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Anisotropy of superconducting $Pb_2Sr_2(Y_{0.7}Ca_{0.3})Cu_3O_{8+\delta}$ and $Pb_2Sr_2Y_{1-y}Cu_3O_{8+\delta}$

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The London-penetration-depth anisotropy ratio γ of $Pb_2Sr_2(Y_{0.7}Ca_{0.3})Cu_3O_{8+\delta}$ and Ca-free $Pb_2Sr_2Y_{1-y}Cu_3O_{8+\delta}$ ($y \approx 0.1$) single crystals has been measured by the equilibrium torque magnetometry method. γ values are found to be in the range of 10 to 11 and are comparable to that of $YBa_2Cu_3O_{7-\delta}$ rather than $Bi_{2+x}Sr_2Ca_{1-x}Cu_2O_{8+\delta}$ although $Pb_2Sr_2(Y_{0.7}Ca_{0.3})Cu_3O_{8+\delta}$ is structurally similar to both, with its c-axis lattice constant 30% larger than that of $YBa_2Cu_3O_{7-\delta}$. The γ of a $Pb_2Sr_2(Sm_{0.5}Eu_{0.5})Cu_3O_{8+\delta}$ crystal was measured to be 13±3.

I. INTRODUCTION

After the discovery of the family of $Pb_2Sr_2ACu_3O_{8+\delta}$ (A represents $R_{1-x}Ca_x$ or R, where R =rare-earth element) layered copper oxides,[1] the so-called Pb-2213 phase continues to be of interest. Extensive structural studies have been carried out with x-ray diffraction,[1-4] neutron diffraction,[5] and electron microscopy.[6-8] Structurally the Pb-2213 phase is similar to both $YBa_2Cu_3O_{7-\delta}$ and $Bi_{2+x}Sr_2Ca_{1-x}Cu_2O_{8+\delta}$. The A layer is sandwiched between two CuO_2 layers with a separation of approximately 1.62 Å. Between sets of the double-pyramidal CuO_2-A-CuO_2 layers, $Pb_2Sr_2ACu_3O_{8+\delta}$ has PbO-CuO_δ-PbO sequenced layers. The double PbO layers are similar to the double BiO layers in $Bi_{2+x}Sr_2Ca_{1-x}Cu_2O_{8+\delta}$ and the CuO_δ layer is analogous to the chain layer of the oxygen-depleted $YBa_2Cu_3O_{7-\delta}$.

The parent compounds $Pb_2Sr_2RCu_3O_{8+\delta}$ are insulators[1,9,10] in which the Cu^{2+} spins in the CuO_2 layers order antiferromagnetically.[9,11] Substitution of the trivalent rare earth by Ca^{2+} provides hole doping in the CuO_2 layers and induces superconductivity. Examples are $Pb_2Sr_2(R_{1-x}Ca_x)Cu_3O_{8+\delta}$ with R =Y, Er, Gd, Dy, Tm, and Sm, and these superconductors can have a T_c higher than 80 K.[12] However, the superconducting volume fractions are usually less than 10%.[13,14] The study of $Pb_2Sr_2(Y_{1-x}Ca_x)Cu_3O_{8+\delta}$ ceramic samples with $0 \leq x \leq 0.8$ and $\delta \sim 0$ shows that the optimal doping condition is at half substitution (x =0.5) resulting in $T_c \approx 83$ K.[15] The Ca-free $Pb_2Sr_2R_{1-y}Cu_3O_{8+\delta}$ with R-site cation vacancies (y=0.1–0.2) are also superconductors for R =Y, Eu, Ho, Dy, etc.[14] The R-site vacancies may function as zero-valence ions and therefore provide hole carriers to the neighboring CuO_2 layers through charge balance.

$Pb_2Sr_2ACu_3O_{8+\delta}$ can incorporate excess oxygen up to $\delta \approx 1.9$ from $\delta \approx 0$.[9] The excess oxygen is located in the CuO_δ plane between the two PbO layers, and oxidizes the Cu^{1+} to Cu^{2+} and the Pb^{2+} to Pb^{4+}. The oxidization process in the PbO-CuO_δ-PbO layers seeks extra hole carriers from the adjacent CuO_2 layer and suppresses the superconductivity.[9,16] Therefore a N_2 annealing process is usually employed to minimize the oxygen content δ in superconducting $Pb_2Sr_2ACu_3O_{8+\delta}$.

