Upper Critical Field and Normal-State Resistivity of the High- T_c Superconductors $\text{RBa}_2\text{Cu}_3\text{O}_y$ ($\text{R}=\text{Y, Gd, Dy, Ho, Er and Yb}$)

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Electrical resistivity of the superconducting $\text{RBa}_2\text{Cu}_3\text{O}_y$ ($\text{R}=\text{Y, Gd, Dy, Ho, Er and Yb}$) compounds, which were prepared by prefiring and sintering in air or oxygen, was measured in zero field and various magnetic fields up to 15 T. The experimental results show that the sintering in oxygen is indispensable for the formation of the superconducting $\text{RBa}_2\text{Cu}_3\text{O}_y$ phase. The upper critical field at 0 K in the superconducting $\text{RBa}_2\text{Cu}_3\text{O}_y$ compounds with the transition temperature of ~ 90 K has been determined to be ~ 170 T, and it is suggested that the purified superconducting phase has the normal-state resistivity of $\lesssim 3 \mu\Omega\cdot\text{m}$, if the previous theory of the type-II superconductivity is applied.

(Received August 22, 1989)

Keywords: superconductor, $\text{RBa}_2\text{Cu}_3\text{O}_y$, electrical resistivity, upper critical field

I. Introduction

The recent discoveries of superconductivity with critical transition temperature, T_c , near 30 K in $(\text{La}_{1-x}\text{Ba}_x)_2\text{CuO}_4$ compounds⁽¹⁾ and T_c near 90 K in the Y-Ba-Cu-O system⁽²⁾ have provided the impetus for research in this field. The superconducting phase in the Y-Ba-Cu-O system was identified to be $\text{YBa}_2\text{Cu}_3\text{O}_y$ ($y \approx 7$), which is of the orthorhombically distorted perovskite structure⁽³⁾. Since the latter discovery, most $\text{RBa}_2\text{Cu}_3\text{O}_y$ compounds ($\text{R}=\text{rare earth metals, except for Pm}$) have been found to have an orthorhombic, oxygen-deficient perovskite structure and exhibit superconductivity with $T_c \approx 90$ K (except for the compounds with $\text{R}=\text{Ce, Pr and Tb}$, which are normal above 4.2 K)⁽⁴⁾⁻⁽⁹⁾.

It has been found that the superconducting properties of $\text{RBa}_2\text{Cu}_3\text{O}_y$ with the layer structure⁽¹⁰⁾ strongly depend on the oxygen content and the crystal direction. McKinnon *et al.*⁽¹¹⁾ have found that the orthorhombic and superconducting phase is present over the range $6.3 < y \leq 7$, whereas the tetragonal and semiconducting phase lies from $y = 6.0$ to 6.3. The superconducting transition temperature decreases as y decreases, and the transition is sharp at $T_c = 90$ K near $y = 7.0$ and at 55 K near $y = 6.5$. On the other hand, Hagen *et al.*⁽¹²⁾ have found that the anisotropy between the resistivities along the c -axis and in the a - b plane is ~ 250 just above T_c in a $\text{YBa}_2\text{Cu}_3\text{O}_y$ single crystal. As a result of the nearly two dimensional structure, the anisotropy of the superconducting properties has been also observed in critical-current-density⁽¹³⁾ and upper-critical-field measure-

ments⁽¹⁴⁾⁻⁽²²⁾.

Since the superconducting properties strongly depend on the oxygen content and the crystal direction, it is important to know which factor determines the sample quality of superconducting $\text{RBa}_2\text{Cu}_3\text{O}_y$ materials. The superconducting transition of this compound has been found to become pronouncedly broad under magnetic fields⁽¹⁷⁾⁽²²⁾. This broadening is considered to be attributed to two factors, i.e. (i) the intrinsic dissipation which is associated with flux motion and is thermally activated⁽²³⁾⁻⁽²⁵⁾ and (ii) to the usual origin: i.e. the presence of weak superconducting links between the grains or the inhomogeneity of the sample, possibly arising from inhomogeneous distribution of oxygen atoms. Therefore, the studies on the upper critical field may be used for the determination of the sample quality as will be shown later.

