International Journal of Sciences: Basic and Applied Research (IJSBAR)

ISSN 2307-4531
(Print & Online)

http://gssrr.org/index.php?journal=JournalOfBasicAndApplied

Preparation and Superconductivity of $Fe_{1-x}SeSn_x$ Superconductor

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Abstract

Sn doped $Fe_{1-x}Se$ has been prepared by solid-state reaction through vacuum sintering process at 845^{o}C for 3 hours and cooled in the closed air tube furnace. Precursor powder (FeSe) was mixed with Sn powder (with purity of 98.5%) obeying a stoichiometric ratio of Fe:Se:Sn = (1-x) :1 : (x), where x= 1-wt% Sn, 5-wt% Sn and 10-wt% Sn, respectively. The precursor was then inserted into dies to form pellet. The pellet was then inserted into a crucible and put on quartz tube that aimed to prevent the oxidation of iron during the heating process. Finally, the sample sintered in the furnace tube at 845^{o}C with heating rate of 5^{o}C/minute held for 3 hours.The result obtain show that Sn did not actually enter the crystal lattice β-FeSe, existing as an inclusion Fe instead on 2Θ=44.38^{o}. Structurally, the addition Sn doped FeSe superconductors material can increase the volume fraction of the main tetragonal (β-FeSe) from 30.13% to 72.02% at addition of 1 wt%Sn; up to 54.59% at addition of 5 wt%Sn and 51.17% at addition of 10 wt%Sn. The morphology observation also displayed FeSe with the addition of Sn dopant has a large crystallite size with dissipation and the presence of porosity. And result temperature critical at 13.33 K to 15.87 K. All the factors mentioned above are contributed to the high Tc of Sn doped $Fe_{1-x}Se$ sample. The present work suggests that addition of 1 wt% Sn might be a promising element in improvement the superconductivity of $Fe_{1-x}Se$.

Keywords: cryogenics; FeSe; sintering; superconductors.

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1. Introduction

Superconductor material can be defined as a material has no resistance (R=0) and shows a perfect diamagnetism (Meissner effect). This phenomena, called superconductivity occurred under a certain temperature value (zero critical temperature). In the superconducting state, superconductor materials conduct electric current without any losing of power [1]. Since the discovered in 1991 by Onnes using liquid helium and cooled to temperature 4.2 K with resistivity of $3\times10^{-6}\Omega$ at temperature 0.01 K [2], a great amount research have been done around the world. In addition, the discovery of high Tc superconductors by Bednorz and Muller stimulated a large number of works and research worldwide [3] The Fe-based superconductor discovered in 2008 with a value of Tc 26 K in $LaFeAs(O_{1-x}F_x)$ in the same year Hsu and his colleagues conducted a Fe-based superconductor research with the addition of Se which value of Tc 8 K with simple structure crystal in the from tetragonal phase, β-FeSe. Which is currently the best choice for study of the structure and the conductivity of the FeSe superconductors [4,5]. The addition of Sn doping on FeSe superconductors not just to achieve conductivity, but also a method to explore the mechanism of conductivity [6]. However, research FeSe doped with metals such as (Ti, V, Cr, Mn, Co, Ni, and Cu) [7]. The addition of Sn on $Fe_{1-x}Se$ is rarely studied. In the $FeSe_{1-x}$ with addition of Sn result growth of β-FeSe grain was obtained that could accelerate the formation of FeSe. In this paper, we report analysis phase formation, morphological structure observation and superconductivity measurement. Systematically studied the doping effect of Sn on the phase formation and superconductivity in $Fe_{1-x}Se$, with the expectation to optimize the improve superconductivity.

2. Materials and Methods

Preparation of FeSe superconductor was started by mixing Fe powder and Se powder. The powder of Fe (99.99%) and Se powder (99%) were mixed in an agate mortar by stoichiometric ratio 1:1. Precursor powder (FeSe) was mixed with Sn powder (with purity of 98.5%) obeying a stoichiometric ratio of Fe:Se:Sn = (1-x) :1 : (x), where x= 1-wt% Sn, 5-wt% Sn and 10-wt% Sn, respectively.The precursor was homogenized using agate mortar for 3 hours. The precursor was then inserted into dies to form pellet. The pellet was then inserted into a crucible and put on quartz tube that aimed to prevent the oxidation of iron during the heating process. Finally, the sample sintered in the furnace tube at 845^oC with heating rate of 5^oC/minute held for 3 hours. The sample cooled with closed close air at room temperature. The same procedure was done to prepare sample with Sn addition. Phases identification was carried out using *X-Ray Diffraction* (XRD) with Cu Kα (λ = 1.5418Å) at the angle of $2\theta = 10^o – 60^o$. Critical temperature, T_c was observed by plotting the resistance graph versus time in a *Teslastron Cryogenic Magnetic.* The Four Point Probe (FPP) method was applied with silver paste in the range of 5K- 300K (liquid Helium).

