Observation of anomalous reentrant superconductivity in $Sr_{1-x}K_{y}BiO_{3}$

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(Received 14 September 2000; revised manuscript received 24 April 2001; published 11 July 2001)

We report the observation of a reentrant superconducting-normal resistive transition in $Sr_{1-x}K_xBiO_3$ superconductors. In contrast to previously reported reentrant resistive transition behaviors, the reentrant resistivity appearing at zero magnetic field is suppressed to zero by applying an external magnetic field (H) or increasing the electrical transport current (I) : an observation of the recovery of a zero resistive superconducting state induced by *H* or *I*. An analysis of the normal-state resistivity data indicates the important role that disordered junction barriers between superconducting grains might play on the observed reentrant resistivity behavior. Possible physical origins of this anomalous phenomenon are discussed.

DOI: 10.1103/PhysRevB.64.064502 PACS number(s): 74.72.Hs, 74.25.Fy

Previously, several experimental results for the reentrant superconducting-normal resistive transitions (RRT's) have been reported in various superconducting systems, including granular superconductors, Al thin films, and borocarbide $(RNBC)$ superconductors.^{1–4} If the general behavior of the RRT and its physical origins are considered, we can categorize the previously reported RRT's into two main groups: magnetic and nonmagnetic. The magnetic origin, observed in superconductors containing magnetic elements such as *RNBC* superconductors with $R = Tm$, Er, Ho, and Dy, involves the pair-breaking effect due to local magnetic moments. $¹$ The nonmagnetic origin was found in granular</sup> superconducting systems. In this case, the observed RRT was attributed to the destruction of the Josephson weak links caused by the increase of the tunneling resistance in the grain barriers at low temperatures.^{2,3} The RRT observed in granular Al films was also discussed on the basis of Fulde's theory in a dirty superconductor. 4 Also in cuprate superconductors observations of the RRT were reported.⁵⁻⁷ Especially the RRT found in Bi-2212/2221 intergrowth single crystals was explained by the weakening of the Josephson coupling between trilayers by applying a magnetic field or a large driving current.6,7 In all these reports, the superconducting region between the normal states was observed to be reduced by either an increase of *I* or *H*, and a further increase of *I* or *H* resulted in the eventual breakdown of the superconducting state. In addition to the RRT discussed above, a different normal-superconducting transition induced by *H* was observed in $Eu_{x}Sn_{1-x}Mo_{6}S_{8}^{8}$. The superconducting state of this compound at low temperatures $(T<1.15 K)$ breaks down at low *H* ($H > 1$ T) but at high *H* ($H > 3$ T), the superconducting state is recovered. This phenomenon was explained in terms of the Jaccarino-Peter compensation effect, the magnetic interaction of conduction electrons with the rare-earth ferromagnetic ions (Eu^{3+}) .⁹

Contrary to previously observed RRT's, another type of the RRT has recently been reported in the $Sr_{1-x}K_xBiO_3$ $(SKBO)$ compound.^{10,11} At zero magnetic field the reentrant resistivity suddenly appears at a temperature below T_c , and it becomes suppressed to zero by applying *H* or increasing *I*;

that is, one observes the recovery of the zero resistive superconducting state by applying a magnetic field or increasing the electrical transport current. The peculiarity of this observation is that the observed RRT behavior with respect to *H* and *I* cannot be simply understood by the general Josephson coupling mechanism between superconducting grains, which becomes the normal state by applying *H* and *I*. This requires a consideration of another special tunneling mechanism with a specific character of the tunneling barriers in our observations. In this paper, we present the magnetotransport data of various SKBO samples confirming the previous reports on this RRT behavior and carry out the phenomenological analysis that would give physical insight into the observation of this anomalous RRT phenomenon.

