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Effects of Zn substitution on the superconductivity and pseudogap in $HgBa_2CuO_{4+\delta}$ with various doping levels

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A series of high-T_c superconductors $HgBa_2Cu_{1-x}Zn_xO_{4+\delta}$ (Hg-1201, $x=0$ and 0.03) with various doping levels was synthesized, and the effects of Zn substitution on superconductivity and pseudogap were investigated on the basis of dc susceptibility, thermoelectric power, and electrical resistivity data. T_c decreased with Zn substitution over a wide doping range, but most strongly at a heavily underdoped state, without any appreciable change in hole concentration irrespective of doping level. A disappearance of superconductivity or a sharp drop in T_c by Zn substitution is not observed in the vicinity of $p=1/8$, which suggests no enhancement of charge/spin stripe order. Analyses of temperature dependence of Seebeck coefficient S and resistivity revealed that there is no clear change with Zn substitution in the characteristic temperature $T^\star$ associated with the pseudogap, while the transport data are affected below $T^\star$ by Zn substitution.

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I. INTRODUCTION

The Zn-substitution effects in high-temperature superconductors (HTSC's) have been extensively studied since they were expected to provide key insights into the mechanism of HTSC's. As to the effect on superconductivity, it is understood that Zn primarily acts as a strong scattering center which destroys Cooper pairs in a *d*-wave superconductor.[1,2] One of the recent interesting topics for Zn-substitution effects is the pinning of the spin/charge stripe order.[3,4] Koike *et al.* reported that T_c is strongly suppressed by Zn substitution at hole concentration $p\approx 1/8$ in $La_{2-x}Sr_xCuO_4$ (La-214),[3] and $Bi_2Sr_2Ca_{1-x}Y_xCu_2O_{8+\delta}$ (Bi-2212),[4] but not in $YBa_2Cu_3O_7$ (Y-123).[5] It seems that the stability of stripe order is sensitive to a crystal structure including lattice distortion. Therefore it is desired to study the same phenomenon for other HTSC's with a simple and less distorted structure like Hg-based HTSC's.

The Zn effect on the pseudogap is also very important to understand the nature of the pseudogap[6] and its difference from the superconducting gap. In transport properties such as resistivity,[7] Hall coefficient,[8] and thermoelectric power,[9] no significant change in pseudogap temperature $T^\star$ with Zn substitution at optimal and under doping states has been observed. Williams *et al.* also found a negligible change in the spin-gap energy in $YBa_2Cu_4O_8$ (Y-124) (Ref. 10) in the NMR study. On the other hand, there is a report in which the pseudogap temperature decreased by Zn substitution in the temperature dependence of T^{-1} in NMR spectra of the same material.[11]

Most of the Zn-substitution effect experiments were limited to La-214, Y-123, Y-124, and Bi-2212. Among them, the Zn effects on La-214 is peculiar. The magnitude of S for the Zn-unsubstituted sample in La-214 (Refs. 12 and 13) is relatively large and $S(T)$ is different from those in Y-123 and Y-124 (Ref. 9) at the same doping level, while $S(T)$ with the Zn-substituted sample seems to be rather similar to those of other HTSC's[13]. The behavior of the spin-gap temperature T_s, which may be related to the pseudogap, in the NMR study for La-214 is also quite different from those of other HTSC.[14] Therefore it is hard to regard La-214 as a prototype of HTSC's with a single CuO_2 layer in the study of pseudogap. An attractive system is $HgBa_2CuO_{4+\delta}$ (Hg-1201), which exhibits the highest T_c of 98 K (Ref. 15) among HTSC's with a single CuO_2 layer in contrast to La-214 with T_c of 40 K (Ref. 16) and $Bi_2Sr_{2-x}La_xCuO_{6+\delta}$ (Bi-2201) with 35 K.[17] The compound with a single CuO_2 layer is appropriate for the study of Cu-site substitution because there is only one substitution site. The following, except for the high-T_c value, are the advantages of Hg-1201. First, the crystal structure is very simple and there is almost no distortion of the CuO_2 planes, while there is a tilting of the octahedron in La-214 (Ref. 18) and the BiO modulation in Bi-2201.[19] Second, the hole concentration can be widely controlled by changing oxygen content.[20] In spite of these advantages, no Cu-site substitution effect has been studied for Hg-1201 so far, probably owing to a difficulty in sample preparation.

