TOPICAL REVIEW

Physics and chemistry of layered chalcogenide superconductors

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Abstract

Structural and physical properties of layered chalcogenide superconductors are summarized. In particular, we review the remarkable properties of the Fe-chalcogenide superconductors, FeSe and FeTe-based materials. Furthermore, we introduce the recently discovered BiS\textsubscript{2}-based layered superconductors and discuss their prospects.

Keywords: Fe chalcogenide, FeSe, FeTe, superconductivity, magnetism, BiS\textsubscript{2}-based superconductor

1. Introduction to layered chalcogenide superconductors

Layered materials have provided us with many interesting fields in physics and chemistry. Owing to their two-dimensional crystal structure and electronic states, anomalous electronic and magnetic properties have often been observed. In particular, exotic superconductivity is likely to prefer such a layered crystal structure. For example, superconductivity at a high transition temperature ($T_c$) has been achieved in layered materials, such as cuprates \cite{1-4}, Fe-based \cite{5-13} and MgB\textsubscript{2} \cite{14, 15} superconductors. We expect that a new high-$T_c$ superconducting family will be found in layered materials.

Among the layered superconductors, the ‘chalcogenides’ are one of the notable groups, because of the variety of materials and the observation of exotic superconductivity. Sulfur (S), selenium (Se) and tellurium (Te) are categorized as chalcogens. Here we introduce the structural and physical properties of some layered chalcogenide superconductors. CdI\textsubscript{2}-type TiSe\textsubscript{2} has a simple layered structure, composed of a stack of TiSe\textsubscript{2} layers as depicted in figure 1(a), and exhibits a charge-density-wave (CDW) state. Intercalation of transition metals into the interlayer sites or application of external pressure suppresses the CDW state and induces superconductivity \cite{16, 17}. A topological insulator Bi\textsubscript{2}Se\textsubscript{3} also possesses a layered structure with a van der Waals gap as shown in figure 1(b). Similar to TiSe\textsubscript{2}, intercalation of Cu in the interlayer sites induces superconductivity in Bi\textsubscript{2}Se\textsubscript{3} \cite{18, 19}. One of the notable characteristics of chalcogenides is crystallization of a simple layered structure with van der Waals gaps. In such a structure, ions can be easily intercalated in the interlayer sites and dramatically change the physical properties of the chalcogenide layers.

The most remarkable layered chalcogenides are the Fe chalcogenides, FeSe and FeTe, which are the simplest Fe-based superconductors. The crystal structure of FeSe is shown in figure 1(c). FeSe exhibits a superconducting transition around 10 K, and shows a marked increase of $T_c$ up to 37 K under high pressure. In contrast to FeSe, FeTe undergoes an antiferromagnetic transition at 70 K. However, a partial substitution of Te by S or Se suppresses the antiferromagnetic ordering and induces superconductivity. This family is very interesting because the physical properties markedly change upon the covalent substitutions of S, Se and Te. Recently, superconductivity above 40 K was observed in metal- or molecule-intercalated FeSe. To that respect, studies on not only fundamental physics but also applications of...
Figure 1. Crystal structures of the typical layered chalcogenides (a) TiSe$_2$, (b) BiSe$_2$ and (c) FeSe.

Figure 2. Crystal structures of the typical Fe-based superconductors (a) LaFeAO, (b) FeSe and (c) Fe$_{1+d}$Te. For Fe$_{1+d}$Te, excess Fe occupies the interlayer sites with an occupancy of 7–25% as indicated with partially filled circles.

Fe-chalcogenide superconductors will be addressed. In the second section, we summarize the physical and structural properties of Fe-chalcogenide superconductors.

Very recently, we discovered a new superconducting family of the layered bismuth sulfides [20–22]. In this family, the BiS$_2$ layers are the common superconducting structure essential for superconductivity as the CuO$_2$ planes in the cuprates and the Fe$_2$AN$_2$ (An: anions of P, As, S, Se or Te) layers in the Fe-based family. In section 3, we introduce the discovery of the novel BiS$_2$-based layered superconducting family and the latest results.

