Effect of sputtering power on the microstructure of Mg doped ZnO thin films

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Abstract. In this paper, Mg doped ZnO thin films were prepared on glass substrates by RF magnetron sputtering. The surface morphology, composition, structure, thickness and refractive index of the films were studied by SEM, XRD and ellipsometer. The results show that the sputtering power has no great influence on the growth direction of the film. When the sputtering power changes from 40 W to 100 W, the crystalline state of the film becomes better, the grain size and the thickness and the refractive index of the film increase with the rising of the sputtering power.

1. Introduction
ZnO is a new type of II VI wide band gap semiconductor material. The band gap width at room temperature is about 3.3 eV, which is hexagonal wurtzite structure. Zno has good chemical stability, rich materials and low price; its transmittance can reach more than 90% in the visible light wavelength range; especially, the band gap width of ZnO film can be adjusted in the range of 3.3 to 7.9 eV by Mg doping, resulting in its detection range of ultraviolet light will expand to the range of 200 nm to 280 nm. ZnO thin films have been widely used in light-emitting diodes, laser diodes, UV detectors, transparent electrodes, gas sensors and photoconductivity. Now they have been widely used in all kinds of photoelectric detection devices and become the most potential new materials for photoelectric devices. [1-3]

Many preparation methods have been applied for ZnO thin films, such as pulsed laser deposition (PLD), chemical vapor- deposition (CVD), magnetron sputtering, molecular beam epitaxy (MBE) and sol-gel method. Among these methods, magnetron sputtering method has the advantages of high sputtering speed, fast growth, compactness, high purity and firm adhesion with the substrate, and the substrate is widely used because of the increasing temperature under the bombardment of high-energy particles without heating. It is easy for magnetron sputtering to dope in the ZnO thin films, which is uniform, stable and fast film-forming. The films discussed in this paper are prepared by high speed RF magnetron sputtering. [4-7]

2. Experimental part
Mg doped ZnO thin films were prepared on ordinary glass substrates by RF magnetron sputtering with JPG450 type ultra-high vacuum magnetron sputtering equipment. The morphology, structure,
thickness and refractive index of the films were characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD) and ellipsometry.

Before sputtering, the glass substrate is cleaned by acetone, anhydrous ethanol and deionized water. The target materials are ZnO and Mg doped ZnO ceramic targets with the mole percentages of 75% and 25%, respectively. The purity of the target materials reaches 99.99%. The target base distance is set at 70 mm. By changing the sputtering power, the RF magnetron sputtering method is used to prepare magnesium doped ZnO film. Other process parameters are as follows: substrate temperature is 300 ºC, background vacuum is 2×10^{-4} pa, gas flow of oxygen and argon is controlled as 2:1, sputtering time is 4 hours, sputtering pressure is 3 Pa.

3. Result and discussion

3.1. Surface morphology analysis of Mg doped ZnO thin films

SEM was used to observe the surface morphology of the films by focusing the electron beam on sample surface, and interacts with the sample to generate various physical signals. The signals are received, amplified and converted into modulation signals by the detector. Finally, images reflecting various characteristics of the sample surface are displayed on the fluorescent screen. SEM is an effective analytical tool for the study of sample surface, which has the characteristics of large depth of field, strong stereo sense of image, large magnification range, continuously adjustable, high resolution, large sample room space and simple sample preparation. Figure 1 shows the SEM pattern of the film at same magnification.

![Figure 1.](image)

It can be seen from Figure 1 that there are granular film growth on the glass surface, but the surface morphology of the film is different under various sputtering power. With the increased sputtering power, the film particles become larger and the surface becomes more and more loose and rough.

3.2. Composition analysis of Mg doped ZnO thin films

By comparing the EDS spectra of Mg doped ZnO thin films with different sputtering power, the following figure can be obtained:
Figure 2. Percentage spectrum of Mg atoms in Mg doped ZnO thin films at different sputtering power.

It can be seen from Figure 2 that the content of Mg in the film increases with the increase of sputtering power when other conditions are not changed. As the content of Mg and Zn in the target remains unchanged, the increasing of Mg content will cause the decrease of the content of other components, indicating that the sputtering power has a certain impact on the composition of the film.

3.3. XRD analysis of Mg doped ZnO thin films

The working principle of XRD is diffraction phenomenon. The processed diffraction pattern of X-ray signal is obtained by X-ray diffraction on the object surface.

Figure 3. X-ray diffraction patterns of Mg doped ZnO films with different sputtering power (a)40 W,(b)60 W,(c)80 W,(d)100 W.