Recently we have developed a synthetic method of high quality Hg-1201 using nearly carbon-free materials [21] and studied the dependence of T_c and T^* on hole concentration p over a wide doping range.[22] The $T_c(p)$ variation as a function of hole concentration was basically parabolic in $0.05<p<0.20$ as is seen in other HTSC's. We found a very weak plateau at around $p=1/8$ in our $T_c(p)$ curve.[22] Although it is not clear whether this plateau is related to 1/8 anomaly or not, it is of great interest to investigate the Zn-substitution effect on this problem. Another important result was the carrier doping dependence of the pseudogap temperature. We successfully determined the temperature using the scaling technique[23,24] for the Seebeck coefficient and resistivity.[22] $T^\star$ at the optimally doped state is much higher than T_c.

In the present study, we proceed to study the Zn-substitution effect on the superconducting and transport

TABLE I. Annealing condition, superconducting critical temperature T_c, copper valence, oxygen content, Seebeck coefficient S at 290 K, and hole concentration p of $HgBa_2Cu_{1-x}Zn_xO_{4+\delta}$ ($x=0$ and 0.03). Annealing time for all samples is about 5 days. Oxygen content calculated on the assumption of stoichiometric composition in metals. Hole concentration p was estimated from S using the empirical relation. ΔT_c is difference between T_c of $x=0$ and that of $x=0.03$.

(a) $x=0$

Label	Annealing condition	T_c (K)	Oxygen content	Cu valence	S at 290 K (μV/K)	p
a	Vac. 400 °C	36	4.032($\pm$0.06)	2.064($\pm$0.03)	84.9	0.052
b	Vac. 350 °C	48	4.033	2.066	66.3	0.062
c	Ar 450 °C	72	4.043	2.086	29.2	0.082
d	Ar 300 °C	82	4.052	2.104	13.3	0.103
e	O_2 0.01% 300 °C	89	4.058	2.116	7.2	0.123
f	O_2 100% 300 °C	95	4.073	2.146	4.3	0.132
g	O_2 5atm 300 °C	98	4.081	2.162	0.6	0.168

(b) $x=0.03$

Label	Annealing condition	T_c (K)	Oxygen content	Cu valence	S at 290 K (μV/K)	p	ΔT_c (K)
a	Vac. 400 °C	>2	4.032($\pm$0.06)	2.045($\pm$0.03)	79.4	0.052	<34
b	Vac. 350 °C	28	4.027	2.056	66.6	0.061	20
c	Ar 450 °C	53	4.043	2.089	25.9	0.086	19
d	Ar 300 °C	69	4.052	2.107	12.3	0.107	13
e	O_2 0.01% 300 °C	75	4.063	2.130	7.5	0.118	14
f	O_2 100% 300 °C	82	4.073	2.140	3.4	0.141	13
g	O_2 5atm 300 °C	83	4.081	2.167	2.0	0.153	15

properties of Hg-1201 over a wide doping range. It was revealed that a pinning of the spin/charge stripe is not induced by Zn doping in this system from a dependence of T_c on hole concentration. It was also found that changes in pseudogap temperature with Zn substitution were almost negligible over a wide doping range.

II. SAMPLE PREPARATION AND EXPERIMENTS

Samples with nominal composition of $HgBa_2Cu_{1-x}Zn_xO_{4+\delta}$ ($x=0$ and 0.03) were prepared from HgO (99.9%), BaO, CuO (99.999%), and ZnO (99.9%) by the solid-state reaction method.[25] BaO was purified from $BaCO_3$ (99.995%) according to the procedures described in Ref. 21. Mixed powders were pressed into bar-shaped blocks in a dry box filled with high-purity Ar gas. The pressed sample was sealed in a quartz tube, heated for 20 h at 930 and 850 °C for $x=0$ and 0.03, respectively. Then, it was quenched in cold water. In this study, we used only one sample bar with $x=0$ and another with $x=0.3$. Each sample was divided into about 20 pieces, which were annealed under various conditions listed in Table I to achieve various doping levels. This procedure excludes a difference in cation ratio among the samples at a fixed x and enables us to make a reliable comparison among them.