2. Fe chalcogenides

In 2008, Kamihara et al. [5] reported superconductivity in the Fe-based compound LaFeAsO$_{1−x}$F$_x$. Although the parent compound LaFeAsO is an antiferromagnetic metal, the F substitution suppresses the magnetic ordering and induces superconductivity with a $T_c$ as
Figure 4. Temperature dependence of resistivity in FeSe under high pressure measured using (a) a piston-cylinder cell and (b) an indenter cell.

Figure 5. Pressure dependence of $T_c$ and the Se height in FeSe.

high as 26 K. As shown in figure 2(a), LaFeAsO has a layered structure with a stacking of the blocking La$_2$O$_2$ layers and the superconducting Fe$_2$As$_2$ layers. Many FeAs-based compounds analogous to LaFeAsO, for example, SmFeAsO$_{1-x}$F$_x$ and Ba$_{1-x}$K$_x$Fe$_2$As$_2$, were found to be superconducting with a maximum $T_c$ as high as 55 K [7, 8]. All the Fe-based superconductors share a common layered structure based on the planar layer of an Fe square lattice. In Fe-pnictide superconductors, blocking layers containing alkali, alkali-earth or rare-earth and oxygen/fluorine elements are alternatively stacked with Fe–As conduction layers [5–9, 23–26].

Superconductivity in layered Fe chalcogenides was initially found in FeSe by Hsu et al. [10] soon after the discovery of superconductivity in the LaFeAsO system. Contrary to the FeAs-based superconductors, FeSe is composed of only superconducting Fe$_2$Se$_2$ layers as shown in figure 2(b). Because of the lack of a blocking layer Fe-chalcogenides have the simplest crystal structure among the Fe-based superconductors. Furthermore, Fe$_{1+x}$Te, which has a crystal structure analogous to FeSe as shown in figure 2(c), exhibits antiferromagnetic ordering as observed in the parent (non-doped) phases of FeAs-based superconductors. Thus, Fe-chalcogenides are the key materials for elucidating the mechanism of Fe-based superconductivity [10–12].

2.1. FeSe

PbO-type FeSe is well known as a commercial material, but the discovery of superconductivity in this compound was triggered by Fe-based superconductors reported in 2008 [10].
enhancement of $T_c$ in FeSe is applicable to all the Fe-based superconductors, not only FeSe [36–38]. Figure 6(a) shows the anion height dependence of $T_c$ in typical Fe-based superconductors [36], and a schematic of anion height from the Fe layer is given in figure 6(b). The anion height dependence of $T_c$ is a symmetric curve peaking at $\sim 1.38\,\text{Å}$, as indicated by the handrawn fitting curve. The data for FeSe fall on this fitting curve only for pressures above 2 GPa, at which the anomaly was observed in the pressure dependence of $T_c$ as shown in figure 5. This observation indicates that intrinsic superconductivity in FeSe might be induced by the application of pressure above 2 GPa.

2.2. High-$T_c$ superconductivity in FeSe-related materials

As achieved by the application of the external pressure, the $T_c$ of FeSe can be enhanced by decreasing the anion height upon intercalation of a metal element or a molecule into the interlayer sites. Guo et al. [13] reported superconductivity with $T_c$ above 30 K in K-intercalated FeSe, K$_x$Se$_2$F$_2$Se$_2$. Not only metal ions but also molecules such as Li$_x$(NH$_3$)$_y$(NH$_3$)$_{1-y}$ can be intercalated into the interlayer site of FeSe: Li$_x$(NH$_3$)$_y$(NH$_3$)$_{1-y}$FeSe$_2$ shows a high $T_c$ above 40 K [39]. To date, many FeSe-based superconductors with $T_c$ above 30 K have been documented [13, 39–46]. Furthermore, Wang et al. [47] recently reported that single unit-cell FeSe films show signatures of superconducting transition with an onset temperature of 53 K. These facts indicate that the mechanisms of high-$T_c$ superconductivity in Fe-based compounds are common in FeAs-based and Fe-chalcogenide-based superconductors.