We have measured the electrical resistivity of the polycrystals of $\text{RBa}_2\text{Cu}_3\text{O}_y$ compounds, where $\text{R}=\text{Y, Gd, Dy, Ho and Yb}$, in zero field and various magnetic fields up to 15 T. In this paper, the influence of atmosphere in the sample preparation on the superconducting transition temperature, T_c , and the upper critical field, H_{c2} , has been investigated and we have proposed that the normal-state resistivity is a suitable physical property to determine the sample quality of superconducting $\text{RBa}_2\text{Cu}_3\text{O}_y$ materials. This will be justified on the basis of the discussion of the upper critical fields.

II. Experimental Procedure

The sample of $\text{RBa}_2\text{Cu}_3\text{O}_y$ compounds was made by the solid-state reaction method. A mixture of R_2O_3 (99.99%), BaCO_3 (99.9%) and CuO (99.9%) with the atomic ratio $\text{R}:\text{Ba}:\text{Cu}=1:2:3$ was fired at 1203 K for 36 ks. The prefired mixture was ground and then pressed into a pellet under ~ 160 MPa for the subsequent sintering. After the examinations of the superconducting and

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Table 1 Lattice parameters, a , b and c , in the orthorhombic unit cell for the $\text{RBa}_2\text{Cu}_3\text{O}_y$ ($\text{R}=\text{Y, Gd, Dy, Ho, Er}$ and Yb) compounds which were prefired and sintered in air or oxygen.

Sample		Lattice parameters (nm)		
		a	b	c
Y	Air	0.382	0.387	1.161
	O ₂	0.380	0.386	1.147
Gd	Air	0.384	0.389	1.162
	O ₂	0.382	0.388	1.161
Dy	Air	0.381	0.387	1.160
	O ₂	0.383	0.386	1.158
Ho	Air	0.382	0.389	1.167
	O ₂	0.382	0.388	1.181
Er	Air	0.381	0.387	1.157
	O ₂	0.377	0.384	1.133
Yb	O ₂	0.377	0.385	1.147

normal-state properties in the samples sintered under several conditions (temperature and time), we found an almost satisfactory condition for obtaining superconducting compounds in an atmospheric pressure. The pellet of $\text{RBa}_2\text{Cu}_3\text{O}_y$ compounds was sintered in this condition: i.e. at 1223 K for 36 ks, and cooled to room temperature in the furnace. The prefiring and sintering were performed in air or oxygen.

Crystal structure of these specimens were examined by the powder X-ray diffraction with $\text{CuK}\alpha$ -radiation. The X-ray powder pattern of all $\text{RBa}_2\text{Cu}_3\text{O}_y$ specimens could be completely indexed assuming a 3c-type orthorhombic cell⁽²⁶⁾ with the lattice parameters $a=0.377\text{--}0.384$ nm, $b=0.384\text{--}0.389$ nm and $c=1.133\text{--}1.181$ nm, as shown in Table 1.

The electrical resistivity was measured by a conventional four-probe method with a measuring current of 2–3 mA/mm². Data were taken in zero field and eight fixed fields, 2, 4, 6, 8, 10, 12, 14 and 15 T, from 4.2 to 300 K. The measurement were carried out in a 16.5 T superconducting solenoid at the High Field Laboratory for Superconducting Materials, Institute for Materials Research, Tohoku University. The temperature was measured by the calibrated carbon-glass and platinum resistance thermometers. Temperature measurements were accurate within ± 0.5 K. T_c and H_{c2} values were determined by the midpoint of the resistivity in the superconducting transition region.

III. Experimental Results and Discussion

Three kinds of atmospheres were used to prepare for the $\text{RBa}_2\text{Cu}_3\text{O}_y$ compounds: samples (a) and (b) were prepared in air and oxygen, respectively, during prefiring and sintering, whereas sample (c) was sintered in oxygen after prefiring in air. Figure 1(a) and (b) shows the temperature dependence of the resistivity of the samples (a) and (b), respectively, in the temperature range between 4.2 and 300 K. The resistivity decreases almost linearly

Fig. 1 Temperature dependence of the electrical resistivity for the $\text{RBa}_2\text{Cu}_3\text{O}_y$ ($\text{R}=\text{Y, Gd, Dy, Ho, Er}$ and Yb) specimens produced in (a) air and (b) oxygen.

above 150 K with decreasing temperature. All the samples exhibit the superconductivity of 90 K range, as reported previously^{(4)–(6)}. On the other hand, the normal-state resistivity of the sample (b) is a factor of 2–4 smaller than the value of the sample (a). We have found that the resistivity of the sample (c) lies midway between those of the samples (a) and (b).

