Iraqi Journal of Science, 2022, Vol. 63, No. 12, pp: 5232-5241 DOI: 10.24996/ijs.2022.63.12.13





ISSN: 0067-2904

Synthesis and Dielectric Properties of MgO:ZnO Composites

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Received: 5/10/2021

Accepted: 23/2/2022

Published: 30/12/2022

Abstract:

The dielectric properties of the fabricated composites MgO:ZnO with various mixing ratios (100,75:25,50:50,25:75, and 100 wt. %)were investigated. The structure analysis was conducted using X-ray diffraction. The structure phase, crystallite size and purity of the fabricated MgO:ZnO composites were confirmed using X-ray diffraction spectra. The results declared that the diffraction spectrum of 100% MgO composite samples were compatible with cubic structure along the plane (200) while the structures of residual composite's samples were compatible with hexagonal structures. The crystal size of the most pronounced plane (101) for crystal growth was changed from 30.4 nm to 53.2 nm by increasing ZnO ratio from 25 to 100wt%. The dielectric properties were studied as function of frequency over the range (50Hz-10MHz). The a.c conductivity $\sigma_{a.c}(\omega)$ showed power low dependence for the full frequency range except for the composites samples of 0 and 75 % wt. ZnO which showed d.c region in the low frequency range. The exponent (s) values which represents the slope of $\ln \sigma_{a,c}(\omega)$ and $\ln(\omega)$ changed in non-regular manner by increasing the ZnO ratio. The dielectric constant $\epsilon 1$ and the dielectric loss ϵ_2 increased with the increase of the ZnO ratio up to 75% ZnO and then decreased with further increase of ZnO ratio. The dielectric loss peaks observed in the plot diagram of ε_2 against ln(ω) is found to shift towards the high-frequency side which indicates the decrease of relaxation time and prompt movement of charge carriers .The polarizability values (α) estimated from the COLE –COLE diagram increased from 0.112 to 0.467 when the ratio of ZnO changed from 0 to 50wt.% which referred to reduction of the intermolecular forces. While (α) reduced drastically at 75wt.% ZnO which referred to the growing of the intermolecular forces.

Keywords: A.C conductivity, dielectric properties of MgO:ZnO composites, x-ray diffraction

التحضير والخواص العزلية لمتراكبات MgO:ZnO

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الخلاصة

الخواص العزلية لمتراكبات اوكسيد المغنيسيوم اوكسيد الزنك بنسب خلط مختلفة تم دراسة .تم تحليل التركيب بواسطة حيود الاشعة (100,75:25,50:50,25:75, and 100 wt. %) السينية .تم التاكد من الطور , الحجم البلوري وكذلك النقاوة من طيف الاشعة السينية .اظهرت النتائج ان طيف الحيود لاوكسيد المغنيسيوم كان متطابقا مع الطور المكعبي وفي الاتجاه (200) في حين ان بقية المتراكبات كانت متطابقة مع الطور السداسي .الحجم البلوري للمستوي المفضل للنمو (101) تغير من مع زيادة نسبة الخلط من 25 الى 100 wt% الى 30.4 nm . تم دراسة الخواص $\sigma_{a.c}(\omega)$. الم 100 wt% العزلية كدالة للتردد ضمن المدى . (50Hz-10MHz) . اظهرت التوصيلية المتناوبة (ω) العزلية كدالة للتردد ضمن المدى . (50Hz ZnO) . اظهرت التوصيلية المتناوبة (ω) اعتمادا اسيا على الترد ماعدا النموذجين Mo . ZnO والذين اظهرا توصيلية مستمرة عند الترددات الواطئة . قيم العامل الاسي (S) والذي يمثل ميل الرسم بين (ω) الم مع الم وجد الم عدى التردد ماعدا النموذجين (S) والذي يمثل ميل الرسم بين (ω) الم وجد انها تريد ماعدا النموذجين (S) والذي يمثل ميل الرسم بين (ω) الم وجد انها تريد منتظمة مع زيادة نسبة اوكسيد الزنك. لوحظ قمم في طيف معامل الفقد $_{23}$ مع (ω) الم والذي وجد انها ترحف باتجاه الترددات العالية والذي يشير الى هبوط في زمن الاسترخاء وحركة سريعة والذي وجد انها ترحف باتجاه الترددات العالية والذي يشير الى هبوط في زمن الاسترخاء وحركة سريعة الماملات الشحنة . الاستقطابية والتي تم حسابها من منحني وجد انها ازدادت من 1100 الى الماملات الم الم والذي يشير الى منوط في زمن الاسترخاء وحركة سريعة الماملات الشحنة . الماملات الماملات الماملات الم معامل الفقد $_{23}$ مع (ω) الماملات والذي وجد انها ترحف باتجاه الترددات العالية والذي يشير الى هبوط في زمن الاسترخاء وحركة سريعة الماملات الشحنة . الاستقطابية والتي تم حسابها من منحني وجد انها ازدادت من 1100 الى الماملات الم الجزيئية في حين هبطت قيم الاستقطابية بشدة عند نسبة %7000 منا وكميد الزنك من 10 الى والذي يشير الى تنامي القوى الرابطة الجزيئية.