The high-T_c superconductors are highly anisotropic due to their layered structures. The anisotropy is usually expressed by a dimensionless parameter γ defined as $\gamma \equiv \lambda_c/\lambda_{ab}$, where λ_c and λ_{ab} are the superconducting penetration lengths for currents along the c axis and in the ab plane, respectively. The equilibrium torque magnetometry method offers an unambiguous way to measure γ directly. This is because an anisotropic superconductor experiences a torque $\tau = M \times H$ in an arbitrarily oriented magnetic field H. The angular dependence of $\tau(\theta)$ gives a direct measure of γ of the superconductor where θ is the angle between the c axis and the applied H.[17-19] This torque method has been used to study the anisotropy of the $YBa_2Cu_3O_{7-\delta}$ compounds[20-24] and the $Bi_{2+x}Sr_2Ca_{1-x}Cu_2O_{8+\delta}$ compounds.[25] $\gamma \approx 8.5$ for $YBa_2Cu_3O_{7-\delta}$ and γ is on the order of 100–200 for $Bi_{2+x}Sr_2Ca_{1-x}Cu_2O_{8+\delta}$.

Because of its similarity in structure to both $YBa_2Cu_3O_{7-\delta}$ and $Bi_{2+x}Sr_2Ca_{1-x}Cu_2O_{8+\delta}$, it is interesting to know whether the γ values of the $Pb_2Sr_2ACu_3O_{8+\delta}$ compounds are close to that of $YBa_2Cu_3O_{7-\delta}$ or of $Bi_{2+x}Sr_2Ca_{1-x}Cu_2O_{8+\delta}$. In this paper, we present anisotropy measurements of $Pb_2Sr_2(Y_{0.7}Ca_{0.3})Cu_3O_{8+\delta}$ and $Pb_2Sr_2Y_{1-y}Cu_3O_{8+\delta}$ ($y \approx 0.1$). Results for $Pb_2Sr_2(Sm_{0.5}Eu_{0.5})Cu_3O_{8+\delta}$ superconducting single crystals are also included. Crystals with Er and Eu substitution rather than Y were also examined. More details are discussed in the following sections.

II. EXPERIMENTS

Superconducting single crystals of $Pb_2Sr_2R_{1-y}Cu_3O_{8+\delta}$ and $Pb_2Sr_2(R_{1-x}Ca_x)Cu_3O_{8+\delta}$ were prepared by a PbO/NaCl flux growth method.[12-14] The as-grown crystals were extracted undamaged from

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the PbO flux by using a methanol solvent in an ultrasonic bath.[13] Typical dimensions of the crystals were $1\times1\times0.3$ mm^3. A N_2 annealing process at 500 °C for 100 h was used to reduce the excess oxygen content. Previous magnetization measurements of these crystals indicated superconducting transition widths of ~5 to more than 20 K and a small superconducting volume fraction ($<10\%$).[13,14] A $Pb_2Sr_2(Sm_{0.5}Eu_{0.5})Cu_3O_{8+\delta}$ crystal was made in a similar way.

The rotating magnetic torque magnetometer described previously[21] was used. The torque on the sample, which caused a small deviation of the detection spring, was approximately proportional to the capacitance change between the spring and a counterelectrode. Each sample in the magnetometer was cooled slowly in zero field to below 100 K for the torque experiment. The torque experiments were performed in a 1.8 T field at a temperature several kelvins below the onset T_c for most samples. During the experiment, the sample probe was rotated 190° in both directions while a computer recorded the capacitance between the spring and the counterelectrode as a function of angle.

The torque signal was very small ($<10^{-9}$ N m) with some samples due to a small volume fraction of superconductivity. It was then necessary to set the temperatures 10–15 K below the T_c to increase $\tau(\theta)$, but the $\tau(\theta)$ was hysteretic due to fluxoid pinning. In this case the average of the curves for both directions of rotation was used for the fitting procedures described below.

