Flux-pinning behavior and the interlayer coupling of the $Hg_{0.7}Cr_{0.3}Sr_2CuO_{4+\delta}$ superconductor

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Previously the compound of $Hg_{0.7}Cr_{0.3}Sr_2CuO_{4+\delta}$ was believed to exhibit an enhanced flux-pinning behavior compared to the pristine $HgBa_2CuO_{4+\delta}$. As candidates for its origin, possible extended defects of a superlattice in the c direction or a strong coupling deduced from a reduced distance between CuO_2 planes have been proposed. To clarify these, we extensively measured the magnetization of the c-axis aligned grains of this compound. Contrary to previous belief, the irreversibility line and magnetic hysteresis show reduced pinning properties. From the analysis of the reversible magnetizations based on the vortex fluctuation model, the high-field scaling analysis, and the Hao-Clem model, the interlayer coupling was also confirmed to be weak. The proposed extended defects seem not to be effective in pinning vortices in $Hg_{0.7}Cr_{0.3}Sr_2CuO_{4+\delta}$.
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I. INTRODUCTION

The $Hg_{0.7}Cr_{0.3}Sr_2CuO_{4+\delta}$ (nominal composition) superconductor [Hg/Cr(Sr)-1201] has attracted much attention due to the possible enhancement of flux pinning by chemical substitution.[1,2] Shimoyama *et al.*[1] reported that they observed a large difference between the zero-field-cooled (ZFC) and field-cooled (FC) magnetizations in this material in a low external field. They inferred that the pinning was enhanced due to the strong coupling between CuO_2 planes, which was deduced from a shorter interlayer distance than that of the pristine $HgBa_2CuO_{4+\delta}$ [Hg(Ba)-1201].[2] However, in the hysteresis in low fields, it is difficult to discern the intrinsic pinning properties from others such as the intergrain coupling effect and the surface barrier effect.[3] The pinning properties of this compound need to be examined in high-field measurements.

Another suggestion of the enhanced flux pinning comes from the electron and the neutron diffraction measurement by Chmaissem *et al.*[4] According to their report, the Hg/Cr(Sr)-1201 has a structure in which the Cr- and Hg-rich regions are ordered in all three crystallographic directions with a supercell of the approximate dimension $19.2\times19.2\times17.4$ Å^3. For example, the two regions alternate in the c direction. This supercell structure is the peculiar feature of the Hg/Cr(Sr)-1201, which is not shown in similar compounds $Hg_{1-x}Cr_xBa_2CuO_{4+\delta}$.[5] The Cr- and Hg-rich regions are not likely to be good pinning centers since they are smaller than the typical size of a vortex core and are ordered regularly. Chmaissem *et al.* speculated that the defects in the superstructure might enhance flux pinning possibly by forming columns of Hg-containing cells along the c axis like columnar defects[6,7] generated by ion radiation. However, no direct evidence of the enhanced flux pinning was observed in their structural studies.

In this paper, we extensively study the interlayer coupling between CuO_2 planes and the effects of the proposed extended defects on flux pinning by magnetization measurement. For this purpose, we prepared a c-axis grain-aligned sample of Hg/Cr(Sr)-1201, because these extended defects are claimed to be oriented in the c direction.

In Sec. III A, the irreversibility line and the hysteresis of this sample are presented in comparison with those of the pristine Hg(Ba)-1201. In Sec. III B, the reversible magnetizations are analyzed with the vortex fluctuation model,[8] the high-field scaling analysis,[9] and the Hao-Clem model,[10] which reveal the strength of the interlayer coupling. In contrast to the previous conjecture, our result shows that the flux pinning and the interlayer coupling of the Hg/Cr(Sr)-1201 are rather weaker than in the pristine superconductor.

