Effect of partial substitution of iron for group IV elements on the structure and superconducting properties of the $Fe(Se_{0.2}Te_{0.8})_{0.82}$ compound

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The structure and superconducting properties of the $Fe_{1-x}(A)_x(Se_{0.2}Te_{0.8})_{0.82}$ ($x = 0.1$, 0.2) compound, where A is Cu, Zn, Bi or Group IV elements (Pb, Sn, Ge), were studied. It was shown that the effect of substitution of some iron atoms on the structure and superconducting properties of the Fe(Se,Te) compound depends on whether a solid solution with a substituting element was formed. If complete dissolution of the replacement element occured (Cu, Zn), then a significant degradation of the superconducting properties was observed. If a separate chalcogenide phase was formed (substitution by Pb, Sn, Ge), then a slight change in the superconducting properties was observed. Such a slight change and even improvement in the superconducting properties upon precipitation of the chalcogenide phase can be associated with both the positive effect of an increase in the relative content of iron and a decrease in the number of iron atoms in the interstitial sites of the β-phase. The preservation of the superconducting properties of the Fe(Se,Te) compound with a small substitution of iron for group IV elements can be used to create wires based on Fe(Se,Te), since impurity chalcogenide particles can act as effective pinning centers.

Keywords: Iron-based superconductor, FeSeTe, critical parameters, substitution, structure, electrical resistivity, magnetoresistance, pressure effect.

DOI: 10.21883/TP.2023.04.55943.252-22

Introduction

Among iron-containing superconductors, the $Fe(Se_{1-x}Te_x)$ compound with a critical temperature of 15 K for the optimal composition favorably differs by a relatively simple structure (tetragonal structure of the PbO — β- phase), the absence of toxic elements in its composition and stability when stored in air [1]. The large critical magnetic fields of this compound (more than 50 T at 4.2 K) make it possible to consider it as a promising material for creating superconducting wires in the helium temperature range. It was earlier shown that the compound $Fe(Se_{1-x}Te_x)$ can be obtained from a melt with short synthesis times [2], and $Fe(Se_{1-x}Te_x)$ superconducting coatings can be formed on the surface of a pure iron strip during the deposition of coatings from chalcogen vapor or from chalcogenide melt with critical superconductivity parameters close to those of bulk polycrystals [3]. The superconductivity of iron chalcogenides, including doped and intercalated compounds FeSe and $FeTe_{1-x}Se_x$, is considered in the review [4].

Two experimental observations, an increase in the critical temperature of the initial FeSe compound from $T_{cr} = 8$ K to $T_{cr} = 15$ K at the substitution of selenium by tellurium [5], as well as a significant increase in the critical temperature of superconducting iron chalcogenides under pressure (up to 37 K) [6], initiated a large number of works on the partial replacement of elements in the $Fe(Se_{1-x}Te_x)$ compound to increase the critical parameters of superconductivity. An increase in the critical temperature T_{cr}

above 30 K was found in the compound $A_xFe_2Se_2$ [7–9], and up to 45 K in the compound $A_x(NH_3)_yFe_2Se_2$ [10], where A are alkaline, alkaline earth and some rare earth elements. Unfortunately, these compounds are extremely unstable when stored in air, and stability is one of the advantages of the initial materials.

In a number of papers, the partial substitution of iron by metal atoms was studied and it was found that in most cases the substitution does not occur, impurity atoms form compounds with chalcogens, which precipitate out as inclusions, which leads to a change in the stoichiometry of the initial compound and degradation of its superconducting properties [11]. Only some metals (Co, Ni, Cu) enter the lattice of the compound, substituting up to 10% of iron atoms, but the critical temperature decreases sharply even at a few percentages of impurity. Thus, the substitution of 2% of iron atoms by copper atoms leads to the disappearance of superconductivity [11]. Most of the studies on the substitution of elements in the $Fe(Se_{1-x}Te_x)$ compound was carried out in materials obtained by solid-phase (ceramic) technology, since it was assumed that melting incongruity prevents the preparation of the compound from the melt.

In the present work, the structure and superconducting properties of the compound $Fe_{1-x}(A)_x(Se_{0.2}Te_{0.8})_{0.82}$ ($x = 0.1$, 0.2) were studied, where A stands for Cu, Zn, Bi or elements of the IV group (Pb, Sn, Ge). The region of existence of the β-phase in the Fe−Se state diagram is shifted towards an excess of iron; therefore, the composition $Fe(Se_{0.2}Te_{0.8})_{0.82}$ with a little excess of iron was chosen

as an initial one. The superconducting properties of this compound were studied by us earlier [2], and in Ref. [3] it was shown that the ratio of chalcogens $Se_{0.2}Te_{0.8}$ is more promising to obtain coatings than $Se_{0.5}Te_{0.5}$.

