

Relation of the incommensurate modulation structure and normal-state properties of the Ba-doped $\text{Bi}_2\text{Sr}_2\text{CuO}_{6+\delta}$ system

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(Received 31 August 1993; revised manuscript received 6 December 1993)

In this paper the variations of the normal-state characteristic associated with the metal-insulator transition for $\text{Bi}_2\text{Sr}_{2-x}\text{Ba}_x\text{CuO}_y$ were investigated by means of x-ray photoemission spectroscopy, Raman scattering, as well as the measurement of thermoelectric power. The experimental data reveal that in the Ba-doped $\text{Bi}_2\text{Sr}_2\text{CuO}_y$ system the Coulomb correlation of Cu 3d electrons tends to strengthen with the partial substitution of Ba for Sr. The metal-insulator transition in this system is very likely to originate from the change of Cu 3d electrons from partially delocalized to localized states. In addition, the experiment also shows that in the Ba-doped 2:2:0:1 phase the change in the incommensurate modulation structure results in intensifying the structural distortion of the 2:2:0:1 phase. The variation of the Cu 3d electrons with Ba substitution possibly comes from such a structural distortion. This suggests that for high- T_c cuprates the superconductivity is closely related to the crystal microstructure.

I. INTRODUCTION

It is commonly realized that the study of normal-state properties is necessary to clarify the mechanism for high- T_c superconductivity. In recent years, researches in this field have focused mainly on the first member $\text{Bi}_2\text{Sr}_2\text{CuO}_{6+\delta}$ (i.e., the Bi-2:2:0:1 phase) of the homogeneous series of layered superconductors $\text{Bi}_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_{2n+4+\delta}$ ($n = 1, 2, \text{ or } 3$) due to its lower T_c . However, the preparation of a 2:2:0:1 single-phase sample in the Bi-Sr-Cu-O system is difficult owing to the presence of other insulating phases, and most of the early studies in this material were on multiphase ceramics.¹⁻⁵ In substituted Bi-Sr-Cu-O systems, i.e., $\text{Bi}_{2+x}\text{Sr}_{2-x}\text{CuO}_y$,⁶ $\text{Bi}_2\text{Sr}_{2-x}\text{La}_x\text{CuO}_y$,⁷⁻⁹ and $\text{Bi}_{2-x}\text{Pb}_x\text{Sr}_2\text{CuO}_y$,^{10,11} the ideal 2:2:0:1 single phase can easily be acquired. However, the Bi-2:2:0:1 phase possesses a complicated incommensurate modulation structure, the supercell of which is monoclinic. Furthermore, the characteristics of this kind of monoclinic superstructural modulation are sensitive to the preparation conditions and chemical composition of the samples. So, for crystals grown under specific conditions, it is difficult to describe the real structural features by the average structure model proposed by Tarascon *et al.*¹² Evidently, the influence of the incommensurate superstructural modulation on the normal-state properties should be taken into account in investigation of the 2:2:0:1 phase.

As far as the incommensurate modulation structure of Bi cuprates is concerned, a common opinion of many authors is that it has no remarkable relation with the super-

conductivity of Bi-based compounds. Yet, Onoda and Sato¹³ and Gao *et al.*¹⁴ indicated that the incommensurate superstructural modulation was closely related to the oxygen configuration of the Cu atoms. For the Bi-2:2:0:1 and 2:2:1:2 phases, the deviation of the Cu-atom positions from the *a-c* plane caused by the modulation wave in the 2:2:0:1 phase is much larger than that in the 2:2:1:2 phase, which may be one of the origins of the rather low T_c value of the Bi-Sr-Cu-O compound. By this argument, it can be postulated that the variation of the modulation structure of the Bi-2:2:0:1 phase is certain to change the properties of the Cu-O bond, thus affecting its superconductivity and normal-state properties.

