Tunneling spectroscopy of superconducting $Nd_{1.85}Ce_{0.15}CuO_{4-\delta}$

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(Received 5 September 1997)

The superconducting electronic states of $Nd_{1.85}Ce_{0.15}CuO_{4-\delta}$ (NCCO) are studied by applying the phase-sensitive capability of tunneling spectroscopy. Unlike the $YBa_2Cu_3O_{7-\delta}$ cases, the conductance spectra measured on (100)-, (110)- and (001)-oriented surfaces showed V-shaped gap structures, and no zero-bias conductance peaks were observed even around steps on the surfaces. The present results reject the possibility of $d_{x^2-y^2}$-wave and d_{xy}-wave symmetries for the pair potential in NCCO. The experimental spectra are well fitted to theoretical curves of anisotropic s-wave symmetry with a large smearing factor. [S0163-1829(98)05513-1]

I. INTRODUCTION

Tunneling spectroscopy has long been accepted as a phase-insensitive probe of the superconducting electronic states. The basic concept of this method originated from the equivalency of tunneling conductance spectra with density of states (DOS) of samples at the low-temperature limit.[1] In the case of normal-insulator-superconductor (N-I-S) junctions with conventional BCS superconductors, the conductance spectra correspond to the BCS density of states. In fact, many experimental results of tunneling spectroscopy have been analyzed in terms of this standpoint, and various aspects of the electronic states have been revealed by tunneling spectroscopy. However, most of the trials to apply this concept for the high-T_c superconductors have failed except for c-axis surface measurements of $Bi_2Sr_2CaCu_2O_{7-\delta}$ (BSCCO).[2] Otherwise, i.e., when the tunneling direction is in the ab plane, not only gap structures but also zero-bias conductance peaks (ZBCP's) are observed in large area on $YBa_2Cu_3O_{7-\delta}$ (YBCO) surfaces.[3] These results cannot be understood in terms of the classical tunneling theory.

A hint to reveal the origin of the ZBCP is presented by theoretical aspect. Based on an analysis of the boundary condition on d_{xy}-wave superconductor, the formation of zero-energy states (ZES) on the surface is predicted.[4] By developing this concept, a theory for tunneling spectroscopy is constructed which correctly takes into account of the anisotropy of the pair potential.[5–8] These theories claims the phase-sensitive capability of tunneling spectroscopy.[7] The fundamental concept is that the DOS at the surface is not always equivalent to that in the bulk. In the case of d-wave superconductors, pair potentials with different the signs and the amplitudes are woven in k space. This means that the effective pair potential for the traveling quasiparticle strongly depends on its wave vector $\mathbf{k}$. As a result, the sudden change of the effective pair potential occurs through the reflection at the surface depending on the surface orientations. This effect leads to the formation of the orientational-dependent surface states.[9] In the case of $d_{x^2-y^2}$-wave superconductors, the formation of ZES are expected,[10] and they are observed as ZBCP's in tunneling spectroscopy. Actually, various types of experimental spectra on YBCO surfaces are well analyzed along this concept, which suggests the validity of the novel theory and the $d_{x^2-y^2}$-wave nature of the pair potential in YBCO.[6,11,12]

For most of the high-T_c superconductors, their carriers are believed to be holes, which indicates that they are hole-doped superconductors. On the other hand, NCCO belongs to an exception. It has negative Hall coefficients, which implies that the carriers are electrons.[13] The crystal structure is simple, i.e., a single Cu-O plane, lacking the Cu-O chain and the apex-oxygen site. This simplicity allows us to study the intrinsic electronic properties of the Cu-O plane. For NCCO, unlike the hole-doped high-T_c superconductors, strong trends for conventional behaviors have been presented based on various experimental methods. For example, the resistivity has a T^2 temperature dependence,[14] and the penetration depth measurements show good fits for a simple s-wave BCS-type function.[15–17] In addition, the important role by the phonon for the superconductivity is suggested through

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$\alpha^2F(\omega)$ information based on tunneling measurements.[18] The Fermi-surface structures observed by angle-resolved photoemission spectroscopy (ARPES) show consistent results with the calculation based on the band picture.[19] However, as far as we know, no phase-sensitive measurements, which present most convincing evidence for the determination of the pairing symmetry,[20] have been reported.

