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Pressure dependence of $T_c$ in $LnFeAsO_{1-y}$ ($Ln = La, Ce, Nd, Tb$)

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Abstract. We measured the temperature dependence of electrical resistivity of non-fluorine-substituted, oxygen-deficient $LnFeAsO_{1-y}$ ($Ln = La, Ce, Nd, Tb$) under hydrostatic high pressure up to 18 GPa in order to observe pressure dependence of superconducting transition temperature $T_c$. In LaFeAsO$_{0.65}$, onset $T_c$ initially enhances below 4 GPa, then decreases by applying further pressure. However, the zero-resistivity temperature does not show enhancement by applying pressure. In the case of NdFeAsO$_{1-y}$ and TbFeAsO$_{1-y}$, $T_c$ shows monotonic decrease as increasing pressure. $T_c$ is much more likely to go down faster if $Ln$ with small ionic size was taken. Therefore, the bulk superconductivity is suppressed finally at ~7 GPa in TbFeAsO$_{0.7}$.

1. Introduction

It was a pleasant surprise for the scientific community when Kamihara et al. discovered iron pnictide superconductivity in LaFeAsO$_{1-x}$F$_x$ [1]. The highest superconducting transition temperature ($T_c$) in this series of iron pnictide compounds, $LnFeAs(O_{1-x}F_x)$ ($Ln$-1111 type family where $Ln$ is various lanthanide elements), is around 55 K, which was observed in Sm-1111 [2] or Nd-1111 [3]. Even though $T_c$ in $Ln$-1111 is three times smaller than that of the record in high-$T_c$ cuprates [4,5], $Ln$-1111 family of superconductors still has the second-highest $T_c$. It is well-known that $T_c$ in $Ln$-1111 systematically changes on the changing the $Ln$ ion [6]. The relationship between crystal structure and $T_c$ was first pointed out by Lee et al. [7]. They predicted the dependence of $T_c$ on the bond angles of As-Fe-As in the Fe-As tetrahedron. External pressure drastically changes lattice length and also changes $T_c$. The pressure dependence of $T_c$ will help in understanding the mechanisms and important factors that vary $T_c$ in the $Ln$-1111 type family.

Hydrostatic high-pressure is the best tool to investigate the relationship between crystal structure and $T_c$ in $Ln$-1111 because it changes the lattice parameter without causing randomness in the sample. However, generating hydrostatic high-pressures up to 10 GPa is very difficult. Very few electrical resistivity measurements have succeeded under such hydrostatic high-pressure conditions in $Ln$-1111, and few apparatuses are applicable for such measurements [8,9]. As non-isotropic high-pressure provides randomness to the compounds, the transition curve of the resistivity becomes broad [10].
Therefore hydrostaticity is necessary to observe the intrinsic behavior of superconductivity at high pressures. In order to achieve this condition, we have developed a cubic-anvil-type high-pressure apparatus [11], that enables us to measure physical properties precisely at high-pressures up to 20 GPa temperatures as low as that of liquid helium. In this study, we show an accurate pressure phase diagram of $T_c$ in $Ln$-1111 ($Ln = \text{La, Ce, Nd, Tb}$) by measuring electrical resistivity and ac magnetic susceptibility in hydrostatic high-pressure conditions.

2. Experimental

All samples were synthesized using high-pressure techniques. The high-quality polycrystalline samples are suitable for isotropic pressure studies. Many studies on Ln-1111 have used fluorine-substituted samples. However, we have succeeded in demonstrating superconductivity in an oxygen deficient, non-fluorine-substituted sample ($LnFeAsO_{1-y}$) [3,6]. The details of the synthesis techniques are described elsewhere [6]. On examining the $T_c$ of the samples, we concluded that they are optimally doped compounds with the exception of Tb-1111 in resistivity measurement. Judging from $T_c$ and lattice parameters, the Tb-1111 sample in resistivity observation was relatively under-doped. We determine transition temperature $T_c$ in this study as the temperature at which zero resistivity occurs. Zero resistivity is the most fundamental feature of superconductivity and its temperature generally matches the onset temperature of the Meissner signal. Since $T_c$ is very sensitive to the crystal structure in the Ln-1111-type family, randomness in the sample will cause some parts to show a $T_c$ higher than the bulk $T_c$. This will result in a possible overestimation of the onset $T_c$ in resistivity measurement. To obtain the pressure phase diagram for bulk superconductivity, zero-resistivity temperature is appropriate for $T_c$. In this paper, we refer to our previous results [9,12], with redefining $T_c$ as zero-resistivity temperature.

The resistivity at high pressure is measured in cubic-anvil-type high-pressure apparatus [11]. Samples are set in a Teflon case and homogeneously compressed with gasket material independently from six directions. Teflon case is filled with a liquid pressure medium (Daphne7474, distributed by Idemitsu Kosan Co. [13]). The temperature of the sample is monitored using a calibrated resistance-temperature sensor (Lake Shore Cryotronics, RF-100). We also measured the ac magnetic susceptibility of Tb-1111 at ac field of 10 Hz and 4 Oe with a miniature diamond anvil cell (DAC) designed for use in MPMS (Quantum Design Co.) [14].

3. Results and discussions

The temperature dependence of resistivity at high pressures in La-1111 is shown in Figure 1. The sample shows sharp transition and zero resistivity at low pressure. In La-1111, the onset $T_c$ is enhanced at first from $\sim$30 K to $\sim$35 K. Above a pressure of $\sim$4.2 GPa, $T_c$ starts to decrease. This result is consistent with a previous study [15].