In our earlier work,¹⁵ it was found that the ideal 2:2:0:1 single phase could be easily obtained by the partial substitution of Ba for Sr in the $\text{Bi}_2\text{Sr}_{2-x}\text{Ba}_x\text{CuO}_y$ ($0.1 \leq x \leq 0.5$) system. But such a substitution changed the structure and properties of the Bi-2:2:0:1 phase. The microstructural analysis in Ref. 15 showed that the most striking feature of the structural distortion induced by the Ba substitution is that the wavelength of the Bi-concentrated band modulation decreased from $4.86b$ with $x = 0.1$ to $4.14b$ with $x = 0.5$. Measurement of the superconductivity of this Ba-doped system showed that the appropriate substitution of Ba for Sr ($x \leq 0.2$) could raise the T_c of the 2:2:0:1 phase, to 20 K for the sample with $x = 0.2$; however, with greater Ba substitution ($x > 0.2$) a metal-to-insulator transition occurred and the superconductivity of the 2:2:0:1 phase disappeared as x exceeded 0.3. In the discussion of Ref. 15, we directly ascribed the variation of the superconductivity of the 2:2:0:1 phase with Ba substitution to the change in the modulation

structure. In this paper, we present a detailed study with the emphasis on the varying behavior of the normal state of the Ba-doped 2:2:0:1 phase during the metal-to-insulator transition, aimed at clarifying the dependence of normal-state properties on the incommensurate modulation structure.

II. EXPERIMENTAL METHODS

The $\text{Bi}_2\text{Sr}_{2-x}\text{Ba}_x\text{CuO}_y$ ($0 \leq x \leq 0.5$) polycrystalline samples used in the present investigation were prepared by regular solid-state reaction. The detailed preparation conditions were described in Ref. 15, where the microstructural features and superconductivity of these Ba-doped 2:2:0:1 phases have already been reported.

The investigation of the normal-state properties of these samples was carried out by means of measurements of thermoelectric power (TEP), the photoelectron spectrum, and the Raman spectrum. The TEP was measured using a differential method. The x-ray and ultraviolet photoelectron spectroscopy (XPS and UPS) measurements were performed on a ESCALAB MKII surface-analysis system. The samples were scraped before being transferred into the analysis chamber in a vacuum of 1×10^{-9} mbar. Raman spectra were measured on a Spex-1403 Raman spectrophotometer using a Raman backscattering technique. The 5145-Å line from an argon-ion laser was used as an excitation light source. All measurements were made at room temperature.

III. EXPERIMENTAL RESULTS

A. Measurement of TEP

Figure 1 shows the TEP of the samples with $x = 0.1, 0.2,$ and 0.4 in the temperature range 80–280 K. From this figure, it can be seen that the $\text{Bi}_2\text{Sr}_{1.9}\text{Ba}_{0.1}\text{CuO}_y$ ($x = 0.1$) sample has negative values of TEP in the measured temperature range, and the TEP decreases linearly as the temperature increases. This suggests that the transport in the normal state of the sample with $x = 0.1$ is metallic. However, the TEP of the $\text{Bi}_2\text{Sr}_{1.8}\text{Ba}_{0.2}\text{CuO}_y$ ($x = 0.2$) sample is negative around room temperature, but becomes positive with $T < 210$ K. For the insulating

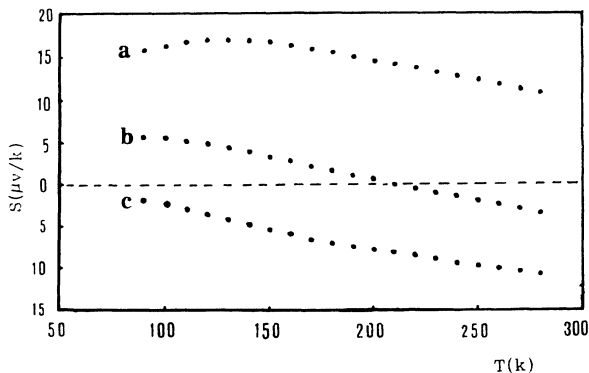


FIG. 1. Temperature dependence of the TEP for $\text{Bi}_2\text{Sr}_{2-x}\text{Ba}_x\text{CuO}_y$ (a, $x = 0.4$; b, $x = 0.2$; c, $x = 0.1$).

sample $\text{Bi}_2\text{Sr}_{1.6}\text{Ba}_{0.4}\text{CuO}_y$ ($x = 0.4$), the TEP is positive and large, and a broad peak at the temperature of 140 K can be observed. Such a dependence of the TEP on the Ba content in the Ba-doped 2:2:0:1 phase shows a great similarity with the varying behavior of the TEP observed in the $\text{Bi}_2\text{Sr}_2\text{Ca}_{1-x}\text{Y}_x\text{Cu}_2\text{O}_y$ system.^{16,17} For that system, Munakata and co-workers¹⁶ indicated that the TEP value was mainly controlled by the Cu valence. The decrease in the Cu oxidation level was certain to result in an increase in the TEP value, as well as a transition from negative to positive values. Consequently, based on the argument proposed by Munakata *et al.*, it can be speculated that the substitution of Ba^{2+} for Sr^{2+} in the 2:2:0:1 phase also reduces the Cu oxidation level, i.e., the Cu average valence decreases with increase of Ba content. This postulation is confirmed by the following XPS analysis of the Ba-doped system.