The electrical resistivity measurements in magnetic fields were made at 2, 4, 6, 8, 10, 12, 14 and 15 T. Figure 2 shows the temperature dependence of resistivity for the $\text{YBa}_2\text{Cu}_3\text{O}_y$ specimens produced (i.e. prefired and sintered) in (a) air or (b) oxygen. In zero field, an abrupt decrease of the resistivity occurs around 89 and 90 K with the transition width $\Delta T_c=7$ and 6 K for samples (a) and (b), respectively. On the other hand, the superconducting transition is broadened when finite magnetic fields are applied. As previously reported in the Y–Ba–Cu–O superconductor⁽¹⁷⁾⁽²²⁾⁽²⁵⁾, the high temperature side of the transition (i.e. the onset temperature) is only a little influenced by the magnetic field, but the transition width depends strongly on the field strength. This broadening of the superconducting transition under magnetic fields is remarkable especially in the sample (a).

Similar features in the ρ – T curves at 0–15 T can be also found in the $\text{RBa}_2\text{Cu}_3\text{O}_y$ compounds where $\text{R}=\text{Gd, Dy, Ho}$ and Er , as shown in Figs. 3–6. As can be seen

Fig. 2 Temperature dependence of the electrical resistivity for the $\text{YBa}_2\text{Cu}_3\text{O}_y$ specimens produced in (a) air and (b) oxygen under various magnetic fields.

Fig. 4 Electrical resistivity for the $\text{DyBa}_2\text{Cu}_3\text{O}_y$ specimens produced in (a) air and (b) oxygen.

Fig. 3 Electrical resistivity for the $\text{GdBa}_2\text{Cu}_3\text{O}_y$ specimens produced in (a) air and (b) oxygen.

Fig. 5 Electrical resistivity for the $\text{HoBa}_2\text{Cu}_3\text{O}_y$ specimens produced in (a) air and (b) oxygen.

from these figures, the normal-state resistivity is smallest and the superconducting transition is sharpest for the $\text{HoBa}_2\text{Cu}_3\text{O}_y$ compound among the specimens produced either in air or oxygen. For the $\text{HoBa}_2\text{Cu}_3\text{O}_y$ specimen produced in oxygen, $\rho_r = 3.3 \mu\Omega \cdot \text{m}$, $T_c = 91 \text{ K}$ and $\Delta T_c = 4 \text{ K}$ in zero field, whereas the midpoint and the width of the superconducting transition are 85 K and 30

K, respectively, at 15 T. For the $\text{YbBa}_2\text{Cu}_3\text{O}_y$ compound, we could not produce a single phasic sample in air. Figure 7 shows the temperature dependence of resistivity for the $\text{YbBa}_2\text{Cu}_3\text{O}_y$ specimen produced in oxygen. In zero field, $\rho_r = 16.7 \mu\Omega \cdot \text{m}$, $T_c = 87 \text{ K}$ and $\Delta T_c = 4 \text{ K}$, and

Fig. 6 Electrical resistivity for the $\text{ErBa}_2\text{Cu}_3\text{O}_y$ specimens produced in (a) air and (b) oxygen.

Fig. 7 Electrical resistivity for the $\text{YbBa}_2\text{Cu}_3\text{O}_y$ specimen produced in oxygen.

the broadening of the superconducting transition remains fairly small even at 15 T.

In the H_{c2} studies using the single crystal of $\text{YBa}_2\text{Cu}_3\text{O}_y$ compound, Oh *et al.*⁽¹⁷⁾ and Oda *et al.*⁽²²⁾ have found that the broadening of the resistivity transition under the magnetic field parallel to the c -axis becomes 4–5 times larger than that parallel to the a - b plane, which is interpreted in terms of the critical fluctuations with a short coherence length along the c -axis. By Yeshurun and Malozemoff⁽²³⁾, Tinkham⁽²⁴⁾ and Palstra *et al.*⁽²⁵⁾, the broadening of the resistive transition under magnetic fields in the single crystal of high- T_c superconductors has been discussed without invoking material inhomogeneity in terms of the pinning, creep, and flow of quantum flux lines in the superconducting medium. They have predicted that the transition width scales⁽²⁴⁾ as $\Delta T_c \propto B^{2/3}$, where B is the flux density in sample, and an exponential

temperature dependence of the resistivity well below T_c ⁽²⁵⁾.

