Research article

Enhanced magnetic ordering by impurity Fe substitution on electron-doped superconductors Eu$_{2-x}$$\times$$\text{Ce}_x$$\times$$\text{yCu}_{1-y}$$\times$$\text{Fe}_y$$\times$$\text{O}_4$$\times$$\alpha$$\times$$\delta$

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Fe impurities

T$_c$

A B S T R A C T

Crystal structure and magnetic properties are two important properties of superconducting materials that need to be studied. We partially substituted the Cu with a magnetic impurity Fe in the electron-doped superconducting cuprate of Eu$_{2-x}$$\times$$\text{Ce}_x$$\times$$\text{yCu}_{1-y}$$\times$$\text{Fe}_y$$\times$$\text{O}_4$$\times$$\alpha$$\times$$\delta$ (ECCFO) to investigate the effect of Fe impurity substitution on crystal structure and magnetic properties. In this study, we have chosen concentrations of $x = 0.12$ and $0.15$, and concentrations of $y = 0.01, 0.02$, and $0.05$. The crystal structure was studied by analyzing XRD patterns, and magnetic properties were analyzed using data obtained from susceptibility measurements. For $y = 0$, the lattice parameter $a$ and Cu–O(2) bond length increased with increasing $x$ from 0.12 to 0.15. It is confirmed by magnetic susceptibility measurement that the $T_c$ of $x = 0.15$ (14 K) is larger than that of $x = 0.12$ (12 K). For the effect of the addition of Fe on the structural parameters, the lattice parameter $c$ increased with the addition of Fe, which means the distance between the charge reservoir and the conducting layer is getting farther. The addition of Fe to the ECCFO sample increased the $C$ and $\mu_0$ values. This is because Fe impurity causes an increase in the magnetic ordering of the ECCFO material and reduces its superconductivity.

1. Introduction

The study of the crystal structure and its relation to physical properties, magnetic properties, and critical temperature ($T_c$) value of high $T_c$ superconducting cuprates (HTSC) has attracted the attention of many researchers since this material was discovered in 1986. As is known, HTSC has a distinctive crystal structure where this material has two essential parts, namely the charge reservoir layer and the conducting layer. There are two known types of HTSC, namely hole-doped HTSC and electron-doped HTSC. The hole-doped HTSC was reported to have several conducting layers, and the number of these conducting layers determines $T_c$ and other properties. On the other hand, the most studied electron-doped HTSC have only one conducting layer. This difference causes the $T_c$ for hole-doped HTSC to be much higher than that of electron-doped HTSC. In addition, for electron-doped HTSC, the oxygen content of the material is also more difficult to control during synthesis than hole doping HTSC. This condition causes the value of $T_c$ of electron-doped HTSC to depend on the amount of charge carrier (doping) and the amount of oxygen content. It also causes the research and reports of hole-doped HTSC to be far more numerous [1, 2, 3, 4, 5, 6, 7] than that of electron-doped HTSC [8, 9, 10, 11, 12, 13, 14, 15, 16]. However, even though the synthesis process is very difficult, research on electron-doped HTSC should be performed to study the mechanism of superconductivity, especially regarding the symmetry in properties between hole-doped HTSC and electron-doped HTSC.

Among the electron-doped superconductors, the material with the parent compound Eu, namely Eu$_2$$\times$$\text{Ce}_x$$\times$$\text{CuO}_4$ (ECCO), has received much attention when studying the magnetic properties of these materials. This is because Eu$^{3+}$ is a material with no magnetic moment in the ground state, so when studying the effect of impurities substitution on its structure and magnetic properties, the information obtained comes from the basic nature of the superconducting material, which is disturbed by the impurities. Studies on the effect of magnetic impurities such as Ni or nonmagnetic impurities such as Zn on the properties of electron-doped HTSC have been reported [6, 9, 10]. It was reported that magnetic impurities have a stronger effect than nonmagnetic impurities in reducing superconductivity [13]. This phenomenon contrasts with the effect of these two types of impurity in hole-doped HTSC [13].

