



Positive and negative chemical pressure effects investigated in electron-doped FeSe films with an electric-double-layer structure

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We investigated chemical pressure effects in electron-doped (e-doped) FeSe by fabricating an electric-double-layer structure with single-crystalline FeSe films on LaAlO₃ with Se substituted by isovalent Te and S. Our method enables transport measurements of e-doped FeSe. Electron doping by applying a gate voltage of 5 V increases T_c of the FeSe_{1-x}Te_x and FeSe_{1-y}S_y films with $0 \leq x \leq 0.4$ and $0 \leq y \leq 0.25$. Applying both positive and negative chemical pressures monotonically suppresses T_c of the e-doped FeSe. T_c and its evolution with chemical pressure is very different between undoped and e-doped FeSe, which is considered to be related to the difference in change of density of states at Fermi energy as a function of chemical pressure in the two systems. These results might be one of the manifestations of the possible difference of superconductivity mechanisms in these systems.

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I. INTRODUCTION

Iron chalcogenide superconductor FeSe [1] has attracted much attention in the field of superconductivity. FeSe shows a wide variety of intriguing physical properties [2–5], such as the nematic state apart from magnetic order, topological superconductivity, and other exotic superconducting states. FeSe also shows drastic enhancement of the superconducting transition temperature T_c in various ways. The T_c value of FeSe under ambient pressure is approximately 9 K, but it increases up to 40–50 K by applying hydrostatic pressure [6,7] and by doping electron through intercalation [8–11] and the electric-field effect [12–14]. In addition, a large gap was observed in monolayer FeSe films on SrTiO₃ (STO) [15]. The following angle-resolved photoemission spectroscopy (ARPES) measurements revealed that the gap opens below 65 K [16,17], which was argued to be the manifestation of superconductivity.

Although undoped FeSe has both electron and hole Fermi surfaces, such as other iron-based superconductors, electron-doped (e-doped) FeSe with enhanced T_c as high as 40–50 K has electron Fermi surfaces alone [18]. Monolayer FeSe on STO has the similar electronic state to e-doped FeSe by charge transfer from the substrate [19]. An interesting question is whether the mechanism of superconductivity in the heavily e-doped FeSe is the same as that of undoped FeSe. For undoped FeSe, pairing between different Fermi surfaces (interband pairing) is a plausible mechanism, whereas such a mechanism is impossible for e-doped FeSe with an electron Fermi surface alone. Another important issue is if there is any interface effect between the film and the substrate which enhances the superconductivity in the monolayer FeSe on STO. Indeed, resistive transition at as high as 65 K has not been

confirmed in monolayer FeSe, below which the energy gap was observed by ARPES. Although Ge *et al.* reported zero resistivity at a very high temperature of approximately 100 K [20], its reliability and reproducibility have been controversial [21]. Rather, recent *in situ* resistivity measurements reported that the highest temperature where the zero resistivity was observed T_c^{zero} was approximately 30 K [22,23], much lower than the gap-opening temperature and lower than T_c^{zero} of the e-doped FeSe. It is also suggested that the energy gap at high temperatures observed by the ARPES measurements in ultrathin samples is a pseudogap due to the strong superconducting fluctuations [23]. To tackle these problems, systematic investigation of the physical properties in e-doped FeSe is of particular importance.

Isovalent Te and S substitutions for Se expand and shrink the lattice of FeSe, respectively, without doping carriers, and, thus, are referred to as the negative and positive chemical pressures, respectively. Te and S substitutions involve various effects other than the pressure effect, such as effects of the substitution-induced disorders, the different electronegativity of S, Se, and Te, etc. We refer to Te- and S-substitution effects including all of these factors as chemical pressure effects in this paper. Although chemical pressure effects are extensively studied in undoped FeSe, there are few reports on Te and S substitutions for e-doped FeSe and research on electron doping so far focused only on FeSe. Although some papers reported intercalation experiments on powder samples of FeSe_{1-x}Te_x and FeSe_{1-y}S_y [24–28], it is difficult to measure transport properties with powder samples [29,30].

