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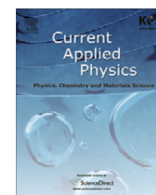

H_2/Ar and vacuum annealing effect of ZnO thin films deposited by RF magnetron sputtering system

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ABSTRACT

The properties of ZnO films were investigated as functions of annealing temperatures in H_2/Ar and vacuum. The resistivities and mobilities of ZnO films decreased with increase of annealing temperatures in vacuum and H_2/Ar ambients. However, the carrier densities of ZnO films increased with increase of annealing temperatures in vacuum and H_2/Ar ambients. The resistivities of ZnO_2 films annealed at 300 °C were ~2186 Ω cm and ~798 Ω cm in H_2/Ar and vacuum ambients, respectively. The resistivities of ZnO films annealed in vacuum and H_2/Ar ambients at 600 °C were similar with ~0.040 Ω cm and ~0.035 Ω cm, respectively. The hydrogen donor was more dominant than the oxygen vacancy or Zn interstitial donor in ZnO films annealed in ambient H_2/Ar at low temperatures. The average optical transmission was >82% and an orientation of the deposition was [0 0 2] for all ZnO films annealed in vacuum and H_2/Ar ambients.

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1. Introduction

Transparent conductive oxide (TCO) films are being more widely used recently due to the rapid development of the optoelectronic industry, with applications such as liquid crystal displays, solar cells, and organic light emitting diodes (OLEDs). Sn-doped In_2O_3 (ITO) has been mostly used as the TCO because of its excellent conductive and transparent properties in the visible region. However, ITO contains In, which is a limited resource in the earth and very expensive. Therefore, it is highly desirable to substitute the ITO in a TCO film in optoelectronic devices with a more common, less expensive material that has a high transparency in the visible region.

ZnO is often considered as a substitution material for the ITO film as the TCO electrode in optoelectronic devices [1,2]. A ZnO film has several advantages, such as high transparent conduction in visible region, low cost, non-toxicity, and an abundance of ZnO in the earth. ZnO thin film has a hexagonal wurtzite structure and 3.37 eV direct wide energy band gap, and its II–VI form is a semiconductor.

It is still difficult to grow a ZnO thin film that has sufficient conductivity and stability, although much research has been done over the last several decades. Based on experimental results, undoped ZnO is n-type with zinc interstitial (Zn_i) or an oxygen vacancy (V_o) donor [3,4]. Van de Walle reported that hydrogen is also a donor source in ZnO films [5,6]. The hydrogen ion is stable and serves as a shallow donor with a high carrier concentration in ZnO films. Recently, it has been reported that the resistivity of the ZnO film annealed in ambient hydrogen decreases $\sim 2 \times 10^{-3}$ Ω cm when the film was deposited with ZnO (doped with Al, Ga or In) on a metal target using a sputtering system [7]. It is necessary to investigate the hydrogen gas annealing effect in ZnO films. ZnO films have been deposited by various deposition techniques such as sputtering, chemical vapor deposition, pyrolysis and pulsed laser deposition systems. The sputtering approach is most commonly used because the system can readily deposit a large surface area and is fast compared with other systems.

In this study, we deposited ZnO films on a Corning glass substrate at 200 °C using a RF magnetron sputtering system and ZnO films were annealed in vacuum and H_2/Ar ambients from 300 °C to 600 °C. We aimed to investigate the electrical, structural and optical properties ZnO films as functions of annealing temperatures in vacuum and H_2/Ar ambients using the van der Pauw method at room temperature, X-ray diffraction (XRD), scanning electron microscopy (SEM), and UV/Visible spectrophotometry.

2. Experimental

ZnO films were deposited on the Corning glass (#1737) substrate at 200 °C substrate using a RF magnetron sputtering system

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Table 1
Process conditions of the RF magnetron sputtering system for ZnO film deposition.

Deposition parameter	Value
RF, power	100 W
Ar gas	25 sccm
Substrate temperature	200 °C
Distance between target and substrate	10 cm
Rotation speed	3 rpm
Deposition time	60 min
Background pressure	5×10^{-6} torr
Working pressure	2.0×10^{-2} torr

with pure Ar gas. Before the ZnO film deposition process, the chamber was pumped down to 5×10^{-6} torr base pressure using turbomolecular and rotary pumps. The working pressure of ZnO film deposition was maintained at 2×10^{-2} torr by MFC control of Ar gas. The Corning glass substrate was treated to sequential ultrasonic cleaning with acetone, ethanol and deionized water. Two inches ZnO (99.99%) target was used, and the process conditions appear in Table 1.

The thickness of deposited ZnO thin film was about 400 nm, and the annealing process was done in a quartz tube with $\sim 10^{-3}$ torr vacuum and $\sim 10^{-2}$ torr H_2/Ar (4 ratio of Ar to H_2), respectively, for 30 min each. The tests of structural, electrical and optical properties were done at room temperature. The structural properties were investigated with X-ray diffraction (PANalytical B.V., X'Pert-Pro) and scanning electron microscopy (FEI, Quanta 200) for crystal orientation and surface appearance, respectively. The electrical properties were measured for resistivity, carrier density and mobility using the Van der Pauw method at room temperature (Ecopia, HMS-3000). The optical properties were measured with a UV/Visible spectrophotometer in the 300–800 nm range (Perkin Elmer, Lambda-950).