Phase identification was carried out using x-ray powder diffraction (XRD) and the lattice parameters were determined from the XRD data by the least-square method. The compositions were analyzed by an inductively coupled plasma emission spectrometer (ICP-ES). The copper valences of the samples were determined by the iodometric titration method,[26] and the oxygen contents were estimated from the copper valence on the assumption of a stoichiometric composition in metal. Dc susceptibility was measured by a superconducting quantum interference device (SQUID) magnetometer with 20 Oe in the zero-field-cooling and field-cooling modes. Thermoelectric power was measured using the four-probe method with a temperature gradient of 0.3–0.5 K.[27] The sample temperature was measured using a calibrated Si diode sensor and the temperature difference between the two ends of the sample was measured by two pairs of Cu-constantan thermocouples. The absolute thermoelectric power was evaluated by subtracting the thermoelectric power of the lead (Cu). Measurement of electrical resistivity was carried out by the conventional four-probe method with a current of 1–20 mA.

An XRD study confirmed that there is no detectable impurity phase in our samples both before and after the annealing. The lattice parameter a of the Zn-substituted sample was longer than that of the Zn-free sample in all doping levels.[25,28] This is the evidence that Zn ions were substituted for Cu ions, since an enhancement of lattice parameter a was also reported in Zn-substituted La-214 (Ref. 29) and Y-123.[30] ICP analysis revealed no significant Hg deficiency.

Determination of the hole concentration p for Zn-free and

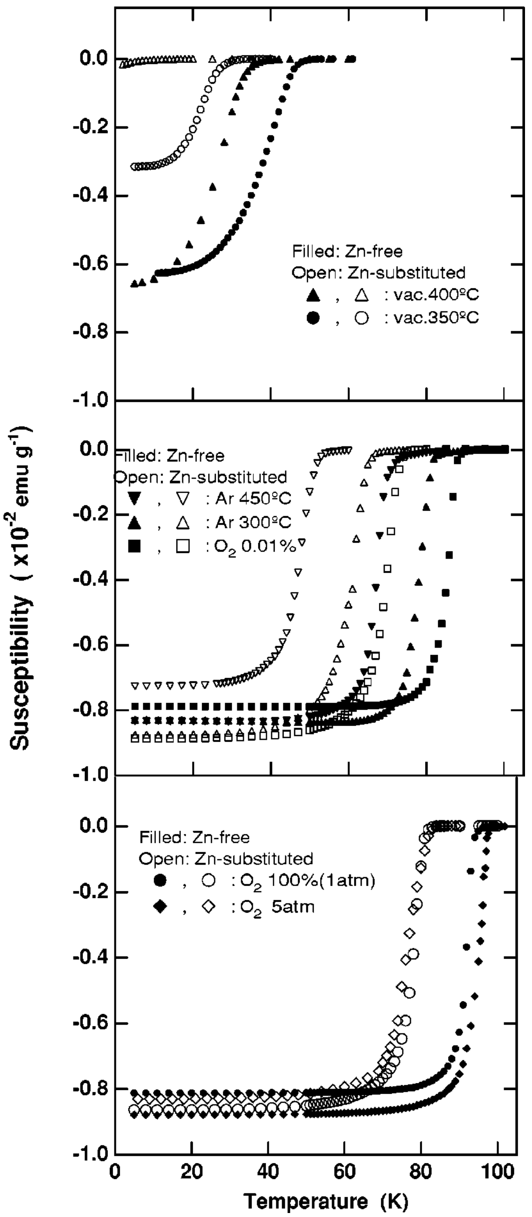

FIG. 1. Temperature dependence of dc susceptibility for HgBa$_2$Cu$_{1-x}$Zn$_x$O$_{4+\delta}$ ($x=0$ and 0.03) ($x=0$ and 0.03) annealed under various conditions.