The broadening in our polycrystal specimens may be attributed not only to this intrinsic dissipation^{(23)–(25)}, but also to the presence of weak superconducting links between the grains and to the inhomogeneous distribution of oxygen atoms. Actually, the resistive transition under magnetic fields shows two steps around 90 and 55 K in the $\text{DyBa}_2\text{Cu}_3\text{O}_y$ and $\text{ErBa}_2\text{Cu}_3\text{O}_y$ samples produced in air. Since there is no extra phase in all samples according to the X-ray examination, this 55 K transition is probably due to the oxygen-pure region in the sample.

Figure 8 shows the temperature dependence of the upper critical field, H_{c2} , for the $\text{RBa}_2\text{Cu}_3\text{O}_y$ specimens ($\text{R}=\text{Y}, \text{Gd}, \text{Dy}, \text{Ho}, \text{Er}$ and Yb), which were produced in air or oxygen. In this figure, the closed and open circles are the data of the specimens produced in air and oxygen, respectively. As seen from the bars in this figure, the transition width in each compound prepared in air is larger than that produced in oxygen, especially under magnetic fields. The temperature slope of H_{c2} near T_c , $(-dH_{c2}/dT)_{T_c}$, is also smaller in the former. For instance, the endpoints of the superconducting transition at 15 T are 50 and 70 K for the $\text{HoBa}_2\text{Cu}_3\text{O}_y$ specimens produced in air and oxygen, respectively. The $(-dH_{c2}/dT)_{T_c}$ values are estimated as 1.5 and 2.8 T/K in these specimens, respectively.

Oda *et al.*⁽²²⁾ have found that H_{c2} exhibits an anisotropy between the c -axis and a - b plane where the resistivity was measured for a $\text{YBa}_2\text{Cu}_3\text{O}_y$ single crystal under magnetic fields up to 26 T. The temperature slope of H_{c2} at $T \leq T_c$, $(-dH_{c2}/dT)_{T_c}$, is 0.56 T/K for the c -axis and 3.3 T/K for the a - b plane. Our results for all the compounds produced in both air and oxygen lie between these two values. However, the X-ray and metallographic examinations show that our sample is a polycrystal with small grains oriented at random. The difference in $(-dH_{c2}/dT)_{T_c}$ of our specimens produced in air and oxygen may be caused by the inhomogeneous distribution of oxygen atoms. The inhomogeneity exists especially in the former specimens, as in the case of the broadening of the transition width.

The residual resistivity, ρ_r , was determined as the normal-state resistivity just above the onset temperature of superconducting transition. When sintered in oxygen, ρ_r becomes a factor of 2–4 smaller and $(-dH_{c2}/dT)_{T_c}$ becomes 2 to 3 times larger than the values for the samples produced in air. Concerning the superconductivity and the crystal structure of the tetragonal $\text{LaBa}_2\text{Cu}_3\text{O}_y$ compound, Nakai *et al.*⁽²⁷⁾ have found that the occupation probability in the oxygen atomic sites lying between the barium atomic layers is 0.2 for the semiconducting phase ($y \leq 6.2$), whereas it approaches 0.5 for the superconducting phase with $T_c \approx 50$ K. Thus, it is concluded that by sintering in oxygen, the occupation probability of oxygen atoms homogeneously increases and the $\text{RBa}_2\text{Cu}_3\text{O}_y$ compound is purified as the superconducting phase with small ρ_r .

