



# Characterization of the phase composition, crystal structure and superconducting properties of $\text{Fe}_{1.02}\text{Se}_y\text{Te}_{1-y-x}\text{S}_x$



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## ABSTRACT

Two series of the  $\text{Fe}_{1.02}\text{Se}_{0.5}\text{Te}_{0.5-x}\text{S}_x$  (I) and  $\text{Fe}_{1.02}\text{Se}_{0.4}\text{Te}_{0.6-x}\text{S}_x$  (II) samples with the sulfur for tellurium substitution and with the invariable Se concentrations have been synthesized and studied by means of X-ray diffraction, scanning electron microscopy, electrical resistivity and magnetic susceptibility measurements. The superconducting PbO-type phase is found to persist in the first series up to  $x = 0.4$  and in the second one up to  $x = 0.5$ . Despite the lower ionic radius of sulfur in comparison with tellurium the replacement of tellurium by sulfur does not lead to contraction of the unit cell volume of the superconducting phase in both I and II series with ternary mixture of chalcogens. Variations of the lattice parameters caused by the S for Te substitution in the  $\text{Fe}_{1.02}\text{Se}_{0.5}\text{Te}_{0.5-x}\text{S}_x$  and  $\text{Fe}_{1.02}\text{Se}_{0.4}\text{Te}_{0.6-x}\text{S}_x$  samples are found to be less pronounced than that reported for the  $\text{Fe}_{1.02}\text{Te}_{0.5}\text{Se}_{0.5-x}\text{S}_x$  system and are accompanied by lowering of the critical temperature. The behavior of the lattice parameters and critical temperature of Fe(S,Se,Te) materials with the ternary mixture of chalcogens at substitutions is ascribed to the changes in the volume fraction and chemical compositions of the coexisting tetragonal and hexagonal phases.