Zn-substituted samples is of great importance to discuss the doping dependence of the Zn-substitution effects. However, it is very difficult to evaluate p directly. In this work, we estimate p by two different approaches. One is based on the empirical relation between S and p,[31,32] and the other is chemical analysis of copper valence. The hole concentrations estimated by the two methods are in good agreement with each other, as listed in Table I. There was no significant change in p with Zn substitution for the samples annealed in the same condition.

III. RESULTS AND DISCUSSION

A. Dependence of T_c on hole concentration

Figure 1 shows temperature dependence of dc susceptibility for pure and Zn-substituted Hg-1201 annealed in various conditions that are listed in Table I. The large ($\approx 80\%$) superconducting volume fractions and sharp transitions indicated a homogeneous distribution of metals and oxygen in our samples. T_c values were determined from temperatures at which a negative signal starts to be detected, as summarized in Table I. T_c of the Zn-substituted sample is lower than that of the unsubstituted one that was annealed under the same conditions. T_c was depressed about 7 K per 1% Zn substitution, which is smaller than those of La-214 (12–13 K per 1% Zn),[1,29,33] Y-123 (8–9 K),[30,34–36] Y-124 (12 K),[10,37] when we assumed that Zn is only located in the CuO$_2$ layer. The weaker depression in Hg-1201 is an interesting issue. As one of the possible origins, we can consider the characteristic crystal structure of Hg-1201, namely, the long distance between the Cu and the apical oxygen [2.790(2) Å (Ref. 21)] that could relax a lattice distortion introduced by Zn substitution. Although the bond distance of Cu-O (apical) has not been directly measured for the Zn-substituted sample, the lattice parameter c that reflects the change in Cu-O (apical) distance is almost constant with Zn substitution. This is in sharp contrast to the reduction in c for La-214.[29] If T_c is sensitive to the lattice distortion, particularly to the distortion of CuO$_6$ octahedra or the buckling of the CuO$_2$ planes,[38] the distortion relaxation mechanism may weaken the pair breaking effect.[39] This suggests an additional pair breaking in the distorted lattice system such as La-214.[40]

Figure 2(a) displays dependence of T_c on hole concentration for pure and Zn-substituted samples together with our previous data.[22,41] The concentration was estimated from the empirical relation between S at 290 K and p.[32] The difference in T_c between pure and substituted samples became larger with decreasing the hole concentration p, as listed in Table I. T_c is still high ($=35$ K) at $p \approx 0.05$ in the pure Hg-1201, while it is nearly zero for the Zn-substituted sample with the same hole concentration. The tendency that T_c suppression is stronger at lower hole concentration is consistent with the results in La-214 (Ref. 33) and Y-123.[36,42]

Next, looking at the data of Fig. 2(a), we can observe a weak plateau in T_c curve as a function of p at around $p \approx 0.1$, as pointed out in our previous paper.[22] This reminds us of the 60-K plateau in the T_c-p curve for YBa$_2$Cu$_3$O$_y$,[43] which was interpreted as an indication of the change in charge transfer from the CuO chain to the CuO$_2$ plane. Though Cao *et al.* did not find the plateau in Hg-1201,[44] we considered that it is due to a rough step of doping level in their experiment. Recently, Koike proposed a different interpretation of this feature in terms of the "1/8 anomaly" associated with the spin/charge stripe order.[3–5] If Zn substitution is effective for pinning of the fluctuating stripes, as observed in Bi-2212 with (Ca,Y) solid solution,[4] it is of interest whether a similar effect can be observed in Hg-1201. To study a detailed doping dependence of T_c versus hole concentration, we annealed both Zn-free and Zn-substituted samples in Ar gas at temperatures between 280 and 450 °C with narrower intervals than the case of the samples in Table I. Using thermogravimetric analysis we confirmed that the weight of the sample, namely, oxygen content, continuously change with annealing temperature. The T_c values of the samples as a function of

