

Interplay between electronic and atomic short-range order in $\text{La}_{2-x}\text{Ca}_x\text{CuO}_{4-\delta}$: Temperature and doping dependence

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Complex oxides are well-known to develop oxygen ordering patterns with well-defined periodicities, but their interplay with the electronic structure remains largely unexplored. Here, we report resonant and nonresonant x-ray diffraction data indicating a four-unit-cell periodic superstructure related to oxygen vacancy ordering in $\text{La}_{2-x}\text{Ca}_x\text{CuO}_{4-\delta}$ films with Ca dopant concentrations from the underdoped ($x = 0.15$) to the extremely overdoped ($x = 0.50$) regime. Whereas the oxygen superstructure is temperature independent up to 300 K, a strongly temperature-dependent electronic charge density wave (CDW) is observed in the underdoped and slightly overdoped regimes ($x \leq 0.20$). The periodicity, in-plane, and out-of-plane correlation lengths of the CDW are locked in by the oxygen superstructure. Our results highlight the interdependence of electronic and atomic ordering phenomena and underscore the necessity of determining the temperature dependence to distinguish electronic and lattice atomic contributions to superstructures.

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Introduction. Atomic short-range order (ASRO) is ubiquitous in complex metal oxides. Partial order in the lattice site occupation by different ionic species has long been known to decisively influence the electronic properties of oxides [1], and is rapidly gaining prominence as a key performance characteristic of electrochemical devices [2,3]. Whereas machine learning tools are increasingly applied to explore the thermodynamic stability of partially ordered metal oxides and to optimize their functionality [4], the interplay between ASRO and electronic ordering phenomena in complex oxides remains largely unexplored. Research on less complex solids such as graphene and semiconductor surfaces has offered detailed microscopic insight into the nucleation of electronic charge density waves (CDWs) by lattice defects [5,6]. Conversely, theoretical research on disordered metallic alloys has shown that the periodicity of ASRO is set by maxima of the electronic charge susceptibility, which naturally occur at wave vectors connecting parallel segments of the Fermi surface [7–12]. Although band theory is typically associated with translationally symmetric lattices, it is now well established that the notion of a Fermi surface remains relevant even in the high-temperature

disordered phase of alloys, where dynamic short-range order is established as a precursor to ASRO [10–12].

Here we explore the relationship between ASRO and the electronic structure of the high-temperature superconducting cuprates, where ASRO has been widely studied in the form of partially ordered superstructures of oxygen ions [13–26]. As oxygen ions act as dopants, their local correlations affect the local doping level and can induce spatial inhomogeneities in the electronic phase behavior. The discovery of short-range-ordered CDWs with periodicities between three and four unit cells has recently opened another perspective on the electronic structure of the cuprates. The CDW periodicity reflects features of the electronic charge susceptibility, which is strongly renormalized by Coulomb interactions between conduction electrons and difficult to compute in the strongly correlated electron system [27–29]. CDWs have been widely observed in underdoped cuprates [27–35] and were more recently reported above the optimal doping level in certain systems: up to hole doping level of $p = 0.24$ in $(\text{Bi}, \text{Pb})_{2.12}\text{Sr}_{1.88}\text{CuO}_{6+\delta}$ (Bi2201) [36], $p = 0.25$ in $\text{Tl}_2\text{Ba}_2\text{CuO}_{6+\delta}$ (Tl2201) [37], and $p = 0.21$ in bulk $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$ (LSCO) [38–40].

Research on the relationship between ASRO and CDW order has thus far largely focused on the influence of the nonstatistical distribution of dopants on the CDW correlation length [41]. According to one recent report [42], however, electronic CDW order with an unusual periodicity of ~ 6 unit cells is present in the extremely overdoped regime of LSCO with $p > 0.25$. Contrary to the underdoped systems, the purported CDW correlations are virtually temperature independent at least up to room temperature, raising the question to what extent lattice superstructures arising from cation or oxygen order may be involved at a more essential level.

