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Hall and thermoelectric-power coefficients of superconducting $Nd_2CuO_{4-x}F_x$

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Both the Hall coefficient (R_H) and the thermoelectric-power coefficient (S) have been measured using the same polycrystalline samples of superconducting $Nd_2CuO_{4-x}F_x$ ($0 \leq x \leq 0.3$). For the samples with $0.1 \leq x \leq 0.3$, the R_H of every sample is found to be negative in the temperature range between 30 and 275 K. Furthermore, as x increases, the charge-carrier density (p_H) estimated from R_H at 100 K increases in proportion to x ($p_H = x$) up to around 0.18, then much more rapidly for $x \geq 0.18$. On the other hand, the S of every sample is also found to be negative in the temperature range between 30 and 300 K. The absolute value of S is found to reduce with x up to around 0.18 and then level off to a constant value as x increases further to 0.3. The decrease in $|S|$ due to F doping is discussed using a semiconductor model. In addition, we point out a possibility that a magnon drag plays a significant role in S for samples with $x < 0.18$.

I. INTRODUCTION

The occurrence of superconductivity in $L_2CuO_{4-x}F_x$ (L=Nd and Pr) (Ref. 1) exhibited the possibility of a control of charge-carrier density by anion substitution in the high-T_c superconducting cuprates. Nevertheless, at this moment, superconductivity caused by anion substitution has been observed only in these compounds.

Recently, several reports[2–6] have shown that the structural and physical properties of $Nd_2CuO_{4-x}F_x$ were parallel to those of $Nd_{2-y}Ce_yCuO_4$. That is, the Hall coefficient (R_H) of $Nd_2CuO_{4-x}F_x$ (x=0.1, 0.16, 0.2, and 0.3) was negative in the temperature range between 30 and 275 K.[3] In addition, the temperature dependence of R_H was similar to the case of $Nd_{2-y}Ce_yCuO_4$.[7] Hence this compound was considered to be an "n-type" superconductor. However, to our knowledge, all the data for $Nd_2CuO_{4-x}F_x$ were measured using polycrystalline samples. Thus the data might include the effects of grain boundaries and/or coexisting secondary phases.

On the other hand, it is well known that the thermoelectric power (S) is often less affected by sample imperfections. Additionally, the analysis of the temperature dependence of S in the normal state can provide information on the scattering mechanism of the charge carriers. Therefore the measurement of S is one of the essential experiments to investigate changes in the charge-carrier density and electronic structure due to F doping. Nevertheless, to our knowledge, the data of S for $Nd_2CuO_{4-x}F_x$ have not been reported, except that James, Zahurak, and Murphy mentioned[1] that the values of S were negative at room temperature. For $Nd_{2-y}Ce_yCuO_4$, some groups reported the results of the S measurements.[8,9] For all the samples, S was small and nearly independent of temperature in the range between 100 and 250 K, although either sign of S were observed.

Furthermore, the detailed relationship between R_H and fluorine content is useful for understanding the change in the charge-carrier density, even for polycrystalline samples. In this paper we report both the Hall and thermoelectric-power coefficients measured using the same samples of polycrystalline superconducting $Nd_2CuO_{4-x}F_x$. In addition, we discuss the relationship between the charges in R_H and S due to F doping.

II. EXPERIMENT

Polycrystalline samples of $Nd_2CuO_{4-x}F_x$ (0 and $0.1 \leq x \leq 0.3$) were synthesized by a conventional solid-state reaction technique. Stoichiometric mixtures of the starting materials, such as Nd_2O_3, CuO, and NdF_3, were mixed throughly by a planetary ball mill using ethanol as solvent. After drying, the mixtures were calcined at 860 °C for 10 h in a 20% O_2-Ar gas mixture flow. After regrinding, the calcined power was pressed into rectangular bars and sintered at 1100 °C for 14 h in a 1% O_2-Ar gas mixture flow. Then the samples were annealed at 910 °C for 14 h in a nitrogen gas flow and furnace cooled to room temperature at a rate of about 5 °C/min.

