d-wave superconductivity of the hole-doped $(SrCa)_{10}Cu_{17}O_{29}$ ladder compound

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(Received 27 July 1999)

We performed tunneling experiments on single crystals of hole-doped two-leg ladders $(SrCa)_{10}Cu_{17}O_{29}$ with $T_c \cong 75$ K and, for reference purposes, on textured bulk samples of $Bi_2Sr_2CaCu_2O_8$ (Bi2212). Point-contact and break-junction (Bi2212 only) junction configurations were used. The point-contact junctions, both superconductor–insulator–normal-metal and superconductor–normal-metal, were using Ag counterelectrodes. In all experiments the tunneling direction was along Cu_2O_3 planes (for the ladders) and CuO_2 planes (for Bi2212). The recorded tunneling conductance spectra were reproducible with sharp, well-defined energy-gap structures. All junctions exhibited a pronounced zero-bias conductance peak disappearing at the critical temperature. Experimental results are consistent with the formation of zero-energy Andreev bound states at the surface of the superconductors and $d_{x^2-y^2}$-wave symmetry of the order parameter Δ in both compounds.

I. INTRODUCTION

After the discovery of high-temperature superconductivity much interest was invested in low-dimensional quantum spin systems, including the so-called spin-ladder systems[1] of $((M_{1-x}M'_x)_2Cu_2O_3)_m(CuO_2)_n$ type (designated further on as $\Lambda_{m/n}$), where M stands for alkaline earth metal and M' for trivalent metal. These materials consist of alternate planes containing Cu_2O_3 two-leg ladders and single CuO_2 chains separated by M-M' layers. It has been shown theoretically that a two-leg spin ladder suitably doped may become superconducting with a $d_{x^2-y^2}$-wave symmetry.[1–3] Superconductivity was observed experimentally in some ladder compounds. The transition to superconducting state was observed at ambient pressure in single crystals of the $(M_2Cu_2O_3)_5(CuO_2)_7$ or$\Lambda_{5/7}$ compound,[4,5] while the samples of $Sr_xCa_{14-x}Cu_{24}O_{41.84}$ ($x=0.4$ and $x=2.5$) were superconducting at $3-4.5$ GPa at $9-12$ K.[6,7] No attempt was made in these experiments to determine the symmetry of the order parameter.

In this paper we report on the results of tunneling and Andreev reflection spectroscopy of the energy gap in the bismuth-doped single crystals of composition $(Ca_{5.84}Sr_{4.02}Bi_{0.14})Cu_{15.84}O_{29}$, a superconductor with $T_c \cong 75$ K. Similar measurements were performed on highly textured $Bi_2Sr_2CaCu_2O_8$ (Bi2212) bulk samples. All our measurements indicate in both compounds $d_{x^2-y^2}$ symmetry of the superconducting energy gap $\Delta(\varphi)=\Delta_0\cos(2\varphi)$ with $2\Delta_0/kT_c \cong 6.5$ (ladder) and $2\Delta_0/kT_c \cong 7.5$ (Bi2212).

II. EXPERIMENT

The superconducting and nonsuperconducting single crystals with ladder-type structure were obtained by the flux method using Bi_2CuO_4 and $BaCuO_2$ as fluxes. The flux method was chosen because it allows one to adjust easily the crystal growth parameters. A ''melted band'' method was developed for the growth of the $\Lambda_{5/7}$ - type crystals from a partly melted Bi-containing load at maximal temperatures below 960 °C.[8] At negative temperature gradient along the furnace radius the load melts only near the side walls of the crucible, while at the center and top it remains solid with the solvent melted out. The extracted solvent flows down into the zone near the crucible bottom. Single crystals of $\Lambda_{5/7}$ grow in a relatively cold zone. It is reasonable to assume that the growth of the single crystals is similar to the self-supporting mechanism determined for whiskers.[9] The investigated single crystals grow in a needlelike shape along the ladder c axis. The maximum size of the samples used in the measurements was approximately $0.4\times0.7\times4$ mm^3.