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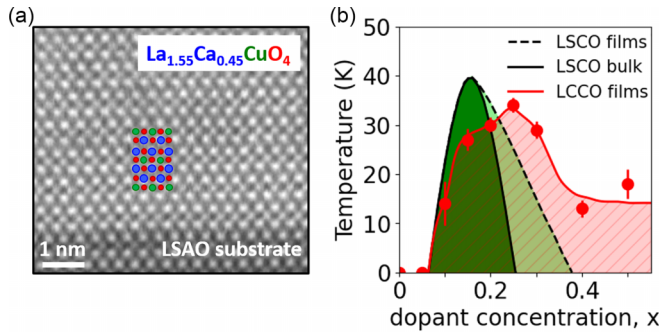


FIG. 1. (a) Scanning transmission electron microscopy image of an $x = 0.45$ LCCO film, confirming high crystallinity. (b) Ca dopant concentration dependence of superconducting T_c in 10-unit-cell-thick LCCO films compared with that of bulk LSCO and LSCO films [45–48]. Figures adapted from Ref. [43].

Motivated by these reports, we have used a combination of Cu L_3 -edge resonant x-ray scattering and nonresonant hard x-ray diffraction (XRD) to investigate CDW correlations and lattice superstructures in thin films of $\text{La}_{2-x}\text{Ca}_x\text{CuO}_{4-\delta}$ (LCCO) over a wide range of Ca dopant concentrations $x = 0.10$ – 0.50 . LCCO is of particular interest because of its low level of structural disorder due to the nearly identical radii of La and Ca ions. Our films were extensively characterized in prior work [43] (see also Fig. 1(a) and Supplemental Material Ref. [44]), and it was found that superconductivity in this system persists to a Ca concentration of at least $x = 0.50$, well beyond what is known for systems with higher structural disorder such as LSCO [Fig. 1(b)]. Here we report a temperature-independent bulk four-unit-cell periodic lattice superstructure over an exceptionally wide Ca concentration range, $x = 0.15$ – 0.50 , which we attribute to oxygen-vacancy ordering. For $x = 0.15$ and 0.20 , we additionally observe an electronic CDW, distinguished from the oxygen superstructure by its pronounced temperature dependence, with its periodicity and both in-plane and out-of-plane correlations locked to those of the oxygen superstructure. Our results indicate that lattice and electronic instabilities are highly interdependent, highlighting the need to treat both phenomena self-consistently. At low temperatures, the static oxygen superstructure clearly serves as a template for the electronic CDW. Conversely, the unusual stability of the four-unit-cell oxygen superstructure and its coincidence with the CDW raise the question how electronic structure contributes to the formation of dynamical oxygen short-range order at high temperatures, where oxygen diffusion kinetics is appreciable ($\sim 280^\circ\text{C}$ [45]), and hence to the static oxygen superstructure observed in our low-temperature experiments. In analogy to ASRO in metallic alloys, our results call for high-temperature electronic-structure calculations to address this issue.

Experimental details. LCCO films of 10 and 100 unit cell thickness (~ 13.2 nm and 132 nm, respectively) were grown on LaSrAlO_4 (LSAO) (001) substrates using molecular beam epitaxy and extensively characterized, as reported previously [43] (see also Figs. 1(a), 1(b) and Supplemental Material Ref. [44]). The resonant inelastic x-ray scattering (RIXS) measurements were performed using the ERIXS spectrometer at the ID32 beamline at the European Synchrotron Radiation

