IOSR Journal of Applied Physics (IOSR-JAP)
e-ISSN: 2278-4861.Volume 12, Issue 1 Ser. I (Jan. – Feb. 2020), PP 56-60
www.iosrjournals.org

Effect of KI Addition on FeSe Superconductors by Powder in-Sealed Tube Method

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Abstract: *The results of the effect KI addition to phase identification, critical temperature and electrical resistance in FeSe superconductors are reported. The synthesis process of FeSe superconductors is done conventionally by hand milling for 3 hours followed by a solid state reaction at 845°C for 3 hours and cooled in the open air. Structurally, KI addition to FeSe superconductors material was found to reduce the volume fraction of the main (tetragonal) FeSe superconductors from 85.66% to 79.28% at the addition of 1 wt % and 60.36% at the addition of 5 wt % FeSe superconductors namely 13.33 K to 12.27 K. In this report, we discuss the phases formed, the morphology of the doped material and the resistivity value of each as a function of temperature varying from 5 K to room temperature.*

Keywords: FeSe Superconductors, KI Addition, Temperature

Date of Submission: 30-12-2019 Date of Acceptance: 15-01-2020

I. Introduction

Since the discovery of Fe-based superconductors with a value of Tc = 26K in LaFeAs (O1-xFx) (Kamihara et al, 2008). This discovery has brought new excitement to the superconducting material research community and attracted much attention to finding new Fe-based superconducting compounds. Within a few months in the same year hsu et al reported a new Fe-based superconductor with Tc = 8K in FeSe, although the Tc value obtained was small, the FeSe superconducting material had a simple crystal structure. PbO type FeSe crystal structure is tetragonal phase with space group P4/mmm (β-FeSe). This FeSe superconducting material has the simplest structure among iron-pnictides and other chalchogenides superconductors and finds many interesting properties from them. This is a good starting point for studying and understanding the electrical properties of FeSe superconductors in the form of stoichiometry, doping treatment and the methods applied. Different stoichiometric variations are believed to be extreme sensitivity in the β-FeSe phase.

The treatment of doping and the methods applied provide a way to achieve superconductivity in FeSe superconductors. Substitution of chemical elements in Fe or Se in FeSe can increase superconductivity, such as the addition of Te, S that has been reported to researchers before (Margadonna, 2008). However, FeSe research with doping Family Metal/transition metals (Ti, V, Cr, Mn, Co, Ni, Cu) and non transition metal shows superconductivity suppression due to deformation of the B-FeSe structure and negative effects of the concentrations used. From the elements that have been mentioned in the identification of the nature of superconductivity in previous researchers, this research was carried out with the addition of KI innovation to investigate superconductivity.

In this paper, we report the sintered FeSe sample at 845 ° C, this temperature selection was made on the statement of Li et al (2012) reporting that the higher the heating temperature was treated at 150 ° C-750 ° C, the result of heating 750 ° C in the phase The main β-FeSe is increasingly dominant with a percentage of ~ 80% in FeSe material [10]. The choice of temperature was also chosen based on the DTA test that had been done by the previous researchers, where the results of the DTA test at 750 ° C showed more maximum results. Stoichiometric concentration sequences were selected for phase identification, morphology and resistance properties in FeSe superconductors.

II. Experimental Procedure

2.1 Synthesis of FeSe by Addition of KI

In this study, FeSe superconducting samples with the addition of KI were synthesized using the powder in sealed tube (PIST) method. Fe powder (Merck, with a purity of 99.5%), Se powder (Merck,

with a purity of 99.5%) was used as a precursor in the stoichiometric ratio FeSe = 1: 1 which was prepared by conventional solid reactions. The precursors are homogenized using agate mortar for 3 hours. Then the precursor is inserted into a stainless steel 316 tube where the left and right ends are sealed by pressing using a hydraulic press that aims to prevent the oxidation of iron during the heating process. Finally, the sample in a closed SS316 tube is sintered in a tube furnace at 845 ° C with a heating rate of 7 ° C/minute held for 3 hours. The sample is then cooled by expelling it in the open air at room temperature. Subsequently the precursor sample in powder form was mixed with the addition of KI of 1% and 5% by weight (Merck, with 99.99% purity). This powder with a mixture of KI is synthesized like the previous precursors process, put into SS316 and pressed again using a press machine. Subsequently all samples were sintered for 3 hours in a furnace tube with a heating rate of 7 °C/min and held for 3 hours.

2.2 Material Characterization

Phase identification and crystal structure were characterized using X-Ray Difraction (XRD). PANalytical diffractometer with a Cu Kα radiation source (λ = 1.5046 Å) performed at an angle of 2θ = 10 - 70°. Morphological observations of the samples were carried out using JEOL JSM-6390LA Scanning Electron Microscopy (SEM). Electrical properties (superconductivity) are measured using the Teslatron cryogenic magnetic system using the four-point probe (FPP) method at temperatures of 5 - 300 K.

