Surface morphology and effect of particle sizes on the electrical conductivity of FeAl$_2$O$_3$ composite

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A composite sample of FeAl$_2$O$_3$ were synthesized by using mixture of paraffin wax and a powder of Al$_2$O$_3$ (0.07 gm.) with iron granular Fe (0.01 gm.) of different particle sizes (63μm, 73μm, 212μm and 300μm). Where paraffin wax was melted using a temperature source (heater) with 328 K, then the granular volume of composite FeAl$_2$O$_3$ powder was added to paraffin wax by manual mixing until the mixture was homogeneous and then deposited on slices of aluminum in laboratory conditions. The Characteristics of (current-voltage) were measured to various particle sizes. The electrical conductivity was estimated as a function of various temperature range. The conductivity values of this composite doped of FeAl$_2$O$_3$ increasing with temperature increases. Also, the activation energies Ea for the composite samples have been found (4.1x10$^{-4}$–2.9x10$^{-4}$) eV. Beside that the mechanisms of electrical conductivity were studied and identified in support of Schottky and Frankel effect mechanisms.

Keywords: Al$_2$O$_3$; Iron granular; Conduction Mechanism; I-V characteristics.

1. INTRODUCTION

The use of inorganic materials in optoelectronic devices have still considered commercial although the polymeric materials are widely being used in various devices as insulating materials and for important scientific and technological applications according to their properties such as light weight, mechanical flexibility, versatile of chemical structure, low manufacturing cost and mass production. The researches in this field take two main branches; Firstly, is to get new materials to having suitable electrical properties that serve as an effective layer. Secondly is to fabricate the device using an easy and efficient technique. Accordingly, the results obtained from investigating the electrical properties of the devices are based on satisfying the two later conditions. One of the main scientific concerns at present is the study of the important physical parameters including the electrical properties of composites and blend [1-3] as well as the effect of the thermal insulator matrix material on electrical properties and the magnetic properties of the metal- insulator compound [4-6] and it is considered one of the important and sensitive method in studying the properties of physical medium [7-99]. The process of identifying conduction mechanisms is determined due to the measurements (current-voltage) characteristics and (conductivity-temperature) curves [10,11]. The conduction mechanism in the metal-insulator compound may be depended on the thickness or the degree of deformation of the synthesized materials as well as the applied of the electric field intensity. The different physical
properties of the host material can be modified with different quality and concentration of organic filler used [12,13].

Metallic and semiconductor fillers such as graphite and carbon black are used to modify the electrical and thermal properties [14,15]. In past few years studies on optical and electrical characteristics of composite materials have been the subject of considerable research efforts due to their wide applications in the various technological devices. The physical properties of composite materials and determination of carrier concentration, as well as the mobility of Sb$_2$(Te$_{1-x}$Se$_x$)$_3$ thin films, have been studied [16,17]. The influence of sodium zirconate nanoparticles on the chemical structural and electrical properties of PVA nanocomposite films have been reported [18]. The effect of cobalt chloride on the electrical properties of poly [O-Toluidine] was reported [19,20]. Synthesis and study characteristics as well as the optical properties of (PANI) filled by Graphene nanofilms were also investigated [21,22]. In the present article the preparation and the study electrical properties of FeAl$_2$O$_3$ / paraffin wax composite film (Al$_2$O$_3=0.07$ gm) and (Fe=0.01gm) for various particle's size (63μm,73μm,212μm and300μm). The characteristic of (current-voltage) was measured at temperatures range (293-323 K). Also, the electrical conductivity was estimated at those temperature levels.

2. EXPERIMENTAL

2.1. Synthesis of CCTS solution

The films FeAl$_2$O$_3$ composite were synthesized from mixture of paraffin wax and a powder of Al$_2$O$_3$ and Fe. The fillers of Fe element powder was filtering by micro sifted using granular sieves whose size is 63 μm to 300 μm then 0.07 gm of aluminum oxide mixed with a granular iron element for each size by using a pie dish and a mixing spatula. Then a 0.01 gm of paraffin dough was added to the mixture. After that, the composition consisting of paraffin paste and aluminum oxide with the iron were deposited on the aluminum slices of dimensions (2.5 x 4 cm$^2$). These slices were left in the laboratory conditions for 24 hours. Then, the aluminum electrode were deposited in the shape of circles whose radius is 1 mm by using the evaporation system under the low pressure of $10^{-3}$ torr. After completion of electrode deposition, the samples were put on base of the special electrode measuring circuit which is consisting of a regulated voltages supplied by PHYWE 2592 power. Also, the digital hot plate was used to raise the temperature degree of the sample. The current values were measured by digital-meter type measuring amplifier D53200 L.H.Co. The temperature sensor of samples is placed over the layer films. The thickness of the composites samples was measured and indicated in table (1).

