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Bulk charge density wave and electron-phonon coupling in superconducting copper oxychlorides

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Bulk charge density waves (CDWs) are now reported in nearly all high-temperature superconducting (HTS) cuprates, with the noticeable exception of one particular family: the copper oxychlorides. Here, we used resonant inelastic X-ray scattering (RIXS) to reveal a bulk CDW in these materials. Combining RIXS with non-resonant IXS, we investigate the interplay between the lattice excitations and the CDW, and evidence bond-stretching (BS) phonon anomalies at the CDW wave-vector. We propose that such electron-phonon anomalies occur in the presence of dispersive charge excitations emanating from the CDW and interacting with the BS phonon. Our observations in a structurally simple cuprate promises to better connect bulk and surface properties and bridge the gap between theory and experiment.

The experimental studies of CDWs in HTS cuprates were initiated with bulk-sensitive scattering measurements of La-based cuprates [1] and surface-sensitive scanning tunneling spectroscopy (STS) on Bi-based cuprates and copper oxychlorides ($\text{Ca}_{2-x}\text{Na}_x\text{CuO}_2\text{Cl}_2$) [2, 3]. Then, the evolution and diversification of the techniques fertilized the experimental field of CDW orders in cuprates, providing deeper insights on a larger scale of compounds [4–12]. However, despite numerous efforts, a unified description of the CDW is not yet established due to certain dichotomies between surface and bulk studies as well as distinct properties found in different cuprate families [7, 8]. Such discrepancies have raised substantial questions about the nature and universality of the CDW formation mechanism in HTS cuprates and stimulated scenarios involving either real-space local interactions or Fermi surface nesting [1, 4, 13–16]. Experimental studies have collected evidence for both scenarios and no consensus has been reached so far.

Combining surface and bulk probes on the same material can provide information on the CDW formation mechanism and its relation with both the superconduct-

tivity and the pseudogap. This approach was applied to the Bi-based family, which are, to date, the only HTS cuprates where a CDW was evidenced by both surface and bulk probes [17, 18]. However, they have a large unit cell and a tendency to form complex, incommensurate lattice superstructures [19]. $\text{Ca}_{2-x}\text{Na}_x\text{CuO}_2\text{Cl}_2$ (Na-CCOC), whose surface electronic structure has been thoroughly explored [3, 4], is an appealing alternative offering unique characteristics. Fortuitously, this system has a simple tetragonal $I4/mmm$ crystal structure [Fig. 1 (a)] without any structural instabilities [20] and presents strong 2D electronic properties [21], representing an ideal test bench to study the intrinsic physics from CuO_2 planes [4]. Although Na-CCOC appears as an ideal choice for studying bulk and surface CDW correlations, the existence of such order in its bulk electronic properties remains an open question. This is a central issue, as Na-CCOC was among the first HTS cuprate in which STS measurements successfully revealed a surface CDW, that is observed over a doping range from $x = 0.08$ to $x = 0.12$ [3, 22] but no bulk CDW was detected in previous X-ray study [23]. Addressing this issue is crucial to

