Resultant peak dissection by match 3 in XRD of Mg doped ZnO nanoparticles

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Abstract

Pure nanomaterials have limited applications, so to enhance the quality of the material, dopants play an important role in the application. It is important to find out whether the crystal structure of the dopant is rearranged or it preserves its crystal structure even after it is being doped in the pure material. Zinc Oxide has hexagonal structure and MgO has cubic structure. When Mg is doped in ZnO at various temperatures, MgO rearranges itself and it also has hexagonal structure in some cases and in other cases, it preserves or changes its crystal structure other than hexagonal structure represented as secondary peaks.

Match 3 tool has been used for Mg doped ZnO at 500^{O}C wherein MgO has rearranged itself into hexagonal structure and ZnO has rearranged itself into cubic structure. EDAX studies confirm that the synthesized material contains only Zn, O and Mg which confirm that the secondary peaks are due to structural interchange and not due to any other impurities.

Keywords: Match 3, EDAX, XRD, secondary peaks, structural change.

Introduction

Zinc oxide is an n-type material[1] which is available abundantly and at less cost and also shows various interesting properties[7]. It can be used with suitable dopant[9] and the tuning of the properties[6] can be easily done. Due to its tuning property, the zinc oxide may be used in optical, thermal, electrical industries and medical applications.[10] The crystallographic orientation does not affect the physical properties[12] easily but the size of the atom plays an important role in the application. Hence to increase the quality of the crystal and size of the particle, the nature of material should be studied at various temperatures with suitable concentrations of dopant.[2]

Magnesium oxide is chosen due to its wide band gap[3] and the ionic radii match with the zinc oxide[17]. Thus the band gap of zinc oxide can be altered and increased. Many researchers reported that the optical band gap and thermo dynamical studies are done based on the rock salt structure because wurtzite hexagonal ZnO under high pressure takes a phase transition to rock salt structure[19]. When the dopant atom replaces suitably in the place of parent atom position, a single phase behavior will be shown. Else, when secondary phases are present, the material shows the bi-phasic behavior[4] which affects the application of the material. The doping concentration plays an important role when it is greater than 4 atomic%.

In cases where there is only one phase and it represents the parent molecular structure, then it can be understood that the dopant molecule has completely merged with the structure of parent molecule. But if there are secondary peaks, then it is usually understood as improper synthesis method due to which the dopant molecule does not fit properly into the parent molecular structure. In some cases, these secondary phases are not detected due to the low intensity of the XRD peaks which merges with the background noise, which is usually present in XRD pattern of nano powders. Even if there are secondary peaks, due to noise reducing software, the secondary peaks, which have very low intensity are also suppressed and it cannot be detected and these peaks never find a place in the final XRD output[16].

In the case of Mg doped ZnO, Mg usually disrupts the tetragonal structure of ZnO[18] and this phenomena creates oxygen vacancies. Suppose, if there are secondary peaks, it is important to analyze the cause for these secondary peaks and a detailed structural analysis should be done. From the XRD pattern of Mg doped ZnO at 500^{O}C, which we have synthesized, secondary peaks are seen. Hence, it is important to analyze the structural aspects of these secondary peaks, suitable software is needed to conclude the structure of the material. Match 3 software is used to analyze the structure of the synthesized material. The secondary peaks may also be due to impurities present in the sample and hence to confirm, whether any impurities are present in the sample, EDAX analysis is also done.

Material and Methods

Zinc nitrate, magnesium nitrate, citric acid, urea and poly ethanol glycol (Nice Company AR Grade) were purchased and used without further purification.

60 g of zinc nitrate was dissolved in a 75 mL of double distilled water which was mixed with 60 g of citric acid (dissolved in 10 mL of water) and 60 g of urea (dissolved in 10 mL of water) and kept in a magnetic stirrer for 10min. After 10 minutes, 3.5% molecular weight of magnesium nitrate was taken and dissolved in 10 mL of water which was mixed with a zinc nitrate solution. This event was kept in a magnetic stirrer for one hour at a temperature of 80 °C. Then, 10 mL PEG (poly ethylene glycol) was added and again kept in the hot plate for 10 hours at a constant temperature. Later

it was kept in Muffle furnace for 500 °C of temperature up to 3 hours.

Results and Discussion

Structural Analysis (XRD Technique): The synthesized material is characterized using the XRD instrument of scanning mode 2 theta/theta of scanning type continuous scanning X-Ray 40 kV/30 mA and the data is recorded using CuKβ radiation. XRD of Mg doped ZnO Nano particles are shown in the figure 1 in the 2 theta range of30-60°. The spectra show the standard JCPDS peaks[13] at preferential orientation and also secondary peaks at [110][11] and [103][8] planes. Usually, in XRD analysis, it is assumed that the standard peaks represent the wurtzite hexagonal structure of ZnO and secondary peaks may be due to the MgO sub lattice which is cubic phase in nature[14]. Due to this, it is assumed that if XRD analysis shows secondary peaks, it is a sign of improper synthesis and the dopant molecule and the parent molecule does not react and the dopant molecules remain seperately and the parent molecules remain seperately.