At this stage, the measurement of orientational-dependent tunneling spectroscopy of NCCO is meaningful in two different aspects; one is to make secure the validity of the tunneling formula for anisotropic superconductors, and the other is to determine the pairing symmetry in NCCO by applying the phase-sensitive capability of tunneling spectroscopy. In this paper, we present the scanning tunneling spectroscopy (STS) measurements of NCCO single crystals for (100)-, (110)-, and (001)-oriented surfaces. By comparing these results with those for YBCO, clear differences in superconducting states between electron-doped and hole-doped cuprates are demonstrated. The results are well analyzed by assuming extended s-wave symmetry of the pair potential. The electronic states of NCCO are discussed based on the analysis of the gap structures in the tunneling spectra.

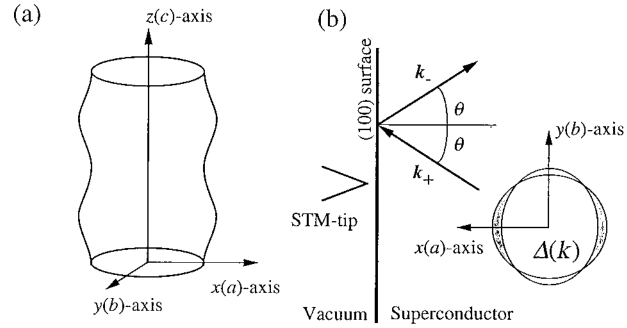

FIG. 1. (a) Schematic illustration of the Fermi surface in high-T_c superconductors. A cylindrical form with slight modulation for the z direction is assumed. (b) Schematic illustration of the elastic reflection at the surface. In the figure, the anisotropic pair potential of $d_{x^2-y^2}$-wave symmetry is shown. The surface state is formed because the effective pair potential for the quasiparticle changes through the reflection process.

II. TUNNELING SPECTROSCOPY FORMULA

In this section, the theory of tunneling spectroscopy for anisotropic superconductors in N-I-S structures and experimental results of YBCO are simply reviewed.[5–7] We assume that the pair potential in superconductor $\Delta(\mathbf{k})$ has $\mathbf{k}$ dependence. A spin-singlet superconductor with a cylindrical Fermi surface as shown in Fig. 1(a) is assumed in the following discussion. For the z direction, a slight modulation of the Fermi surface exists which allows the electron conduction for this direction. The electron transfer from the normal metal to the superconductor is so small as to be able to neglect higher-order tunneling terms. This assumption is valid for most of the experimental situations of tunneling spectroscopy. In this situation, the conductance spectrum corresponds to the surface DOS of the isolated superconductor.[7]

The concept and physical origin of the surface states in anisotropic superconductors are already described in Ref. 9. The most simple explanation is as follows. The conducting electrons in superconductor are specularly reflected at the surfaces. The wave vectors of the electrons change from $\mathbf{k}_+$ to $\mathbf{k}_-$ through the reflections as shown in Fig. 1(b). Accordingly the effective pair potentials for the electrons also change from $\Delta_+\equiv\Delta(\mathbf{k}_+)$ to $\Delta_-\equiv\Delta(\mathbf{k}_-)$. This sudden change of the pair potential results in the formation of bound states just at the surface whose energy levels corresponds to the bound states formed between two pair potentials Δ_+ and Δ_-. Angle-resolved surface DOS $\rho_\kappa(E,\theta)$ is given by

$$\rho_\kappa(E,\theta)=\frac{|\Delta_+\Delta_-|+(E-\Omega_+)(E-\Omega_-)\exp(i\varphi_- - i\varphi_+)}{|\Delta_+\Delta_-|-(E-\Omega_+)(E-\Omega_-)\exp(i\varphi_- - i\varphi_+)}, \tag{2.1}$$

where

$$\Omega_\pm\equiv\sqrt{E^2-|\Delta_\pm|^2},$$

$$\exp(i\varphi_\pm)\equiv\frac{\Delta_\pm}{|\Delta_\pm|}.$$

Note that Eq. (2.1) explicitly includes the phases of the effective pair potentials $\varphi_\pm$, which indicates the phase sensitivity of the surface DOS. The normalized conductance spectrum at temperature T is given by

$$\sigma_S(eV,\Gamma,T)=\frac{\int d\theta\int_{-\infty}^{\infty}dE\,\sigma_N(\theta)\mathrm{Re}[\rho_\kappa(E-i\Gamma,\theta)]\{-\partial f(T,E+eV)/\partial(eV)\}}{\int d\theta\,\sigma_N(\theta)}. \tag{2.2}$$