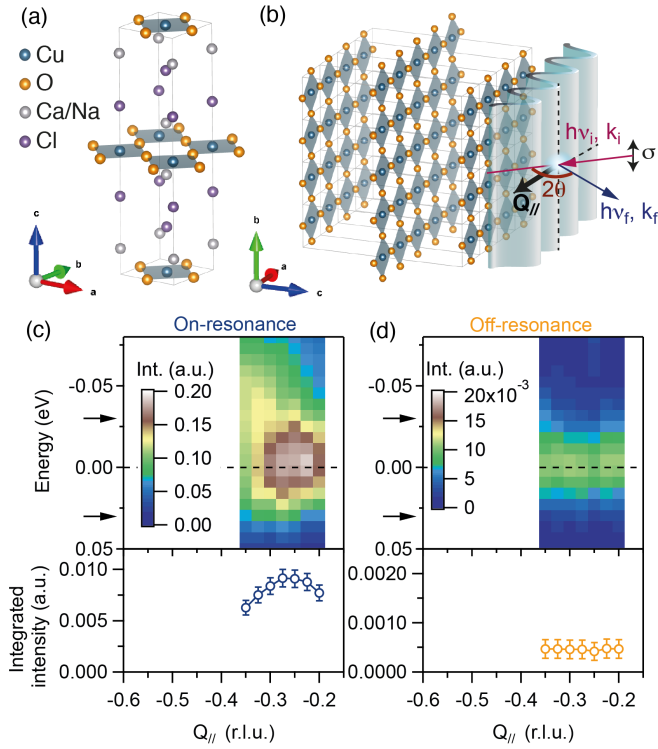


FIG. 1: (a) Crystal structure of Na-CCOC [24]. Cl ions are located on the apical sites. The four surrounding O form the square coordination of Cu, which is highlighted in blue. a , b and c correspond to the lattice axes of the sample. (b) Sketch of the scattering geometry used for our RIXS experiment with respect to the crystal structure of Na-CCOC and in the presence of a CDW. The incident beam was polarized with linear vertical polarization (σ -polarization). All the RIXS data shown in this manuscript are plotted as function of the projected in-plane momentum transfer Q_{\parallel} (along the a -axis) in reciprocal lattice units (r.l.u.). (c), (d) RIXS intensity maps recorded on- (c) and off- (d) resonance (top panels) as function of the energy and Q_{\parallel} . The integrated intensities in the quasi-elastic regions (indicated with black arrows on the RIXS intensity maps) are plotted on the bottom panels.

clarify whether the CDW seen by STS is representative of the bulk electronic properties or only reflects a charge order pinned at the surface [25].

In this Letter we combine high energy-resolution RIXS at the Cu L_3 -edge [Fig. 1 (b)] and IXS measurements (see Supplemental Material Note 1 for details about experimental methods) to investigate CDW and electron-phonon coupling (EPC) anomalies in Na-CCOC. Our results provide the first direct observation of a bulk and incommensurate CDW in this material. Concomitant to this observation, our RIXS and IXS data revealed anomalies on the BS phonon characterized by (i) a softening and broadening at the CDW wave-vector, (ii) a RIXS intensity anomaly at $Q_A > Q_{CDW}$ and (iii) a funnel-like RIXS intensity emanating from the CDW. Together these findings, which are not observed in the undoped

sample, point toward CDW-induced EPC anomalies that are interpreted in terms of dispersive charge excitations emanating from the CDW and interacting with the BS phonon.

The RIXS technique has recently emerged as decisive to detect incipient electronic orders such as CDW and its corresponding excitations/fluctuations [7, 9–12, 26–29]. Figure 1 (c) displays a RIXS intensity map recorded on-resonance and at 25 K in Na-CCOC. Enhanced quasi-elastic intensity peaking at an in-plane momentum transfer of $Q_{\parallel} \sim -0.26$ r.l.u. (reciprocal lattice units) is detected. In contrast, the intensity drops by two orders of magnitude in the same energy- and momentum-range, when the incident photon energy is off-resonance [Fig. 1 (d)]. The observed quasi-elastic peak is therefore dominated by the resonant process, indicating that it is associated with the electronically active Cu states.

More quantitative assessments can be achieved by analyzing the high-resolution ($\Delta E \sim 50$ meV) RIXS intensity map displayed in Fig. 2 (a). The RIXS intensity integrated over a small energy window (-25 meV $< E < 25$ meV) around the zero-energy loss is shown in Fig. 2 (b). A quasi-elastic peak is confirmed. The peak position is found at $Q_{CDW} \sim -0.27 \pm 0.01$ r.l.u., while its FWHM $\sim 0.08 \pm 0.01$ r.l.u., indicates a correlation length of $\xi \sim 15 - 20$ Å. Note that measurements at positive Q_{\parallel} confirm the observations of a quasi-elastic peak at $Q_{CDW}^{\dagger} \sim 0.28 \pm 0.01$ r.l.u., with a FWHM $\sim 0.06 \pm 0.01$ r.l.u. (see Supplemental Material, Note 2). This peak is the signature of an incommensurate short-range CDW, that is static or quasi-static in nature (on a timescale of ~ 160 fs, limited by the experimental energy resolution). Upon increasing the temperature to 245 K, the CDW peak is absent, as shown in Fig. 2 (b), and only the specular scattering background remains, indicating that the CDW correlates when decreasing the temperature.

