Studies on Physical and Electrical Characterization of Al₂O₃-ZnO Composite Material and its Thick Films

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Abstract: Al₂O₃ - ZnO composite material was obtained by adding the Aluminium chloride (AlCl₃ ·6H₂O) (Hexahydrate) powder of different weight percent (0.5, 1 3, 5 and 7 wt %) and Analar Reagent grade (99.9 % pure) ZnO powder by mixing mechnochemically in acetone medium. The prepared materials were sintered at 1000°C for 12h in air ambience and ball milled to ensure sufficiently fine particle size. The structural properties of the powder materials were investigated by X-ray diffraction analysis. The observed powder materials show the polycrystalline nature and the crystallite size found to be in the range of 10 to 36 nm. The thick films of undoped ZnO and Al₂O₃ doped-ZnO were prepared by screen printing technique. The surface morphology of the films was studied by SEM and it shows the films are porous in nature and petal-shaped grains of sizes varies from 190nm to 276nm were observed. The final composition of each film was determined by EDAX analysis. The At. wt. % of Zn and O in each sample was not as per stoichiometric proportion. The electrical conductivity and activation energy of all films were determined.

Keywords: ZnO, Al₂O₃, Thick Films, Crystalline size, Electrical Conductivity, Activation Energy.

1. Introduction

The requirements of semiconductor material are increased along with the expanding of global electronic industry [1][2]. Semiconductor metal oxides as high performance catalysts for energetically and environmentally improved catalytic combustion of sulfur and hydrocarbons. In order to enhance the catalytic activity (oxidation), the oxides were doped with it using mechanical mixing of some impurities. The aim of present research work is to enhance the catalytic activity (oxidation) of Zinc Oxide (ZnO) using mechanical mixing of Al₂O₃. Metal oxides are extremely important technological materials for use in electronic and photonic devices. The conventional oxide-mixing techniques; powders are produced more homogeneous after the sintering process. In addition, the grain size obtained is much smaller [3]-[4].

Al₂O₃ is weak n-type semiconductor material with wide band gap(8.8 ev) for bulk material in different crystalline form and good thermal stability. It works as good catalyst with semiconductor when the gases such as H₂S and Ethanol come in contact with it.

Zinc oxide (ZnO) is an n-type direct band semiconductor of a wide bandgap (3.4 eV). It is an important multifunctional material with wide ranging applications in varistors [5],surface acoustic wave (SAW) devices[6], transparent conducting oxide electrodes [7], solar cells [8], blue/UV light emitting devices [9], gas sensors [10]-[11], etc. Its conductivity can be tailored by controlling the deviation from stoichiometry and by doping [12]. The undoped ZnO has high n-type conductivity due to defects such as oxygen vacancies and Zn interstitials, which form donor levels [13]. Group IIIa elements (Al, Ga, In) have been used to improve the electrical conductivity and thermal stability of ZnO films. For this work, Al³⁺ substitution on Zn²⁺ (was chosen due to the small ion size of Al³⁺ compared to that of Zn²⁺ (Al³⁺ (0.53Å) and Zn²⁺ (0.74 Å)).

2. Experimental Procedure

2.1 Preparation of Functional Material

Al₂O₃ - ZnO composite material were obtained by adding the Aluminium chloride (AlCl₃ ·6H₂O) (Hexahydrate) powder of different weight percent (0.5, 1 3, 5 and 7 wt %) and Analar Reagent grade (99.9 % pure) ZnO powder by mixing mechnochemically in acetone medium. The prepared materials were sintered at 1000°C for 12h in air ambience and ball milled to ensure sufficiently fine particle size.

2.2. Preparation of Thick Films

The thixotropic paste was formulated by mixing the fine powder of functional material with a solution of ethyl cellulose (a temporary binder) [14] in a mixture of organic solvents such as butyl cellulose, butyl carbitol acetate and terpineol, etc. The ratio of the inorganic to organic part was kept at 75:25 in formulating the paste. This paste was screen-printed [15]-[16] on a glass substrate in a desired pattern (1.5cm x 0.5cm). The films were fired at 550°C for 30 min. in air atmosphere

2.3. Thickness measurements of films

The range of thicknesses of the films was observed in the range from 55 to 65μm. The reproducibility in thickness of the films was possible by maintaining the proper rheology and thixotropy of the paste.

