Pressure-induced high-T_c superconducting phase in FeSe: Correlation between anion height and T_c

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In this study, we performed high-pressure electrical resistivity measurements of polycrystalline FeSe in the pressure range of 1–16.0 GPa at temperatures of 4–300 K. A precise evaluation of T_c from zero-resistivity temperatures revealed that T_c shows a slightly distorted dome-shaped curve with maximum T_c^{offset} (30 K) at 6 GPa. With the application of pressure, the temperature dependence of resistivity above T_c changes dramatically to a linear dependence, that the high-T_c phase appears above 3 GPa. We found a striking correlation between T_c and the Se height: the lower the Se height, the more enhanced is T_c. Moreover, this relation is broadly applicable to other iron pnictides, strongly indicating that high-temperature superconductivity can appear only around the optimum anion height (~1.38 Å). On the basis of these results, we suggest that the anion height should be considered as a key determining factor of T_c of iron-based superconductors containing various anions.

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I. INTRODUCTION

Recent findings on the superconductivity in $LaFeAsO_{1-x}F_x$ (Ref. 1) and related materials have triggered a great deal of interest in iron compounds because of the possible connection between superconductivity and magnetism,[2] which undergoes a phase transition from antiferromagnetic to superconducting ground states (and vice versa) tuned by external pressure[3] or chemical doping.[1] In particular, PbO-type FeSe, which is one of the iron-based superconductors discovered a long time ago,[4] has attracted attention as a key material for elucidating the superconducting mechanism, because of its extremely simple structure (composed only of the superconducting $FeSe_4$ layer) and its excellent response to external pressure.[5] Among all similar materials, FeSe shows the greatest enhancement of its T_c at high pressure:[6] T_c varies from 13.6 K (T_c^{onset} at ambient pressure) to 37 K (T_c^{onset} at 4.5 GPa), indicating a growth rate as high as 5.2 K/GPa. As a result, using FeSe, it is possible to demonstrate the strong correlation between the structural parameter and T_c.[7]

The maximum T_c value of iron pnictides is apparently attained when the FeX_4 (X: anion) tetrahedron assumes a regular shape.[8] However, this rule is not applicable to FeSe, because the $FeSe_4$ tetrahedron is distorted from the regular shape,[6] while T_c increases significantly with application of pressure. Although several studies have investigated FeSe subjected to high pressure,[9,10] the pressure dependence of T_c, particularly above 6 GPa, is controversial because of the ambiguous definition of T_c and the large anisotropic compressibility of the layered structure,[6] which are attributable mainly to difficulties in measurements under high-pressure conditions. To overcome all these problems, we used a cubic-anvil-type high-pressure apparatus[11] that ensures quasihydrostaticity up to 16 GPa by the isotropic pressurization from six directions, even after the solidification of the liquid pressure-transmitting medium at low temperature and high pressure. Using this apparatus, we reconfirmed the T_c-P (pressure) phase diagram of FeSe.

In this study, we measured the electrical resistivity of a high-quality FeSe polycrystal at pressures ranging from 0 to 16 GPa. A precise evaluation of zero-resistivity temperature shows that the pressure dependence of T_c has a slightly distorted dome-shaped curve with the maximum T_c ($T_c^{\text{offset}}=30$ K) in the range of $0 \leq P \leq 11$ (GPa) and that the temperature dependence of resistivities above T_c changes dramatically between 2 and 3 GPa, which suggests the existence of a phase transition. We found a striking correlation between T_c and anion (selenium) height, which is the distance of the anion from the nearest iron layer; that is, T_c varies with the anion height. Moreover, this relation is broadly applicable to other ferropnictides, indicating that the high-temperature superconductivity in these materials only appears around the optimum anion height (~1.38 Å). We suggest that the anion height should be considered as a key determining factor of T_c of iron-based superconductors containing various anions.