III. Results and Discussion

Figure 1 shows the X-ray diffraction pattern of FeSe samples with the addition of 1% by weight KI (YP31) and 5% by weight (YP33). The phases produced by the FeSe superconducting material are multi-phase, identified by the β-FeSe space group (P4/nmm) phase as the main phase and the δ-FeSe space group (P63/mmc) phase as the impurity phase. The peak intensity produced on the maximum FeSe material at an angle of 2θ = 28.4 ° is the highest peak of the other peaks. The results of the XRD pattern showed a significant difference in the FeSe material without doping and after the addition of KI. The addition of KI of 5wt % Identified new phases, namely the cubic phase Fe3O4 space group (F d-3 m (227) and the Fe0929O phase space group (F m-3 m (225) which is considered to be the impurity phase. The presence of the cubic phase Fe3O4 impurity phase and the Fe0929O phase may be caused by the reaction between SS316 material and Fe precursors during the heating process The emergence of the impurity phase in the addition of KI causes a decrease in the main phase to eliminate the superconductivity of the material in the sample.The results of this diffraction pattern will be adjusted to the results of resistivity testing using a tool Cryogenic Magnet.

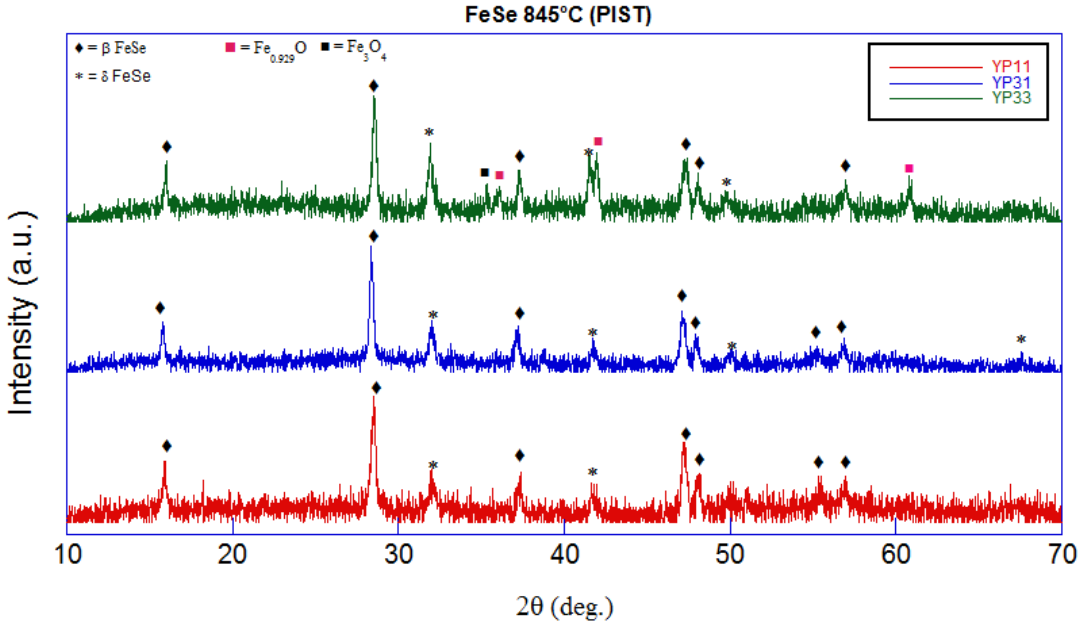

Figure 1. XRD Patterns of the KI Doped FeSe Sintered at 845°C for 3 h

The plot of temperature vs resitance of the of FeSe with addition 0, 1 and 5 wt % KI samples are shown in Figure 2.