3. Physical Characterization

3.1. Structural analysis

In order to understand the structural properties of AlO₃ - doped ZnO powder materials at different dopant concentration, X-ray diffraction analysis of these sintered
powders were carried out in the 20-80° range using CuKα radiation.

Figs. 1(b-f) show the XRD patterns of 0.5, 1, 3.5, and 7 wt. % Al2O3-ZnO composite material. All the patterns exhibit an intensive (002) XRD peak, indicating that they have c-axis-preferred orientation due to the self-texturing mechanism as discussed by Deng et al [23]. The 2θ values of the diffraction peaks (002) are located at 34.40°, which are very close to that of standard ZnO crystal. No Al2O3 or ZnAl1O2 phase was detected from the XRD spectra. This may be due to aluminum replacing zinc substitutionally in the hexagonal lattice or aluminum segregating to the noncrystalline region in grain boundary.

From the XRD results, it is concluded that the material properties are strongly dependent on aluminum concentration. The diffraction peak intensity of Al2O3-ZnO films decreased with increased doping concentrations. This indicates that an increase in doping concentration deteriorates the crystallinity of the films, which may be due to the formation of the stresses by the difference in ion sizes between zinc and the dopant [17, 18] and the segregation of dopants in grain boundaries for high doping concentrations.

The average crystallite size was calculated from XRD pattern using Debye Scherer’s formula [19].

\[ D = \frac{0.9 \lambda}{\beta \cos \theta} \]  

Where D- is average crystallite size, β - is the broadening of the diffraction line measured at half maximum intensity(FWHM), \( \lambda \) - is wavelength of the x- ray radiation and (0.1542 nm). \( \theta \) - is the Bragg angle.

Table 1: XRD data of undoped ZnO and Al2O3-ZnO composite material

| Sample          | Sr. No. | 2θ deg. | d (Å) | FWHM | D(nm) | Plane (hkl) |
|-----------------|---------|---------|-------|------|-------|-------------|
| Undoped ZnO     | 1       | 31.70   | 2.82  | 0.265| 35    | Z-100       |
|                 | 2       | 72.60   | 1.30  | 0.427| 26    | Z-004       |
| 0.5 wt.% Al2O3-ZnO | 1     | 31.80   | 2.81  | 0.259| 35    | Z-100       |
|                 | 2       | 72.60   | 1.29  | 1.112| 36    | Z-100       |
| 1 wt.% Al2O3-ZnO | 1       | 31.80   | 2.80  | 0.258| 35    | Z-100       |
|                 | 2       | 72.60   | 1.30  | 0.584| 19    | Z-004       |
| 3 wt.% Al2O3-ZnO | 1       | 31.80   | 2.81  | 0.259| 35    | Z-100       |
|                 | 2       | 72.60   | 1.30  | 0.611| 18    | Z-004       |
| 5 wt.% Al2O3-ZnO | 1       | 31.80   | 2.80  | 0.259| 35    | Z-100       |
|                 | 2       | 72.60   | 1.30  | 1.112| 10    | Z-004       |
| 7 wt.% Al2O3-ZnO | 1       | 31.80   | 2.81  | 0.259| 35    | Z-100       |
|                 | 2       | 72.60   | 1.298 | 1.112| 10    | Z-004       |

d = Interplaner distance in Å

D = Crystallite Size in nm

Table 1 shows the variation of crystallite size with doping concentration of Al2O3 in ZnO samples. Slightly broadening of diffraction lines may be attributed to small crystalline effects [20]. From the XRD pattern, the lattice constants of hexagonal ZnO phase can be calculated using the equation [20]-[21].

\[ \frac{1}{d_{(hkl)}^2} = \left( \frac{h^2 + kl + k^2}{a^2} \right) + \left( \frac{l^2}{c^2} \right) \]  

Figure 1 shows the XRD spectra of Al2O3-doped ZnO composite material.
times larger than the average crystallite sizes calculated from 190nm to 276nm. The majority of these grains appear several were observed in all samples. The grains of sizes varies from 0.53Å [22]. This should result in decreased lattice parameters po
gains would be greater than optimum level, it reduces the porosity of the film surface.