II. METHOD

FeSe has a simple tetragonal structure that is composed only of edge-shared $FeSe_4$ tetrahedral layers. However, it is difficult to fabricate a good-quality superconducting FeSe sample because a large amount of excess iron is absolutely imperative for the occurrence of superconductivity[12] and extreme caution is required to prevent the formation of magnetic impurities from easily oxidizable iron. Polycrystalline samples of FeSe used in this study were prepared by a solid-state reaction using Fe (99.9%, Kojundo-Kagaku) and Se (99.999%, Kojundo-Kagaku) powders. The powders were mixed in a molar ratio of 100:99 (nominal composition of $FeSe_{0.99}$) in an argon-filled glove box and sealed in an evacu-

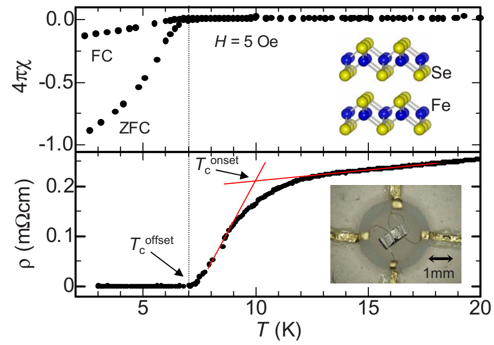

FIG. 1. (Color online) Temperature dependence of magnetic susceptibility (top main panel) and electrical resistivity (bottom main panel) of polycrystalline FeSe at ambient pressure. The cross point of two extrapolated lines denotes T_c^{onset}. The top and bottom insets show the crystal structure of FeSe and the setting of the sample in the high-pressure apparatus (see text for details), respectively.

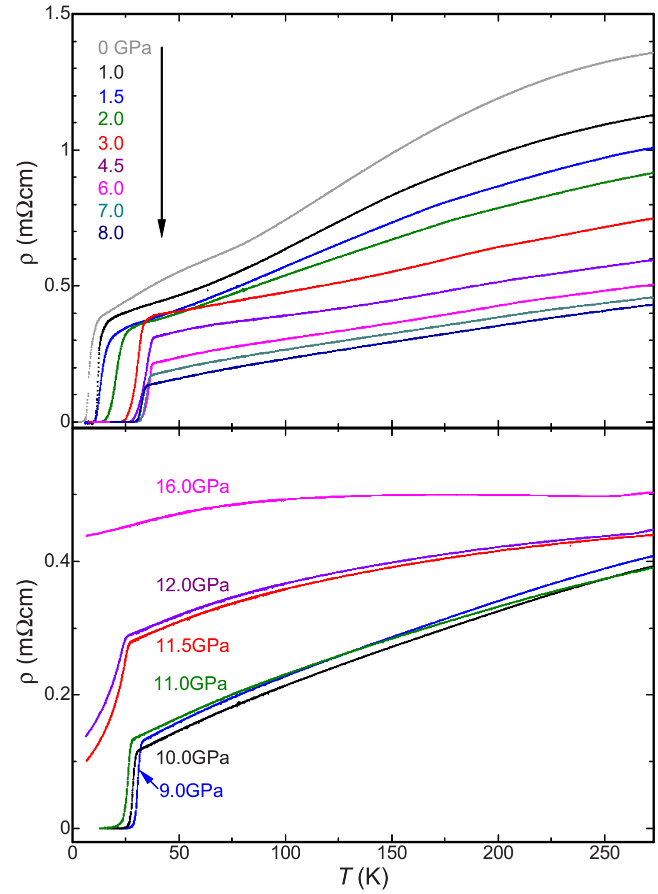

FIG. 2. (Color online) Temperature dependence of resistivity at ambient and several other pressures (top panel: 0–8 GPa and bottom panel: 9–16 GPa) for polycrystalline sample of FeSe.

ated quartz tube. Then, the powders were sintered at 1343 K for 3 days, annealed at 693 K for 2 days, and finally quenched in liquid nitrogen. Further details of sample preparation are described in Ref. 12. The quality of the obtained sample was verified by powder x-ray diffraction using an x-ray diffractometer with a graphite monochromator (MultiFlex, Rigaku). The results confirmed that the sample quality was similar to that of previously reported high-quality samples.[12] The electrical resistivity and magnetic susceptibility of the sample were measured using a physical property measurement system (PPMS, Quantum Design) and magnetic property measurement system (MPMS, Quantum Design), respectively. Electrical resistivity measurements were performed in the cubic-anvil-type apparatus[11] with Daphne 7474 oil as the liquid pressure-transmitting medium, which ensured precise measurements up to 16 GPa under nearly hydrostatic conditions in this study, even if Daphne 7474 solidifies at 3.7 GPa at room temperature.[13] Good hydrostaticity of pressure is essential for obtaining a precise pressure dependence of T_c, because FeSe has an inhomogeneous compressibility,[6] which stems from the layered structure stacked loosely by a van der Waals interaction (see upper inset of Fig. 1). Pressure was calibrated using a calibration curve that was previously obtained by observations of several fixed-pressure points (Bi, Te, Sn, and ZnS) at room temperature. The resistivity measurements were performed by a conventional dc four-probe method, as shown in the lower inset of Fig. 1, with an excitation current of 1 mA. The samples used in these experiments had dimensions of $1.0 \times 0.4 \times 0.2\ \text{mm}^3$.

