ISSN: 0975-8585

Research Journal of Pharmaceutical, Biological and Chemical Sciences

Structural, Morphological, Optical and Sensing Studies of MgO Thin Film.

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ABSTRACT

MgO thin film was deposited on to the preheated substrate at 230°C using home built spray pyrolysis technique. The precursor solution of magnesium chloride hexahydrate with 0.05M concentration was sprayed as fine mist under optimized condition to achieve spherical and rod type nanoparticles which is in nanorange.XRD studies confirms the polycrystalline nature of the film. UV visible studies shows that, maximum energy absorbed in lower wavelength region (λ_{max}=331.94) results a wideband gap material. The field emission scanning electron micrograph of the surface indicates sphere like particles and rod like particles are formed.The sensing response of MgO film towards ammonia vapour was studied at room temperature (≈30°C) and the results are reported.
Keywords: MgO thin film; Spray pyrolysis; Polycrystalline; Ammonia;

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ISSN: 0975-8585

INTRODUCTION

MgO thin films were widely used in various fields for different applications. Recently it was reported as protecting layer of dielectric in ac plasma display panel to improve the life time and discharge characteristics [1]. MgO is a highly dielectric solid with NaCl structure [2] and has low chemical reactivity hence widely used as a buffer layer for high temperature superconducting films and ferroelectric films [2, 3]. MgO has interesting properties such as high thermal stability, wide band gap, low optical loss and low refractive index [4]. There are many method to prepare MgO film such as Metal organic chemical vapour deposition (MOCVD), Sputtering technique, Pulsed laser deposition (PLD) but recent interest to grow metal oxide film through spray pyrolysis became interest, because it can able to yield high uniformity and high purity film in open atmosphere [4].In this study, high quality MgO thin film have been successfully deposited by the home built spray pyrolysis, and the characteristics of obtained MgO film was studied by XRD, FE-SEM and UV visible spectrometer..The sensing response of MgO film towards ammonia vapour was studied at room temperature (≈30°C) and the results are reported.

EXPERIMENTAL

The schematic diagram of home built spray pyrolysis is illustrated in Fig 1. This set up comprised of spray gun, heater, thermostat with thermocouple and exhaust fan. The 0.05M magnesium chloride hexahydrate was taken as a precursor salt and it completely dissolved in 50ml demineralised water. The precursor solution was then sprayed as a fine mist on to the substrate. Before that the substrate was cleaned with deionised water and heated at 230^{o}C. The temperature was maintained using a microcontroller-supported thermostat and a K-type thermocouple with an accuracy of ±1°C. The spray time and successive spray intervals were fixed at 5 s and 75 s, respectively, to avoid any sudden cooling of the substrate. Then the aqueous solution was sprayed at the flow rate of 3ml per minute. The droplet from the nozzle was carried by the compressed dry air to the substrate. The size of the each droplet depends upon the flow rate and diameter of inner nozzle. The optimized parameter is given in the table 1.

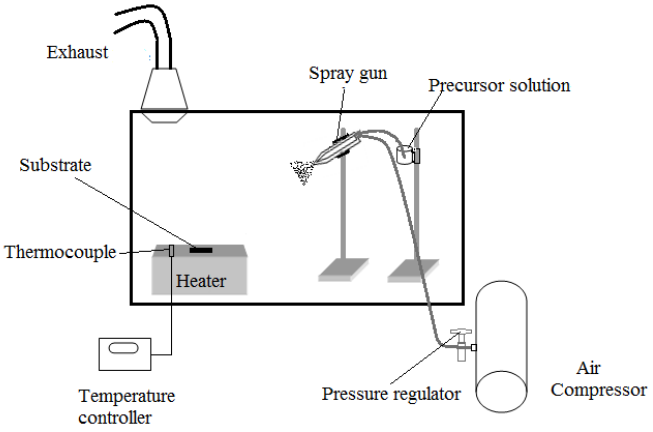

Figure 1: Schematic diagram of home built spray pyrolysis setup

ISSN: 0975-8585

Table 1: optimized deposition parameter of MgO thin film

Distance between spray gun & nozzle	40cm
Compressed dry air pressure	2kg/cm^2
Angle between spray nozzle and substrate	45^o
Substrate temperature	230^{o}C
Spray rate	3ml/min

The deposited film was then characterized without annealing by X-ray powder diffractrometer (XRD, D8 Focus, Bruker, Germany) with CuKα radiation wavelength of 1.5406 Å for the structural studies. The surface morphology of the film were obtained from JEOL-6701 Field Emission Scanning Electron Microscopy (FE-SEM), the optical studies were carried out through UV Visible spectrophotometer (Perkin Elmer Lambda 25). The vapour sensing properties of the film have been studied using home-constructed test chamber of 1.5L capacity. Before doing the sensing studies, the film was conditioned at 300°C for 24 hours to remove undesirable pre-adsorbed organic and water molecules. Ohmic electrical contacts were made on the film (12mmX 10mm) using thin copper wire and silvered passed .The change in electrical resistance of the film was recorded using Pico-ammeter, during the processes of injection and venting of ammonia vapour.