Especially for magnetic impurity Ni substitution, various studies were also conducted to study various properties such as Quantum Critical

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Point (QCP) [2], the hole-trapping effect [3], and the stripe-pinning effect [3]. These properties are fundamental to study. Considering the effect of this magnetic impurity, it can significantly change the magnetic properties of hole-doped and electron-doped materials. In order to study the effect of magnetic impurities with greater magnetic strength, it is also necessary to study the effect of other types of magnetic impurities that are stronger than Ni. In this case, Fe is widely used. In hole-doping superconducting materials, the effect of Fe impurities on superconductivity has been studied extensively [6]. It was reported that Fe impurity could cause a spin-glass-like magnetic state due to Ruderman–Kittel–Kasuya–Yosida (RKKY) interaction and also a localized state with stripe correlations [6].

The effect of magnetic impurities on the hole- or electron-doped superconducting materials has been studied for the optimum- and under-doped region. Studying magnetic impurities in the optimum-doped region is intended to determine the effect of these impurities on their superconductivity properties. Meanwhile, the study on the effect of magnetic impurities in the underdoped area is intended to reveal various phenomena that occur in materials in the underdoped area, such as the 1/8 anomaly, pseudogap, coexistence between magnetism and superconductivity, and spin-glass state, which will help explain the emergence of superconductivity phenomena.

To our knowledge, studies of strong magnetic impurities such as Fe on the crystal structure and the magnetic properties of ECCO, especially in the optimum- and under-doped regions, have not been widely reported. At the optimum-doped condition \( x = 0.15 \), we reported the transport and magnetic properties in the normal state and found that the electron mobility decreased and the magnetic moment per unit volume increased with the addition of Fe [11]. However, detailed studies on crystal structure and magnetic properties, especially regarding changes in the magnitude of the effective magnetic moment which are directly related to their magnetic properties, have not been reported. In addition, there are also no reports on the effect of Fe on ECCO materials for under-doped regions. This paper reported a study on the effect of partial substitution of Fe on Cu in \( \text{Eu}_{2-x-y} \text{Ce}_x \text{Cu}_{1-y} \text{Fe}_y \text{O}_{4+0.5} \) samples with \( x = 0.12 \) (underdoped) and 0.15 (optimum doped) for the concentration of \( y = 0.01, 0.02, \) and 0.05. All samples were characterized by X-ray Diffraction (XRD) and Superconducting Quantum Interference Device (SQUID) measurements to analyze the crystal structure and magnetic properties.

2. Materials and methods

2.1. Sample preparation

\( \text{Eu}_{2-x-y} \text{Ce}_x \text{Cu}_{1-y} \text{Fe}_y \text{O}_{4+0.5} \) samples with \( x = 0.12 \) and 0.15 for \( y = 0.01, 0.02, \) and 0.05 were prepared by solid state method. The concentration of Ce and Eu needed to be changed to keep the sample in under- and optimum-doped region because the Fe used in this study was \( \text{Fe}^{3+} \). Detailed information of sample preparation has been reported in our previous paper [14]. After sintering at 1000 °C, the sample has excess oxygen (\( \alpha \)), which must be reduced or removed by the annealing process. The values of \( \alpha \) is estimated to be less than 0.1, while the \( \delta \) value calculated after annealing is expected to be equal to the value of \( \alpha \) to form an ECCO phase with an oxygen content of four [17].

2.2. Characterization

All bulk samples were characterized by XRD (Bruker AXS D8 ADVANCE) with Cu-Ka radiation. Measurements were performed in the range of 2 \( \theta = 10°-70° \). XRD patterns were analyzed to determine crystal size, lattice parameters \( a \) and \( c \), Eu–O and Cu–O bond lengths.

