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The Role of F-doping and the Sintering Temperature on the Superconductivity and Lattice Constants in LaOFeGe Compounds

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ABSTRACT

The most prominent indicator of superconductivity is the superconducting transition temperature (T_c) that refers to three points. The onset transition temperature (T_{onset}) is defined as the deviation point away from the *ρ(T)* straight line (onset of the drop in resistivity). The midpoint transition temperature ($T_{midpoint}$) is defined as the temperature, where resistivity becomes 50% of its value at T_{onset}. The zero-resistance transition temperature ($T_{\rho\approx0}$) is defined as the temperature, in which the resistance is identically zero or only immeasurably small. These indicators of temperatures associated with some factors. In this article, the X-ray diffraction and electrical resistivity measurements of $LaO_{1-x}F_xFeGe$ samples are reported. This compound was successfully synthesized via a solid state reaction method with the presence of germanium Ge in the conduction layer. Furthermore, some factors affecting the superconducting transition temperature were studied, which are the F-doping dependence of T_{onset}, $T_{midpoint}$, and lattice parameters, and sintering temperature dependence of T_{onset}.

1 Introduction

Iron-based LnOFeAs phase is not a superconductor and displayed an anomalous change in the slope of ρ(T) resistivity measurement curve. The anomaly transition point related to the spin-density wave fluctuations and structural phase transition was at 150 (Kamihara et al., 2008), 145 (Chen et al., 2008), 155 (Prakash et al., 2010), 140 (Martinelli et al., 2008), 135 (Wang et al., 2008), and 124 K (Jun Li et al., 2008) for compounds LaOFeAs, CeOFeAs, PrOFeAs, SmOFeAs, GdOFeAs, and TbOFeAs, respectively. Conversely, nickel-based quaternary oxypnictides LaONiP and LaONiAs exhibited superconducting transition in resistivity measurements with critical transition temperature T_{onset}=4 K ($T_{\rho\approx0}$=2 K) (Watanabe et al. 2007, Tegel et al., 2008) and T_{onset}=2.4 K (Tρ≈0=2 K) (Watanabe et al., 2008), respectively. Moreover, in iron-based 1111-phase, only the LaOFeP compound (Liang et al., 2007, Hamlin et al., 2008) was a superconductor at T_{onset}=5 K ($T_{\rho\approx0}$=3.2 K) (Kamihara et al., 2006).

Superconductivity could be obtained from LnOFeAs phase through replacement of O^{2-} with F^- (i.e., F-doping), with the resulting phase being the $LnO_{1-x}F_xFeAs$ compound. Superconducting transition temperature T_{onset} of $LaO_{1-x}F_xFeAs$ was at 17 (Dong et al., 2008), 28 (Dong et al., 2008), 24.6 (Gao et al., 2008), and 30 K (Kamihara et al., 2008) for x=0.03, 0.06, 0.10, and 0.11, respectively. The F-doping dependence of T_c and T_{onset} on $LaO_{1-x}F_xFeAs$ (Kamihara et al., 2008), after superconductivity appears, T_c is nearly unchanged up to x=0.14, and the highest T_c=26 K (T_{onset}= 30 K) is attained at the F-content x=0.11.

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Replacement La in $LaO_{1-x}F_xFeAs$ compound with other rare earth elements (Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, and Tm) led to superconductors with T_{onset} > 28 K. T_{onset}=42.5 (Prakash et al., 2009), 52 (Ren et al., 2008), 52 (Jia et al., 2008), 56.1 (Wang et al., 2010, Iida et al., 2013), 36.6 (Peng et al., 2008), 45.9 (Bos et al., 2008, Kuzmicheva et al., 2018), and 45.4 K (Bos et al., 2008) for compounds $CeO_{0.8}F_{0.2}FeAs$, $PrO_{0.89}F_{0.11}FeAs$, $NdO_{0.82}F_{0.18}FeAs$, $SmO_{0.8}F_{0.2}FeAs$, $GdO_{0.83}F_{0.17}FeAs$, $TbO_{0.8}F_{0.2}FeAs$, and $DyO_{0.9}F_{0.1}FeAs$, respectively. Two rare earth elements, Eu and Tm, did not display superconducting transition in resistivity measurements of $LnO_{0.84}F_{0.16}FeAs$ phase; instead, resistivity ρ(T) displayed metallic behavior (Gen-Fu et al., 2008).