Here, V is a bias voltage, θ is the angle of quasiparticle trajectory to the surface normal, $\sigma_N(\theta)$ represents the angle-dependent tunneling distribution of emitted electrons from the normal metal to the superconductor, Γ corresponds to a smearing factor due to the lifetime broadening effect,[21] and $f(E)$ is the Fermi-distribution function. In the case of planer junctions, the Gaussian dependence of θ are used for $\sigma_N(\theta)$. For the tunneling junction formed between the normal metal and high-T_c superconductors, due to the large discrepancy in the Fermi wavelengths between the two conductors, $\sigma_N(\theta)$ is regarded as a constant value (independent of θ) for simplicity. To get a theoretical fitting to the experimental spectra, once $\sigma_N(\theta)$ is determined from the analysis of the junction configuration, the unknown parameters are $\Delta(\mathbf{k})$ and Γ.

Assume a situation where the tunneling direction is in the ab plane. In the case of $d_{x^2-y^2}$-wave superconductors with (100)-oriented surface, since $\Delta_+=\Delta_-$ applies for every direction, the calculated spectrum shows gap structure independent of θ. However, in $d_{x^2-y^2}$-wave superconductors with (110)-oriented surface, the calculated spectrum shows

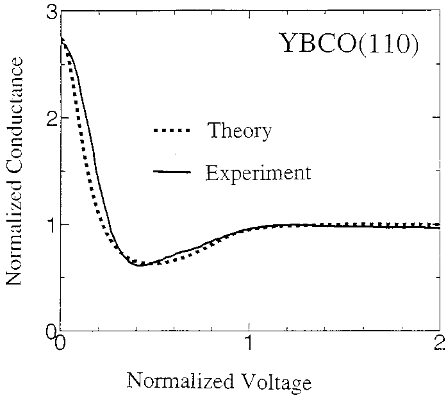

FIG. 2. Tunneling conductance spectrum of a YBCO(110) film at 4.2 K. Similar ZBCP's have been observed spatially continuously in large area of YBCO(110) surfaces. The dotted line represents a theoretical fitting by assuming $d_{x^2-y^2}$-wave symmetry, $\Delta_d=16.5$ meV, and $\Gamma=0.15\Delta_d$ (see Ref. 7 for details.)

ZBCP's reflecting the sign change of the two effective pair potential ($\Delta_+=-\Delta_-$). Since the sign change does not occur for s-wave symmetry, ZBCP's are not expected for metal superconductors. Figure 2 shows an experimental conductance spectrum obtained on a YBCO(110) surface and a theoretical fitting curve. Similar spectra have been observed at most of the area on YBCO(110) spatially continuously.[11,12] While on YBCO(100) surfaces, ZBCP's have been observed at a relatively small area compared to the cases of the YBCO(110) surfaces.[12] This result is consistent with the present theory by assuming a $d_{x^2-y^2}$-wave nature of the pair potential. Similar ZBCP's are expected to be observed on the surfaces of superconductors whose pair potentials have negative regions in **k** space. Next, we assume the tunneling spectroscopy for (001) orientation. In this case, $\Delta_+=\Delta_-$ applies for all directions. For a fixed θ, the conductance spectrum corresponds to the conventional BCS DOS. The conductance spectrum of the N-I-S junction is obtained by the integration of all θ, and the gap structure reflects the gap distribution in **k** space. Due to the rotational symmetry around the tunneling direction, the tunneling electron has equal weight distribution in all direction on the Fermi surface. Thus the conductance spectrum for the (001) direction corresponds to the bulk DOS. This analysis is consistent with the good agreement of theory with experiments on (001)-oriented BSCCO (Ref. 2) and YBCO.[22]