<table>
<thead>
<tr>
<th>Practical size</th>
<th>Thickness mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al$_2$O$_3$ gm</td>
<td>Fe gm</td>
</tr>
<tr>
<td>0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>0.01</td>
<td>0.03</td>
</tr>
<tr>
<td>0.01</td>
<td>0.05</td>
</tr>
<tr>
<td>0.01</td>
<td>0.07</td>
</tr>
</tbody>
</table>

3. RESULTS AND DISCUSSION

3.1 The surface morphology

The study of the surface and the homogeneous distribution of the film give desirable properties to be used in several applications, including solar cells and in communication devices, as well as in its use in
the manufacture of compact television screens and optical switches devices. The images films of FeAl$_2$O$_3$ composite of were illustrated in figure 1 corresponding to 63μm, 73μm, 212μm and 300μm particles size respectively. It gives morphology for films. It was showed that the roughness of the surfaces of these films are low and formed a smooth surface with increasing the particle sizes of FeAl$_2$O$_3$ composite films and the surfaces reveal without pinholes and porosity. The films exhibited a better surface with a very low deformity which indicated that the composites were complete forming FeAl$_2$O$_3$ from mixing paraffin wax with a powder of Al$_2$O$_3$ and iron. Figure 1 shows the, uniform scanning in the direction of the x-axis and polynomial fitting of the surface as a function of different particles sizes.

![Figure 1](image1.png)

**Figure 1** Scanning in the direction of the x-axis vs surface fitting of the FeAl$_2$O$_3$ films, (a) 63μm, (b) 73μm, (c) 212μm and (d) 300μm.

Figure 2 shows the small raw complete homogeneity of iron powder in the film and free of cracks and islands through the drawing of all the films, we notice that the small arrow indicates the homogeneous distribution and the absence of unwanted iron powder clusters and their intertwining with alumina. In addition, no obvious aggregation was observed from iron in the film samples.
3.2 MEASUREMENT OF ELECTRICAL PROPERTIES.

The current-voltage characteristic was conducted at various temperatures; 293-318 K and at weights of Al₂O₃=0.07 gm and Fe=0.01 gm for different particle sizes which are shown in figure 3 to figure 5 respectively. It is noted from the figures that, when a raise the temperature of the samples causes to rise in current, and, the shapes have the linear behavior which showed ohmic behavior at all applied voltage. Not down values of current and voltage (which Agrees with the current), it is followed by increasing the temperature of the sample to a temperature higher than its predecessor, which is leave for a while of 30 min. to reach the degree of thermal stability, then repeating the measurements more than once of current-voltage, until the highest permissible temperature range is reached.
Figure 3 The I-V characteristic for all particles size of composite FeAl$_2$O$_3$.

The relationship between Electrical conductivity and temperature was measured as a function of all particle sizes (FeAl2O3) composites (63μm, 73μm, 212μm and 300μm) for various temperatures (293-318K). The measurements of the Electrical conductivity showed a rise in electrical conductivity as the temperatures increases. This behavior in semiconductors for composite materials is considered traditional for most studies in this field due to the increased movement of charge carriers through the semiconductor connection. Figure 5 shows the variation of electrical conductivity of FeAl$_2$O$_3$ composite for a range of temperatures (293-318K) as a function of different particle sizes. It can be noticed that the electrical conductivity increases with the increasing particle sizes for all the ranges of temperature as shown in Table 2.

Table 2 Experimental Values of α’s from Schottky and Frankel theories and the dielectric constant for composite samples at Particle size 63 μm.