RESULT AND DISCUSSION

Structural, morphological and optical characterization

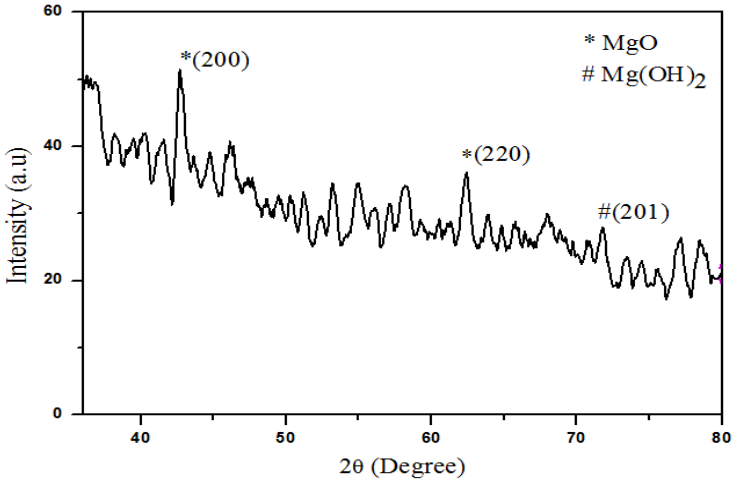

Figure 2: X – ray diffraction pattern of MgO thin film

Fig 2 depicts the x-ray diffraction pattern of MgO film. This pattern revealed three peaks as (200), (220) and (311) plane which indicated polycrystalline nature of the film with respect to standard JCPDS (86-0753). The deposited MgO film was crystalline phases with (200) preferred orientation and the average crystallite size was found to be 14nm as obtained from well known Scherrer formula.

ISSN: 0975-8585

$$D = \frac{S\lambda}{\beta \cos\theta} \text{ ---------- (1)}$$

Where S is the shape factor (0.9), λ is the wavelength of X- rays, β is the Full width half maxima (°), θis the diffraction angle (°). Due to the influence of substrate temperature high crystallinity film was obtained. In addition to MgO peak Mg $(OH)_{2peak}$ of (201) plane was observed. This may be due to insufficient annealing temperature. The lattice constant was calculated using the relation formula $\alpha = \frac{\lambda}{\sqrt{3}\sin\theta}$ and found to be α=2.445A° and dislocation density 3.2×10^{15} (lines/m^2) determined by $\delta = \frac{1}{D^2}$.

The FE-SEM image of the MgO thin film obtained from 50 ml precursor solution for three different magnifications is shown in the Fig 3. Large number of sphere like particles and rod like particles were seen on the film surface. The different shape occurred mainly due to substrate temperature, distance between the nozzle and substrate and the chosen concentration of precursor solution. Fig 3, (a,b,c) shows porous in the film so it reduce the film density. As the impurity in the film the irregular aggregation of nano particles were found around the rod and sphere particles. The size of the particles was found to be 22 nm and agrees with X-ray diffraction pattern .

