Comparison of the empty electronic states of $Bi_2Sr_2CaCu_2O_8$ (001) and $Bi_2Sr_2CuO_6$ (001) at 60 and 300 K

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Inverse photoelectron spectroscopy has been used to study the unoccupied electronic states of $Bi_2Sr_2CaCu_2O_8$ (001) and $Bi_2Sr_2CuO_6$ (001) at 60 and 300 K. Both compounds exhibit low emission between E_F and E_F+2 eV derived from Cu $3d$–O $2p$ and Bi $6p$–O $2p$ hybrids and structure centered at $\sim$4 and 9.5 eV that can be attributed to empty Bi $6p$ and Sr $4d$ levels. Results for $Bi_2Sr_2CaCu_2O_8$ show additional structure near E_F+10 eV because of contributions from Ca $3d$ levels. Plasmons are found for both compounds at 15 and 21.2 eV. Off-normal studies of $Bi_2Sr_2CaCu_2O_8$ (001) at 60 K showed O $2p$ holes with $p_{x,y}$ character. Results obtained at 300 K showed decreased O $2p$ hole emission and increased Cu $3d^{10}$ emission for both $Bi_2Sr_2CaCu_2O_8$ and $Bi_2Sr_2CuO_6$ compared with the 60 K results.

I. INTRODUCTION

A great deal of attention has been placed on elucidating the mechanism(s) responsible for high-T_c superconductivity. Frequently, comparisons have been made among similar compounds that exhibit very different T_c's. For instance, $La_{2-x}Sr_xCuO_4$ has O $2p$ hole states and $T_c\sim32$ K, while the antiferromagnetic insulator La_2CuO_4 does not.[1] In general, $RBa_2Cu_3O_{7-x}$ systems exhibit superconductivity at 93 K and have O $2p$ hole states for $x<0.5$, but the O $2p$ hole states are quenched for $x>0.5$ and superconductivity is not observed (R denotes the rare-earth elements except Tb, Ce, and Pr).[1–4] Intriguingly, O $2p$ hole states have been reported for $PrBa_2Cu_3O_{7-x}$, but high-temperature superconductivity has not been found.[4–5] For $YBa_2Cu_3O_{7-x}$, 10% substitution of Zn for Cu suppresses superconductivity without apparently altering the O $2p$ hole states.[6] Hence O $2p$ holes are necessary, but not sufficient, for high-T_c superconductivity.[4]

Variations in T_c have also been observed for the $Bi_2Sr_2Ca_{n-1}Cu_nO_y$, where n represents the number of CuO_2 planes in each crystal sublattice. Transition temperatures of $\sim$10, 85, and 110 K have been obtained for the $n=1$, 2, and 3 compounds, respectively.[7] Photoemission studies have shown similar core-level and valence-band structure for the $n=1$ and 2 compounds,[8] with slightly higher relative emission near E_F for $n=2$ due to the second CuO_2 plane.[8,9] They also showed that these compounds cleave between Bi-O layers.[8]

In this paper, we show that the empty electronic states of $Bi_2Sr_2CaCu_2O_8$ (001) and $Bi_2Sr_2CuO_6$ (001) are nearly indistinguishable, as revealed by tunable-energy inverse photoelectron spectroscopy (IPES). Temperature-dependent studies were undertaken, in part because of the proposed structural phase transition at $\sim$210 K,[10] and they show increased O $2p$ hole emission and decreased Cu $3d^{10}$ emission at 60 K. No temperature-dependent changes were observed for the Bi, Sr, and Ca levels, and this indicates that the changes were confined to the CuO_2 planes. Momentum-resolved inverse photoelectron spectroscopy (KRIPES) results acquired at 60 K for off-normal incidence for $Bi_2Sr_2CaCu_2O_8$ (001) showed O $2p$ hole states with $p_{x,y}$ character.