In this paper, we report fabrication of the electric-double layer transistor (EDLT) structure with single-crystalline thin films of FeSe_{1-x}Te_x and FeSe_{1-y}S_y, which are available for a wide range of composition [31–33]. Our method using epitaxial films enables transport measurements of e-doped samples. We investigated the chemical pressure effect on the superconductivity of e-doped FeSe_{1-x}Te_x and FeSe_{1-y}S_y thin films for

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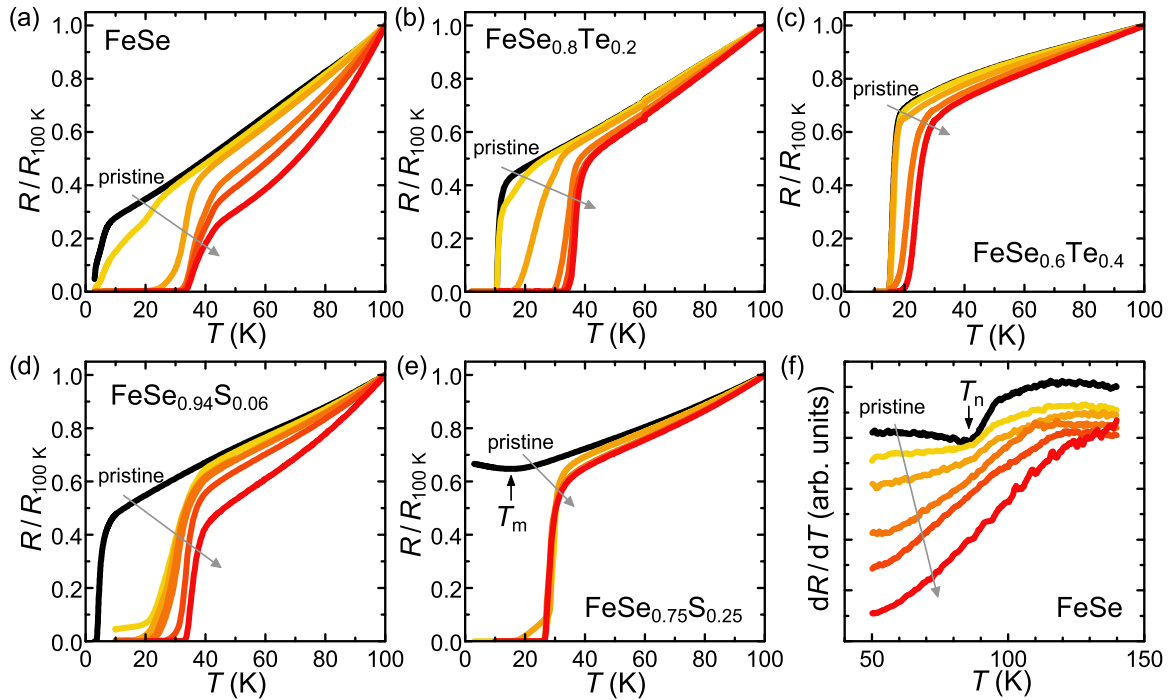


FIG. 1. (a)–(e) Shift of the temperature dependence of the resistance R of the etched $\text{FeSe}_{1-x}\text{Te}_x$ and $\text{FeSe}_{1-y}\text{S}_y$ thin films as the etching process was repeated for $x = y = 0$, $x = 0.2$, $x = 0.4$, $y = 0.06$, and $y = 0.25$, respectively. Arrows indicate the shift over the etching processes. (f) The temperature dependence of the temperature derivative of resistance of the etched FeSe.