Copper and zinc were introduced to compare the results of measurements on samples obtained by melting with the results for ceramic and single-crystal samples. Doping with IV group elements has been little studied earlier, since it was assumed that, due to the lower thermodynamic stability of iron monochalcogenides compared to Ge, Sn, Pb chalcogenide compounds, the formation of two-phase systems will occur and, therefore, the effect of substitution on the structure and properties of the source material will be weak. For example, in Ref. [11] it was found that lead doping of the compound $Fe_{1.05}Te_{0.7}Se_{0.3}$ leads to its complete precipitation in the form of the compound $Fe-Pb-Te$ and thereby changes the ratio of chalcogens in the original compound.

Tin also does not enter the lattice upon substitution in the $Fe-Se-Te$ [12] system, but forms the $SnSeTe$ compound. But at the same time, a small addition of Sn paradoxically promotes the formation of a superconducting phase, the composition of which is closer to ideal, suppresses the formation of the Fe_7Se_8 impurity, contributes to an increase in J_c due to better grain bonding, and leads to an increase in T_{cr} by 3 K. However, at a high concentration of tin (10% and more), the superconducting properties deteriorate due to an increase in the amount of the $SnSe_{0.3}Te_{0.7}$ impurity [13].

The positive effect of adding lead or tin atoms has also been found in iron-containing superconductors with the general formula AFe_2As_2, where A means an alkaline earth metal (Ca, Ba, Sr) or Eu [14–18]. The addition of 5 at.% Pb [14] promotes the growth of grains and improves the bond between them, thereby increasing the value of J_c. The addition of Sn showed similar positive effects in these compounds [19].

In a number of publications, the substitution for antimony of both iron and chalcogen atoms was studied and it was found that, despite the formation and precipitation of FeSb inclusions, the superconducting properties of the matrix do not deteriorate, and the normal state of the material has a lower resistance and a noticeable magnetoresistance [20.21].

Thus, despite the general trend of deterioration of superconducting properties in $Fe-Se-Te$ compounds with partial substitution of iron atoms, there are a number of examples of the positive effect of substitutions, primarily with elements of the IV group. In the present paper, we have carried out a detailed study of the effect of the substitution of iron atoms for elements of the IV group on the structure and superconducting properties of the $Fe(Se_{0.2}Te_{0.8})_{0.82}$ compound .

1. Experiment

Samples of composition $Fe_{1-x}(A)_x(Se_{0.2}Te_{0.8})_{0.82}$, where $A = Sn$, Pb, Ge, Bi, Cu, Zn ($x = 0.05$, 0.1, 0.2), were obtained by cooling the melts in evacuated and sealed quartz ampoules. The heat treatment mode consisted in heating and holding for 6 h at a temperature of 700°C, then raising the temperature to 930−950°C and holding 3 h, cooling of the melt in the off-furnace mode at a rate of 6°C/min. The composition $Fe(Se_{0.2}Te_{0.8})_{0.82}$, with a critical temperature of 12.3 K, was chosen for substitution. The structure and composition of the samples were determined by X-ray diffraction and X-ray phase analysis using a DRON 2 setup (CuK_α radiation with a Ni filter). The phase composition of the samples was determined and the structure parameters were determined (with an accuracy of $1 \cdot 10^{-3}$ Å) for each phase. Measurements of resistance R and magnetic moment M were carried out using a PPMS-14 setup in the temperature range of 300−2 K and magnetic fields up to 14 T. The effect of high pressure on the dependences $R(T, H)$ was studied using autonomous high pressure chambers with a manganin pressure sensor.

2. Results and discussion

The resulting ingots are intergrown flat silver-gray crystals with transverse dimensions of an individual crystal up to 5 mm. Figure 1 shows an image of one of these crystals, which shows its lamellar structure, reflecting the crystallographic structure of the compound.

X-ray analysis showed that the initial compound $Fe(Se_{0.2}Te_{0.8})_{0.82}$ consists of β-phase crystallites with lattice parameters $a = 3.818$ Å, $c = 6.192$ Å. The superconducting parameters of these crystals, ($T_{cr} = 12.3$ K, $\Delta T_{cr} = 0.6$ K, $dH_{cr2}/dT(T_{cr}) = -4.9$ T/K), are comparable with the highest values of these parameters available in the literature.