II. EXPERIMENTS

The sample was synthesized by the solid-state reaction of the precursor and HgO.[11,12] First, an aqueous solution of stoichiometric amounts of $Sr(NO_3)_2$ and $Cu(NO_3)_2.3H_2O$ was

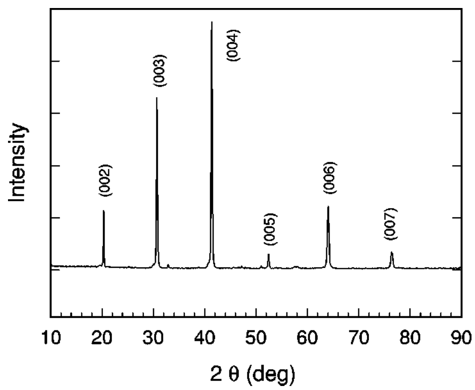

FIG. 1. X-ray-diffraction pattern for grain-aligned Hg/Cr(Sr)-1201.

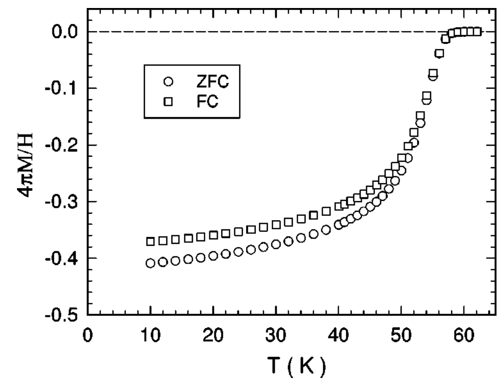

FIG. 2. Temperature dependence of the dc susceptibility $4\pi M/H$ in the zero-field-cooled (ZFC) and field-cooled (FC) condition for applied field $H=6$ Oe.

dried and heated slowly to 900 °C. An appropriate amount of Cr_2O_3 was then added and fired at 900 °C for 24 h. The obtained precursor is of nominal composition of $Cr_{0.3}Sr_2CuO_y$. HgO powder was mixed with powder of the black precursor in a nitrogen-filled dry box. Then they were pressed into pellets, sealed in an evacuated quartz tube, sintered at 880 °C for 8 h, and finally quenched to room temperature. The as-prepared samples were oxygen-annealed at 300 °C for 1 h and cooled slowly to room temperature.[12]

The structure and composition were determined by x-ray diffraction (XRD) and neutron diffraction.[11] Like Hg(Ba)-1201, the sample contains a tetragonal structure and a single CuO_2 plane per unit cell. The obtained cell parameters [$a=3.845(2)$ Å, $c=8.715(6)$ Å] are smaller than those of Hg(Ba)-1201 ($a=3.88$ Å, $c=9.51$ Å),[13] especially by 8.3 % in the c direction. Chromium was found to replace Hg partially at the (0.0, 0.0, 0.0) site with the ratio of occupancy Hg:Cr = 53:47. The structure determined by neutron diffraction is consistent with previous works on similar samples.[4]

For the magnetic measurement, the bulk sample was ground into small grains of less than 20 μm and aligned in epoxy with an external field of 7 T. The resultant epoxy binder contained the sample of 18.6 mg at the volume density of 4%. Excellent alignment was confirmed by the XRD as shown in Fig. 1. Using a Quantum Design superconducting quantum interference device magnetometer, we measured the magnetic hysteresis, and the temperature dependence of magnetization under ZFC and FC condition, applying external fields of various strength up to 4 T. For analysis, the background contribution from epoxy and impurities was subtracted from the observed values.

Figure 2 shows the temperature dependence of susceptibility in the c-axis direction. From the diamagnetic signal, superconducting transition occurs at 58 K. The superconducting volume fraction was evaluated as about 41% from the low-temperature data, without including the geometric factors.