B. Electronic structure of the $\text{Bi}_2\text{Sr}_{2-x}\text{Ba}_x\text{CuO}_y$ system

1. Analysis of XPS spectra

In Fig. 2(a) we show the O 1s XPS spectra of the samples with $x = 0, 0.2, 0.5$. The signal from a contaminant around 531 eV is very weak for all three samples, which means that the surfaces of these samples are all clean. Comparing the O 1s XPS spectra of the three samples, we note that the O 1s binding energy exhibits an obvious change with the partial substitution of Ba for Sr. For the sample with $x = 0$, the O 1s binding energy is about 528.1 eV, while for the samples with $x = 0.2, 0.5$, the O 1s binding energy is increased to 529.1 eV, i.e., the substitution of Ba for Sr gives rise to a 1-eV shift in the O 1s energy level. Again, when the three O 1s spectra are decomposed, four components are obtained for the sample with $x = 0$ and five components for the samples with $x = 0.2, 0.5$. These components are denoted by 1, 2, 3, 4, and 5 in Fig. 2(a). Meyer and Weaver¹⁸ and Parmigiani *et al.*¹⁹ suggested that these different components of the O 1s peaks originate from the O 1s emission of the different

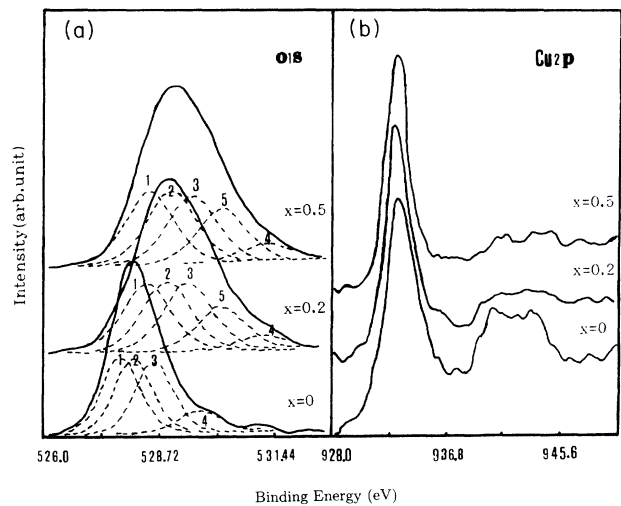


FIG. 2. (a) O 1s XPS spectra of $\text{Bi}_2\text{Sr}_{2-x}\text{Ba}_x\text{CuO}_y$ ($x = 0, 0.2, 0.5$); (b) Cu $2p_{3/2}$ XPS spectra of $\text{Bi}_2\text{Sr}_{2-x}\text{Ba}_x\text{CuO}_y$ ($x = 0, 0.2, 0.5$).

atomic layers. According to their work, the following assignments can be made. First, the lowest-energy component (peak 1) is probably associated with oxygen atoms of the Cu-O planes. Peak 2 arises from oxygen atoms bound to strontium atoms and oxygen atoms bound to Bi atoms in off-plane sites. The third component (peak 3) is likely to come from oxygen atoms in the BiO planes. Weaver *et al.*, based on their previous work,²⁰ suggested that the small, highest-binding-energy component (peak 4) arose due to the two different ground states of Cu. However, in Ref. 18 Meyer and Weaver assigned this component to a plasmon energy-loss feature, proposed by Fujimori *et al.*²¹ Obviously the second explanation is reasonable. Such a plasmon satellite has now been identified for all of the superconducting oxides. As for the component peak 5 in the samples with $x = 0.2, 0.5$, we think that it is likely to be associated with the oxygen atoms bound to the substitutional Ba^{2+} ions. All these experimental data reveal that Ba substitution for Sr in the Bi-2:2:0:1 phase changes the arrangement of the energy levels of oxygen atoms located at the different atomic layers.