II. EXPERIMENT

The IPES experiments were conducted in a four-chamber system operating at a base pressure of 1×10^{-10} Torr.[11] A collimated monoenergetic electron beam (current ~10 μA, beam spot 1×5 mm^2) was provided by a Pierce-type electron gun manufactured by Kimball Physics. The angular divergence of the electron beam ($<3°$ at 25 eV) gave a momentum resolution of ~0.1 Å^{-1}. Emitted photons were dispersed by an $f/3.5$ grating onto a position-sensitive detector. The combined energy resolution of the spectrometer (electrons and photons) was 0.3–1.0 eV for photons detected at energies of 10–44 eV. The samples were cooled to 60 K with a closed-cycle helium refrigerator via a copper braid. The temperature was determined with a Au–0.07 at. % Fe versus chromel thermocouple attached to the same holder.

Single crystals of $Bi_2Sr_2CaCu_2O_8$ (001) and $Bi_2Sr_2CuO_6$ (001) were prepared by grinding high-purity reagents with stoichiometric ratios for [Bi]:[Sr]:[Ca]:[Cu] of 2:2:2:3 and 2:2:0:3, respectively.[12] After sintering at 850 °C, they were reground and placed in Al_2O_3 crucibles. Following annealing at 1050 °C (2 h) and 950 °C (12 h), they were cooled to 850 °C at a rate of 2 °C/h, then cooled to room temperature at a rate of 50 °C/h. The stoichiometries were determined to be $Bi_2Sr_2CaCu_2O_8$ (2:2:1:2) and $Bi_2Sr_2CuO_6$ (2:2:0:1). The superconducting transition

temperature for the 2:2:1:2 samples was measured to be 85 K, while the 2:2:0:1 samples were not superconducting. The single crystals shared a common [001] axis but had randomly oriented *a-b* domains.

The single crystals ($8\times8\times0.25$ mm^3) were mounted on Cu posts with conducting epoxy and were covered with an epoxy bead. They were transferred into the measurement chamber after bakeout, and the beads were pried off *in situ* at 60 or 300 K to expose mirrorlike (001) surfaces. Immediately after cleaving, a surface-sensitive KRIPES spectrum was acquired with an incident electron energy of 18 eV. Periodic checks showed no changes that could be related to electron bombardment or residence in vacuum. Low-energy electron-diffraction (LEED) studies showed multiple domains that were randomly oriented about the *c* axis, i.e., [001].

III. RESULTS AND DISCUSSIONS

A. IPES of $Bi_2Sr_2CaCu_2O_8$ and $Bi_2Sr_2CuO_6$ at 60 K

In Fig. 1 we present photon-distribution curves (PDC's) acquired at 60 K from 2:2:1:2 (solid lines) and 2:2:0:1 (dashed lines). These spectra are referenced in energy to the Fermi level E_F and the incident electron energies E_i are referenced to E_F. All spectra were acquired with incident electrons normal to the (001) surfaces, and a constant photon-energy feature at $h\nu=21.2$ eV is marked with arrows, and it can be seen to move to the right in Fig. 1 as E_i is increased. The spectra for 2:2:1:2 and 2:2:0:1 are renormalized to the emission intensity at $h\nu=21.2$ eV to facilitate comparison. Analogous IPES spectra have been shown previously[13] for polycrystalline 2:2:1:2.

The spectra in Fig. 1 reveal low emission for states from E_F to E_F+2 eV for both 2:2:1:2 and 2:2:0:1. These states are due to the Cu $3d$–O $2p$ and Bi $6p$–O $2p$ hybrids.[14–16] The shoulder at E_F+4 eV is attributed to higher-lying Bi $6p$ levels that are predicted to be 2.5 eV above E_F,[14–16] i.e., ~1.5 eV closer to E_F than observed. We will discuss the states near E_F in Fig. 2.