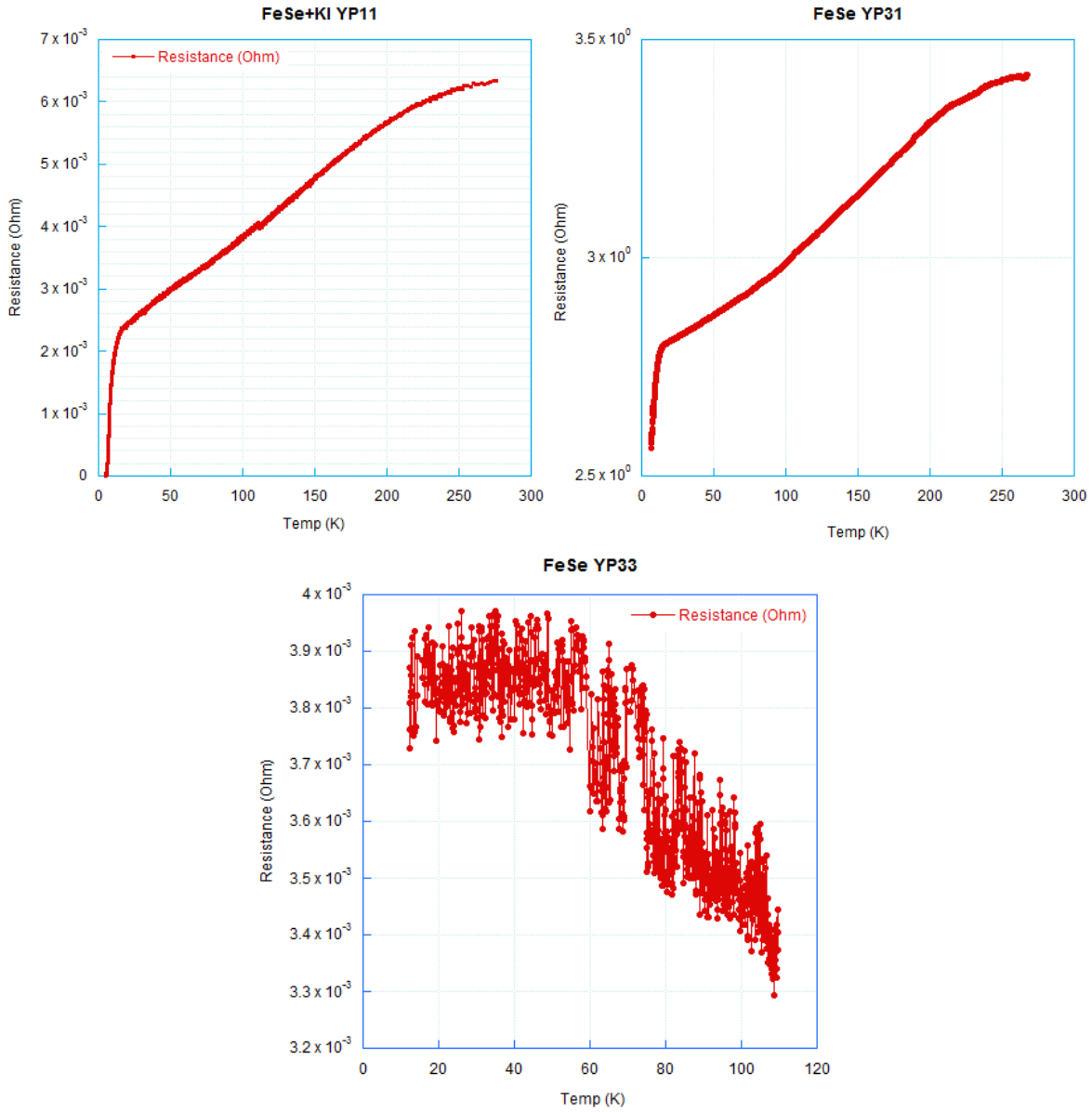

Figure 2. Plot of Temperature vs Resistivity of the Mn Doped FeSe Samples

Figure 2 shows that the qualitative analysis of the addition of KI with 1 wt % and 5 wt % composition variations shows that not all samples produce superconducting curves and have a critical temperature (Tc). The results that show superconducting phenomena only occur at 1% KI addition (YP31), YP33 sample shows non-superconducting FeSe material. The addition of KI by 1% indicates a Tc value of 13.4 K, with a fairly large resistivity (ρ) value of 78.2 and a fairly small RRR value of 1.21. The results obtained are also synchronous with the XRD Analysis results, where the volume fraction in the main phase of FeSe superconductor with 1% KI addition shows the greatest value of the other KI addition samples, which means the β-FeSe phase tends to be more dominant in this sample.

The morphology of FeSe samples was reported by adding 1 wt % KI and 5 wt %.