Figure 2(b) shows the Cu $2p_{3/2}$ XPS spectra of the samples with $x = 0, 0.2$, and 0.5 . First, for the undoped sample ($x = 0$) there is a main peak at about 933 eV and a satellite peak around 942 eV. This type of Cu $2p_{3/2}$ XPS spectrum is completely similar to that of other series of cuprates, such as $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$, $\text{La}_{2-x}\text{Sr}_x\text{CuO}_{4-\delta}$, $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$, and CuO .²²⁻²⁴ Based on the knowledge of XPS in these cuprates, we assign the main peak to the final state of Cu $2p^5 3d^{10}L$ where L represents a hole in the ligand oxygen, while the satellite peak indicates the presence of the Cu $2p^5 3d^9$ final state. The ground states corresponding to the two different final states are Cu $3d^{10}L$ and Cu $3d^9$, respectively, and are mixed in the proportion of 1:1 due to their closer energy.²⁵ Second, for the case of $x > 0$, a striking feature arising in the Ba-doped samples, and becoming more pronounced with the increase of Ba content can be seen: though no detectable difference is observed for the position of the main and satellite peaks in the Ba-doped samples, the ratio of the intensities of the satellite and main peaks (I_{SP}/I_{MP}) decreases markedly with increase of Ba content. This suggests that the partial substitution of Ba for Sr in the 2:2:0:1 phase reduces the ratio of the ground state Cu $3d^9$. That is to say, the average valence of Cu exhibits a tendency to decrease with an increase of Ba content. This point agrees well with the result deduced from the measurements of TEP.

2. Analysis of UPS valence-band spectra

Figure 3(a) shows the UPS valence-band (VB) spectra of the samples with $x = 0, 0.2, 0.5$. In order to compare the features of the VB spectra of the three samples at about the Fermi level, the VB region with lower binding energy is magnified and shown in Fig. 3(b). From Fig. 3(b), it can clearly be seen that the line shape of the VB spectra of the three samples is very similar in the region of lower binding energy (0–3.3 eV). They all exhibit no clear Fermi level (FL), and the density of states at the FL is much lower. Nevertheless, the VB spectrum at higher

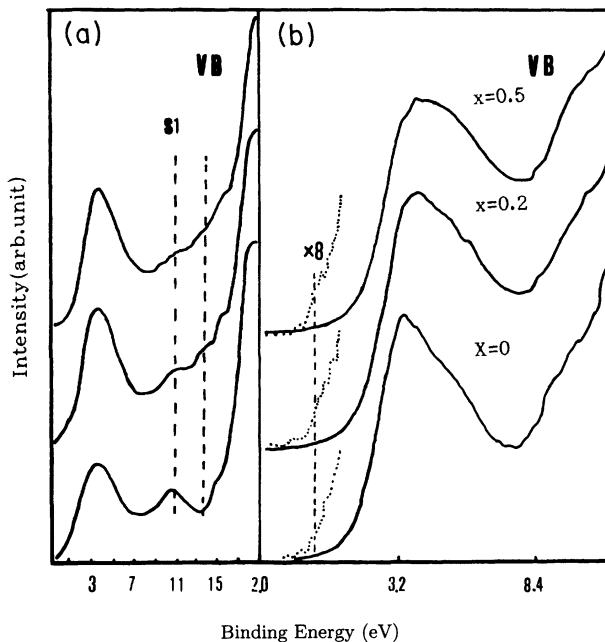


FIG. 3. UPS valence-band spectra of $\text{Bi}_2\text{Sr}_{2-x}\text{Ba}_x\text{CuO}_y$ ($x = 0, 0.2, 0.5$).

energy (3.1–12 eV) shown in Fig. 3(a) displays an obvious change with Ba substitution. The most apparent feature is that the relative intensity of the typical peak S1 at about 10.7 eV decreases with increase of Ba content. But the emission at the higher-binding-energy side of the peak S1 increases correspondingly. Reference 26 indicated that the main component of the VB region was contributed by the Cu-O hybridization state. The VB region with lower binding energy (0–3.3 eV) is of O $2p$ character, while the VB region with higher binding energy (3.3–12 eV) is of Cu $3d$ character, and it reflects strong correlation of the Cu $3d$ electrons. Hence, from this argument, it can be believed that the variation of the VB spectrum at higher binding energy observed in the Ba-doped system essentially reveals the nature of the change in the Coulomb correlation of Cu $3d$ electrons induced by Ba-substitution. With increase of Ba content, the Coulomb correlation is enhanced.

