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*RESEARCH ARTICLE*

# SYNTHESIS AND CHARACTERIZATION OF FESE SUPERCONDUCTORS BY THE ADDITION OF MG DOPING USING THE VACUUM METHOD

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## *Abstract*

In this work, the powder preparation Fe0.99Se-Mg 1wt%. Iron, selenium powder, and magnesium powder are used as raw materials. The raw materials is milled using a mortar agate for 3 hours after weighed in the ratio of the atom Mg:Fe;Se = 0.01:0.99:1. To be characterized by the formation of Fe0.99Se-Mg 1wt%, the milled powder is then compressed into a pellet sample and sintered at 745˚C and 845˚C for 3 hours. All sample are sintered on furnance and cooled with room temperature. XRD are used to analyse forming phase. Based on the calculation of diffraction pattern, sample are sintered at 745˚C has a higher mass fraction of tetragonal phase than a sample that is sintered at 845˚C. the lattice parameter at 745˚C sample are a = 3.7790 Å and c = 5.5111 Å. According to the resistivity measurement results, it appears that the sample are sintered at 745˚C have a critical temperature ~ 13.4K.



## Introduction:-

Superconductor were first discovered by dutch physicist Heike Kamerlingh Onnes in 1911. Onnes succeeded in liquefying helium (He) by cooling it at 4 K or 269˚C. Then onnes began to study the electrical characteristics of metal at very cold temperature. Onnes conduct an electrical current on pure mercury (Hg) wire and lowering that temperature, Onnes measures the resistance. At 4.2 K, the resistance suddenly disappears but the current still flows through the mercury wire continuously . [1].

Magnetic materials such as iron cannot be used as superconductor material because it has a direct current with a magnetic field, but with the invention made by Kamihara et al in 2008, iron can be used as a superconducting material. One iron-based superconductor (Fe) is a FeSe superconductor. Most FeSe superconductor have critical temperature onset (Tconcet) of 8K . [ 2 ].

Since the discovery of Fe-based superconductor materials in 2008, research on superconductors has increased over time, because Fe which has ferromagnetic characteristic should not have superconductivity (generally superconductor diagmanetic materials). Because of the appear of Fe-based superconductors, research on superconductors changed their direction and began to explore this type of superconductor. The main barrier to the manufacture of FeSe superconductors is the emergence of δ-FeSe phases (tetragonal structures with P4/nmm crystal symmetry), which do not have superconductor characters, during the synthesis of β-FeSe superconductors. [ 3 ].

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The FeSe superconductor has several parameters that must be considered to get the type of β-FeSe tetragonal crystal structure. Superconductivity control is generally controlled by two main parameters consisting of intrinsic properties such as Tc and microstructure (grain boundaries and impurities), extrinsic character such as critical current density (Jc). The stoichiometric composition is the most influential parameter in superconducting structures. [4]

According to Feng Lan, Mg doping in FeSe material below 150K has a lower resistivity compared to FeSe material without Mg doping. This can occur because Mg doping is associated with the effect of electron scattering (interactions) under normal conditions. [5]

According to Wenbin Qiu Mg doping can induce variations in electron carrier concentration and include electron doping, which significantly influences the performance of the final superconductor in FeSe- film. Optimal Mg doping has a positive effect on the performance of FeSe superconductors, but degradation or even collapse will occur in superconductivity when excess Mg content is incorporated into the FeSe material. [6]

In this paper, phase formation analysis, morphological structure observations and superconductivity measurements at Fe0.99Se-Mg 1 wt% .

## Research Methods:-

In this work , the FeSe prepared Iron (99%), Selenium (99%) is used as a precursor and Magnesium powder (98%) . The process of formation of Fe0.99Se-Mg 1wt% using methods in solids (dyes) by sintering in vacuum. In First step, the precursor weighed in accordance with the ratio of nominal precursors, Mg:Fe:Se = 0.01:0.99:1. The precursors are homogenized using mortar agate for 3 hours then the precursor is inserted into the dies so that the sample is in the form of a pellet which will then be placed in a boat cup and put on a quartz tube which aims to prevent oxidation during the heating process. Then the sample will be in sintering in a tube furnace at 745˚C and 845˚C with a heating rate of 7°C / min detained for 3 hours. The sample is then cooled with closed air at room temperature.

Analysis of phase identification and crystal structure was characterized using X-Ray Diffraction (XRD). PANalytical Diffractometer with a Cu Kα radiation source (λ = 1.5418Å) performed at an angle of $2\theta = 10^{o} – 60^{o}$. Morphological observations were carried out using JEOL JSM-6390A Scanning Microscopy Electron (SEM). Superconductivity is measured using Teslastron Magnetic Crayogenic system using Four Point Probe (FPP) method at temperatures of 5-300 K.

## Results and Discussion:-

The XRD pattern on the sample Fe0.99Se-Mg 1wt% 745˚C and 845˚C are shown in picture 1. Establishment phase β-FeSe not occur with perfectly at temperatures low. According to Figure 1, an increase in the peak phase of β-FeSe occurs when the material is sintered at 745˚C. It is directly inverse to the peak phase of δ-FeSe , where the intensity of the peak phase of δ-FeSe decreases with increasing temperature sintering.

