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Impedance and dielectric analysis of polycrystalline undoped and Fe doped SnS_2

Areej S. Alqarni*, B.O. Alsobhi, A.A. Elabbar, O.A. Yassin[1]

Department of Physics, Faculty of Science, Taibah University, B.O. Box 30002, Prince Naif road, Almadinah Almunawarah, Saudi Arabia

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ABSTRACT

Polycrystalline $Sn_{1-x}Fe_xS_2$ samples with (x=0, 0.125, 0.250 and 0.375) have been prepared by the molten salt solid state reaction method. The X-ray diffraction (XRD) shows that all the samples crystallize in the hexagonal structure, with P-3m1 space group in preferred orientation of (011). The electrical properties have been studied by complex impedance spectroscopy over the frequency range (20 Hz up to 1 MHz) at room temperature. The Nyquist plot for all samples have been fitted using ZMAN software. The impedance analysis showed that all samples exhibit both bulk and grain boundary contributions and it was found that by increasing the iron content, the resistance increases, but, the dielectric constant and dielectric loss tangent decrease which leads to decrease in conduction. The absorption coefficient (α) has been calculated from the complex dielectric constant. Interestingly, there was a significant correlation between the electromagnetic wave absorption and the reduction in the peak intensity of the XRD patterns indicating that when the iron content increases the sample seems to be a good absorber of electromagnetic waves.

1. Introduction

Considerable attention has been paid to the layered semiconductor compound tin disulfide which has many remarkable applications [1–8]. For example, it has been used as high-power lithium ion batteries [1], optoelectronic devices [2], gas sensors and chemical sensors [3,4], photoluminescence material [5], photoconductive [6], pigment [7] as well as anode materials [8]. Moreover, its properties can be tuned by doping. For instance, its photocatalytic activity can be enhanced when zinc or cerium elements are inserted [8,9].

Recently, it has been reported that doping SnS_2 with vanadium (V) or tungsten (W) elements makes it a promising parent material candidate for intermediate band solar cells IBSC applications [10]. Fe doped SnS_2 was also reported as a good candidate for intermediate band solar cells because iron doping enhanced its optical absorption [11]. On the other hand, many published works have shown that SnS_2 can easily be prepared and widely investigated. Useful examples of SnS_2 synthesis methods are hydrothermal [12], microwave-assisted [13], spray pyrolysis [10] and molten salt solid state reaction [14].

Studies on the electrical properties of SnS_2 are very rare. In spite of that, there is little published data on its electrical properties. In 1996 the dielectric properties of SnS_2, single crystals have for the first time been studied [15] and there is no much work have been reported on its dielectric and impedance properties. Electrochemical impedance spectroscopy (EIS) measures the dielectric properties of a material as a function of frequency. It can be used to elucidate the relaxation mechanism(s) of the transport properties of the materials under study. The response of the material to the applied frequency is usually represented in terms of the most commonly used Bode and Nyquist plots.

For a Debye single relaxation process the Nyquist plot is a half circle with its center falls on the x-axis. However, when there is more than one relaxation process the shape in the plot is composed of many joined circles or not well defined half-circle (quasi half circle). The last situation takes place when the relaxation frequencies are close to each other which can be separated by fitting the data to a certain equivalent circuit model.

In this work the purpose is to prepare $Sn_{1-x}Fe_xS_2$ (x=0, 0.125, 0.250 and 0.375) powder samples and study their impedance and dielectric properties to inspect the effect of the Fe-doping on relaxation process and the absorption of the electromagnetic radiation.

* Corresponding author.
E-mail addresses: aqarni@taibahu.edu.sa (A.S. Alqarni), sobhibo@yahoo.com (B.O. Alsobhi), elabbar60@yahoo.com (A.A. Elabbar), oyassin63@gmail.com, oyassin@taibahu.edu.sa (O.A. Yassin).
[1] Permanent address: Department of Physics, Faculty of Science, Al-Neelain University, Khartoum 11121, Sudan.

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2. Experimental details

2.1. Synthesis of samples

The polycrystalline samples of $Sn_{1-x}Fe_xS_2$ (x=0, 0.125, 0.250 and 0.375) were prepared by the molten salt solid state reaction method. All reagents were purchased from Sigma-Aldrich. First, thiourea (N_2H_4CS), tin(II) chloride dihydrate $SnCl_2{\cdot}2H_2O$ and iron(II) chloride hexahydrate $FeCl_2{\cdot}6H_2O$ were mixed and ground by an agate mortar. Next, the powders were placed inside a tubular furnace at 280 °C using alumina boats. After heating for 3 h in a flowing N_2 gas, they are left to cool naturally. After that, powders were grounded again in an agate mortar to become soft and then washed so many times using distilled water to remove any possible impurity such as $(NH_4)_2SnCl_6$ which is easily soluble in water. Lastly, they were dried at 90 °C to obtain the samples.

2.2. Characterization

The structural properties have been studied using a Shimadzu 6100 X-ray diffractometer. The were collected in the 2θ range from 10° to 80° with a step size of 0.02. To study the impedance and dielectric properties of the samples, impedance spectroscopy (GW INSTEK LCR-8105 G) was used at room temperature in the frequency range of 20 Hz to 1MHz with the ac-amplitude of 100 mV for a series circuit (X-R). Before the impedance measurement, the powder is pressed into disk pellets of 13 mm diameter and 2 mm thickness using a hydraulic press. It was densified by annealing at 280 °C for 2 h in a flowing of N_2 gas. Each pellet sample is loaded between two circular copper electrodes with similar dimensions. The impedance data were acquired using a computer with automated data collection software.