$x \leq 0.25$ and $y \leq 0.4$. All the films showed the increase in T_c after the electron doping. T_c of the electron-doped FeSe films decreases with both Te and S substitutions. The obtained superconducting phase diagram of electron-doped $\text{FeSe}_{1-y}\text{S}_y$ and $\text{FeSe}_{1-x}\text{Te}_x$ films is rather different from that of the undoped films and that of monolayer $\text{FeSe}_{1-x}\text{Te}_x$ films, which is considered to be related to the difference in change of density of states (DOS) at Fermi energy E_F as a function of chemical pressure in the two systems. These results might be one of manifestations of the possible difference of superconductivity mechanisms in these systems.

II. METHOD

All the films were grown using a pulsed laser deposition method, details of which are described elsewhere [33–35]. We used LaAlO_3 (LAO), CaF_2 , and STO as substrates. All the films used in this paper have thickness of approximately 50 nm. As for STO substrates, conducting electrons induced by oxygen vacancies in STO enter the film on it and might affect the superconductivity of the film as in monolayer FeSe on STO. However, the STO substrates used in this paper are insulating, suggesting the amount of doped electrons are small. In addition, the films are so thick that the effect of the doped electrons on the superconductivity is negligible [14,36].

The EDLT structure was fabricated on the grown films with N,N -dethyl- N -methyl- N -(2-methoxyethyl)ammonium (DEME)-bis(trifluoromethanesulphonyl)imide as the gate dielectric [12–14,37,38]. Once the sample is taken out from the growth chamber to the atmosphere, the surface of the films is degraded and becomes inactive to the electrostatic doping. To obtain enhanced T_c by electron doping, we removed the

surface dead layer by the *in situ* electrochemical etching of the film with the ionic liquid [37]. The gate voltage V_g of 5 V, was applied at 220 K, and then the temperature was raised and kept at around 240 K to induce etching. In the experiments with V_g of 2.5 V, we raised the temperature to 330 K to induce etching. Details of the etching process are described in our previous papers [37,38]. We found that an e-doped layer is formed at the surface of the film during the etching process by an electrochemical reaction, possibly by the intercalation of DEME^+ cations in FeSe or by FeSe which adsorbs DEME^+ cations on the surface and that the reacted layer exhibits the high T_c in the FeSe-EDLT systems [37,38]. A single etching process was finished when the film thickness was decreased by approximately a few nanometers. We estimated the thickness of the etched films from the time integral of the gate current. We repeated the cycle of an etching process and an accompanying measurement of the temperature dependence of the resistivity. The electrochemical etching and the resistivity measurements were performed in a helium atmosphere using a Quantum Design physical property measurement system.

III. RESULTS AND DISCUSSION

Figures 1(a)–1(e) show the shifts in the temperature dependence of the resistance (R - T) of the electrochemically etched $\text{FeSe}_{1-x}\text{Te}_x$ and $\text{FeSe}_{1-y}\text{S}_y$ films when the etching process were repeated. The R - T curves and T_c values change as the etching process is repeated. The change in the T_c values saturates after repeating the etching several times as we previously reported in FeSe and $\text{FeSe}_{0.8}\text{Te}_{0.2}$ [37,38]. There is no broadening of the superconducting transition width over

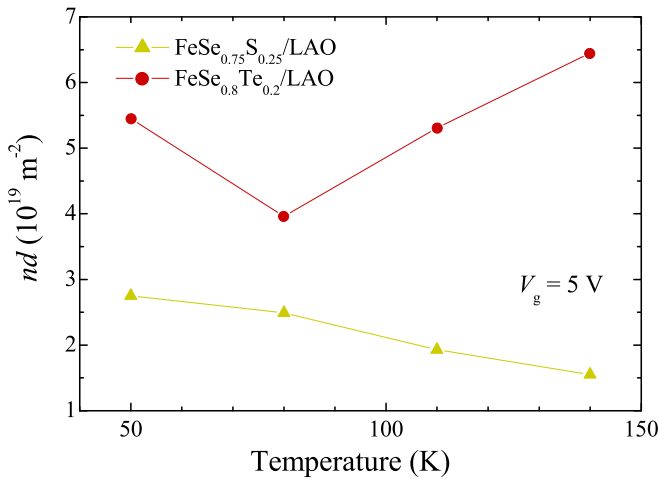


FIG. 2. Estimated carrier densities n multiplied by thickness of the surface-doped layer d for $x = 0.2$ [37] and $y = 0.25$ films.