III. RESULTS AND DISCUSSIONS

A. Flux-pinning behaviors

The flux-pinning behavior of a superconductor, which is important in application, can be well characterized by means of an irreversibility line and a magnetic hysteresis. The irreversibility line $H_{irr}(T_{irr})$ is a boundary which divides the mixed-state phase diagram into two regions, a reversible and an irreversible magnetization region. Explanations of this line have been suggested in terms of the conventional flux creep,[14,15] the vortex lattice melting,[16,17] or the vortex glass transition.[18]

In the irreversible magnetization region, spatial inhomogeneities or defects pin and prevent vortices from moving freely. This is the origin of magnetic hysteresis. In this region, there exists a nonzero depinning critical current J_c below which the superconductor can sustain supercurrents without dissipation. The J_c, which is often used to quantify the pinning strength, can be determined from the magnitude of magnetic hysteresis. The enhancement of the $H_{irr}(T_{irr})$ and the J_c, i.e., the wider irreversible region of zero linear resistance at larger currents, can be accomplished by introducing point defects, or extended defects such as radiation-induced columnar defects.[6,7]

For the reversible magnetization region, the vortices move freely because their thermal fluctuations dominate over the pinning. For the high-temperature superconductors, the high transition temperature and the small interlayer coupling can make this reversible region wider, and thus suppress the irreversibility line.

The irreversibility line of the Hg/Cr(Sr)-1201 in a reduced temperature scale compared with that of the pristine material is shown in Fig. 3. The irreversibility temperature T_{irr} was

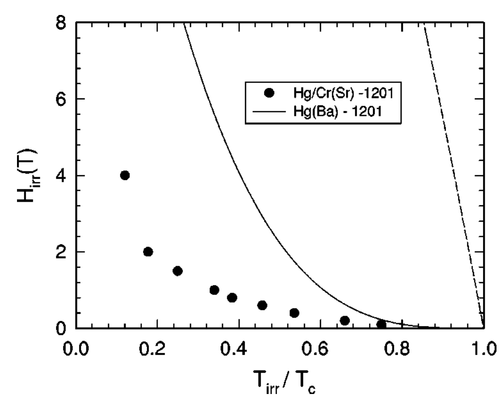

FIG. 3. Irreversibility line of Hg/Cr(Sr)-1201 and Hg(Ba)-1201. The dashed line represents the mean-field upper critical field $H_{c2}(T)$ of Hg/Cr(Sr)-1201.

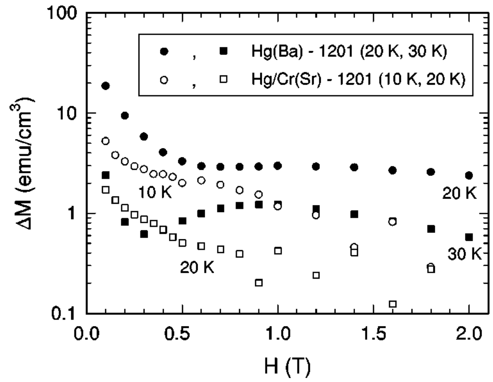

FIG. 4. Magnetic hysteresis ΔM for Hg/Cr(Sr)-1201 and Hg(Ba)-1201.

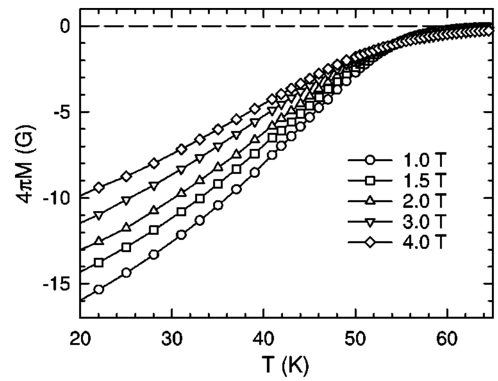

FIG. 5. Temperature dependence of the reversible magnetizations of Hg/Cr(Sr)-1201 for various strengths of magnetic fields.

determined from the merging point of the ZFC and the FC magnetizations, M_{ZFC} and M_{FC}, with a criterion of the $M_{\mathrm{ZFC}}-M_{\mathrm{FC}}=0.01$ emu/cm^3.