The strong emission observed between E_F+6 and 14 eV in Fig. 1 is due to empty Sr $4d$–derived and Ca $3d$–derived levels. Comparison of spectra acquired at $E_i=35$ and 39 eV indicates a significant increase in emission from the feature centered at ~9.5eV above E_F. Since this enhancement occurs for both compounds, we attribute it to the Sr $4p$-d giant-dipole-resonant transition. Such empty-state resonances have been observed for many of the early transition elements, and they have been discussed in detail for Sc.[17] A further increase in E_i to 43 or 47 eV produces additional enhancement for 2:2:1:2 that can be ascribed to the Ca $3p$-$3d$ giant-dipole-resonant transition (centered at ~10 eV with maximum intensity at $E_i=45$ eV). The higher-energy resonance for Ca compared to Sr is expected since the binding energy of the Ca $3p$ core level is ~5 eV greater than that of the Sr $4p$ core level.[18] The empty-state values of ~9.5 and 10 eV above E_F for the Sr $4d$ and Ca $3d$ bands are consistent with the highly ionic character of the cuprate oxides [compare to centroids for metallic Sr $4d$ and Ca $3d$ bands at ~5 eV (Ref. 19) and ~6 eV (Ref. 20) above E_F].

A constant photon-energy feature centered at $h\nu=21.2$ eV can be seen in Fig. 1 for both 2:2:1:2 and 2:2:0:1. Although not shown, another constant photon-energy feature appears at $h\nu=15$ eV. In a previous IPES study of polycrystalline 2:2:1:2,[13] we attributed the constant photon features at 15 and 21.2 eV to radiative plasmon losses. These plasmon loss features have a full width at half maximum (FWHM) of 3 eV, and they have similar properties to the plasmon-loss features reported for Sb.[21] As shown in Fig. 1, the 21.2-eV plasmon-loss feature is indistinguishable for 2:2:1:2 and 2:2:0:1, as is the $h\nu=15$-eV feature. The energies of the two plasmon features differ by a factor of 1.4, suggesting that the lower-energy feature can be attributed to a surface plasmon. Since the 2:2:1:2 and 2:2:0:1 compounds have similar planar character and electron densities, it is not surprising that they exhibit similar bulk plasmon losses. Furthermore, since both cleave between Bi-O planes, they will have similar surface properties, and the radiative decay of the surface plasmons should be equivalent.

In Fig. 2 we compare emission from the empty electronic states of 2:2:1:2 (left panel) and 2:2:0:1 (right panel) between E_F and E_F+4 eV. The spectra were acquired within 30 min of cleaving at 60 K with normal incident electrons of $E_i=16$ eV (dashed lines) and 18 eV (solid

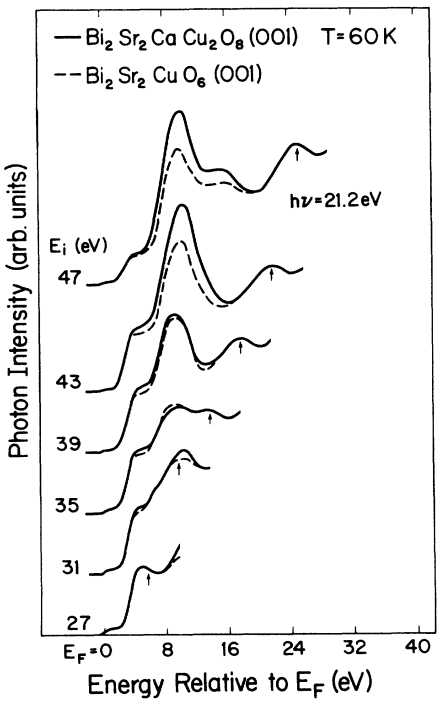

FIG. 1. Normal incidence IPES spectra as a function of incident electron energy E_i for 2:2:1:2 (solid line) and 2:2:0:1 (dashed line). Cu $3d$–O $2p$ and Bi $6p$–O $2p$ states lie between E_F and E_F+2 eV, the Bi $6p$ levels produce structure at E_F+4 eV, and the Sr $4d$ levels are centered at $E_F+9.5$ eV. The spectra for the two compounds differ for $E_i=43$ and 47 eV because the Ca $3p$-$3d$ giant dipole resonance in 2:2:1:2 highlights the Ca $3d$ states. The constant photon-energy feature at $h\nu=21.2$ eV represents radiative plasmon decay.

