signal is quite different from the CDW reflection in hole-doped cuprates. Moreover, the ARPES result suggests that the fermiology cannot account for such wave vectors. These results call into question the universality of the CDW phenomenon in the cuprates.

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I. INTRODUCTION

The charge density wave (CDW) in hole-doped cuprates has been widely appreciated as an important ingredient for understanding high-temperature superconductivity (HTSC) [1–17]. This is because the CDW order is shown to compete or, more intimately, intertwine with HTSC [5–8,10–17]. However, the CDW order in the hole-doped cuprates is distinguishable from the classical one-dimensional charge modulation driven by Peierls' mechanism [18,19]. In other words, the CDW phenomena in HTSC are not simply explained by the band structure. Therefore, characterizing the correlation length of CDW order and its competition with superconductivity is crucial to a comprehensive understanding of the high- T_c phase diagram [14–16]. In this context, to ascertain the nature of CDW order in hole-doped cuprates, many experimental approaches, such as x-ray scattering [5–13,16,17], nuclear magnetic resonance (NMR) [4,20,21], scanning tunneling microscopy (STM) [2,10,22–24], and quantum oscillations [3,25], have been

performed. In particular, x-ray scattering by resonant soft x-ray scattering (RSXS) and resonant inelastic x-ray scattering (RIXS) [5,9–11,16,17] have played a critical role in showing that CDW order in the CuO_2 layer exists, thanks to the high sensitivity to Cu charge density of the resonant process at the Cu L_3 edge (Cu $2p \rightarrow 3d$ dipole transition) [5,16]. Furthermore, substantial theoretical work has also been carried out [15,26]. These characterizations of CDW order in hole-doped cuprates have shed light on the essential physics underlying HTSC [14,15].

Meanwhile, RSXS efforts in $\text{Nd}_{2-x}\text{Ce}_x\text{CuO}_4$ and $\text{La}_{2-x}\text{Ce}_x\text{CuO}_4$ have recently expanded these studies to the electron-doped cuprates, where they found putative CDW order in both systems [27,28]. This finding is an important step forward for understanding CDW phenomena in the high- T_c cuprates. However, at the same time, there are many aspects of the feature (described below) that are distinct from those seen in the hole-doped cuprates [5–8,16,17, 27–29]: (1) The doping range of the observed CDW order extends well beyond the superconducting (SC) dome [28] [see Fig. 1(a)]; (2) the CDW onset temperature is much higher than the onset temperature (T^*) [27–28] of the mysterious pseudogap phase [29]—in general, the onset temperature of the CDW in hole-doped cuprates is lower than T^* [5–8,11,19] or similar to T^* [9,22]; (3) no temperature dependence of the correlation length is observed [27,28]; (4) there is no CDW response when SC is suppressed by an external magnetic field up to 6 Tesla [28]. These distinctions

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FIG. 1. Phase diagram of annealed and as-grown NCCO and their RSXS measurements. Phase diagram of the (a) annealed and (b) as-grown NCCO as a function of Ce doping. The red, green, and blue areas denote the long-range AFM order, SC, and the proposed CDW, respectively. Dashed lines in panel (c) denote AFM and SC boundaries after annealing as displayed in panel (a). RSXS data of the (b) annealed and (d) as-grown NCCO ($x = 0.14$). These were measured at the Cu L_3 edge (931 eV) with $T = 30$ K. The vertical lines indicate the peak position at $h = -0.26 \pm 0.01$ reciprocal lattice units (r.l.u.).

from the hole-doped cuprates leave open the question of the nature and origin of the putative CDW order, as well as its universality in high- T_c cuprates.

In this work, we carry out comparative RSXS and angle-resolved photoemission spectroscopy (ARPES) studies of as-grown (nonsuperconducting) and postannealed (superconducting) $\text{Nd}_{1.86}\text{Ce}_{0.14}\text{CuO}_4$ (NCCO) crystals, aiming to address such questions. As reported in the previous studies [27,28], we can observe the putative CDW reflection at $Q = (-1/4, 0, l)$ in the SC NCCO. Interestingly, we also found the same reflection in the non-SC NCCO. Corresponding ARPES results on both NCCO crystals demonstrate that a CDW arises for reasons that have nothing to do with the fermiology. Detailed RSXS studies indicate that this superconductivity-insensitive order in NCCO is in contrast with the hole-doped cuprates, in particular, $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ (YBCO). These findings and corresponding implications support one of two possible scenarios proposed by da Silva Neto *et al.* [28] to understand this reflection's origin. These results also imply that the CDW is different in the electron- and hole-doped cuprates.