2.3. FeTe

The crystal structure of FeTe is very similar to that of FeSe. However, FeTe, but not FeSe, exhibits a magnetic/structural transition at 70 K, and the physical properties of FeTe are different from those of FeSe [48, 49]. FeTe is not superconducting except in the special case of the tensile-stressed FeTe thin film [50], which showed superconductivity at 13 K. Neutron scattering studies indicate that the spin structure is different in FeTe and the Fe-pnictide parent compounds as shown figure 7 [48]. For FeSe and Fe-pnictide superconductors, the antiferromagnetic spin fluctuations with a wave vector $Q_s = (0.5, 0.5)$ were found to correlate with superconductivity [51–55]. On the other hand, FeTe shows magnetic wave vector $Q_d = (0.5, 0.5)$ [51, 52], which is not favorable for superconductivity [56–58]. The emergence of a Fermi surface nesting associated with $Q_d$ could be induced by excess Fe at the interlayer site as illustrated in figure 2(c). Excess Fe supplies a substantial amount of electrons and it has a magnetic moment. FeTe contains 7–25% of excess Fe in its crystal structure, which affects its physical properties [56, 58].

2.4. FeTe-based superconductors

2.4.1. $FeTe_{1-x}Se_x$. As mentioned above, FeTe exhibits antiferromagnetic ordering associated with a lattice distortion at 70 K. Furthermore, FeTe possesses excess Fe (7–25%) at
Figure 8. Temperature dependence of normalized magnetic susceptibility in FeTe$_{1-x}$Se$_x$ around $T_N$ (a) and around $T_c$ (b). The arrows indicate the magnetic transition temperature. ZFC stands for zero-field cooling.

Figure 9. $T_c$ as a function of applied pressure. Reproduced with permission from [61] ©2009 American Chemical Society.

Figure 10. Nominal $x$ dependence of $x_E$ determined by electron probe microanalysis. Reproduced with permission from [65] ©2011 IEEE.

Figure 11. Temperature dependence of (a) resistivity and (b) magnetic susceptibility in FeTe$_{0.8}$S$_{0.2}$ exposed to air.

The interlayer sites. However, partial Se substitutions suppress the low-temperature structural/magnetic phase transition and reduce excess Fe, thereby inducing superconductivity [11, 59, 60]. Figure 8 shows the temperature dependence of magnetic susceptibility in FeTe$_{1-x}$Se$_x$ around $T_N$ (a) and around $T_c$ (b). The long-range magnetic ordering is suppressed with increasing Se concentration and completely
that the bulk superconductivity region spreads for Te induces superconductivity in FeTe, the S of Te by Se completely suppressed the magnetic ordering. The temperature dependence of magnetic susceptibility for the samples were also reported for thin films of FeTe\(_{1-x}\)Se\(_x\) [63, 64]. The \(T_c\) of FeTe\(_{0.5}\)Se\(_{0.5}\) films increases with decreasing lattice constant \(a\) reaching 21 K. The tensile stress can raise \(T_c\) similar to external pressure.

2.4.2. \(\text{FeTe}_{1-x}\)S\(_x\). As the partial Se substitution for Te induces superconductivity in FeTe, the S substitution for Te also suppresses magnetism and induces superconductivity [12]. However, in the case of FeTe\(_{1-x}\)S\(_x\), the optimal substitution cannot be achieved due to the low S/Te solubility limit as shown in figure 9. Although the antiferromagnetic ordering can be completely suppressed by 20% S substitution, bulk superconductivity is not observed. This can be understood as the appearance of bulk superconductivity in FeTe\(_{1-x}\)S\(_x\) is affected by the higher content of excess Fe resulting from low S concentration. In particular, the FeTe\(_{0.5}\)Se\(_{0.5}\) samples synthesized using a solid-state reaction method show a broad transition in the temperature dependence of resistivity, and diamagnetic signal corresponding to superconductivity is not observed. Ingenious attempts have been carried out to enhance this weak superconductivity and exotic annealing effects were discovered.

