Superconductivity at $T_c = 44$ K in Li$_x$Fe$_2$Se$_2$(NH$_3$)$_y$

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Abstract. Following a recent proposal by Burrard-Lucas et al. [unpublished, arXiv: 1203.5046] we intercalated FeSe by Li in liquid ammonia. We report on the synthesis of new Li$_x$Fe$_2$Se$_2$(NH$_3$)$_y$ phases as well as on their magnetic and superconducting properties. We suggest that the superconducting properties of these new hybrid materials appear not to be influenced by the presence of electronically-innocent Li(NH$_2$) salt moieties. Indeed, high onset temperatures of 44 K and shielding fractions of almost 80% were only obtained in samples containing exclusively Li$_x$(NH$_3$)$_y$ moieties acting simultaneously as electron donors and spacer units. The c-axis of the new intercalated phases is strongly enhanced when compared to the alkali-metal intercalated iron selenides $A_{1-x}$Fe$_2$-$y$Se$_2$ with $A = $ K, Rb, Cs, Tl with $T_c = 32$ K.

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1 Introduction

The discovery of iron-based superconductors in 2008 [1] had boosted hopes to find compounds that would rival the $T_c$ records of the copper-based superconductors [2]. The highest transition temperatures of Fe based superconductors to date are in the vicinity of 56 K and were reported already within the first year after the initial discovery [3,4]. Meanwhile a variety of families has been identified [5,6,7,8] all of which share a common structural recovery [3,4]. Meanwhile a variety of families has been identified [5,6,7,8] all of which share a common structural recovery [3,4].

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ducting properties are controlled by electronic doping and lattice expansion due to the presence of the Li$_x$(NH$_3$)$_y$ moieties.

These results could be the starting point to employ tailor-made electronic donor molecules (e.g. metallocenes) which allow for a systematic variation of the donor capabilities of the guest species and the interlayer separation in Fe$_2$Se$_2$ hybrids and thus a systematic control of the critical temperatures.

2 Experimental details

Polycrystalline samples of tetragonal FeSe were synthesized from high purity Fe pieces (99.99%) and Se (99.999%) shots. Stoichiometric mixtures of the starting materials were placed in double-wall ampoules and were slowly heated to 1100°C, kept at this temperature for 48 h and then cooled with a rate of 60°C/h to 410°C. At 410°C the ampoules were kept for 100 h and then quenched in ice water. X-ray diffraction and SQUID measurements documented single phase character of the materials lacking any impurity phases and displaying the appropriate tetragonal space group $P4/nmm$, with lattice constants $a = b = 0.3771$ nm and $c = 0.5524$ nm. Furthermore, a well-defined transition into the superconducting state with an onset temperature of 9.5 K was identified. The high transition temperature and the ratio of $c/a = 1.4648$ signal a composition close to Fe$_{1.02}$Se$_3$. We note, that careful inspection of powderized samples of tetragonal FeSe revealed its metastable character. Indeed, a tribochemical transition and formation of Fe$_7$Se$_8$ as a ferrimagnetic impurity $^{30}$ is observed upon grinding of tetragonal FeSe samples. Hence, excessive grinding of the samples should be avoided before the subsequent intercalation reaction in liquid ammonia.

The various intercalation reactions of tetragonal FeSe host lattices with lithium were carried out under inert gas conditions in liquid ammonia employing a Schlenk line. In order to prevent the formation and intercalation of significant amounts of lithium amide rather small batches of FeSe (100-600 mg) were intercalated under avoidance of high lithium concentrations in liquid ammonia. Hence, an excess of Li metal (99.9%; Sigma Aldrich) was avoided to synthesize amide-free Li$_x$Fe$_2$Se$_2$(NH$_3$)$_y$ hydride materials. Furthermore, the cooling bath temperature was always kept at about -75°C during the intercalation period (typically 1-4 hours) and the removal of the remaining NH$_3$ solvent was accomplished via condensation into a cooling trap using a vacuum pump. The dry sample was allowed to warm up before the transferral to a glove box (argon inert gas) which is equipped with an inlet system adopted for the sample holders of the subsequent magnetic measurements. Hence, all sample manipulations during synthesis and physical property measurements were strictly performed under inert gas condition. Two samples with different amounts of lithium were synthesized by this approach: Li$_{0.4}$Fe$_2$Se$_2$(NH$_3$)$_{0.6}$ and Li$_{0.9}$Fe$_2$Se$_2$(NH$_3$)$_{0.5}$. Elemental analysis of the two samples yielded N/H ratios of 1:3.06 and 1:2.97, respectively, in line with the successful intercalation of Li(NH$_3$)$_2$ moieties and avoidance of any significant Li(NH$_2$)$_2$ impurity phases. Both samples yielded $T_c$ values of 44 K and shielding fractions as high as 80%. This result clearly suggests that the Li(NH$_2$)$_2$ impurities found in the materials prepared by Burrard-Lucas et al. do not trigger the superconducting properties of the LiFe$_2$Se$_2$ hydride phases.