X-ray diffraction patterns confirmed that the obtained single crystals were monophase samples. X-ray structural analysis has shown that the crystals were indeed grown with indices $m=5$ and $n=7$. All the reflexes with intensity higher than 2% of the largest peak could be interpreted with an accuracy better than 2% for the interplane distances. The lattice constants of the investigated crystals as determined from the structural analysis were $a=11.346(2)$ Å, $b=12.809(3)$ Å, $c=19.52(1)$ Å. The chemical composition of the crystals was determined by electron probe microanalysis, and was found to be described by the formula $(Ca_{5.84}Sr_{4.02}Bi_{0.14})Cu_{15.84}O_{29}$. The characteristic feature of the superconducting $\Lambda_{5/7}$ samples is high Ca content (Ca amount higher than Sr) and Cu-site deficiency with respect to the stoichiometric composition.

$BiSr_2CaCu_2O_8$ (Bi2212) samples in the form of $10\times1\times0.1$ mm^3 rectangular bars were prepared by compacting powdered Bi2212 compound at $30-40$ kbar between two steel anvils. A grid of parallel copper wires with $D=0.1-0.2$ mm diameter fastened to the anvil surface served as the powder container during compression. In this manner the powder was compacted into dense plane-parallel bars about 0.1 mm thick. The current and voltage terminals were then painted on by silver paste, the bars annealed at $T=845$ °C for 16 h, compressed again, and finally annealed at $T = 830\,^\circ\mathrm{C}$ for 14 h in order to obtain a well pronounced texture.

Superconductor–normal-metal (S-N) (Andreev) and superconductor–insulator–normal-metal (S-I-N) (tunnel) junctions with silver N counterelectrodes were fabricated by two methods. The first, used for Bi samples, was based on pressing a fine silver powder into the ceramic material. The powder composed of particles with $d = 3\ \mu\mathrm{m}$ diameter was spread over the surface of the ceramic sample and fixed with a drop of silicon varnish. The sample was then subjected to hydrostatic pressure of up to 15 kbar. From Sharvin's formula[11] for the point-contact resistance $R_N = 4\rho l/3\pi a^2$, where we assume Ag resistivity $\rho \cong 1\ \mu\Omega$ cm and the mean free path $l \sim 0.1\ \mu\mathrm{m}$, we estimate the resultant point-contact radius to be $a \cong 1000$ Å.

The second method was used for the ladder samples and relied on baking the silver powder smeared on the sample surface. First the low-resistance voltage and current contact pads were prepared by baking the silver powder at $500-600\,^\circ\mathrm{C}$ for 2 h in air. Then the powder was spread over a small spot at the intended junction location and baked at $400-450\,^\circ\mathrm{C}$ for $40-60$ min in air. Leads were attached to the junction spot by a splash of indium solder. The outer diameter of such a junction was $1-1.5$ mm, while the area of the spectroscopic S-N microjunction estimated from Sharvin's formula is much smaller, about $10\ \mu\mathrm{m}^2$. Only a single S-N contact resulted from both described methods.

Superconductor-insulator-superconductor (S-I-S) junctions were prepared as "break-junctions," a form which is particularly appropriate for the study of high-temperature superconductors.[12] Superconducting gap features and their temperature dependence can be obtained in this configuration with high stability and reproducibility, and the peaks in tunneling conductance allow direct measurement of 2Δ. To prepare such junctions, the Bi2212 sample bars were glued with silicon varnish to a flexible steel plate. The external surface of the sample except for the contact pads was also covered by a 0.5-mm-thick layer of this varnish. After polymerizing, the sample-plate assembly became a solid unit. To form a break junction, the steel substrate was bent until the wafer was broken (without breaking the varnish coat). Then the external force was released and the substrate returned to the initial position.