3. Results and discussion

3.1. Structural properties

The single phase formation has been confirmed using X-ray diffraction (XRD). Fig. 1 shows the XRD patterns of $Sn_{1-x}Fe_xS_2$ (x=0, 0.125, 0.250 and 0.375). In this figure, the points and the solid line represent the experimental data and the profile fitting created by the FullProf software for a model with a hexagonal lattice and space group $P\bar{3}m1$, respectively. All Bragg's reflections have been indexed and no additional peaks from any possible impurities, like oxides, cubic FeS_2 or $(NH_4)_2SnCl_6$ have been detected. Beside the FullProf result, some matching works [3,16] of the observed peaks and the standard PDF card-01–089-2028 also confirmed that the doping with Fe ions does not change the hexagonal crystal structure of tin disulfide. This result has been previously reported by us [11] in a different preferred orientation of (001) and that may be due to gradually heating during the preparation steps. It has been observed that the intensity of the peaks become weaker when the dopant concentration increase. Fig. 2 shows the variation of the intensity of the detected beam with dopant concentration x. A possible explanation for this is that when the iron content increases the sample seems to absorb a larger amount of the incident X-ray.

3.2. Impedance and dielectric analysis

The electrical properties of semiconductors and insulators may be represented in terms of the complex impedance (Z*), complex dielectric constant (ε*), and dielectric loss (tan δ). These quantities are related to each other [17] as follows

$$Z^* = R + iX = R_s + i\frac{1}{\omega C_s} = R_s - \frac{1}{i\omega C_s} = Z' - iZ'' \quad (1)$$

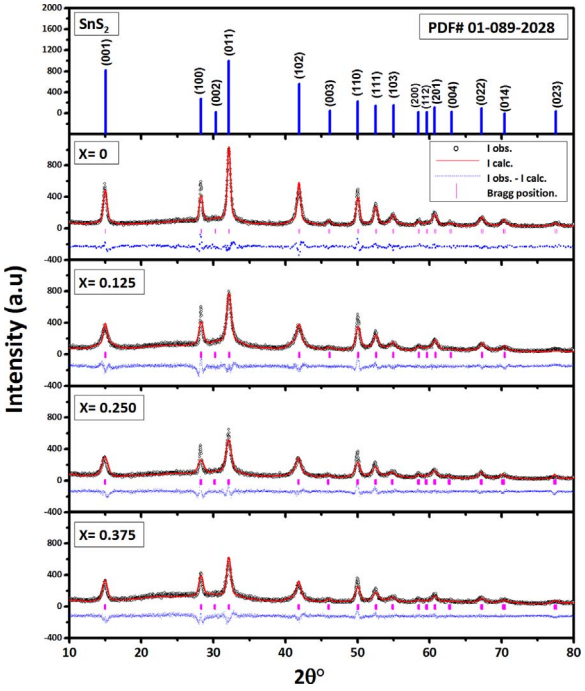

Fig. 1. : The X-ray diffraction patterns of $Sn_{1-x}Fe_xS_2$ (x=0, 0.125, 0.250 and 0.375) with the standard PDF Card - 01–089-2028. The red solid lines are the profile fitting obtained using Fullprof software.

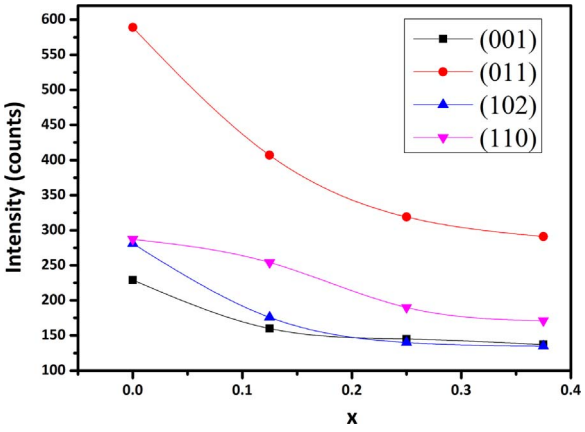

Fig. 2. : Shows the variation of the peaks intensity with the dopant concentration x.

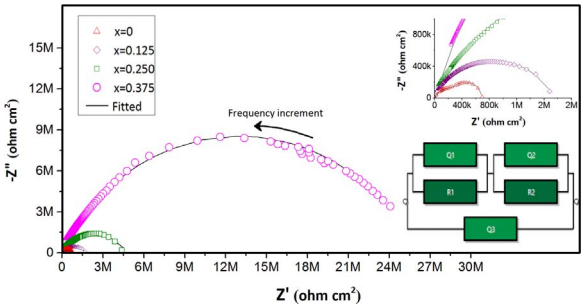

Fig. 3. : Nyquist plots for $Sn_{1-x}Fe_xS_2$ (x=0, 0.125, 0.250 and 0.375) measured at room temperature, The solid lines are the fits obtained using ZMAN software, the arrow shows the direction of frequency increment. The upper and lower insets show a zoomed view of the high frequency region and equivalent circuit used for modeling the data, respectively.

$$\varepsilon^* = \varepsilon' - i\varepsilon'' = [i\omega C\, Z^*]^{-1} \quad (2)$$

$$\varepsilon' - i\varepsilon'' = [i\omega C\,(Z' - iZ'')]^{-1} \quad (3)$$