2.5. Annealing effects of FeTe-based superconductors

Figure 11(a) shows the temperature dependence of resistivity for a FeTe\(_{0.8}\)Se\(_{0.2}\) sample that was kept in the air for up to 2 years. Exposure to air induced a distinct superconducting transition with zero resistivity, which was absent in the as-grown state [66, 67]. After 2 years, the \(T_c^{\text{zero}}\) reached 7.8 K. The superconducting signal of magnetic susceptibility was markedly enhanced with increasing air-exposure time as shown in figure 11(b).

The weak superconductivity was improved by annealing in oxygen [68, 69]. Figure 12 shows the temperature dependence of magnetic susceptibility for the samples annealed under various conditions. Only the oxygen-annealed sample shows a distinct superconducting behavior. Kawasaki et al. [70] reported that annealing in oxygen is effective for not only FeTe\(_{1-x}\)S\(_x\) but also FeTe\(_{1-x}\)Se\(_x\). Figure 13 shows the temperature dependence of magnetic susceptibility for O\(_2\)-annealed samples with various Se concentrations around \(T_S\) (a) and around \(T_c\) (b). Even a small (10%) substitution of Te by Se completely suppressed the magnetic ordering and induced bulk superconductivity. The difference in superconducting signals between as-grown and O\(_2\)-annealed samples is obvious as shown in figures 8 and 13.

Figures 14(a) and (b) show phase diagrams based on magnetic susceptibility measurement for the as-grown and O\(_2\)-annealed samples. The as-grown samples exhibited a long-range AFM for \(x \leq 0.15\) and weak superconductivity for \(0.1 \leq x \leq 0.4\) where ‘weak superconductivity’ means non-bulk (filamentary or partial) superconducting state interfered by excess Fe. Only the FeTe\(_{0.5}\)Se\(_{0.5}\) sample was found to be a bulk superconductor. For O\(_2\)-annealed samples, the coexistence of AFM ordering and weak superconductivity was observed only for \(x \leq 0.1\). As the long-range AFM ordering was completely suppressed, the O\(_2\)-annealed samples with \(x \geq 0.1\) became bulk superconductors. It is clear from figure 14 that the bulk superconductivity region spreads with O\(_2\) annealing.

Next, we discuss the relation between bulk superconductivity and antiferromagnetic fluctuations. As mentioned in section 2.1, Antiferromagnetic spin fluctuations with the wave \(Q_s = (0.5, 0.5)\) were observed in FeSe and other FeAn compounds. In contrast, FeTe exhibits antiferromagnetic wave vector \(Q_d = (0, 0.5)\). Neutron scattering measurements revealed that \(Q_s\) and \(Q_d\) are observed over a wide composition range where FeTe\(_{1-x}\)Se\(_x\) exhibits weak superconductivity [57, 71]. For as-grown FeTe\(_{1-x}\)Se\(_x\) samples, weak superconductivity was observed in the range \(0.1 \leq x \leq 0.4\), suggesting that the nesting vectors \(Q_s\) and \(Q_d\) coexist. It is expected that the nesting vector \(Q_s\) becomes dominant for FeTe\(_{0.5}\)Se\(_{0.5}\) where bulk superconductivity sets in. On the other hand, for the O\(_2\)-annealed samples, the bulk superconductivity region extends down to \(x = 0.1\), which implies that the nesting vector \(Q_d\) is strongly suppressed by annealing in oxygen. It was concluded that the oxygen ions intercalated between superconducting layers compensate the over-doped electron carriers and suppress the magnetic wave vector \(Q_d\), which is responsible for the appearance of bulk superconductivity.