Measurements of the spatially resolved conductance spectra on rough surfaces are also good stages to detect the anisotropic surface bound states. Mainly two effects are expected on these surfaces. One is the effect of the misorientation of the crystal axis due to the surface corrugation,[7] and the other is the breakdown of the specularity through the reflection process at the surfaces.[10,23] Since the coherence lengths of high-T_c superconductors are short compared to those of metal superconductors, the spatial distribution of the conductance spectra is expected to reflect the atomic-scale configuration at the surface.[24] Calculated results based on the extended Hubbard models show that the conductance spectra are sensitive to the roughness when the pair potential has d-wave symmetry, while they are insensitive to the roughness when the pair potential has s-wave symmetry. Using the sensitivity of the tunneling spectrum to the surface roughness, we can check whether the pair potential has s-wave symmetry or d-wave symmetry. Actually, calculated spectra on the (100)-oriented surfaces of a $d_{x^2-y^2}$-wave superconductor with roughness show complex gap structures including ZBCP's.[24] This result explains the origin of the experimental observations of a wide variety of conductance spectra on YBCO(100) surfaces.[3,6]

III. RESULTS AND DISCUSSIONS

The NCCO single crystals used in this study were fabricated by traveling-solvent floating-zone method. After the growth, the samples were annealed in an oxygen with a pressure of 10^{-4} atmosphere at 1000 °C for four days. We used two crystals with T_c's of 17.5 K (referred to as sample A) and 17.2 K (sample B) evaluated by magnetization measurements. The doping rate of the sample B was set to a relatively underdoped region compared to that of the sample A (optimum dope). Details about the crystal growth conditions are described in Ref. 25. The orientations of the crystals were identified by the Laue diffraction patterns, and the surfaces with specified orientations were prepared by the mechanical cleavages in the air. All the samples were mounted on the scanning tunneling microscopy (STM) unit soon after the cleavage, however, the surfaces were exposed to the air about 1 h before cooling down to low-temperature (LT). Mechanically sharpened Pt and Pt-Ir wires were used for STM tips. High-energy resolution of our LT-STM system has demonstrated by the observation of superconducting gap in NbN films. Details about our LT-STM system are described in Ref. 26. The main advantages of the spectroscopy by STM (STS) compared to that by thin-film junctions are; (i) the spatially resolved capability, (ii) the potential for the adjustable measurements by changing the bias condition. The latter is especially important because it helps us to avoid the influences of the nonequilibrium effect and the higher-order tunneling terms. The conductance spectrum data shown below were obtained under tip-biased condition with the typical resistance of 1–0.1 GΩ at 4.2 K. We usually check that the spectra are insensitive to the variation of the tip-sample distance.

The observations of clear STM topographic images of the surfaces are one of the important criterions to check the measurement condition. In our cases, although atomic images could not be obtained, images of atomic terraces and steps were measured. Figure 3 shows an STM topographic image observed on (110)-oriented surface of sample B with the bias voltage of 2 V and the current of 100 pA. Each bump corresponds to the monolayer step whose height corresponds to the half of the lattice constant for (110) orientation.

Figures 4 and 5 show typical conductance spectra obtained on samples A and B for various surface orientations. It is clear that all spectra have V-shaped gap structures. Less dependence of the gap structure on tunneling direction seems to reflect rather isotropic electronic structure in NCCO. The amplitude of the gap seems somewhat smaller for (001)-oriented surface compared to other orientations. However, since the observed gap amplitudes depend on the position even on the same surface, we cannot conclude that the gap amplitude has orientational dependence. The amplitudes Δ_{pp}(4.2 K) measured by peak-to-peak value in the spectra