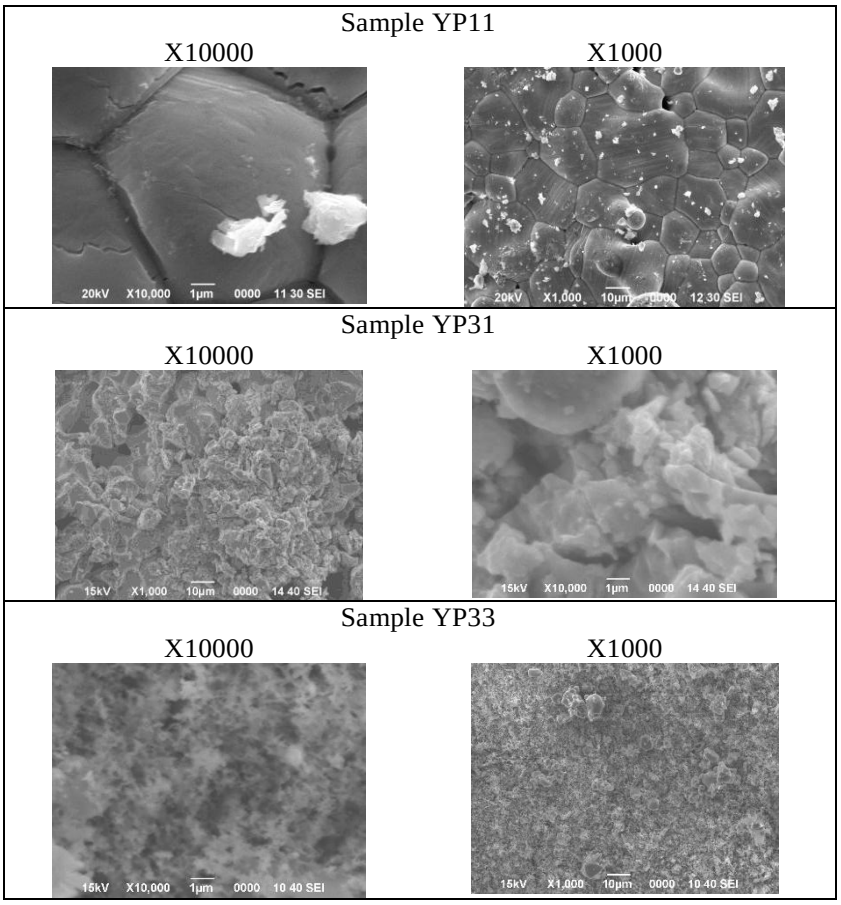

Figure 3. Surface Morphology of The Fracture Samples

Figure 3 above shows the SEM test results from samples YP11 to YP33 FeSe material with the addition of KI of 1% and 5% by weight sintered at 845^0 C for 3 hours using the PIST method. Addition of KI of 1% and 5% by weight weakens the main phase of the FeSe superconductor and the superconductivity of the FeSe sample itself. This can be seen from the results of the morphological distribution of the pattern produced by the addition of KI 5wt % Appears to damage the tetragonal phase in the crystalline shape of the sample and the morphology of the pattern appears unclear and irregular. The morphological results are also synchronous with the XRD results which show that the addition of KI gives rise to new phases, namely Fe304 and Fe0929O as impurity phase, which means that the impurity phase tends to be more dominant than the main phase of FeSe superconductor.

Table 1. Resume of Refined XRD Patterns Data, Electrical Resistivity and Critical Temperature of Samples

	0 wt. % KI (YP11)	1 wt.% KI (YP31)	5wt.% KI (YP33)
FWHM of Tetragonal (degree)	0.2833	0.1889	0.2667
FWHM of Hexagonal (degree)	0.2222	0.2111	0.2166
Fv Tetragonal (%)	85.66	79.28%	60.36%
Fv impurity (%)	14.34	20.72%	39.64
x (Mn content, at. %)	2.25	3.75	3.75
R_{250} K (Ω)	0.0062	3.4027	-
R_{20} (Ω)	0.0024	2.8100	-
RRR	2.5833	1.21	-
Tc (K)	13.33	12.27	-

According to qualitative and quantitative analysis, there were oxidation phases in the sintered addition of 1 wt % And 5 wt % KI samples. This shows that the FeSe sample with the addition of KI oxidation process occurred during preparation and the addition of KI to the FeSe material impairs the superconductivity of the FeSe material itself. Furthermore, these results can be used as a reference that FeSe-based superconductors about the effect of adding KI to superconductivity.

IV. Conclusions

In this study, the synthesis of FeSe with the addition of KI has been successfully carried out using the powder in sealed tube (PIST) method. We observed the effect of KI addition on the phases formation, morphology and superconductivity of FeSe superconducting material. The addition of 1 wt % And 5 wt % KI causes the emergence of new phases that become impurities in the FeSe superconducting phase which causes a decrease in tetragonal phase content. Addition of 1 wt % And 5 wt % KI showed a decrease in tetragonal phase content, irregular grain density and decreased critical temperature in FeSe samples with the addition of KI.

Acknowledgements

This work was supported by research center for Materials and Metallurgy, Indonesian Institute of Sciences (LIPI) and and Universitas Sumatera Utara, Indonesia.

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Yeni Sartika Manurung,et.al. "Effect of KI Addition on FeSe Superconductors by Powder in-Sealed Tube Method." *IOSR Journal of Applied Physics (IOSR-JAP)*, 12(1), 2020, pp. 56-60.