II. METHODS

Sample preparation.—Dried powder Nd_2O_3 , CeO_2 , and CuO with the appropriate molar ratio were mixed and

sintered in air at 1050 °C for 24 hours with intermediate grindings. The prefired powders were pressed under hydrostatic pressure to obtain cylindrical rods with a diameter of 6 mm and a length of 100 mm. Subsequently, the rod was sintered at 1200 °C for 12 hours. A single crystal of $\text{Nd}_{1.86}\text{Ce}_{0.14}\text{CuO}_4$ was grown by the traveling-solvent floating-zone method using the sintered rod. A part of the crystal was annealed under Ar gas flow at 900 °C, which resulted in superconductivity with a transition temperature of about 25 K. The amount of oxygen removed by annealing was determined to be about 0.02 from the weight loss of the sample. A change in disorder of NCCO from the annealing process was characterized by extended x-ray absorption fine-structure (EXAFS) and x-ray diffraction measurements [30].

Experimental details of RSXS.—Resonant x-ray scattering experiments near the Cu L_3 edge were carried out at beamline 13-3 of the Stanford Synchrotron Radiation Lightsource (SSRL). The single crystals were *ex situ* cleaved to obtain flat and shiny surfaces perpendicular to the c axis, and the sample temperature was controlled by an open-circle cryostat from 17 K to 380 K. Incident photon polarization was fixed as σ (vertical linear) polarization. The $(h, 0, l)$ scattering plane was aligned from $(0, 0, 2)$ and $(1, 0, 1)$ lattice reflections using photon energy of about 1700 eV. To minimize the background slope change, the channeltron detector was fixed at 176°, and the sample angle was controlled to obtain h dependence. The length of the sample surface along the beam direction was about 1.5 mm, and the horizontal full width of the beam spot was about 0.5 mm. The x-ray beam size was smaller than the sample size until a low grazing angle ($\theta \sim 20^\circ$ and $h \sim -0.55$). Accordingly, most of the x ray was safely shined onto the sample surface during the experiment. The RSXS peaks were fit by the Lorentzian function to obtain their height and FWHM [Figs. 2(b) and 2(c)] after subtracting the highest-temperature data (380 K), which was fit by a polynomial function [black solid line in Fig. 2(a)].

Experimental details of ARPES.—ARPES measurements were carried out at beamline 5-2 of the SSRL using SCIENTA D80 electron analyzers with linearly polarized photons with a photon energy (E_{ph}) = 53 eV. All measurements were done in ultra-high vacuum (UHV) with a base pressure lower than 6×10^{-11} Torr. The total energy resolution was set to 15 meV, and the angular resolution was 0.3°. All single crystals were cleaved *in situ* at 25 K for each ARPES measurement.

III. RESULTS AND DISCUSSION

We first investigate the annealed NCCO crystal using RSXS at the Cu L_3 edge (931.0 eV) using measurements similar to the earlier work [27,28]. As shown in Fig. 1(b), we clearly observe the reflection, as reported previously [27,28] at the same in-plane wave vector $(-0.26, 0)$. Next,

FIG. 2. Temperature dependence of the reflection at $q \sim 1/4$. (a) Temperature-dependent h scans in the as-grown NCCO ($x = 0.14$), measured at the Cu L_3 edge. The shaded areas denote the portion of the peak above the background (black line), which was taken at $T = 380$ K. (b) Plots of peak height (upper panel) and the full width half maximum (FWHM) (lower panel) of the peaks at $h \sim -0.26$ as a function of temperature. The open and filled circles are from the annealed and as-grown NCCO, respectively. The width is about 0.085 r.l.u. over the entire temperature range. The grey shadow is a guide to the eye, and the error bars denote 1 standard deviation (s.d.) as obtained from the peak fitting.

we repeat the same measurements on the as-grown NCCO. Note that there is no SC phase in the as-grown sample [Fig. 1(c)]. Nonetheless, we observe the same reflection at the same in-plane wave vector [Fig. 1(d)]. Note that the as-grown (i.e., non-SC) NCCO, as well as the SC NCCO, followed a technically similar approach in the previous work [27,28], aiming to directly compare the putative CDW between the SC and non-SC samples.