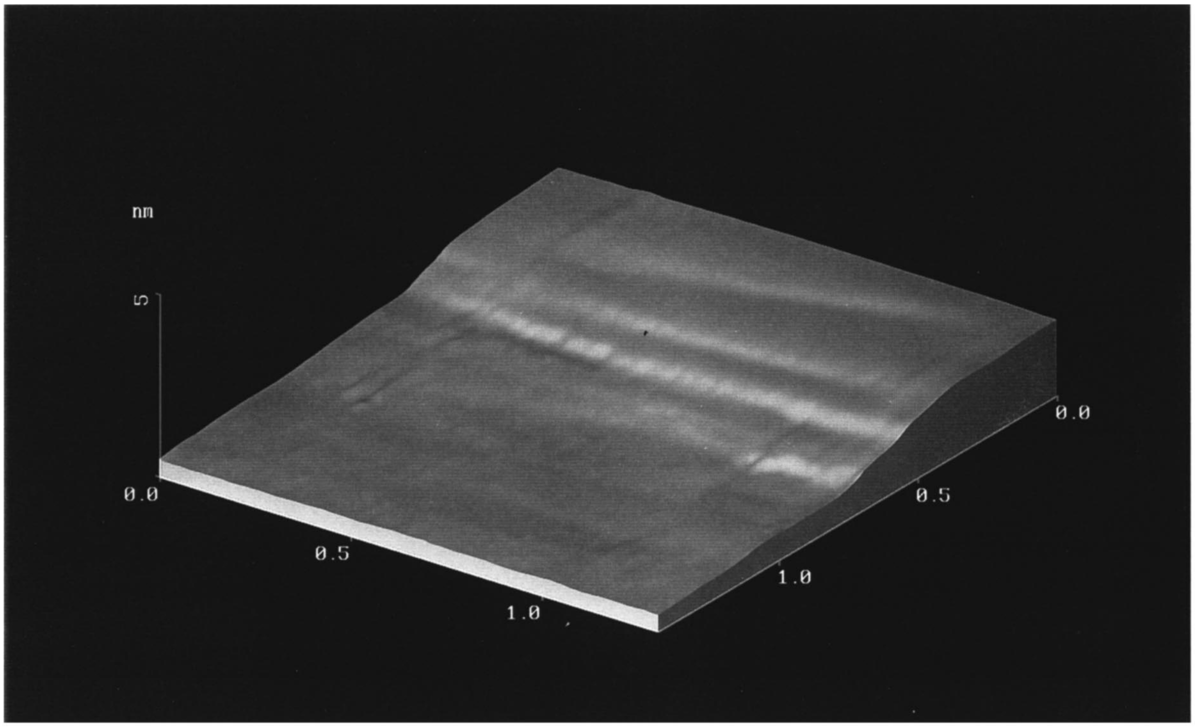

FIG. 3. STM topographic image of NCCO (110)-oriented surface. The bias voltage was 2 V with the current of 100 pA.

are in the range from 3.5 to 5 meV. If we assume BCS-like temperature dependence of the gap amplitude, measured $2\Delta(0)/k_BT_c$ values are in the range from 4 to 6, which is almost consistent with the results in other reports.[18,27,28] The most important feature to note is the lack of ZBCP's for (110) orientation. Along the theory in the previous section, the lack of ZBCP's for (110) orientation rejects the possibility of $d_{x^2-y^2}$-wave symmetry of the pair potential in NCCO. The possibility of d_{xy}-wave symmetry is also rejected based on the lack of ZBCP's for (100)-oriented surface. In fact, we have measured more than one thousand tunneling spectra on various NCCO surfaces. All of these spectra showed either the gap structure (more than 95%) or semiconducting properties (see below), and none of the spectra showed the ZBCP's.

The doping rate of sample B is placed close to the antiferromagnetic (AF) region in the phase diagram. Due to the small inhomogeneity of the doping rate, small regions on the surface of sample B showed semiconducting properties. At these regions, the servomotion of STM was stable around 1 V bias, however we could not attain the servomotion with the bias voltage below 0.1 V. Figure 6 shows the typical conductance spectrum obtained at these regions. The spectrum has V-shaped structure with its value of almost zero around the zero-bias level. There is a broad peak at -30 mV. Although the origin of the peak is not clear, we speculate that it reflects the energy level of spin excitations in the AF phase.

The possibility of d-wave symmetry is also rejected from the STS measurements in the ab-plane tunneling configuration. Figures 7 and 8 show the results of STS on the surfaces of (110)- and (100)-orientations, respectively. These figures show successive 50 points spectroscopy data obtained along lines of 35 nm. In the lower parts of these figures, the tip traces before and after the spectroscopy measurements are also shown. By comparing the tip trace with the spectrum forms, we can check the correlation between the surface topography and the spectrum forms. The shift of the tip traces indicates the existence of a drift of the tip position. It is apparent that the gap structures are insensitive to the surface

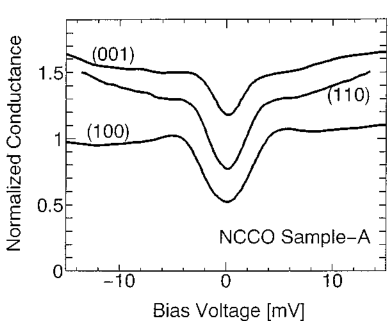

FIG. 4. Typical conductance spectra obtained on the surfaces of NCCO (sample A) for (100), (110), and (001) orientations.