Indeed, a control experiment using an excess of lithium during the intercalation process yielded a product containing significant amounts of Li(NH$_2$)$_2$ with the formal stoichiometry of Li$_{1.8}$Fe$_2$Se$_2$(NH$_3$)$_2$Li(NH$_2$)$_2$ but yielding a lower $T_c$ of 40 K. Since the Fe$_2$Se$_2$ parent lattice only provides voids to accommodate formally one NH$_3$/NH$_2$-moiety per formula unit it remains to be seen, whether Li(NH$_2$)$_2$ represent a true guest species or just an impurity phase. We therefore suggest that the electronically inert Li(NH$_2$)$_2$ moiety will not contribute to the physical properties of these hydride materials.

Magnetization measurements were performed in a magnetic property measurement system MPMS-7 (Quantum Design), in a temperature range between 2 K to 300 K and in magnetic fields up to 7 T. All powder samples were mounted in a special KeI-F sample holder, which has a cylindrical hole with a diameter of 3 mm and a height of 3 mm. The samples were prepared in argon atmosphere and transferred to the magnetometer via an argon-lock. To determine the volume susceptibility of the intercalated samples, the density $\rho$ was derived from the unit cell volume based on the powder diffraction and the analytical data. The calculated density for Li$_{0.4}$Fe$_2$Se$_2$(NH$_3$)$_{0.6}$ is 3.937 g/cm$^3$ and for Li$_{1.8}$Fe$_2$Se$_2$(NH$_3$)$_2$Li(NH$_2$)$_2$ the value is 4.24 g/cm$^3$.

![Fig. 1. Powder diffraction pattern of Li$_{1.8}$Fe$_2$Se$_2$(NH$_3$)$_2$Li(NH$_2$)$_2$ at room temperature. The intensity was normalized to the intensity of the (002) reflection. The result of a Le-Bail fit is indicated as a solid line. The resulting difference spectrum is indicated at the bottom of the figure. The calculated and allowed Bragg reflections of the parent compound (tetragonal Fe$_2$Se$_2$), Fe$_7$Se$_8$ impurities (traces) and the intercalated hydride material (1) are indicated by vertical bars.](image-url)
3 Experimental results and discussion

Phase identification and purity of the parent lattices and of the intercalated hybrid materials was controlled by powder diffraction studies using a Image Plate Guinier Camera G670 (Huber) and monochromatized CuKα1 radiation with λ = 1.540598 Å. A flat sample holder was employed and the inherent air and moisture sensitive samples were prepared inside a glove box. The samples were sealed in between two Mylar foils to prevent sample decomposition. Phase analysis and lattice parameter refinements were performed using the Le-Bail method [37].

In Fig. 1 the x-ray diffraction pattern is shown for Li$_1$,$_3$Fe$_2$Se$_2$(NH$_3$)$_2$[Li(NH$_2$)$_2$]$_{0.5}$. All Bragg intensities of the intercalated species could be indexed by a body centered tetragonal cell with I4/mmm symmetry in agreement with the study by Burrard-Lucas et al. [34]. The lattice parameters were found to be rather insensitive to the amount and ratio of intercalated Li(NH$_3$)$_2$/Li(NH$_2$)$_2$ fractions, for example, a = b = 0.38273(6) nm and c = 1.6518(3) nm (space group I4/mmm) for Li$_0.5$Fe$_2$Se$_2$(NH$_3$)$_2$$_0.5$ and a = b = 0.379607(8) nm and c = 1.69980(11) nm in the case of Li$_1$,$_3$Fe$_2$Se$_2$(NH$_3$)$_2$[Li(NH$_2$)$_2$]$_{0.5}$. Compared to the FeSe starting material the in-plane lattice constants are almost the same, being expanded by less than 0.3%. The c-axis however is enlarged by more than a factor of 3. Compared to the 245 compounds [17] the in-plane lattice constants ($a\sqrt{5}$) are slightly smaller but the c-axis is dramatically increased, a fact which strongly points toward the importance of the FeSe layer separation along c to enhance the $T_c$ values. However, one has to keep in mind that the alkali intercalated 245 compounds exhibit critical temperatures of approximately 32 K, independent of the magnitude of the $c$-axis lattice change, which increases from 1.4 nm in the potassium containing compounds to 1.53 nm in the Cs intercalated compounds [17].