Te and S substitution, suggesting that the inhomogeneity in composition is negligible. We also performed electron-doping experiments for films with $x \geq 0.5$, but the electrochemical reaction does not take place in a reproducible manner with $V_g = 5$ V for films with $x \geq 0.5$.

The R - T curve of the undoped sample for $y = 0.25$ showed an up-turn behavior at low temperatures. This is due to a magnetic transition, which is characteristic in strained film samples of $\text{FeSe}_{1-y}\text{S}_y$ [39]. Because a single etching process increased T_c up to higher temperatures than the magnetic transition temperature in the present paper, the behavior of the magnetic phase over electron doping is not clear. A systematic study of changing the doping amount in $\text{FeSe}_{1-y}\text{S}_y$ would provide new insights into the relation between the superconductivity in e-doped FeSe and the magnetism, which is now underway.

Figure 1(f) shows the temperature dependence of the temperature derivative of the resistance dR/dT of the etched FeSe film. It is well known that an anomaly was observed in the temperature dependence of the resistivity at the nematic transition of FeSe. The undoped FeSe film shows a dip anomaly at around 85 K in dR/dT . The dip anomaly became obscured as the etching process was repeated, which suggests the suppression of the nematic transition by electron doping. Indeed, for FeSe flakes with an EDLT structure, the nematic transition is suppressed almost completely by applying $V_g = 4$ V [40]. Suppression of the nematic transition was also observed for a K-doped-thick FeSe film by an ARPES measurement [41].

In general, estimation of the amount of carriers in the surface-doped layer of the films is not easy because the undoped layer underneath is also metallic. However, taking advantage of the fact that the surface-doped layer completely peels off after the discharge process, we can extract the transport properties of the surface conducting layer from the measurements before and after the discharge process [37]. Figure 2 shows the estimated carrier densities multiplied by the thickness of the surface-doped layer for $x = 0.2$ [37] and $y = 0.25$. Because the thickness of the surface-doped layer is on the order of 10 nm, the electron densities of the doped layer is on the order of 10^{27} m^{-3} , which is obtained by as-

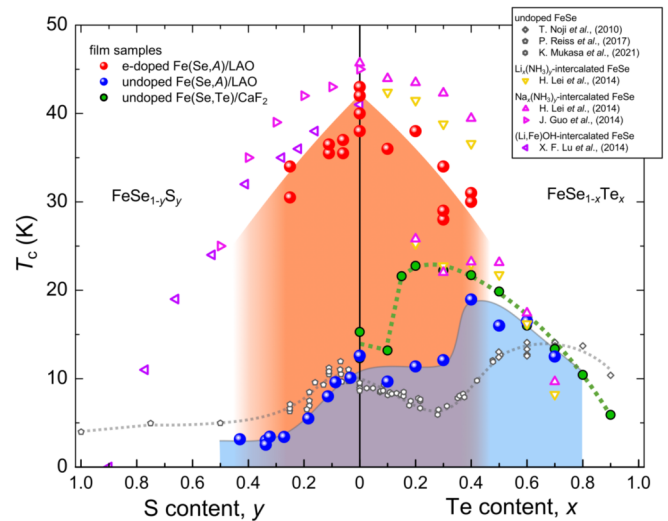


FIG. 3. The Te- and S-content dependence of T_c in undoped [32,33] and e-doped $\text{FeSe}_{1-x}\text{A}_x$ ($A = \text{Te}, \text{S}$) films on LAO. Those of undoped $\text{FeSe}_{1-x}\text{Te}_x$ films on CaF_2 [31,43] and those of bulk undoped [50–52] and intercalated samples [24,27,28] are also plotted. The dotted lines are guides for the eye.

suming only electron-type carriers in the doped layer. This value of electron density is much larger than those of pristine films ($\sim 10^{26}$) [42]. Although we were not able to evaluate the accurate values of the thickness of the reaction layers for these films, we can safely conclude that the amounts of the doped electrons are on the same order for both the Te- and S-substituted films.

