Unconventional Superconductivity in Electron-Doped Layered $Li_{0.48}(THF)_yHfNCl$

H. Tou,[1] Y. Maniwa,[1] T. Koiwasaki,[2] and S. Yamanaka[2,3]

[1]*Department of Physics, Tokyo Metropolitan University, Minami-osawa, Hachi-oji, Tokyo 192-0397, Japan*
[2]*Department of Applied Chemistry, Faculty of Engineering, Hiroshima University, Higashi-Hiroshima 739-8527, Japan*
[3]*CREST, Japan Science and Technology Corporation (JST), Toshima-ku, Tokyo 171-0031, Japan*

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We report magnetic susceptibility measurements on a layered superconductor $Li_{0.48}(THF)_{0.3}HfNCl$ having $T_c \sim 26$ K. The present study revealed that (a) the Fermi level density of states is small, $N^*(E_F) \sim 0.25$ states/(eV spin f.u.), (b) mass enhancement is negligible, $\tilde{\gamma} \sim 1$, (c) electron-phonon coupling is weak, $\lambda_{ep} \ll 1$, (d) exchange enhancement is negligible, $1/(1 + F_0^a) \sim 1$, and (e) electronic density parameter is large, $r_s^{2D} \sim 10.3$ (i.e., low-carrier density). It is difficult to explain the origin of the high T_c in terms of the conventional phonon (BCS) mechanism of superconductivity.

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In 1998, Yamanaka and co-workers discovered a new layered superconductor $Li_x(THF)_yHfNCl$ (THF; tetrahydrofuran C_4H_8O) with $T_c \sim 26$ K [1]. The mother compound β-HfNCl is a semiconductor with a band gap of ~4 eV having layered structure stacking the Cl-[HfN]-[NHf]-Cl slabs along the c axis [1,2]. As shown in Fig. 1(a), Li atoms are cointercalated with THF molecules into the weak van der Waals gap between the Cl layers and the interlayer distance d increases from 9.23 Å in β-HfNCl to 18.7 Å in $Li_{0.48}(THF)_{0.3}HfNCl$ (Li-HfNCl) [1]. On intercalation, electrons are doped into the $(HfN)_2$ layer and the system shows superconductivity with a relatively high T_c of ~26 K [1].

Experimental and theoretical studies have clarified the variety of physical properties, as follows: (1) T_c of ~26 K exceeds 23 K of a conventional BCS superconductor, Nb_3Ge [3]. A question as to why such a high T_c is realized naturally arises. (2) Bulk superconductivity suddenly appears with $T_c \sim 26$ K at the doping content of $x \sim 0.13$. T_c is almost constant (~26 K) up to $x \sim 0.5$ but gradually decreases to $T_c \sim 24.4$ K with further doping [1], suggesting a low-carrier-density system. (3) Band calculations revealed a two-dimensional (2D) electronic structure originating in planer Hf-$d_{xy}, d_{x^2-y^2}$ orbitals strongly hybridized with N-p_x, p_y orbitals having broad bandwidths of $w \sim 1.5$ eV, suggesting a weakly correlated electron system [4–6]. (4) Actually, we reported the quasi-2D superconducting (SC) properties in Li-HfNCl from dc-magnetization and NMR measurements for c-axis oriented pellets [7]. (5) An exotic SC nature near the Bose-Einstein condensation limit was reported from muon spin relaxation measurements [8].

In the present work, the magnetic susceptibility measurements of the c-axis oriented Li-HfNCl pellets were carried out in the temperature (T) range of 4–300 K for magnetic fields applied parallel to the ab plane ($H \parallel ab$) and the c axis ($H \parallel c$). From analyses of the present data together with the previous results [7], we clarify the unsuccessful explanation of T_c within a traditional BCS framework.