The SKBO compound is a recently discovered superconducting bismuthate having a similar structure to $Ba_{1-x}K_xBiO_3$, where the Ba^{2+} site is replaced by $Sr^{2+}.12-14$ To synthesize the SKBO compound, first the stoichiometric amounts of $Sr_2Bi_2O_5$, KO_2 , and Bi_2O_3 with a nominal Sr/K ratio of 0.4/0.6 was ground and sealed in a Au capsule inside an Ar-filled glove box. After placing the Au capsule in a belt-type high-pressure furnace, pressure was increased to 2 GPa and the temperature was increased to 700° C. This was maintained for 60 min. Four samples were synthesized for the magnetotransport measurements. Sample 1 and sample 2 were made using the above synthesis condition. For sample 3, the reaction time was 20 min while for sample 4 the synthesis pressure of 5 GPa was applied. Regardless of different synthesis conditions all samples show identical x-raydiffraction patterns, that of a single perovskitelike phase of SKBO with typical lattice parameters $a = b = 5.94$ Å and *c* $=8.43$ Å 15 The maximum amount of impurity phase found in certain samples is less than 5%, around the resolution limit by x ray. The energy dispersive spectroscopic (EDS) analysis generally gives the K content $x=0.45-0.6$, and the average grain size estimated from the scanning electron microscope (SEM) image is about 0.5 μ m in all samples. No definite grain-boundary structures or secondary phases in the grain boundaries were observed in the SEM images of the measured samples. It is noted that the sample characterization by

FIG. 1. Resistive transition curves of our four SKBO samples as a function of temperature in various *H* fields.

x-ray diffraction, EDS, and SEM does not show any detectable differences between the samples although, as shown below, the measured magnetotransport data vary from sample to sample. For the magnetotransport measurement, the sample was cut into a rectangular bar shape of typical size 1.5 mm \times 0.875 mm \times 0.25 mm. The sample surface was polished carefully to remove possible surface contamination and gold electrodes were evaporated onto the sample surface in a four-probe configuration. Silver paste was used to attach gold wires onto the gold electrodes. A 7-T superconducting magnet system (Janis Research Co.) was used with a Keithley 220 dc current source and a Keithley 182 nanovoltmeter.

Figure 1 shows the temperature dependence of resistivity for our SKBO samples in various magnetic fields with *I* $=100 \mu A$. In sample 2 and sample 3 the RRT is clearly observed below T_c at $H=0$. In sample 2 the observed reentrant resistivity (ρ_{re}) below the reentrant temperature T_{re} \sim 5 K increases with decreasing temperature at zero magnetic field. Surprisingly, as the magnetic field is applied to the sample, the reentrant resistivity (ρ_{re}) suddenly disappears. Further increase of H gives a shift of T_c to lower temperatures without any appearance of ρ_{re} below T_c . A similar RRT behavior at $H=0$ is also observed in sample 3 with a different reentrant temperature $T_{re} \sim 8$ K. For sample 3 the applied magnetic field does not completely suppress ρ_{re} as in case of sample 2, but decreases ρ_{re} with an increase of *H*. Competition between the suppression of T_c and the

FIG. 2. *I*-*V* curves of SKBO samples showing the RRT or quasi-RRT behavior at $T=2$ K with $H=0$. Inset: *I*-*V* curves of sample 3 measured at various temperatures with $H=0$.

decrease of ρ_{re} induced by *H* gives a resistivity minimum at $T \sim 6.6$ K at $H=0.5$ T in sample 3. In contrast to sample 2 and sample 3, the RRT is not found in sample 1 below T_c and in sample 4 a quasi-RRT $(RRT$ without a zero resistivity state) is observed with the same decreasing behavior of ρ_{re} with increasing *H*. The first sample that had been measured was sample 2. In that measurement no changes of the RRT behaviors by cooling or heating the sample with or without the magnetic field across T_{re} were found with reproducible RRT behaviors. The RRT behaviors didn't show any dependencies on the magnetic-field direction or field history either. Also no hysteresis in *I*-*V* curves was observed depending on the direction of the current flow. So in subsequent measurements on other SKBO samples (sample 3, sample 4) we measured the samples with random sequences.

Another peculiar feature is observed in the *I*-*V* characteristics of the samples exhibiting the RRT or quasi-RRT behavior (see Fig. 2). Below T_{re} an ohmic behavior is first found in the low current region $(I \sim 100 \mu A)$. As *I* increases further, the reentrant voltage sharply decreases and eventually becomes zero, similar to the *H* dependence of ρ_{re} . If the sample temperature is increased (see the inset of Fig. 2), the overall magnitude of the reentrant voltage decreases and, in the region T_{re} \leq $T \leq T_c$, a zero resistivity state is observed, followed by the usual ohmic behavior above T_c . For sample 1 showing no RRT, the $I-V$ curves below T_c simply follow the general characteristics of a superconducting sample.¹⁶ Namely, the superconductivity disappears and the sample resistivity increases as the applied current becomes higher.

