Influence of Heat Treatment on the Properties of MgO Thin Films as Dielectric Layer

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Abstract- **Physical properties such as high melting point, low heat capacity and high bonding strength of magnesium oxide, MgO could be get give an advantage to MgO to be used as the dielectric layer. Besides that, MgO also has large band gap (7.8 eV) and high dielectric constant (9.8) could be potentially used as a buffer layer for ferroelectric material. In this work, MgO with 0.2M concentration were prepared using sol-gel technique. Magnesium acetate tetrahydrate, ethanol and nitric acid used as precursor, solvent and stabilizer respectively. The MgO solutions were then deposited on the cleaned glass by using spin-coating technique. The thin film deposition is repeated for 8 times for uniformity purpose. The electrical and dielectric properties of MgO are identified by observing its resistivity, dielectric constant, dielectric loss and its surface morphology in terms of annealing temperature. Since high deposition temperature is needed in order to obtain high quality of MgO films and also due to the limitation of glass, the annealing temperature is varied from 400°C to 500°C with 25°C interval. The experimental results show that annealing temperature is inversely proportional to the thickness of MgO films due to the merging activity of the particle. MgO film annealed at 500ºC has been chosen as a good dielectric layer since it has a good electrical, dielectric and structural properties.**

Keywords- heat treatment; annealing; MgO; dielectric; sonication

I. INTRODUCTION

Currently, the size of electronic devices is continuously decreased while the performance is improved. Scaling down the device size however, resulted in used of existing thin dielectric layer (SiO_2) which will lead to high leakage current. Hence, material with high dielectric constant such as magnesium oxide, MgO is used to replace SiO_2 [1-3].

MgO with the NaCl- type cubic structure is a good insulator material due to its properties such as it has high dielectric constant (9.8), wide band gap (7.8eV) and higher breakdown field (12MV/cm) [4]. Moreover, MgO also can be used as a template for ferroelectric material where ferroelectric characteristic will improve the capacitance value [5, 6].

Annealing temperature is the most profound parameter that will affect the thin films properties. Moreover, annealing temperature value is directly proportional to the diffusion rate that will enhance the formation of material structure and alter the thin films properties. Annealing is the process to change the thin films properties such as strength and hardness by applied temperature. Therefore, this research work is focus on the effect of annealing temperature to the dielectric properties of MgO.

II. EXPERIMENTAL PROCEDURE

MgO solution was prepared by using magnesium acetate tetrahydrate (CH_3COO_2) Mg*4H_2, ethanol and nitric acid as precursor, solvent and stabilizer respectively. Then the MgO solution was sonicated at 50°C for 20 minutes and followed by heating, stirring and ageing process.

The thin films were deposited on the cleaned glass substrate by using sol-gel spin coating. The glass substrate was cleaned by using acetone, ethanol and deionized water to remove all residual on the substrate. The rotation speed and time was set to 3200 rpm and 30 second respectively. The MgO thin films were then heated at 200°C for 5min and these process was repeated for 8 times. Finally the thin films were annealed at different temperatures such as 400, 425, 450, 475, and 500 ºC for 1 hour respectively.

Au/ MgO/ glass have been used as the test structure (fig.1) for the electrical characterization in this research. Electrical characterization was conducted using point probes I-V measurement (BUKOH KEIKI-EP2000) to determine the resistivity of the films. Gold with thickness of 60 nm was used as the metal contact to the films since it has a good contact to several materials. Impedance spectroscopy (Solartron 1260A-1296) was used to investigate the dielectric properties of prepared MgO films while atomic force microscopy, AFM (XE-100 Park System) was used to observe the surface topology of the MgO films.

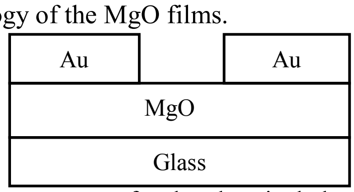

Figure 1. Test structure for the electrical characterization of MgO thin films

III. Results & Discussion

A. Electrical Properties of Nanostructured MgO

Fig.2 shows the *I-V* curves of MgO films at different annealing temperatures measured by two point probes (BUKOH KEIKI-EP 2000). The voltage was ramp up to 10V to investigate the electrical behavior of the prepared films. As can be seen in figure, for all samples the current linearly increased with applied voltage.