The mean-field upper critical field H_{c2} (dashed line) obtained from the Hao-Clem model (see the following section) is presented for comparison. The solid line in the figure represents the irreversibility line of the Hg(Ba)-1201 from Ref. 19. The magnetization of this pristine material was also obtained in the same grain-aligned configuration. This figure shows that the irreversibility line is not improved via the chromium and strontium substitution into the Hg(Ba)-1201. On the contrary, this line is much suppressed from that of the pristine material.

The weak pinning property is observed also in the hysteresis of magnetization. Figure 4 shows the magnetization hysteresis ΔM of the Hg/Cr(Sr)-1201 and the pristine material. This ΔM is the difference of magnetizations measured in increasing and decreasing fields. The data of the pristine Hg(Ba)-1201 is from Welp *et al.*[20] The ΔM of the pristine material was also measured on a grain-aligned sample. For comparison of data sets at similar T/T_c, the ΔM at 10, 20 K ($T/T_c=0.17$, $T/T_c=0.35$) of the Hg/Cr(Sr)-1201, and the ΔM at 20, 30 K ($T/T_c=0.21$, $T/T_c=0.32$) of the Hg(Ba)-1201 are presented. The values were normalized by the superconducting volume fraction of each sample, which was 100% for the pristine one. We note that the ΔM of the Hg/Cr(Sr)-1201 is much smaller than that of the pristine material.

It is hard to compare the pinning strength of the above two samples directly since the critical current J_c is determined not only from the hysteresis ΔM, but also from the size D of the supercurrent loop. For example, according to the Bean's critical-state model, J_c is 20 $\Delta M/D$. We assume that the supercurrent is confined in each grain, and that the size of its loop is equal to the granular size. The size of the Hg/Cr(Sr)-1201 grains is estimated to be 5 $\sim$ 20 μm from the SEM image. The fineness of the sieve used for grain alignment was 20 μm. This granular size is comparable to that ($\sim$ 10 μm) of the pristine sample.[20] Therefore, the pinning strength or J_c of the Hg/Cr(Sr)-1201 is also believed to be much lower than that of the Hg(Ba)-1201.

These reduced pinning behaviors are contrary to the previous speculations based on the reduced interlayer distance[1,2] or the proposed extended defects.[4] The intrinsic columnar defects proposed by Chmaissem *et al.* may not be pinning centers strong enough to enhance the pinning behavior of the Hg/Cr(Sr)-1201.

B. Interlayer coupling

To find the interlayer coupling strength of the Hg/Cr(Sr)-1201, we analyzed the reversible magnetizations. Depending on the coupling strength, the magnetization can be described better by considering the superconductors either as a stack of two-dimensional (2D) superconducting layers weakly linked to each other, or as the anisotropic 3D superconductors. For this study, the vortex fluctuation model,[8] the high-field scaling analysis,[9] and Hao-Clem model[10] were utilized.

Near the transition temperature $T_c(H)$, positional fluctuation of vortices is severe and deviates the magnetization from the mean-field one. In 2D-like superconductors such as $Bi_2Sr_2CaCu_2O_{8+\delta}$ (Bi2212), this vortex fluctuation effect is significant down to relatively low temperatures,[21] while it appears only near T_c in 3D superconductors.[22,23] For the 2D-like case, the resultant magnetization is well described by the vortex-fluctuation model by Bulaevskii *et al.*,[8] which considers the Josephson-coupled layered structure.

The contribution of the vortex-fluctuation effects can be studied in two ways. One way is to analyze the magnetization based on the vortex-fluctuation model. The other is from the analysis based on the Hao-Clem model. As a mean-field model, the Hao-Clem model does not consider the fluctuation effects. Therefore, in the fluctuation-dominant region, its application results in the unreasonable deviation of the Ginzburg-Landau (GL) parameter κ from the expected values. For 2D-like superconductors, the κ is observed to increase abruptly far below the T_c instead of decreasing slowly as T increases.