The table shows the initial composition of some of the studied samples, the parameters of the main tetragonal phase, the content of the main impurity phase, the critical temperature T_{cr} (according to the $R = 0.5R_n$
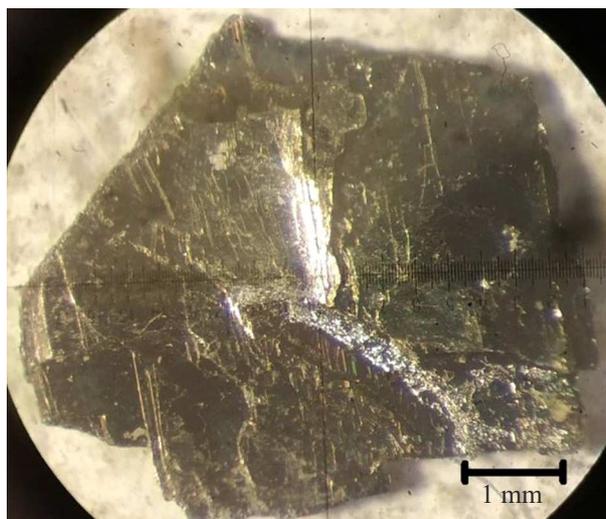
Figure 1. An image of one of the $Fe_{0.8}Pb_{0.2}(Se_{0.5}Te_{0.5})_{0.82}$ crystals chipped from a molten ingot showing its lamellar structure, reflecting the crystallographic structure of the compound.

Initial composition of some studied samples, parameters of the main tetragonal phase, content of the main impurity phase, critical temperature T_{cr}, resistive transition width ΔT_c, and slope of the second critical field dH_{cr2}/dT around T_{cr}

| Composition	Main tetragonal PbO-type phase		Main impurity phase	T_{cr}, K	ΔT_{cr}, K	dH_{cr2}/dT, T/K
	a, Å	c, Å				
$Fe(Se_{0.2}Te_{0.8})_{0.82}$	3.818	6.192	Traces	12.3	0.6	-4.9
$Fe_{0.9}Ge_{0.1}(Se_{0.2}Te_{0.8})_{0.82}$	3.819	6.175	$\sim 10\%$ GeSeTe	14	2.4	-6.3
$Fe_{0.9}Sn_{0.1}(Se_{0.2}Te_{0.8})_{0.82}$	3.808	6.162	$\sim 10\%$ SnTe	10.3	5.0	-4
$Fe_{0.9}Pb_{0.1}(Se_{0.2}Te_{0.8})_{0.82}$	3.804	6.162	$\sim 10\%$ PbTe	12.7	1.5	-6.07
$Fe_{0.95}Cu_{0.05}(Se_{0.2}Te_{0.8})_{0.82}$	3.817	6.156	Traces of Cu	4	–	–
$Fe_{0.95}Zn_{0.05}(Se_{0.2}Te_{0.8})_{0.82}$	3.795	6.067	Traces	11.6	7	–

criterion, where R_n is the resistance in the normal non-superconducting state at the beginning of the transition), resistive transition width $\Delta T_{cr} = \left(T(0.9R_n) - T(0.1R_n)\right)$, and slope of the second critical field dH_{cr2}/dT about T_{cr}.

2.1. Substitution for copper, zinc and bismuth

When iron is replaced by copper and zinc, only traces of impurity phases are observed, i.e., complete dissolution of the substituting component occurs. The parameter c substantially decreases upon substitution, while the parameter a of the crystal lattice depends on the impurity concentration. The resistivity increases significantly, which may be due to stresses caused by fluctuations in the size of the unit cells of solid solutions. The superconducting properties under these substitutions change in different ways: zinc slightly reduces the critical temperature (with a large expansion of the superconducting transition). Copper causes a significant suppression of the superconducting transition: when iron is replaced by 5% copper, the superconducting transition shifts so that the beginning of the transition occurs at 4 K and the end is lower than 2 K, and upon substitution by 10% of atoms, the transition is not observed up to $T \sim 2$ K. In Ref. [11], in the course of studying $Fe_{1-x}Cu_xTe_{0.67}Se_{0.33}$ single crystals, it was found that superconductivity was suppressed already upon substituting 1.5% by copper. The causes of superconductivity suppression when iron is substituted by copper are discussed in [22].

Figure 2 shows the dependence $R(T)$ for the initial compound $Fe(Se_{0.2}Te_{0.8})_{0.82}$ and for the compound with 5% substitution of iron atoms by copper atoms, and 10% substitution by germanium and lead atoms.