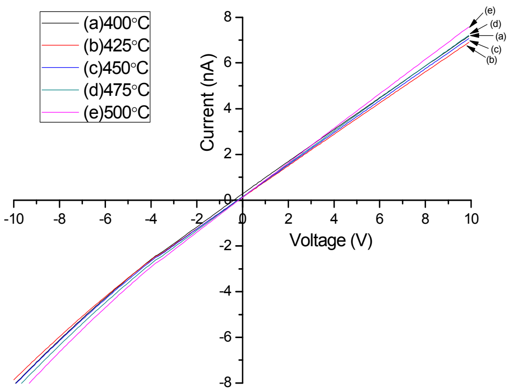

Figure 2. The electrical behavior of MgO films in terms of I-V characteristic

Further electrical investigation was done using the following equation;

$$\rho = R\frac{wt}{l} \qquad (1)$$

where ρ is the resistivity (Ω.cm) of the films; *R* is resistance (Ω); *w* (cm) and *l (*cm) is the width and length between metal contact respectively; and *t* is the thickness of the films. The resistivity value of each MgO film was plot in the graph as shown in fig. 3.

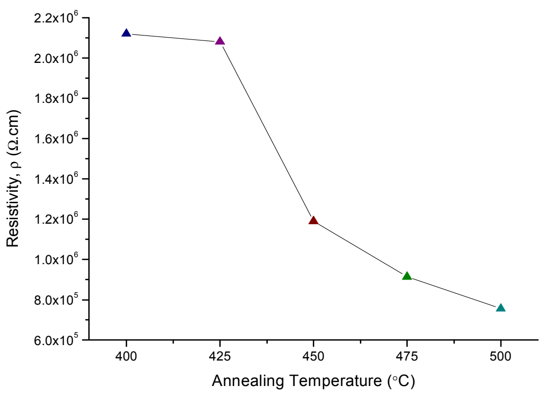

Figure 3. The resistivity of MgO films at different annealing temperature

From the graph, we observed that increasing in the annealing temperature from 400 to 500ºC has resulted on the decreased on resistivity. The resistivity values of the films are 2.12×10^6, 2.08×10^6, 1.19×10^6, 9.14×10^5 and 7.56×10^5 Ω.cm. Since from equation 1, thickness of the film has positive correlation with the resistivity, hence decrease in the resistivity is due to the decrease in the thickness of the MgO films. It has been proven by the value of the thickness that tabulated in table 1.

B. Dielectric Properties of Nanostructured MgO

The dielectric properties of nanostructured MgO thin films which are dielectric constant and dielectric loss were characterized using impedance spectroscopy system (Solartron 1260A-1296). As shown in fig. 4(a), the dielectric constant of the MgO thin films were changed as the annealing temperature varied from 400°C to 500°C with 25°C interval. MgO thin film annealed at 500°C give highest dielectric constant which is 10.114 at 1 kHz frequency. This is due to the electric dipole polarization, relaxation and large grain size effects [7]. Besides, it also might due to the low porosity in the film.

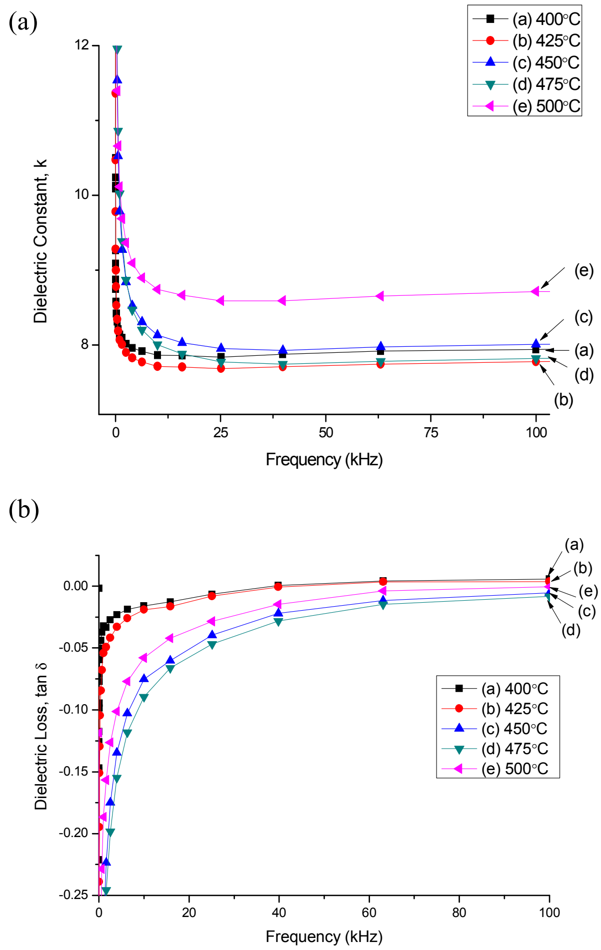

Figure 4. (a) Dielectric constant and (b) dielectric loss as functions of frequency for nanostructured MgO thin film deposited at five different annealing temperatures.