We now focus on the inelastic features and investigate the interplay between the lattice excitations and the CDW. Indeed, the study of the phonon spectra can provide insight on the driving force behind its formation mechanism, possibly revealing Kohn anomaly resulting from Fermi surface nesting or any momentum-dependent EPC [30]. Inelastic neutron scattering (INS) and IXS have identified anomalies in the phonon spectra close to the CDW wave-vectors in several HTS cuprates, suggesting an intimate relation with the lattice [31–39]. These anomalies could be connected to the origin of the CDW, and have been discussed in term of CDW-induced modification of the lattice [26], hybridization between different phonon modes [33, 38] or even the presence of collective charge excitations [40]. Despite of being intensively studied over the past 30 years, the very nature of these phonon anomalies remains puzzling in HTS cuprates, calling for a new paradigm in phonon investigation. Because of its sensitivity to EPC and the recent technical

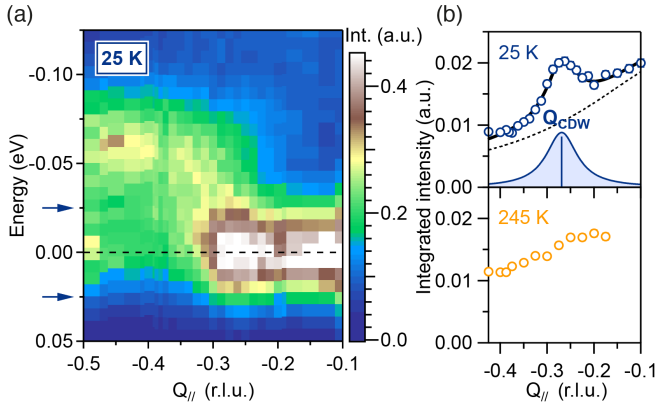


FIG. 2: (a) RIXS intensity maps recorded at 25 K with an energy resolution of $\Delta E \sim 50$ meV, as function of the energy and $Q_{||}$. (b) Momentum-dependence of the integrated quasi-elastic intensity at 25 K (top) and 245 K (bottom). The quasi-elastic region is defined as between the two blue arrows on (a). The black solid line is a fit to the data consisting of two Lorentzians, one for the CDW and the other for the tail of the specular reflection peak at $Q_{||} = 0$, plus a constant. The fit of the CDW peak is highlighted in blue. The CDW wave-vector is indicated with a vertical solid line. The background corresponding to the tail of the specular reflection peak is shown with a dotted line.

progress made on its energy-resolution, the RIXS is now becoming a key complementary technique to investigate phonon spectra and EPC in HTS cuprates.