When replacing iron with bismuth, there was a spread in the properties of the samples obtained in one technological cycle. The reason for this spread may be the inhomogeneity of the distribution of bismuth in the casting, associated with the sedimentation of heavy bismuth oxide in the melt. An example of such sedimentation is the formation of different phases (phases with different stoichiometry) along the height of a Bi−HTSC ingot, obtained by remelting under increased gravity [23].
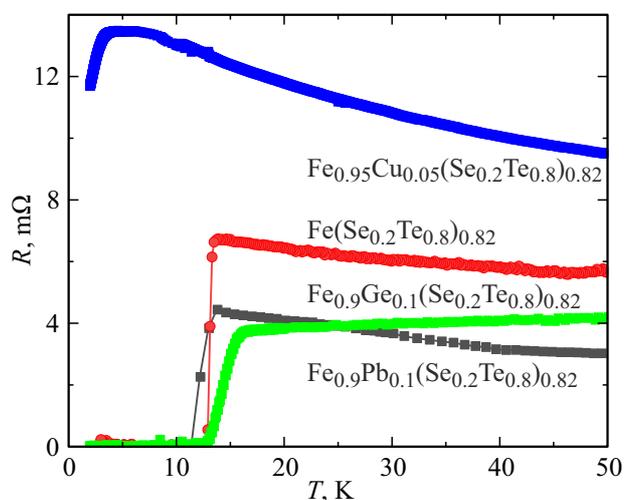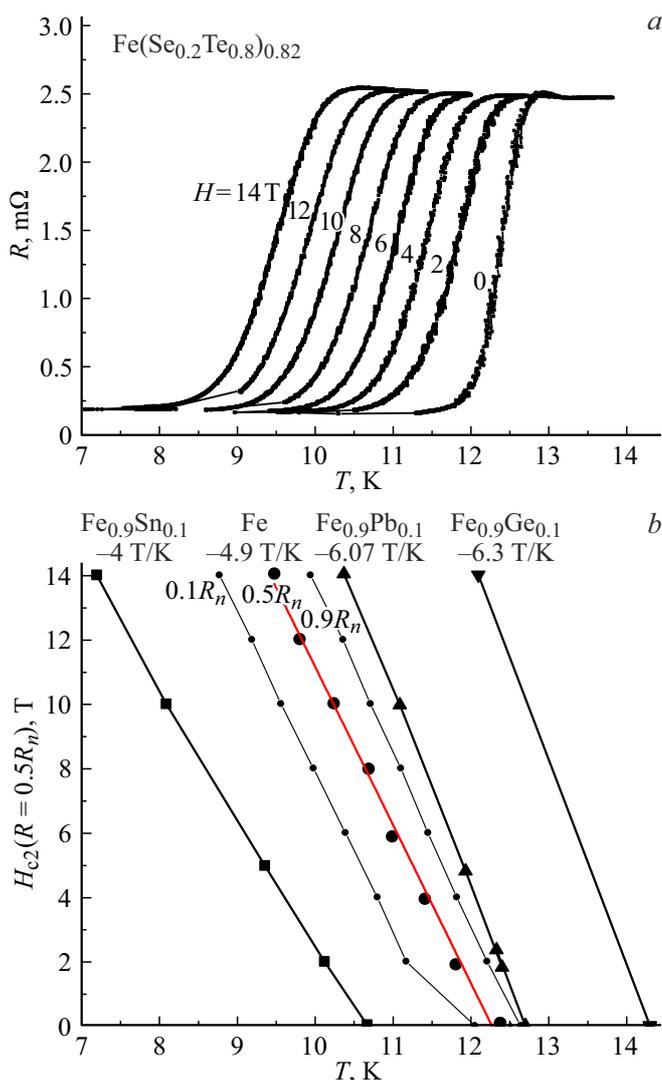

Figure 2. Dependence of $R(T)$ for the initial $Fe(Se_{0.2}Te_{0.8})_{0.82}$ and for samples $Fe_{0.9}Pb_{0.1}(Se_{0.2}Te_{0.8})_{0.82}$, $Fe_{0.95}Cu_{0.05}(Se_{0.2}Te_{0.8})_{0.82}$ and $Fe_{0.9}Ge_{0.1}(Se_{0.2}Te_{0.8})_{0.82}$.

2.2. Substitution for tin and lead

When part of the iron was replaced by Sn and Pb, the resulting samples consisted of a tetragonal solid solution (β-phase) with lattice parameters different from those of the initial compound and a significant amount of a separate phase of tin or lead telluride. Thus, for the $Fe_{0.9}(Sn)_{0.1}(Se_{0.2}Te_{0.8})_{0.82}$ system, the β-phase parameters are $a = 3.808$ Å, $c = 6.162$ Å and all of the introduced tin forms the SnTe compound ($T_m = 806°C$) rather than forming a solid solution with iron chalcogenide. For the $Fe_{0.9}Pb_{0.1}(Se_{0.2}Te_{0.8})_{0.82}$ system, the β-phase parameters are $a = 3.804$ A, $c = 6.162$ A and lead also does not enter the β-phase, but forms PbTe telluride ($T_m = 924°C$).