As an additional phenomenon, nano powders have background noise and if the intensity is low, it gets undetected if noise reducing software are used to supress the noise. In the present case, clear secondary peaks are seen in the XRD spectra even when the sample was synthesized at 500^{O}C which is high temperature. Hence, Match 3 software is used to analyze these secondary peaks.

XRD Match 3 Software: The XRD Match 3 software is used to confirm the phase structure of the parent and the dopant atom and also to study the lattice parameters.

The Match 3 report for the temperature 500 °C is tabulated in the table 1 and the spectra is shown in the figure 2. The Match 3 spectra show that there is a interchange of phase structure of parent (ZnO which is hexagonal) and dopant (MgO which is cubic) in nature. Hence the presence of secondary phase is due to the structural interchange between the parent and the dopant molecules and not due to the improper synthesis of the sample. It can be seen that under high pressure, ZnO which has hexogonal structure changes its structure to cubic structure[5], but in the present case, no pressure has been applied but there is a structural change due to the method of synthesis and the percentage of dopant. Further, MgO, which has a cubic structure, has changed its structure to hexagonal which has been reported here.

Compositional Analysis: The compositional analysis is carried out by EDAX to study the presence of the atoms in the synthesized Mg doped ZnO sample. Figure 3 shows the EDAX spectra for synthesized ZnO at the temperature 500^{o}C. The spectra show the presence of the constituents zinc (Zn), oxygen (O) and magnesium in the sample. It does not show any impurity atom, hence the synthesized sample is pure in nature and it reveals that secondary peaks are not due to any impurity of atom. The elemental percentage of Zn, Mg and O elements present in the sample is tabulated in table 2.