The temperature dependent magnetic susceptibilities of two intercalated Fe$_2$Se$_2$ samples are depicted in Fig. 2 showing the respective zero-field-cooled (zfc) and field-cooled (fc) runs. The earth magnetic field was compensated during the zfc sequences (down to 2 K) by a procedure described in detail in Ref. [38]. In the subsequent heating run we applied a small magnetic field ($B = 0.5$ mT) to record the magnetization data up to 55 K. This procedure reflects the complete shielding effect of the sample at low temperature ($\chi_V = -1$ in the ideal case), whereas the fc measurements account for the Meissner expulsion. Figure 2 shows the temperature dependence of the volume susceptibility $\chi_V(T)$ of two selected samples, namely Li$_0.5$Fe$_2$Se$_2$(NH$_3$)$_2$$_0.6$ with the lowest normal-state susceptibility (Fig. 2a) and Li$_1$,$_3$Fe$_2$Se$_2$(NH$_3$)$_2$[Li(NH$_2$)$_2$]$_{0.5}$ with the largest normal-state susceptibility values (Fig. 2b).

In case of Li$_0.5$Fe$_2$Se$_2$(NH$_3$)$_2$$_0.6$ (Fig. 2b) we find a shielding fraction of about 80% as observed in the zfc measurements and observe the $\chi_V(T)$ signature of another superconducting transition below 10 K which we ascribe to traces of the non-intercalated parent compound FeSe. The fc experiments point towards a small lower critical field and a moderate pinning effect leading to a Meissner phase which amounts approximately 20% of the sample volume. Both volume fractions seem to be significant and rather large when compared to the results by Ying et al. [33] and Burrard-Lucas et al. [34]. A well-defined onset of superconductivity in Li$_0.5$Fe$_2$Se$_2$(NH$_3$)$_2$$_0.6$ appears close to 44 K in both, the zfc and fc experiments. This is one of the highest transition temperatures reported so far in

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**Fig. 2.** Temperature dependence of the magnetic susceptibility of intercalated FeSe samples as obtained in zero-field-cooled and field-cooled runs. In field cooling cycles and as probing dc fields, external magnetic fields of 0.5 mT have been used. (a) Volume susceptibility of the batch with the lowest normal-state susceptibility Li$_0.5$Fe$_2$Se$_2$(NH$_3$)$_2$$_0.6$. (b) Volume susceptibility of the batch with the highest normal-state susceptibility Li$_1$,$_3$Fe$_2$Se$_2$(NH$_3$)$_2$[Li(NH$_2$)$_2$]$_{0.5}$. The superconducting onset temperatures are indicated by arrows.

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the iron-selenides at ambient pressure. For temperatures $T > T_c$, we find a small and almost vanishing paramagnetic Pauli-like magnetic susceptibility only.

In case of $\text{Li}_{0.8}\text{Fe}_2\text{Se}_2(\text{NH}_3)_{0.6}$ (Fig. 2a) the relative behavior of the fc and zfc susceptibility values is similar to the amid free sample (Fig. 2b) with exception of the somewhat lower critical temperature $T_c = 40$ K and the large normal-state susceptibility contribution of $\chi V = 0.4$. Accordingly, the fc curve is completely shifted to positive susceptibility values. Subtracting this normal-state susceptibility of 0.4, we find that both samples exhibit shielding fractions of about 80% as observed in the zfc measurements.

We will outline below that this large normal-state susceptibility might originate from a ferrimagnetic impurity ($\text{Fe}_8\text{Se}_6$) which is absent in $\text{Li}_{0.8}\text{Fe}_2\text{Se}_2(\text{NH}_3)_{0.6}$ but present in $\text{Li}_{1.8}\text{Fe}_2\text{Se}_2(\text{NH}_3)[\text{Li}(\text{NH}_2)]_{0.5}$. A similar susceptibility contribution has been observed in the Li(ND$_2$) containing sample $\text{Li}_{0.8}\text{Fe}_2\text{Se}_2(\text{ND}_2)_{0.2}(\text{ND}_3)_{0.8}$ reported by Burrad-Lucas et al. [31], which displays the highest superconducting volume fraction (40-50%) in their earlier report.