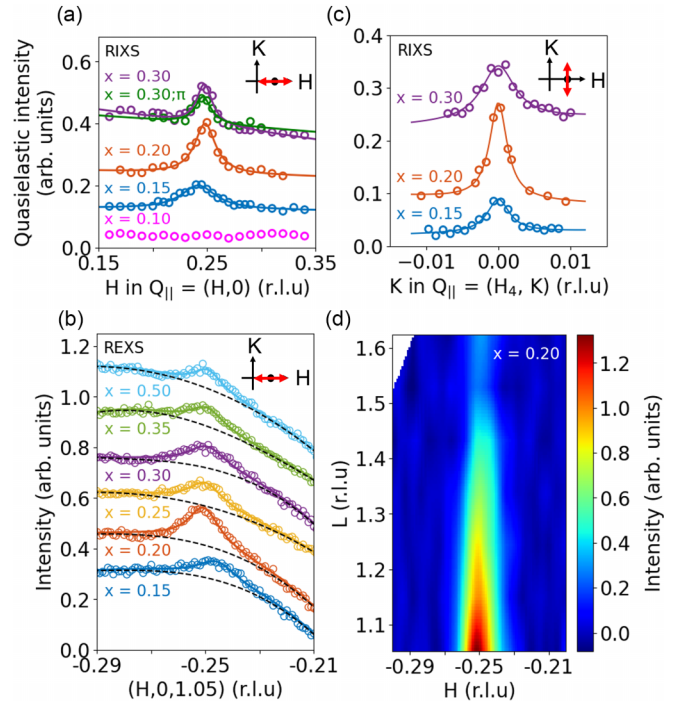


FIG. 2. (a) H scan of the energy-integrated quasielastic scattering intensity obtained from RIXS for various Ca concentrations. (b) H scan of the REXS intensity at $L = 1.05$, obtained for various Ca concentrations. The dashed black lines represent the fitted third-order polynomial background. (c) K scan of the energy-integrated quasielastic scattering intensity obtained from RIXS across $Q_4 = (H_4, 0)$. The solid lines in (a)–(c) represent the results of fits to the data as described in the text. (d) Reciprocal space map of the background-subtracted REXS intensity in the $H-L$ plane for the $x = 0.20$ sample. The data were linearly interpolated to create the color map. All data were obtained at $T = T_c$.

Facility in Grenoble. The scattering angle was fixed at 149.5° . Since our focus is on static CDW and lattice superstructures, the RIXS measurements were performed with an energy resolution of 45 meV, and we discuss only the resolution-limited (quasi)elastic scattering part of the RIXS spectra (see Supplemental Material Ref. [44] for examples of full spectra). The resonant energy-integrated x-ray scattering (REXS) measurements were performed at the beamline UE46-PGM1 at the BESSY-II synchrotron of the Helmholtz-Zentrum in Berlin. All resonant x-ray measurements were performed at the Cu L_3 absorption edge (~ 931.5 eV) with σ incident polarization, unless otherwise specified. The nonresonant hard XRD measurements were performed with an incident x-ray energy of 10 keV at the MPI beamline of the KARA synchrotron. The resonant x-ray and hard x-ray measurements were performed on 10 unit cell and 100 unit cell thick films, respectively.

Results. We first present data from Cu L_3 edge RIXS, which are highly sensitive to charge correlations of the Cu $3d$ conduction electrons. Figure 2(a) displays the momentum dependence of the energy-integrated quasielastic scattering intensity (integration range -0.15 eV to 0.1 eV) along the copper-oxide bond direction (H) at various Ca concentrations. Note that the momentum coordinates are given in reciprocal lattice units, so $H_4 = 1/4$ corresponds to four unit cells in

real space. Clear peaks near H_4 are observed for $x = 0.15$, 0.20, and 0.30, whereas no peak is observed for $x = 0.10$. Upon switching the incident beam to π polarization, the peak in the $x = 0.30$ sample becomes weaker, confirming that the observed quasielastic scattering signal originates from charge correlations [31]. Further confirmation of the four-unit-cell reconstruction was obtained from REXS measurements as shown in Fig. 2(b). The highest peak amplitude is observed for $x = 0.20$, in agreement with the RIXS results. Due to the advantage of faster measurements possible with REXS, more samples could be explored, and analogous peaks were also observed in samples with Ca concentrations of $x = 0.25$, 0.35, and 0.50. Using least-squares fits to Lorentzian profiles for the peak and a first- (third-) order polynomial for the smooth background in RIXS (REXS), we extracted the characteristic wave vector, which evolves from $H \sim 0.245$ for $x = 0.15$ to ~ 0.25 for all $x \geq 0.20$. An equivalent peak was observed in the $(0, K)$ direction (see Supplemental Material Ref. [44]), as expected based on the tetragonal symmetry of the crystal structure. For simplicity, we refer to both wave vectors as Q_4 . The correlation length parallel to Q_4 , $\xi_{\parallel} \sim 60$ Å, extracted from the fits, is typical of electronic CDWs in cuprates [28]. The in-plane correlation length perpendicular to Q_4 was extracted from scans parallel to the K (H) direction across $(H_4, 0)[(0, K_4)]$. The value obtained in this way, $\xi_{\perp} \sim 200$ – 300 Å, is about 4–5 times larger than $\xi_{\parallel}$ [see Supplemental Material Ref. [44] and Fig. 2(c)], indicating the uniaxial nature of the corresponding domains [49].