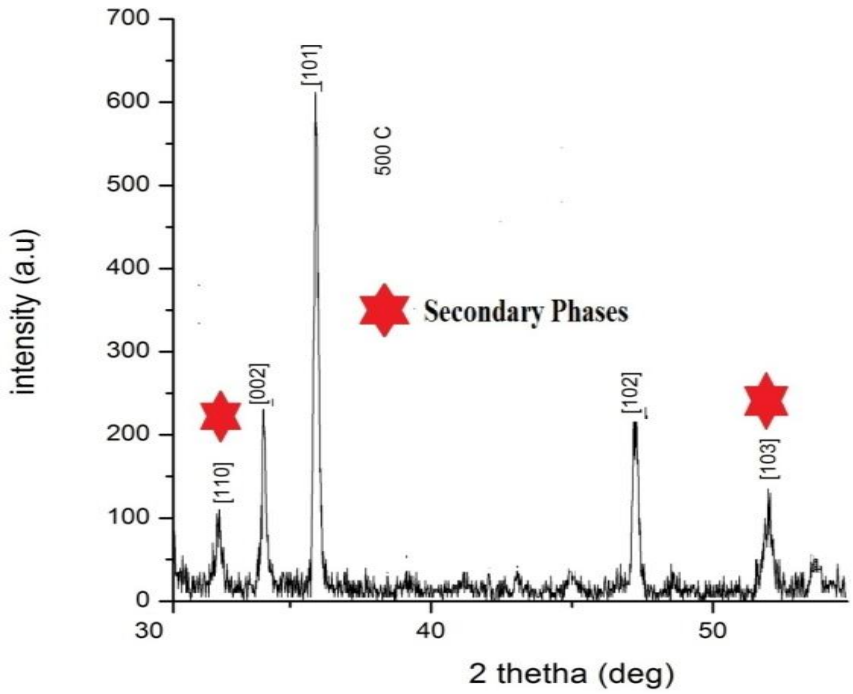

Figure 1: XRD spectra of Mg doped ZnO at 500 °C

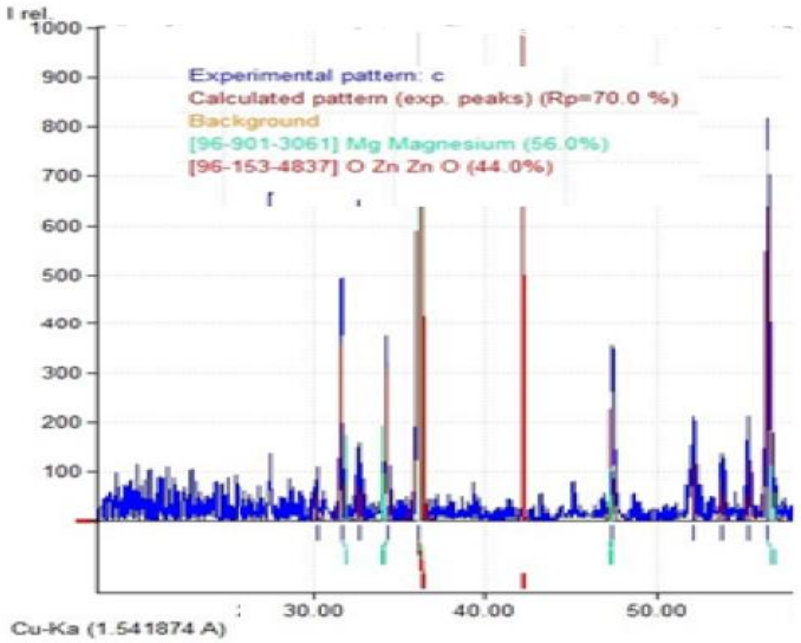

Figure 2: XRD match 3 software spectra for the temperature 500º C

Table 1
XRD Match 3 report of Mg doped ZnO at 500 °C

Formula sum	**Mg (56.0 %)**	**ZnO (44.0%)**
Entry number	96-901-3061	96-153-4837
Figure-of-Merit	0.796248	0.369
Total number of peaks	32	20
Peaks in range	26	10
Peaks matched	16	3
Intensity scale factor	0.72	1.00
Space group	P 63/m mc	F m-3 m
Crystal system	**Hexagonal**	**Cubic**
Unit cell	a= 3.2425 Å, c=5.2663Å	a= 4.28 Å
Calc.density	1.684 g/cm³	6.895 g/cm³

Table 2
The atomic percentage for Mg doped ZnO at the temperature 500º C

Element	**Atomic % 500ºC**
Zn	35.59
O	60.26
Mg	4.15

Conclusion

The synthesized Mg doped ZnO at the temperature 500ºC shows secondary phases in XRD spectra, which usually is a sign of improper synthesis where it shows that there is no interaction between the dopant and the parent molecule. But, in the present case, Match 3 XRD software is used and it reveals that the secondary peaks are due to ZnO which has changed its structure from hexogonal to cubic and the MgO, which usually has a cubic structure, has changed its structure to hexagonal structure. EDAX studies also show that the sample is free from any other impurities and the secondary peaks are only due to the parent molecule which is ZnO in this case and not due to improper synthesis of the sample or due to the dopant molecule.

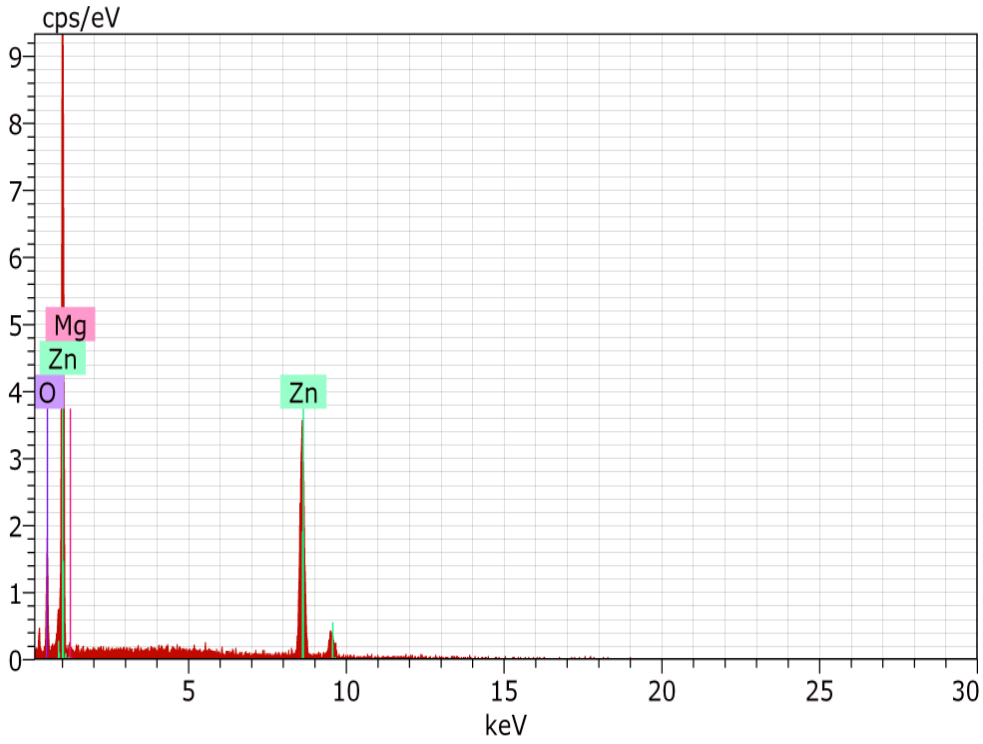

Figure 3: EDAX pattern at 500ºC

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