Furthermore, the dielectric loss of prepared MgO films that shown in fig. 4(b) are reduced as the annealing temperature increased form 400ºC to 475ºC. However the dielectric loss value increased again when annealing temperature reached 500ºC could be due to the more charge deffect in the MgO film.

C. Mgo Thin Films Growth

Annealing temperature affected MgO thin films growth. As tabulated in table 1, increased in annealing temperatures resulted in reduced of thin film thickness. The films thickness decreased from 433.185nm to 128.265nm as a result of increasing the annealing temperature from 400ºC to 500°C.

TABLE I:

THICKNESS OF MgO THIN FILMS ANNEALED AT 400, 425, 450, 475 AND 500°C

Annealing Temperature (ºC)	**Oxide Thickness (nm)**
400	433.185
425	307.205
450	194.695
475	153.735
500	128.265

During annealing process, the temperature applied will influence the diffusion of MgO atom. As the temperature increase, the merging activities of MgO particle become faster hence produce denser films and large grain size [8, 9].

D. Surface Topology of MgO Films

Atomic force microscopy (XE-100 Park System) was used to observe the surface topologies which include the surface roughness of the MgO films. From AFM images shown in fig. 5, it is proven that MgO films become denser as the annealing temperature increased. Therefore MgO films became thinner due to the more compact MgO particle in the films.

Surface roughness of the films annealed at 400, 425, 450, 475 and 500ºC are 0.1, 0.134, 0.126, 0.226 and 0.242nm respectively. Although the roughness values are increased with the annealing temperature, but all the prepared MgO films are uniformed.

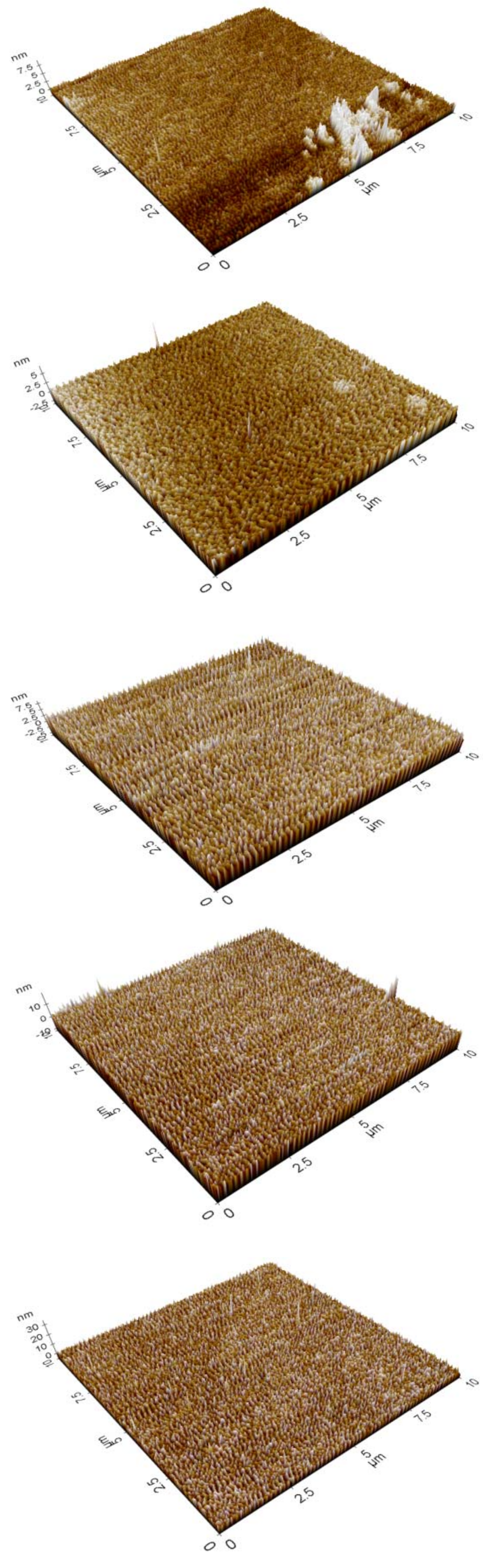

Figure 5. Surface topology of MgO films annealed at (a) 400ºC, (b) 425ºC, (c) 450ºC, (d) 475ºC and (d) 500ºC