The microjunctions formed this way inside the sample, were—as witnessed by their I-V characteristics—either of the tunneling type with $R_N \sim 50-100\,\Omega$, or of the S-C-S type, where C stands for constriction, with Andreev conductivity ($R_N \sim 1-5\Omega$). As a rule these junctions exhibited a crisp singularity at $eV = \Delta$, typical of well defined single junctions. Rarely faulty (multiple) junctions were obtained with smeared singularity indicating a variation of break angle with respect to crystal planes, but such samples were eliminated from further investigations. The investigated junctions were highly stable and their conductance could be repeatedly measured in the temperature range from 4.2 to 350 K. The varnish coating plays in the described fabrication method a very important role by protecting the ceramic compound from degradation and preventing mechanical instabilities. Because thin ($D \leqslant 0.1$ mm) highly textured ceramic bars are employed, the junction is formed from a single microcrystal ($d \sim 10\ \mu\mathrm{m}$) and its geometry is favorable for probing in-plane tunneling characteristics.

According to Ref. 10, the atomic force and scanning electron microscopy of cleaved surfaces of Bi2212 crystals reveal broad flat regions separated by large cleavage steps. The surface roughness of the flat regions was found to be approximately 15 Å (corresponding to the distance between BiO layers). The large cleavage steps were several mm apart and typically $1-10\ \mu\mathrm{m}$ high. We assume that in our case the situation was similar and the tunneling was realized predominantly in the direction of base CuO_2 planes, with the barrier roughly perpendicular to these planes. This assertion is confirmed by the comparison of the obtained experimental dI/dV versus V curves with the theory of tunneling spectroscopy of d-wave superconductors (see below). We found that the angle α between the normal vector to the junction interface and the CuO_2 plane of the microcrystal was small, $\alpha \leqslant 10°$. The cross section of a typical microjunction was about $0.1\times 0.1\ \mu\mathrm{m}^2$.

The differential conductance dI/dV as a function of voltage V was recorded using standard lock-in techniques[13] with a small ac modulation of 10 mV superimposed on a slowly varying bias voltage applied to the sample. Data acquisition was automated using computer control of programmable current sources and voltmeters. In some cases the dynamic conductance was determined through numerical differentiation. For the Bi2212 no filtering or numerical smoothing of the data was employed at any stage of data analysis. No correlation was noticed between the apparent junction quality and spectral behavior. The junctions were measured as a function of temperature in a standard helium cryostat equipped with a temperature-controlled sample holder.

III. TUNNELING AND ANDREEV SPECTROSCOPY

Tunneling spectroscopy provides a sensitive probe of the quasiparticle density of states $N(E)$ and thus renders possible a direct measurement of the superconducting energy gap Δ.[13] In a S-I-N junction the conductance dI/dV at $T = 0$ is proportional to $N(E)$, $dI/dV \sim N(eV)$. However, for the high-T_c superconductors a wide variety of tunneling spectra have been reported, generally deviating from the usual BCS behavior (see, for example Ref. 14 and references therein). Among the anomalies is the so-called zero-bias conductance peak (ZBCP) observed in many cases, especially for a-axis tunneling. Recent theories of tunneling spectroscopy for anisotropic superconductors take into account in a natural manner the formation of the Andreev bound states on the surface of superconductors and successfully explain the ZBCP in terms of $d_{x^2-y^2}$-wave superconductivity.[15,16] As far as Bi2212 is concerned, there are now ample reasons to believe that this material has d-wave pairing symmetry. For example, the observation of half-flux quantum in a tricrystal ring[17] indicates that the superconducting order parameter in Bi2212 changes sign along $|k_x| = |k_y|$ directions in (k_x, k_y) space, which is consistent with the $d_{x^2-y^2}$-pairing symmetry. We have used this result as a starting point for revealing the pairing symmetry of the ladder $\Lambda_{5/7}$.