Next, we look at the correlations of the superstructure signal along the c axis, by mapping out its L dependence using REXS (see Fig. 2(d) and Supplemental Material Ref. [44]). Although the Q range accessible with Cu L_3 -edge photons is limited to $L \geq 1.05$, the salient features of the out-of-plane correlations are apparent from the resulting data. In particular, the peak intensity increases upon approaching $L \sim 1$ over the entire Ca concentration range $x = 0.15 - 0.50$, unlike previous observations of CDWs in bulk single crystals of LSCO and $\text{La}_{2-x}\text{Ba}_x\text{CuO}_4$ (LBCO), where the strongest out-of-plane CDW correlations appear at half-integer L [30,39,50]. (Note that the L -dependence measurements were not attempted with RIXS due to their time-consuming nature.)

Since electronic CDWs are generally expected to display a strong temperature dependence, determining the temperature dependence of the superstructure peaks would allow us to assess whether they have an electronic nature. Figure 3(a) displays the T dependence of the integrated peak intensity for $x = 0.15$, 0.20, and 0.30 obtained using REXS. A strong temperature dependence and onsetlike behavior at $T_{\text{onset}} \sim 150$ – 200 K is observed for $x = 0.15$ and $x = 0.20$. A downturn of the Q_4 intensity below T_c can be discerned for $x = 0.15$ (albeit within the error bar), evidencing competition between the CDW and superconductivity. These observations are consistent with prior reports of electronic CDW in bulk single crystals [30,39,50–53]. On the other hand, the intensity is nearly temperature independent for $x = 0.30$. Limited T -dependence data collected on $x = 0.35$ (see Supplemental Material Ref. [44]) also show nearly temperature independent behavior.

Several studies on bulk LSCO have revealed nearly temperature-independent CDW signals in certain regions

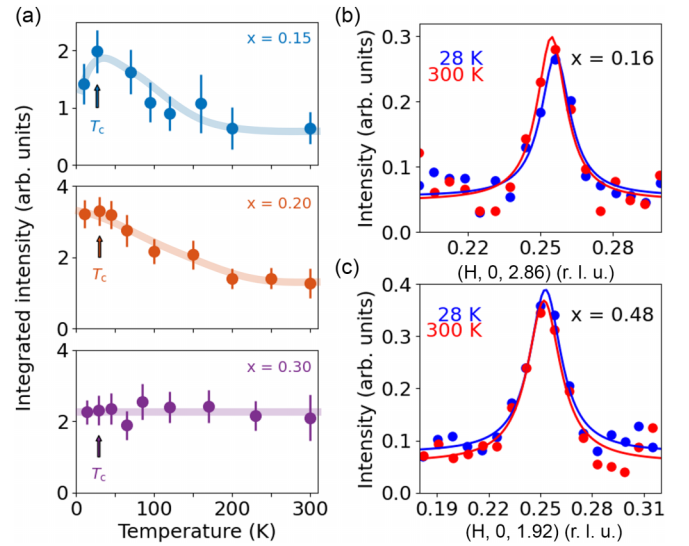


FIG. 3. (a) Temperature dependence of the integrated intensity of the superstructure peak obtained from REXS for $x = 0.15$, 0.20, and 0.30. The T_c of each sample is indicated. H scans across $Q_4 = (H_4, 0)$ obtained for 100-unit-cell-thick $x = 0.16$ and $x = 0.48$ samples, respectively, at 28 K and 300 K, with nonresonant hard XRD. The solid lines were obtained from Lorentzian fits.