It follows from the table and Fig. 2, 3 that when 10% of iron atoms are replaced by lead atoms, the critical temperature slightly increases from 12.3 to 12.7 K, and the slope of the temperature dependence of the second critical field H_{C2} near T_c increases from 4.9 T/K for the original compound to 6.07 T/K for $Fe_{0.9}Pb_{0.1}(Se_{0.2}Te_{0.8})_{0.82}$. When 10% of iron

Figure 3. *a* — dependence $R(T)$ for $Fe(Se_{0.2}Te_{0.8})_{0.82}$ in the temperature range $T = (7-14)$ K at different fields $H = (0-14)$ T. *b* — dependence of the second critical field H_{cr2} on temperature T near the critical temperature T_{cr} ($R = 0.5R_n$) for the initial compound and for compounds with substitution of 10% of iron atoms for lead, tin, and germanium atoms.

atoms are replaced by tin atoms, the critical temperature and the slope of the second critical field somewhat decrease ($T_{cr} = 10.3$ K and $dH_{cr2}/dT = 4$ T/K, respectively), which is significantly less than when substituting for copper. The slope of the temperature dependence of the second critical field H_{cr2} near T_{cr} was determined from the middle of the superconducting transition ($R = 0.5R_n$). For the original unalloyed composition, the temperature dependences of H_{cr2} determined for $R = 0.1R_n$ and $0.9R_n$ are also given.

Thus, when iron is partially replaced by elements Pb and Sn of the IV group, the resulting compounds have properties similar to those of the initial compound, namely, the parameters of the main tetragonal phase change insignificantly, almost all of the introduced impurity precipitates

in the form of a separate chalcogenide phase, the critical temperature and the slope of the second critical field change slightly, and the transition width noticeably increases. The broadening of the transition can be associated with an increase in the inhomogeneity of the sample in terms of the content and ratio of chalcogens due to the precipitation of the chalcogenide phase. At the same time, resistance measurements showed insignificant changes in resistivity upon precipitation of chalcogenide phases. In this connection, the increase in the critical field and dH_{cr2}/dT for samples with phase precipitation cannot be explained only by an increase in resistivity. Such a change in properties upon substitution of iron for elements of the IV group differs significantly from the changes upon substitution for Cu and Zn, and also, as shown in [11], upon substitution for many other metal elements.

Another difference in the changes caused by the substitution for group IV elements is the appearance of a large magnetoresistance in samples with substitution, which is practically absent in the original compound.

Figure 4 shows the $R(T)$ dependences for the $Fe_{0.9}Pb_{0.1}(Se_{0.2}Te_{0.8})_{0.82}$ sample, measured without a magnetic field and at $H = 5$ T. It can be seen that as the temperature drops below about 200 K, there is an ever increasing magnetoresistance. The inset in Fig. 4 shows the dependences $R(H)/R(0)$ for systems with tin and lead measured at 16 K in a 14 T field. The resistance of the sample with lead increases at this temperature by 37%. This value is greater than the magnetoresistance 28% (under close conditions 10.5 K and 12 T) for the $Fe_{1.11}Se_{0.95}Sb_{0.05}$ system, which was considered previously record-breaking for chalcogenide iron-containing superconductors [21].

The magnitude of the magnetoresistance in the normal state depends on the type and amount of the substituting element, and on the initial ratio of chalcogens. For example, the magnitude of the magnetoresistance at 15 K
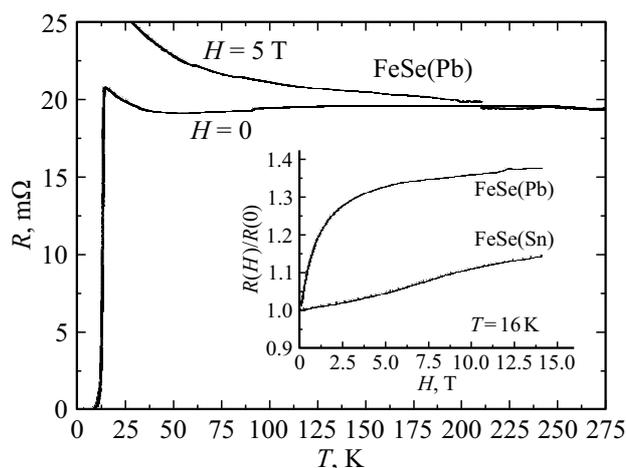
Figure 4. Temperature dependence of the resistance in the system $Fe_{0.9}(Pb)_{0.1}(Se_{0.2}Te_{0.8})_{0.82}$ in a magnetic field $H = 0$ and 5 T. The inset shows the magnetoresistance in the normal state for the $Fe(Se_{0.2}Te_{0.8})_{0.82}$ system with partial replacement of iron by tin and lead.
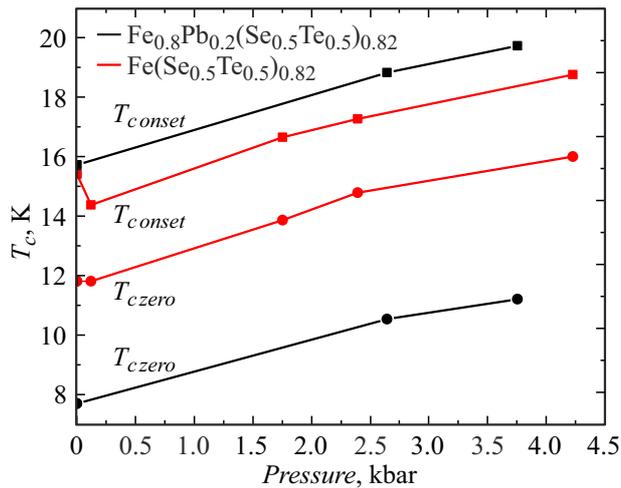
Figure 5. Dependence of the temperature of the beginning T_{Conset} and the end T_{Czero} of the superconducting transition on the applied pressure for the initial compound $Fe(Se_{0.5}Te_{0.5})_{0.82}$ and lead-doped $Fe_{0.8}Pb_{0.2}(Se_{0.5}Te_{0.5})_{0.82}$.