III. RESULTS

As shown in Fig. 1, in our sample, zero resistivity and the Meissner effect were observed simultaneously at 7 K at ambient pressure. In order to evaluate the precise pressure dependence of T_c, we defined both T_c^{offset} (determined from the zero-resistivity temperature) and T_c^{onset} (determined from the cross point of two extrapolated lines drawn for the resistivity data around T_c). FeSe does not show Meissner diamagnetism at T_c^{onset}. Therefore, there is no guarantee that a kink in the resistivity immediately represents a signature of superconductivity.

Figure 2 shows the temperature dependence of electrical resistivities under application of external pressures ranging from 0 GPa (ambient pressure) to 16 GPa. With the application of pressure (ambient pressure to 16 GPa), the room-temperature resistivity decreases by a factor of more than 3; it reaches a minimum at 10 GPa and subsequently increases between 10 and 16 GPa. In the pressure range from 0 to 6 GPa, T_c (both T_c^{onset} and T_c^{offset}) increases rapidly but not monotonically; further, the resistivity curves gradually change shape from the one at ambient pressure (see top panel of Fig. 2). Meanwhile, as shown in the bottom panel of Fig. 2, T_c^{offset} suddenly vanishes at 11.5 GPa. This disappearance is attributed to a rapid enhancement of resistivities between 11 and 11.5 GPa. Although T_c^{onset} still remains above 11.5 GPa, it disappears completely at 16 GPa. Figure 3 shows the pressure dependence of T_c^{onset}, T_c^{offset}, and the width of the superconducting transition, ΔT_c ($=T_c^{\text{onset}}-T_c^{\text{offset}}$). Beautiful but slightly distorted dome-shaped curves are observed as cuprates[14] and heavy fermions.[15] However, the pressure dependence of ΔT_c shows a complicated trend. At low pressures up to 2 GPa, ΔT_c increases exponentially, indicating a salient broadening of the transition width, whereas T_c^{offset} in-

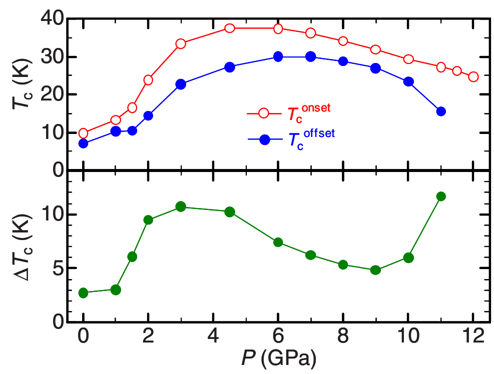

FIG. 3. (Color online) Pressure dependence of T_c^{onset} (open circle) T_c^{offset} (closed circle, top main panel) and width of superconducting transition $\Delta T_c(=T_c^{\text{onset}}-T_c^{\text{offset}})$ (bottom main panel). The solid lines are obtained by connecting the data points.

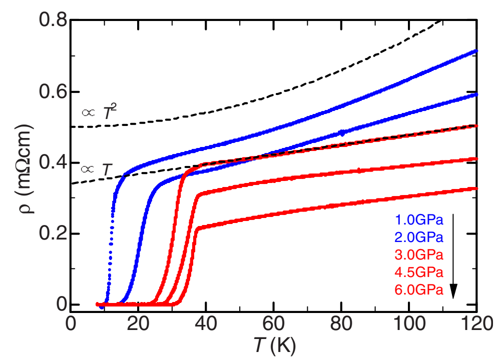

FIG. 4. (Color online) Enlarged view of resistivity below 120 K between 1 and 6 GPa. The dotted lines are guides to the eye, showing the dependence of T and T^2. For simplicity, we have not shown the data at 1.5 GPa.

creases gradually. Thereafter, ΔT_c decreases moderately but increases again above 9 GPa, resulting in a dome-shaped T_c curve. In Sec. IV, we shed light on the details of the above-mentioned behaviors, in comparison with those reported in previous studies, to elucidate the nature of iron-based superconductors.