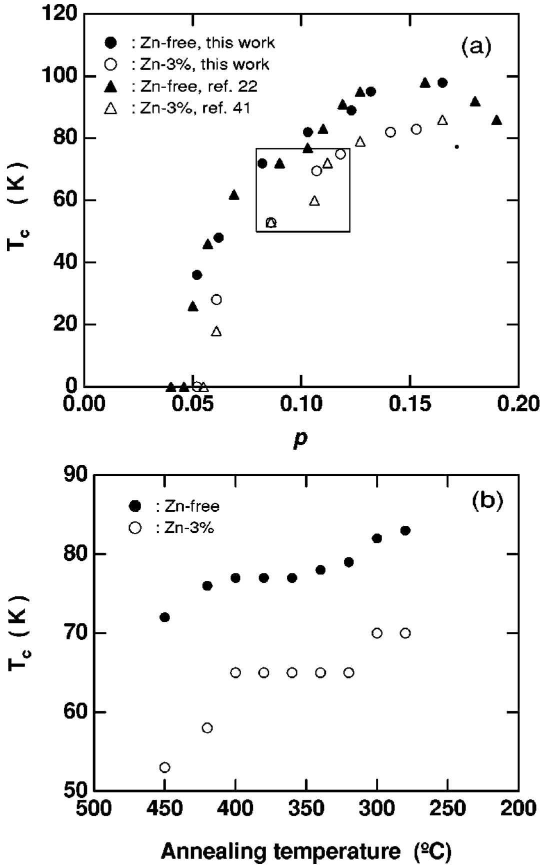

FIG. 2. (a) Superconducting critical temperature T_c of $HgBa_2Cu_{1-x}Zn_xO_{4+\delta}$ ($x=0$ and 0.03) as a function of hole concentration. An area of rectangle corresponds to the figure (b). (b) Variation of T_c for $HgBa_2Cu_{1-x}Zn_xO_{4+\delta}$ ($x=0$ and 0.03) samples annealed at 280–450 °C in a flowing Ar gas. Hole concentrations of these samples are 0.082–0.103 and 0.086–0.107 for pure and Zn-substituted samples, respectively.

annealing temperature are plotted in Fig. 2(b). A clear plateau feature is observed for both pure and Zn-substituted samples between the samples (c) and (d), namely, we can see a region where T_c is insensitive to the hole concentration that correlated to the annealing temperature. However, no remarkable drop of T_c like the 1/8-anomaly appeared by Zn substitution. Therefore we conclude that the plateau exists in a certain doping range of the T_c curve for Hg-1201, as seen in Y-123,[43] but Zn-induced drop of T_c due to the stripe pinning is not observed in this system, as is similar to the case of Y-123.[5]

B. Zn-doping effects on transport properties

Thermoelectric power (TEP) is a useful tool to study the transport properties of polycrystalline HTSC's,[36] because it is not sensitive to the grain-boundary condition. In our previous paper,[22] we demonstrated that the TEP of well sintered polycrystalline Hg-1201 gives almost comparable value for the in-plane (parallel to CuO_2 layer) TEP. Figure 3(a) shows temperature dependence of the TEP for the pure and Zn-substituted samples. Seebeck coefficient S increases with decreasing the carrier doping level in both series of samples.