As presented in Fig. 3 (a), the energy-loss RIXS spectrum at $Q_{||} = -0.4$ r.l.u. and 25 K shows an excitation centered at ~ 60 meV, between the elastic peak and the tails of the magnetic excitations observed at higher energy. The dispersion of this excitation is obtained by plotting the peak position as function of $Q_{||}$ [Fig. 3 (b)]. To identify this excitation, we have performed IXS measurements on another Na-CCOC sample from the same batch. As shown in Fig. 3 (d) displaying IXS spectrum at $Q = (2.6 \ 0 \ 0)$ and 25 K, a peak corresponding to a phonon mode is observed at 60 meV, whose dispersion is plotted in Fig. 3 (e). Its energy and dispersion agree well with the 60 meV excitation observed in the RIXS data, indicating that both excitations, *i.e.* the one seen in RIXS and the one seen in IXS, have common origin. A comparison to density functional theory (DFT) calculations shown on Fig. 3 (b) and (e) and other HTS cuprates indicate that this mode corresponds to the Cu-O BS phonon [31–36, 41, 42]. Interestingly, as seen by both RIXS and IXS, the BS phonon dispersion softens. This softening presents two components. First, a doping-induced softening in a form of a cosine-like dispersion along $(q \ 0 \ 0)$, which has been already reported in a earlier IXS study [36] and reproduced well by the DFT. Then, a pronounced dip is observed at $Q \sim 0.3$ r.l.u., *i.e.* close to the CDW wave-vector, which is not captured by the DFT [43, 44]. This dip coincides with a peak width broaden-

ing occurring between 0.2 and 0.4 r.l.u. [Fig. 3 (f)] and resembles the phonon anomaly observed in conventional metallic systems such as NbSe₂, where the EPC is known to have a substantial implication on the formation of the CDW [30]. Our observations of BS phonon anomalies at the CDW wave-vector indicate that the CDW affects the lattice dynamics, and in the present case the BS mode, by renormalizing its frequency and linewidth. This points toward an intimate connection between the EPC and the CDW that deserves further examination.

To study the role of the EPC, we can examine the RIXS phonon intensity. Indeed, this technique has recently been used to quantify the EPC strength in several HTS cuprates, confirming a momentum dependent EPC that is strongest at the zone boundary for the BS mode [45, 46]. This is due to the intrinsic nature of the RIXS process, in which the phonons are excited through the EPC interaction [47, 48]. Consequently, the RIXS phonon intensity is proportional to the EPC strength and can thereby reflect interactions between phonons and underlying charge excitations [10–12]. As a function of the momentum, the EPC should scale as $I(Q) \propto \sin^2(\pi H) + \sin^2(\pi K)$ for the BS mode [48], *i.e.* $\sin^2(\pi Q_{||})$ for our scattering geometry. However, Fig. 3 (g) shows that a deviation is observed in our data. Indeed, the momentum-dependence of the BS phonon integrated intensity presents an anomaly at $|Q_A| > |Q_{CDW}|$, as indicated by the difference plot shown in the bottom panel of Fig. 3 (g), and confirmed by the analysis of the positive $Q_{||}$ data (see Supplemental Material, Note 2). A Lorentzian fit of the difference gives $Q_A \sim -0.36 \pm 0.02$ r.l.u. This deviation confirms the presence of EPC anomalies in the vicinity of the CDW.

Similar EPC anomalies have been observed in La_{1.8-x}Eu_{0.2}Sr_xCuO_{4+δ} [49, 50] and Bi-based cuprates [10–12] using RIXS. The former seems to occur in a narrow momentum region near the CDW wave-vector and has been discussed in term of enhanced EPC within the CDW phase that further stabilizes the CDW modulation [49]. The latter however occurs for $|Q| > |Q_{CDW}|$ and has been attributed to originate from an interference between the BS phonon and charge excitations emanating from the CDW [10–12]. Due to their dispersive nature, these excitations broaden in momentum with increasing energy to form a funnel-like intensity emanating from the CDW. Once crossing the BS phonon, an interference occurs that leads to an intensity anomaly in the RIXS data. Since this interference is dictated by the EPC, which increases monotonically with the momentum for the BS mode, the intensity anomaly is more visible for $|Q| > |Q_{CDW}|$, *i.e.* where the EPC is strong [10–12]. Our observation of an intensity anomaly occurring at $|Q_A| > |Q_{CDW}|$ therefore favors the second scenario, *i.e.* the interference between the BS phonon and charge excitations dispersing from the CDW. This is further supported by the RIXS intensity map presented in Fig. 2 (a) showing an additional