Here, we shall discuss the superconducting and normal-state properties of $\text{RBa}_2\text{Cu}_3\text{O}_y$ compounds in terms of

Fig. 8 Temperature dependence of the upper critical field for the $\text{RBa}_2\text{Cu}_3\text{O}_y$ ($R=\text{Y, Gd, Dy, Ho, Er and Yb}$) specimens produced in air (closed circles) and oxygen (open circles). The bar shows the transition width of superconductivity.

the previous theory of type-II superconductivity, since no one has known what governs the high- T_c oxide-superconductors. The upper critical field at 0 K is related⁽²⁸⁾ at dirty limit to the equation:

$$H_{c2}^A(0) = 0.69T_c(-dH_{c2}/dT)_{T_c}. \quad (1)$$

As shown in Table 2, the $H_{c2}^A(0)$ value is given to be 140–230 T for the $\text{RBa}_2\text{Cu}_3\text{O}_y$ compounds produced in oxygen. The Clogston upper-critical-field limit is defined⁽²⁹⁾ as

$$H_p(0) = 1.84T_c. \quad (2)$$

The estimated $H_p(0)$ value (160–170 T) is lying in the

Table 2 Superconducting transition temperature, the residual resistivity, the temperature slope of upper critical field at $T \leq T_c$, and the upper critical fields at 0 K in the $\text{RBa}_2\text{Cu}_3\text{O}_y$ compounds produced in oxygen.

R	T_c (K)	ρ_r ($\mu\Omega \cdot \text{m}$)	$(-dH_{c2}/dT)_{T_c}$ (T/K)	$H_{c2}^A(0)$ (T)	$H_p(0)$ (T)
Y	90	11.5	2.3	141	166
Gd	93	7.9	2.5	159	171
Dy	88	14.0	2.6	156	162
Ho	91	3.3	2.8	175	167
Er	90	5.4	3.7	227	166
Yb	87	16.7	3.9	231	160

range of $H_{c2}^A(0)$. On the other hand, the upper critical field at 0 K can be expressed⁽²⁸⁾ as

$$H_{c2}^B(0) = 3.11\gamma\rho_r T_c, \quad (3)$$

where the electronic specific-heat constant, γ and ρ_r are in $\text{mJ m}^{-3}\text{K}^{-2}$ and $\Omega\cdot\text{m}$, respectively.

There are no data on the γ value directly determined from the low temperature heat-capacity measurements for the strong-coupling type-II superconductors, $\text{RBa}_2\text{Cu}_3\text{O}_y$. However, the heat-capacity jump at the transition temperature in superconductors, ΔC , leads to the γ value according to the Bardeen-Cooper-Schrieffer theory⁽³⁰⁾: i.e.

$$\Delta C = 1.43\gamma T_c. \quad (4)$$

From the ΔC value, the previous investigators have estimated $\gamma = 1.1\text{--}2.3 \text{ mJ K}^{-2} \text{ mol}^{-1}$ for the $\text{YBa}_2\text{Cu}_3\text{O}_y$ ^{(31)–(34)} and $\text{DyBa}_2\text{Cu}_3\text{O}_y$ ⁽³⁵⁾ compounds. If we adopt the medium value reported by Phillips *et al.*⁽³²⁾ ($1.5 \text{ mJ K}^{-2} \text{ mol}^{-1}$) as γ , the $H_{c2}^B(0)$ value can be calculated by using the values of T_c , ρ_r and the lattice parameters, a , b and c , which were estimated from this work. A value of $H_{c2}^B(0)$ similar to $H_{c2}^A(0)$ or $H_p(0)$ is obtained only for the case of $\text{HoBa}_2\text{Cu}_3\text{O}_y$ which has $\rho_r = 3.3 \mu\Omega\cdot\text{m}$: i.e. $H_{c2}^B(0) = 172 \text{ T}$. The $H_{c2}^B(0)$ values calculated for other $\text{RBa}_2\text{Cu}_3\text{O}_y$ specimens are anomalously large, which is probably due to too large value of ρ_r . This may mean that the purified superconducting phase of $\text{RBa}_2\text{Cu}_3\text{O}_y$ compounds has the normal-state resistivity of $\lesssim 3 \mu\Omega\cdot\text{m}$.

The degree of purity of the high- T_c oxide-superconductor clearly appears in the resistivity measurements under magnetic fields. However, the present work has definitely shown that the normal-state resistivity can be a factor which determines the purity of the superconducting $\text{RBa}_2\text{Cu}_3\text{O}_y$ materials without the examination of ρ vs T under magnetic fields.

Acknowledgments

The authors would like to thank Professor K. Nakazawa who has supported, and Mr. M. Saito, Mr. T. Kusakabe, Mr. H. Daigaku, Mr. K. Chiba and Mr. Y. Wada for help in the experiments.

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