lines). The four IPES spectra of Fig. 2 were normalized to constant height at E_F+4 eV. (Typically, we normalize to electron doses, but the samples were electrically grounded during the low-temperature studies. Normalization at a fixed energy allows comparisons for a given material, but quantitative comparisons between 2:2:1:2 and 2:2:0:1 should not be made.) These IPES spectra show the well-defined E_F cutoff characteristic of the Bi-Sr-(Ca)-Cu-O compounds. The cutoff appears more distinctly for these compounds than for La-Sr-Cu-O and Y-Ba-Cu-O because of the added Bi $6p-$O $2p$ states at E_F.[4]

In Fig. 2, we overlay the $E_i=16$- and 18-eV spectra to emphasize the enhanced structure between E_F and E_F+2 eV that occurs for $E_i\sim 18$ eV in all of the cuprate superconductors.[4] This enhancement has been attributed to an O-derived resonance whereby the incident electron beam induces O $2s-$O $2p$ transition. Radiative decay channels of the core hole, $O(2s^1 2p^6)\rightarrow O(2s^2 2p^5)+h\nu$, couple with the IPES continuum and produce resonant enhancement.[22] The differences between the $E_i=16$- and 18-eV spectra are shown at the bottom of Fig. 2, multiplied by three for visual comparison. The difference curves show nearly identical O $2p$ hole character for 2:2:1:2 and 2:2:0:1, despite the large differences in transition temperatures (85 versus 10 K). This suggests that there are no fundamental differences in the O $2p$ hole character for these two Bi-Sr-(Ca)-Cu-O compounds, at least on the energy scale of these measurements.

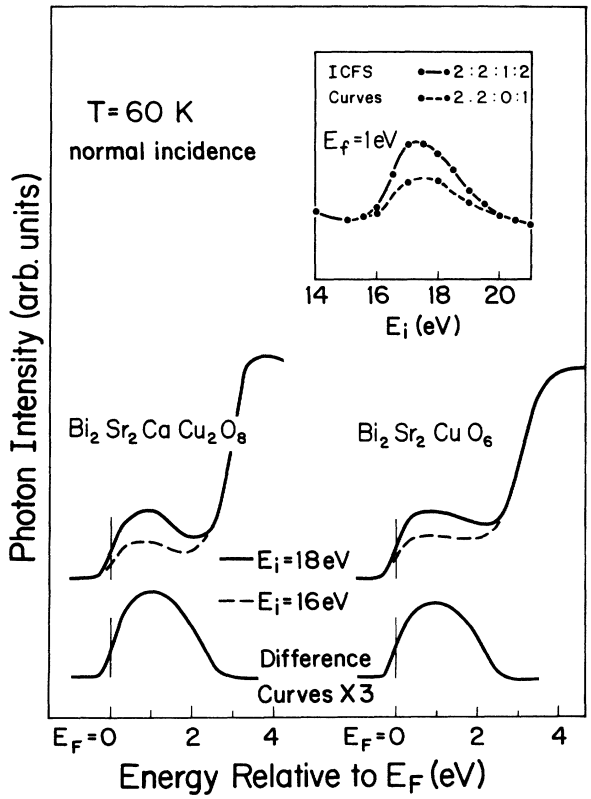

FIG. 2. IPES spectra for 2:2:1:2 and 2:2:0:1 to emphasize the empty states between E_F and E_F+4 eV. Spectra for $E_i=16$ and 18 eV correspond to off and on the O $2s$-$2p$ resonant transition. The O $2p$ character is nearly identical for 2:2:1:2 and 2:2:0:1. Inverse-constant final-state energy spectra shown in the inset display a Fano line shape and an enhancement that is twice as large for 2:2:1:2 because of the extra CuO_2 plane.