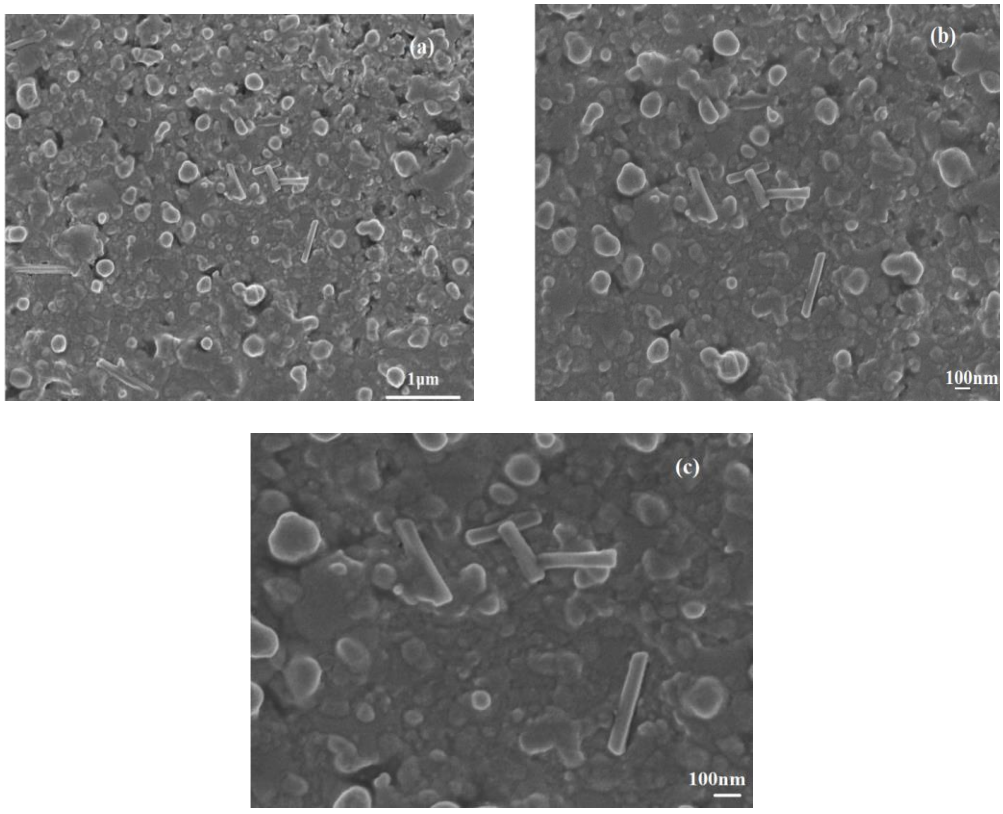

Figure 3: Field-emission scanning electron micrograph of MgO thin film at different magnification (a) 20,000 (b) 30,000(c) 40,000

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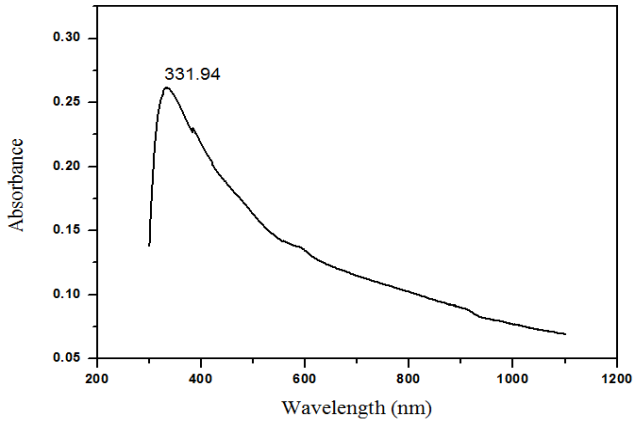

Figure 4: Absorption spectrum of MgO film.

Fig 4 represents the UV-visible absorption spectrum of MgO film. This spectrum was recorded at the room temperature in the wavelength range from 200 to 1200 nm. To investigate the optical property of thin film is important for many industrial application The absorption peak obtained at near ultra violet region in 331.9 nm which posses maximum absorption in lower wavelength. Due to wide band gap, maximum energy absorbed in lower wavelength region.

Ammonia sensing studies

The sensing studies of MgO thin film was taken at the room temperature. The sensing set up consists of digital picometer , glass chamber, digital voltmeter ,vacuum pump, probe and copper wire. For the sensing studies , calibrated volume of liquid ammonia vapour(25%) was introduced into the test chamber and the concentration of the generated vapour was obtained using the following relation(equation 2).

$$C_{(ppm)} = \frac{\delta \times v_{\Gamma} \times R \times T}{M \times P_b \times v_b} \text{----------(2)}$$

Where C is the concentration of liquid ammonia(ppm), δ is the density of ammonia (g/mL), V_{Γ} is the injected ammonia volume (μL), R is the universal gas constant(8.3145J/mol K), T is the absolute temperature(K), M is the molecular weight, P_b is chamber pressure (atm) and V_b is the volume of the chamber (L). The ammonia vapours response mechanism under ambient condition as follows: At low temperatures, oxygen molecules will be adsorbed as molecular oxygen ionsO_2^- on the film surface as indicated in equation (3). [5]. These ions form a depletion layer over the surface of the MgO film, resulting in a high electrical resistance

$$O_2 + e^- \rightarrow O_{2(ads)^-} \text{----------(3)}$$

When the film is exposed to ammonia, nanocrystallites of MgO present on the surface of the film act as a catalyst for the exothermic reaction between the adsorbed oxygen ions and

ISSN: 0975-8585

ammonia. Thus, the adsorbed oxygen ions are desorbed and the trapped electrons are released into the film. This process reduces the surface electrical resistance of the MgO film and leads to the detection of ammonia. This process can be expressed as indicated in equation (4)

$$4NH_3 + 3O_{2(ads)^-} \rightarrow 2N_2 \uparrow + 6H_2O \uparrow + 3e^- \text{ ----------(4)}$$

The resistance of the film was observed from fixed base resistance to saturation resistance after injecting the ammonia vapour in different concentration 5ppm, 10ppm, 15ppm, 20ppm, 25ppm. The response percentage of ammonia vapour was calculated by equation (5).

$$S = \frac{R_o - R_g}{R_g} \times 100 \text{ ----------(5)}$$

Where R_g and R_0 are saturated and base resistance of the film.