The most striking feature in the low-pressure region (<2 GPa) is that T_c^{offset} has a relatively flat plateau; that is, an increase in T_c almost levels off between 1 and 1.5 GPa. A similar behavior was also observed during the measurements of dc magnetization[16] and electrical resistivity[17] of FeSe by using high-pressure piston-cylinder units. Therefore, this flat plateau is probably an important characteristic of FeSe. Previous ^{77}Se-NMR measurements[18] showed that antiferromagnetic spin fluctuations significantly enhanced in the plateau region and that there exists a possibility of a magnetic phase transition or spin freezing. The superconductivity in iron-based compounds is thought to be closely related to a neighboring antiferromagnetic ordered phase. The appearance of pressure-induced superconductivity adjacent to a magnetic-ordered phase is a characteristic feature of exotic superconductors such as $CeRh_2Si_2$,[19] $CeNi_2Ge_2$,[20] and $CeIn_3$,[21] with superconductivity appearing around a quantum critical point.

On application of further pressure over 7 GPa, T_c^{offset} reduces monotonically to lower values and disappears completely above 11.5 GPa. Then, the superconducting transition becomes less sharp, as is indicated by the broadening of the transition width ΔT_c. After the disappearance of T_c^{offset}, the resistivity over the entire temperature range would improve greatly with increasing pressure, indicating the occurrence of the metal-semiconductor transition. At ~9 GPa, tetragonal FeSe starts being transformed from a tetragonal structure to a hexagonal (NiAs-type) structure, and this structure undergoes a transition from a metallic superconducting state to the semiconducting state.[6] A recent synchrotron x-ray study on FeSe at various pressures[22] has revealed that the structural transition to the hexagonal phase is completed at around 12.4 GPa, which is consistent with the fact that all traces of superconductivity (see bottom panel of Fig. 2) completely vanish by 16 GPa, without any trace of an anomalous decrease in resistivity. Therefore, the remarkable increase in transition width ΔT_c above 9 GPa corresponds to the transition to the hexagonal phase, and this corresponds to the closure of the superconducting dome. The observed onset of T_c above 11.5 GPa indicates a subtle fraction of the superconducting phase, which may no longer manifest Meissner diamagnetism.

IV. DISCUSSION

Figure 4 shows an enlarged view of the resistivities below 120 K between 1 and 6 GPa. We can distinguish the gradual change in the shape of resistivity curves: with increasing pressure, the temperature dependence curve of resistivity changes from nearly quadratic to linear. In particular, the change between 2 and 3 GPa is drastic, implying the occurrence of a certain phase transition. A linear dependence of electrical resistivities on temperature is commonly observed in cuprate superconductors[23–25] and is considered to be one of the primary indicators of non-Fermi-liquid behavior. An incoherent scattering of fermion quasiparticles via magnetic interactions leads to resistivity of the form $\rho(T)=\rho_0+AT^{\alpha}$, where ρ_0, A, and α are arbitrary constants. However, no linear term is expected according to conventional Fermi-liquid theory. It should be noted that in our study, the temperature dependence of resistivities is not entirely quadratic even below 3 GPa, indicating the presence of anisotropic carrier scattering by spin fluctuations. Meanwhile, the temperature dependence of resistivity in the high-temperature superconducting phase (>3 GPa) of FeSe is highly reminiscent of the linear temperature dependence observed in high-T_c cuprates, interpreted as a "strange metal" phase,[26] where this phase is ascribed to antiferromagnetic spin fluctuations. T-linear dependence of resistivities has also been reported for other ferropnictides, for example, $Ba(Fe,Co)_2As_2$,[27] implying that antiferromagnetic spin fluctuations and superconductivity are closely related to each other in iron-based compounds, as discussed in the context of heavy fermion and cuprate superconductors.