In the critical fluctuation region around $T_c(H)$, magnetizations for various high magnetic fields are scaled into a single curve for an appropriate variable depending on dimensionality. For 2D superconductors, good scaling behavior is observed for the variable $[T-T_c(H)]/(TH)^{1/2}$.[24,25]

In Fig. 5, the temperature dependence of reversible magnetization is displayed for the field between 1 and 4 T parallel to the c axis. A prominent feature is that the magnetization curves for different external fields are crossing at $T^*=54.5$ K with a value of $M^*=-0.165$ G. This is typical for the highly anisotropic layered superconductors with strong fluctuation effect.

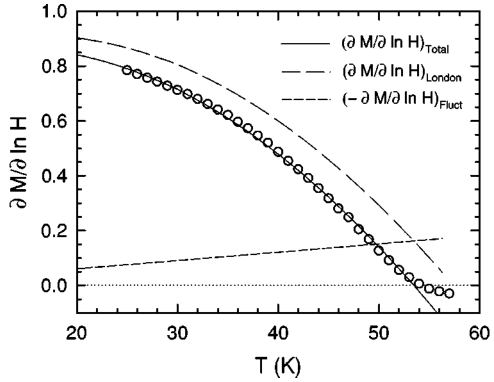

FIG. 6. Temperature dependence of the slope $\partial M/\partial(\ln H)$ of Hg/Cr(Sr)-1201. The solid line represents the theoretical curve derived from the vortex-fluctuation model, and the dashed lines are from the London and the fluctuation contribution, respectively.

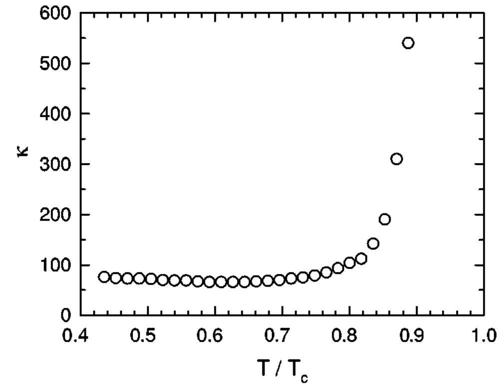

FIG. 7. Temperature dependence of the Ginzburg-Landau parameter $\kappa(T)$ determined from the Hao-Clem model.

For highly anisotropic layered superconductors, vortex lines are described by weakly coupled quasi-2D vortices, and the coherence in the positions of these pancakelike vortices along the c direction is easily lost due to thermal fluctuation. The vortex-fluctuation model by Bulaevskii *et al.*[8] considers this contribution in addition to the mean-field magnetization in a system of Josephson-coupled layers.

The vortex-fluctuation effect can be analyzed quantitatively by comparison of the derivative $\partial M/\partial \ln H$ from the experiment (open circle) and from the vortex-fluctuation model (solid line), which is given as

$$\left(\frac{\partial M}{\partial \ln H}\right)_{\text{Total}} = \left(\frac{\partial M}{\partial \ln H}\right)_{\text{London}} + \left(\frac{\partial M}{\partial \ln H}\right)_{\text{Fluct}}, \tag{1}$$

$$\left(\frac{\partial M}{\partial \ln H}\right)_{\text{London}} = \frac{\phi_0}{32\pi^2 \lambda_{ab}^2(T)}, \tag{2}$$

$$\left(\frac{\partial M}{\partial \ln H}\right)_{\text{Fluct}} = -\frac{T}{\phi_0 s}, \tag{3}$$

as shown in Fig. 6. In the comparison, the adjustable parameters are the zero-temperature penetration depth $\lambda_{ab}(0)$ and the interlayer distance s. In this case, $\lambda_{ab}(0) = 2.67\times 10^3$ Å and $s = 22$ Å as estimated in consideration of superconducting volume fraction. The data in the entire reversible region above 25 K(0.44 T_c) agree well with the theory.