and $H = 14\,T$ in the compound $Fe_{0.8}(Pb)_{0.2}(Se_{0.5}Te_{0.5})_{0.82}$ is 27%, for $Fe_{0.9}(Pb)_{0.1}(Se_{0.2}Te_{0.8})_{0.82}$ it is more than 37%, and about 15% for $Fe_{0.9}Sn_{0.1}(Se_{0.2}Te_{0.8})_{0.82}$.

Due to the fact that substitution with lead leads to significantly different results than with substitution by many other elements, as shown above, it was decided to test the effect of pressure on the superconducting properties of lead-doped samples and compare them with unalloyed samples.

For the measurements, the $Fe_{0.8}Pb_{0.2}(Se_{0.5}Te_{0.5})_{0.82}$ compound was used, because for other substitutions at this doping level (20% substitution of iron) superconductivity already disappears, and when replaced by Pb, the compound possesses superconductivity with $T_{cr} = 10.2\,K$. The $Fe_{0.8}Pb_{0.2}(Se_{0.5}Te_{0.5})_{0.82}$ sample was placed in a high-pressure chamber, where it was subjected to all-round compression. The dependences of resistance on temperature were measured at magnetic fields up to $14\,T$ and pressures up to $7.1\,kbar$ ($300\,K$) and up to $3.75\,kbar$ ($5\,K$).

The applied pressure leads to a monotonic increase in the superconducting transition temperature for both the initial compound and the doped sample. Figure 5 shows the temperature dependences of the beginning and end of the superconducting transition on the applied pressure for the sample doped with lead and for the initial one. It can be seen that the increase in these temperatures with increasing pressure is almost identical for both compounds, with dT_{cr}/dP being $\approx 1\,K/kbar$. This value is somewhat higher than the values obtained for poly- and single-crystal compounds of the same composition. For example, in Refs. [24,25] the value of $dT_{cr}/dP \approx 0.7\,K/kbar$ was obtained, which may be due to the higher quality of our samples obtained by melting.

Measurements in magnetic fields showed (Fig. 6) that the slope of the critical field near the critical temperature $dH_{cr2}/dT\,(T_{cr})$ does not depend on the applied pressure and is equal to $4.24\,T/K$ for $Fe_{0.8}Pb_{0.2}(Se_{0.5}Te_{0.5})_{0.82}$
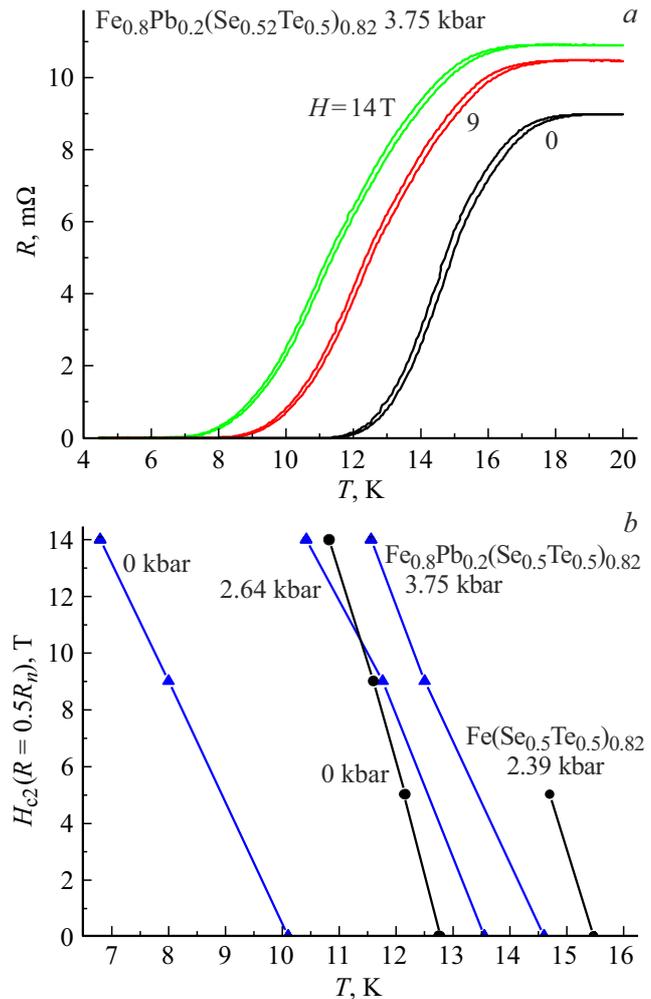
Figure 6. a — temperature dependence of resistance for $Fe_{0.8}Pb_{0.2}(Se_{0.5}Te_{0.5})_{0.82}$ in external field 0, 5, 9, 14 T. b — dependence of T_{cr} on the magnetic field at different pressures for samples with and without lead substitution.