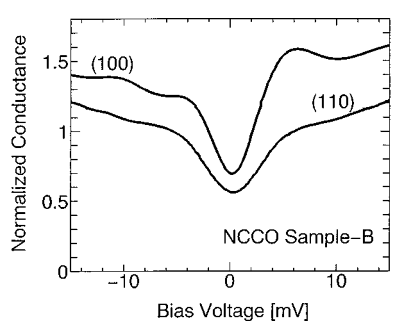

FIG. 5. Typical conductance spectra obtained on the surfaces of NCCO (sample B) for (100) and (110) orientations.

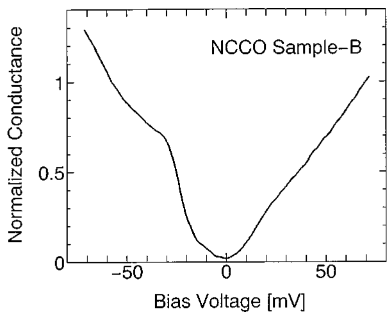

FIG. 6. Typical conductance spectra obtained on the nonmetallic surfaces of NCCO (sample B) for the (100) orientation. There is a broad peak at -30 mV.

morphology. This feature is completely different from that observed on YBCO surfaces, i.e., the spectra have strong spatial dependence and vary between the gap structure and the ZBCP.[6] As described in the previous section, the robustness of the gap structure on the surface roughness in NCCO suggests that the pair potential in NCCO has no sign change at everywhere in k space, and clearly reject the possibilities

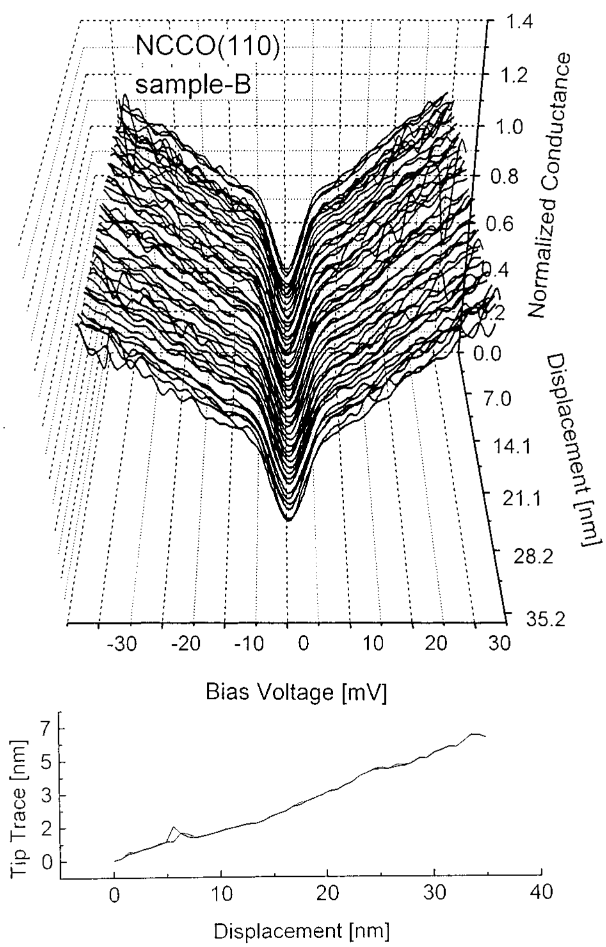

FIG. 7. A experimental result of STS data on the (110)-oriented surface of sample B. The figure shows successive 50 points spectroscopy measurement obtained along a line of 35 nm. Almost spatially uniform gap structures are observed. In the lower part of the figure, the tip traces before and after the spectroscopy measurement are also shown.