Superconductivity in LnOFeAs phase can also be obtained by partially replacing the trivalent ion Ln^{3+} with a bivalent dopant, such as Sr^{2+}, Pb^{2+}, or a tetravalent dopant, such as Th^{4+} in the LnO layer. As a result, superconducting transition T_{onset} of superconductors Sr-doped $La_{0.87}Sr_{0.13}OFeAs$ (Wen et al., 2008) and Pb-doped $La_{8.0}Pb_{0.2}OFeAs$ (Che et al., 2008) was at 25.6 ($T_{\rho\approx0}$=15 K) and 11.6 K ($T_{\rho\approx0}$=9.7 K), respectively. In Th-doping, which provides the insulating layer with an extra positive charge, superconductors $Nd_{0.8}Th_{0.2}OFeAs$ (Xu et al., 2008), $Gd_{0.8}Th_{0.2}OFeAs$ (Wang et al., 2008), $Tb_{0.9}Th_{0.1}OFeAs$ (Jun Li et al., 2008), and $Tb_{0.8}Th_{0.2}OFeAs$ (Jun Li et al., 2008) were successfully synthesized, exhibiting transition temperature T_{onset} in resistivity measurements at 47, 56, 45, and 52 K, respectively, all higher than the recorded values for Sr- and Pb-doped compounds.

The third method of doping in the LnO layer consists of replacing the trivalent ion Ln^{3+} and monovalent ion F^- with two dopants, and is referred to as double doping. For K doping in $LaO_{1-x}F_xFeAs$ compound, the onset of superconducting transition was practically unaffected by the addition of K, with onset T_{onset} occurring at 26.20 and 26.45 K for $(La_{0.85}K_{0.15})(O_{0.85}F_{0.15})FeAs$ and $(La_{0.8}K_{0.2})(O_{0.8}F_{0.2})FeAs$, respectively (Prakash et al., 2008). Replacing potassium (K^{1+}) with Ce or Yb increased transition temperature T_{onset} to 29 K for $La_{0.2}Ce_{0.8}O_{0.9}F_{0.1}FeAs$ (Gen-Fu et al., 2008) and 31.3 K for $La_{0.9}Yb_{0.1}O_{0\cdot8}F_{0\cdot2}FeAs$ (Prakash et al., 2010).

Superconductivity can be also achieved by doping in the conduction layer MPn (M=transition metals, and Pn=pnictogen). For Co-doped $LaOFe_{1-x}Co_xAs$ samples, the T_{onset} was at 11.2, 14.3, and 6 K for x=0.05, 0.11, and 0.15, respectively (Sefat et al., 2008). Co-doping for $PrOFe_{1-x}Co_xAs$ samples showed T_{onset} at 4.7, 14.2, and 5.9 K for x=0.05, 0.1, and 0.15, respectively (Prakash et al., 2010). For $SmOFe_{1-x}Co_xAs$ samples, T_{onset} (=15.2 K) was unchanged at two levels of doping x=0.10 and 0.15 (Qi et al., 2008), whereas T_{onset} was affected by the change in doping from x=0.10 to x=0.15 for Co-doping in La-oxypnictide and Pr-oxypnictide samples. Ir-doped $SmOFe_{0.85}Ir_{0.15}As$ compounds provided the critical transition temperature T_{onset} close to 17.3 K (Chen et al., 2009), which is greater than that for Co-doping. In (Singh et al., 2009), researcher reported one case of increased transition temperature T_{onset} with doping in the conducting layer when $LaO_{0.8}F_{0.2}FeAs$ was synthesized with Sb-doping. The doping in this case was in both layers and led to the enhancement of transition temperature to 30.1 K for compound $LaO_{0.8}F_{0.2}FeAs_{0.95}Sb_{0.05}$. Singh et al. (Singh et al., 2014) prepared the $SmO_{0.88}F_{0.12}FeAs_{1-x}P_x$ compound with double-doping in both layers, and they observed a suppression in the superconducting transition temperature.