2.6. Soft chemical treatment for deintercalation of excess Fe

It was found that the alcoholic beverages induced superconductivity in FeTe\(_{0.8}\)Se\(_{0.2}\) [72]. FeTe\(_{0.8}\)Se\(_{0.2}\) samples...
Figure 13. Temperature dependence of magnetic susceptibility in O$_2$-annealed samples with various Se concentrations around $T_N$ (a) and around $T_c$ (b). The arrows indicate the magnetic transition temperature.

Figure 14. Phase diagrams showing $T_c$ and $T_N$ as a function of $x$ for (a) as-grown FeTe$_{1-x}$Se$_x$ and (b) O$_2$-annealed FeTe$_{1-x}$Se$_x$.

Figure 15. The shielding volume fraction of FeTe$_{0.8}S_{0.2}$ samples heated in various liquids as a function of ethanol concentration.

were immersed in red wine, white wine, beer, Japanese sake (rice wine), shochu (distilled spirit) or whisky and then heated at 70 °C for 24 h. The obtained shielding volume fractions are summarized in figure 15 as a function of ethanol concentration; they vary between 6 and 9% for water–ethanol mixtures, but are much higher (21–63%) for alcoholic beverages, especially red wine. This effect was also confirmed for FeTe$_{0.9}S_{0.1}$ (figure 16). It was explained by the deintercalation of excess Fe from the interlayer sites [73]. After analyzing the ingredients of alcoholic beverages we found that the solutions of malic acid, citric acid and β-alanine also induced the superconductivity in FeTe$_{0.8}S_{0.2}$ as shown in figures 17(a) and (b). Inductively coupled plasma spectroscopy results indicated that Fe ions were deintercalated from the sample to these solutions as shown in figure 17(c). Therefore, annealing in alcoholic beverages suppresses the magnetic moment of excess Fe simply by removing Fe, resulting in superconductivity. A similar enhancement of superconductivity by the deintercalation of excess Fe was reported in [74, 75]. This technique can be generally applied to the layered Fe-chalcogenide superconductors.
Figure 16. (a) Temperature dependence of magnetic susceptibility and (b) the shielding volume fraction of FeTe$_{0.9}$Se$_{0.1}$ samples heated in various liquids.

Figure 17. (a) Temperature dependence of magnetic susceptibility for FeTe$_{0.8}$S$_{0.2}$ annealed in various solutions. (b) The shielding volume fractions for the samples annealed in various liquids as a function of pH. (c) The pH dependence of Fe concentration in solutions after annealing.

Figure 18. Crystal structures of Bi$_4$O$_4$(SO$_4$)$_{1-x}$Bi$_2$S$_4$ (a) and LaOBiS$_2$ (b); $x = 0.5$ corresponds to Bi$_4$O$_3$S$_3$.

3. BiS$_2$-based layered superconductors

Discovery of a common superconducting layer is very important because many analogous superconductors can be designed by changing the spacer layers as in the high-$T_c$ cuprates and Fe-based superconductors. Recently, we found that the BiS$_2$ layer can be a basic superconducting layer in a new class of layered superconductors.

Figure 19. Temperature dependence of resistivity of Bi$_4$O$_3$S$_3$ in magnetic fields up to 5 T.
The first BiS$_2$-based superconductor is Bi$_4$O$_3$S$_3$ [20]. Its crystal structure (figure 18(a)) comprises a stack of rock-salt-type BiS$_2$ layers and Bi$_4$O$_3$(SO$_4$)$_{1-x}$ layers (blocks), where $x$ indicates the lack of SO$_4^{2-}$ ions at the interlayer sites. The parent phase ($x = 0$) is Bi$_4$O$_3$S$_3$, and Bi$_4$O$_3$S$_2$ corresponds to $x = 0.5$. Such defects at the interlayer sites are common for layered materials.