C. Raman-scattering analysis of the $\text{Bi}_2\text{Sr}_{2-x}\text{Ba}_x\text{CuO}_y$ ($0 \leq x \leq 0.5$) system

Figure 4 shows the Raman spectra of the samples with $x = 0, 0.2, 0.3, 0.4$, and 0.5 . For the samples with $x = 0, 0.2, 0.3$, two stronger Raman modes at 451 and 626 cm^{-1} can clearly be observed, and the 626- cm^{-1} mode broadens slightly with increase of Ba content. However, for the samples with $x = 0.4, 0.5$, the higher-frequency mode shifts to lower frequency and simultaneously broadens significantly, such that it overlaps with the lower-frequency mode at 451 cm^{-1} . In the light of the assignment of the phonon Raman modes of the Bi-2:2:0:1 phase in Refs. 27 and 28, the phonon modes observed in

the Raman spectra of $\text{Bi}_2\text{Sr}_{2-x}\text{Ba}_x\text{CuO}_y$ can be assigned. That is, the ~ 626 - and 451-cm^{-1} lines correspond, respectively, to the A_g mode of vibration of O_{Bi} atoms along the a axis and to the A_g mode of vibration of O_{Sr} atoms along the c axis (O_{Bi} and O_{Sr} refer to the oxygen atoms in the Bi_2O_2 and SrO layers, respectively).

From the above analysis of the O 1s XPS spectra, it can be postulated that the vibrational frequency of oxygen atoms bound to the substitutional Ba ions (O_{Ba}) should be larger than that of the O_{Bi} atoms. Hence, broadening in the Raman mode in the frequency range $450\text{--}620\text{ cm}^{-1}$ cannot be considered to result from the vibration of O_{Ba} atoms but comes from the structural distortion caused by Ba substitution. There is no doubt that in the Ba-doped system the structural distortion is related to the change in the incommensurate superstructural modulation caused by the Ba substitution. In accordance with the argument of Onoda and Sato,¹³ the modulation wave in the Bi-2:2:0:1 phase causes expansion of each layer, i.e., displacements of Bi, Sr, and Cu in the chains which run along the c axis, it can be thought that for the Ba-doped 2:2:0:1 phase, a decrease in the modulation wavelength is certain to enhance such a relative displacement. Therefore, we can say that the structural distortion of the Ba-doped 2:2:0:1 phase intensifies with decrease in the modulation wavelength. In this case, the features of the Cu-O bond should change accordingly. In Ref. 15 we reported that for the $\text{Bi}_2\text{Sr}_{2-x}\text{Ba}_x\text{CuO}_y$ system the lengths of the a and b axes increased with increase of the Ba content. Usually, the lengths of the a and b axes are considered to be controlled by the in-plane

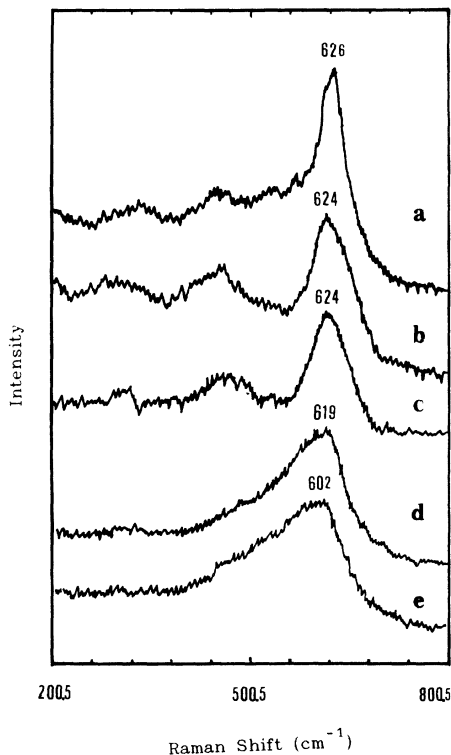


FIG. 4. Raman spectra of $\text{Bi}_2\text{Sr}_{2-x}\text{Ba}_x\text{CuO}_y$ (*a*, $x=0$; *b*, $x=0.2$; *c*, $x=0.3$; *d*, $x=0.4$; *e*, $x=0.5$).

Cu-O bond distance.²⁹ Thus the increase in the lengths of the a and b axes caused by Ba substitution reflects the distortion of the Cu-O bond. But, unfortunately, such a change of the Cu-O bond with the modulation structure cannot be detected directly by Raman scattering, due to the fact that the copper and oxygen atoms in the CuO_2 layers are not Raman active in the 2:2:0:1 phase.