The ZnO film doped with 1 wt. % Al2O3-ZnO phases was not detected. This might be due to relatively low sintering temperature (~1000°C) at which their crystallization might not have occurred, suggesting formation of the secondary phases probably in the amorphous form.

Table 2: Lattice constants Al2O3-doped ZnO composite material

| Phase          | hkl-Plane | 2θ  | d (Å)  | a (Å)  | c (Å)  |
|----------------|-----------|-----|--------|--------|--------|
| ZnO (Undoped) | 101       | 36.20 | 2.4794 | 3.240  | 5.291  |
| 0.5 wt.% Al2O3-ZnO | 101 | 36.20 | 2.4720 | 3.231  | 5.276  |
| 1 wt.% Al2O3-ZnO | 101 | 36.20 | 2.4688 | 3.226  | 5.269  |
| 3 wt.% Al2O3-ZnO | 101 | 36.20 | 2.4753 | 3.235  | 5.283  |
| 5 wt.% Al2O3-ZnO | 101 | 36.20 | 2.4720 | 3.231  | 5.276  |
| 7 wt.% Al2O3-ZnO | 101 | 36.20 | 2.4720 | 3.231  | 5.276  |

From Table 2, it has been observed that there is variation of lattice constants from JCPDS value (a = 3.253Å, c = 5.213Å). The ionic radius of Zn2+ is 0.74Å and of Al3+ is 0.53Å [22]. This should result in decreased lattice parameters as per Vegards Law. However, such a trend is not observed as the sintering temperature is very low, and hence diffusion of Al into Zn sites might not have occurred to a complete extent. The possible formation of Al2O3 or ZnAl2O4 phases was not detected. This might be due to relatively low sintering temperature (~1000°C) at which their crystallization might not have occurred, suggesting formation of the secondary phases probably in the amorphous form.

3.2. Micro structural analysis of the films

The surface morphology and chemical composition of the films were analyzed using a scanning electron microscope [SEM model JEOL 6300 (LA) Germany] coupled with an energy dispersive X-ray analysis. (EDAX, JED-2300, Germany). Figs. 2 (a-e) show the surface morphology of thick films of pure ZnO and Al2O3-ZnO (0.5, 1, 3.5, and 7 wt. %) thick films. Plane-view SEM investigation reveals a porous structure of the ZnO films with different Al2O3 doping concentrations. Petal-shaped grains of various sizes were observed in all samples. The grains of sizes varies from 190nm to 276nm. The majority of these grains appear several times larger than the average crystallite sizes calculated from X-ray diffraction data (10-36 nm) thus, indicating that most of the grains comprise multiple crystallites. No systematic variation in the microstructure of the ZnO films as a function of the doping concentration was detected.

The ZnO film doped with 1 wt. % Al2O3 was observed to be the most porous. The Al2O3 additives would present on the ZnO grains are in an optimum level leading to high porosity. In Figs.2 (d, e and f), the Al2O3 additives distributed on ZnO grains would be greater than optimum level, it reduces the porosity of the film surface.

Table.3 gives quantitative elemental analysis of Al2O3-doped ZnO thick films at different level. The EDAX analysis shows presence of only Zn, Al and O as expected, no other impurity elements were present in the thick films. The EDAX result shows variation in Zn/O ratio and Al/Zn ratio with variation in doping concentration. The At. wt. % of Zn and O in each sample was not as per stoichiometric proportion. The entire samples were observed to be oxygen deficient.

The composition of pure and Al2O3-doped ZnO thick films with different mass ratio were analyzed by energy dispersive X-ray analysis. (EDAX, 6360LA).