In our tunneling measurements on monocrystalline ladder and highly textured Bi2212 samples we have always ob-

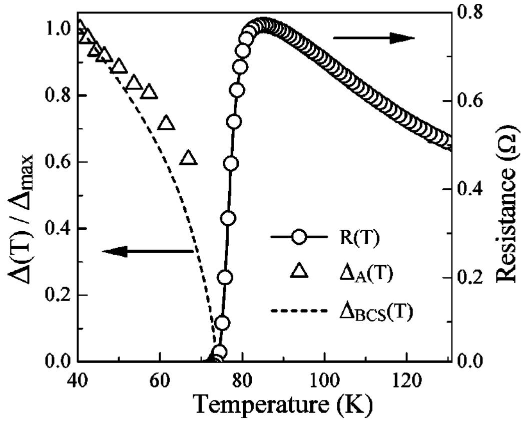

FIG. 1. Temperature dependence of c-axis normal resistance R_N and $\Delta_A(T)$ for single-crystal $(SrCa)_{10}Cu_{17}O_{29}$.

served clear and rather narrow ZBCP. Taking into account the above mentioned theoretical developments, we consider that this observation confirms the existence of Andreev bound states on the junction interface due to the $d_{x^2-y^2}$-wave symmetry of the pair potential in both Bi2212 and $\Lambda_{5/7}$ superconductors.

The anisotropic energy gap in a superconductor can also be deduced from the Andreev reflection measurements. As a result of Andreev reflection from the N-S interface, there is an interchange of electrons and holes, with a hole reflected in the opposite direction to the one in which the incident electron had been moving. This leads to doubling of the junction conductance $\sigma(V)=dI/dV$ at $V=0$, i.e., $\sigma(0)/\sigma(eV\cong\Delta)\cong 2$.[18] Such behavior in the reflection is retained even for large electron incidence angles. As a result, if the N-S junction is elaborated on a single crystal, Andreev reflected electrons carry information about the value of $\Delta(\mathbf{k})$ for all directions of the wave vector $\mathbf{k}$. Thus, by a single measurement of the conductance of a point-contact N-S junction it is possible to completely reconstruct the anisotropy of the energy gap $\Delta(\mathbf{k})$ of the superconductor. The Δ values obtained from Andreev spectroscopy will be further denoted as Δ_A.

In Fig. 1 is shown the temperature dependence of c-axis normal resistance R_N and $\Delta_A(T)$ for the investigated two-leg ladder $\Lambda_{5/7}$. The voltage dependence of $\sigma(V)=dI/dV$ of a $(Sr,Ca)_{10}Cu_{17}O_{29}$-Ag junction is shown in Fig. 2. No surface

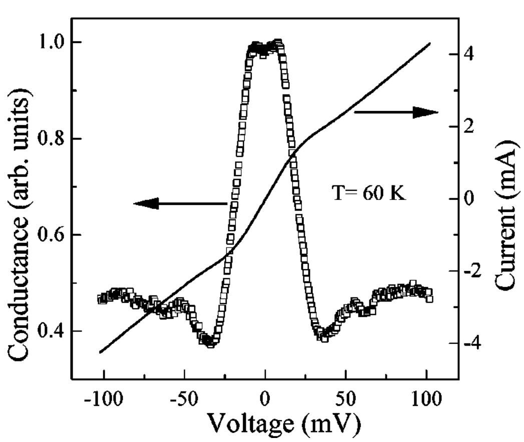

FIG. 2. *I-V* characteristic and conductance of the $(SrCa)_{10}Cu_{17}O_{29}$-Ag junction.

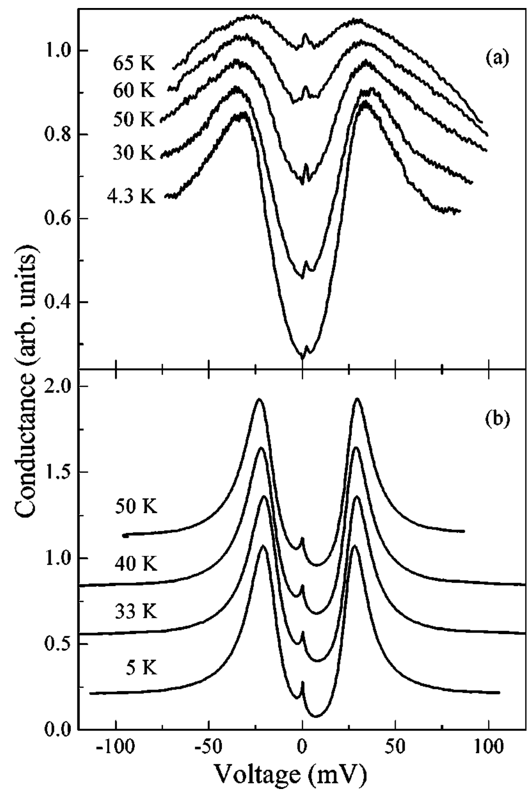

FIG. 3. Tunneling conductance of $(SrCa)_{10}Cu_{17}O_{29}$ (a) and Bi2212 (b).

degradation effects caused by oxygen migration[19] were observed.