The critical superconducting transition temperature for 1111-oxyarsenide compounds is affected by a number of factors, mainly the density of conduction carriers in the insulating and conduction layers, which is altered with doping. Although hole or electron-doping suppressed the anomalous behavior to induce superconductivity, some phases did not show superconductivity: $La_{0.80}Sr_{0.20}OFeAs$ (T_{onset}=25.6 K for x=0.13) (Wen et al., 2008), $LaOFe_{0.80}Co_{0.20}As$ (T_{onset}=14.3 K for x=0.11) (Sefat et al., 2008), $PrOFe_{0.70}Co_{0.30}As$ (T_{onset}=14.2 K for x=0.10) (Prakash et al., 2010), $EuO_{0.84}F_{0.16}FeAs$, and $TmO_{0.84}F_{0.16}FeAs$ (Gen-Fu et al., 2008). For nonsuperconducting $La_{0.80}Ce_{0.20}OFeAs$ compound (Che et al., 2008), the doping was at the same oxidation state (La^{3+} was replaced with Ce^{3+}), and as a result, anomaly point T_{anom} still existed and appeared at 155 K.

Concentration and lattice parameters are also factors that affect superconducting transition temperature. Doping dependence of T_c, a, and c for hole-doped $La_{1-x}Sr_xOFeAs$ sample showed that a and c increased monotonously with Sr-doping concentration and a consequent increase in T_c. This expansion in lattice constants is because the radius of Sr^{2+} is larger than that of La^{3+} (Mu et al., 2008). In contrast with electron doping, T_c increased with the shrinkage of lattice parameters, whereby the electron-doped $SmO_{1-x}F_xFeAs$ sample showed that a and c decreased monotonously with the increase in F-doping concentration x in the range of $0 \leq x \leq 0.20$, and T_c increased with the increase in concentration x in the same range (Yang et al., 2009).

When hole doping was applied in the FeAs layer for $PrOFe_{1-x}Co_xAs$ sample, the lattice parameters decreased with the increase in Co concentration x ($0 < x < 0.3$), but T_c increased from 4.7 K for x=0.05 to 14.2 K for x=0.1. However, transition temperature T_c decreased with higher Co-doping concentration $x > 0.1$ (T_c=5.9 K for x=0.15, T_c=4 K for x=0.20 and 0.30) (Prakash et al., 2010). A similar behavior of doping dependence of T_c, a, and c in double-doped compound $La_{1-x}Ce_xO_{0.9}F_{0.1}FeAs$ (x=0, 0.2, 0.4, 0.6, and 0.8) was observed. Lattice parameters a and c slightly decreased with the change in Ce concentration (a=4.029˚A and c= 8.726˚A at x=0, and a=3.994˚A, c=8.598˚A at x=0.8) whereas T_c increased from 24.99 K at x=0 to 29 K at

x=0.8 with the presence of an abnormal point in the phase diagram $T_c(x)$ at x=0.60 (T_c= 28.01 K) (Che et al., 2008). In contrast to the case of $La_{1-x}Ce_xO_{0.9}F_{0.1}FeAs$, the transition temperature for $LaO_{0.8}F_{0.2}FeAs_{1-x}Sb_x$ compound (double-doping in both layers) decreased from 30.1 K at Sb concentration x=0.05 to 28.6 K at Sb concentration x=0.10. However, by increasing the doping level from x=0.05 to x=0.10, lattice parameters *a* and *b* increased because of the larger size of the Sb ion compared with the As ion (Singh et al., 2009).

The effect of sintering temperature on the superconducting properties of $SmO_{0.8}F_{0.2}FeAs$ was reported by Wang et al. (Wang et al., 2010). Onset transition temperature of the samples sintered at 850 ºC was 53.5 K. Samples sintered at 1000 ºC displayed a transition temperature of 56.1 K whereas those sintered at 1200 ºC displayed a transition temperature of 50.8 K. Samples sintered at 1000 ºC had the highest transition temperature, the lowest *ρ(T)*, and the highest residual resistivity ratio *ρ*(300 K)/*ρ*(57 K), indicating low impurity scattering and enhanced carrier density (Wang et al., 2010). However, the highest onset transition temperature was related to specific sintering temperature, where T_{onset} was 41 K for $SmO_{0.7}F_{0.3}FeAs$ sample sintered by a two-step approach at 500 ºC for 15 h and then at 900 ºC for 40 h (Wang et al., 2010), and it was 54.6 K for sintering at 1160 ºC for 40 h (Ma et al., 2008).