Detailed sample preparation techniques for the powder Li-HfNCl sample were reported in Ref. [1]. The powder sample was compressed into a pellet of 4 mm in diameter and 0.5 mm in thickness under a pressure of 1.5 kbar in argon atmosphere. The sample was roughly cut into disks of ~2 mm in diameter and sealed in a quartz tube with helium gas at 350 torr [7]. X-ray locking curve measurements revealed that the c axis was aligned in a Gaussian distribution with a half-width of less than 3.5° in the pellet. We prepared two oriented pellets (No. 1 and No. 2). Sample No. 1 is the same batch as in the previous paper [7]. The static magnetic susceptibility was measured using a commercial SQUID magnetometer (MPMS, Quantum Design). In order to subtract the diamagnetism of the quartz tube, the susceptibility of the sample was obtained from subtraction between two measurements for the tube containing the sample and the "empty" tube itself.

In Fig. 1(b), we show the dc-magnetic susceptibility data (No. 1) of Li-HfNCl for the zero-field cooling protocol, where the Meissner shielding occurs at $T_c = 25.56$ K. An evident difference in susceptibilities for $H \parallel ab$ and

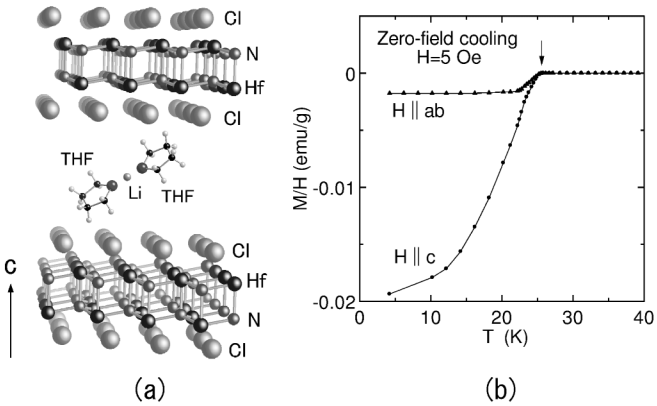

FIG. 1. (a) Schematic structural model and (b) dc-magnetic susceptibility of $Li_{0.48}(THF)_{0.3}HfNCl$ with field ($H = 5$ Oe) parallel to the c axis ($H \parallel c$) and to the ab plane ($H \parallel ab$). Arrow shows T_c(= 25.56 K) for $H = 5$ Oe.

$H \parallel c$ reflects the anisotropic nature of the SC state due to the layered structure [7]. The volume fraction of the SC state for $H \parallel c$ at 4 K is ~95% (~78% for No. 2) of the theoretical value $-1/4\pi$. Here, the correction on the demagnetizing field was performed assuming the disk.

The T dependence of susceptibility (χ_0) of Li-HfNCl (No. 1) for $H \parallel ab$ and $H \parallel c$ is shown in Fig. 2. A typical magnetization curve of Li-HfNCl (No. 1) for $H \parallel ab$ is shown in the inset. A ferromagnetic background corresponds to ~150 ppm spins per formula unit (f.u.) independent of field direction, which should be due to impurity domains. Therefore we determined χ_0 from the difference between the values at $H = 40$ and 20 kOe in order to subtract the ferromagnetic background. For sample No. 2, χ_0 agrees with that for No. 1 within the accuracy of $\pm 2 \times 10^{-6}$ emu/mol. The susceptibility of β-HfNCl is also shown, represented by a dashed line, which gives a Curie tail corresponding to 0.15% spins/f.u.

A negative value of χ_0 implies that the Pauli susceptibility χ_s is of the order of the diamagnetic susceptibility $\chi_{\rm dia}$. Actually, the Pauli susceptibility becomes $\chi_s(T_c) \sim 1.7 \times 10^{-5}$ emu/mol at T_c, which is about one-tenth of that of conventional BCS superconductors, by subtracting the diamagnetism; $\chi_s = \chi_0^i - \chi_{\rm core} - \chi_L^i - \chi_{\rm orb}^i$ ($i = ab$ and c), where $\chi_{\rm core}$, χ_L^i, and $\chi_{\rm orb}^i$ are the core diamagnetic susceptibility, Landau diamagnetism, and orbital susceptibility, respectively. $\chi_{\rm core}$ is estimated from the increments of values, $\chi_{\rm Li^+}^{\rm core} \sim -6.0 \times 10^{-7}$ emu/mol, $\chi_{\rm HfNCl}^{\rm core} \sim -3.4 \times 10^{-5}$ emu/mol (estimated from the T-independent part of the pristine HfNCl), and $\chi_{\rm THF}^{\rm core} = -1.57 \times 10^{-5}$ ($y = 0.3$) emu/mol (calculated by the Pascal's method for organic molecules [9]). χ_L is given by $-(m/m^*)\chi_s/3$, where m is the free electron mass and m^* is the effective mass. Because the mass anisotropy is very large $\Gamma \sim 14$ [7], χ_L would be negligible for $H \parallel ab$, whereas $\sim\chi_s/3$ for $H \parallel c$. $\chi_{\rm orb}^{ab}$ and $\chi_{\rm orb}^{c}$ are estimated to be $\sim 10^{-6}$ and 0 emu/mol, respectively, using the prescription by Clogston *et al.* [10].