To understand why the same reflection is present for both as-grown and annealed NCCO, we examine the correlation length and the intensity of the reflection as a function of temperature. Figure 2(a) shows data of as-grown NCCO at $T = 30, 240, 300$, and 380 K. The background (black solid line) is the fit to the RSXS data at $T = 380$ K (see Sec. II). With increasing temperature, the scattering intensity at $Q \sim (-0.26, 0, l)$ becomes weaker. However, even at room temperature, which is already much higher than T^* (about 80 K in annealed NCCO [33,34]), there is still a bump around $(-0.26, 0, l)$ as presented in the annealed case [27,28]. Figure 2(b) summarizes the temperature dependence of the peak intensity (upper) and its width (lower) along with the results from the annealed NCCO. Interestingly, the temperature behaviors in both the as-grown and annealed NCCO are indistinguishable. The peak intensity shows the same gradual development from 380 K, while the peak width is constant as a function of temperature [lower panel in Fig. 2(b)]. The estimated correlation length from a Lorentzian fitting function is $2/\text{FWHM} \times (a/2\pi) \sim 15$ Å, while the lattice constant of NCCO, a , is

3.95 Å. The estimated correlation length is just comparable to 1 period of the real space length (15.8 Å) of the proposed CDW with a q vector position $\sim 1/4$. Even at $T = 17$ K, which is lower than the T_c of the annealed NCCO, no change in the width is seen, indicating no interaction with its SC. This is also consistent with the implication from the reported scattering result with 6 Tesla [28]. However, this field independence [28] in NCCO is in contrast to the strong enhancement of the CDW strength found in vortex cores of Bi-based cuprates [2], which have a similar correlation length as NCCO. Furthermore, we performed 2θ dependences of the peak in as-grown NCCO [30], showing a strange behavior, in which the in-plane h vector shifts with varying 2θ angle (i.e., at different l vectors). These properties strongly contrast with the CDW-order character observed in the hole-doped cuprates, in particular, Y/La-based cuprates [5–8].

In Ref. [28], the authors proposed two possible scenarios for interpreting the scattering feature in annealed NCCO. First, a disorder-pinned scattering intensity could come from a CDW. The second scenario is that the reflection is a signature of fluctuations in the inelastic excitations. Considering that the annealing process removes oxygen from the crystal and generates secondary phases, superconducting NCCO should have more defects and disorder [35–38]. However, the scattering results in the as-grown and annealed NCCO crystals are nearly identical, despite the difference of the disorder, which was monitored by EXAFS and x-ray diffraction [30]. In the absence of quenched disorder (i.e., the very clean limit), a CDW phase has spontaneously broken symmetry. It appears below a sharply defined thermodynamic phase-transition temperature, resulting in CDW order with zero width (infinite correlation length). In this limit, the scattering feature results from a strictly elastic (*static*) scattering structure factor. On the other hand, in the presence of disorder, this correlation length becomes finite. However, despite the difference in the strength of disorder between the as-grown and annealed NCCO, the estimated correlation length is identical. Therefore, changes in disorder from annealing are unlikely related to this reflection, conflicting with the first scenario in Ref. [28].

Moreover, a correlation between the fermiology and the CDW has been discussed in both hole-doped [9,10] and electron-doped [27,28] cuprates, and the possible importance (and some problems) of the Fermi surface (arc) instabilities for the CDW has been discussed [9,10, 27,28,39]. Note that the superlattice peak in Bi-based cuprates is not relevant to this discussion. To directly compare the RSXS wave vector with the Fermi surface, we have performed ARPES measurements on the same crystals measured with RSXS, annealed [Fig. 3(a)] and as-grown [Fig. 3(b)]. The distance between the parallel segments at $k_x \sim \pi$ are estimated and summarized in Fig. 3(c). We find $(h, k) \sim (0.283 \pm 0.002, 0)$ and $(0.270 \pm 0.002, 0)$ for the

FIG. 3. Fermi surface topology in ARPES. We show Fermi surface intensity mappings for (a) annealed and (b) as-grown NCCO measured by ARPES at $T = 25$ K with $E_{ph} = 53$ eV. Intensities are integrated within ± 10 meV of E_F . Note that the mappings are not folded. The dashed red curves for the Fermi surface are guides to the eye. Red arrows indicate the polarization of incident x-ray (c) line cuts along the k_y direction around $(\pi, 0)$. Here, k_x is integrated over the range $[0.95\pi, 1.05\pi]$. The open (filled) circles denote the nesting wave-vector positions of the annealed (as-grown) NCCO. The vertical lines indicate the in-plane wave vector measured by RSXS.