2 Materials and Methods

A polycrystalline sample of $LaO_{1-x}F_xFeGe$ (x=0, 0.11, 0.13) was synthesized by heating a mixture of high-purity starting materials (i.e., dehydrated La_2O_3, Fe, LaF_3 powders, Ge grains, and La pieces), which were weighed with stoichiometric amounts of molar mass according to nominal compositions in the following formula for undoped compound LaOFeGe:

$$La+3Fe+3Ge+La_2O_3 \rightarrow 3LaOFeGe \quad (1)$$

For F-doped compound, the stoichiometric formula is:

$$(1+x)\ La + 3Fe + 3Ge + (1-x)\ La_2O_3 + x\ LaF_3 \rightarrow 3LaO_{1-x}F_xFeGe \quad (2)$$

The present method involves two steps. First, 2 g of the starting materials (Table 1) were mixed thoroughly, pressed into a pellet 13 mm in diameter through a manual hydraulic press under a load of 12 metric tons, and then placed between two boats of Tungsten inside a chamber evacuated at 10^{-5} Torr. The pellet was heated at high current by electrical poles at both ends of the boats. Current was applied in the following order: 120 A for 2 h, 160 A for 3 h, and 100 A for 2 h. The product was smashed and grinded at each electric current set-up to make it more homogeneous and was then pressed into a pellet. Second, the final pellet was sealed in an evacuated silica tube at 10^{-3} Torr and then annealed in a furnace under the following conditions: 500 °C for 12 h, 600 °C for 24 h, and 800 °C for 12 h. Then, the pellet was cooled to room temperature gradually. In heat treatments, an electric current of 160 A through the boats (heating source) causes a temperature of around 1000 ºC, which is appropriate for solid-state reaction with consideration for the comparison of melting-point temperatures of the starting materials. Heat treatment for a long time was conducted in the furnace, where the samples were first heated at 500 ºC and then sintered at a temperature range of 600 ºC (i.e., 2/3 of the melting point of La and Ge) to 800 ºC (i.e., 6/7 of the melting point of La and Ge), which is the most suitable temperature range to obtain an adequately annealed sample. Sample preparation, except for the annealing, was conducted in a glove box under high-purity Nitrogen. The samples were cut and polished into a thin bar shape 6 mm long, 1 mm wide, and 1 mm thick for use in measuring DC resistivity.

Table 1. Stoichiometric amounts for starting materials of nominal compositions $LaO_{1-x}F_xFeGe$ according to formulas 1 and 2.

	Starting materials (gm)				
Content X	La Purity: 99.9%	Fe 99.9%	Ge 99.999%	La_2O_3 99.5%	LaF_3 99%
0	0.3268	0.3941	0.5127	0.7664	0
0.11	0.3623	0.3937	0.5121	0.6813	0.0506
0.13	0.3688	0.3936	0.5119	0.6659	0.0598

3 Results

Figure 1 shows the X-ray diffraction patterns of $LaO_{1-x}F_xFeGe$ (x=0, 0.11, 0.13). All main peaks of the samples can be well determined based on the tetragonal oxypnictide structure LnOMPn, which is indexed to the tetragonal ZrCuSiAs-type structure reported previously (Kamihara et al., 2008, Liang et al., 2007, Hamlin et al., 2008, Kamihara et al., 2006, Che et al., 2008, Chen et al., 2008, Gao et al., 2008). A small amount of the impurity phases LaOF and Fe_2Ge were detected. Secondary phases were observed in most LnOMPn oxypnictide phases (Kamihara et al., 2008, Gao et al., 2008).

The lattice parameters *a* and *c* (table 2) were calculated by the least-squares method of the measured peak positions using equation:

$$\sin^2(\theta_{hkl}) = \frac{\lambda^2}{4}\left(\frac{h^2+k^2}{a^2} + \frac{l^2}{c^2}\right) \quad (3)$$

For electrical resistivity measurements *ρ(T)*, table 2 shows the F-doping dependence of T_{onset} ,

$T_{midpoint}$ and lattice parameters for $LaO_{1-x}F_xFeGe$ samples. The effect of sintering temperature on the transition temperature of F-content samples as shown in table 3.