Tunneling spectra of the ladder $\Lambda_{5/7}$ and Bi2212 are shown in Fig. 3. The peaks in the tunneling conductance at $eV\cong\Delta_T$ are clearly visible, as well as ZBCP. The distance between maxima of the $\sigma(V)$ curves yields twice the energy gap Δ_T.

The Δ_A dependence on temperature for Andreev $(Sr,Ca)_{10}Cu_{17}O_{29}$-*Ag* junctions is similar to the well-known BCS $\Delta(T)$ curve for the energy gap of a superconductor (cf. Fig. 1 and Fig. 4). On the other hand, the tunneling gap $\Delta_T(T)$ does not show any clear temperature dependence and remains temperature independent up to T_c, as can be seen from Fig. 3. This can be related to the pseudogap formation in Bi2212 (Ref. 20) and in ladders.[3]

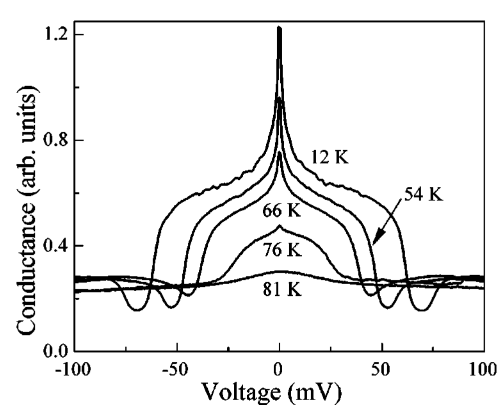

FIG. 4. Conductance spectra of Bi2212-Bi2212 break junction.

IV. GAP SYMMETRY

To get a feeling for the nature of the experimental results, we calculated the conductivity $\sigma(V)$ of a d-superconductor–normal-metal junction. For simplicity, we assumed that the anisotropy of the energy gap Δ is most pronounced in the ladder (ca)-plane (corresponding to the (ab) plane in the conventional cuprates) and we used the quasi-two-dimensional isotropic band structure. Then the expression for the tunneling conductance can be written as[16]

$$\sigma(V)=G_{NS}(V)\sim G_{NN}\frac{1}{T}\int\int \sigma_S(E+i\Gamma,\theta)\,\mathrm{sech}^2\left(\frac{E-eV}{2k_BT}\right)\exp\left[-\left(\frac{\theta}{\beta}\right)^2\right]dE\,d\theta, \quad (1)$$

where G_{NN} is the junction conductance in normal state, θ is the injection angle of the tunneling electron, β is the tunneling cone width,[13] and the quantity σ_S is given by

$$\sigma_S(E,\theta)=\frac{16(1+|\Gamma_+|^2)\cos^4\theta+4Z^2(1-|\Gamma_+\Gamma_-|^2)\cos^2\theta}{|4\cos^2\theta+Z^2\{1-\Gamma_+\Gamma_-\}|^2}, \quad (2a)$$

$$\Gamma_\pm(E,\theta)=\frac{E-\Omega(E,\theta\pm\alpha)}{\Delta(\theta\pm\alpha)}, \quad (2b)$$

$$\Omega(E,\theta)=\sqrt{E^2-|\Delta(\theta)|^2}, \quad (2c)$$

where α denotes the angle between the vector normal to the junction interface and the x axis (x is taken along the c direction of the ladders); the Blonder-Tinkham-Klapwijk[21] (BTK) parameter Z represents the barrier strength and the Dines parameter Γ accounts for the smearing of the singularity in Gor'kov's anomalous Green's function.[19] In the S-I-N case, assuming barrier of thickness $d=2$ nm, height $U\cong 2.5$ eV, and free electron mass m, we obtain $\beta^2=\sqrt{2mU}/\hbar d\approx 0.03$. The tunneling electrons are thus collimated within a narrow cone ($\beta\cong 6°$). In the Andreev S-N point contact this cone width becomes infinite.

