

EFFECT OF PARTICLE SIZE ON THE ENERGY BANDS OF MgO SYNTHESIZED BY THE COMBUSTION METHOD

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Nanostructured materials usually possess characteristics different from conventional micron sized particles. The effect of particle size on the energy bands of MgO synthesized by the combustion method is the object of this study. Magnesium oxide in this work is synthesized using a combustion method. The samples obtained are annealed at two different temperatures which are 600 °C and 900 °C for 24 hours producing 2 different ranges of particle size. The phase and purity of samples are studied using X-Ray diffraction (XRD). The morphology and particle size of the magnesium oxide samples are examined by a field emission scanning electron microscope (FESEM). UV-Vis NIR spectrophotometer is used to determine the band energies of the materials. Results showed that the particle size of MgO nanomaterials affect their band energies.

Keywords: MgO, Combustion, Band energies

INTRODUCTION

MgO is one of the metal oxides that have been widely used for industrial applications such as in pharmaceuticals [1], catalysis [2], superconducting materials [3], reflecting and antireflecting coatings [4] toxic waste remediation [5], etc. It has unique and interesting properties including electrical insulation, chemical inertness, high temperature stability, optical transparency and secondary-electron emission capabilities [6-7]. Recently, many studies have been focused on the synthesis of MgO nanostructures [1-5]. There are many ways for producing MgO including sol-gel, combustion, hydrothermal, chemical gas phase deposition and flame spray pyrolysis [8-11]. The route of synthesis of nanomaterials can affect the properties of materials such as the size and shape of their particles [6- 12]. Selvamani *et.al* [10], used hydrothermal methods to get rectangular MgO sheets while Niu *et.al* [13] reported that coral-like MgO nanoparticles have been obtained using single-step self-assembly method. Kumar *et. al.* [12] synthesized nano MgO powders using the sol-gel method and they have succeeded in producing parallelepiped, sphere, rod and rectangular shapes of MgO crystal. Wang *et.al* also used sol-gel method [8] and obtained the needle shape and cubic structure of nanocrystalline MgO. Rao *et. al.* [9] used a combustion method and they produced

spherical nanostructured MgO. In this paper, we report the morphology and particle size of MgO materials synthesized by the combustion method but using a different route from Rao.

METHOD AND MATERIALS

In this study, MgO nanomaterials were prepared using a combustion method. Magnesium nitrate hexahydrate, $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was used as a starting material. The materials used were according to stoichiometric calculations. This starting material was dissolved in deionised water followed by addition of triethanolamine. The mixture was slow-heated until combustion occurs. Then, an annealing process at two different temperatures of 600 °C and 900 °C for 24 hours were done. The samples were named as M1 and M2 respectively. The phase identification was carried out using an X-Ray Diffractometer, the Pan Analytical X'Pert Pro System. The morphology and particle size of the samples were examined by Field Emission Scanning Electron Microscope (FESEM), (JEOL JSM-7600F). The band energies of the samples were measured in the wavelength range of 190 nm to 500 nm by using a UV-Vis NIR spectrophotometer (Perkin Elmer Lambda 950 UV-Vis Spectrophotometer).

RESULTS AND DISCUSSIONS

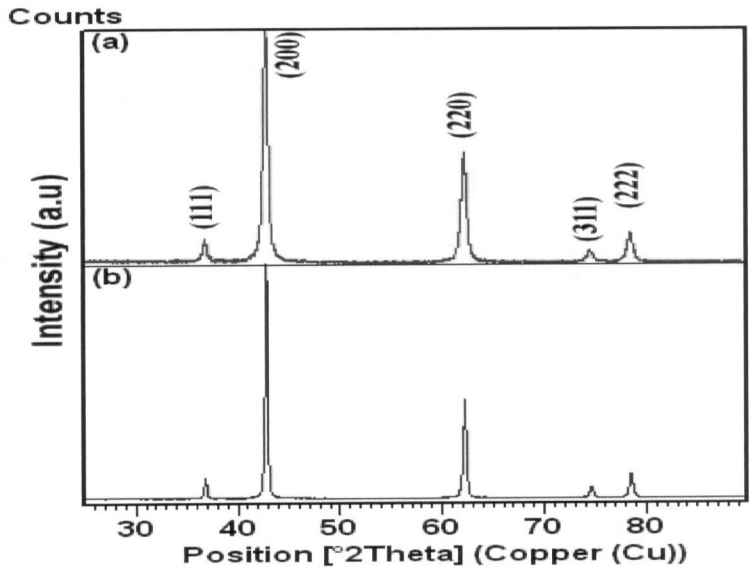


Fig. 1. XRD patterns of (a) M1 (b) M2 samples

Fig. 1a and 1b show the X-Ray diffraction patterns of M1 and M2 samples. The XRD patterns are matched to the MgO cubic structure, ICDD reference pattern number 01-087-0651 with space group Fm-3m. Both samples are pure with no impurities or other phases present. Observations of the XRD patterns show that the M1 sample has

broadened peaks implying that sample M1 has very small crystal size compared to M2 sample. On examination of the (200) peak, the FWHM of the M1 and M2 samples is 0.54 and 0.29 respectively. This implies that the crystallite size of M1 is smaller than that of M2 sample which is confirmed by the SEM results.