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## 1. Introduction

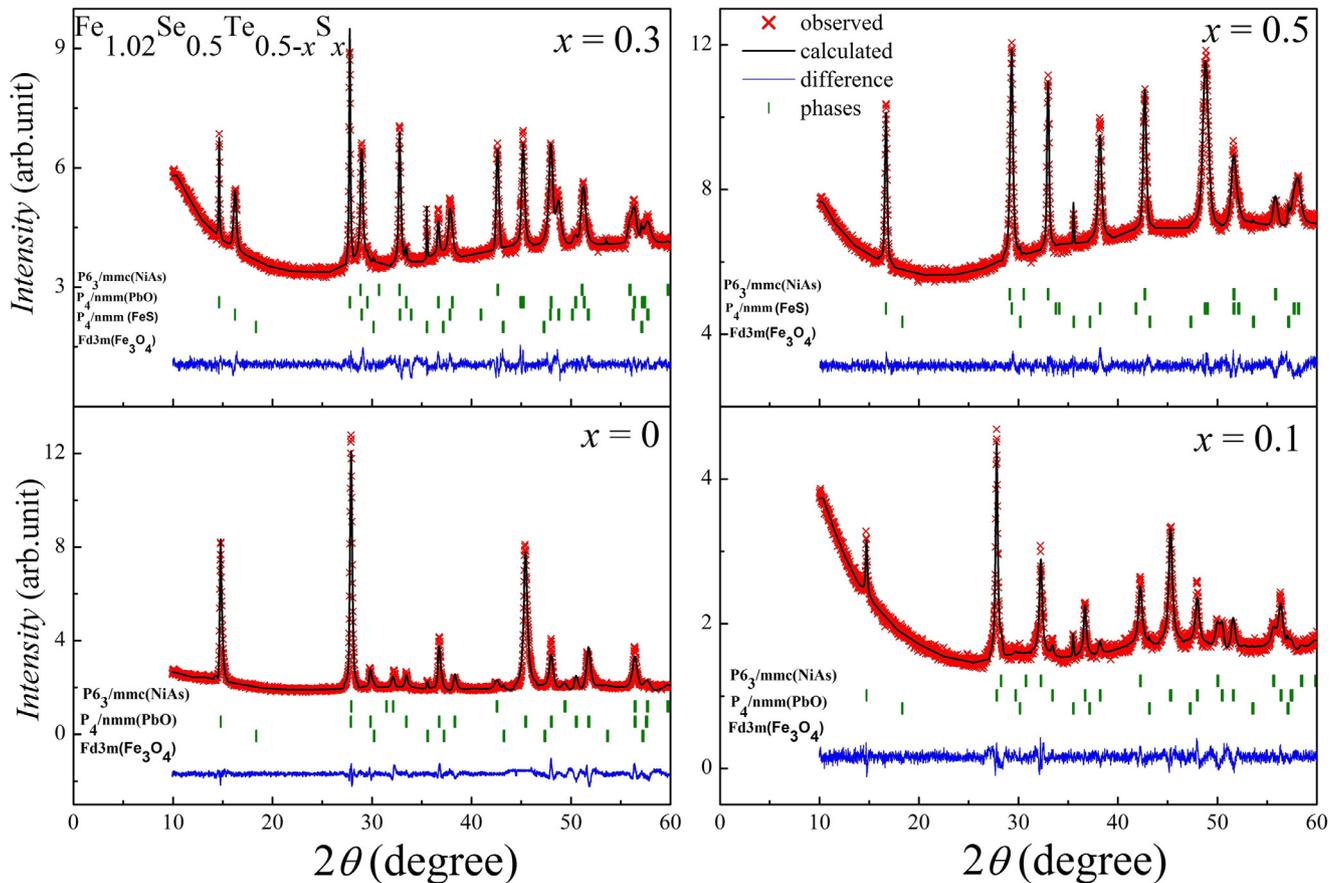
The discovery of new iron-based superconductors has initiated advanced search for new superconducting compounds and key factors influencing their properties [1–5]. The common features of these materials are: (i) the layered crystal structure consisting of a set of square lattices of Fe atoms with pnictogen or chalcogen atoms; (ii) the similarity in the morphology of the Fermi surface; and (iii) the interplay between superconductivity and magnetism [4–6]. The FeSe (11) type compounds have a tetragonal crystal structure of the PbO-type ( $P4/nmm$  space group) with layers in which Fe cations are tetrahedrally coordinated by chalcogen and with no atoms located within the van der Waals gap. Owing to the simplicity of the crystal structure and similarities in the Fermi surface the FeSe-type compounds can be used as model systems for studying the mechanisms of superconductivity in other Fe-based superconductors having more complex crystal structures. The superconducting critical temperature ( $T_c$ ) of FeSe is found to be strongly affected by doping and by substitutions in both the Fe and chalcogen sublattices, as well as by application of hydrostatic pressure and strains (see, e.g., Ref. [4–6] for overview). The  $T_c$  values increased up to  $\sim 80$ – $100$  K were recently reported for the

FeSe monolayer film grown on the  $\text{SrTiO}_3$  substrates [7,8]. In previous studies [9–14], the substitution effects on the crystal structure and properties of iron chalcogenide superconductors were investigated in the compounds with different binary mixtures of chalcogens (Se–Te, Se–S, Te–S). As was shown recently in [15], the presence of all three chalcogens in the  $\text{Fe}_{1.02}\text{Te}_{0.5}\text{Se}_{0.5-x}\text{S}_x$  compounds results in unusual change of the lattice parameters with substitutions.

Bearing in mind that the  $\text{Fe}(\text{Se},\text{Te})_y$  samples often exhibit an impurity hexagonal phase of the NiAs-type together with the main superconducting phase having a tetragonal PbO-type phase [16] the aim of the present work is to study how the coexistence of the tetragonal and hexagonal phases and the changes in their volume fractions, chemical composition and lattice parameters may affect the superconducting properties of the 11-type iron-chalcogenide materials. We have synthesized the  $\text{Fe}_{1.02}\text{Se}_{0.5}\text{Te}_{0.5-x}\text{S}_x$  and  $\text{Fe}_{1.02}\text{Se}_{0.4}\text{Te}_{0.6-x}\text{S}_x$  samples with the fixed Se content and studied the effect of the substitution of sulfur for tellurium taking into account the changes in the phase composition, lattice parameters and volume fractions of both phases bearing in mind a narrow homogeneity range of the superconducting phase and a different chalcogen solubility in both tetragonal and hexagonal phases.

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**Fig. 1.** Observed (symbols) and calculated (line) patterns for the samples  $\text{Fe}_{1.02}\text{Se}_{0.5}\text{Te}_{0.5-x}\text{S}_x$  (series I) with various sulfur concentrations. Vertical bars indicate positions of Bragg reflection for the existing phases. The difference between calculated and observed intensities is shown in the bottom.