The inset of Fig. 2 shows inverse-constant final-state (ICFS) curves for 2:2:1:2 (solid line) and 2:2:0:1 (dashed line) taken at a final-state energy of E_F+1 eV. This final-state energy corresponds to the peak in intensity of the empty O $2p$ states. The Fano line shape exhibited by the ICFS curves is consistent with enhancement due to an O $2s$-$2p$ resonant transition. The ICFS curves are renormalized to the same background emission between $E_i=20$ and 21 eV and show that the amount of enhanced emission above background is approximately twice as large for the 2:2:1:2 compound. This suggests greater O $2p$ hole character in the 2:2:1:2 compound. The consequence can be seen in the IPES spectra acquired at $E_i=18$ eV. Indeed, the peak for 2:2:1:2 occurs clearly at E_F+1 eV whereas 2:2:0:1 has a somewhat flatter distribution between E_F and E_F+2 eV. These results can be related to the existence of two CuO_2 planes in 2:2:1:2 compared to one in 2:2:0:1 and help to verify that the O $2p$ hole states originate in CuO_2 planes. While there are more O $2p$ hole states in 2:2:1:2, their distribution and character is similar for 2:2:0:1 and 2:2:0:1.

B. Off-normal results for $Bi_2Sr_2CaCu_2O_8$ (001) at 60 K

In Fig. 3 we present off-normal KRIPES spectra taken at $E_i=16$ eV (left) and $E_i=18$ eV (right) for $Bi_2Sr_2CaCu_2O_8$ (001). The PDC's were acquired at 60 K immediately after cleaving. The angle between the incident electrons and the surface normal is represented by θ, as labeled. The $\theta=0°$ spectrum is repeated as a dashed line overlaid on each off-normal curve. Since the samples have randomly oriented domains aligned along the c axis, the off-normal spectra do not follow any specific direction within the (001) plane of k space. Since $Bi_2Sr_2CaCu_2O_8$ is a two-dimensional compound, the initial state is determined by the momentum parallel to the surface. Therefore, the $\theta=0°$ spectrum represents Γ, and spectra taken at increased θ represent a fixed radial distance (but arbitrary azimuthal direction) in the (001) plane.

Figure 3 shows increased emission near E_F for the $\theta=10°$ spectra. If those spectra sampled the zone along [110] or [100], then the $\theta=10°$ spectra would represent either 0.25 Γ-X or 0.35 Γ-M. A KRIPES study by Claessen *et al.*[23] also observed increased emission near E_F at ~ 0.5 Γ-X. We suggest that this increased emission represents Cu $3d-$O $2p$ hybrids that are expected to cross E_F at ~ 0.3 Γ-X.[14-16] Indeed, angle-resolved photoemission results[24-26] show this band crossing at 0.33 Γ-X.

For $\theta>10°$, the increased emission spreads away from E_F and eventually occurs throughout the region between E_F and E_F+3 eV. This increased emission is larger for the $E_i=18$-eV spectra, suggesting that it can be associated with the O $2p$ hole character from the CuO_2 planes, even though some of the increase could be related to Bi-O hybrids. Spectra taken at $\theta=25°$ and 30° show this increased emission to broaden asymmetrically toward E_F, centered near $E_F+0.75$ eV. For $\theta=40°$, this structure is