From powder x-ray-diffraction (XRD) analysis,[2] all the samples with $x < 0.3$ were found to be a single phase of the Nd_2CuO_4-type tetragonal structure. On the contrary, the diffraction patterns for the samples with $x \geq 0.3$ included weak peaks due to NdOF and CuO and unidentified phases besides those for the $Nd_2CuO_{4-x}F_x$ phase. Thus the maximum fluorine solubility was found to be $x_{cr}=0.3$. Electron-probe-microanalysis (EPMA) observations supported these results. The chemical analysis for the fluorine content in the samples with x = 0.1, 0.2, 0.5, 1.0, and 2.0 showed that the F content of each sample was reduced to ~85% of the nominal content after heat treatment of the sample.[3] (Henceforth, the quantity x always represents a nominal composition.) Electrical-resistivity and magnetic-susceptibility measurements indicated that the values of $T_{c,\text{on}}$ were ~27 K for the samples with $x \geq 0.12$, though the zero-resistance states were observed only for the samples with $x \geq 0.2$. The highest value of $T_{c,\text{zero}}$ was observed at 22 K for the sample with $x = 0.22$. The preparation and structural and superconducting properties of the samples were described in detail elsewhere.[2,3]

Hall-coefficient measurements were carried out for thin rectangular plate samples in a constant magnetic field of $H = 8$T at temperatures between 20 and 275 K. The sample dimensions were about 120 μm thickness, 2.5 mm width, and 6 mm length. Electrical contacts with resistance under 1 Ω were made with indium using an ultrasonic soldering iron. The dc-current density, applied within the plate, was about 20 A/cm^2.

The thermoelectric-power coefficient was measured by a dc method in the temperature range between 30 and 310 K using the same sample for the Hall-coefficient measurement. The temperature gradient (ΔT) in the sample was measured using the two pairs of copper-Constantan thermocouples. These thermocouples were attached by silver epoxy on the indium electrodes on the sample. The magnitude of ΔT was kept 0.3–0.4 K during the measurement. To eliminate the effects of the reference leads (Cu wires), the absolute thermoelectric power of Cu was subtracted from the measured thermoelectric voltage.

III. RESULTS

A. Resistivity and Hall coefficient

The temperature dependences of the resistivity (ρ) and the Hall coefficient (R_H) are shown in Figs. 1(a)–1(d) for the $Nd_2CuO_{4-x}F_x(0.1 \leq x \leq 0.3)$ samples. The value of R_H for every sample is negative in the temperature range between 50 and 275 K; furthermore, the magnitude of $|R_H|$ decreases with increasing x [Figs. 1(b) and 1(d)]. This behavior is in good agreement with the result of ρ measurements [Figs. 1(a) and 1(c)]; that is, ρ reduces with x, indicating an increase in the charge-carrier density caused by F doping. In addition, for the samples with $x < 18$, $|R_H|$ of every sample increase monotonically as temperature lowers [Fig. 1(b)]. On the other hand, for the samples with $x \geq 0.18$, $|R_H|$ of every sample exhibits a broad peak around 100 K [Figs. 1(b) and 1(d)]; that is, as the temperature lowers, $|R_H|$ increases down to about 100 K, and then decreases; in particular, for the samples with $x = 0.24$ and 0.3, both R_H's rise steeply and then become positive at temperatures below ~40 K.

Figures 2(a) and 2(b) show the temperature dependences of ρ and R_H for three samples of $Nd_2CuO_{3.8}F_{0.2}$ prepared from the same calcined power in different sintering runs. Although every sample exhibits a sharp superconducting transition at ~27 K, the R_H-vs-T curve seems extremely sensitive to the samples; that is, for sample Nos. 1 and 2, R_H is negative at all temperatures below 275 K; on the other hand, for sample No. 3, R_H becomes positive at the temperatures below 30 K. This result is similar to the case for the single-crystal samples of $Nd_{1.85}Ce_{0.15}CuO_4$.[10]

The relationship between R_H and $1/x$ is shown in Fig. 3. In this figure solid and open circles represent the data for R_H at 275 and 100 K, respectively. Each $|R_H|$ at 275