annealed and as-grown NCCO, respectively. As expected, because of stronger AFM and the excess oxygen [35–38], the distance in the as-grown sample is smaller than in the annealed sample. This observation is in good agreement with previous work reporting the annealing dependence of the Fermi surface [40–42]. Both in the annealed and as-grown samples, the wave vectors connecting the two Fermi surfaces at $k_x \sim \pi$ are larger than the in-plane wave vector of the scattering reflection [the vertical lines in Fig. 3(c)]. Apparently, the wave vectors connecting the hot spots due to either the Fermi arc formation by the pseudogap or (fluctuating) antiferromagnetism are even larger. Furthermore, the annealing dependence on the nesting wave vector is in sharp contrast with the annealing independence of the reflection. Our direct comparison of the RSXS and ARPES data on the same set of samples makes it possible to conclude that the Fermi surface (arc), i.e., fermiology, is not directly relevant to the CDW. This conclusion is supported by recent numeric work [39], which argues that Fermi surface nesting is unlikely to cause CDW formation in the high- T_c cuprates.

FIG. 4. Energy dependence of the reflection at $q \sim 1/4$. (a) The 2D map of the RSXS intensity of the as-grown NCCO at $T = 30$ K. (b) The integrated energy profile of the as-grown I_{RSXS} near $h \sim -0.26$ (red circle). The integrated range is indicated by red dashed lines in Fig. 3(a). The profile is compared to the x-ray absorption spectroscopy (XAS) spectra using fluorescence yield (FY). We show the h scans of (c) annealed and (d) as-grown NCCO ($T = 30$ K) at different photon energies: Cu L_3 edge, pre-edge, and postedge are 931 eV, 922 eV, and 936 eV, respectively. Data are vertically shifted without scaling for better visibility. The red vertical bars indicate the h positions of the features.

Based on our findings, which are different to the CDW features found in the hole-doped cuprates [5–11,16,17], including the directly compared fermiology, the appreciation of the universality of the CDW features in cuprates still seems in the early stages. Concurrently, one may ask the critical question, *where does this reflection come from?* In order to gain additional insight into this critical question, we study the energy dependence (i.e., resonant profile) of the reflections in both as-grown and annealed NCCO. Figure 4(a) shows the E_{ph} vs h map of the as-grown NCCO. The energy profile does not show a peak solely around $h \sim -0.25$. Regardless of the h position, the scattering intensity is pronounced only near the Cu L_3 edge. Even after integrating the RSXS intensity around $h \sim -0.25$ as a function of E_{ph} [see Fig. 4(b)], it is still indistinguishable from background signals, which are mainly from inelastic fluorescence (e.g., $d-d$ excitations) [43].

Recall that one of the possible causes for the reflection suggested in the previous work [28] was an inelastic excitation (i.e., the second scenario). To consider this possibility, we plot h scans at three different energy positions (Cu L_3 edge, pre-edge, and postedge) as shown

in Figs. 4(c) (annealed) and 4(d) (as-grown). Like the temperature dependences shown in Fig. 2, the energy dependences of both samples are nearly identical. In comparison with the pre-edge (922 eV), a peak feature at $h \sim -0.26$ is pronounced at the Cu L_3 edge (931 eV) and becomes weaker for the postedge (936 eV). In addition, we observe a pronounced intensity at $h \sim -0.40$ at the Cu L_3 edge, which is invisible at the pre-edge. Interestingly, there are substantial features at $h \sim -0.39$ even at the postedge, while the feature at $h \sim -0.26$ is shifted to -0.27 . Note that this shifting feature is more pronounced when a fluorescence background is eliminated [30], and the shifting value is much larger than the change in the real part of the atomic form factor (i.e., refractive index) as a function of the x-ray photon energy [44]. These facts, as well as the temperature dependence, contradict the CDW features from the hole-doped cuprates, in particular, YBCO. To determine whether an elastic contribution coexists or not, high-resolution RIXS studies should be carried out in future work.

IV. CONCLUSION

In summary, we investigated the recently observed putative CDW scattering in the electron-doped superconducting NCCO using RSXS and ARPES measurements. We reproduced the reported reflections at $q \sim 1/4$ in both superconducting and nonsuperconducting NCCO. The detailed RSXS studies, such as temperature, energy, and angle dependences, of both reflections show no interaction with the SC order. Our findings show that the reflection observed in NCCO at $q \sim 1/4$ is not analogous to the CDW observed in the hole-doped cuprates. The ARPES measurement on the same set of samples shows that no Fermi surface (arc) instability can account for the observed CDW, except the nesting origin of the CDW. Moreover, these results indicate that the peak signal may come from inelastic contributions. Unfortunately, our RSXS data cannot resolve which inelastic contribution generates this peak, presenting a rich opportunity for RIXS studies and further theoretical interpretations.

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