![Figure 1. The electrical resistivity of polycrystalline LaFeAO$_{0.65}$ (nominal composition) under various pressures. Superconductivity survives at the highest pressure of 14.5 GPa. After the measurement at 14.5 GPa, we reduced load to check the reproducibility (3 ton).]}
However, the zero resistivity temperature is seen to decrease monotonously on applying pressure. This result clearly suggests that there is no enhancement in ‘bulk’ \( T_c \) in La-1111 due to pressure. Furthermore, no enhancement of \( T_c \) in La-1111 is observed in magnetization measurements \([16]\). We also confirmed the reproducibility of our measurement. After reducing applied load, the zero resistivity temperature and resistivity value are almost completely recovered. However, onset \( T_c \) is still high and the transition curve becomes broad, as shown in Figure 1 (see the result of ‘3 ton’).

Figure 2 shows the resistivity results in Tb-1111 up to 13 GPa. \( T_c \) is decreased by the application of pressure. Above 6.5 GPa, \( T_c \) is decreased to lower than 4.2 K. Although the resistivity drop remained in higher pressure, bulk superconductivity clearly vanished. AC magnetic susceptibility data also supports the resistivity results. The Meissner signal also disappeared above 6.5 GPa as shown in Figure 3. In the susceptibility measurements, the transition temperatures are relatively higher than that of the resistivity measurements data because the sample was improved \([6]\). But pressure dependence of \( T_c \) is qualitatively the same. Nd-1111 also shows negative pressure dependence of \( T_c \) \([9]\), but it is clear that superconductivity in Tb-1111 is much more sensitive to external pressure than that in Nd-1111.

![Figure 2](image1.png)\(\rho(\text{m}\Omega\text{cm})\) vs. \( T (\text{K}) \) for TbFeAsO\(_{0.7}\) (nominal composition) at high pressures. Bulk superconductivity vanishes above \~6.5 GPa.

![Figure 3](image2.png)\(\chi [\text{emu}]\) vs. \( T [\text{K}] \) for Tb-1111 at various pressures. At 6.5 GPa, it becomes difficult to confirm the Meissner signal from the sample.
We obtained a $T_c$-Pressure phase diagram of $Ln$-1111 ($Ln$ = La, Ce [12], Nd [9], Tb), as shown in Figure 4. By defining superconducting transition temperatures as the temperature at which zero resistivity occurs, we can conclude that the $T_c$ of all the compounds show monotonic and negative pressure dependence. In La-1111, spontaneous randomness of the structure occurs when hydrostatic pressure is applied to the crystal. This randomness induces a ‘pseudo’ enhancement of $T_c$. However, it is not a bulk behavior. Negative pressure dependence of $T_c$ is an intrinsic property in La-1111. Ce-1111 is a special case [12] in the $Ln$-1111 family. Owing to the pressure-induced enhancement of correlation to 4f electrons in the Ce ion, superconductivity suddenly vanishes at around 4 GPa. Nd-1111 and Tb-1111 shows similar behavior. Both show negative pressure dependence of $T_c$, and superconductivity in Nd-1111 is more robust for external pressure when compared to Tb-1111.

![Figure 4. Obtained $T_c$-Pressure phase diagram of $Ln$-1111 ($Ln$ = La, Ce [12], Nd [9], Tb). Because of the quality of the sample, $T_c$ of Tb-1111 are different between the electrical resistivity results and the magnetic measurements. However, the superconductivity vanishes above ~6.5 GPa in both observations.](image)

The pnictogen height, which is the distance between the arsenic atom and iron square lattice, has been considered as one of the potential factor [17] that determines $T_c$. In this model, there is an optimum value of height to explain the highest value of $T_c$ in $Ln$-1111. Tb-1111 and Nd-1111 has nearly the same $T_c$ and anion height [17]. However, pressure dependence of $T_c$ is very different between these two materials. Therefore, this model would not be adequate. The bond angle [7,18] is another strong candidate as $T_c$ determinative factor, but we cannot elucidate the effect of the bond angle from this study. It is not clear how the bond angle behaves at high pressure. Precise structural study in hydrostatic high pressure and low temperature is required. Accurate information of atom positions at high pressure is needed to determine the key parameter affecting $T_c$ in $Ln$-1111. Since all $Ln$-1111 samples show monotonic negative pressure dependence, a very simple parameter such as the volume of iron arsenic tetrahedron could be one of the $T_c$ determinative factors. Since La-1111, which has the largest volume, does not exhibit the highest $T_c$, the volume of the iron arsenic tetrahedron is not obviously a unique parameter. There would be some independent key factor for determining $T_c$. For example, the bond angle and the volume of iron arsenic tetrahedron are the independent parameter in $Ln$-1111.

In summary, the electrical resistivity of a high-quality $Ln$-1111 ($Ln$ = La, Ce, Nd, Tb) polycrystalline sample was measured at hydrostatic pressures up to 18 GPa. All compounds show negative pressure dependence for $T_c$. La-1111 exhibits spontaneous randomness of structure under pressure, which causes a pseudo enhancement of the onset temperature for superconductivity. Since
superconductivity in Tb-1111 is very sensitive to the applied pressure, its $T_c$ is suppressed, whereas superconductivity in Nd-1111 survives up to a high pressure of 18 GPa.

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