The SQUID (MPMS 7 Quantum Design) is used to measure the susceptibility in the temperature range from 2 to 20 K in the field-cooled process at \( H = 5 \) Oe. All samples for SQUID measurements were in

Figure 1. Results of XRD characterization of ECCFO with \( x = 0.12 \) (a), 0.15 (b) for \( y = 0, 0.01, 0.02, \) and 0.05.
powder form with masses ranging from 50 mg - 70 mg. XRD and SQUID measurements were carried out at the Department of Applied Physics, the Graduate School of Engineering, Tohoku University, Japan.

3. Results

Figure 1 shows the XRD patterns of Eu$_{2-x-y}$Ce$_x$Cu$_{1-y}$Fe$_y$O$_{4+\alpha-\delta}$ samples with $x = 0.12$ (a) and 0.15 (b) for $y = 0, 0.01, 0.02,$ and $0.05$. From the identification of XRD patterns results, it was found that all samples had identical peaks with the standard sample according to the ICSD reference (code: 98-007-1188) with a Goodness of Fit (GOF) value ranging from 0.8404 to 0.8823. These results indicated that all samples have a tetragonal T' crystal structure (Figure 2(a)). The purity of the sample was found to be around 98.5%–99.6%. A small amount of CeO$_2$ impurity was found in samples with $x = 0.12$, and $y = 0.01, 0.02,$ and $0.05$. This slight impurity is probably caused by inhomogeneous and uncontrollable oxidation processes. In addition, the concentration of the material at $x = 0.12$ which is closer to the antiferromagnetic phase in the phase diagram of ECCO (Under-doped regions), may also be the reason for the appearance of the impurity peak which was identified as the peak of the initial precursor of CeO$_2$. However, the main structure of the ECCO material, namely the T’ structure, is still clearly observed. This indicates that all samples with the T’ structure have been successfully synthesized.

Figure 2 shows the crystal structure of Eu$_{2-x-y}$Ce$_x$Cu$_{1-y}$Fe$_y$O$_{4+\alpha-\delta}$ with a tetragonal structure consisting of Eu atoms (green color) partially filled by Ce (yellow color), Cu atoms (blue color), and Oxygen atoms (red

| $x$  | $y$  | Crystallite Size (nm) | Lattice Parameter (Å) | Bond length (Å) |
|------|------|-----------------------|-----------------------|-----------------|
|      |      |                       | $a$                   | $c$             | Eu–O(1) | Eu–O(2) | Cu–O(2) |
| 0.12 | 0    | 100.37                 | 3.9074                | 11.8578         | 2.2916  | 2.6341  | 1.9537  |
|      | 0.01 | 87.20                  | 3.9071                | 11.8607         | 2.2916  | 2.6343  | 1.9536  |
|      | 0.02 | 93.63                  | 3.9073                | 11.8615         | 2.2917  | 2.6345  | 1.9537  |
|      | 0.05 | 95.07                  | 3.9073                | 11.8633         | 2.2920  | 2.6348  | 1.9539  |
| 0.15 | 0    | 73.87                  | 3.9107                | 11.8494         | 2.2929  | 2.6352  | 1.9544  |
|      | 0.01 | 81.67                  | 3.9084                | 11.8492         | 2.2915  | 2.6336  | 1.9542  |
|      | 0.02 | 77.68                  | 3.9086                | 11.8570         | 2.2925  | 2.6355  | 1.9543  |
|      | 0.05 | 118.22                 | 3.9088                | 11.8560         | 2.2916  | 2.6336  | 1.9544  |
color) (Figure 2(a)). Figure 2 also shows the Cu–O(2) conduction layers (Figure 2(b)), Eu–O(1) bond lengths (Figure 2(c)), and Eu–O(2) bond lengths (Figure 2(d)).