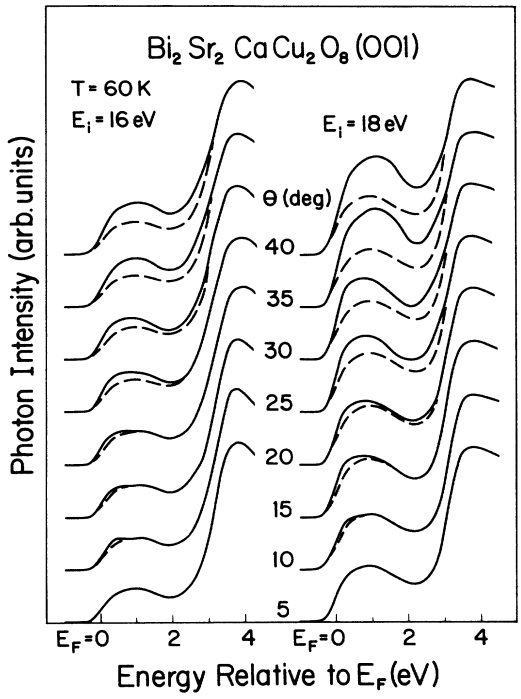

FIG. 3. Off-normal KRIPES spectra acquired at 60 K for $Bi_2Sr_2CaCu_2O_8$ 001). The $\theta=0°$ spectrum is shown dashed and is overlaid on each off-normal curve to emphasize changes. Increased O $2p$ hole emission is found for increased angle θ, consistent with $p_{x,y}$ character.

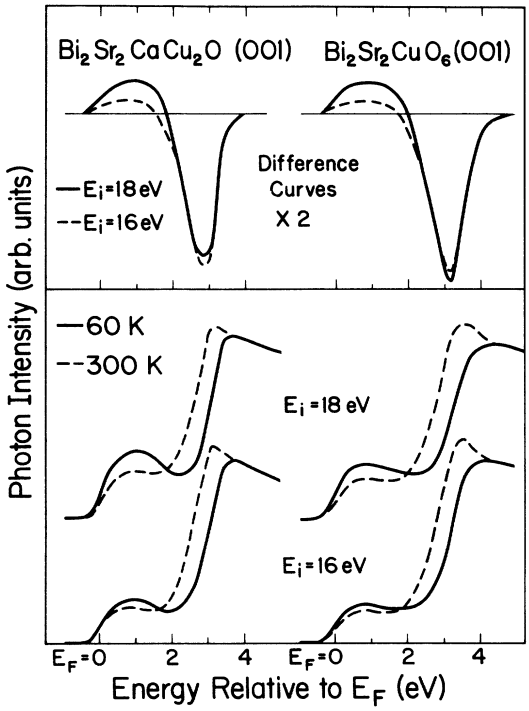

FIG. 4. Temperature-dependent empty-state emission for $Bi_2Sr_2CaCu_2O_8$ and $Bi_2Sr_2CuO_8$. As can be seen in the difference curves, cooling from 300 to 60 K gives rise to increased emission near 1 eV that we attribute to O states and decreased emission near 3.2 eV that we attribute to d states.

centered near $E_F=1$ eV. Band-structure calculations predict the Cu $3d-$O $2p$ antibonding hybrids to disperse across E_F along Γ-X and peak near E_F+2 eV at the X point.[14–16] If this study was conducted along Γ-X, then the $\theta=25°$ and 30° spectra would represent ~ 0.7 Γ-X and $\theta=40°$ spectra would represent the X point. We suggest that the observed behavior qualitatively parallels that predicted by theory, showing that the Cu $3d-$O $2p$ antibonding hybrids have dispersive character along the a-b plane. Similar conclusions for $Bi_2Sr_2CaCu_2O_8$ have been reached by Himpsel *et al.*[27] based on optical absorption studies.

