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CHARACTERISATION OF MgO PRODUCED BY COMBUSTION SYNTHESIS METHOD DOPED WITH Dy^{3+}

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ABSTRACT

The objective of this study is to explore the possibility of synthesizing MgO: Dy^{3+} by using combustion synthesis method. Magnesium oxide powders doped with Dy have been prepared by this method and synthesisation occurs. The average particle size, d spacing and intensity are estimated from XRD analysis. The morphology and structure was analyzed by scanning electron microscopy. EDX was used for the elemental analysis of the sample.

KEYWORDS: Combustion Synthesis, XRD, SEM, EDX, Nanoparticles

INTRODUCTION

Over the past few decades many advances have been made in the area of preparation of nanomaterials. Nanotechnology has made nanocrystalline materials become an area of intense research activity [1–4]. Nanocrystalline materials are polycrystal- line materials with grain size below 100 nm [5, 6]. The change in the crystalline size and shape will alter the properties, which were formerly thought to be constant for a given material. Nanocrystals of common metal oxides have been shown to be highly efficient and active adsorbents for many toxic chemicals, including air pollutants, and chemical warfare agents [7]. Magnesium oxide (MgO, periclase), as an exceptionally important material for using in catalysis [8,9], toxic waste remediation, or as additives in refractory, paint, and superconductor products [10] has been attracting both fundamental and application studies [11]. Many different synthetic routes provide nanoscale MgO including sol–gel [12], hydrothermal/solvothermal [13,14], laser vaporization [15], chemical gas phase deposition [16], aqueous wet chemical [17], surfactant methods [18], polyol-mediated thermolysis process [19], and microwave-assisted method [20].

In recent years metal and semiconductor received considerable attention as active components in wide variety of research and technological application due to their optical, electric and magnetic properties compare to the bulk modular parts[21,23].Magnesium oxide is an interesting basic oxide that has many application in catalysis, absorption and synthesis of refectory ceramics [24,27]. MgO is a wide band gap insulator (7.8ev) with rock salt crystal structure (fcc) at ambient pressure, the Mg ions occupying octahydral sites in anion closed packed structure ([28,29]. Dy $^{3+}$ ions are well known activator dopants for many different inorganic lattice producing white light emission by suitably adjusting yellow and blue emission[30].Although the PL such as borate, niobate and phosphate has drawn attention[31,32].

MgO: $Dy3^{+}$ has commonly being prepared using combustion syntheses method at temperature of 550° C. This method is one of the best method because it is relatively simple,efficient,low cost and time consuming method. The scope of this work is to analyze crystalline nature, spectrum and atomic percentage of sample.

Experimental Procedure

The starting raw materials are magnesium nitrate [$Mg(NO_3)_2.6H_2O$] urea[NH_2CONH_2]and dysprosium nitrate. These raw materials were firstly weighted first and were taken in mortar pistal and mixed it properly for one hour. After mixing, these materials are placed in crucible was then introduced into muffle furnace at 550˚C for 20 min as the ignition occurs the reaction occurs vigorously for few seconds and the fluffy substance was obtained. Based on mass ratio of the experiment the overall reaction equation could be expressed as follows:

$$6[Mg\ (NO_3)_2.6H_2O] + 6[Dy(NO_3)_3.6H_2O] + 28[NH_2CONH_2] \rightarrow 6MgO{:}Dy + 43N_2 + 28CO_2$$

XRD analysis of prepared sample was done using PAN analytical Xpert diffractometer.The surface morphology was analysed by using Zeiss.Evo 18 special edition.The elemental analysis of the sample was analysed by EDX

.P hotoluminescence spectra were investigated.

RESULTS AND DISCUSSIONS XRD

The crystalline structure of material was analysed by PAN analytical X-pert diffraction with Cu-kα radiation (λ=1.54060 A˚ or 0.154nm).The synthesized sample was observed by moving radiation detector with scan speed of 2˚/min at the range of 10˚-80˚ where monochromatic wavelength of 1.54 A˚ (Cu-kα) was used. The XRD patterns shows very broad peaks. The broadening of the diffraction peaks of the samples indicates that the particle sizes are in the nano-scale range.

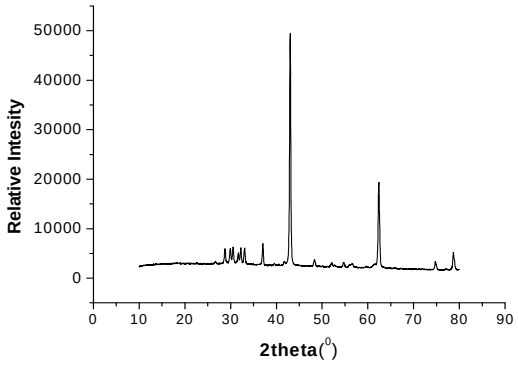

Figure 1: Powder XRD PATTERNS of MgO and Dy^{3+}

Graph and D Spacing

Indexing process of powder is done, with the help of miller indices the value of hkl is being calculated.Following is the detail. There were number of braggs reflection can be seen with respect to(111),(200),(220) reflection.Peak value indexing from d spacing

Table 1

2θ	d(A˚)	$1000/d^2$	$(1000/d^2)$/C.F	HKL
36.9545	2.43052	169.280	2.980	111
42.9718	2.10308	226.090	3.980	200
62.3481	1.48827	451.497	7.497	220

Particle Size

The average grain size of magnesium oxide doped with dysprosium nanoparticles is determined using Debye Scherrer formula.

$$D= K \lambda/\beta\cos\theta \quad (1)$$

$$D= 0.9\lambda/\beta\cos\theta \quad (2)$$

Where K=0.9, λ=wave length of X-Ray (0.1541 nm), β= FWHM (full width at half maximum),Θ= the diffraction angle and 'D' is particle diameter size. The value of d which is known as interplaner between the atoms can be calculated by braggs law i.e.

$$2d \sin\theta=n\lambda \quad (3)$$

The average particle size is calculated in the below table i.e 20nm.