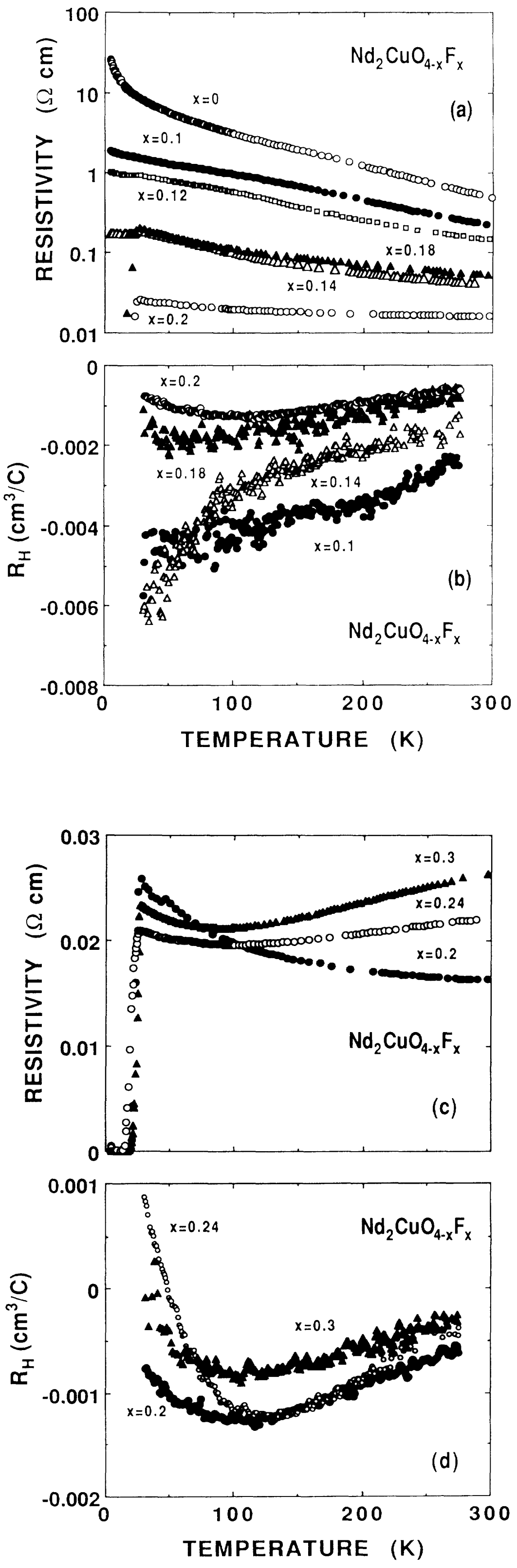

FIG. 1. Temperature dependence of resistivity (ρ) and Hall coefficient (R_H) for $Nd_2CuO_{4-x}F_x$: (a) ρ vs T for the samples with $0 \leq x \leq 0.2$, (b) R_H vs T for the samples with $0.1 \leq x \leq 0.2$, (c) ρ vs T for the samples with $0.2 \leq x \leq 0.3$, and (d) R_H vs T for the samples with $0.2 \leq x \leq 0.3$.

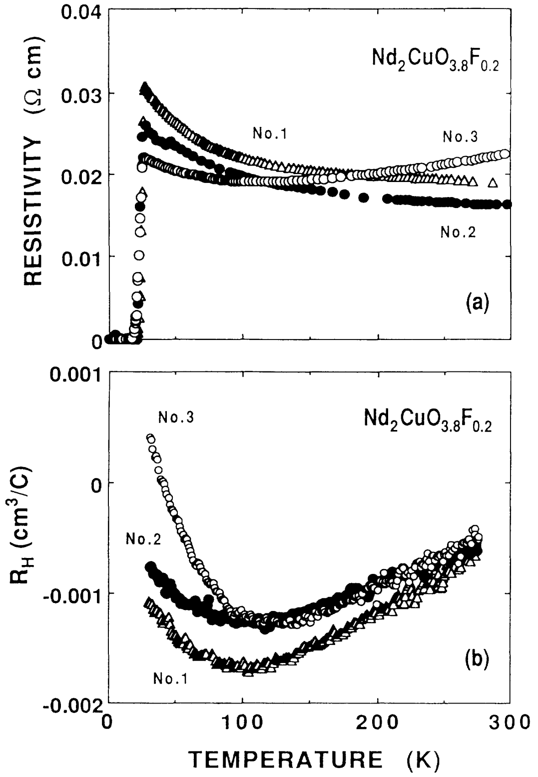

FIG. 2. (a) Resistivity and (b) Hall coefficient as a function of temperature for three $Nd_2CuO_{3.8}F_{0.2}$ samples.

and 100 K seems to increase monotonically with $1/x$; this suggests that the number of mobile charge carriers increases monotonically with x. this also indicates that charge carriers (holes or electrons) are doped into the Mott-Hubbard insulator as a result of F doping, as has been proposed[11] for $La_{2-z}Sr_zCuO_4$ and $Nd_{2-y}Ce_yCuO_4$.