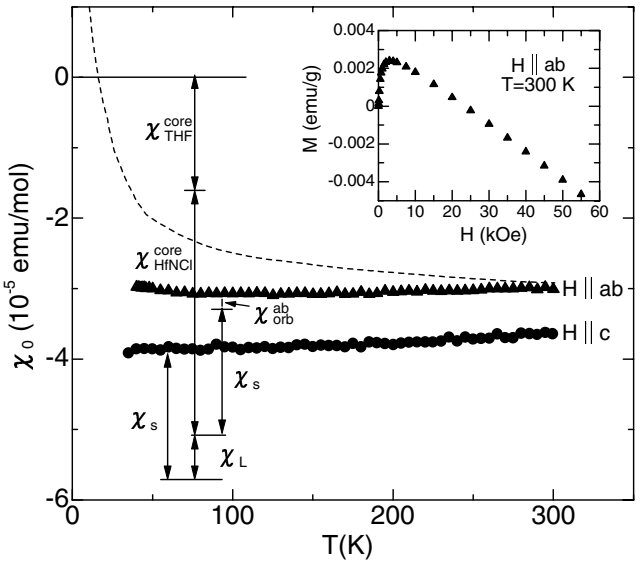

FIG. 2. Temperature dependence of susceptibility of $Li_{0.48}(THF)_{0.3}HfNCl$ for $H \parallel c$ (circles) and $H \parallel ab$ (triangles). The dashed curve is the susceptibility of β-HfNCl. The procedure of the decomposition of the parameters is given in the text. The inset gives the magnetization curve for $H \parallel ab$ at $T = 300$ K.

Now, we discuss the electronic properties within the Fermi liquid model [11], where the mass m is enhanced by $\tilde{\gamma} = m^*/m$ as a result of the electron-electron interaction. The renormalized Fermi level density of states $N^*(E_F) = N(E_F)\tilde{\gamma}$ can be estimated from the following relationship: $\chi_s = 2N_A\mu_B^2 N^*(E_F)/(1 + F_0^a)$, where $N(E_F)$ is the bare density of states and F_0^a is the Landau parameter. The obtained value for χ_s gives us $N^*(E_F)/(1 + F_0^a) \sim 0.26$ states/(eV spin f.u.).

Alternatively, we can estimate $N^*(E_F)$ from the thermodynamics in the SC state; $H_c(0)^2 V_{\rm mol}/(8\pi) = (F_n - F_s)_{T=0} \sim (1 + \lambda_{ep})N^*(E_F)\Delta^2/2$ with $V_{\rm mol} = 59.93$ cm^3/mol, where F_n and F_s are the free energy in the normal and SC states, respectively, and λ_{ep} is an electron-phonon coupling constant. Assuming the weak-coupling value of $2\Delta = 3.53 k_B T_c$, and using $H_c(0) = 845$ Oe [7], the effective density of states is calculated to be $(1 + \lambda_{ep})N^*(E_F) \sim 0.24$ states/(eV spin f.u.), where k_B is the Boltzmann factor. Correspondingly, the electronic specific heat coefficient γ_{el} is calculated to be 1.1 mJ/(mol K^2) from $\gamma_{el} = (1 + \lambda_{ep})(2/3)\pi^2 k_B^2 N^*(E_F)$.