3. Results and Discussions

Figure 1 shows the X-Ray Diffraction pattern of FeSe samples with the addition of Sn 0 wt% (C1), 1 wt% (C2), 5 wt% (C3) and 10 wt% (C4). The phases produced by the FeSe superconducting material are multi-phase, identified by β-FeSe space group (P4/nmm) phase as the main phase and δ-FeSe space group (P63/mmc) as the impurity phase. Figure 1. Shows phases of Fe increased at angle 2Θ 44.38^o. Comparative analysis of the peak

position of β-FeSe for the undoped sample and the doped Sn make slightly difference, which gives us a reasonable bases believe that the element Sn does not enter the crystal lattice of β-FeSe in $Fe_{1-x}Se$ sample shows parameter lattice a=b=3.7723Å, c=5.5225Å. And deviations are less than 0.001Å in the doped samples, which is within the range allow-able error.

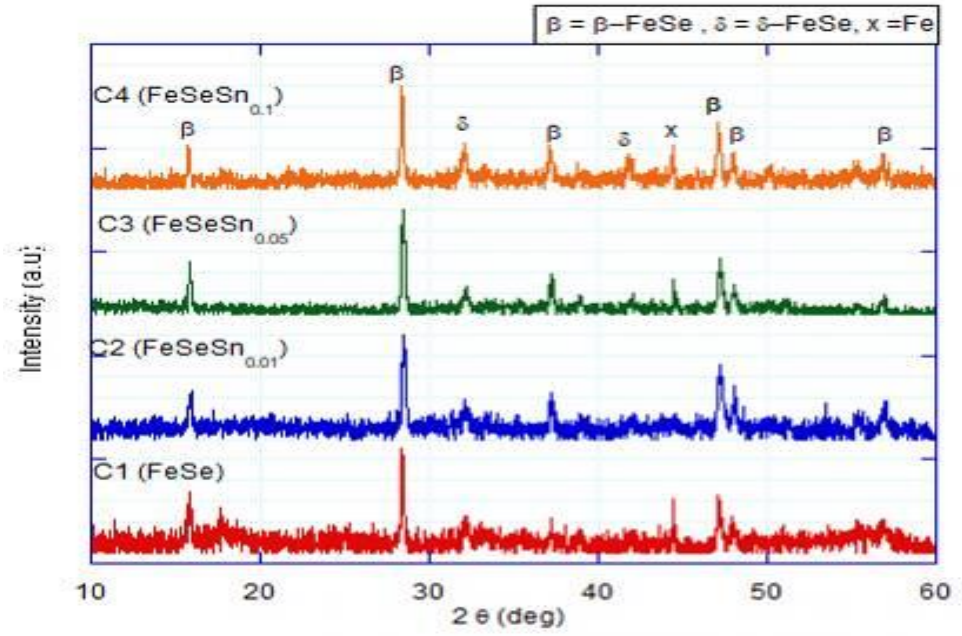

Figure 1: XRD Patterns of the Sn on FeSe Superconductors.