Table 2

2θ	HKL	FWHM β(rad)	Size of Particle D(nm)	D Spacing (nm)
36.9545	111	0.05761	10nm	0.243052
42.9718	200	0.02608	23 nm	0.210308
62.3481	220	0.05761	28 nm	0.148827

Intensity of XRD Peaks

The maximum intensity of experimental MgO doped Dy powder XRD is 100% for(200).Peak intensity are shown in the below table

Table 3

HKL	111	200	220
2θ of peak	36.9545	42.9718	62.3401
Height(cts)	2120.29	23904.14	10226.46
Relative intensity (%)	8.87	100	42.78

SEM

The SEM image is carried out by using Zeiss. Evo 18 Special Edition in order to analyse the structure and morphology of doped samples. SEM was used for the morphological study of MgO

Doped with Dy. The instrument was accelerated at voltage of 10 Kv and the samples were scanned at a working distance of 8.5 mm. The SEM images for the MgO doped with dysprosium samples are shown in Fig. 2, Fig 3, Fig 4, respectively.

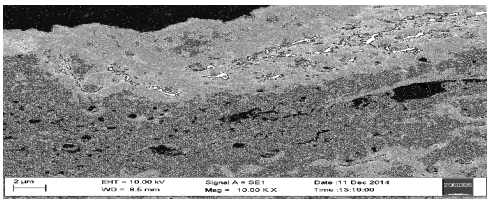

Figure 2: The SEM Image of MgO Doped Dy for 2 µm

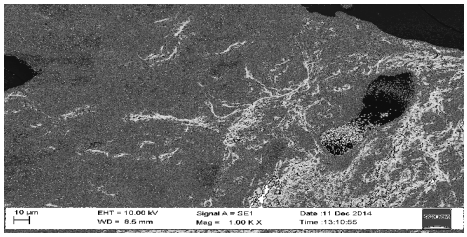

Figure 3: The SEM Image of MgO Doped Dy for 10 µm

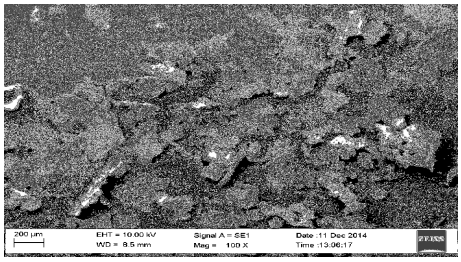

Figure 4: The SEM Image of MgO Doped Dy for 100 µm

EDX Analysis

The spectrum obtained by EDX samples is shown in Fig. 5 (a). From the sample spectrum 100% of Mg metal was observed in the sample corresponding to peak shown in the Fig. 5 (b). In sample the inclusion of Dy $^{3+}$ is shown in the corresponding peaks. From the data it is observed that the synthesized sample contains about Mg, O and Dy with 55.71%, 44.32% and 0.87% of atomic percentage respectively which agrees with expected value. From the element count percentage, 55.71% of Mg and 0.87% of Dy have been observed.

Table 4

Element	Weight%	Atomic%
O	42.69	55.71
Mg	50.56	44.32
Dy	6.75	0.87

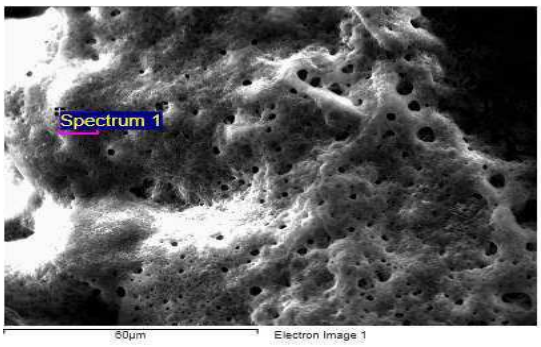

Figure 5 (a): The EDX Image of MgO Doped Dy

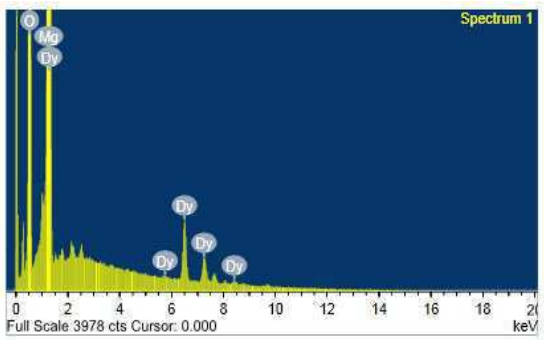

Figure 5 (b): The EDX Image of MgO Doped Dy

CONCLUSIONS

In summary, magnesium oxides doped with Dy have been synthesized successfully by combustion syntheses method. The crystalline structure of material was analysed by PAN analytical X-pert diffractometer. A morphology index based on FWHM of XRD data have been developed. The average grain size of magnesium oxide doped with dysprosiun nanoparticles is determined 20nm. The SEM image is carried out by using Zeiss. Evo 18 Special Edition in order to analyse the structure and morphology of doped samples. The spectrum obtained by EDX samples is 100% for Mg metals and PL property of sample was determined.

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