For applied pressures greater than 3 GPa, T_c shows the dome-shaped curve, with maximum $T_c^{\text{offset}}=30.02$ K at 6 GPa, whereas between 3 and 9 GPa, ΔT_c continues to decline steadily. As has been noted previously, the shape of the FeX_4 tetrahedron is closely related to the value of T_c. In the case of iron pnictides, T_c appears to attain maximum values when

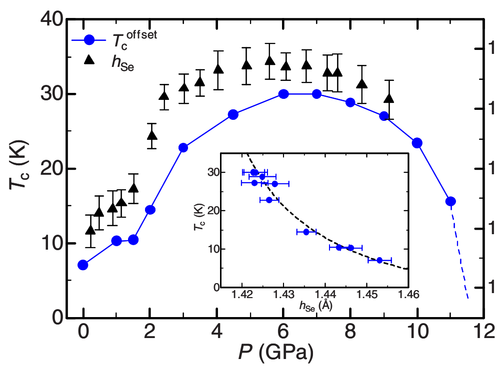

FIG. 5. (Color online) Pressure dependence of T_c^{offset} and Se height h_{Se} (inversely scaled), as obtained from Ref. 6. The inset shows T_c^{offset} as a function of the Se height. The dotted line is a guide to the eye.

the As-Fe-As bond angles come close to 109.47°,[8] which corresponds to a regular tetrahedron. However, this rule is not applicable to FeSe.[6] Therefore, we focus on the relationship of T_c with Se height. Figure 5 shows the pressure dependence of T_c^{offset} and Se height (inversely scaled), obtained from Ref. 6. Astonishingly, T_c^{offset} varies in accord with the Se height, even in the plateau in the low-pressure region. Although there is a subtle shift in the pressure dependence, which may be due to the difference in ways of applying pressures (cubic or diamond anvil), there is a clear correlation between both parameters. Furthermore, T_c^{offset} is inversely proportional to the magnitude of the Se height, as can be observed from the inset of Fig. 4, indicating that the smaller the Se height, the more enhanced is T_c. However, this seems to be contradictory to the behavior observed in other pnictides.[8] In other pnictides, it is observed that T_c is higher when the pnictogen is located at greater heights in the crystal structures; this behavior is also supported by the theoretical aspect.[7] In any case, FeSe is a suitable material for demonstrating the importance of anion position as discussed below, which is inherently linked to the mechanism of superconductivity in iron-based compounds.

We now turn to consider, in a more universal sense, the nature of the iron-based superconductivity in FeSe with respect to pressure tuning of T_c, which is the focus area in this study. Figure 6 shows the maximum T_c as a function of anion height (h_{anion}) for various iron-based superconductors.[28,29] In this study, we successfully derived the T_c-h_{anion} diagram of iron (partially nickel)-based superconductors. The clear correlation between T_c and h_{anion} is a certain indicator of the importance of anion positions in these iron-based superconductors. As shown in Fig. 6, the anion height dependence of T_c is well described by a Lorenz curve. As the value of anion height increases, T_c of the iron-based superconductors starts to increase dramatically up to ~55 K at a height of 1.38 Å, which corresponds to the optimum value of a 1111 system. However, above the optimum anion height (1.38 Å), T_c decreases rapidly with increasing h_{anion}, passing through our measured FeSe region (1.42–1.45 Å); finally, the value of h_{anion} becomes equal to that for nonsuperconducting FeTe (1.77 Å).[30] It should be noted that superconductors with direct substitution in the FeX_4 tetrahedral layer or a large de-

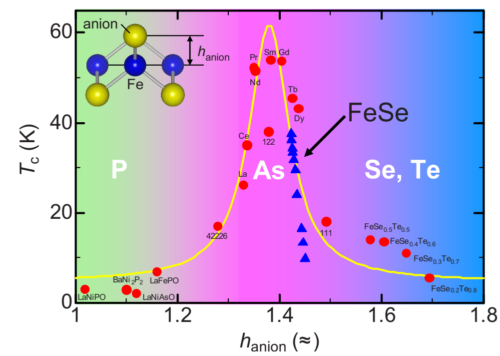

FIG. 6. (Color online) T_c as a function of anion height (h_{anion}) for various iron (and nickel)-based superconductors, as obtained from Ref. 28 (triangle: FeSe, circle: other pnictides). Lanthanides (*Ln*) indicate *Ln*FeAsO (1111 system). 111, 122, and 42226 represent LiFeAs, $Ba_{0.6}K_{0.4}Fe_2As_2$, and $Sr_4Sc_2Fe_2P_2O_6$ (Ref. 29), respectively. The yellow line shows the fitting result by the Lorenz function. The inset shows a schematic view of h_{anion}.