Temperature dependence of resistivity is shown in figure 19. A gradual decrease of resistivity is observed below 8.6 K with the zero resistivity appearing at 4.5 K. Magnetic susceptibility measurements indicate that Bi$_4$O$_3$S$_3$ is a bulk superconductor. To investigate the origin of superconductivity in Bi$_4$O$_3$S$_3$, we performed band calculations. They indicate that Bi$_4$O$_3$S$_3$ ($x = 0.5$) is a metal, whereas the parent phase of Bi$_4$O$_3$S$_3$ ($x = 0$) is a band insulator containing Bi$^{3+}$ ions. For Bi$_4$O$_3$S$_3$, the Fermi level lies within the bands that mainly originate from the Bi 6p orbitals as shown in figure 20. In particular, the Fermi level coincides with the peak of the partial density of states of the Bi 6p orbital within the BiS$_2$ layer.

The second BiS$_2$-based system is REO$_{1-x}$F$_x$BiS$_2$ (RE = rare earth). So far, superconductivity was observed in LaO$_{1-x}$F$_x$BiS$_2$ [24], NdO$_{1-x}$F$_x$BiS$_2$ [25], CeO$_{1-x}$F$_x$BiS$_2$ [76] and PrO$_{1-x}$F$_x$BiS$_2$ [77]. The crystal structure of LaO$_{1-x}$F$_x$BiS$_2$ is shown in figure 18(b). These materials also possess the BiS$_2$ layers as superconducting layers. The structure is simpler than that of Bi$_4$O$_3$S$_3$; hence, we can consider this system as a prototype of BiS$_2$-based superconductors. Temperature dependencies of resistivity in magnetic fields for LaO$_{1-x}$F$_x$BiS$_2$ and NdO$_{1-x}$F$_x$BiS$_2$ are shown in figures 21(a) and (b), respectively. Superconducting transition occurs at 10.6 and 5.6 K for LaO$_{1-x}$F$_x$BiS$_2$ and NdO$_{1-x}$F$_x$BiS$_2$. We note that the blocking layer of La$_2$O$_2$ or Nd$_2$O$_2$ is analogous to those in FeAs-1111 system such as LaFeAsO. Owing to the similarities of crystal structure in the BiS$_2$-based and Fe-based superconductors, we can easily design new BiS$_2$-based superconductors by changing the blocking layers. We believe that $T_c$ can be further enhanced by changing the blocking layers in the BiS$_2$-based superconductor and that the discovery of new BiS$_2$-based materials will open a new field in physics and chemistry of low-dimensional superconductors. Preliminary reports have indicated the possibility of unconventional superconductivity in the BiS$_2$-based superconductors [78–81].

4. Conclusions

We introduced the crystal structure and physical properties of layered chalcogenide superconductors. Chalcogenides tend to crystallize in a layered structure; hence, the
intercalations/deintercalations of ions or molecules at the interlayer site markedly change the physical properties and induce exotic superconductivity. The most remarkable family is the Fe chalcogenides, which are the simplest Fe-based superconductors. In this series, the key factors to induce superconductivity are the suppression of antiferromagnetism of Fe planes and the reduction of magnetic moment of excess Fe at the interlayer site. The latter can be achieved by oxygen intercalation via annealing in an oxygen-containing atmosphere or by deintercalation of excess Fe via annealing in a liquid. Interestingly, red wine is most effective for this purpose. We also introduced the newly discovered Bi$_2$S$_2$-based superconducting family. The Bi$_2$S$_2$ layer is likely to play an important role in the superconductivity, as the CuO$_2$ plane in cuprates and FeAn (FeAs, FeP, FeSe or FeTe) layers in Fe-based superconductors. New Bi$_2$S$_2$-based superconductors should be discovered by varying the blocking layers. We also believe in the existence of unidentified exotic chalcogenide superconductors other than the families introduced here.

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