Xiaoting Li, et al reported that the temperature of the heating that more high- boost phase β-FeSe [ 7 ]. The results have showed that the phase δ- fese will turn into a phase of β-FeSe at a temperature sintering high followed by cooling the air that quickly.

Figure 1 shows the phases of Fe increased at an angle 2θ = 44:40˚ where the presence of a temperature rise Fe phase becomes dead. X-ray diffraction pattern calculations using three phases are formed, namely β-FeSe (tetragonal crystal structure with p4/nmm space group), δ-FeSe (hexagonal crystal structure with p63/mmc space group) and iron (cubic crystal structure with space group im-3m). Crystal lattice parameters were calculated from sample Fe 0.99 Se-1wt% Mg at a temperature of 745˚C and 845°C respectively, are a = 3, 7790 Å, c = 5, 5111 Å and a = 3.6100 Å, c = 5.8700 Å .

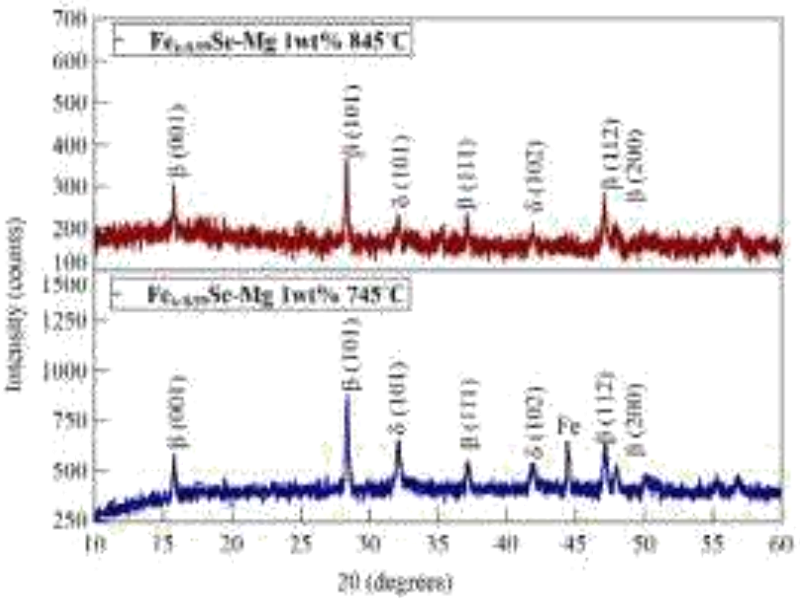


**Figure 1:-** XRD pattern of Fe0.99Se-Mg 1wt%.

The conclusion obtained is by increasing the temperature of the sample undergoing phase changes in Fe where there is no temperature at 845˚C and by calculating the volume fraction of the β-FeSe phase as the main phase and δ-FeSe as the impurity phase obtained at 845˚C decreases which will affect the superconducting properties of the FeSe material will be shown in Table 1. SEM image of the sample shown in Figure

The samples were processed shows the grain size enlarged, yes ng appears to bind to one another with a density yan g good and the least porosity. This is possible by the addition of Mg can accelerate the growth of grains by increasing the diffusion of atoms to form β-FeSe phases. Samples at a temperature of 745˚C shows the growing size of the crystallinity of this case according to the FWHM values obtained in XRD testing phase of impurity getting smaller so that the crystallinity of the sample is getting better (see Table 1) .