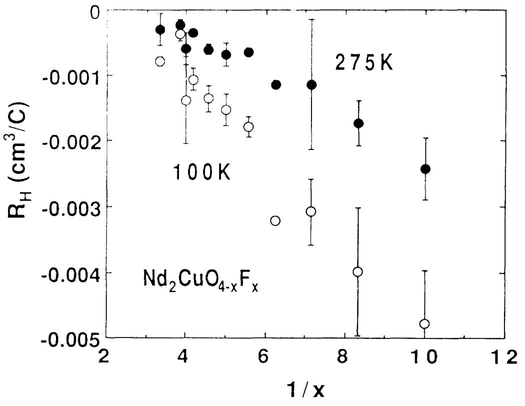

FIG. 3. (a) Relationship between Hall coefficient and inverse fluorine content ($1/x$) for the $Nd_2CuO_{4-x}F_x$ samples. The solid and open circles represent the data at 275 and 100 K, respectively.

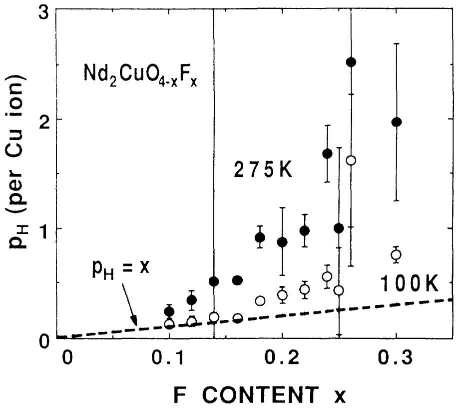

FIG. 4. Relationship between the normalized Hall density (p_H) and fluorine context (x) for the $Nd_2CuO_{4-x}F_x$ samples. The solid and open circles represent the data at 275 and 100 K, respectively. The dashed line represents the relation $p_H = x$.

Figure 4 shows the relationship between the normalized charge-carrier density (p_H) and x. Here p_H is defined as

$$p_H = \frac{V}{|R_H|eN} , \tag{1}$$

where e is the elementary charge, V is the unit-cell volume, and N is the number of Cu ions per unit cell. Using the values of $V = 0.18926$ nm³ and $N = 2$ for Nd_2CuO_4,[12] we can calculate the magnitude of p_H. As x increases, each p_H of 275 and 100 K increases linearly up to around 0.18 and then much more rapidly for $x > 0.18$. If one doped F ion introduces one free electron into the sample, p_H should be equivalent to x; at least in the range with $x < 0.18$ at 100 K, the experimental data are in good agreement with the relation $p_H = x$. On the other hand, the change in the slope (dp_H/dx) at $x \sim 0.18$ may indicate the change in the electronic structure, implying the transition from an insulator to a metal around $x = 0.18$.

B. Thermoelectric power

Figures 5(a)–5(c) show the temperature dependence of thermoelectric power (S) for the samples with $0 \leq x \leq 0.3$. The value of S of every sample is found to be negative in the temperature range between 30 and 300 K; furthermore, $|S|$ reduces with x. Hence this indicates that the dominant charge carriers are assigned as electrons.

For the samples with $x \geq 0.1$, $|S|$ of every sample rises as the temperature lowers and then reduces steeply; that is, $|S|$ exhibits a broad peak around 120 K for the sample with $x = 0.1$. It is worth noting that the temperature at which $|S|$ exhibits a broad peak lowers with x [Fig.5(b)]. Even for the undoped sample, there may exist a broad peak around 220 K in the S-vs-T curve [Fig. 5(a)]. For the samples with $x \geq 0.2$, $|S|$ of every sample seems to be nearly independent from x [Fig. 5(c)]. As the temperature lowers, $|S|$ decreases linearly at temperatures above ~ 150 K, rises slightly in the temperature range between 150 and 60 K, and then reduces rapidly and changes sign. The temperature dependence of S will be discussed later.

The relationship between S and x is shown in Fig. 6(a); as x increases, each $|S|$ at 275 and 100 K decreases steeply and then levels off to constant values above $x = 0.2$.