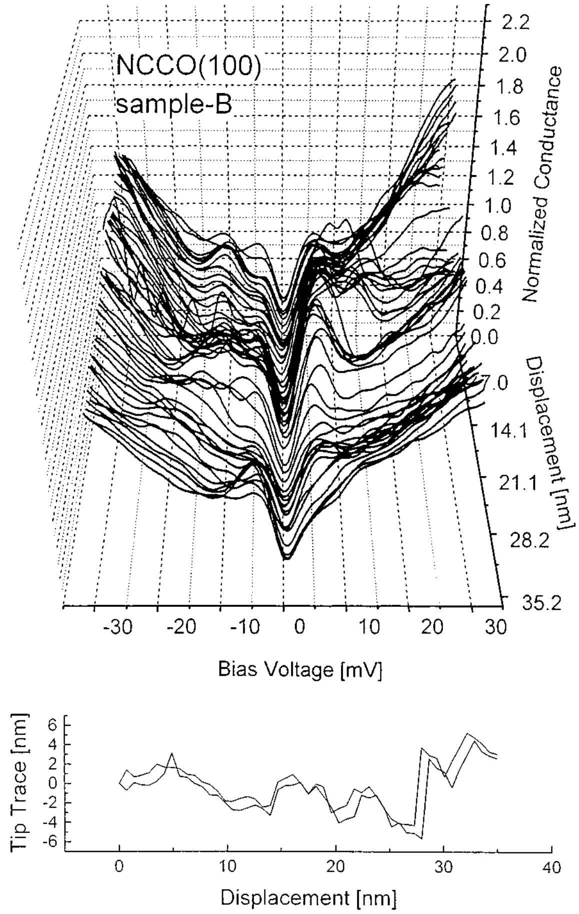

FIG. 8. An experimental result of STS data on (100)-oriented surface of sample B. The figure shows successive 50 points spectroscopy measurement obtained along a line of 35 nm. Although the gap structures are uniformly observed, some of the spectra show distorted background. They are obtained at the position near or around the steps on the surface as can be seen from the tip trace in the lower part of the figure.

of d-wave symmetry. On the other hand, the background forms of the spectra on the (100) surface have strong spatial dependence. Around the large steps, the spectra show strong asymmetries. This feature reflects the existence of the interference effect of the wave functions due to the random scattering at the surface.[24]

The ZBCP's have been reported in various types of tunneling junctions in the hole-doped high-T_c superconductors. For the origin of the ZBCP's in the hole-doped cuprates, several papers propose the Kondo-type scattering via localized magnetic impurities near surfaces or inside the barrier.[29–31] We have rejected the magnetic impurity effect as the origin of ZBCP's based on the tunneling formula for anisotropic superconductors and the experimental results of STS on YBCO surfaces.[6,11] The disappearance of ZBCP's in NCCO also supports our previous results because (i) the ends of Cu-O planes are not responsible for the localized spins, (ii) the disappearance of ZBCP's is consistent with the pairing symmetry determined by other measurement methods as described in Sec. I.

Although the pairing symmetries with no sign change in k space are strongly suggested, we cannot easily conclude the

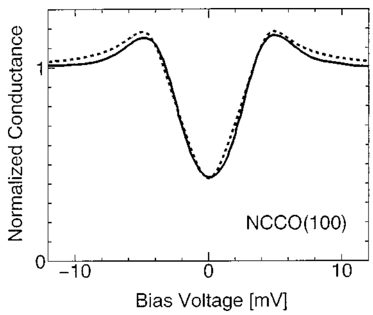

FIG. 9. A comparison between theory and experimental spectra. The solid line represents an experimental spectrum obtained on the (100)-oriented surface of sample A normalized by a parabolic background. The dotted line represents a theoretical curve for anisotropic s-wave symmetry.

conventional s-wave pairing. As is clear from the spectra in the figures, the gap structures are completely different from those expected for a conventional BCS superconductor.[26] The conductance at zero-bias level is not zero but about half of its background normal conductance. The gap structures are V-shaped forms. The peak structures around Δ are not present in some spectra. These features are commonly observed on the surfaces of NCCO, and quite similar to those observed on YBCO surfaces. These facts may suggest the similarity of the electronic states between YBCO and NCCO except the pairing symmetry.