To check on the amount of s- and d-wave order-parameter symmetry in the investigated compounds, we used the gap function

$$\Delta(\varphi)\approx\Delta_0[A\cos(2\varphi)+B\cos(4\varphi)+C\cos(6\varphi)+D]. \quad (3)$$

Here the coefficients B and D are related to the s-wave pairing and A and C to $d_{x^2-y^2}$ - wave symmetry.[22] The parameters A–D in Eq. (3) were adjusted so that the results provided by Eq. (1) would reasonably fit the experimental $\sigma(V)$ dependence of Fig. 3 and Fig. 4, depending on Z value. The calculated function $\sigma(V)=dI/dV$, shown in Fig. 5 reflects the main features of the experimental curves, especially the ZBCP peak at small bias voltages due to the Andreev bound states (Fig. 4).

In the Andreev bound-state model[15,16] the zero-bias anomaly (ZBCP) arises from the bound states formed by the constructive interference of electronlike and holelike quasiparticles. The superposition of electronlike and holelike waves on the free boundary can be written as

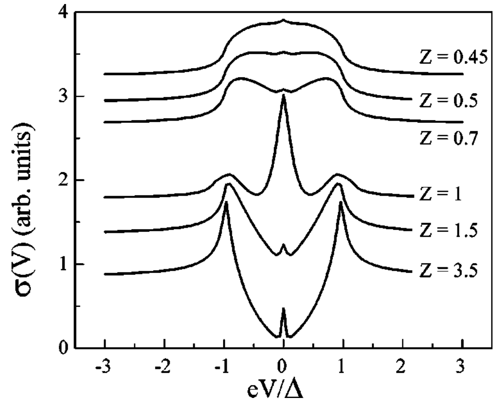

FIG. 5. Normalized tunneling conductance $\sigma(V)$ plotted as a function of eV/Δ for a normal-metal–insulator–$d_{x^2-y^2}$-wave-superconductor junction with the electron transmission in the crystal CuO_2 plane ($\alpha=6°$).

$$\Psi(\mathbf{r})\sim A\exp(ik_{\varpi}x)\begin{pmatrix}u_+\\ v_+\end{pmatrix}+B\exp(-ik_{\varpi}x)\begin{pmatrix}u_-\\ v_-\end{pmatrix}, \quad (4)$$

where $u_\pm/v_\pm=(E-\Omega)/\Delta_\pm$, $|u^2|+|v^2|=1$, and $\Delta_\pm=\Delta(\theta\pm\alpha)$. By requiring the wave function (4) to vanish at $x=0$ (the constructive interference), we obtain for the bound states the condition $u_+u_--v_+v_-=0$, or $\Gamma_+\Gamma_-\approx 1$. This condition gives the energy of bound states of a quasiparticle formed at the surface of a semi-infinite superconductor.

The Andreev bound state is formed if the scattering induces a change in sign of the order parameter $\Delta_\pm$ along the trajectories of the particles. Hence, the ZBCP's are not possible for s-wave symmetry of the order parameter. On the other hand, for the hole-doped ladder compound interladder interactions should be taken into account explicitly, and the superconductivity might be regarded as a two-dimensional phenomenon.[7] The conductivity along the b axis always has insulating behavior.[23] Therefore, for trajectories of the particles orthogonal to the Cu_2O_3 plane no Andreev bound states must be formed.

Qualitative analysis verifies the predominance of d-wave pairing symmetry in Bi2212 and $(Sr,Ca)_{10}Cu_{17}O_{29}$, with less than 5% of s-wave component. The angle α between the normal to the interface and the c (ladder) or a (Bi2212) axis was found to be small, $\alpha\leqslant 10°$. At higher α values and higher than 5% content of s-wave component it was impossible to reproduce the narrow ZBCP feature (Fig. 5). Let us remark that such a narrow ZBCP has often been observed in tunneling experiments performed on many different high-T_c superconductors (Bi2223, Hg2223) when the point-contact and break-junction technique was used (see, e.g., Ref. 24, and the many earlier references therein on this subject).