According to the negative values of $d\rho/dT$, R_H, and S, we assume that the normal states of the samples with $x<0.2$ are of an n-type semiconductor; then S is expressed as

$$S=-\frac{k_B}{e}\left[\ln\left[\frac{N_0(T)}{n_e}\right]+\frac{5}{2}+r\right],$$
$$N_0(T)=2(2\pi m_e k_B T/h^2)^{3/2}, \tag{2}$$

where k_B is the Boltzmann constant, n_e is the number density of electrons in the conduction band, m_e is the electron rest mass, and h is the Planck constant, respectively; the coefficient r is expected to be approximately constant. Indeed, as shown in Fig. 6(b), $|S|$ seems to increase in proportion to $\ln(1/x)$ in the range between $1/x=6$ and 10 $(0.1\leq x\leq 0.2)$; therefore the decrease in $|S|$ due to F doping indicates an increase in the charge-carrier density. This is in good agreement with the result of Hall-coefficient measurements (Fig. 4). On the other hand, for each sample with $x\geq 0.2$, the temperature dependence of S may be explained using a normal-metal model, as will be discussed later.

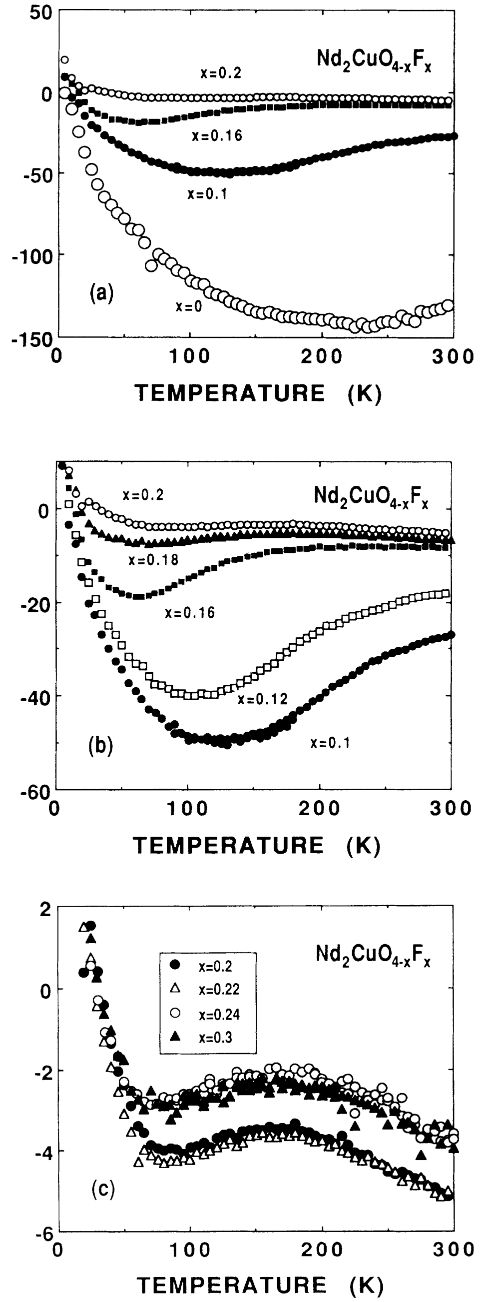

FIG. 5. Temperature dependence of thermoelectric power for the Nd$_2$CuO$_{4-x}$F$_x$ samples: (a) $0\leq x\leq 0.2$, (b) $0.1\leq x\leq 0.2$, and (c) $0.2\leq x\leq 0.3$.