Figure 3 shows the composition dependence of T_c of the electrochemically etched $\text{FeSe}_{1-x}\text{Te}_x$ and $\text{FeSe}_{1-y}\text{S}_y$. T_c of the e-doped films systematically decrease with increasing Te and S content. These results demonstrate that both the positive and the negative chemical pressure suppress the superconductivity in e-doped FeSe. The gradual and monotonic decrease in T_c over chemical pressure may be related to the absence of nematic transition in e-doped films for all ranges of x and y as will be discussed later. For comparison, the data for powder samples of $\text{Li}_x(\text{NH}_3)_y$ [24], $\text{Na}_x(\text{NH}_3)_y$ [24,27], (Li,Fe) OH-intercalated [28] $\text{Fe}(\text{Se}, \text{A})$ ($A = \text{Te}, \text{S}$) and undoped $\text{FeSe}_{1-x}\text{Te}_x$ films on CaF_2 [31,43] are also shown in Fig. 3. Our results resembles those of bulk powder samples of intercalated $\text{FeSe}_{1-x}\text{Te}_x$ and $\text{FeSe}_{1-y}\text{S}_y$. Small variations in the T_c values among these systems will be due to the difference in the intercalated ions. Difference in intercalant results in the difference in lattice parameters, for example, distance between the neighboring Fe planes, chalcogen height, etc. It may also result in the difference in amount of the doped carriers.

As for the undoped samples, the composition dependence of T_c is different among bulk and films on LAO and CaF_2 , particularly, for $\text{FeSe}_{1-x}\text{Te}_x$. We attribute the difference to the difference in lattice strain in the samples. It should be noted that films on CaF_2 and LAO are under strong and weak compressive strains, respectively, at least, for Te-substituted samples. T_c of $\text{FeSe}_{0.5}\text{Te}_{0.5}$ increases with increasing compressive strain, and T_c of a film on CaF_2 is approximately 1.5 times higher than that of bulk. In addition, for samples

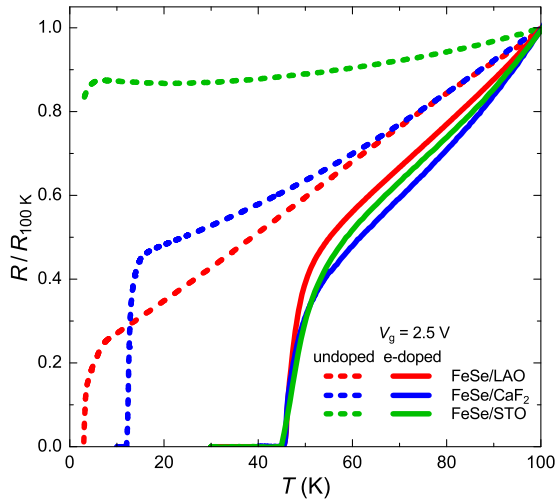


FIG. 4. Temperature dependence of the normalized resistance of FeSe films on LAO, CaF₂, and STO substrates with an EDLT structure before and after the electrochemical etching/doping with $V_g = 2.5$ V.

under stronger compressive strain, the highest T_c is achieved at smaller x and the change in T_c in the small x side of the superconducting dome become steeper. Thus, the composition dependence of T_c seems to change systematically with the strength of the strain.

