Experimental Report

Proposal: **5-15-606** **Council:** 4/2014

Title: Crystal structure of two Fe1+y(Te1-xSex) x=0.4 single crystals as a function of a heat treatment

This proposal is a new proposal

Researh Area: Physics

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Samples: Fe1+y(Te1-xSex) x=0.4: two single crystals

Instrument	Req. Days	All. Days	From	To
D10	9	4	14/10/2014	18/10/2014

Abstract:

In the proposed experiment we suggest to study two Fe1+y(Te1-xSex) x=0.4 single crystals that differ in the sample preparation. Although having the same overall composition, one sample is homogeneous, the other consisting from two phases (this sample appears to be superconducting). In the latter case have inclusions the same orientation with respect to the matrix enabling thus the crystal structure determination of both the matrix and the secondary phase. We suggest the room-temperature and a heat-treatment (up to 800 K) study of the inhomogeneous sample along with the room temperature study of the homogeneous sample. We anticipate that at elevated temperatures the inhomogeneous sample becomes homogeneous again and both systems will have the same crystal structure. The study should be able to resolve controversal claims in the literature concerning the pahse purity and question which phase hosts the superconductivity.

Report of the D10 experiment on $FeSe_{0.4}Te_{0.6}$

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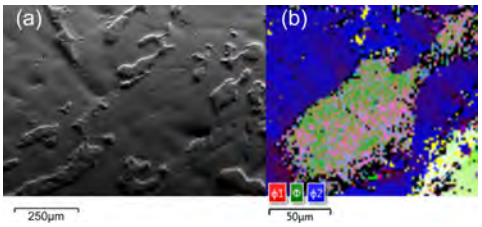

FIG. 1. SEM (a) and EDX (b) images of the surface of superconducting $FeSe_{0.4}Te_{0.6}$.

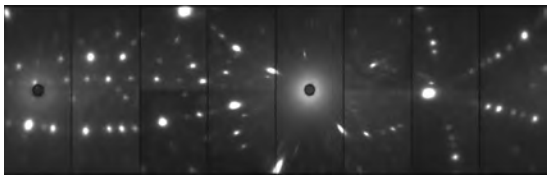

FIG. 2. Neutron Laue diffractogram obtained with CYCLOPS at ILL Grenoble. Alongside the structural main peaks of the crystals matrix, several minor peaks due to the inclusions are visible.

I. INTRODUCTION

As one of the structurally and stochiometrically simplest iron-based superconductor $FeSe_xTe_{1-x}$, the investigation of this compound has been of great interest. However, the mechanism of superconductivity remains still unclear. Often superconducting $FeSe_xTe_{1-x}$ samples are not perfect single crystals, but show a secondary phase.

Several recent publications revealed that the impact of those secondary phases must be taken into account to interpret and understand the properties of the Fe-Se-Te system.[1][2] In particular, it turns out that those foreign phases are a mandatory ingredient for the observation of bulk superconductivity.

EDX measurements with our superconducting $FeSe_{0.4}Te_{0.6}$ sample show rather big (up to 250m) inclusions of this secondary phase (Fig. 1). This EDX-data and x-ray powder diffraction suggest the composition $Fe_3Se_{2.1}Te_{1.8}$ .The goal of the experiment was to perform single crystal diffraction on one of those inclusions.

II. EXPERIMENTS

We analyzed the structure of the inclusion with the 4-circle neutron diffractometer D10 at ILL Grenoble. The experiments have been performed at room temperature with a wavelength of $1.26\mathring{A}$. The sample was glued directly to the sampleholder, since no effects of stress were expected. First we have determined the orientation and structure of the main phase by focusing only on the most intense reflections. The picture in Figure 2 was obtained with the neutron Laue instrument CYCLOPS. The clear difference between main phase and small reflection from inclusions is visible and simplifies the distinction. Afterwards, a scan along one octand has been performed showing several additional non-indexed peaks. These reflections were used to determine the orientation matrices of at least three different $Fe_3Se_{2.1}Te_{1.8}$ grains inside the matrix. The most favourable of them was used to acquire a series of reflections for structure determination. In Figure 1 is noticeble, that besides rather large inclusions with lengths up to 250m, a lot of tiny inclusion exist as well. Furthermore, the contact layer between matrix and inclusion appears to be heavily distorted. Both effect leads to recognizable Debye-ring next to the reflection, which limits the precision of the structure determination. Nevertheless, via careful handeling of the intensity integration it was possible to obtain a good result.

We were able to show via the unrelated UB-matrices that there is no distinct relationship in the orientation between the $FeSe_{0.4}Te_{0.6}$ matrix and the $Fe_3Se_{2.1}Te_{1.8}$ inclusions. Furthermore the Debye-rings and SEM-data suggests a polycrystalline layer in the contact area of both. The refinement of the $Fe_3Se_{2.1}Te_{1.8}$ single crystal data yield a hexagonal order associated with Fe_3Se_4 and closely correspond to our previous investigations.[2] Since we showed the possibility to acquire single crystal data of the secondary phase, it is now a feasible option to study in-situ the influence of annealing.

[1] A. Wittlin, P. Aleshkevych, H. Przybylinska, D. Gawryluk, P. Dluzewski, M. Berkowski, R. Puzniak, M. Gutowska, and A. Wisniewski, Superconductor Science and Technology **25**, 065019 (2012).

[2] M. Schulze, K. Prokes, S. Hartwig, N. Schfer, S. Landsgesell, C. Blum, D. Abou-Ras, M. Hacisalihoglu, E. Ressouche, B. Ouladdiaf, A. Bachmann, C. Hess, B. Buechner, and S. Wurmehl, To be published (2015).