In the latter case the authors found 10.3% hexagonal FeSe impurities via Rietveld analysis [31]. Consequently, the reduction or complete avoidance of FeSe impurities during sample preparation might provide one of the key control parameter of the superconducting properties of the intercalated FeSe species. Accordingly, in all compounds there seems to be a correlation between the superconducting transition temperature and the positive normal-state susceptibility values.

In order to elucidate the relationship between the superconducting state and the high positive normal-state susceptibility, magnetization measurements at 2 K and 55K were performed. The magnetization versus magnetic field is shown in Fig. 3a for $\text{Li}_{0.8}\text{Fe}_2\text{Se}_2(\text{NH}_3)_{0.6}$ and in Fig. 3b for the system $\text{Li}_{1.8}\text{Fe}_2\text{Se}_2(\text{NH}_3)[\text{Li}(\text{NH}_2)]_{0.5}$ in fields up to 7 T. In case of the LiNH$_2$ free sample (Fig. 3c) the intercalated compound exhibits the typical hysteresis loop of a type-II superconductor. As in most of the 245 iron selenides and probably as a fingerprint of 2D superconductors the lower critical field is close to zero and hence, in these systems the Meissner phase only exists close to zero external fields. The small asymmetry of the magnetization hints for a a small magnetic contribution.

This contribution is in case of the LiNH$_2$ containing sample (Fig. 3b) clearly identified as a ferrimagnetic impurity. The hysteretic loop at 55 K (Fig. 3d) shows this underlying magnetic contribution, which is most likely due to the presence of the Fe$_2$Se$_6$ impurities and/or additional free Fe-ions. This phase is ferrimagnetic with a saturation magnetism of 0.2 $\mu_B$/Fe-atom and a critical temperature $T_K = 425$ K [30]. From the linear slope of this magnetization curve between -10 and 10 mT a susceptibility contribution can be derived which is in good agreement with the observed magnetic normal-state contribution in Fig. 2b. In order to estimate the lower critical field $B_{c1}$ of the samples linear fits to the initial slopes (solid lines) were performed as depicted in the insets of Figs. 3c) and (d). $B_{c1}$ at 2K is determined by the deviation of the magnetization data from this straight line resulting in $B_{c1}(2K) = 0.8 \pm 0.1$ mT for $\text{Li}_{0.8}\text{Fe}_2\text{Se}_2(\text{NH}_3)_{0.6}$ and 0.5 $\pm 0.1$ mT in the case of $\text{Li}_{1.8}\text{Fe}_2\text{Se}_2(\text{NH}_3)[\text{Li}(\text{NH}_2)]_{0.5}$.

In summary, we synthesized superconducting hydride materials $\text{Li}_x\text{Fe}_2\text{Se}_2(\text{NH}_3)_x$ via intercalation of lithium in liquid ammonia. $\text{Li}_{0.5}\text{Fe}_2\text{Se}_2(\text{NH}_3)_{0.6}$ with a maximal su-

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**Fig. 3.** Magnetic hysteresis loop of intercalated FeSe samples at 2 K: (a) of the batch with the lowest paramagnetic background ($\text{Li}_{0.8}\text{Fe}_2\text{Se}_2(\text{NH}_3)_{0.6}$) and (b) of the batch with the highest paramagnetic background ($\text{Li}_{1.8}\text{Fe}_2\text{Se}_2(\text{NH}_3)[\text{Li}(\text{NH}_2)]_{0.5}$). The insets (c) and (d) display an expanded region of the magnetization curves which allows to estimate the lower critical field, $H_{c1}(2K) = 8$ mT and 5 mT in $\text{Li}_{0.8}\text{Fe}_2\text{Se}_2(\text{NH}_3)_{0.6}$ and $\text{Li}_{1.8}\text{Fe}_2\text{Se}_2(\text{NH}_3)[\text{Li}(\text{NH}_2)]_{0.5}$, respectively. Inset (e) exhibits the hysteresis loop at 55 K reflecting the ferrimagnetic contribution of the samples with the highest magnetic background.
perconducting onset temperature of 44 K is almost free of magnetic impurities with a normal-state susceptibility close to zero. The Meissner fraction of this compound is about 20% and the shielding fraction close to 80%. The enhancement of the critical temperature results from the significant increase of the c-axis lattice parameter and electron doping via lithium ions. In addition, we synthesized Li$_1.5$Fe$_2$Se$_2$(NH$_3$)$_2$Li[Li(NH$_2$)$_2$].0.5. Here we found a reduced superconducting transition temperature and a significant amount of magnetic impurities. We hope that these results are the starting point to systematically vary the separation of the Fe$_2$Se$_2$ layers by introducing tailored electronic donor molecules.

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