Table 3: Quantitative elemental analysis of undoped ZnO and Al2O3-ZnO films

| Sample              | At. Wt. % of Elements |
|---------------------|-----------------------|
|                     | O    | Zn    | Al    | Total |
| ZnO (Undoped)       | 21.18 | 78.82 | -     | 100   |
| 0.5 wt.% Al2O3-ZnO  | 16.71 | 83.44 | 0.35  | 100   |
| 1 wt.% Al2O3-ZnO    | 15.43 | 83.45 | 2.12  | 100   |
| 3 wt.% Al2O3-ZnO    | 17.25 | 80.22 | 2.53  | 100   |
| 5 wt.% Al2O3-ZnO    | 18.31 | 77.83 | 3.86  | 100   |
| 7 wt.% Al2O3-ZnO    | 18.44 | 76.28 | 5.27  | 100   |

Figure 2: SEM images of (a) undoped ZnO, (b) 0.5 wt.% (c)1 wt.% (d) 3 wt.% (e) 5 wt.% and (f) 7 wt.% Al2O3 – ZnO films.
4. Electrical Characterization

4.1 I-V Characteristics of the films

Fig. 3 shows the I-V characteristics of pure and Al$_2$O$_3$ doped ZnO films at room temperature in air ambience. I-V Characteristics are observed to be symmetrical in nature, indicating the ohmic nature of aluminium contacts.

4.2. Electrical conductivity of the films

The conductivity of Al$_2$O$_3$ -doped ZnO thick films at constant temperature was calculated using the Eq. (4)[23],

$$\sigma = \frac{1}{(R \times b \times t)} \left( \frac{1}{\beta - m} \right)$$  \hspace{1cm} (4)

Where
- $R$ = Resistance of thick film at constant temperature
- $t$ = thickness of the film sample
- $l$ = length of the thick film
- $b$ = breadth of the thick film

Fig.4 shows that the conductivity films increases with increase in dopant concentration of Al$_2$O$_3$ up to 3 wt. % then decreases with increase in dopant concentration of Al$_2$O$_3$ i.e from 5 wt. % to 7 wt. %. Thus, it was found that the conductivity of Al$_2$O$_3$ - ZnO films is strongly dependent on the Al$_2$O$_3$ dopant concentration. In general, at low doping concentration of i.e. below the solubility limit, Al$^{3+}$ ions substitute Zn$^{2+}$ ions in the ZnO lattice site and increase the electrical conductivity by increasing the donor concentration [24] – [26]. These reactions easily take place because Al$^{3+}$ (0.53 Å) and Zn$^{2+}$ (0.74 Å) have similar ionic radii.

$$\text{Al}_2\text{O}_3 + \text{ZnO} \rightarrow \text{ZnO}_2 + 2\text{Al}^{2+} + 3\text{O}_2^- + \frac{3}{2} \text{O}_2(\text{g}) + 2\text{e}^-$$  \hspace{1cm} (5)

Where $O_2^-$ is the oxygen at oxygen site

At high doping concentration Al atoms may also enter the structure and have preference for interstitial sites. This interstitial substitution occurs either directly or by substitution of Zn atoms on its regular site at first and then moving to the interstitial position. At the interstitial position, the aluminium atoms absorb an electron. In this case, the aluminium behaves as an acceptor and decreases the conductivity [27].

$$\text{Al}_2\text{O}_3 + \text{ZnO} \rightarrow \text{ZnO}_2 + 2\text{Al}^{2+} + 3\text{O}_2^- + \text{O}_i^-$$  \hspace{1cm} (6)

Where $O_i^-$ is the negatively charged oxygen interstitial, $O_o^-$ the oxygen at oxygen site,

Eq. (5) indicates a decrease in resistance whereas Eq. (6) lead to an increase in resistance. It may be concluded from Fig. 4 that in the present sample Eq. (6) is predominantly operative for undoped and 0.5, 1, 3 wt. % Al$_2$O$_3$ doped ZnO, indicating an increase in conductivity, and the mechanism from Eq.(6) is possibly favorable at higher dopant concentrations of Al$_2$O$_3$ (5, 7 wt. %) in indicating decrease in conductivity.