Two terms, the mean-field (London) term $(\partial M/\partial \ln H)_{\text{London}}$ and the vortex-fluctuation term $(\partial M/\partial \ln H)_{\text{Fluct}}$, are also plotted. As they show, the fluctuation contribution is not negligible for the entire range of Fig. 6. For example, the fluctuation term at 27 K(0.47 T_c) is about 10% of the London term. It verifies that the Hg/Cr(Sr)-1201 is 2D-like since the fluctuation effect remains significant in the wide range of temperature.

The s value obtained here is somewhat larger than the one determined from the XRD. This deviation was commonly observed in other 2D-like systems, and its reconciliation was tried by correcting the superconducting volume fraction[26,27] or by using a theoretical result by Koshelev.[28,29]. In the fluctuation magnetization model for 2D superconductors,[28] Koshelev considered the Gaussian fluctuation from all the Landau levels to estimate the magnetization at the crossing point as $M^* = -0.346\times k_B T^*/\phi_0 s$. For the values of M^* and T^* from our data, the s is estimated to be 7.6 Å comparable with the s from the XRD.

As stated before, the strength of interlayer coupling can be studied by identifying the fluctuation-dominant region. One way to do this is to study the magnetization in terms of the Hao-Clem model. The Hao-Clem model[10] describes the field dependence of the mean-field magnetization $M(H)$ in the entire region of mixed state. Hao and Clem, in their model, calculated it by minimizing the GL free energy of the vortex lattice system variationally. Important superconducting parameters such as the GL parameter κ and the thermodynamic critical field $H_c(T)$ can be obtained by comparison of the reversible magnetization with the theoretical curve $M(H)$.

Since the Hao-Clem model does not consider the fluctuation effect, its application to the region where the fluctuation prevails gives the obtained parameters deviating from reasonable values. In this analysis, this deviation was used as a tool to find the fluctuation-dominant region.

The parameter $\kappa(T) = \lambda/\xi$ is known to be a slowly decreasing function of temperature.[30] However, Fig. 7 shows that $\kappa(T)$ increases rapidly, starting from $T \simeq 0.7T_c$. This anomalous behavior can be understood as due to fluctuation effects. The wide fluctuation-dominant region indicates that the interlayer coupling of the Hg/Cr(Sr)-1201 is very weak. This is in great contrast to the case of 3D superconductors where $\kappa(T)$ starts to increase very near T_c.[22,23]

From the analysis based on the Hao-Clem model outside the fluctuation-dominant region, several superconducting parameters such as $\kappa = 72$, $T_c = 57.5$ K, $H_c(0) = 3.19\times 10^3$ Oe, $H_{c2}/dT|_{T_c} = -1.14$ T/K, and $\lambda_{ab}(0) = 2.35\times 10^3$ Å (dirty limit) were obtained.

The weak interlayer coupling of the Hg/Cr(Sr)-1201 is also confirmed through the high-field scaling analysis suggested by Ullah and Dorsey.[9] According to them, the magnetization in the critical fluctuation region follows the scaling formula

$$\frac{M(T,H)}{(TH)^{1-1/d}} = F_d\left(A_d \frac{T - T_c(H)}{(TH)^{1-1/d}}\right). \tag{4}$$

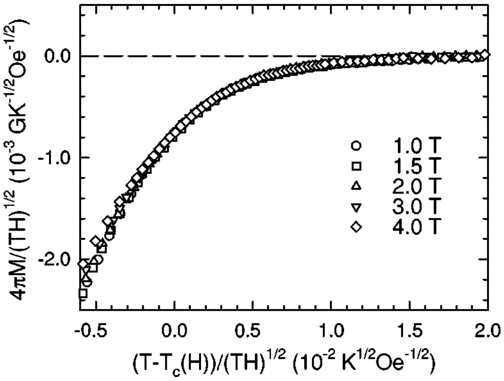

FIG. 8. 2D scaling of magnetizations of Hg/Cr(Sr)-1201 using the analysis suggested by Ullah and Dorsey.