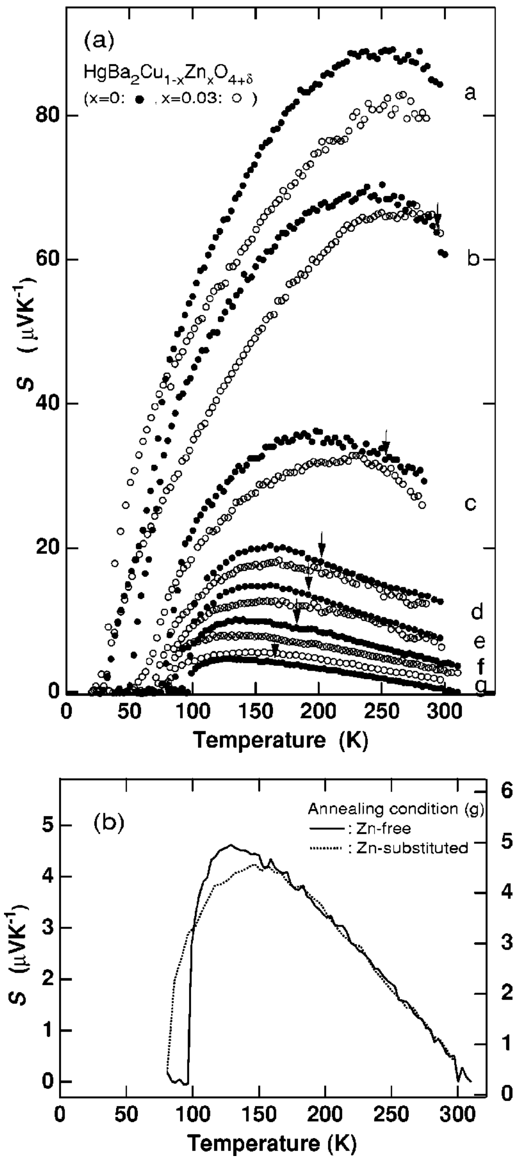

FIG. 3. (a) The temperature dependence of thermoelectric power of $HgBa_2Cu_{1-x}Zn_xO_{4+\delta}$ ($x=0$ and 0.03) annealed under various conditions listed in Table I. The arrows indicate pseudogap temperatures T^* which were estimated from the scaling in Fig. 4 (see text). (b) Thermoelectric power of optimally doped samples [annealing condition (g)]. The vertical axis for the Zn-substituted sample is slightly shifted downward.

There is no significant change in S at 290 K with Zn substitution, which basically agrees with the TEP in Y-123 reported by Tallon *et al.*[9] An appreciable Zn effect is the suppression of the TEP below $T^\star$ over a wide doping range. In order to demonstrate the Zn-substitution effects clearly, we display in Fig. 3(b) comparison of the $S(T)$ curve for the Zn-free and substituted sample labeled as "g." S is clearly suppressed below the temperature indicated by an arrow. The changes in $S(T)$ of Hg-1201 with Zn substitution are consistent with those of Y-123 and Y-124, which were reported by Tallon *et al.*,[9] but is quite different from that of La-214,[13] in spite of the structural similarity between Hg-1201 and La-214. This confirms that La-214 cannot be regarded as a stan-

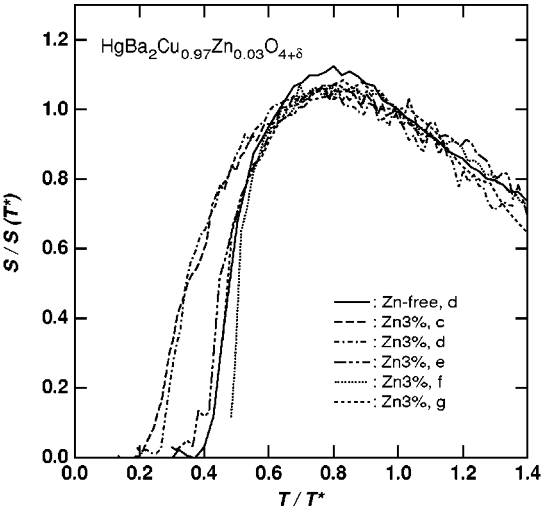

FIG. 4. Scaling curves of thermoelectric power for Zn-substituted Hg-1201 samples. A scaling curve for the unsubstituted samples is plotted by a solid line as a comparison.

dard HTSC with a single CuO_2 layer in the study of physical properties.

Recently, the successful scaling behavior of TEP on the basis of $T^\star$ was demonstrated for Bi-2212.[23,24] This scaling method is convenient to determine the value of $T^\star$ from TEP data. In our recent study,[22] we found that the scaling of the $S(T)$ curves was also successful in Hg-1201, which enabled us to estimate the pseudogap temperatures at various doping levels. We should note that the pseudogap temperature labeled as $T^\star$ here is the characteristic temperature associated with the so-called lower pseudogap temperature at which anomalies begin to be observed in the NMR relaxation rate (T_1^{-1}),[14] the in-plane resistivity,[1,7] the tunneling spectrum,[45] and angle-resolved photoemission spectrum.[46] Another pseudogap temperature that is much higher than $T^\star$ is observed in, for example, the NMR Knight shift[42,47] and the Hall coefficient[48] is discussed in terms of the enhancement of antiferromagnetic correlation.