From the electrical, dielectric and structural properties obtained, it shows that, the MgO films annealed at 500ºC give better characteristics compared to others. The film is then exposed to the structural characterization (fig.6) using field emission scanning electron microscopy, FESEM (JEOL-JSM 7600F). We observed that it has uniform surface with roughness of 0.242nm and the particle is in nanometer scale which is 37.5nm. From FESEM image also, it proven that as the annealing temperature increased, the grain size became large and reduce the porosity due to the higher growth rate. Although the films is uniform, yet the film still do not have any structure and further investigation by vary deposition parameter need to be done.

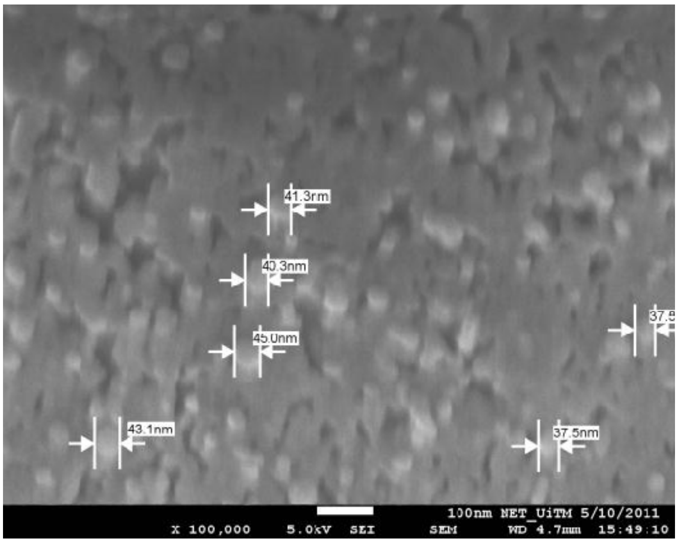

Figure 6. FESEM image of MgO film annealed at 500ºC

Besides that, the elemental analysis (fig.7) was done using Energy-Dispersive X-ray Spectroscopy (EDS) in order to confirm the produced film is magnesium oxide, MgO.

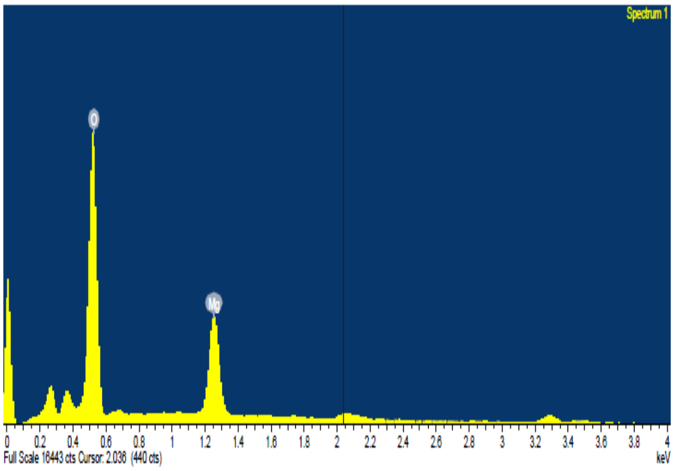

Figure 7: EDS analysis of MgO film annealed at 500ºC

Since dielectric is the most important layer that contributes to the properties of the device such as capacitor, therefore the dielectric films must good in the electrical, dielectric and structural properties. From the results that we have obtained, the MgO films annealed at 500ºC is found to be suitable candidate to be used as dielectric layer.

CONCLUSION

The MgO thin films were prepared at five different annealing temperatures by using sol-gel spin coating method. We observed that, thin film annealed at 500ºC has better dielectric properties which the dielectric constant is 10.114 at thickness of 128.265. Moreover, the nanometer dimension particle as small as 37.5nm is detected at thin films annealed at this temperature. From the results, it can be concluded that higher annealing temperature will improve the surface morphology and enhance the dielectric properties of the film.

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