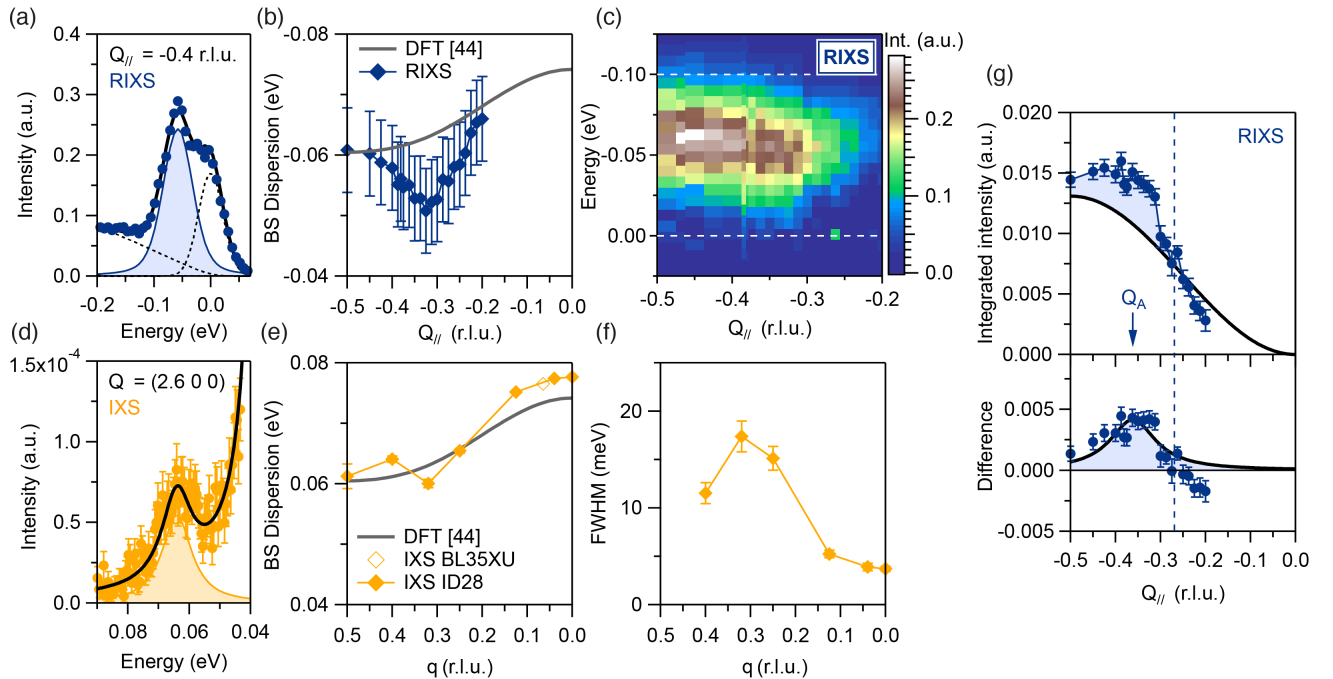


FIG. 3: (a) RIXS spectrum at 25 K and -0.4 r.l.u. demonstrating the fit quality. The fitted phonon is highlighted in blue. (b) BS phonon dispersion at 25 K obtained with the RIXS data. DFT calculations, extracted from ref. [44], are shown with a grey solid line. (c) RIXS intensity map at 25 K, with subtracted elastic and paramagnon peaks. The raw data are the same than those shown in Fig. 2 (a). (d) IXS spectrum at 25 K and $Q = (2.6\ 0\ 0)$ demonstrating the fit quality. The fitted phonon is highlighted in orange. (e), (f) BS phonon dispersion (e) and width (f) obtained from the IXS data at 25 K. IXS spectra have been measured at both BL35XU (SPring-8) [one data point at $Q = (3.065, 0.003, -0.073)$] and ID28 (ESRF) [full dispersion along $Q = (3\ 0\ 0) - (q\ 0\ 0)$] beamlines. (g) Momentum-dependence of the RIXS integrated intensity of the elastic/paramagnon-subtracted data at 25 K. The integrated energy window is 0-100 meV [white dashed lines in (c)]. The black solid line is the $\sin^2(\pi Q_{\parallel})$ curve. The bottom panel represents the difference between the integrated intensity and the $\sin^2(\pi Q_{\parallel})$ curve, with Lorentzian fit (solid line). The dashed line indicates Q_{CDW} . The arrow highlights the anomaly wave-vector Q_A .