The temperature dependence of resistivity for the SKBO samples at $H=0$ is replotted in Fig. 3. Figure 3 shows that the residual resistivity obtained by extrapolating $\rho(T)$ for the normal state to 0 K increases monatomically as the behavior

FIG. 3. Temperature dependence of resistivity of various SKBO samples at zero magnetic field. In the inset, the solid line represents the fitting result obtained by using Eq. (2) .

of the sample below T_c changes from a simple superconducting transition to the RRT and then to the quasi-RRT with an increase of a $\rho(20 \text{ K})/\rho(273 \text{ K})$ ratio (see Table I). Also, sample 1 is found to have a sharper superconducting transition (ΔT_c =1.3 K) with a smaller ρ (20 K) while sample 4 has a larger $\rho(20 K)$, compared to those of the RRT samples.

Through the series of measurements on various SKBO samples, we observed a systematic change from superconducting to the insulating behavior below T_c via the RRT and the quasi-RRT as the normal-state transport properties of the sample go from metallic to insulating.^{15,16} This implies a percolating behavior of superconductivity in our system and the anomalous behaviors below T_c are related to the normalstate transport property of the sample. To analyze the normal-state transport data of the samples in connection to the RRT or quasi-RRT behaviors, we used a tunneling percolation model proposed by Pury and Caceres.¹⁷ In this phenomenological model the electrical behavior of granular superconductors at low temperatures was investigated by considering the random transition rates in a percolating system composed of a metallic junction between two metallic grains and a tunneling junction due to an insulating barrier between grains. The normal-state transport property is mainly governed by the concentration of the metallic bond *p* and the temperature dependence of the transition rates. The tunneling percolation model predicts the temperature dependence of resistance in a disordered media to behave as $R(T) = CT/w_{eff}$, where w_{eff} is an effective transition rate for carriers and *C* is a constant depending on the carrier properties and geometrical form factors of the sample. If we assume *p* in the samples showing the RRT or quasi-RRT is near the percolation threshold limit $p \sim p_c$, then w_{eff} calculated from the effective-medium approximation at $p = p_c$ will be

$$
w_{eff}(T) = \sqrt{\frac{d-1 - d(1-p_c)^2}{d-1}} \sqrt{w_t(T)w_s(T)},
$$
 (1)

where $w_s(T)$ is a transition rate in the metallic junction and $w_t(T)$ is a transition rate in the tunneling junction. The dimensionality *d* of the percolating network is 3 in our system and p_c (= $\sqrt{(1/d)}$)=0.577.¹⁷ First we assume a temperature dependence for the metallic junctions of $w_s(T) = C_1 T^{-1}$ where C_1 is a constant. To check the validity of this assumed form for $w_s(T)$ we imagine a system in the metallic limit $p > p_c$. In this limit $w_{eff}(T)$ is simply $w_s(T)$,¹⁷ which gives a T^2 dependence for resistivity on substituting $w_s(T)$ into $R(T) = CT/w_{eff}$. The previous observations of the T^2 dependence for resistivity below $T \sim 100$ K in other superconducting bismuthates confirms our assumed $w_s(T)$ reasonably well.^{18,19} For $w_t(T)$, instead of assuming a simple activationtype temperature dependence used in modeling the insulating barrier in the general Josephson junctions, we use the equation $w_t(T) = C_2 \exp[-U/(T+T_0)]$ for the tunneling junctions. This equation has the same form as that for transport pro-

TABLE I. Transport properties of SKBO samples showing the RRT or quasi-RRT. $T_{c_{onset}}$ is the temperature where the ρ drop occurs and $T_{c_{zero}}$, the temperature where $\rho=0$. Fitting parameters α and β are obtained by using Eq. (1). ρ_0 , *U*, and U/T_0 are obtained by fitting Eq. (3) to the normal-state resistivity. For sample 4, which shows the quasi-RRT, *Tre* is defined as the temperature at which resistivity starts to increase below $T_{c_{onset}}$

	Sample 2	Sample 3	Sample 4
$T_{c_{onset}}/T_{c_{zero}}$	12.5/10.1	12.6/10.5	12.0/quasi-RRT
T_{re} (K)	~1	\sim 8	\sim 8
$\rho_{re}(2 \text{ K})(m\Omega \text{ cm})$	0.95	7.00	3.23
$\rho(20 \text{ K})(m\Omega \text{ cm})$	3.97	4.29	4.88
$\rho(20 \text{ K})/\rho(273 \text{ K})$	0.81	0.97	1.29
$\rho_0(m\Omega \text{ cm}), U(K)$	3.72,220	4.34,331	4.55,558
U/T_0	12.02	31.03	19.77
α, β	1.64×10^{-4} , 1.634	2.98×10^{-5} , 2.513	5.98×10^{-4} , 1.409