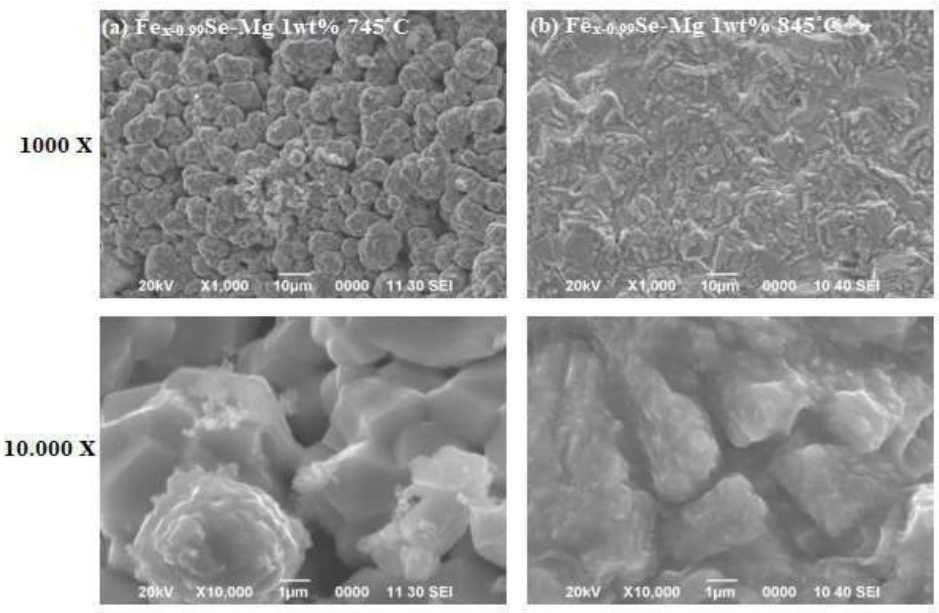


**Figure 2:-** SEM image for surface morphology of a sample fracture.

**Table 1:-** Resume of XRD Pattern Data, Resistivity and Critical Temperature in Samples.

| | **1wt% 745˚C** | **1wt% 845˚C** |
|---|---|---|
| | | |
| **F. Volume β-FeSe (%)** | 76 | 44 |
| **F. Volume δ-FeSe (%)** | 24 | 20 |
| **FWHM β-FeSe (deg)** | 0.25 | 0.26 |
| **FWHM δ-FeSe (deg)** | 0.64 | 0.40 |
| **R250K (Ω)** | 0.5335 | - |
| **R20K (Ω)** | 0.2416 | - |
| **RRR** | 2.2084 | - |
| **Tc (K)** | 13.8 | - |

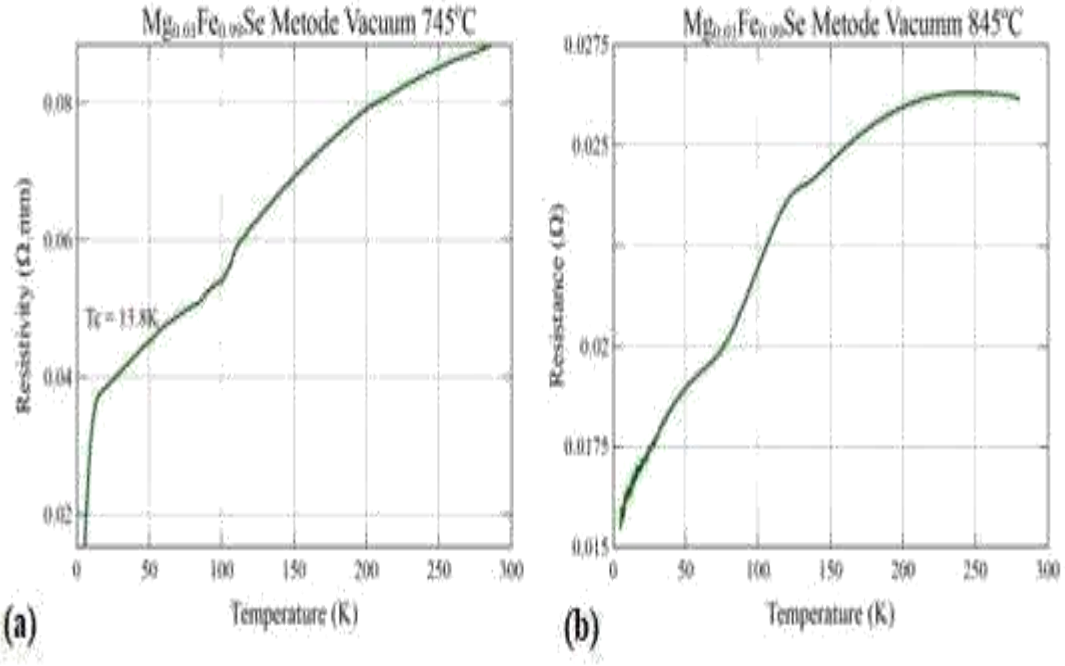


**Figure 3:-** Graph of Relationship of Resistivity and Temperature Sample Fe0.99Se-Mg 1wt%.

Figure 3 is the result of crayogenic magnet testing showing that not all samples with the addition of Mg produce superconducting curves and have a critical temperature (Tc). The results that show the superconducting phenomenon occur at 745˚C, namely by having a Tc of 13.8 K, with a RRR value of 2.2084. This is consistent with the volume fraction β-FeSe which shows the greatest value. And the smaller the FWHM value, the larger the crystallinity size in the sample [8]. The higher RRR value has smaller residual resistance, so that the material has a high conductivity purity.

In this study, Mg did not enter the β-FeSe crystal lattice which meant there was no significant deformation (see Fig.1). Temperature 745˚C can accelerate the transformation of δ-fese to β-FeSe phase (see T abel 1). Moreover, the value of FWHM β -FeSe enlarged, the more is not good in crystallinity also affect the superconducting properties (see Figure 3). For the reasons mentioned above, an increase in temperature can increase the superconductivity of Fe1-xSe, but in excessive amounts an increase in temperature cannot suppress the superconductivity of Fe1-xSe.

## Conclusions:-

In this work , phase formation and electrical resistivity of Fe0.99Se-Mg 1wt% prepared using the solid reaction method with a vacuum sintering process have been reported. β-FeSe as the main phase is formed at a sintering temperature of 745°C. Based on diffraction patterns , the tetragonal FeSe phase increases due to an increase in sintering temperature. The method that is used in this experiment can occur oxidation if it's not done with either . The lattice parameters in the 745˚C sample are a = 3.7790 Å and c = 5.5111 Å . According to the results of resistivity measurements, it appears that the sample sintered at 745°C has a critical temperature of ~ 13.4K

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