C. IPES of $Bi_2Sr_2CaCu_2O_8$ and $Bi_2Sr_2CuO_6$ at 300 K

In Fig. 4, we show spectra that represent the temperature-dependent empty electronic states of 2:2:1:2 and 2:2:0:1. The bottom plane compares the 60-K (solid lines) and 300-K (dashed lines) IPES spectra taken at $E_i=16$ and 18 eV, normalized to emission at E_F+4 eV. These spectra were obtained from samples cleaved at the measurement temperature. Identical spectra were obtained from samples cleaved at 60 K and warmed to 300 K, or vice versa. Difference curves are shown in the top panel, with the solid and dashed lines representing the differences in the $E_i=16$- and 18-eV spectra, respectively. Unfortunately, the experimental resolution was ~ 0.4 eV and changes related to the development of the superconducting gap could not be detailed.[24]

Figure 4 reveals increased emission between E_F and E_F+2 eV and decreased emission between E_F+2 and 4 eV at 60 K for both 2:2:1:2 and 2:2:0:1. No temperature-dependent empty electronic state changes were observed above E_F+4 eV. Comparison of the $E_i=16$- and 18-eV spectra indicates that the increased emission centered at E_F+1 eV can be associated with O $2p$ character since the changes are larger for incident electron energies near the O $2s$ threshold energy. The absence of such a dependency on E_i suggests that structures centered at $E_F+2.8$ eV and $E_F+3.2$ eV for 2:2:1:2 and 2:2:0:1, respectively, are not related to changes in O $2p$-derived features.

The character of the room-temperature feature at $E_F+2.8$ eV in 2:2:1:2 has been the focus of recent speculation. IPES results by Drube *et al.*[28] showed its magnitude to be somewhat cleave dependent, and they attributed it to either localized Cu $3d$ levels or to Bi $6p$ levels. Pattnaik and Newns[29] discussed this feature within the framework of a heavy-fermion normal state and attributed it to a localized Cu $3d$ level. Since x-ray photoemission spectroscopy (XPS) studies of $Bi_2Sr_2CaCu_2O_8$ showed the Bi $4f$ emission to be temperature independent,[30] it is unlikely that it is due to empty Bi $6p$ levels. Hence, we associate the $E_F+2.8$-eV feature in 2:2:1:2 with localized Cu $3d$ levels, and attribute the feature at $E_F+3.2$ eV in 2:2:0:1 to the equivalent Cu $3d$ level.

These changes in the empty O and Cu features suggest enhanced charge transfer from the O to the Cu at low temperature, possibly because of a structural transition. Indeed, He *et al.*[10] have observed a discontinuity in the specific heat and changes in x-ray-diffraction images at 210 K for both 2:2:1:2 and 2:2:2:3 Bi-Sr-Ca-Cu-O. They

suggest that these anomalies are consistent with a phase transition occurring in either the CuO_2 or BiO planes. With this in mind, we associate with changes to a structural phase transition in the CuO_2 planes. Note that enhanced charge transfer was also observed for $YBa_2Cu_3O_{7-x}$ whereby increased O $2p$ hole and reduced Cu^{2+} satellite emission was found at 60 K.[4] He *et al.*[31] also observed ultrasound anomalies near 170 and 250 K for $YBa_2Cu_3O_{7-x}$.

IV. CONCLUSIONS

We have found decreased O $2p$ hole emission and increased Cu $3d$ emission for IPES spectra acquired at 300 K when compared to equivalent spectra acquired at 60 K. These changes can be explained by a phase transition occurring in the CuO_2 planes at 210 K. We also find that the empty electronic states for 2:2:1:2 are nearly identical to those for 2:2:0:1 at 60 K. In particular, resonant enhancement of the O $2p$ hole levels showed similar O $2p$ hole character even though $T_c \sim 85$ K for 2:2:1:2 and $T_c \sim 10$ K for 2:2:0:1. However, the O $2s$-$2p$ resonant enhancement was twice as large for 2:2:1:2 because of the extra CuO_2 plane in the 2:2:1:2 sublattice. Finally, off-normal KRIPES results for $Bi_2Sr_2CaCu_2O_8$ (001) at 60 K found O $2p$ holes with $p_{x,y}$ character.

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