Irreversibility and critical current density of FeSr$_2$ErCu$_2$O$_{6+y}$

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Abstract. FeSr$_2$ErCu$_2$O$_{6+y}$ (ErFe1212) and non-superconducting FeSr$_2$ErCu$_{1.9}$Zn$_{0.1}$O$_{6+y}$ were synthesized to study the property of the superconductivity and the irreversibility of ErFe1212. A large irreversibility in the temperature dependence of magnetization and a hysteresis in the magnetization curve were observed in ErFe1212. By comparison with non-superconducting FeSr$_2$ErCu$_{1.9}$Zn$_{0.1}$O$_{6+y}$, it was found that the most part of the hysteresis at high magnetic field originates from the magnetism of Fe ion and some part of the hysteresis at low magnetic field originates from the superconductivity. Using the magnetization curve of ErFe1212 and FeSr$_2$ErCu$_{1.9}$Zn$_{0.1}$O$_{6+y}$, the $J_c$ of ErFe1212 in individual grains at 10 K under 0.1 T was estimated by the Bean model and $J_{c\text{ intra}}$ was $2.6 \times 10^9$ A/m$^2$. The critical current density across inter-grain boundaries at 10 K estimated by $V-I$ measurement was $J_{c\text{ inter}} = 5.7 \times 10^4$ A/m$^2$. A large difference between $J_{c\text{ inter}}$ and $J_{c\text{ intra}}$ was observed in ErFe1212. $J_{c\text{ intra}}$ and $J_{c\text{ inter}}$ of ErFe1212 are 2.2 and 5.2 times larger than these of YFe1212, respectively.

1. Introduction

The crystal structure of FeSr$_2$YCu$_2$O$_{6+y}$ (YFe1212) is related to that of YBa$_2$Cu$_3$O$_{7-\delta}$, in which Cu ion is partially replaced by Fe ion and Ba ion is fully replaced by Sr ion. In YFe1212, superconductivity has been found to be associated with CuO$_2$ plane and magnetism also has been associated with FeO$_6$ layers. YFe1212 shows superconductivity only after three separate annealing process [1]. The first annealing is performed in N$_2$ flow, and this relates to suppression of Fe substitution at Cu(2) sites. The second and third annealing are in O$_2$ flow and in a high-pressure O$_2$ atmosphere, respectively, to supply carriers to the CuO$_2$ planes[2, 3]. The optimized annealing condition is annealing in N$_2$ flow at 790 °C, annealing in O$_2$ flow and under high O$_2$ pressure at 270 °C[4]. Superconductivity of YFe1212 was much affected by the annealing temperature in N$_2$ flow rather than the annealing temperature in O$_2$ flow. After this annealing process, YFe1212 shows zero resistivity below 40 K.

Two stage transition was observed in resistivity measurement under the magnetic field. The critical current densities of YFe1212 in individual grains, $J_{c\text{ intra}}$, and across inter-grain boundaries, $J_{c\text{ inter}}$, were studied[5]. $J_{c\text{ inter}}$ was about six orders of magnitude lower than $J_{c\text{ intra}}$ at 2 K under 0.1 T and this seems to be responsible for the two stage transition.

The superconducting properties of YBa$_2$Cu$_3$O$_{7-\delta}$ are affected by the substitution of rare earth (RE) ions for Y ion, which are sandwiched between CuO$_2$ plane. Most type of RE ions substituted for Y ion and the effects of the substituting ions on the superconducting
transition temperature[6] and magnetic irreversibility field[7] have been reported. The Y sites in YFe1212 is also occupied by RE ions. The crystal structure of FeSr2ErCu2O6+y (ErFe1212) and FeSr2NdCu2O6+y was studied[8] and the superconducting properties and Fe ion distribution of ErFe1212, FeSr2EuCu2O6+y, FeSr2GdCu2O6+y and FeSr2NdCu2O6+y were studied[9]. A large irreversibility was observed in the temperature dependence of magnetization for ErFe1212 and the origin of this irreversibility was unclear. On the assumption that this large irreversibility originates from the superconductivity, it implies that ErFe1212 has large critical current density.