Introduction:

Recent research have focused on investigating the properties of mixed metal oxides due to many applications in different fields like physics, chemistry and engineering science. Materials with novel properties can be obtained by combining more than one metal oxide to the oxide matrix. The fabrication of metal oxides attracted valuable consideration because of their very important applications in optoelectronic and photonic devices [1-5]. Zinc oxide, an n-type semiconductor with a band gap of 3.37 eV at R.T, has considerable uses due to its optoelectronic, catalytic, electrical, and photochemical properties [6]. Magnesium oxide semiconductor is of a wide band gap with characteristic structures that lead to unique electronic, optical, magnetic, mechanical, thermal, and chemical properties [7]. The development of new composites from mixing both oxides opens a new field of applications. Recent research is concerned with the fabrication of ZnO/ MgO for their applications in advanced materials technologies. Different methods were used to synthesise ZnO/MgO nanoparticles [8-19]. These composites in small sizes attracted attention because of the confinement effect [20-22].

The present work is concerned with the synthesis of zinc oxide/magnesium oxide (ZnO/MgO) composites as well as the study of their dielectric properties. Many valuable information can be obtained from the dielectric properties measurements like the dc conductivity, dielectric constant, dielectric loss, polarizability, capacitance, and relaxation time.

Materials, experimental procedure, and basic concepts

MgO and ZnO oxides (particle size of 45-50µm) with high purity were provided from Sigma Aldrich. Composites samples were synthesized according to the following mixing ratios of MgO:ZnO of (100:0, 75:25, 50:50, 25:75, and 0:100 wt. %) using solid solution interaction operation. The samples were sintered at a temperature of 1273°C for 5 hours. The materials were grinded to obtain good homogeneity; and pressed into pellets by subjecting a pressure of 8 tons for 20 sec. The structure of the synthesized composites was examined using X-ray diffraction. The dielectric properties were measured using a dielectric analyzer (type Hewlett Packard model-HP4274A) which were done by inserting the pellets samples between two electrodes .The obtained data include the resistance, capacitance and loss factor as a function of frequency(f) in the range (50-10 MHz).The alternative current conductivity σ (tot) (ω) at certain angular frequency (ω =2*3.14*f) is expressed by two terms $\sigma_{tot} = \sigma_{a.c}(\omega) + \sigma_{d.c}$: the first does not depend on frequency but on temperature, the second depends on frequency which may be written as $\sigma_{a.c}(\omega) \propto \omega^s$ or $\sigma_{a.c}(\omega) = A\omega^s$ (2) where Hasan

A is constant, The value of (s) can be measured from the decline of $\ln \sigma_{a.c}(\omega)$ against $\ln(\omega)$.

The following equations were used to measure the a.c conductivity (σ_{ac}), dielectric constant ε_1 and dielectric loss ε_2 :

$$\sigma = \frac{t}{R.A^*} \tag{3}$$

$$\varepsilon_1 = C.t / \varepsilon_0 .A^* \tag{4}$$

$$\varepsilon_{2} = \sigma_{ac} / \omega \varepsilon_{o} \tag{5}$$

Where: ω , t, R, A*, C and ε_0 are the angular frequency =2* π *f, film thickness, the sample resistance, the effective area for capacitance, and the **p**ermittivity of free space =8.854x10⁻¹⁴ (F/cm), respectively.