cesses in a fluctuation-induced tunneling model given by Sheng.²⁰ In Sheng's theory for fluctuation-induced tunneling, the tunneling process between metallic islands is governed by the change of the disordered insulating barrier due to thermal fluctuation. Here, *U* indicates the temperature scale for the fluctuations, U/T_0 is proportional to the barrier size in the zero-temperature limit, and C_2 is a constant. Substituting $w_s(T)$ and $w_t(T)$ into Eq. (1) and using $R(T) = R_0$ $+ C T/w_{eff}$ [we add a constant resistance term R_0 to $R(T)$ where R_0 is associated with scattering in the zerotemperature limit], we have the temperature dependence of resistivity for our system as

$$
\rho(T) = \rho_0 + C_3 T^{3/2} \left[\exp\left(\frac{U}{T + T_0}\right) \right]^{1/2}.
$$
 (2)

Fitting parameters of the SKBO samples displaying the RRT or quasi-RRT are given in Table I. The increase of *U* and ρ_0 follows a slight change of the temperature dependence of resistivity in the normal state from metallic (sample 2) to semiconducting $(sample 4)$. In connection with the RRT behavior below T_{re} , this increase corresponds to an increase of T_{re} for the sample. The magnitude of $\rho_{re}(T)$ for each sample can be related to the relative barrier size estimated from U/T_0 . The investigation on the normal-state resistivity data using the tunneling percolation model indicates that the role of disordered junction barriers between superconducting grains is important in the observed RRT phenomenon. We empirically found that the temperature dependence of ρ_{re} for $H=0$ can be fitted to the following power-law-type equation:

$$
\rho_{re}(T) = \alpha (T_{re} - T)^{\beta}.
$$
 (3)

Interestingly, the fitting value of β shown in Table I seems to be proportional to U/T_0 obtained from the normal-state analysis.

In regard to *I* dependencies of the RRT, we first consider the current-induced self-magnetic field effect. The selfmagnetic field (H_{self}) generated by the transport current (*I* $=100 \mu A$) in the resistance measurements is estimated to be approximately 0.001 G by using the equation H_{self} $=1.25I/C_r$, where C_r , the circumference of the sample, is about 1 mm from the typical size of the measured samples $(1.5 \text{ mm} \times 0.875 \text{ mm} \times 0.25 \text{ mm})$.²¹ The estimated H_{self} is found to be very small compared to the applied external magnetic field. If the magnetic field (H_0) , which limits the Josephson critical current flowing across the grain boundary, is further considered, given an average grain size *L* ≈ 0.5 µm from the SEM image of the sample and the penetration depth $\lambda_L \approx 0.5 \mu$ m, the equation $H_0 \approx 2.07$ $\times 10^{-7} / (\lambda_L L)$ yields $H_0 \sim 40 \text{ G}^{21}$ This generally gives an order of 10^3 A/cm² as the critical current across the junction barriers. Actually the above calculations of H_{self} and H_0 are strictly based on the general Josephson coupling, which becomes weaker by applying *H* or increasing *I*. This is opposite to the observed RRT behaviors in our sample, suggesting a different coupling mechanism between the superconducting grains in the RRT observed region. The observed currentcontrolled negative differential resisitivity (NDR) below T_{re} reminds us of the NDR behavior in a percolating network system. A simple phenomenological model introduced by Peinke *et al.*²² could explain the NDR behavior observed in other systems in terms of a percolative formation of lowresistivity phases induced by $I^{23,24}$ If we assume that a percolative formation of a resistive path gives ρ_{re} below T_{re} , then the *I* dependence of the RRT indicates an unusual increase of the superconducting domains with a decrease of resistivity domains due to *I*.