scattering intensity smoothly connecting the CDW and the BS phonon and forming a funnel-like intensity emanating from the CDW [Fig. 3 (c)]. Together, these observations point toward the existence of dispersive excitations associated with the CDW. By connecting Q_{CDW} and Q_A , we determined a velocity for these excitations: $v_{\text{CDW}} \sim 0.4$ eV $\cdot\text{\AA}$, consistent with the velocities found in Bi-based cuprates [10, 11]. This result differs from a recent RIXS study on $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$, where no EPC anomalies have been detected and in which the phonon softening has been attributed to a CDW-induced modification of the lattice without any coupling to electronic excitations [26].

The CDW nature of the observed anomalies is confirmed by recording RIXS spectra on the undoped $\text{Ca}_2\text{CuO}_2\text{Cl}_2$ (CCOC) sample. Fig. 4 (a) presents the energy-momentum intensity map, with the fitted elastic peak and magnons subtracted. As expected, a good agreement is obtained by comparing the dispersion of the 70 meV RIXS excitation (white diamonds) to the one of the BS phonon measured by IXS on another CCOC sample from the same batch (black diamonds) [44]. The CCOC BS phonon is almost dispersionless, with no soft-

ening in the probed momentum range. In addition, the momentum-dependence of the quasi-elastic peak confirms the absence of the CDW in CCOC [Fig. 4 (b)]. As anticipated, the phonon integrated intensity shown in Fig. 4 (c) increases monotonically with Q_{\parallel} . More importantly, its momentum-dependence is well captured by the $\sin^2(\pi Q_{\parallel})$ trend, consistent with predictions due to EPC for the BS phonon [45, 48]. These observations confirm that the phonon and EPC anomalies observed in Fig. 3 exist only in doped CCOC, where the CDW is present.

To summarize our results, the temperature-hole doping phase diagram of Na-CCOC is displayed in Fig. 4 (d). Na substitution for Ca in CCOC dopes the Mott insulating state, inducing a metallic state with no long-range magnetic order and superconductivity at higher doping [51–53]. Our study unambiguously reveals a bulk CDW at 25 K in the underdoped region of the phase diagram, *i.e.* close to the doping concentration of $x \sim 0.1$. This is consistent with STS studies which observe surface CDWs for $x = 0.08, 0.10$ and 0.12 [3]. Similarly to the STS data, our results suggest that the CDW is short-ranged ($\sim 15 - 20$ \AA). This is comparable to the correlation lengths found in Bi-based cuprates, known for exhibiting