Table 1 shows crystallite size, the lattice parameters $a$ and $c$, bond length of Eu–O(1), Eu–O(2), and Cu–O(2) for all samples. It can be seen that the value of the lattice parameter $a$ is in between 3.9071 Å and 3.9107 Å, while the lattice parameters $c$ has a value in between 11.8479 Å and 11.8672 Å. Eu–O(1) bond was 2.2915 Å to 2.2929 Å, Eu–O(2) bond was 2.6336 Å to 2.6355 Å, and Cu–O bond was 1.9536 Å to 1.9554 Å for $y = 0, 0.01, 0.02$, and $0.05$, respectively. To make it easier to observe the tendency of the data in Table 1, the data for lattice parameter $a$, Lattice parameter $c$, Volume, (d) Cu–O(2) bond length, (e) Eu–O(1) bond length, and (f) Eu–O(2) bond lengths are shown in Figures 3(a) to 3(f). For $y = 0$, the lattice parameter $a$ and Cu–O(2) bond length increased with increasing $x$ from 0.12 to 0.15 as shown in Figures 3(a) and 3(d). This causes the conduction layer that contributes to the value of $T_c$ to be larger. On another side, the lattice parameters $c$ and crystal size decreased with increasing $x$ as shown clearly in Figure 3(b). The changes in lattice parameters and crystal size can probably affect the magnitude of $T_c$. The smaller the lattice parameter $c$ and the crystal size, the closer the distance between the charge reservoir and the conducting layer so that the transfer process can be carried out easily and requires smaller energy, which can probably result in the $T_c$ value of sample $x = 0.15$ being higher than that of $x = 0.12$.

Changes in parameter $c$ may affect other physical parameters which ultimately affect the value of $T_c$ as described in the Inter-Layer Tunneling (ILT) Theory proposed by Anderson and co-workers [17, 18, 19, 20].

![Figure 3](image1.png)  
Figure 3. (a) Lattice parameter $a$, (b) Lattice parameter $c$, (c) Volume, (d) Cu–O(2) bond length, (e) Eu–O(1) bond length, and (f) Eu–O(2) bond length for Eu$_{2-x-y}$Ce$_x$JCu$_{1-y}$Fe$_y$O$_{4+\alpha-\delta}$ with various $y$ and $x$.

![Figure 4](image2.png)  
Figure 4. The susceptibility curves versus temperature of ECCFO with (a) $x = 0.12$, (b) 0.15 for $y = 0, 0.01, 0.02$, and 0.05.
For the effect of the addition of Fe on the structural parameters in the sample with $x = 0.12$ and $0.15$, the value of Eu–O(1) bond (Figure 3(e)), Eu–O(2) bond (Figure 3(f)), and Ca–O(2) bond (Figure 3(d)) does not provide clear information about the changes of parameters. For $x = 0.12$, the lattice parameter $c$ (Figure 3(b)) was observed to change remarkably. The lattice parameter $c$ increased with the addition of Fe, which means the distance between the charge reservoir and the conducting layer is getting larger. For $x = 0.15$, the effect of adding Fe can be observed systematically from changes in the value of the lattice parameter $a$ and the crystallite size. The lattice parameter $a$ decreased while the crystallite size increased with the addition of Fe impurities. The decrease in the lattice parameter $a$ and the increase in the crystallite size causes the changes in the size of the conduction layer and also probably affects to the superconducting properties. Figure 4 shows the susceptibility ($\chi$) curves versus temperature of ECCFO with $x = 0.12$ (a), $0.15$ (b) for $y = 0$, $0.01$, $0.02$, and $0.05$. For $y = 0$, the trace of superconductivity indicated by the changes of magnetic behavior from paramagnetic to diamagnetic is observed at $12$ K and $14$ K for $x = 0.12$ [14] and $0.15$, respectively. This temperature is known as the onset of $T_c$. The value of the onset of $T_c$ increases with the increasing value of $x$, which corresponds to a decrease in the value of the lattice parameter $c$, as discussed in the XRD analysis. For $y \geq 0.01$, diamagnetic properties disappeared indicating that the superconductivity also disappeared by substituting Fe on Cu in the ECCFO sample. To obtain information on magnetic parameters such as Curie constant and effective magnetic moment, the susceptibility data for non-superconducting samples with $y \geq 0.01$ were analyzed by using the Curie equation. The effective magnetic moment is obtained using Eq. (1).

$$\mu_{eff} = \sqrt{\frac{3Ck_B}{N_A\mu_B^2}} = 2.828\sqrt{C}$$  \hspace{1cm} (1)

$\mu_{eff}$ is the effective magnetic moment, $C$ is the Curie constant, $k_B$ is the Boltzmann constant, $N_A$ is the Avogadro constant, and $\mu_B$ is the Bohr magneton.