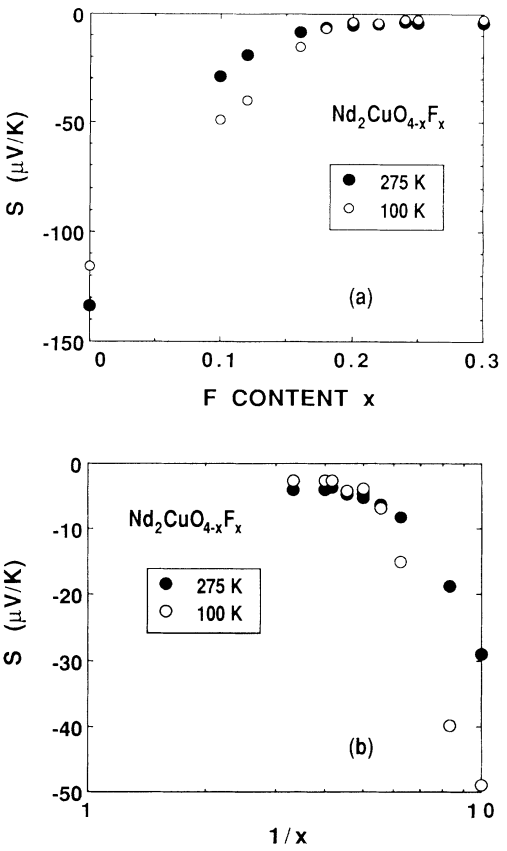

FIG. 6. Relationship between thermoelectric power (S) and fluorine content (x) for Nd$_2$CuO$_{4-x}$F$_x$: (a) S vs x and (b) S vs $\ln(1/x)$. The solid and open circles represent the data at 275 and 100 K, respectively.

IV. DISCUSSION

A. Hall coefficient

Assuming that the conduction is done by electrons, we estimated the charge-carrier density (p_H) using Eq. (1). However, as already mentioned, the result indicates that the doped F ions not only work as "electron donors," but also alter the electronic structure. For the Ce- or F-doped samples, recent calculations of the electronic structure using the tight-binding approximation[13,14] showed that the Fermi level (E_F) increased linearly with x and the density of states at E_F decreased with the shift in E_F. In addition, the present R_H data for $Nd_2CuO_{4-x}F_x$ $(0.12<x<0.2)$ at 275 K seem to be in good agreement with the calculated R_H (J$\|ab$, H$\|$c) for $Nd_{2-y}Ce_yCuO_4$ (in the superconducting and metallic region, such as $0.12<y<0.19$).[15] These suggest the existence of the transition from an electron-doped Mott-Hubbard insulator to a normal metal with increasing x.

For the high-temperature limit of a Mott-Hubbard insulator with a strong electron-electron repulsion, the relationship between S and p_H is expressed as[16]

$$S(T\rightarrow\infty)=-\frac{k_B}{e}\ln\frac{2(1-p_H)}{p_H} . \tag{3}$$

Assuming that $p_H=x$ from the result of the R_H measurements, we can estimate $S(T\rightarrow\infty)$ using Eq. (3). However, as shown in Fig. 7, the calculated $S(T\rightarrow\infty)$ is about 10 times larger in magnitude than the measured S(275 K), although the x dependence of S(275 K) seems to be parallel to that of $S(T\rightarrow\infty)$; this indicates the following possibilities: (a) p_H derived from R_H ($p_H=x$ for the samples with $x<0.18$) is not always equivalent to the number density of mobile carriers because of such effects as the grain boundaries and coexisting secondary phases which were not detected by XRD analysis, (b) the temperature (275 K) is not high enough for the high-temperature limit, or (c) the system is not a Mott-Hubbard insulator with a strong electron-electron repulsion.

B. Thermoelectric power

Thermoelectric power (S) consists of three main processes:[17,18] conduction-electron thermal diffusion (S^d); phonon drag, which implies that in their streaming motion the phonons can carry conduction electrons along (S^p); and finally magnon drag (S^m). As a first approximation, the contributions are treated as independent of each other and are added together:

$$S=S^d+S^p+S^m . \tag{4}$$

For metals with a spherical Fermi surface, S^d is expressed as the well-known Mott formula

$$S^d=\pi^2k_B^2T/(3eE_F) , \tag{5}$$

where E_F is the Fermi energy. For free electrons, S^d is negative and increases linearly with T in absolute value. Indeed, for every sample with $x>0.2$, an approximately linear relationship between S and T is observed at temperatures above 180 K [Fig. 5(c)]; thus, in the temperature range above 180 K, S^d is considered to be the predominant factor in Eq. (4).

