THE STUDY OF STRUCTURAL, ELECTRONIC AND SPIN STATES OF IRON IONS IN THE COMPOUNDS OF THE FAMILY FeSeTe BY MOSSBAUER SPECTROSCOPY



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Experimental

Powder samples Fe_{1+x}Te and FeSe_{1-x}Te_x were prepared at the Chemistry Department of Lomonosov Moscow State University from Fe (freshly reduced by hydrogen at 400°C for 1 hr), Se and Te. All chemicals were of analytically or extra pure grade. Mixtures of chemicals with aimed stoichiometry $Fe_{1+x}Te(x = 0 - 0.2)$ and $FeSe_{1-x}Te_x(x = 0 - 0.2)$ 0.1—0.8) were thoroughly ground, pressed into pellets, sealed in evacuated quartz tubes (at the residual pressure of ca. 0.05 Torr), and annealed twice at 700°C for 48 hrs with one intermediate re-grinding and re-pelletizing. All FeTe samples were nonsuperconducting. Only two of FeSeTe samples were superconduting and enough singlephase: FeSe_{0.5}Te_{0.5} ($T_c \approx 14$ K) and FeSe_{0.2}Te_{0.8} ($T_c \approx 13$ K).

Initial platelike crystals FeSe_{1-x} were grown in the Lebedev Physical Institute RAS (Moscow, Russia) from a potassium chloride flux. Elements have been used high purity Fe (99.98%), Se (99.99%) and KCI (99.99%) or pre-synthesized FeSe. The crystal growth was carried out in evacuated and sealed quartz ampoules placed in a furnace with a vertical heater at 835°C. All samples were superconducting with $T_c \approx 9$ K.

Powder X-ray diffraction studies were performed in the Lomonosov Moscow State University on the Bruker D8 Advance diffractometer (Cu-K_α1 radiation, Ge-111 monochromator, reflection geometry) between 20 = 13—100° and in the National Research Center "Kurchatov Institute" (Moscow, Russia) on the "Belok" station installed on the synchrotron source (λ = 0.9886 Å) with MARRESEARCH CCD detector (2048×2048).

The Mossbauer absorption spectra from ⁵⁷ Fe nuclei were recorded in the Shubnikov Institute of Crystallography RAS (Moscow, Russia) in 5—295 K temperature interval in transmission geometry with standard MS-1100Em spectrometers operating in the constant acceleration regime and equipped with a closed-cycle helium cryostat RTI CryoFree-104. The source of γ-quanta [⁵⁷Co(Rh)] was at room temperature. Isomer shifts were measured relative to the reference α-Fe sample at room temperature.

80 - y = 0.08

60

20

Rel. absorption

T (K)

 $Fe_{1+x}Te$

The low-temperature Mossbauer spectra for x = 1.125 exhibit complex magnetic structure with Neel point about 62 K. But there is no clear understanding of the nature of such a complex behavior.



It is well known from the literature that iron in the regular tetrahedral sites develops a bicollinear antiferromagnetic structure in *ab*-plane, transforming into an incommensurate 3D magnetic order upon increasing x. The crystal structure of Fe_{1+x} Te with the layers consisting of edge-sharing FeTe₄ tetrahedra. The interstitial iron sites, shown as beach ball spheres, are partially occupied and disordered (but how?).

FeSe_{1-x}Te_x

Fe_{1+v}Se_xTe_{1-v}

O T_{SDW}

 OT_{SG}

 OT_c

0.8

1.0

aramagnetic Metal

0.6

There are two of the most interesting problems for FeSeTe system. 1. Why is FeSe system superconductor but structurally similar FeTe system is not?

> 2. What is the role of the magnetic ordering of iron ions in the formation of the superconducting state?

> Mossbauer studies of compounds with the different Se/Te relative content can give some answers. Room temperature (RT) Mossbauer spectra for six **FeSe_{1-x}Te_x** samples are mainly paramagnetic (left figure), samples with x = 0.1, 0.2, 0.3 have low intensity magnetic sextets from FeSe impurity phases.

Se content, (x)J. WEN et al // Rep. Prog. Phys. 2011. 74. 124503.

0.4

0.2

In all FeSeTe samples iron does not have excess. We hoped to get different low temperature magnetic ordering at the 5 K for samples with x = 0.5 and 0.8, because

upon the increasing of the relative Te concentration FeSeTe lattice should become more like the FeTe lattice. But there are **no difference and complex magnetic structure of the** spectra for both samples at 5 K (right figure). This result confirms that the excess iron

 $FeSe_{1-x}Te_x$

can be responsible for the formation of the complex magnetic ordering in the both FeSeTe and FeTe systems.





Magnetic-crystallographic phase diagram for Fe_{1+x} Te constructed by plotting the δ of the propagation vector $k = (\delta 0 1/2)$ versus concentration of interstitial iron at base temperature [E. RODRIGUEZ et al // Phys. Rev. B. 2011. 84.064403].



Another phase diagram with complex magneto-structural transition and different magnetic states [S. ROßLER et al // Phys.Rev. B. 2011. 84. 174506]. We hope that our low-temperature Mossbauer data for all Fe_{1+x}Te samples will give additional useful information about the nature of that magnetic-structural transition.

Velocity (mm/s)

90 K

-12 Velocity (mm/s)

12

FeSe_{1-x}

Two samples (x = 0.9 and 0.8) were studied, for both RT spectra consist of the main paramagnetic doublet which correspond iron ions in regular tetrahedral sites and two

> low intensity magnetic sextets which correspond two 295 K different hexagonal phases FeSe and Fe₇Se₈. Temperature 160 K dependences of the hyperfine magnetic field of the sextets shows sharp jump in region of the superconducting 120 K transition. We assume that nonsuperconducting magnetic hexagonal phases may feel superconducting transition in the main nonmagnetic superconducting phase.



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Rel.