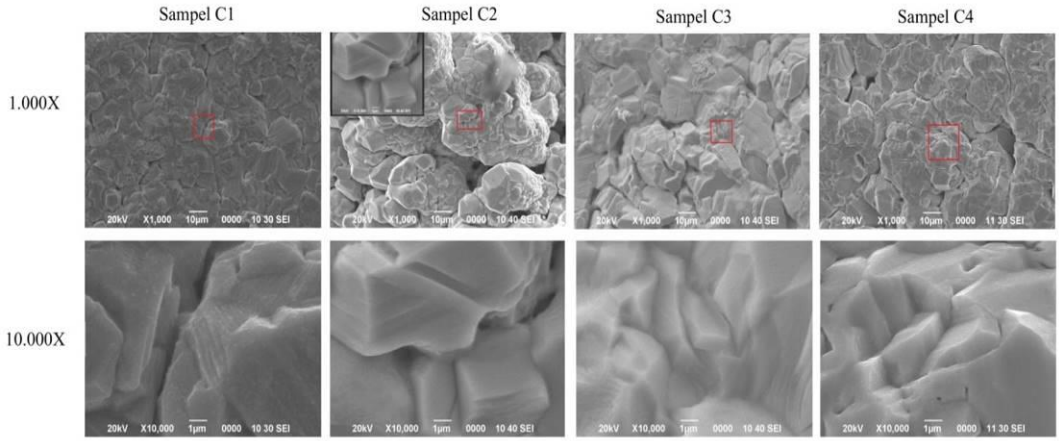

Figure 2: Surface Morphology of The Fracture Samples

The conclusion is the addition of Sn doped on FeSe sample does not make change structural are significant and with calculate volume fraction of phase β-FeSe as the main phase and δ-FeSe as the impurity phase obtained the addition of Sn 1 wt% at increasing that will effect the superconducting properties of FeSe materials. Which is also shown in Table 1. The SEM images of the sample shown in Figure 2. The doped samples exhibit enlarged grains, which seems to connect tightly with good density and least porosity. This possible with the addition of Sn can accelarate the growth of grains by improving atomic difussion to phase β-FeSe. The sample doped with 1

wt%Sn showed an increase of crystallinity and values FWHM obtained XRD result of the phase impurity smaller so that the crystallinity in the sample improved (Table1).

Table 1: Resume of Refined XRD Patterns Data, Electrical Resistivity and Crictical Temperature of Sample

	0 wt% Sn (C1)	1 wt% Sn (C2)	5 wt% Sn (C3)	10 wt% Sn (C4)
F. Volume β-FeSe (%)	30.13	72.02	54.59	51.17
F. Volume δ-FeSe (%)	69.86	27.97	45.40	48.22
FWHM β-FeSe (deg)	0.18	0.29	0.21	0.20
FWHM δ-FeSe (deg)	0.23	0.14	0.38	0.44
$R_{250}K$ (Ω)	0.79530	0.63422	-	-
$R_{20}K$ (Ω)	0.25637	0.19194	-	-
RRR	3.1020	3.3041	-	-
Tc (K)	13.33	15.83	-	-

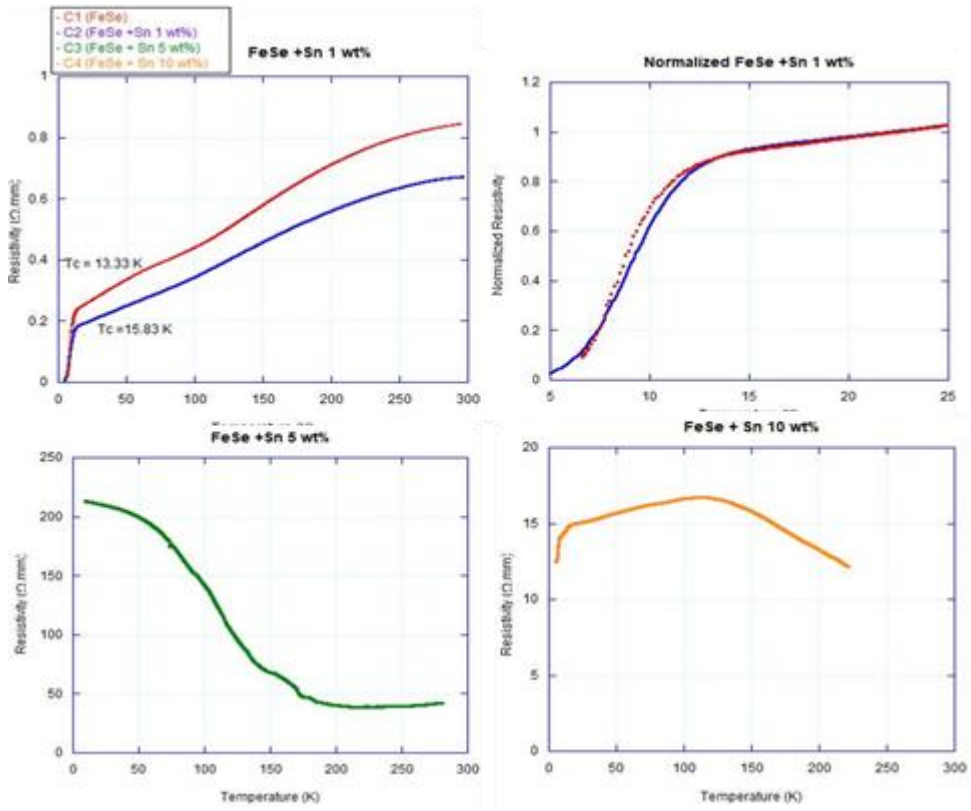

Figure 3: Plot Temperature vs Resistivitty of the Sn doped FeSe Samples

The onset transition temperature, *T*c onset is defined as the temperature where there is a sudden drop in resistance and can be obtained from the crossing point of the linear fit of the highest slope and the metallic high temperature part of the ρ(T) curve for each sample. The zero-resistance temperature, *T*c zero is the temperature where the resistance drops to zero and can be estimated from the extrapolation of the linear part of the resistivity to the temperature axis [8]. Figure 3: Shows the result of cryogenic magnet does not all samples with the