of the temperature-doping phase diagram, which were attributed to short-range CDW order [39,40]. Recent studies on $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (YBCO) have also pointed out that a charge fluctuation regime persists in an extended region of the phase diagram, displaying similar temperature-independent behavior [54,55]. Such fluctuations are significantly shorter-ranged than the static CDW. However, the correlation lengths we extract from our measurements are nearly temperature- and Ca concentration-independent with $\xi_{\parallel}$ remaining at ~ 60 – 80 Å (see Supplemental Material Ref. [44]), implying that the T -independent component cannot be attributed to short-range CDW order. Instead, the crossover in the temperature dependence of the integrated intensity observed in our LCCO films with increasing Ca content strongly suggests that the peak at higher Ca concentrations ($x \geq 0.30$) does not have an electronic origin, whereas the peak at lower Ca concentrations ($x \leq 0.20$) displays signatures of a partially electronic origin common to CDWs in other cuprates.

Further confirmation of the nonelectronic nature of the T -independent intensity comes from nonresonant hard XRD measurements. Whereas the resonance at the Cu L_3 edge greatly enhances the contribution of Cu $3d$ electronic charge modulations to the total diffraction signal, allowing the detection of electronic CDWs, nonresonant measurements are sensitive to the total electron density and are therefore dominated by lattice atomic contributions. Since electronic CDWs involve only a fraction of the electronic charge, they are nearly invisible in nonresonant XRD. Figures 3(b) and 3(c) show H scans across Q_4 obtained for films with $x = 0.16$ and 0.48, respectively, using nonresonant hard XRD. Clear temperature-independent peaks are observed near $H = 0.25$ for both samples. This observation demonstrates the presence of a temperature-independent superstructure of nonelectronic origin in the entire Ca dopant concentration range used in

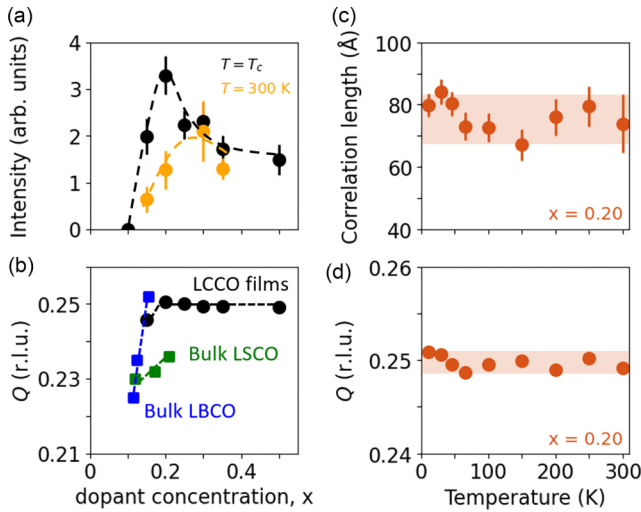


FIG. 4. Ca dopant concentration dependence of (a) the integrated intensity of the superstructure peak and (b) its characteristic wave vector Q , extracted from fits to the REXS data at $T = T_c$. The integrated intensity at 300 K and the CDW wave vectors for bulk LSCO [39,50] and LBCO [60] are included in (a), (b), respectively, for comparison. The dashed lines are guides to the eye. (c), (d) Temperature dependence of the (c) correlation length, $\xi_{||}$ and (d) Q of the superstructure peak in the $x = 0.20$ sample.

our study. This temperature-independent superstructure is also visible in REXS as a temperature-independent intensity at $x = 0.30$, and as a temperature-independent intensity overlapping with the strongly temperature-dependent intensity from the electronic CDW at $x = 0.15$ and 0.20 [Fig. 3(a)].