## 2. Experimental details

Two series of polycrystalline samples with nominal compositions  $\text{Fe}_{1.02}\text{Se}_{0.5}\text{Te}_{0.5-x}\text{S}_x$  ( $x = 0, 0.1, 0.2, 0.3, 0.4, 0.5$ ) (series I) and  $\text{Fe}_{1.02}\text{Se}_{0.4}\text{Te}_{0.6-x}\text{S}_x$  ( $x = 0, 0.1, 0.2, 0.3, 0.4, 0.5, 0.6$ ) (series II) were prepared by a solid state reaction method. In order to achieve a better homogeneity of final samples we used a ternary alloy of chalcogens as a precursor instead of a simple mixture of pure chalcogens. At first, the starting materials: selenium, tellurium and sulfur (all of 99.99% purity) were sealed into evacuated quartz tubes and heat treated at 200 °C for 2 h and then for 2 h at 400 °C. The resulting alloy of chalcogens was ground ground into a powder and mixed with an appropriate amount of the iron powder (of 99.98% purity). The obtained mixture was pressed into pellets. In order to reach homogeneity we used a prolonged annealing procedure: the first annealing at 500 °C for 10 h, then at 700 °C for 38 h; the second annealing at 700 °C for 120 h. The obtained samples were again ground, pelletized, and then additionally heat treated third time at 700 °C for 72 h. This method of the sample preparation differs from that used in our previous work [15], where the  $\text{Fe}_{1.02}\text{Te}_{0.5}\text{Se}_{0.5-x}\text{S}_x$  compounds were prepared from the mixture of pure chalcogens and iron powder. Beside these polycrystalline samples, two coarse-grained samples with nominal compositions  $\text{Fe}_{1.02}\text{Se}_{0.5}\text{Te}_{0.4}\text{S}_{0.1}$  and  $\text{Fe}_{1.02}\text{Se}_{0.4}\text{Te}_{0.3}\text{S}_{0.3}$  were prepared by melting at 950 °C with following cooling to room temperature for 48 h. The structure and phase purity of the samples were examined at room temperature by powder X-ray diffraction using a Bruker D8 Advance diffractometer with Cu K $\alpha$  radiation. The FULLPROF program (Le Bail fit) [17] was used for analysis of diffraction patterns. The chemical composition of the melted samples

was determined by using a Carl Zeiss AURIGA Crossbeam Workstation equipped with an energy dispersive X-ray (EDX) spectrometer. The electrical resistivity of polycrystalline samples was measured by a four-probe dc method in the temperature range from 8 up to 300 K with using a closed-cycle refrigerator. For the magnetization measurements, a SQUID magnetometer MPMS XL-7 EC was used. From the magnetization data, the superconducting volume fractions were estimated taking into account the demagnetizing factor of the samples.

## 3. Results and discussion

Figs. 1 and 2 display the X-ray diffraction patterns for the samples belonging to the series I and series II with the sulfur concentration ( $x = 0, 0.1, 0.3, 0.5$ ) correspondingly along with the Rietveld refinements using the Fullprof program. For the series I (Fig. 1), the patterns at  $x \leq 0.1$  exhibit the Bragg reflections belonging to: (i) the dominating tetragonal PbO-type phase ( $P4/nmm$  space group), (ii) the impurity phase with a hexagonal NiAs-type structure ( $P6_3/mmc$  space group), and (iii) iron oxide  $\text{Fe}_3\text{O}_4$  with cubic structure were detected as well. As can be seen from Fig. 1, the peaks of the hexagonal phase begin to grow with increasing sulfur content. Starting from  $x = 0.3$  the peaks of a tetragonal FeS-based phase with the mackinawite structure ( $P4/nmm$  space group) appear and begin to grow as well. We noticed that the lattice parameters of the mackinawite-type phase observed in all our samples differ from those reported for FeS. Thus, for the  $\text{Fe}_{1.02}\text{Se}_{0.5}\text{Te}_{0.2}\text{S}_{0.3}$  sample, the lattice parameters of this phase were determined as:  $a = 3.73 \text{ \AA}$  and  $c = 5.46 \text{ \AA}$  which are higher than  $a = 3.68 \text{ \AA}$  and

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