In this study, the proper annealing condition was determined and the magnetic and electric property of ErFe1212 were studied to reveal the origin of this irreversibility and the superconducting property.

2. Experimental

Polycrystalline samples of ErFe1212 were prepared by a solid state reaction using a stoichiometric mixture of Er2O3, SrCO3, CuO and Fe2O3 powders. The mixture was calcined at 850 °C for 24 h in air, ground and then pressed into pellets. The pellets were sintered at 950 °C for 24 h in air. Then, they were subsequently annealed at Tann = 730 ~ 850 °C for 24 h in N2 flow, at 270 °C for 24 h in O2 flow, and finally at 270 °C for 24 h in O2 atmosphere under a high pressure of 17.6 MPa. The non-superconducting FeSr2YCuxZn0.1O6+y were synthesized and annealed by the same process.

The crystal structure and phase purity of the samples were characterized using powder x-ray diffraction. The magnetization curves and the temperature dependence of magnetization were measured using a SQUID magnetometer (MPMS-XL5, Quantum Design). The resistivity was measured by a four-probe dc technique. The critical current density in individual grains, Jc\text{intra} was estimated by the Bean model[10] and that across inter-grain boundaries, Jc\text{inter} was estimated from voltage-current measurement.

3. Result and discussion

For as-synthesized sample, all of the peaks in x-ray diffraction pattern can be assigned to single-phase ErFe1212 with a tetragonal crystal structure. After the annealing in N2 flow, the crystal structure changes to an orthorhombic structure and after the annealing in O2 flow, it reverts to tetragonal structure.

The temperature dependence of the resistance for FeSr2ErCu2O6+y after various annealing temperature in N2 flow is shown in Fig. 1. The samples were annealed in O2 flow and under high O2 pressure at 270 °C after the annealing in N2 flow. The resistance began to decrease below around 60 K in the sample annealed below 810 °C in N2 flow. Zero resistivity was observed below Tc\text{zero} = 27 ~ 44 K for the sample annealed at Tann = 750 ~ 810 °C. The annealing temperature in N2 flow dependence of the transition temperatures, Tc\text{onset} and Tc\text{zero}, were shown in Fig. 2. A peak was observed in Tann dependence of Tc\text{zero} and the maximum Tc\text{zero} = 44 K was observed at Tann = 790 °C. Since superconductivity of YFe1212 was much affected by the annealing temperature in N2 flow rather than that in O2 flow [4], we concluded that the proper annealing temperature of N2 flow and O2 atmosphere is 790 °C and 270 °C, respectively. The sample annealed at this proper annealing temperature was used in following study.

Figure 3 shows the temperature dependence of the magnetization under an applied magnetic field of 1.0 mT after zero field cooling and field cooling. The magnetization of ErFe1212 begins to decrease at Tc = 56.6 K and diamagnetism was observed. The temperature dependence of magnetization consists of diamagnetism of superconductivity and the magnetism of Fe ion and paramagnetism of Er ion. A large irreversibility was observed between zero field cooled and field cooled curves below 26.6 K. On the other hand, no diamagnetism was observed and similar irreversibility was observed below 21.6 K in non-superconducting FeSr2YCuxZn0.1O6+y.
Figure 1. Temperature dependence of the resistances for FeSr$_2$ErCu$_2$O$_{6+y}$. The values of the resistivities are normalized with the resistances at 300 K.

Figure 2. Relation between the transition temperature, $T_{c\text{onset}}$ and $T_{c\text{zero}}$, for FeSr$_2$ErCu$_2$O$_{6+y}$ and the annealing temperature in N$_2$ flow, $T_{N_2}$.

Figure 3. Temperature dependence of magnetization under 1.0 mT.

Figure 4. Temperature dependence of resistivity for FeSr$_2$ErCu$_2$O$_{6+y}$.

The temperature dependence of resistivity was shown in Fig. 4. In zero magnetic field, the resistivity was decreased due to the onset of the superconductivity at 60.5 K. Zero resistivity was observed below $T_{c\text{zero}} = 43.3$ K. Under the magnetic field, two stage transition in the resistivity curves were observed and $T_{c\text{zero}}$ was decreased with increase of the magnetic field.