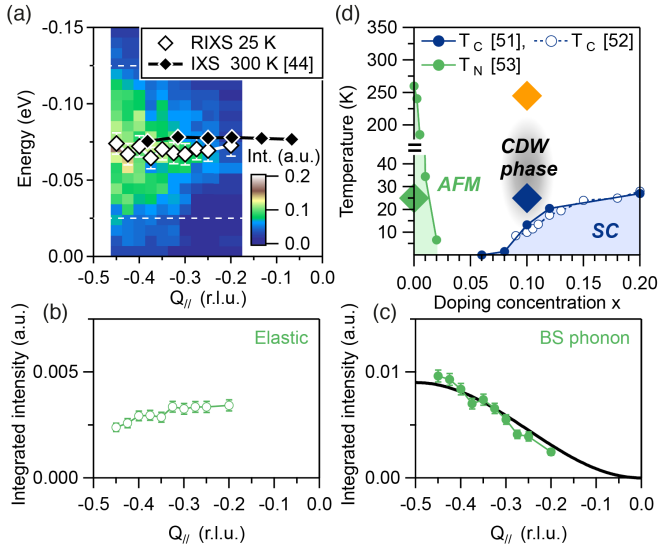


FIG. 4: (a) Elastic and magnons-subtracted RIXS intensity map of CCOC at 25 K. The dispersion of the BS phonon extracted from the RIXS data is displayed with white diamonds. The black diamonds show the dispersion of the BS phonon from IXS measurements, extracted from ref. [44]. (b) Momentum-dependence of the integrated quasi-elastic RIXS intensity of the CCOC data. The integrated energy window is similar to Fig. 2, *i.e.* the two blue arrows in Fig. 2 (a). (c) Momentum-dependence of the integrated RIXS intensity of the elastic and magnon-subtracted data of CCOC. The integrated energy window corresponds to 25-125 meV [white dashed lines in (a)]. The black solid line corresponds to the $\sin^2(\pi Q_{\parallel})$ curve. (d) Temperature-doping phase diagram of Na-CCOC. The onset temperatures, *i.e.* T_N for the AFM phase and T_C for the superconducting one, are extracted from ref. [51–53]. The different doping concentrations and temperatures probed in our study are reported with filled diamonds.

chemical disorder and inhomogeneity [8]. Our RIXS data also demonstrate that the CDW is incommensurate with $Q_{\text{CDW}} \sim -0.27 \pm 0.01$ r.l.u. This is in contrast with the STS studies, where the surface CDW was found to be commensurate [3]. This suggests that the commensurate CDW is not representative of the bulk electronic properties and only located at the surface, possibly stabilized by a soft surface phonon [25]. Another possibility is that the CDW is locally commensurate, locked to a $4a_0$ periodicity, but appears incommensurate in X-ray studies, through discommensuration effects [54].

Concerning the inelastic features, our IXS measurements revealed anomalies in the BS phonon, resulting in a peak width broadening and a dip in its dispersion at Q_{CDW} , which is not reproduced by the DFT. We also evidenced that such lattice anomalies are concomitant to RIXS intensity anomalies which are characterized by a momentum-dependent EPC that deviates from the predictions for the BS mode and a funnel-like intensity emanating from the CDW. Together, these results point toward the existence of additional charge excitations dis-

persing from the short-range CDW at 25 K and interacting with the BS phonon at 60 meV. This extends the observation of such excitations outside the Bi-based family [10–12], whose nature has been discussed in term of a collective mode corresponding to SU(2) fluctuations [55] or a continuum of quantum fluctuations that could melt the CDW below T_C [12].

RIXS experiment was performed at the ID32 beamline of the European Synchrotron Radiation Facility (ESRF, Grenoble, France) supported by a beamtime allocation under the proposal HC-3884. IXS measurements were performed at the ID28 beamline of ESRF supported by a beamtime allocation under the proposal HC-4310. Additional IXS measurements were conducted at BL35XU of SPring-8 (Japan) under the SPring-8 Budding Researchers Support Program (proposal No. 2019B1779). We thank O. Leynaud for access to the X’Press diffractometer platform as well as L.-M. Chamoreau and B. Baptiste for access to the “Plateforme Diffraction” at IPCM, Paris, and providing help for the single-crystal XRD experiments. We thank L. Del-Rey and D. Dufeu for conceiving the sample holders and J. Debray for his help for the preparation of the RIXS experiment. We thank L. Laversenne and N. Bendiab for access to the glove-boxes. We thank P. Rodière and M.-H. Julien for fruitful discussions. We acknowledge financial support from ANR-DFG grant ANR-18-CE92-0014-03 “Aperiodic”, IRS IDEX/UGA and Grants-in-Aid for Scientific Research 18H05208, and 19H05625, from the Japan Society for the Promotion of Science (JSPS).

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