Since the observation of recovery of the superconducting state by applying *H* or increasing *I* is the first observation to our knowledge, no definite physical models are available at present to explain the observed RRT phenomena and it only allows us to discuss possible physical origins of this qualitatively. The analysis on the normal-state transport properties and *I*-*V* characteristics causes us to consider the role of disordered insulating barriers below T_{re} in *H* and *I* dependencies. Obviously the disordered insulating barrier is not a simple insulating barrier expected in the Josephson weak link model used to explain the previously reported RRT. 2 In our case the barriers between superconducting grains become transparent by applying *H* or increasing *I*, leading the supercurrent to flow. It doesn't mean that the general Josephson coupling mechanism at weak links is entirely inapplicable to our case, because at high magnetic field and transport current regions the general Josephson coupling that breaks down by applying *H* or increasing *I* was indeed observed. So the barriers between superconducting grains in our system would be a combination of normal Josephson tunneling junctions and anomalous tunneling junctions responsible for the observed RRT phenomena. For the anomalous tunneling junction that becomes superconducting by *H* or *I*, disorder in the insulating barrier seems to be important specifically. The disorder in this tunneling barrier could be related to U/T_0 from the normal-state transport data, which was proportional to the magnitude of $\rho_{re}(T)$. If disorder in tunneling barriers is considered, one might speculate possible localized spin states induced by disorder in the Josephson tunneling barrier.^{11,16} The localized spin state in the tunneling barrier can be regarded as one form of the π junctions.²⁵ The π junction, which is often called ''the negative Josephson junction,'' proposed by Bulaevskii, Kuzii, and Sobyanin,26 is the Josephson junction with a spin-flip tunneling between superconducting grains. The occurrence of the π junction naturally generates a normal state, which results from the breakdown of the normal superconducting weak links around the π contact.²⁷ At a certain *H* the spin-flip tunneling process can be inhibited by the external magnetic field and the normal Josephson junction increases with a decrease of the π -junction portion, recovering the bulk superconductivity. In addition, it was demonstrated that the π junctions can be produced by correlation effects up to an order of 50% of Josephson junctions formed at weak links in a disordered *s*-wave superconductor near the superconductor-to-insulator transition.^{25,28} The above scenario about the π -junction formations due to disorder seems to be physically plausible in our systems. However, we admit that the above scenario is just one possibility up to now and detailed studies on the observed RRT in a more quantitative manner are ongoing and will be reported in a forthcoming paper. It is emphasized that the SKBO compound contains only nonmagnetic elements with little possibility for any magnetic impurities to be included in synthesizing SKBO, eliminating any magnetic origins for the observed RRT phenomena.

There could be other possibilities for understanding our data. For example, it might be possible to consider a localization effect in the RRT behavior. The reduction of ρ_{re} by applying *H* resembles the negative magnetoresistance in the weak-localization theory in disordered systems.²⁹ In a granular superconductor, if the localization length is assumed to be larger than the Josephson coherence length at low temperatures below T_c , charges might be localized giving a ρ_{re} that decreases with applied *H*. However, the negative magnetoresistance by weak localization would have to be observed in the normal state by applying a high *H* at $T < T_c$,³⁰ which is not the case in our observations. Also there has been a prediction of the reentrant superconductivity in granular superconductors with a small grain size of the order of 100 \AA :³¹ The reentrant normal state below T_c is expected when the Coulomb charging energy is stronger than the Josephson coupling energy at low temperature. In consideration of the charging energy in our observations we found that the grain size of our samples (0.5 μ m in the SEM image) is much larger than the situation where the charging energy in the insulating barrier becomes important. The *H* dependence of the observed RRT in our samples, however, suggests a decrease of the electrostatic charging energy by the magnetic field resulting in the appearance of superconductivity, which looks to be difficult to consider. This again indicates the

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importance of the role of insulating barriers in the observed RRT phenomena.

After measuring the magnetotransport data of our samples, the magnetic susceptibility of each sample was checked using a dc superconducting quantum interference device magnetometer. In the zero-field-cooled mode measurement all samples showed a diamagnetic signal at *H* >50 G without any paramagneticlike signal in the normal state, thus confirming the absence of any magnetic impurities. Interestingly, in sample 2, a gradual decrease of the diamagnetic signal (an appearance of the paramagnetic signal) was observed below $T \sim 4.5$ K at $H \le 50$ G, which recovers the simple diamagnetism by applying $H > 50$ G.¹¹ The investigation on this anomalous behavior of the diamagnetic signal with different SKBO samples is ongoing.

In summary, we have observed the anomalous RRT phenomenon which is the recovery of a zero resistivity superconducting state by applying a magnetic field or increasing the electrical transport current in certain SKBO compounds. From a comparison of the magnetotransport data of various samples, the RRT is found to be closely related to the normal-state transport properties. A phenomenological analysis on the normal-state transport properties along with the RRT behaviors suggests that the observed *H* and *I* dependencies of ρ_{re} might originate from unusual characteristics of the disordered tunneling barriers between superconducting grains.

This work was supported by the Korea Research Foundation $(2000-042-D00032)$ in the Ministry of Education (MOE), Korea.

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