What is the origin of the four-unit-cell superstructure? Tilt distortions of the oxygen octahedra are known to cause structural phase transitions in LSCO and LBCO [56,57]. However, such distortions produce only half-integer superstructural reflections and disappear around $x \sim 0.25$ [56,57]. Furthermore, LCCO is expected to be less prone to such distortions due to the similar ionic radii of La and Ca. In addition, the structure of the film is locked into that of the tetragonal LSAO substrate, making such distortions less likely in films. Two other possibilities arise, namely, Ca dopant ordering and oxygen ordering. For these two types of superstructures, a concomitant periodicity in the charge on the local Cu atom is expected, which would explain the significant contribution of this superstructure to the REXS signal. If the origin of the superstructure were Ca dopant ordering, one would expect a strong dependence of the intensity, periodicity, and correlation length of the superstructure on the Ca dopant concentration x , which is not observed in our measurements (see Figs. 4(a), 4(b) and Supplemental Material Ref. [44]). On the other hand, previous *in situ* RHEED measurements have documented a reversible switching between 4- and 5-unit-cell configurations of the surface structure during growth under vacuum versus ozone-rich atmosphere, respectively [58] (see also Supplemental Material Ref. [44]). Furthermore, prior Raman spectroscopy studies on LSCO films have shown that the overdoped regime is particularly prone to apical oxygen vacancy formation [45]. These observations indicate that the superstructure we detect is most likely associated with oxygen vacancy ordering.

Optical spectroscopy measurements on LCCO films, in conjunction with electronic-structure calculations, indicate a progressively increasing density of holes in the Cu $3d_{3z^2-r^2}$ orbital with increasing Ca content for $x \geq 0.15$ [43,59]. We cannot rule out a charge-and-orbital ordering scenario with full site occupancy at the apical-oxygen position. In this case, however, we would expect a systematic increase of the REXS signal with increasing x , which is not observed in our measurements [see Fig. 4(a)].

From the considerations above, we infer that the superstructure peak at Ca concentrations $x \leq 0.20$ is a superposition of the temperature-independent oxygen superstructure and a temperature dependent electronic CDW. To determine the electronic CDW component of superstructure peaks observed with resonant x-ray scattering, one therefore needs to look at the contrast between low- T and high- T integrated intensities. Figure 4(a) shows the Ca concentration dependence of the integrated intensity of the peaks obtained at $T = T_c$ and 300 K. Indeed, a significant difference between the intensities obtained at the two temperatures exists only for $x = 0.15$ and 0.20 . This confirms that substantial electronic CDW correlations are present only in this Ca concentration range, in agreement with prior reports on bulk LSCO [38,39]. The increase in the characteristic CDW wave vector with Ca content agrees with prior CDW reports on bulk LSCO [39,50] and LBCO [60] [see Fig. 4(b)]. The stronger CDW intensity at $x = 0.20$, compared to $x = 0.15$, may reflect the van Hove singularity in the electronic density of states at $x = 0.19$ [39,61,62]. The correlation length and Q of the superstructure remain unchanged when the peak's character transitions to partially electronic upon cooling from 300 K to low temperatures [Figs. 4(c) and 4(d)]. Furthermore, the L -dependence of the electronic CDW differs from that observed in bulk single crystals, where the oxygen superstructure is absent [Fig. 2(d)]. These underscore that the oxygen superstructure serves as a template for the electronic CDW. We note that locking of electronic charge-order correlations by the crystal structure was also recently reported in LBCO, where the formation of the low-temperature tetragonal phase coincides with a sudden jump and lock-in of an electronic charge-order reflection [60].

Discussion. We now discuss our results in the context of recent experiments on different systems. In particular, a recent study of LSCO films reported superstructure reflections with $Q_6 \sim 1/6$ in the extremely overdoped regime ($x = 0.35, 0.45$ and 0.60) [42] and assigned those reflections to an electronic CDW. If this assignment were correct, this would imply a major revision of the conventional electronic phase diagram of the cuprates, which classifies highly overdoped systems as featureless Fermi liquids. We therefore searched for peaks in a similar Q range in our LCCO films, with negative results (see Supplemental Material Ref. [44]). We note, however, that the reflections reported in Ref. [42] are almost T independent, as the Q_4 reflections we observed in LCCO, and that they display a closely similar in-plane correlation length and L -dependence. These observations point to a lattice superstructure—likely a different oxygen ordering pattern—rather than an electronic CDW in the extremely overdoped LSCO films.