For n-type semiconductors, S^d is rewritten using Eq. (2) and is equal to

$$S^d=-\frac{k_B}{e}\left[\frac{E_C-E_F}{k_BT}+\frac{5}{2}+r\right] , \tag{6}$$

in which E_C is the upper level of the conduction-band energy. Assuming that E_C-E_F is independent from T, $|S^d|$ increases with $1/T$. Figures 8(a) and 8(b) show S as a function of $1/T$ for the samples with $x<0.18$; the linear $1/T$ dependence of S is observed for each sample with $x=0$, 0.1, 0.12, and 0.16 at temperatures above about 240, 170, 140, and 80 K, respectively; however, for the sample with $x=0.18$, S is found not to be proportion-

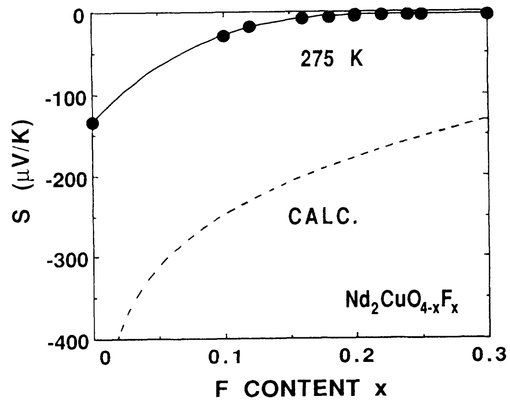

FIG. 7. Relationship between thermoelectric power (S) and fluorine content (x) for $Nd_2CuO_{4-x}F_x$: The solid circles represent the measured data at 275 K; the dashed line represents the calculated dependence of S on x using Eq. (3) and the relation $p_H=x$.

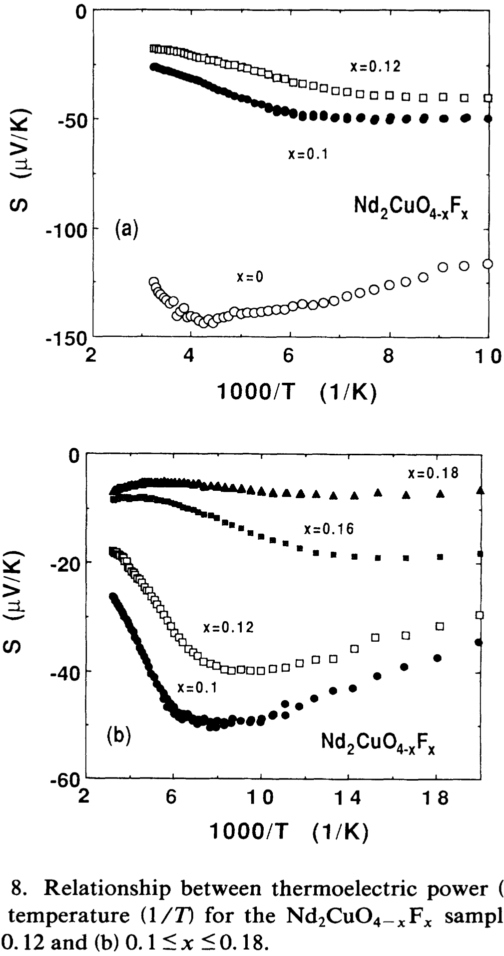

FIG. 8. Relationship between thermoelectric power (S) and inverse temperature ($1/T$) for the $Nd_2CuO_{4-x}F_x$ samples: (a) $0\leq x\leq0.12$ and (b) $0.1\leq x\leq0.18$.

al to $1/T$. Consequently, the samples with $x \leq 0.16$ are considered to be *n*-type semiconductors.

Now, we discuss why S exhibits a complicated temperature dependence. As already described, $|S|$ has a broad peak in the S-vs-T curve [Figs. 5(a) and 5(b)]; in addition, the temperature at which $|S|$ shows a broad peak lowers with increasing x. Theoretical investigations[19] indicate that a phonon- and magnon-drag peak should be observed in the range $\Theta_D/10-\Theta_D/5$ and $T_N/10-T_N/5$, respectively, where Θ_D is the Debye temperature and T_N is the Néel temperature. Furthermore, for the rare-earth metals, S shows changes in slope at T_C and T_N in the S-vs-T curve,[17] where T_C is the ferromagnetic Curie temperature.