The magnetization curves of ErFe1212 were shown in Fig. 5. The magnetization curve consists of the type-II superconductivity and the magnetism of Fe ion and paramagnetism of Er ion. Small hysteresis was observed and it becomes small with increase of temperature. The critical current density in individual grains, $J_{\text{intra}}$, was estimated by the width of magnetization in the hysteresis curve and the Bean model[10].
$J_{\text{c intra}} = \frac{3}{2} \times \frac{\Delta M}{r}$

(1)

$J_{\text{c intra}} = \frac{3}{2} \times \frac{\Delta M - \Delta M'}{r}$

(2)

where $\Delta M'$ is $\Delta M$ of FeSr$_2$YCu$_{1.9}$Zn$_{0.1}$O$_{6+y}$. $J_{\text{c intra}}$ of ErFe1212 at 10 K under 0.1 T is estimated as $2.6 \times 10^9$ A/m$^2$ and this value is 2.2 times larger than that of YFe1212[5].

The voltage-current curve for ErFe1212 is shown in Fig. 7. The values of the voltage and current are normalized by the sample dimension. The critical current density across the intergrain boundaries, $J_{\text{c inter}}$, was determined using a 100 $\mu$V/m offset criterion. The temperature dependence of $J_{\text{c inter}}$ is shown in Fig. 8. $J_{\text{c inter}}$ at 10 K was $5.7 \times 10^4$ A/m$^2$ and was decreased linearly with increase of temperature. $J_{\text{c inter}}$ of ErFe1212 was 5.2 times larger than that of YFe1212[5]. $J_{\text{c inter}}$ was about five orders of magnitude lower than $J_{\text{c intra}}$ under 0.1 T. The broadening in resistivity measurement was probably due to this large difference between $J_{\text{c inter}}$ and $J_{\text{c intra}}$[5].

4. Summary

ErFe1212 and non-superconducting FeSr$_2$ErCu$_{1.9}$Zn$_{0.1}$O$_{6+y}$ were prepared to study the property of the superconductivity and the irreversibility of ErFe1212. ErFe1212 exhibits superconductivity only when it is annealed in a N$_2$ atmosphere and subsequently in an O$_2$ atmosphere. The proper annealing condition of ErFe1212 was determined in advance.
A large irreversibility was observed in the temperature dependence of magnetization below 26.6 K. The temperature dependence of magnetization consists of diamagnetism of superconductivity and the magnetism of Fe ion and Er ion. The magnetism of Er ion kept Curie-Weiss type paramagnetism down to 2 K.

The magnetization curve consists of the type-II superconductivity and the magnetism of Fe ion and paramagnetism of Er ion. A peak of $\Delta M$, that is the difference of the magnetization per unit volume between ascending and descending field process, was observed at around 1.3 T and large $\Delta M$ was observed at zero magnetic field. Since a similar irreversibility was observed for non-superconducting FeSr$_2$ErCu$_2$O$_{6+y}$, the irreversibility at around 1.3 T may originate from the magnetism of the Fe ion.

Some part of the irreversibility under low magnetic field may originate from the superconductivity. Using the magnetization curve of ErFe$_{12}$$\text{La}_{2}$ and FeSr$_2$ErCu$_{1.9}$Zn$_{0.1}$O$_{6+y}$, $J_c^{\text{inter}}$ of ErFe$_{12}$$\text{La}_{2}$ at 10 K under 0.1 T was $2.6 \times 10^9$ A/m$^2$. $J_c^{\text{intra}}$ at 10 K was $5.7 \times 10^8$ A/m$^2$ estimated by $V - I$ measurement. A large difference between $J_c^{\text{inter}}$ and $J_c^{\text{intra}}$ was observed in ErFe$_{12}$$\text{La}_{2}$. The broadening in resistivity measurement was probably due to this large difference. $J_c^{\text{intra}}$ and $J_c^{\text{intra}}$ of ErFe$_{12}$$\text{La}_{2}$ are 2.2 and 5.2 times larger than these of YFe$_{12}$$\text{La}_{2}$, respectively.

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