Compared with the undoped samples, differences in the composition dependence of T_c are much smaller between bulk and the films for the e-doped samples. In addition, we previously observed almost the same T_c in the e-doped FeSe_{0.8}Te_{0.2} films on different substrates [37]. Figure 4 shows the temper-

ature dependence of resistance of FeSe films on LAO, CaF₂, and STO substrates with an EDLT structure before and after the electrochemical etching/doping with $V_g = 2.5$ V. Despite the different T_c 's of the undoped films, which are likely due to the different strength of the strain, the e-doped films shows exactly the same T_c^{zero} values. These results show that T_c does not depend on the strain in e-doped films, which is in contrast to the undoped samples. There are two possible explanations for the similar T_c in e-doped samples. One explanation is that the lattice strain does not affect the superconductivity in e-doped FeSe. The other possibility is that the strain in the e-doped films is relaxed, at least, in the reaction layer at the surface with high T_c . Considering the fact that T_c of intercalated FeSe largely changes by applying physical pressure [44,45] and by applying chemical pressure, it is unlikely that the strain would not change T_c of the e-doped FeSe. Thus, the strain would be relaxed in the reacted layer with high T_c .

The evolution of T_c with chemical pressure is very different between undoped and e-doped FeSe. Although e-doped FeSe shows a gradual and monotonic decrease in T_c over both positive and negative chemical pressures, undoped FeSe shows nonmonotonic dependence of T_c on chemical pressure with a drastic change in T_c at $x = 0.3-0.4$. The composition where the significant change in T_c is observed in undoped FeSe_{1-x}Te_x coincides with the disappearance of the nematic order [32,46]. A Magnetotransport experiment revealed a correlation between T_c and carrier density [42], suggesting that the band reconstruction in the nematic phase predominantly affects the superconductivity rather than the existence of the nematic order itself or the nematic fluctuation possibly developing near the quantum critical point. Indeed, our recent calculation based on density functional theory showed a drastic change in DOS at the Fermi level before and after

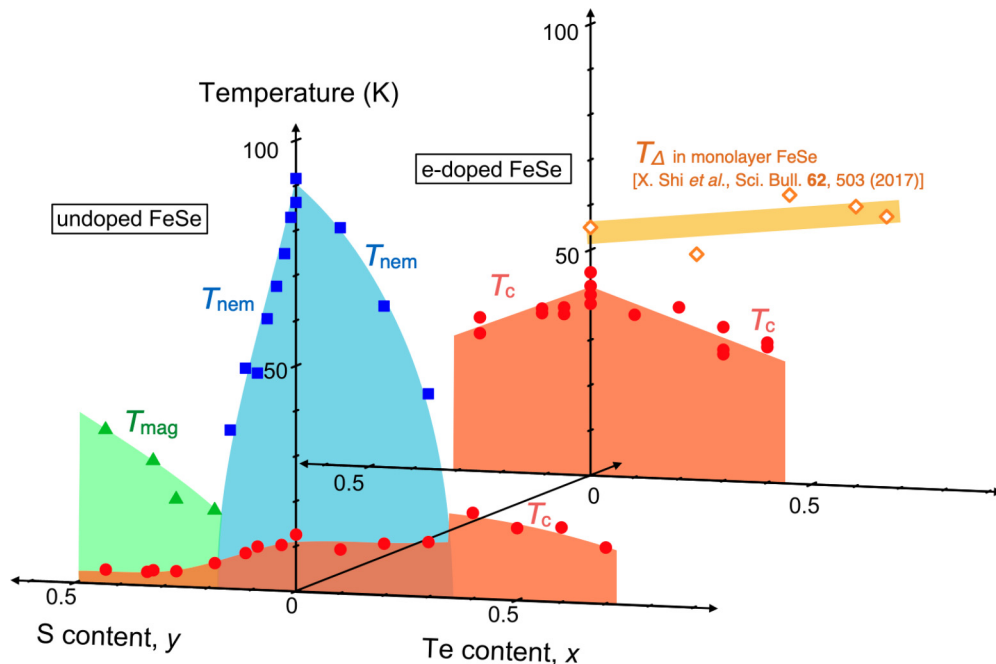


FIG. 5. Obtained phase diagram of the undoped [32,33] and the e-doped FeSe_{1-x}Te_x and FeSe_{1-y}S_y films. T_{mag} represents the magnetic transition temperature in undoped FeSe_{1-y}S_y films [39]. The gap-opening-temperature T_{Δ} of the single-layer FeSe_{1-x}Te_x/SrTiO₃ [53] are also plotted.