The S^p term seems to be insignificant in Eq. (4) at temperatures above 30 K, because the magnitude of Θ_D is generally not sensitive to the dopant concentration. Of course, at temperatures below 30 K, a phonon drag may contribute as a predominant term in Eq. (4); this is because $|S|$ decreases steeply at temperatures below 30 K for every sample; specific-heat measurement[20] showed that the value of Θ_D for single-crystal Nd_2CuO_4 was estimated to be ~ 319 K; hence the phonon-drag peak may be observed at around 30 K. On the other hand, neutron- and x-ray-diffraction analyses indicated[21] that there were no structural phase transitions in the temperature range between 320 and 4.2 K in Nd_2CuO_4 and $Nd_{2-y}Ce_yCuO_4$. Although a structural analysis of the F-doped samples at low temperatures has not been yet reported, all the F-doped samples are considered to belong to the Nd_2CuO_4-type tetragonal structure at temperatures below 310 K; thus the change in slope in the S-vs-T curve is not due to the structural phase transition.

For Nd_2CuO_4, long-range antiferromagnetic ordering of the Cu moments was reported at $T_{N1} \sim 250$ K by neutron-diffraction analyses.[22,23] In addition, two more magnetic phase transitions associated with the Cu-moment reorientations were observed[19,20] at $T_2 \sim 80$ K and $T_3 \sim 30$ K; furthermore, a sharp transition to long-range antiferromagnetic ordering of the Nd moments was observed at $T_{N2} \sim 1.2$ K.[24] For $Nd_{2-y}Ce_yCuO_4$, as the Ce content (y) increased, T_{N1} lowered and then became undetectable at the temperatures above 4 K in the superconducting composition range by a muon-spin-rotation relaxation measurement.[25] Although the dependence of T_2 and T_3 on y have not been reported yet, T_{N2} was found to be nearly independent of y.[24]

For $Nd_2CuO_{4-x}F_x$, to our knowledge, magnetic ordering has not been yet investigated. Thus we assume that the relationship between T_{N1} (and T_{N2}) and x is similar to the case of $Nd_{2-y}Ce_yCuO_4$. Provided T_{N1} lowers with increasing x, there is a possibility that a magnon drag is closely related to the change in slope in the S-vs-T curve; indeed, for the nondoped sample, the temperature at which S exhibits a change in slope [~ 240 K, as shown in Figs. 5(a) and 8(a)] is approximately same as for T_{N1}(~ 250 K); also three other magnetic phase transitions (T_2, T_3, and T_{N2}) may be related to the temperature dependence of S. Hence it is considered that S^m plays the significant role in Eq. (4) at temperatures below T_{N1} for the samples which are an *n*-type semiconductor ($x \leq 0.16$).

For the samples ($x \geq 0.2$) which are of a normal metal, the long-range antiferromagnetic ordering between Cu moments is unlikely to exist, similar to the case of $Nd_{2-y}Ce_yCuO_4$.[25] Thus the temperature dependence of S for each sample with $x \geq 0.2$ is expected to be free from the effects of a magnon drag; however, as shown in Fig. 5(c), S of every sample exhibits changes in slope around 180 and 80 K. In order to explain these changes in S, we must introduce another model, such as a multiband model[26] or a two-dimensional tight-binding model.[27] Also, there is a possibility that the phase separation occurs in the $Nd_2CuO_{4-x}F_x$ phase diagram, as has been proposed[28] for $Nd_{2-y}Ce_yCuO_4$ and $La_{2-z}Sr_zCuO_4$. If the samples with $x \geq 0.2$ consist of the multiple phases with various F content, the effects of S^m should not be ignored.

V. SUMMARY

We reported both the Hall and thermoelectric-power coefficients measured using the same polycrystalline samples of superconducting $Nd_2CuO_{4-x}F_x$ ($0.1 \leq x \leq 0.3$). The Hall coefficient (R_H) was found to be negative at room temperature. The temperature dependence of R_H was similar to that of the polycrystalline *n*-type superconductor $Nd_{2-y}Ce_yCuO_4$. Furthermore, as x increased, the charge-carrier density (p_H) estimated from R_H at 100 K increased in proportion to x up to around 0.18 and then much more rapidly for $x \geq 0.18$. On the other hand, the thermoelectric-power coefficient (S) was also found to be negative in the temperature range between 30 and 300 K. In addition, $|S|$ decreased with increasing x. The change in S due to F doping was discussed using the *n*-type semiconductor model for the samples with $x < 0.18$. In addition, the effect of a magnon drag was discussed in order to explain the temperature dependence of S, especially for the samples with $x < 0.18$.

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