Results and Discussion

The X-ray spectra of MgO:ZnO composites with mixing ratios:(100:0, 75:25, 50:50, 25:75, and 0:100 wt. %) are displayed in Figure 1. It was clearly observed that all diffraction peaks for 100%MgO belonged to magnesium oxide with cubic structure according to the card number 96-100-0054 and the growth occurred at $2\theta = 42.83^{\circ}$ at the plane (200). The composites samples of the ratios (75:25, 50:50, 25:75 wt.%) occurred at 20 of 36.42° at plane (101), according to the card number 96-230-0113, which belonged to cubic and hexagonal structures. The peaks for 100% ZnO matches well the hexagonal structure of zinc oxide. The obtained results are compatible with those of Anandan1 et al. [23] and Siregar and Panggabean [24]. The plane of crystal growth changed from (200) to (101) by the continuous addition of ZnO to MgO. This change was accompanied by gradual reduction of the peak intensity of the (200) plane on the expense of the growing of the peak intensity of the (101) plane, till it vanished at 100%ZnO. The size of crystal has increased from 12.8 to 53.2nm by the effect of the continuous addition of ZnO.



Figure 1: The X-ray diffraction pattern of MgO:ZnO composites.

Figure 2 shows the change of $\ln \sigma_{a.c}(\omega)$ against ln angular frequency(ω) for MgO:ZnO with different ratios. Two regions can be noticed for 100%MgO and 25:75, the first region at high frequency usually called D.C region, this is due to the fast movement of charge carriers and jumping from one available site to another. Succeeded jumping to the nearest neighbor sites participate in the D.C conductivity, where the frequency is lower than that for the jumping named ωp . In the second region, the high frequency region, the frequency overtakes ωp , the conductivity showed proceeding increase with increasing of frequency [25-29].

The other composites showed only one region (the frequency dependent region). It can be observed that the conductivity is clearly affected by increasing the ZnO ratio. The conductivity increased by increasing the ZnO ratio up 75 % and then it decreased. Indeed, the conductivity increased from 3.95×10^{-8} to 3.41×10^{-5} (ohm .cm)⁻¹ when ZnO ratio increased from 0 to 75 % and then fall to 1.78×10^{-6} (ohm .cm)⁻¹ with further increase of ZnO ratio. The exponent values (s) which were deduced from the slope of Figure 2 are shown in Table 1. It is worthy to note that the s values lie in the range 0 < s < 1 where s takes a value of zero when there is an ideal Debye dielectric type while it takes a value of 1 for an the ideal ionic type [30-32].

It can be observed that (s) change in a non-regular manner i.e. decreases and increases by the continuous addition of ZnO. The reduction of (s) suggested that the correlated barrier hopping (CBH) is the most convenient model. This occurred when the electrons get to hope over the potential barrier between two neighbor sites. Here the alternative current conductivity $\sigma_{a.c}(\omega)$ takes place as a results of jumping between two states or dangling bonds (D+D-) [33-34]. The growing of (s) suggested that small poloran (SP) is the most suitable to illustrates the behavior of the fabricated composites. When the factor (s) increases with ZnO ratio, this is because of large amount of domestic lattice distortion, which is named small polaron,



Figure 2: $\ln \sigma_{a.c}(\omega)$ against Ln (ω) of MgO:ZnO composite of different ratios

The relative permittivity is the permittivity of a material expressed as a ratio with the electric permittivity of a vacuum. A dielectric is an insulating material, and the dielectric

constant of an insulator measures the ability of the insulator to store electric energy in an electrical field. The real part of the dielectric constant ε_1 is related to the energy store within the material meanwhile one cycle which supplied from the subjecting alternating electric field. Figure 3 and 4 shows variation of the dielectric constant ε_1 and the dielectric loss ε_2 with Ln (ω)of MgO:ZnO with different ratios 90/10,80/20,70/30 60/40 and 50/50).