Applying the same scaling method to the present data in Fig. 3, we found that all the curves are collapsed to a single curve at $T > 0.7T^\star$, as shown in Fig. 4. $T^\star$ estimated from this analysis is indicated by an arrow in Fig. 3(a). It is noted that the universal curve for the 3% Zn-substitution series is not identical to that for unsubstituted Hg-1201. The latter is plotted as a solid curve in Fig. 4. As is seen in Fig. 3, S is reduced with Zn substitution below $T^\star$. This suggests that Zn substitution has some influence on the pseudogapped electronic state, although $T^\star$ does not change with Zn substitution.

One of the possible ways to understand these two facts, namely, the Zn-insensitive behavior of $T^\star$ and the Zn-induced change in the $S(T)$ curve below $T^\star$, is to assume that the substituted Zn modifies the electronic state only locally, as was reported in the NMR,[49,50] NQR,[51] and ESR (Ref. 52) studies. If the electronic state far from the Zn atom is little affected by Zn, $T^\star$ could remain unchanged. On the other hand, it was reported that the substituted Zn induces staggered magnetic moment in a relatively large area around the Zn atom,[53] which may result in weakening of a spin-gap effect and thus change the transport properties at low temperature.

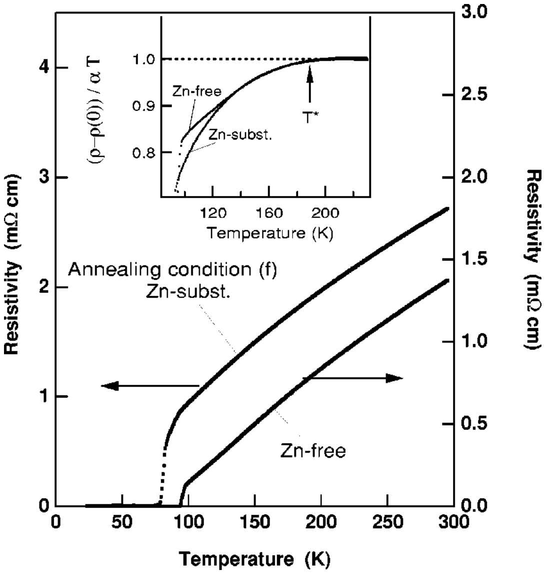

FIG. 5. Temperature dependence of electrical resistivity of slightly underdoped samples $HgBa_2Cu_{1-x}Zn_xO_{4+\delta}$ [$x = 0$ and 0.03, annealed in the condition (f)]. The inset shows dependence of $[\rho - \rho(0)]/\alpha T$ on temperature. Here, α is the slope obtained by a a straight line fit to the data at high temperatures, and $\rho(0)$ is a residual resistivity at 0 K that was estimated from extrapolation of the straight line.

As another transport property, we examined the Zn-substitution effect on resistivity. Figure 5 shows a comparison of $\rho(T)$ curve for slightly underdoped samples with and without Zn. The increase in ρ can be mainly explained by an increase of residual resistivity due to introduced scattering centers.[7] Then, $\rho(T)$ curve was analyzed in the same way as that applied for estimation of $T^\star$ in Zn-substituted Y-123,[7] namely, by subtracting residual resistivity ρ_0 and normalizing by the T-liner term with a slope of α. The result is shown in the inset of Fig. 5. In both pure and Zn-substituted samples, the temperatures at which $\rho(T)$ deviates from the T-linear behavior are almost the same, which suggests no appreciable change in $T^\star$ with Zn substitution. This is consistent with the results for the single crystals and the oriented films of Zn-substituted Y-123.[7,54] The same analysis was performed for the samples with other doping levels and it was found that there is no remarkable Zn effect on $T^\star$.