viation from a divalent state (Fe^{2+}), e.g., an alkali-metal element or Co-doping samples of a 122 system or chalcogen-substituted 11 system, are not particularly suitable for this trend. This is probably due to (1) the considerable disorder in the Fe layers; (2) a large gap among anion heights of different anions, for example, in $FeSe_{1-x}Te_x$, T_c appears to be dominated only by the Fe-Se distance (T_c~14 K at h_{anion}=1.478 Å, which is consistent with the Lorenz curve);[31,32] or (3) coexistence of strong magnetic fluctuation and superconductivity.[33–35] We thus conclude that the appearance of "high-temperature" superconductivity in iron compounds is confined to a specific area that is around the optimum anion height (1.38 Å), which corresponds to the radius of arsenic at ambient pressure. It has been proposed,[7] on the basis of solutions of Eliashberg equations, that the critical temperature of iron pnictides is inherently linked to their structural parameters, particularly pnictogen heights and the *a*-axis lattice parameter. The result obtained in this study is in good agreement with the theoretical prediction, albeit the length of the *a* axis of FeSe monotonically decreases with increasing pressure,[6] which suppresses the enhancement of T_c. An interesting aspect of FeSe, as observed from Fig. 6, is that T_c does not exhibit this trend above 1.43 Å (corresponding to the pressure range of 0–2 GPa), which clearly indicates that the system attains a different electronic state below the characteristic pressure (~2 GPa). The shapes of the resistivity curves above T_c change clearly between 2 and 3 GPa, as pointed out above (see Fig. 4), which implies a significant transformation to the high-T_c superconducting phase. It has been previously suggested that there is a difference in the superconducting gap symmetries of arsenic and phosphide:[36] a full-gap strong coupling *s* wave for high-T_c arsenide compounds and nodal low T_c for phosphide compounds, which is widely perceived in many studies. A theoretical approach[7] has suggested that the pairing symmetry of iron pnictides is determined by the pnictogen heights between a high-T_c nodeless gap for high h_{anion} or a low-T_c nodal gap for low h_{anion}, corresponding to the left-hand side of the Lorenz curve shown in Fig. 6. Although FeSe is located on the right-hand side, i.e., in a region of extremely high h_{anion}, it is highly probable that FeSe also shows a transition from the low-T_c phase to the high-T_c phase under application of external pressures. This is also supported by the fact that the temperature dependence of resistivity above T_c drastically changes between 2 and 3 GPa, which suggests the existence of a certain phase transition. The possibility of two kinds of superconducting phases has also been reported,[16,17] indicated by the two-step increase in the T_c-P curves. The extremely soft crystal structure of FeSe enables the control of h_{anion} in a wide range and the superconducting mechanism can be switched by the application of modest pressure. It may be interesting to explore the gap symmetry of FeSe at high pressure (~6 GPa) by NMR or muon-spin rotation and whether there is any difference between the gap symmetry of FeSe and those of other iron-based superconductors.

V. SUMMARY

In this study, the precise pressure dependence of the electric resistivity of FeSe was measured in the pressure range of 0–16.0 GPa at temperatures of 4–300 K by using a cubic-anvil-type high-pressure apparatus. T_c estimated from zero-resistivity temperature shows a slightly distorted dome-shaped curve with the maximum $T_c^{offset}=30$ K in the range of $0 \leq P \leq 11$ (GPa). The temperature dependence of resistivity above T_c changes dramatically between 2 and 3 GPa; the shapes of the resistivity curves change to linear shapes. This behavior strongly suggests a phase transition between the low-T_c and the high-T_c superconducting phases. A striking correlation is found between T_c and anion (selenium) height: the lower the Se height, the more improved is T_c. Moreover, this relation is broadly applicable to other iron pnictides, indicating that the high-temperature superconductivity in these materials appears only around the optimum anion height (~1.38 Å). On the basis of these results, we suggest that anion height should be considered as a key determining factor of T_c in iron-based superconductors containing various anions.

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