III. RESULTS AND DISCUSSION

In the field range $H_{c2}>H\gg H_{c1}$, the angular dependence of the equilibrium torque per unit volume is given by[18]

$$\tau(\theta)=\frac{\phi_0 H}{64\pi^2\lambda^2}\frac{\gamma^2-1}{\gamma^{1/3}}\frac{\sin(2\theta)}{\epsilon(\theta)}\ln\left[\frac{\gamma\eta H_{c2\|}}{H\epsilon(\theta)}\right], \tag{1}$$

where $\epsilon(\theta)=(\sin^2\theta+\gamma^2\cos^2\theta)^{1/2}$, θ is the angle between the applied field H and the c axis, $\gamma\equiv\lambda_c/\lambda_{ab}$, $\lambda=\sqrt[3]{\lambda_c\lambda_{ab}^2}$, $H_{c2\|}$ is the upper critical field along the c axis, and η is a constant of the order of unity which is dependent on the flux lattice structure. Equation (1) is based on the three-dimensional anisotropic London model.

Due to an uncertainty in determining the exact value of θ during experiments, a small angular offset $\Delta\theta$ was left as a fitting parameter. Thus, four fitting parameters were used: $\gamma, \eta H_{c2\|}$, the amplitude, and $\Delta\theta$. Equation (1) has been tested by experiments with $YBa_2Cu_3O_{7-\delta}$,[20,26,27] and with $Bi_{2+x}Sr_2Ca_{1-x}Cu_2O_{8+\delta}$.[28]

Figure 1 shows the normalized $\tau(\theta)$ data for a $Pb_2Sr_2(Y_{0.7}Ca_{0.3})Cu_3O_{8+\delta}$ crystal in the angular range $90°>\theta>0°$. The maximum torque of the sample was of the order of 10^{-8} N m. The solid curve with $\gamma=10.2$ is the least χ^2 fit of Eq. (1) to the data.

The Be-Cu spring was slightly magnetic and gave a sinusoidal background,[21] which needed to be subtracted before data analysis. Because of the weak torque signals from some samples, in general the $\tau(\theta)$ data after background subtraction were slightly asymmetric in the angular range $180°>\theta>0°$. In order to get a reliable γ value, the angular range for the fitting was chosen to be $100°>\theta>80°$ with more than 50 points. Previous experience indicated that the γ values thus obtained are consistent (to within 3–10 %) with those obtained by fitting in the range $180°>\theta>0°$.[21]

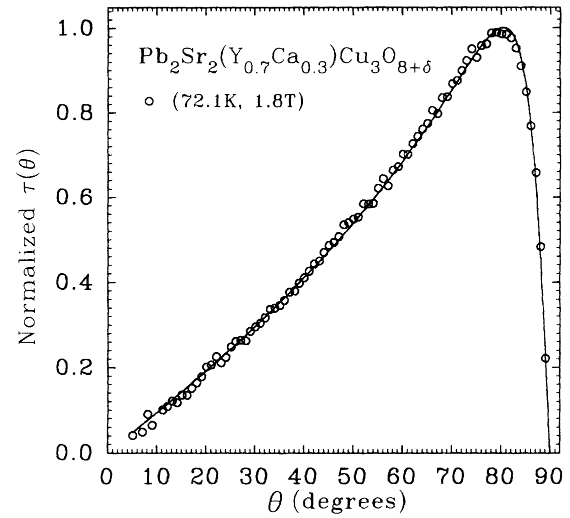

FIG. 1. Normalized $\tau(\theta)$ data for a $Pb_2Sr_2(Y_{0.7}Ca_{0.3})Cu_3O_{8+\delta}$ single crystal. The solid curve is a least-squares fit to Eq. (1). The γ value is 10.2 for these particular data.

Figure 2 shows the $\tau(\theta)$ data for two samples in the angular range $100°>\theta>80°$. In order not to overlap the data, arbitrary units of torque are chosen. The solid curves are the least χ^2 fit; of Eq. (1)