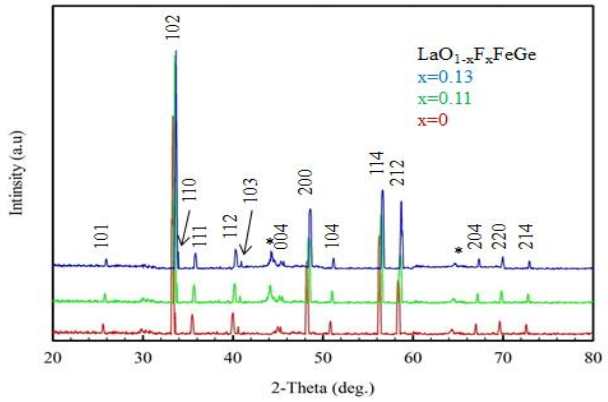

Figure 1. X-ray diffraction pattern of the nominal $LaO_{1-x}F_xFeGe$ samples. The impurity phases LaOF and Fe_2Ge are marked by an asterisk.

Table 2. F-doping dependence of T_{onset} , $T_{midpoint}$, and lattice parameters for $LaO_{1-x}F_xFeGe$ compound.

F content	T_{onset}	$T_{midpoint}$	a [nm]	c [nm]
0	-	-	0.380686	0.815796
0.11	19.7	18.8	0.38178	0.81367
0.13	21.3	20.6	0.379226	0.808415

Table 3. Sintering temperature dependence of transition temperature for $LaO_{1-x}F_xFeGe$ compound.

Sintering temperature [K]	800	850	900
F content		T_{onset} [K]	
x=0.11	19.7	21.2	20.3
x= 0.13	21.3	22.1	22.3

4 Discussion

The lattice parameters of LaOFeGe, a=0.380686 nm and c=0.815796 nm, were found to be smaller than those of the LnOFeAs family reported previously (Kamihara et al., 2008, Chen et al., 2008, Wang et al., 2008, Bos et al., 2008, Chen et al., 2009). Considering that F- has a smaller ionic size than O2-, the peaks shifted to the right-hand side for the F-doped samples. In comparison with the undoped sample, the lattice parameters of F-doped samples decrease with greater F-doping concentration. This result indicates the successful substitution of O by F. For x=0.11, a=0.381786 nm, and c=0.813679 nm and for x=0.13, a=0.379226 nm, and c=0.808415 nm, the c-lattice parameter decreased with the increase in x content (x=0, 0.11, 0.13). By comparing the *a*-lattice parameter with the increase in x content for all samples with each other, the *a* (x=0.13) < *a* (x=0) and *a* (x=0.13) < *a* (x=0.11). However, an expansion in the *a*-lattice parameter was observed, *a* (x=0.11) > *a* (x=0), which is attributed to the impurity phase stacking along the *a*-axis of the x=0.11 phase.

As shown in table 2, T_{onset} and $T_{midpoint}$ increase slightly with increasing F-doping content. For table 3, onset transition temperature of the sample x=0.11 sintered at 800ºC was 19.7 K. Sample sintered at 850ºC displayed a transition temperature of 21.2 K whereas those sintered at 900ºC displayed a transition temperature of 20.3 K. Samples (x=0.11 and 0.13) sintered at 850 ºC had the highest transition temperature.

5 Conclusion

The quaternary compound $LaO_{1-x}F_xFeGe$ were prepared using a two-step solid-state reaction method because the method gives a more homogeneous sintering of the compound in two separate steps of heat treatment. Moreover, this method enables the formation of the initial binary phases when most of the raw materials are direct chemical elements with one or two compounds. The crystal structure of tetragonal LnOMPn oxypnictide (Ln=rare-earth, M=transition metals, and Pn=pnictogen) is characterized by the lattice parameters *a* and *c*. The content x, ionic size of the dopant, and sintering temperature, all affect lattice parameters.

The majority of undoped tetragonal LnOMPn oxypnictides are not superconductors, and only some phases, such as LaOFeP and LaONiP, exhibit a transition to the superconducting state. According to the available data, superconductivity occurs exclusively in oxyarsenide phases under the required condition of electron or hole doping. The previous experimental material suggests that a number of factors affect the critical transition temperature for superconducting 1111 oxypnictides. Doping levels, as well as external factors such as sintering temperature all affect superconductivity. In general, no direct evidence or rule has been discussed to predict the transition behavior in resistivity after doping. However, certain factors cause changes in the interactions among electrons (i.e., electron scattering and electron-phonon scattering).

Conflict of interest: The authors declare that there are no conflicts of interest.

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