4.3. Activation energy of the films

Activation energy can be thought of as the height of the potential barrier or energy barrier separating two minima of potential energy. Activation energy of Al$_2$O$_3$ doped ZnO thick films were calculated from Arrhenius plot for different operating temperature regions by using Eq. (7).[28]

$$\sigma = \sigma_o e^{-\Delta E/kt}$$  \hspace{1cm} (7)

Where
- $\sigma$ = Electrical conductivity (Ω cm)$^{-1}$
- $\sigma_o$ = Electrical conductivity at const. temp.(Ω cm)$^{-1}$
- $\Delta E$ = Activation energy of the electron transport in the conduction band,
- $K$ = Boltzmann constant, $T$ = Absolute temperature.
other doping concentrations. It may be due the barrier height activation energy of undoped and Al operating temperature regions. Fig. 7.

Table 7.4 shows the calculated activation energy of all samples for different doping concentration at different operating temperature regions. Fig. 7.5 shows variation of activation energy of undoped and Al doped ZnO thick films as the function of operating temperature.

The graph shows that the doped film having low activation energy than undoped film. It also shows that the activation energy varies with the dopant concentration. The film doped with 3 wt. % of Al shows lowest activation energy than other doping concentrations. It may be due the barrier height decreases as Al doping increases.

Table 7.4: Activation energy of undoped and Al$_2$O$_3$ -ZnO film with temperature

| $T$(°C) | Undoped | Activation energy (ev) | Al$_2$O$_3$ -doped ZnO |
|---------|---------|------------------------|------------------------|
|         | ZnO 0.5wt.% | 1%wt. | 3%wt. | 5%wt. | 7wt% |
| 100     | 0.032 | 0.076 | 0.031 | 0.007 | 0.067 | 0.028 |
| 150     | 0.028 | 0.092 | 0.023 | 0.005 | 0.080 | 0.053 |
| 200     | 0.011 | 0.094 | 0.028 | 0.004 | 0.078 | 0.061 |
| 250     | 0.024 | 0.092 | 0.026 | 0.002 | 0.071 | 0.071 |
| 300     | 0.064 | 0.087 | 0.011 | 0.005 | 0.045 | 0.086 |
| 350     | 0.100 | 0.070 | 0.007 | 0.028 | 0.004 | 0.102 |
| 400     | 0.139 | 0.051 | 0.003 | 0.076 | 0.017 | 0.116 |
| 450     | 0.177 | 0.026 | 0.052 | 0.086 | 0.025 | 0.130 |

5. Summary and Conclusions

From the results obtained, following conclusions can be made
1) It shows that the Al$_2$O$_3$ –ZnO functional material can be obtained by mechanochemical method.
2) The XRD analysis shows that the observed powder materials show the polycrystalline nature and the variation in the crystallite size due to doping. The crystallite size was found to be in the range of 10 to 36 nm.
3) The SEM analysis shows the surface morphology of the of undoped ZnO and Al$_2$O$_3$ doped-ZnO thick films and it shows the films are porous in nature and petal-shaped grains oriented randomly of sizes varies from 190nm to 276nm were observed.
4) The EDAX analysis shows final composition of each film. The At. wt. % of Zn and O in each sample was not as per stoichiometric proportion. The entire samples were observed to be oxygen deficient.
5) The electrical conductivity for 0.5, 1, 3 wt. % Al$_2$O$_3$ doped ZnO increase while at higher dopant concentrations of Al$_2$O$_3$ (5, 7 wt. %) it decrease.
6) The doped film shows low activation energy than undoped film. It also shows that the activation energy varies with the dopant concentration. The film doped with 3 wt. % of Al$_2$O$_3$ shows lowest activation energy than other doping concentrations. It may be due the barrier height decreases as Al$_2$O$_3$ doping increases.
7) Al$_2$O$_3$ doped-ZnO thick films would be use full for gas sensing application.

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