Here, d is the dimension, and A_d is $[\phi_0 s H_{c2}(0)/16\pi\kappa k_B T_c^2]^{1/2}$ in 2D and $[\phi_0 \xi_c H_{c2}^2(0)/8\pi\kappa k_B T_c^{3/2}]^{2/3}$ in 3D.

As shown in Fig. 8, all the data of diverse external fields collapse clearly on one curve in 2D scaling form. As for the parameters T_c and $dH_{c2}/dT|_{T_c}$ needed in scaling, we adopted the values obtained from the analysis of the Hao-Clem model. The 3D scaling was also attempted, but was poor for any adjustment of the parameters.

The recent Raman study[31] on the Hg/Cr(Sr)-1201 also supports the weak interlayer coupling. The peak due to the apical oxygen in the Hg-O_A bonds is observed to be at a lower frequency than that of the pristine Hg(Ba)-1201. This was interpreted as being due to weaker layer-layer coupling in the Hg/Cr(Sr)-1201 samples than that in the pristine material. Besides, the Raman results indicate the existence of the supercell composed of alternating Hg-rich and Cr-rich phases.

One possible origin of the weak coupling in spite of short interlayer distance may be the reduced carrier density in CuO_2 planes from the depletion of interstitial oxygen O(4) at the (0.5, 0.5, 0) site in the (Hg,Cr)-O plane by Cr substitution. According to the neutron-diffraction study by Chmaissem *et al.*,[4,5] Cr has the valence state of +6, and is incorporated in the form of a $(CrO_4)^{2-}$ tetrahedron, replacing $(HgO_2)^{2-}$ dumbbell in the Hg(Ba)-1201 structure. The additional two oxygen atoms O(3) bonded to Cr are at (0.628, 0.628, 0.0) in the (Hg, Cr) plane, blocking O(4) atoms at (0.5, 0.5, 0.0). However, they do not contribute to the hole doping to CuO_2 planes, while the O(4) atoms do. The hole-doping mechanism by O(4) atoms and the inverted relation between the Cr content and the O(4) content were verified recently in the structural study of the $Hg_{1-x}Cr_xBa_2CuO_{4+\delta}$ with variation of x.[5] This result can be applied to the Hg/Cr(Sr)-1201 case since the local structure and the valence state related to $(CrO_4)^{2-}$ are similar. In the Hg/Cr(Sr)-1201 case, nearly half of the Hg atoms are replaced by Cr atoms, and most O(4) sites are filled by O(3) atoms bonded to Cr, which will significantly decrease the carrier density in CuO_2 planes.[5] The T_c reduction and the weakening of interlayer coupling may be explained at the same time in this scheme.

The reduction of carrier density n is verified by the elongated penetration depth λ in comparison with that of the pristine superconductor, 180 nm,[32] since n is inversely proportional to λ^2.[33] In addition, the recent thermoelectric power (TEP) study[12] on the Hg/Cr(Sr)-1201 gives more direct evidence for the reduced carrier density. Based on the universal dependence of the room-temperature TEP on the hole number p per Cu ion,[34] the p was estimated to be approximately 0.1, which is lower than 0.16 for the maximum T_c of the pristine superconductor.

IV. CONCLUSIONS

We investigated the proposed enhancement of flux pinning and the interlayer coupling in the Hg/Cr(Sr)-1201 through the analysis of various magnetic properties measured on the grain-aligned sample. As compared with the pristine Hg(Ba)-1201, the Hg/Cr(Sr)-1201 exhibits weaker pinning behavior. This is confirmed by the wide reversible region in the field versus temperature plane and the narrow magnetic hysteresis, i.e., the low depinning critical current J_c. The proposed columnarlike defects[4] do not seem to serve as strong pinning centers for vortex lines.

This weak pinning is closely related to the weak interlayer coupling of the Hg/Cr(Sr)-1201 confirmed by the analysis of the reversible magnetization. The weakened layer-layer coupling, in spite of the short interlayer distance, might originate from the reduced carrier density in CuO_2 planes.

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