To see more clearly the gap structure of NCCO, Fig. 9 shows the comparison between theoretical curve calculated from Eq. (2.2) with a experimental spectrum on the (100)-oriented surface of sample A. To get a good fit between theory and experiment, if we assume isotropic s-wave symmetry, an unphysically large Γ ($\Gamma \sim \Delta$) is required. If the maximum value Γ/Δ is assumed to be 0.2 in the analogy of YBCO cases, reasonable fitting is obtained by assuming anisotropic s-wave symmetry. The dotted curve in Fig. 9 is given by assuming

$$\Delta(\theta) = \Delta_0 + \Delta_1 \cos(4\theta) \tag{3.1}$$

with $\Delta_0 = 2.2$ meV, $\Delta_1 = 1.5$ meV, and $\Gamma = 0.2(\Delta_0 + \Delta_1)$. This form of the pair potential has no sign change for all directions which is consistent with the lack of ZBCP's discussed above. The large smearing factor is a feature common to high-T_c superconductors such as YBCO and LSCO. In the case of conventional BCS superconductors, even in the case of dirty superconductors, we have never observed tunneling spectra with such a large Γ. This fact may reject the possibility of the usual impurity effect for the origin of the large Γ. Three effects can be suggested for the candidates which are responsible for the origins of this large smearing factor. The first one is the strong electron correlation effect which is a feature common both to electron-doped and to hole-doped cuprates.[32] However, it is not so clear how the electron correlation effect works as the shortening of the lifetime of the quasiparticles at present. The second effect is the anisotropic pair potential effect. Especially, as explained in Ref. 7, the lack of peaks at $\pm\Delta$ is well understood by taking into account the anisotropic pair potential effect. However, this effect is not enough to wholly explain the origin of such a large smearing factor. The third effect is the surface degradation effect due to the exposure of the surface to the air. At the present technical level, *in situ* preparation of the ab-plane-oriented surface is not possible. Therefore, we cannot exclude the possibility of surface degradation in the present experiments. However, we believe this effect is not so serious because of the small dependence of spectra on the junction resistance. To get a more clear conclusion for the origin of the large smearing effect, more detailed experiments are strongly required.

The final discussion is whether the pair potential in NCCO has anisotropic s-wave or extended s-wave symmetry. In the case of the anisotropic s wave, the pair potential is definitely positive. While in the case of extended s-wave symmetry, the pair potential form strongly depends on the shape of the Fermi surface. The sign change of the pair potential occurs when the Fermi surface intersects the lines given by $\cos(k_x a) + \cos(k_y a) = 0$ in the Brillouin zone, where a is the lattice constant. Since the experimental data by ARPES suggest that the Fermi surface of NCCO across these lines in the vicinity of $(\pm\pi/2, \pm\pi/2)$ in the Brillouin zone,[19] the sign change seems to occur if we assume extended s-wave symmetry. With the decrease of the doping rate, the Fermi surface is reported to be enlarged.[19] Therefore the trend of the sign change is enhanced for the relatively underdoped samples. In our measurements, the doping rate of sample B is set to a relatively underdoped region which takes into account this effect. While in the experimental spectra, no enhancement of the zero-bias level in the conductance spectra have been observed even for sample B. Even admitting the influence of the large smearing effect by Γ, extended s-wave symmetry is less possible for the pairing states of NCCO. Recent theories propose the possibilities of a type of nodeless pairing states for electron-doped superconductors.[33,34] We believe our experimental results are consistent with their calculations.

IV. SUMMARY

The tunneling conductance spectra of NCCO have been measured by using the phase-sensitive capability of LT-STM. The orientational-dependent measurements show the lack of ZBCP's for all orientations, which is prominently different from YBCO results. By comparing the experimental results with tunneling theory for anisotropic superconductors, the possibility of d-wave symmetry of the pair potential is rejected for NCCO. Although the influence of large lifetime broadening effects cannot be excluded, a good fitting of experimental spectrum with theoretical curve is obtained by assuming anisotropic s-wave symmetry. The present results suggest the antisymmetric behavior in the superconducting electronic states of the cuprate for the conversion of the carrier-type between hole doping and electron doping.

Note added in proof. We have recently been informed about a related investigation regarding the lack of ZBCP in NCCO (Ref. 35). We thank J. W. Ekin for sending a reprint.

ACKNOWLEDGMENTS

The authors thank L. Alff for suggesting this topic. They also thank M. Ogata and K. Kuroki for valuable discussions.

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