<table>
<thead>
<tr>
<th>Tk</th>
<th>α$_{exp}$</th>
<th>α$_{sh}$</th>
<th>α$_{P,F}$</th>
<th>ε</th>
</tr>
</thead>
<tbody>
<tr>
<td>293</td>
<td>0.18</td>
<td>0.058</td>
<td>0.116</td>
<td>0.610</td>
</tr>
<tr>
<td>298</td>
<td>0.17</td>
<td>0.057</td>
<td>0.114</td>
<td>0.611</td>
</tr>
<tr>
<td>303</td>
<td>0.16</td>
<td>0.056</td>
<td>0.112</td>
<td>0.612</td>
</tr>
<tr>
<td>308</td>
<td>0.14</td>
<td>0.055</td>
<td>0.110</td>
<td>0.614</td>
</tr>
<tr>
<td>313</td>
<td>0.12</td>
<td>0.054</td>
<td>0.10</td>
<td>0.617</td>
</tr>
<tr>
<td>318</td>
<td>0.10</td>
<td>0.053</td>
<td>0.09</td>
<td>0.620</td>
</tr>
</tbody>
</table>

Figure 4 Electrical conductivity for all particles sizes of composite FeAl$_2$O$_3$ at different temperatures range.
**Figure 5** Electrical conductivity as a function of particles size of FeAl$_2$O$_3$ composites at different temperatures range.

As shown in the previous figure (4), the electrical conductivity was measured by increasing the temperature of the sample. The activation energy of FeAl$_2$O$_3$ composite for various temperatures increases with the increasing the particle sizes as shown in Figures 6 and estimated to be (4.1x10$^{-4}$ – 2.9x10$^{-2}$) eV.

![Activation energy vs particle size graph]

**Figure 6** Activation energy of FeAl$_2$O$_3$ composite as a function of different particles sizes at different temperatures range.

### 3.3 Test of the conduction mechanisms

According to the measurements of the curves (current - voltage) and (conductivity – temperature) can be identified the conduction mechanisms which control the current transmission through composites materials. The multiplicity of these mechanisms in the compound metal-insulator and in the film consisting of a specific physical depends on many parameters such as the thickness of the sample, the temperature applied to the samples, the quantity and type of filling, the applied electric field intensity as well as the degree of deformation. The testing method has been done on the FeAl2O3 composite for different particle sizes (63μm to 300 μm).

A- Hopping Conduction Mechanism test that’s by drawing the ($\sigma$ versus $T^{-1/3}$) and($\sigma$ against $T^{-1/4}$) for the FeAl$_2$O$_3$ composite for different particle sizes(63um. to 300) as shown at figure 7 due to the following relations [23]:

$$\sigma = \exp(\frac{E_a}{KT^{1/3}})$$

(1)
It can be seen from figure (7) that, the relationship of conductivity with temperature is non-linear, which indicates the exclusion of this type of conductivity mechanism.

\[ \sigma = \exp \left( \frac{E_a}{K T^{1/4}} \right) \]  

(2)

**Figure 7** The relationship of conductivity with the reciprocal temperature ($T^{-1/3}$ and $T^{-1/4}$).

**B- SCHOTTY & PAUL FRENEL CONDUCTION MECHANISM**

The non-ohmic behavior of current with voltage is one of the reasons that a Schottky or Paul Frenkel mechanism dominant. This mechanism occurs when a voltage is applied to the contact area between the metal and the semiconductor, where electrons are transferred from the metal to the insulator [24]. The increase in the applied electric field with the temperature leads to a decrease in the voltage barrier. So, this mechanism is related to the thermal emission of electrons. The voltage barrier depends on the surface layer and the quality of this layer. Both effects Paul Frenkel [25] and glow discharge [26] reduce the voltage barrier to Schottky, but in this case, electrons are released from the capture trap levels to the conduction band in the compound. That is, the effect of both mechanisms are shown to decrease the stress barrier, the Schottky effect is on the surface while the Frenkel effect is inside the compound. At high voltage, Schottky's mechanism related with Paul Frenkel's Schottky's constant $\alpha_{sh}$ is related with Paul Frenkel's constant $\alpha_{PF}$ as [27]:

\[ \alpha_{sh} = \sqrt{\frac{q_3}{4 \pi \varepsilon \varepsilon_0}} \]  

(3)

where $q$ is the electron Charge, $\varepsilon$ dielectric constant and $\varepsilon_0$ is the dielectric Constant in the Space.

\[ \alpha_{sh} = \frac{1}{2} \alpha_{PF} \]  