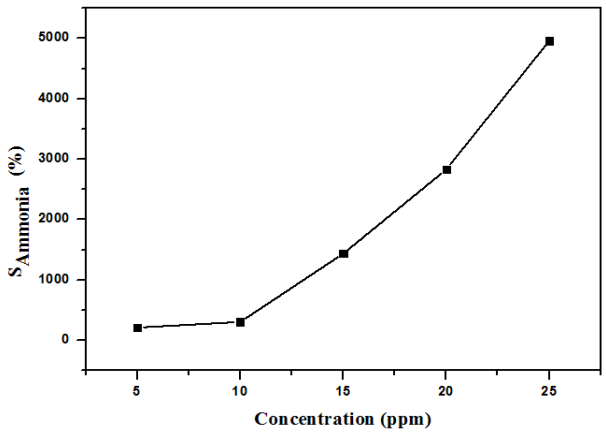

Figure 5: Sensing response variation of MgO film for different ammonia concentration

Fig 5 shows the response percentage of ammonia vapour for 5 different concentration Response is below 500% for low concentration 5ppm and 10ppm then the response was increased 5000% for higher concentration 25ppm so that the film has good response for ammonia vapour. MgO is a highly dielectric material and the resistance obtained in the range of Tera ohm which was depicted in Fig 6. R_0 is a film resistance after injecting the ammonia vapour resistivity decreased with increasing concentration. The ammonia vapour molecule adsorbs on the surface of the film leads to reduce the resistivity of the film. The increase in response is ascribed to the fact that as the ammonia concentration increases, more electrons are liberated resulting in decrease in electrical resistance as shown in figure 6.

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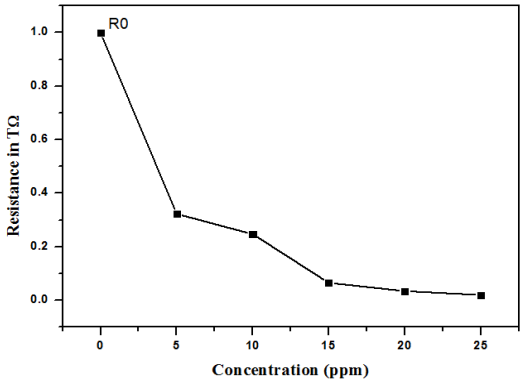

Figure 6:.Variation in electrical resistance of the film as a function of ammonia concentration.

CONCLUSION

The (200) preferred orientation of MgO thin film was deposited on the silicon substrate using home built spray pyrolysis technique and they are polycrystalline in nature. The optimum substrate temperature leads to form spherical and rod shape particles. The appreciable change in resistance observed at ammonia concentration indicates the good sensing behavior of the nanocrystallites MgO film. The observed result suggest that MgO thin films can be utilized to sense ammonia vapours at room temperature for environmental monitoring.

ACKNOWLEDGMENT

The authors wish to express their thanks to SASTRA university for providing infrastructure facilities.

REFERENCES

[1] Soo Gil Kim, Kook Choi, Jae Hwan EunHyeongJoon Kim, CheolSeung. Thin Solid Film 2000; 377-378: 694-698
[2] S Kurtaran, I Akyuz, F Atay. App Surf Sci 2013; 265: 709– 713
[3] Soo Gil Kim, Jin Yong Kim, HyeongJoon Kim. Thin Solid Film 2000;376: 110-114
[4] Ji Ming Bian, Xiao Min Li, Tong Lai Chen, Xiang Dong Gao, Wei Dong Yu. Applied Surface Science 2004; 228: 297–301
[5] R Pandeeswari, RK Karn, BG Jeyaprakash. Sensors and Actuators B: Chemical 2014; 194: 470– 477
[6] Dae Jong Seo, Seung Bin Park, Yun Chan Kang and Kwang Leong Choy. J Nano Particle Rea 2003; 5: 199-210
[7] Moon Hee Lee and Dong Gon Park. Bull Korean Chem Soc 2003; 24: 1437-1443
[8] D Pavlopoulos, Shadi Al Khatiab, TW Button, JS Abell. J Physics 2008; 97: 012098.
[9] Soo Gil Kim and HyeongJoon Kim. J Korean Physical Soc 1999; 35: 180-183
[10] A MosesEzhilRaj, V BenaJothy, C Ravidhas, T Somd, M Jayachandran, C Sanjeeviraja. Rad Physics and Chem 2009;78: 914–921
[11] Xiaorong Fu, Guangming Wu, Shigeng Song, Zhitang Song,Xinzhong Duo, Chenglu Lin. Applied Surface Science 1999; 148: 223–228.