addition of Sn produce superconducting curve and have a critical temperature (Tc). The result that show superconducting phenomena only occur at 0 wt% and 1 wt% indicates a Tc value of 13.33 K and 15.83 K, with value RRR of 3.1020 and 3.3041. This is consistent with volume fraction β-FeSe which the gratest value at the addition of 1 wt% Sn and value of FWHM is smaller, thus the size crystallinity as large as in the sample [9]. The value higher RRR has smaller residual resistance, so it can increase the purity of conductivity for sample. In this study, Sn did not enter the crystal lattice of β-FeSe, which means no significant deformation (Figure 1). The addition of Sn 1 wt% can accelerate the transformation from δ-FeSe to the β-FeSe phase (Table 1). What is more, the value FWHM the enlarged β-FeSe grains more not good in crystallinity also effect the superconducting properties (Figure 2). Due to the reasons mentioned above, the addition of Sn 1 wt% can increase superconductivity of $Fe_{1-x}Se$ but excessive amounts of the additions of Sn does not suppress the superconductivity of $Fe_{1-x}Se$.

4. Conclusion

$Fe_{1-x}Se$ samples with the addition of Sn were prepared by solid-state reaction method with a *vacuum sintering* process. It was found that Sn did not actually enter the crystal lattice of β-FeSe, phase β-FeSe as the main increase of phase would affect the superconductivity of $Fe_{1-x}Se$. The addition of 1 wt%Sn could improve superconducting properties of materials $Fe_{1-x}Se$ with value of Tc obtained 15.83 K while Tc value of FeSe without undoped Sn 13.33 K. FeSe doped Sn 1 wt% have morphology large crystallinity size with the increasingly narrow value FWHM and has increase grain density characterized by the least porosity of samples. And the percentage volume fraction of β-FeSeis the largest of all samples at 72.02%, which can influence superconductivity of $Fe_{1-x}Se$.

Acknowledgements

The authors are grateful to the Metallurgy and Materials Research Center (P2MM), The Indonesian Institute of Science (LIPI) 2019 and the University of Sumatera Utara, Indonesia.

References

[1]. Rusdi. Ariawan. “Superconductor.” University of Udayana, Bali, 2010.

[2]. Kamerlingh. Onnes, H. “On the sudden change in the rate at which the resistance of mercury disappears.” Communications from the Physical Laboratory of the University of Leiden, 124c, 1911.

[3]. Putri Suci Mustika Lubis, Syahrul Humaidi, Agung Imaduddin, Perdamean S, The effect of heat treatment BPSCCO-2212 and LSCO superconductor, 3rd NICTE IOP Conf. Series: Materials Science and Engineering **725** 012043, 2020.

[4]. Y. Kamihara, T. Watanabe, M. Hirano, and H. Hosono, “Iron-Based Layered Superconductor La [O 1 - x F x] FeAs (x) 0 . 05 - 0 . 12) with T c) 26 K,” J. Alloys Compd., pp. 3296–3297, 2008.

[5]. F. Hsu et al., “Superconductivity in the PbO-type structure ⎵ -FeSe,” PNAS, vol. 105, pp. 14262–14264, 2008.

[6]. N. Chen et al., “Influence of Sn doping on the phase formation and superconductivity of FeSe0.93,” J.

Alloys Compd., vol. 588, pp. 418–421, 2014.

[7]. S. Margadonna, Y.Takabayashi, M.T. McDonald, et al, Chem, Commun, 5607–5609, 2008.

[8]. Syahrul Humaidi, Eddy Marlianto, S. Marhaposan, R. Abd-Shukor, Superconducting Properties of Te-Substituted $(Tl_{2-x}Te_x)Ba_2CaCu_2O_{8-\delta}$, Solid State Phenomena, Vol. 290, pp 239-244

[9]. Y.P.Dewi, "Influence of Composition on the Structure and Superconductivity on Fe(Se,Te) in preparatin by the powder metalurgy method." Departement Science and Technology, University of Airlangga, Surabaya, 2016.