These values are comparable to $N(E_F) \sim 0.19$–0.26 states/(eV spin f.u.) obtained from the band calculations [4–6]. Thus, $\tilde{\gamma}/(1 + F_0^a) \sim 1$ and $\tilde{\gamma}(1 + \lambda_{ep}) \sim 1$, implying negligible mass enhancement, $\tilde{\gamma} \sim 1$, weak electron-phonon coupling, $\lambda_{ep} \ll 1$ [12], and negligible exchange enhancement, $1/(1 + F_0^a) \sim 1$. These are consistent with the theoretical prediction, i.e., weak electronic correlation and weak electron-phonon coupling, by Weht *et al.* [6].

The obtained $N(E_F)$ is also very close to that of a 2D free electron gas model: $N^{\rm 2D}(E_F) = mS_{\rm mol}/2\pi\hbar^2 \sim 0.23$ states/eV with $S_{\rm mol} = 6.67 \times 10^8$ cm^2/mol, where $\hbar$ is the Planck constant. Together with the quasi-2D SC properties [7], the electronic state of Li-HfNCl may be approximately described in terms of a 2D electron gas model. The characteristic parameters in the 2D electron gas model [13] are listed in Table I. Here, the Fermi wave number k_F was estimated using the relations $\xi_0 \sim \hbar^2 k_F/(m^*\pi\Delta) \sim 1.35\xi_{\rm GL}$ in the clean limit BCS superconductor [7] and the specific heat coefficient for the 2D case $\gamma_{el}^{\rm 2D} = \pi k_B^2 m^* S_{\rm mol}/(3\hbar^2)$, where $\xi_{\rm GL} = \xi_{ab} \sim 60$ Å [7] and ξ_0 are the Ginzburg-Landau and BCS coherence lengths, respectively. Those for the 3D model, those of Nb, $Ba_{0.6}K_{0.4}BiO_3$ (BKBO) [14], and $YBa_2Cu_3O_7$ (YBCO) [15] were also calculated as references. The obtained value, $k_F \sim 1.3 \times 10^7$ (~1.6×10^7 for the 3D case) cm^{-1}, for the simple electron gas model is consistent in order of magnitude with the theoretical value of 1.95×10^7 cm^{-1} [6], suggesting that the model is applicable to the present system. We note that the estimated SC carrier

TABLE I. Characteristic parameters of Li-HfNCl obtained from the experiments and derived from the electron gas model [13], assuming $\langle\omega_{ph}\rangle = 300$ K. n_s for the 2D case is obtained as n_s^{2D}/d ($d = 18.7$ Å; the interlayer distance). Those for the 3D case were calculated using $\xi_{GL} = (\xi_{ab}^2\xi_c)^{1/3} \sim 38$ Å. As references, the parameters of Nb, BKBO, and YBCO have also been calculated assuming the 3D electron gas model [13]. The parameters used here are as follows: Nb: $2\Delta = 3.8k_BT_c$, $\xi_0 = 390$ Å, and $\langle\omega_{ph}\rangle = 166$ K; BKBO (dirty limit): $\gamma_{el} = 2$ mJ/mol K^2, $2\Delta = 4k_BT_c$, $\xi_0 = 1.83\xi_{GL}^2/l \sim 89$ Å, $\xi_{GL} \sim 40$ Å, $l \sim 33$ Å, $\langle\omega_{ph}\rangle \sim 550$ K [14]; YBCO: $\gamma_{el} \sim 40$ mJ/mol K^2, $2\Delta/k_BT_c \sim 6$, and $\xi_{GL} = (\xi_{ab}^2\xi_c)^{1/3} \sim 7$ Å [15].