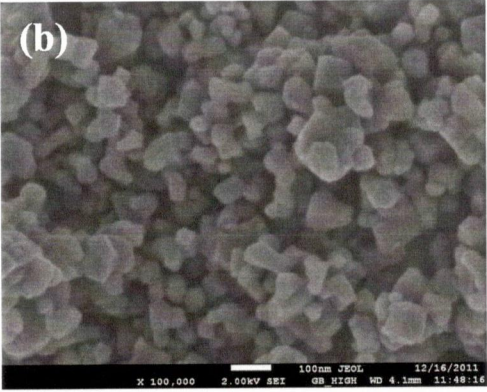
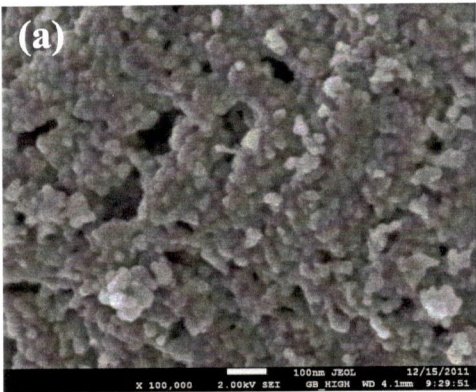


Fig. 2. FESEM images of (a) M1 (b) M2 samples

Fig. 2a and 2b show the SEM micrographs of M1 and M2 samples at a high magnification of 100k times. The images showed that both samples have mostly rounded polyhedral type of crystal. As observed in the highly magnified images, the crystallite size of M1 sample is much smaller than M2 sample. The crystallite size of M1 sample is between 20 nm to 60 nm while M2 sample has

crystallite size between 40 nm to 150 nm. The SEM results confirm that of the XRD data earlier. This finding showed that the higher annealing temperature caused the crystal growth to occur faster since the crystallite size of the highest annealing temperature is largest. Thus, higher annealing temperature seems to affect the rate of crystal growth.

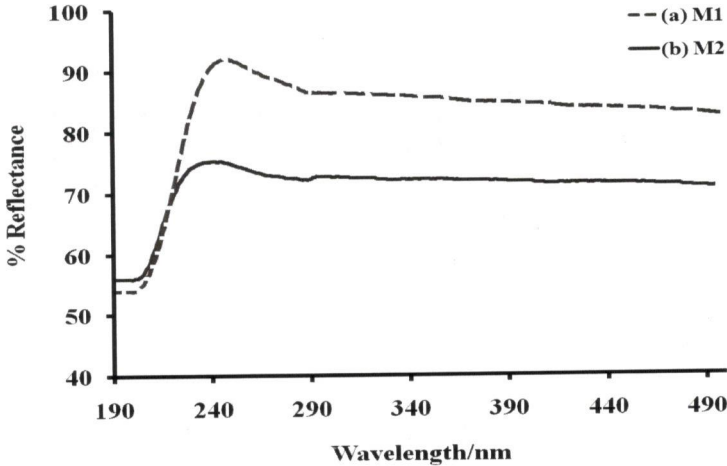


Fig. 3. Reflectance spectra of (a) M1 and (b) M2 samples

Fig. 3 shows the reflectance spectra of the samples. The reflectance spectra for both samples were observed in the wavelength range of between 190 to 500 nm. As observed, the M1 sample has higher reflectance compared to that of M2 sample. From this reflectance curve, it can be seen that the M1 sample was found to be shifted to the higher wavelength of M2 sample. The absorption edge for

the M1 sample was identified at 237 nm which is higher than M2 sample which is at 227 nm. From these results, we can say that the M1 sample has a lower band energy compared to that of the M2 sample. The band energies was evaluated using Tauc plots (Fig. 4) according to the details given by Rusdi *et. al.* [14].

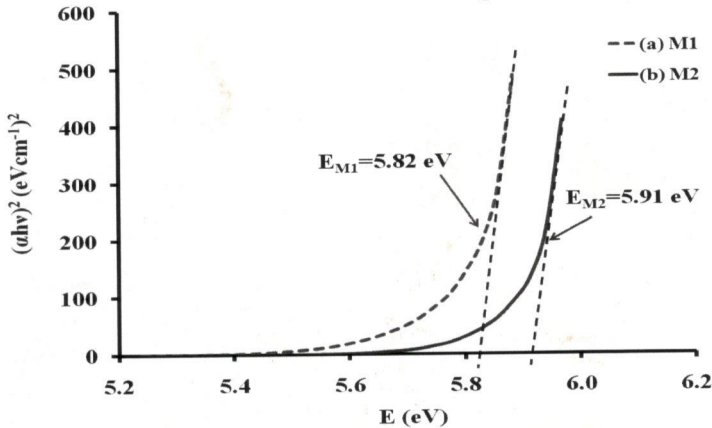


Fig. 4. Tauc plot for (a) M1 and (b) M2 samples

The band energies obtained are 5.82 eV for the M1 sample and 5.91 eV for M2 sample. It showed that the MgO sample with smaller crystallite size has lower band energy. The band energy value obtained agrees with Kumar *et. al.* [12] who obtained band energies of nano sized MgO of about 6.2 eV, that is, less than the conventional value of 7.8 eV [15]. This can be explained by the extremely small crystal size of the MgO nanopowders. They are between 20 to 150 nm. This difference in band energies from bulk powder values can be explained by the increased surface area to volume ratio of the nano size MgO samples.

CONCLUSION

Pure and single phase materials of nanostructured MgO are obtained by a simple and cost-effective combustion method. MgO with bigger crystallite size has higher band energies compared to that of smaller crystallite size. The band energies obtained (5.82 eV, 5.91 eV) for the nanoparticles are smaller than the band energy of bulk MgO (7.8 eV).

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