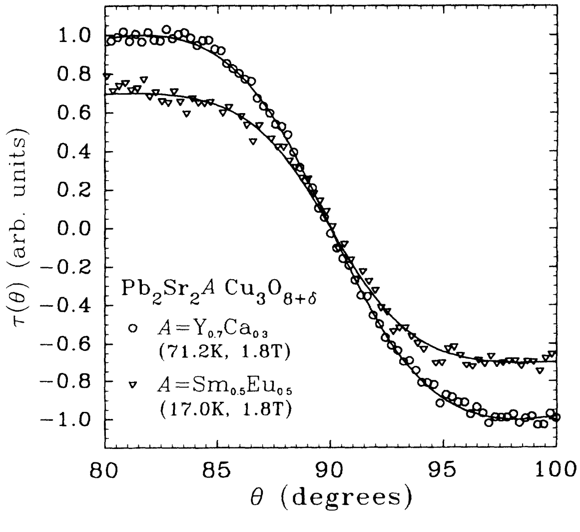

FIG. 2. $\tau(\theta)$ in arbitrary units for field directions within 10° of the ab plane. The solid curve is a least-squares fit to Eq. (1). The best-fit γ for these data sets are 10.4 and 11.9 for $Pb_2Sr_2(Y_{0.7}Ca_{0.3})Cu_3O_{8+\delta}$ and $Pb_2Sr_2(Sm_{0.5}Eu_{0.5})Cu_3O_{8+\delta}$, respectively.

to the data. The best-fit γ values for these data sets are 10.4 and 11.9 for $Pb_2Sr_2(Y_{0.7}Ca_{0.3})Cu_3O_{8+\delta}$ and $Pb_2Sr_2(Sm_{0.5}Eu_{0.5})Cu_3O_{8+\delta}$, respectively.

The uncertainty of the fitting parameter $\Delta\theta$ always converged to about 2×10^{-2} deg for $\gamma\approx10$ and is a negligible factor ($\approx1\%$ correction) in the γ determination. Also, the angular correction for the deviation of the spring resulted in an increase of the γ value of 1–2 %.

The γ value, the diamagnetic onset T_c and the transition width of our $Pb_2Sr_2(Y_{0.7}Ca_{0.3})Cu_3O_{8+\delta}$ and $Pb_2Sr_2Y_{1-y}Cu_3O_{8+\delta}$ crystals and a $Pb_2Sr_2(Sm_{0.5}Eu_{0.5})Cu_3O_{8+\delta}$ crystal are summarized in Table I.

Attempts to measure the γ values of non-yttrium $Pb_2Sr_2(R_{1-x}Ca_x)Cu_3O_{8+\delta}$ crystals were made. The γ values of $Pb_2Sr_2(Er_{0.5}Ca_{0.5})Cu_3O_{8+\delta}$ and $Pb_2Sr_2Eu_{1-y}Cu_3O_{8+\delta}$ were in the range 24–30, and were considerably larger than those of $Pb_2Sr_2(Y_{0.7}Ca_{0.3})Cu_3O_{8+\delta}$ and $Pb_2Sr_2Y_{1-y}Cu_3O_{8+\delta}$. Given that the γ values of $RBa_2Cu_3O_{7-\delta}$(R =Y, Er, Lu, Dy, and Gd) are all close to 8.7,[29] it is somewhat surprising for the $Pb_2Sr_2ACu_3O_{8+\delta}$ compounds to have a large spread in γ values for different rare-earth elements. One possible cause is that the sample quality of the $Pb_2Sr_2(Er_{0.5}Ca_{0.5})Cu_3O_{8+\delta}$ and $Pb_2Sr_2Eu_{1-y}Cu_3O_{8+\delta}$ was less good because their T_c's were lower ($\approx$55 K). Previous measurements show that the optimal T_c values can be higher than 70 and 80 K for $Pb_2Sr_2Eu_{1-y}Cu_3O_{8+\delta}$ and $Pb_2Sr_2(Er_{0.5}Ca_{0.5})Cu_3O_{8+\delta}$, respectively.[12,14] This implies that our $Pb_2Sr_2Eu_{1-y}Cu_3O_{8+\delta}$ and $Pb_2Sr_2(Er_{0.5}Ca_{0.5})Cu_3O_{8+\delta}$ crystals with $T_c\approx55$ K are less than optimally doped. Studies of oxygen-deficient $YBa_2Cu_3O_{7-\delta}$ crystals show a large increase of γ values along with a suppression of T_c when the doping level is decreased.[24] The same phenomenon *probably* accounts for the larger γ values of our $Pb_2Sr_2(Er_{0.5}Ca_{0.5})Cu_3O_{8+\delta}$ and $Pb_2Sr_2Eu_{1-y}Cu_3O_{8+\delta}$ crystals. More samples with optimal preparation conditions are needed to settle this issue.