Parameters	Li-HfNCl		Nb	BKBO	YBCO
T_c (K)	25.5		9.3	30	92
γ_{el} (mJ/mol K^2)	1.1		7.8	~2	~40
$N(E_F)$ (states/eV)	0.25		0.5	~0.3	~3
Model	2D	3D	3D	3D	3D
T_F (K)	735	596	18 400	3430	3350
k_F (10^7 cm^{-1})	1.29	1.63	17.9	3.95	8.23
n_s (10^{20} cm^{-3})	1.42	2.19	1940	20.9	190
r_s	10.3	22.3	2.0	13.8	(44)
k_{TF} (10^7 cm^{-1})	37.8	6.3	20.8	11.95	(44.5)
μ^*	(0.35)	(0.34)	0.11	0.23	···
λ_{ep}	(2.42)	(2.38)	0.91	1.3	···

density n_s is quite low $\sim 1.4 \times 10^{20}$ cm^{-3} in comparison with the value calculated from the valency, similarly to the case for the organic superconductor (BEDT-TTF)$_2$-X [16]. Corresponding to the low-carrier density, $T_F^{2D} \sim 735$ K is also relatively low, which is in good agreement with the value of $T_F^{2D} = 987$ K obtained by μSR measurement [8].

The remarkable findings in the present studies are summarized as follows: (a) the Fermi level density of states is small, $N(E_F) \sim 0.25$ states/(eV spin f.u.), (b) the mass enhancement is negligible, $\tilde{\gamma} \sim 1$, (c) the electron-phonon coupling is weak, $\lambda_{ep} \ll 1$, (d) the exchange enhancement is negligible, $1/(1 + F_0^a) \sim 1$, and (e) the electronic density parameter is large, $r_s^{2D} \sim 10.3$ (i.e., low-carrier density).

Why is such a high T_c realized in this system? In conventional BCS superconductors with high-carrier density $\sim 10^{23}$ cm^{-3}, we have the versatile prescription for T_c given by McMillan and Allen and Dynes [17]:

$$T_c = \frac{\langle\omega_{ph}\rangle}{1.2} \exp\frac{-1.04(1 + \lambda_{ep})}{\lambda_{ep} - \mu^*(1 + 0.62\lambda_{ep})}, \quad (1)$$

where $\langle\omega_{ph}\rangle$ and μ^* are the typical phonon energy and the Coulomb pseudopotential, respectively. This equation can be applied when the screening is sufficient to disregard the long-range Coulomb interaction and the Morel-Anderson model well describes retardation effects of the screened short-range Coulomb interaction [18]:

$$\mu^* = \mu/[1 + \mu \ln(\omega_{el}/\langle\omega_{ph}\rangle)], \quad (2)$$

with the typical screened Coulomb potential $\mu = (a^2/2)\ln[(1 + a^2)/a^2]$, where $a^2 = k_{TF}^2/(4k_F^2)$, k_{TF} is the Thomas-Fermi wave number. ω_{el} is the typical electron energy scale, e.g., the Fermi energy E_F or the plasma frequency ω_{pl} for a single band metal. In usual metals, it gives $\mu^* \sim 0.1$–0.13.

Since $\tilde{\gamma} \sim 1$ and $1/(1 + F_0^a) \sim 1$ for the present case, μ^* is expected to be small. If we assume $\mu^* = 0.1$ and $\langle\omega_{ph}\rangle = 300$ K, then we have $\lambda_{ep} \sim 1.17$ in a region of strong coupling limit by using Eq. (1). Alternatively, we can directly estimate μ^* by using Eq. (2). In the single band model, μ^* is calculated to be ~0.35 using the values listed in Table I. Then λ_{ep} turns out to be significantly large, $\lambda_{ep} \sim 2.42$ [19]. These are inconsistent with both the present result and the theoretical prediction [6], i.e., $\lambda_{ep} \ll 1$. Even if $\lambda_{ep} > 1.17$ is valid, the electron-phonon interaction $V_{ep} \sim \lambda_{ep}/N^*(E_F) > 4.7$ eV, which is much larger than the bandwidth $w \sim 1.5$ eV [4–6], would be too strong to realize the conventional BCS superconductivity, and instead may lead to lattice instability. Finally, we can assume $\mu^* = 0$, but even then the observed T_c cannot be attributed solely to the higher frequency modes, e.g., $\langle\omega\rangle_{ph} \sim 850$ K by nitrogen motion, as discussed by Weht *et al.* [6]. Anyhow, it seems to be quite difficult to explain the observed T_c within the framework of the conventional BCS mechanism.