For Bi2212 Andreev N-C-S junctions the BTK parameter Z used to obtain theoretical fit was small, $Z\cong 0.45$. The Z value reflects the difference between Fermi velocities V_{FN} and V_{FS} for N and S metals, respectively,

$$Z=\left[Z_B^2+\frac{(1-V_{FS}/V_{FN})^2}{4V_{FS}/V_{FN}}\right]^{1/2}, \quad (5)$$

where $Z_B\approx 0$ is the parameter of strength of the real barrier. For the Fermi velocities of Bi2212 we have obtained the value $V_{FS}\cong 4.5\times 10^7$ cm/s.

V. DISCUSSION

During the last five years considerable progress has been made towards the resolution of the debate whether the order parameter in the high-T_c superconductors has nodes, or not (for a recent review see Ref. 25). Several recent phase-sensitive tests of the order parameter symmetry have provided very convincing evidence for d-wave pairing in the high-T_c cuprate superconductors (see, for example, Refs. 17 and 22). On the other hand, the consistent observation of Josephson tunneling between c-axis-oriented (twinned) Y-Ba-Cu-O and Pb has yielded strong evidence for mixed d- and s-wave pairing in Y-Ba-Cu-O.[26] There is also a theoretical possibility for the existence of mixed $d+s$ pairing in Y-Ba-Cu-O and Bi2112 as a consequence of their orthorhombicity.[22] Therefore, it is important to demonstrate experimentally the existence of pure d-wave high-T_c superconductors.

Our experimental results confirm $d_{x^2-y^2}$ order-parameter symmetry in cuprate superconductor $Bi_2Sr_2CaCu_2O_8$ and supply the looked for evidence of such pairing in the ladder superconductor $(SrCa)_{10}Cu_{17}O_{29}$. The observed zero-bias conductance peaks in tunneling conductance are present only if there is a change in sign of the pair potential in different **k** directions. Thus, the experimental observations of ZBCP (i.e., Andreev bound states) give definite evidence for the change of sign of the order parameter on the Fermi surface.

All superconductive cuprates are doped correlated insulators.[27] The underdoped ladders are Mott insulators with a spin gap in contrast to the antiferromagnetic high-T_c cuprates.[28] However, the transport properties of the doped ladders and cuprates have a common feature. Due to the pseudogap opening the charge transport is confined in the two-dimensional CuO_2 plane for high-T_c cuprates and in the Cu_2O_3 plane for spin-ladder compounds.[7,29] In a conventional CuO_2 superconductor there are linear ''rivers of charge'' (stripes) threading through the antiferromagnetic background.[30] These self-organized structures are a consequence of the tendency of the correlated antiferromagnet to expel doped holes.[31] The doped ladder compound $(SrCa)_{10}Cu_{17}O_{29}$ may have similar one-dimensional metallic and spin-gapped regions in close electrical contact built into their structure and not necessarily self-organized. The two-leg Cu_2O_3 ladder (which has a spin gap[1,4] and a pseudogap[29,32]) in intimate contact with doped CuO_2 chains could therefore display the mechanism of superconductivity analogous to conventional high-T_c cuprates.

VI. CONCLUSIONS

In summary, we have investigated the tunneling and Andreev S-N junction spectra of ladder $(SrCa)_{10}Cu_{17}O_{29}$ and cuprate Bi2212 over a wide range of energies and temperatures. The results are consistent with $d_{x^2-y^2}$-wave symmetry of the order parameter Δ in both compounds. For Bi2212 we observed characteristics which might indicate pure $d_{x^2-y^2}$-wave symmetry.

ACKNOWLEDGEMENT

This work was partly supported by the Polish Committee for Scientific Research (KBN) under Grant Nos. 2 P03B 102 14 and 2 P03B 148 14.

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