C. Change in electronic phase diagram with Zn substitution

In Fig. 6, we summarized $T^\star$ and T_c as a function of hole concentration. In this electronic phase diagram, one can see no significant change in $T^\star$ with Zn substitutions while a systematic decrease is found in T_c. From analyses of in-plane resistivity,[7] TEP,[9] and Hall coefficient R_H,[8] it is reported that in case of Y-123 $T^\star$ does not change with Zn substitution in spite of a considerable change in T_c. The different effects of Zn substitution on $T^\star$ and T_c imply that the superconductivity is influenced in a long range by impu-

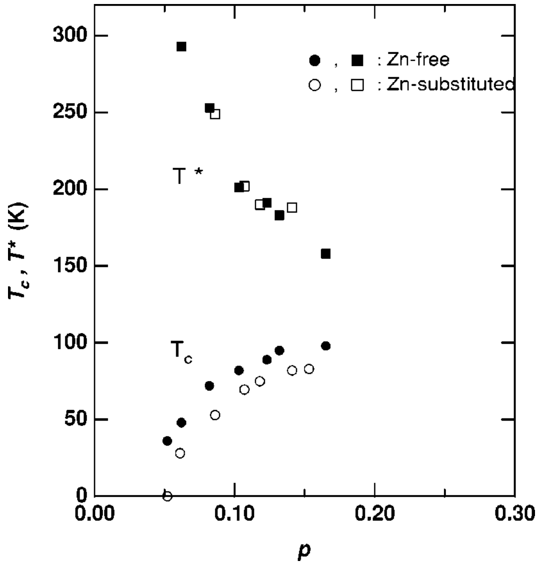

FIG. 6. Electronic phase diagram of pure and Zn-substituted Hg-1201. T_c was determined using dc susceptibility, and T^* was estimated from the scaling of thermoelectric power. Hole concentration p was determined by an empirical relation between S at 290 K and p.

rities even if suppression of the order parameter is pronounced only near the impurities. In contrast, the pseudogap is affected by Zn substitution only locally around Zn atoms, but not far from Zn. This might be related to the lack of phase coherence in the pseudogap state. Although the origin of pseudogap is not clear yet, these results indicate that the observed anomalies below $T^\star$ in the transport properties such as ρ, S, and R_H have the same origin.

Another interesting point is whether the critical hole concentration[36] (p for $T^\star$ reaching 0 K) is common to HTSC's or not. We cannot argue that the critical point is the same in Hg-1201 as that in Y-123, since there are very little data for the overdoped regime in the present work. As is clearly demonstrated in Fig. 6 and reported in our previous paper,[22] $T^\star$ is far above T_c even at the optimal doping level, which makes it unlikely that $T^\star$ goes to zero at $p=0.19$. In spite of such a quantitative difference between our results and the data in Ref. 36, it is highly possible that a stronger T_c depression by Zn at a lower doping level is due to the reduction of density of state resulted from the pseudogap, as proposed in a previous study.[36]

IV. SUMMARY

Zn-substitution effects on T_c and transport properties of $HgBa_2Cu_{1-x}Zn_xO_{4+\delta}$ (Hg-1201, $x=0$ and 0.03) in underdoping and optimal doping states were investigated. T_c decreased with Zn substitution without changing the hole concentration. The decease in T_c by the Zn substitution became larger as the doping level was lowered. No dramatic T_c drop was observed near $p=1/8$ both in pure and Zn-substituted samples, while the region where T_c is insensitive to the hole concentration exists. This implies that there is no clear Zn-induced pinning of the stripes in Hg-1201, which is different from the case of La-214 but similar to Y-123. From the analysis of $S(T)$ using the scaling technique, it was demonstrated that no remarkable change in $T^\star$ is induced by Zn substitution over a wide doping range from under- to optimal-doping states. In contrast to this Zn-insensitive behavior of $T^\star$, the value of S is suppressed below $T^\star$ when Zn is introduced into the CuO_2 plane. A possible interpretation is that the electronic state is locally modified only around Zn, not affected at the sites far from Zn.

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