Turning our attention to the relation $2k_F/k_{TF} < 1$ in Li-HfNCl, we expect that the screening effect is significantly different from that in usual metals. Unless the dielectric screening is sufficient ($\epsilon > 12$ for 2D case), the long-range Coulomb interaction should not be ignored. In this context, the McMillan-Allen-Dynes relation as well as the Morel-Anderson model probably cannot be applied anymore. What mechanism can be realized in this system?

A spin-fluctuation mechanism of superconductivity discussed in the strongly correlated electron system may be excluded because of no evidence for the strong electronic correlation, e.g., negligible mass enhancement $\tilde{\gamma} \sim 1$ and/or broad bandwidth of $w \sim 1.5$ eV. Takada, and Rietschel and Sham predicted that the charge fluctuation due to long-range Coulomb interactions (i.e., plasmon) is possible to mediate high T_c superconductivity in a low-carrier-density system when the electron-phonon coupling and short-range Coulomb interactions play a minor role [20]. In this case, both $\tilde{\gamma}$ and $1/(1 + F_0^a)$ are predicted to be of the order of unity. Our findings, (a)–(e), seem to meet the requirement for a charge-fluctuation mechanism of superconductivity [20].

Meanwhile, we note that the situation (the low-carrier-density, small density of states, and high T_c) is somewhat similar to the case for BKBO [14]. Thus comparisons with the physical properties in BKBO may help in understanding the unconventional superconductivity in Li-HfNCl.

In summary, we have performed magnetic susceptibility experiments on the layered superconductor, $Li_{0.48}$-$(THF)_{0.3}$HfNCl. These studies establish that Li-HfNCl is a low-carrier-density system. Taking account of the weak electron-phonon coupling and the small Fermi level den-

sity of states, it is difficult to explain the origin of the high T_c in terms of conventional electron-phonon coupling BCS superconductivity. Some other mechanisms beyond the conventional BCS theory should be sought as the origin of the high T_c in this system. A possible mechanism is a charge-fluctuation mechanism, although this may not be the only interpretation of our measurements. Thus the issue of why such a high T_c is realized, including the mechanism of superconductivity, remains an open question, and further detailed experimental and theoretical studies are required.

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[9] Landolt-Börnstein, *Numerical Data and Functional Relationships in Science and Technology, New Series II/11,* edited by K.-H. Hellwege and A. Hellwege (Springer-Verlag, Berlin, 1981); C: -6×10^{-5}; H: -2.93×10^{-5}; O: -4.61×10^{-5}; and the correction of the Ethel linkage: $\lambda = -0.24 \times 10^{-5}$ emu/mol. Empirically the calculated value would contain the error of ±5%–10%.

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[12] For the square lattice, we have $(1 + \lambda_{ep})N^*(E_F) \sim 0.31$ states/eV and $\lambda_{ep} \sim 0.19$ using $H_c = 995$ Oe (Ref. [7]). This is not meaningfully different from the case for the triangular lattice.

[13] Various relations of the electron gas model with the dispersion relation, $E(k) = \hbar^2 k^2/(2m^*)$. For the 2D system $T_F^{2D} = \hbar^2 k_F^2/(2m^* k_B)$, $n_s^{2D} = k_F^2/(2\pi)$, $r_s^{2D} = 1/(\sqrt{2}\, a_B^* k_F)$, $k_{TF}^{2D} = 2/(a_B^*)$, and $\gamma^{2D} = \pi k_B^2 m^*/(3\hbar^2)$. For the 3D system, $T_F^{3D} = \hbar^2 k_F^2/(2m^* k_B)$, $n_s^{3D} = k_F^3/(3\pi^2)$, $r_s^{3D} = (9\pi/4)^{1/3}/(a_B^* k_F)$, $k_{TF}^{3D} = [4k_F/(\pi a_B^*)]^{1/2}$, and $\gamma^{3D} = k_B^2 m^* k_F/(3\hbar^2)$. Here, $a_B^* = \epsilon/(m^* e^2)$ is the effective Bohr radius and ϵ is the dielectric constant. For simplicity, we assumed $\epsilon = 1$. Especially for YBCO, we note that the present electron gas model may not be available because of the strong electronic correlation.

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