The growing of ε_1 and ε_2 at low frequencies is due to two phenomena: electrode polarization and space charge effects [35]. The long distance travelled by the charge carriers at low frequencies results in high value of dielectric loss ε_2 . The manifestation of small peaks for the composites samples with low ZnO ratio is ascribed to the relaxation phenomena where the dipoles are cable to stratify themselves according to the direction of the applied electric field giving rise to participating in the polarization process. Whereas at high frequencies, the dielectric constant ε_1 and dielectric loss ε_2 reach constant values. As the frequency increases the variation of the applied electric field become more fast that the charge carriers cannot pursue it, the participation of ε_1 and ε_2 to the polarization reach a small value [36,37]. It can be seen from Figure 3 and 4 that the value of ε_1 and ε_2 increased by increasing ZnO up 75% and then fall again; ε_1 increased from 1.23, to 287.68, by increasing ZnO from 0 to 75% and then decreased to 6.24 when ZnO ratio increased to 100 %. This is ascribed to the increase in number of charge carriers with increasing the amount of ZnO ratio. On the other hand, ε_2 increased from 0.717 to 55168 and the decreased to 2.52 when ZnO changed in the mentioned range.

The dielectric loss provided information about the amount of energy dissipated from charge carriers transporting and electrode polarization. The former sources of loss resulted from carriers precipitation at electrodes which are forced to oscillate in the same frequency of the applied field as soon as it was applied giving rise to dipolar relaxation as a peak in the dielectric loss spectra [38]. Stevels[39] explained this, that the increase in dielectric loss as the relaxation phenomenon resulted from many sources of losses like conduction, dipole and vibration. The increase of temperature results in an increase of conduction losses, which gives rise to an increment of the dielectric loss values.

Debye proposed that the relation between log ε_2 versus Ln (ω) forms curves which are symmetrical around angular frequency named ω_D which corresponded to maximum absorption according to the relation $\omega=1/\tau$, where τ is value of relaxation time which is defined as the reciprocal of frequency for jumping from one site to another. It can be seen that the only composites samples with low ZnO ratios i.e. (0, and 25%) declare absorption peaks.

The relaxation time increases but in a non- regular sequence by increasing the MgO ratio, see Table 1. The deduced values of relaxation are 8.58×10^{-6} sec decreased to 2.87×10^{-6} sec by increasing the ZnO ratio from 0 to 25%. The reduction of relaxation time with increasing ZnO is due to prompt movement of charge carriers that are concurrent with the direction of the applied electric field [40,41]



Figure 3: Variation of ε_1 against Ln ((ω) of MgO:ZnO with various ratios



Figure 4: Variation of ε_2 against Ln ((ω) of MgO:ZnO with various ratios

The Cole-Cole drawing (ε_1 against ε_2) is the most important criteria for materials exhibiting one or more relaxation processes with analogous values and following the Cole-Cole formula. The plot equalization of the changing of dielectric loss ε_2 with dielectric storage ε_1 component at ambient temperature are shown in Figure 6. The semicircle figures are a proof of the existence of only one relaxation time. The polarizability can be measured from the relation $\theta = \alpha \frac{\pi}{2}$ where θ is the angle between the semicircle diameter and the dielectric storage axis .As can be seen from Table 1, the values increases as ZnO ratio increases from 0 to 50 % and then reaches a minimum value at 75% ZnO, then rises again for further increase of ZnO. On the other hand, the increase of the semicircle radius by increasing the ZnO ratio gives an indication that the continuous addition of ZnO reduced the relaxation time throughout making the movement of mobile charge carriers faster which is coincided with the conductivity values [42].



Figure 4: The plot of ε_1 versus ε_2 of MgO:ZnO with various ratios

Composites sample	The exponent factor (s)	Polarizability (α)	Relaxation time (τ) (sec)
100%MgO	0.748	0.1112	8.58x10 ⁻⁶
75% MgO: 25%ZnO	0.628	0.4112	2.87x10 ⁻⁶
50% MgO:50%ZnO	0.667	0.4667	-
25% MgO: 75%ZnO	0.540	0.3556	-
100%ZnO	0.948	0.3777	-

Table 1: The values s, α , and τ for MgO:ZnO composites

Conclusions

The structural, and dielectric properties of MgO:ZnO composites prepared using solid state reaction were investigated. Analysis of X-ray diffraction spectra affirms the structural enhancement and increase of crystalline size with the continues addition of ZnO. A reduction of dielectric constant and dielectric loss was noted by increasing the frequency. High dielectric constant value was achieved for the composite with 75 % ZnO which coincide with the high value of the electrical conductivity. The dielectric spectra as function of frequency and maximum peaks loss confirms relaxation in composites where the peak shifts toward the high-frequency side proposed faster charge carrier's movements.

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