(4)

Figure (8) shows the relationship between the current and the square root of the voltage of the FeAl$_2$O$_3$ composite for various particle sizes. It can be seen that the relationship is linear at high fields. From
the slope of the straight line at high electric fields in Figure 9, the experimental constants' values for Schottky and Paul Frenkel were determined from Equations 3 and 4. The experimental values of $\alpha$ in are shown in Table 2 and Table 3 for particle's size of (63 μm and 212 μm.) are being more nearly to theoretical $\alpha_{P.F}$, that indicates the Paul Frenkel is dominated mechanism, so the applied filed is sufficient to modify the thermally excited electron inside the composite FeAl$_2$O$_3$ and estimated $\alpha$'s Values from Schottky and Frenkel theories and the dielectric constant for composite samples.

**Table 3** Experimental Values of $\alpha$'s from Schottky and Frankel theories and the dielectric constant for composite samples at Particle size 212 μm.

<table>
<thead>
<tr>
<th>$T_k$</th>
<th>$\alpha_{exp}$</th>
<th>$\alpha_{sh}$</th>
<th>$\alpha_{P.F}$</th>
<th>$\epsilon$</th>
</tr>
</thead>
<tbody>
<tr>
<td>293</td>
<td>0.053</td>
<td>0.057</td>
<td>0.114</td>
<td>0.611</td>
</tr>
<tr>
<td>298</td>
<td>0.055</td>
<td>0.056</td>
<td>0.112</td>
<td>0.612</td>
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<td>0.108</td>
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<td>318</td>
<td>0.065</td>
<td>0.052</td>
<td>0.105</td>
<td>0.624</td>
</tr>
</tbody>
</table>
From the relationship between log (Io/T^2) versus reciprocal temperature (Richardson plot), where Io the extrapolation of current value to zero applied field, the lowering of the work function for the thermionic emission due to the Schottky effect can be determined from [28]:

$$\Delta \phi = \sqrt{e E \frac{4 \pi \varepsilon \varepsilon_0}{k_B}}$$  \hspace{1cm} (5)

These are shown in figure (10) for composites particle size 63 μm, 73 μm, 212 μm and 300 μm, respectively.
C- ION CONDUCTION MECHANISM TEST

Ion conduction mechanism tested by drawing the (- Lnσ T^{1/2}) against (1000/T) K and then investigated due to the following formula [29,30]:

\[ \sigma = \sigma_0 \sqrt{T} \exp(-E_a KT) \]

where \( \sigma_0 \) constant, \( E_a \) is the activation Energy, \( K \) Boltzmann constant and \( T \) is the sample temperature. Figure (11) shows the drawing of ln (σ T^{1/2}) verses (1000 /T) K, of the FeAl₂O₃ composites for various particle sizes. A number of other mechanisms have been investigated, such as the ion conduction mechanism and the tunneling mechanism, and it was found graphically that both mechanisms are not applicable in our present study because the graphical curves of the practical results do not apply with the theoretical foundations of both mechanisms.

4. CONCLUSIONS

CCTS nanostructure has been prepared successfully and deposited on SiO₂/Si via sol-gel method at 400 °C. UV-vis has demonstrated band gap, 1.35 eV. X-ray diffraction pattern has showed five peaks; (112) plane is attributed to the highest one whereas, particle size was 26.8 nm. Topography and
morphology have indicated the nanostructures coherency and homogeneity as shown in AFM and SEM images, respectively. The roughness was 1.95 nm. Ag IDE was fabricated on CCTS/SiO$_2$/Si via PVD and hard mask. The model of Herve and Vandamme is agreed well for CCTS quaternary alloy nanostructure. Also, the investigated bulk modulus exhibits the same chemical trends as those found elsewhere in the literature. As seen in electrical measurements, there is a proportional relationship between I and V which is interpreted as Schottky barrier. Otherside, impedance and capacitance measurements have showed inversely correlation with frequency, where they are slightly affected with voltage change. Furthermore, device conductivity increases as frequency increases. These results indicated that Ag IDEs deposited on CCTS/SiO$_2$/Si using PVD and hard mask have showed good sensing capabilities and it can be used as biosensor for detecting different types of DNA in future studies.

References

[29] F. Frenkel, Physical Review 54 (1938) 467