The main purpose of XRD is to get the basic information of the structure, composition and morphological structure of the material, and finally determine the molecular and atomic structure of the material. XRD is an important research method for X-ray diffraction analysis of materials to obtain information such as composition, internal atom or molecular structure [8-10]. Figure 3 shows the X-ray diffraction patterns of Mg doped ZnO thin films with different sputtering power.
It can be seen from Figure 3 that there are diffraction peaks position at 31.8°, 36.0° and 56.2° of Mg doped ZnO thin films with different sputtering powers, and the corresponding growth directions are (100), (101) and (110), which indicates that the film does not show the c-axis preferred growth. The data of (100) plane in XRD were fitted, and the experimental data were analyzed and calculated by using Scheller formula, and Table 1 was obtained. Scherrer formula, also known as Scherrer formula, Debye Scheler formula, was first proposed by Debye, a famous German chemist, and his graduate student Scheler. It is a famous formula for XRD analysis of grain size. \[ D = \frac{K \lambda}{B \cos \theta} \]

\(K\) is Scherrer constant, \(B\) is half height width of diffraction peak, then \(K = 0.89\); \(\theta\) is diffraction angle, which is also converted into radian system (RAD); \(\lambda\) is X-ray wavelength, which is 0.154056nm[11-15].

| Sputtering power (W) | Diffraction peaks (°) | Full width at half maximum (FWHM) | Strength | Grain size (nm) |
|---------------------|-----------------------|-----------------------------------|----------|-----------------|
| 40                  | 31.32                 | 0.90671                           | 11.85413 | 13.238          |
| 60                  | 31.42529              | 0.96113                           | 44.23542 | 13.3598         |
| 80                  | 31.42419              | 0.97558                           | 63.36435 | 14.2021         |
| 100                 | 31.54164              | 1.0597                            | 103.96642| 15.0721         |

It can be seen from Table 1 and Figure 4 that although changing the sputtering power does not affect the growth direction of the film, when the power increases from 40W to 100W, the growth strength of the film in this direction increases gradually, and the grain size of the film is also increasing.

**Figure 4.** Grain size and half peak full width of Mg doped ZnO thin films at different sputtering power.

### 3.4. Thickness analysis of Mg doped ZnO thin film

Ellipsometer is a kind of optical measuring instrument used to detect the thickness, optical constants and microstructure of thin films. Because of its high measurement accuracy, it is suitable for ultrathin film, non-contact with the sample, no damage to the sample and no need of vacuum, making ellipsometer a very attractive measurement instrument. The average thickness of the film measured by ellipsometer is shown in Figure 5. The results show that the thickness of the films increases with the sputtering power.
The result show that when the sputtering power is in the range of 40 ~ 100 W, both the grain size and the thickness of the film increase with the increase of sputtering power.

![Graph showing the relationship between thickness and sputtering power.](image)

**Figure 5.** Average thickness of Mg doped ZnO films at different sputtering power.

3.5. Refractive index analysis of Mg doped ZnO thin films

The change of refractive index with sputtering power measured by ellipsometer is shown in Figure 6.

![Graph showing the change of refractive index with wavelength.](image)

**Figure 6.** Refractive index of Mg doped ZnO films at different sputtering power
(a)40 W,(b)60 W,(c)80 W,(d)100 W.

It is reveal that when the sputtering power increases from 60 W to 100 W, the refractive index of the film increases regularly, and decreases with the increase of the wavelength. when above 800 nm, the refractive index is relatively flat. When the sputtering power is 40 W, the change rule of refractive index is different from that of other powers, and generally decreases with the increase of wavelength.

4. Conclusions

Mg doped ZnO thin films with different sputtering powers were prepared on glass substrates by RF magnetron sputtering. The morphology, composition and structure of Mg doped ZnO thin films were studied by SEM, XRD and ellipsometer. The results are as follows: 1) Dense films were obtained on
glass substrates by RF magnetron sputtering, and the Mg doped ZnO thin film was flat; 2) The Mg content in the film increases with the increasing sputtering power, but the Mg content in the film is still not the same as that in the target; 3) The growth direction of the films is not affected by the sputtering power, the films grow along (100), (101) and (110) planes; 4) With the increase of sputtering power, the crystalline state and grain size of the films are improved; 5) The thickness of the film increases with the sputtering power, and can reach more than 300 nm; 6) The refractive index of Mg doped ZnO thin film increases with the sputtering power.

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