Figure 5 (b) shows the value of $\mu_{eff}$ of ECCFO with $x = 0.12$, $0.15$ and $y = 0.01$, $0.02$, and $0.05$. The value of $\mu_{eff}$ for $x = 0.12$ increased from $1.0158$ to $2.4117 \mu_B$, and for $x = 0.15$ increased from $0.0443$ to $2.3090 \mu_B$. For $y = 0.01$, the $\mu_{eff}$ value for $x = 0.12$ is greater than $x = 0.15$.

The addition of Fe to the ECCFO samples increased the $C$ (Figure 5(b)) and $\mu_{eff}$ values. The significant increase in $C$ with increasing Fe content indicates that Fe impurity causes an increase in magnetic ordering in the material as suggested in the Abrikosov-Gor’kov theory of increasing magnetic ordering mechanism [21, 22, 23]. Although in the Abrikosov-Gor’kov theory the increase in magnetic ordering is caused by the impurity of non-magnetic materials, in this study, the addition of magnetic impurities Fe also increased the magnetic ordering, which is indicated by increasing the $\mu_{eff}$ value. Magnetic ordering in ECCFO samples triggered by the appearance of Fe impurities will cause the superconductivity to disappear, as is the case for hole-doped materials where the magnetic order is induced by Fe substitution [6].

4. Conclusions

Electron-doped HTSC of Eu$_{2-x-y}$Ce$_x$Y$_{1.25}$Fe$_y$O$_4$ samples with $x = 0.12$ and $0.15$ for $y = 0.01$, $0.02$, and $0.05$ have been successfully studied to investigate the effect of partially substituted Fe on Cu in ECCFO samples to crystal structure and magnetic properties. It is found that all samples have the main peaks of $T'$ tetragonal structures. For $y = 0$, the lattice parameter $a$ and Cu–O(2) bond length increased with increasing $x$ from 0.12 to 0.15 which results in an increase in the size of the conduction layer. On another side, the lattice parameter $c$ and crystal size decreased with increasing $x$ that causing the distance between the charge reservoir and the conducting layer will be closer so that the transfer process can be carried out easily and requires smaller energy, which may be one of the reasons for the change in the value of $T_c$. For the effect of the addition of Fe on the structural parameters, the lattice parameter $c$ increased with the addition of Fe, which means the distance between the charge reservoir and the conducting layer is getting farther. The value of the onset of $T_c$ was observed at $12$ K and $14$ K for $x = 0.12$ and $0.15$ with $y = 0$, respectively. $T_c$ completely disappeared for $y \geq 0.01$. The addition of Fe to the ECCFO sample increased the $C$ and $\mu_{eff}$ values. This is due to Fe impurity causing an increase in spin ordering in ECCFO material. Further research is needed to examine the effect of this Fe substitution over a wider $x$ range in order to obtain complete information regarding the effect of Fe on the magnetic properties of electron-doped superconducting materials.

Declarations

Author contribution statement

Muhammad Abdan Syakuur: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Wrote the paper.

Rosaldi Pratama; Helma Dwi Anggia; Lucia Patia Rochman; Yati Maryati: Performed the experiments; Analyzed and interpreted the data.

Togar Saragi: Conceived and designed the experiments; Performed the experiments; Wrote the paper.
Risdiana: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

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Data availability statement

Data included in article/supp. material/referenced in article.

Declaration of interest’s statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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