and $4.9\,T/K$ for $Fe(Se_{0.5}Te_{0.5})_{0.82}$. Thus, measurements under all-round compression showed that:

— when pressure is applied, the increase in the critical temperature of the initial compound obtained by melting is somewhat higher than for samples of the same composition [24,25];

— when iron is replaced by a sufficiently large amount of lead, the increase in T_c with pressure practically coincides with the value of dT_{cr}/dP for the initial composition and amounts to $\approx 1\,K/kbar$;

— the slope of the second critical field near T_{cr} is somewhat smaller for the lead-doped sample ($4.24\,T/K$ for $Fe_{0.8}Pb_{0.2}(Se_{0.5}Te_{0.5})_{0.82}$ and $4.9\,T/K$ for $Fe(Se_{0.5}Te_{0.5})_{0.82}$), and is independent of pressure for both compositions.

2.3. Substitution for germanium

When 10% of Fe atoms are replaced by Ge atoms, the structure of the initial $Fe(Se_{0.2}Te_{0.8})_{0.82}$ phase changes
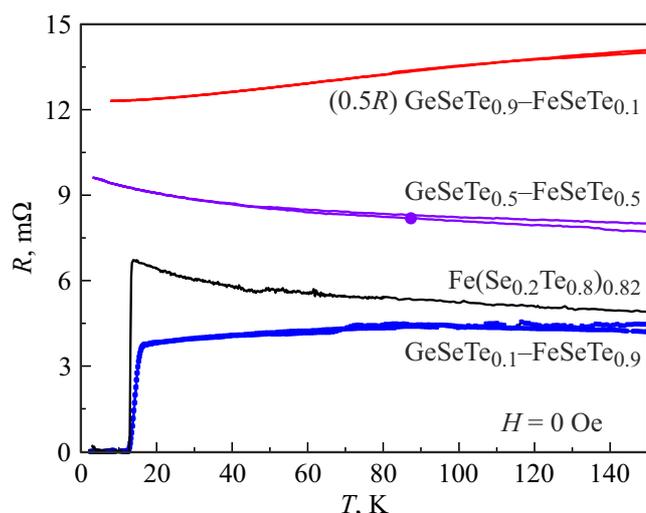
Figure 7. Dependence of resistance on temperature for GeSeTe−FeSeTe system for concentrations of Ge = 0, 0.1, 0.5, 0.9 . For 90% substitution with germanium, the half-percent dependence $R = 0.5\% R_n$ is indicated.

slightly — the parameter a remains practically unchanged, and the parameter c decreases, and almost all of the introduced Ge precipitates as a compound of the GeSeTe system of various stoichiometry. The resulting samples have an increased transition temperature $T_c = 14$ K (Fig. 7) and increased values of the critical magnetic field near the critical temperature $dH_{cr2}/dT(T_{cr}) = 6.3$ T/K. Thus, when ten percent of the iron atoms are replaced by germanium, the critical temperature T_{cr} and the slope of the critical field $dH_{cr2}/dT(T_{cr})$, just as in the case of lead substitution, do not decrease, as for most metal substitutions, but increases.