Prior scanning tunneling microscopy studies reported a doping-independent four-unit-cell periodic charge modula-

tion at the surface of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ (Bi2212) over a wide doping range, which was interpreted as a commensurate CDW [63]. However, these measurements were limited to low temperatures and could therefore not conclusively differentiate between electronic and (possibly sub-surface) oxygen superstructures, necessitating additional measurements with complementary techniques. Our study demonstrates the importance of determining the T dependence to discriminate between electronic and lattice contributions to superstructures in the cuprates.

It is also worth noting that RIXS and REXS experiments on films of the infinite-layer nickelates NdNiO_2 and PrNiO_2 uncovered a superstructure reflection at $Q_3 \sim (1/3, 0)$ with weakly temperature-dependent intensity and correlation length [64–66], similar to our observations for LCCO films with $x > 0.20$. Whereas the reflection was originally attributed to a CDW, follow-up work indicates that the observed signal is at least partially associated with oxygen ordering in insufficiently reduced films [67,68] (see also Ref. [69]).

We note that the presence of oxygen vacancies would inevitably reduce the hole doping from the Ca concentration x , as each oxygen vacancy is expected to contribute two electrons. Prior Hall and optical conductivity measurements are, however, consistent with a hole doping level monotonically increasing with increasing x [43]. Therefore, the density of oxygen vacancies is clearly not large enough to compensate for the holes introduced by Ca substitution. While a direct measurement of oxygen off-stoichiometry—and thus the actual hole-doping level—would be desirable, at the time of writing, quantifying the number density of oxygen vacancies in oxide materials (especially in thin-film structures grown on oxide substrates) remains challenging and an active area of research [70].

Our study indicates that electronic CDWs and oxygen order are separate instabilities that emerge out of a high-temperature state where both electrons and oxygen ions are mobile. Correlation-driven electronic CDWs dominate at low hole concentrations ($x = 0.15 - 0.20$), but fade out around optimal doping. Oxygen vacancies and oxygen-vacancy order, on the other hand, proliferate with increasing doping. Both instabilities overlap over some range of doping, where we observe templating of the CDW state by oxygen order. Our experiments thus establish LCCO as a model system in which

the interdependence of both phenomena can be studied over a large range of the phase diagram, with minimal complications from disorder. We emphasize the key role of the temperature dependence in distinguishing both forms of order.

In the cuprates, the stability of different oxygen-ordered phases has thus far only been described in terms of semiempirical parameters [16–18]. By treating the lattice energetics and electronic structure self-consistently, research on disordered metallic alloys has offered microscopic insight into the formation of ASRO, which is also instructive for the case at hand [7–12]. Specifically, the oxygen ions are mobile during synthesis of oxide compounds at high temperatures, and their positional correlations are expected to be influenced by the charge susceptibility of the conduction electron gas, which exhibits broad maxima near Q_4 over hundreds of meV [71,72], which are expected to persist at high temperatures. As these correlations are frozen in upon cooling, interactions with the lattice favor commensurate superstructures (see Supplemental Material Ref. [44]). The four-unit-cell oxygen superstructure we observe in turn affects the periodicity and correlation volume of the CDW [73]. *Ab initio* electronic structure theory and thermodynamics will be required to fully elucidate the interdependence of ASRO and electronic structure in cuprates, nickelates, and other complex oxides. Control of ASRO during growth [58] could serve as an additional tuning knob to develop oxide materials for enhanced functional properties. As the interplay between ASRO and such properties is also of growing importance for electrochemical devices [2–4], this line of research will open a new frontier at the nexus of quantum and energy science.

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Data availability. The data that support the findings of this article are openly available [74].

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