the nematic transition [47]. An ARPES study of $\text{FeSe}_{1-x}\text{Te}_x$ films also revealed increase of DOS at E_F around the Γ point for films without nematic order. In contrast to the undoped FeSe, the e-doped FeSe does not exhibit nematic order as was suggested by the resistance measurement. So, it is natural to consider that the difference in T_c on the chemical pressure between undoped and e-doped FeSe is related to the difference in change of DOS at E_F as a function of chemical pressure in these two systems. This may be one of manifestations of the possible different superconductivity mechanisms characteristic of the different topology of Fermi surfaces in the two systems.

Figure 5 shows the phase diagram of e-doped and undoped FeSe thin films. The gap-opening temperature T_Δ for monolayer $\text{FeSe}_{1-x}\text{Te}_x$ films on STO are also plotted. T_Δ of the monolayer $\text{FeSe}_{1-x}\text{Te}_x$ films is almost independent of x , whereas our e-doped films of $\text{FeSe}_{1-x}\text{Te}_x$ shows the monotonic decrease in T_c . This might suggest that the superconductivity mechanism of monolayer FeSe is different from that of e-doped FeSe. In fact, theoretical studies suggested that the interfacial effect enhances the superconductivity in monolayer FeSe [48]. Another possible scenario is that the energy gap observed by the ARPES studies in monolayer FeSe is not the superconducting gap and the true values of T_c at which resistive transition occurs in monolayer FeSe is similar to that of e-doped FeSe. A recent combined study of ARPES and resistivity measurements on monolayer FeSe revealed that the temperature where the resistivity start to decrease coincides with the gap-opening temperature, suggesting the opening of a pseudogap at the temperature due to strong superconducting fluctuations [23]. A pseudogap behavior was also reported

in organic-ion-intercalated FeSe by a nuclear magnetic resonance measurement [49]. To settle this problem, investigation of the superconducting fluctuations in e-doped $\text{FeSe}_{1-x}\text{Te}_x$ is of great importance. Transport measurements of the e-doped $\text{FeSe}_{1-x}\text{Te}_x$, combined with a study of the Meissner screening by a mutual inductance measurements will provide new insights into the superconductivity in FeSe-related materials, which is under way.

IV. CONCLUSION

We investigated the chemical pressure effects of single crystalline thin films of $\text{FeSe}_{1-y}\text{S}_y$ and $\text{FeSe}_{1-x}\text{Te}_x$ by fabricating an EDLT structure, and obtained the phase diagram of e-doped $\text{FeSe}_{1-x}\text{Te}_x$ and $\text{FeSe}_{1-y}\text{S}_y$ films. Our method enables transport measurements in e-doped $\text{FeSe}_{1-x}\text{Te}_x$ and $\text{FeSe}_{1-y}\text{S}_y$. Electron doping increases T_c of all the $\text{FeSe}_{1-x}\text{Te}_x$ and $\text{FeSe}_{1-y}\text{S}_y$ films with $0 \leq x \leq 0.4$ and $0 \leq y \leq 0.25$. T_c of the electron-doped films decreases monotonically by the substitution Se for S and Te. This is rather different from those in undoped samples, which is considered to be related to the difference in change of DOS at E_F as a function of chemical pressure in the two systems. These results might be one of manifestations of the possible difference of superconductivity mechanisms in these systems.

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