Previous transport studies of the $Pb_2Sr_2(Y_{0.7}Ca_{0.3})Cu_3O_{8+\delta}$ single crystals showed the resistivity anisotropy ratio $\rho_c/\rho_{ab}\approx30$,[13] which is comparable to $\rho_c/\rho_{ab}\approx40$ of $YBa_2Cu_3O_{7-\delta}$.[30] Given their structural similarity and comparable ρ_c/ρ_{ab} values, it is not surprising that the γ value of $Pb_2Sr_2(Y_{0.7}Ca_{0.3})Cu_3O_{8+\delta}$ is comparable to that of $YBa_2Cu_3O_{7-\delta}(\gamma\approx8.5)$. One of the reasons for the larger γ (10–11) of $Pb_2Sr_2(Y_{0.7}Ca_{0.3})Cu_3O_{8+\delta}$ and $Pb_2Sr_2Y_{1-y}Cu_3O_{8+\delta}$ *may* be their larger c-axis lattice constants. The c-axis lattice constant of $Pb_2Sr_2ACu_3O_{8+\delta}$ lies in the range 15.75–15.80 Å and that of $YBa_2Cu_3O_{7-\delta}$ is ~11.7 Å. This larger c-axis separation presumably reduces the interlayer coupling and in general increases the anisotropy.

Another possible reason for the larger γ values is the lower hole concentration. Recent optical conductivity measurements of $Pb_2Sr_2ACu_3O_{8+\delta}$ (Ref. 31) indicated a smaller normal-state low-frequency electronic background compared to that of $YBa_2Cu_3O_{7-\delta}$, yet the anisotropy was about the same as that of $YBa_2Cu_3O_{7-\delta}$. The lower electronic background implies a lower hole doping level, which would also increase the anisotropy, as is the case for oxygen deficient $YBa_2Cu_3O_{7-\delta}$.[24]

TABLE I. The composition, the diamagnetic onset T_c, γ, and the transition width of our $Pb_2Sr_2ACu_3O_{8+\delta}$ crystals.

Composition	T_c	γ	T_c width
$Pb_2Sr_2(Y_{0.7}Ca_{0.3})Cu_3O_{8+\delta}$	79	10±0.8	~8
$Pb_2Sr_2Y_{1-y}Cu_3O_{8+\delta}$	70	11.3±0.6	~7
$Pb_2Sr_2(Sm_{0.5}Eu_{0.5})Cu_3O_{8+\delta}$	45	13±3	~25

Due to the small superconducting volume fraction and wide transition width, λ cannot be extracted from Eq. (1) by fitting procedures. Previous magnetization measurements by Reedyk *et al.* gave an estimate of $\lambda_{ab}(0)$ of $\approx$2600 Å, by using $\lambda_{ab}^2=\Phi/(32\pi^2dM_{\parallel c}/d\ln H)$ with the field along the c axis.[32] Their measurements with the field in the ab plane and along the c axis indicated an $H_{c1}(0)$ anisotropy of approximately 5, and $(dM_{\perp c}/d\ln H)/(dM_{\parallel c}/d\ln H)\approx2.5$ at fields close to H_{c2}. Such measurements are sensitive to misalignment which could reduce the measured anisotropy, and they represent a lower limit of the magnetic anisotropy. Another example would be the H_{c2} measurements of $YBa_2Cu_3O_{7-\delta}$ along different directions by the dc magnetization method, with anisotropy ratio of approximately 5.[33] The present torque measurements of $Pb_2Sr_2ACu_3O_{8+\delta}$ also show that the anisotropy is larger.

IV. CONCLUSIONS

The γ values of $Pb_2Sr_2(Y_{0.7}Ca_{0.3})Cu_3O_{8+\delta}$ and $Pb_2Sr_2Y_{1-y}Cu_3O_{8+\delta}$ are in the range 10–11. These γ value are comparable to $\gamma\approx8.5$ of $YBa_2Cu_3O_{7-\delta}$. For $Pb_2Sr_2(Sm_{0.5}Eu_{0.5})Cu_3O_{8+\delta}$ single crystals, $\gamma=13\pm3$.

Measurements on $Pb_2Sr_2(R_{1-x}Ca_x)Cu_3O_{8+\delta}$ crystals with R other than Y were attempted. For $Pb_2Sr_2(Er_{0.5}Ca_{0.5})Cu_3O_{8+\delta}$ and $Pb_2Sr_2Eu_{1-y}Cu_3O_{8+\delta}$ crystals with lower T_c, γ values are in the range 25–30. We attribute their larger γ values to a reduced hole doping level as evidence by their lower T_c values.

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