The GeSeTe compound has a high glass-forming capacity, and indeed, in experiments with rapid cooling of the melt, it was found that this phase is fixed in the samples in both the crystalline and amorphous states. At a high content of Ge in the initial composition, a composite material was obtained, in which the crystalline phase of the Fe(Se,Te) composition is immersed in a matrix consisting mainly of the amorphous phase of the GeSeTe composition. Figure 7 shows the dependence of the resistance on temperature for the GeSeTe−FeSeTe system with a change in the concentration of the components. It can be seen that superconductivity is observed only for undoped and lightly doped samples, and at a high content of germanium, the resistance in the normal state increases sharply. Figure 8 shows the corresponding changes in the X-ray diffraction pattern of the samples. It is seen that with an increase in the germanium content, amorphization of the resulting compound occurs, at $x = 0$ clear peaks are visible, and with an increase in substitution, the peaks begin to gradually spread out, and at a value of 0.9, individual peaks are no longer visible.
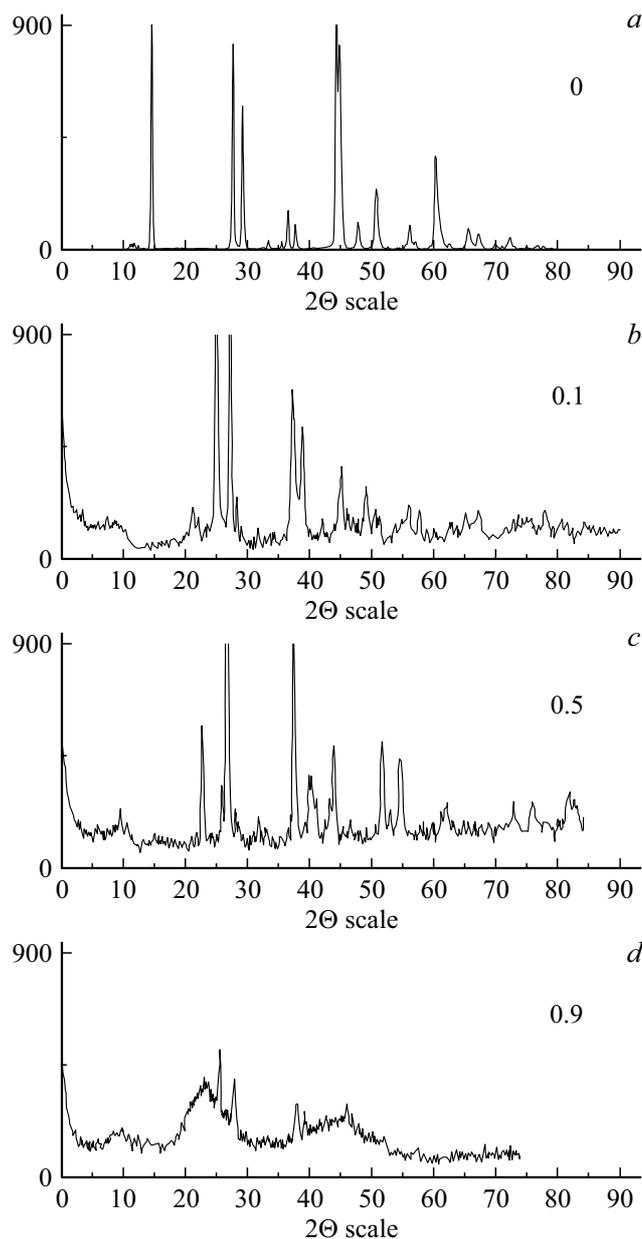
Figure 8. X-ray phase analysis of $Fe_{1-x}(Se_{0.2}Te_{0.8})_{0.82}$ $-Ge_x(SeTe)$ with different substitution of iron for germanium, x: a — 0, b — 0.1, c — 0.5, d — 0.9.

Conclusion

Thus, it is shown that the effect of substitution of a part of iron atoms on the structure and superconducting properties of the Fe(Se,Te) material depends on whether the complete dissolution of the substituting component occurs (and then a significant degradation of the superconducting properties is observed) or whether a separate chalcogenide phase precipitates (and then a slight change in the superconducting properties is observed). This experimental observation looks somewhat paradoxical, since the entire introduced impurity precipitates in the form of the corresponding chalcogenide and does not enter the lattice of the main phase. Neverthe-

less, the main phase shows either improved superconducting properties, i.e. increased T_{cr} and dH_{cr2}/dT (Pb,Ge), or not greatly degraded properties (Sn). This behavior can be due to two circumstances. First, when stoichiometric impurity chalcogenides precipitate in the initial compound, the relative content of iron increases slightly, and as was shown in Ref. [26], β-phase expands towards excess iron. Second, with the introduction of these elements, a decrease in the number of iron atoms in the interstitial positions of the β-phase can occur. The effect of interstitial iron on the superconducting properties of the β phase has been widely discussed in the literature [27,28] and was used to analyze the results on the substitution of antimony for iron in the Fe−Se compound [21].

Thus, the preservation of the superconducting properties of the Fe(Se,Te) compound with a small substitution of iron for elements of the IV group, and the appearance of impurity chalcogenide particles, which can act as effective pinning centers, can be used to create wires based on Fe(Se, Te).

Conflict of interest

The authors declare that they have no conflict of interest.

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