IV. DISCUSSION

A. Transition of Cu 3d electrons from a partially delocalized to a localized state

In the light of the analyses described above of the transport properties and electronic structure of the $\text{Bi}_2\text{Sr}_{2-x}\text{Ba}_x\text{CuO}_y$ system, let us examine the varying behavior of Cu 3d electrons in the metal-insulator transition. Both the measurements of TEP and the XPS analysis reveal that the Cu average valence decreases with Ba substitution for Sr. This means that Ba substitution reduces the Cu oxidation level. Then the analysis of the UPS VB spectra seems to show that such a decrease in the Cu oxidation level results from enhancement of the degree of localization of the Cu 3d electrons. Therefore, it can be argued that in the $\text{Bi}_2\text{Sr}_{2-x}\text{Ba}_x\text{CuO}_y$ system the Cu 3d electrons undergo a transition from partially delocalized to localized, as the 2:2:0:1 phase changes from a metal to an insulator. This result supplies powerful evidence for the two-carrier model of O 2p holes and Cu $3d_{x^2-y^2}$ electrons in the CuO_2 layers, which was proposed in Ref. 30. In terms of the two-carrier model, the change in the sign of the TEP observed in the $\text{Bi}_2\text{Sr}_{2-x}\text{Ba}_x\text{CuO}_y$ system can be understood.

B. Origin of the change in the Cu 3d electrons and the related changes in superconducting properties caused by Ba substitution

It is obvious that in the $\text{Bi}_2\text{Sr}_{2-x}\text{Ba}_x\text{CuO}_y$ system the change in the Cu 3d electrons and the related changes in superconducting properties can only be attributed to the microstructural distortion induced by Ba substitution. On the basis of the Raman-spectrum analyses of Fig. 4, we point out that the crystal microstructural distortion in the Ba-doped 2:2:0:1 phase increases with decrease in the modulation wavelength. Then the distortion in the Cu-O bond undoubtedly influences the Cu 3d electronic state and the transport properties of the CuO_2 layer. According to the argument of Mandal, Poddar, and Ghosh that the in-plane Cu-O bond distance is closely related to the carrier concentration of the CuO_2 layers,²⁹ the dependence of the superconductivity of the Ba-doped 2:2:0:1 phase on the Ba content can be understood. For the sample with $x=0.2$, the distortion in the Cu-O bond makes the carrier concentration of the CuO_2 layers decrease slightly and reach an optimum value where T_c reaches a maximum. However, for the samples with $x > 0.2$, with further intensification in the Cu-O bond distortion, the significant decrease in the carrier concentration results in degrading the superconductivity and the occurrence of the metal-insulator transition. Therefore, it can be ar-

gued that the distortion of the Cu-O bond caused by the change in modulation structure is responsible for the transition of the Cu 3d electrons from a partially localized to a delocalized state.

In addition, many authors have suggested that charge transfer occurs between CuO₂ layers and other normal atomic layers in high- T_c cuprates and plays an important role in the occurrence of high- T_c superconductivity. In the Ba-doped 2:2:0:1 phase, the increase in structural distortion perhaps breaks down such a charge transfer, thus resulting in the metal-insulator transition, as well as the corresponding change in the Cu 3d electrons. Hence the charge-transfer model may also be a possible mechanism for high- T_c superconductivity.

V. CONCLUSIONS

We have studied the variation of normal-state transport properties and electronic structure of the Bi₂Sr_{2-x}Ba_xCuO_y system with Ba substitution by means of measurement of TEP and analyses of XPS and UPS. Furthermore, the relationship between the incommensurate modulation structure and normal-state properties

was discussed. From all these experimental investigations and analyses, the following conclusions can be drawn:

(1) The Cu 3d electrons undergo a transition from a partially delocalized state to a localized state with the occurrence of the metal-insulator transition in the Ba-doped 2:2:0:1 phase.

(2) The changes in the Cu 3d electrons and the related changes in the superconducting properties originate from the crystal microstructural distortion caused by Ba substitution. The presence of the structural distortion is related to the incommensurate modulation structure. Decrease in the modulation wavelength causes intensification of the displacements of the atoms as well as distortion of the Cu-O bond, which makes the degree of localization of the Cu 3d electrons increase. As a result, we strongly suggest that the crystal microstructure has a direct relation with high- T_c superconductivity.

ACKNOWLEDGMENTS

This research was supported by the National Center for Research and Development on Superconductivity and the Doctoral Fund of National Education Committee of China.

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