3. Results and discussion

This study investigated the properties of ZnO films as functions of vacuum and H_2/Ar at different annealing temperatures. ZnO films were deposited with the process conditions shown in Table 1 using a RF magnetron sputter system. Fig. 1 shows the resistivities, mobilities and carrier densities of the ZnO films as functions of annealing temperatures in vacuum and H_2/Ar ambients.

In the case of ZnO films annealed in vacuum and H_2/Ar ambients, observations indicated an n-type semiconductor, and the resistivities decreased with increasing annealing temperatures as shown in Fig. 1a. The resistivities of H_2/Ar annealed ZnO thin films were lower than those of vacuum-annealed ZnO films. The results showed that the hydrogen donor contributed to the increase of carrier densities in ZnO films during H_2/Ar annealing. Van de Walle found evidence that hydrogen is a source of conductivity for ZnO films [5,6]. Hydrogen ions are stable considering it is the lowest energy state with respect to the Fermi level position, and it can be incorporated in high concentrations as a shallow donor in ZnO.

The resistivities of ZnO thin films were $\sim$2184 Ω cm and $\sim$798 Ω cm in the vacuum and H_2/Ar annealing at 300 °C, respectively (Fig. 1a). The carrier density and mobility of the ZnO films annealed in vacuum were $\sim 5.8 \times 10^{13}/\text{cm}^3$ and $\sim 49.7\ \text{cm}^2/\text{V s}$, respectively (Fig. 1b and c). The carrier density and mobility of the ZnO film annealed in H_2/Ar were $\sim 5.0 \times 10^{14}/\text{cm}^3$ and $\sim 15.8\ \text{cm}^2/\text{V s}$, respectively (Fig. 1b and c). The resistivity of H_2/Ar annealed the ZnO film was much lower than that of the ZnO film annealed in vacuum. The carrier density of the H_2/Ar annealed

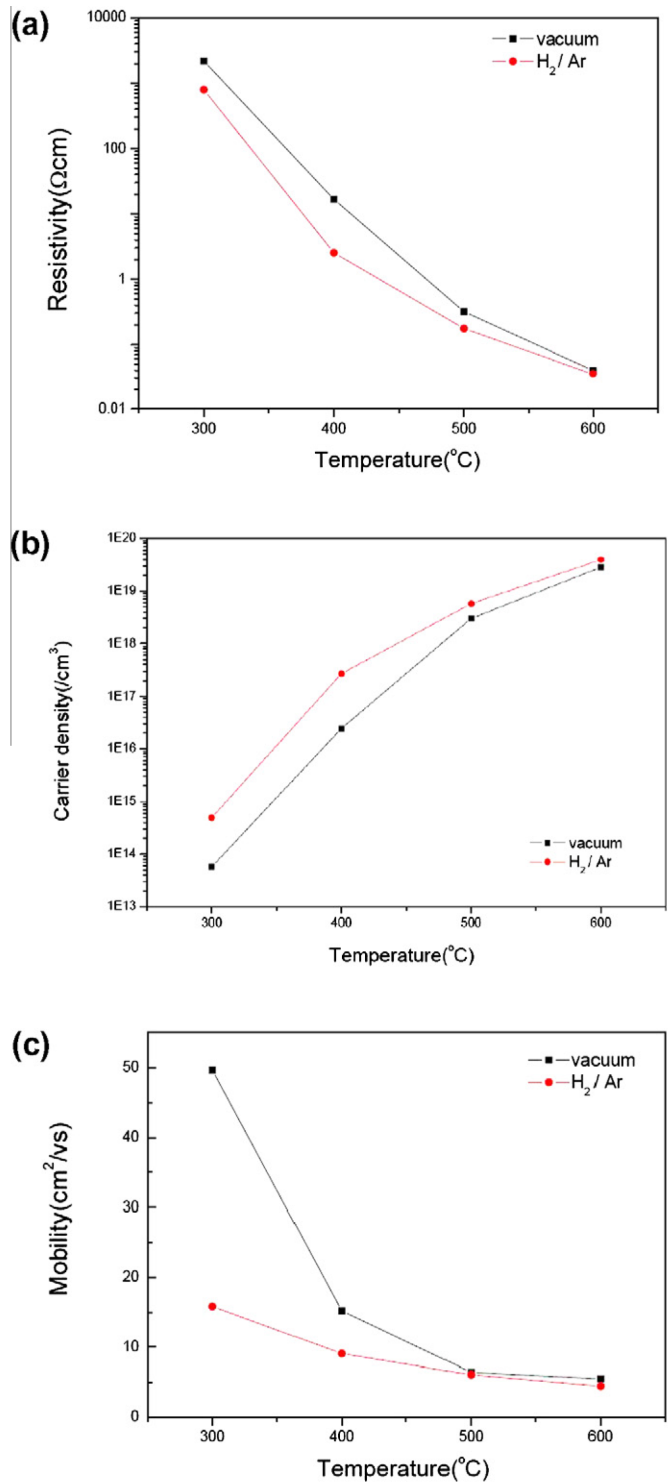

Fig. 1. Resistivities, carrier densities and mobilities of ZnO films as functions of vacuum and H_2/Ar annealing temperatures.

ZnO film was higher than that of the vacuum-annealed ZnO film. The mobility of the H_2/Ar annealed ZnO film was lower than that of the film annealed in vacuum only. This lower mobility value of the H_2/Ar annealed ZnO film was likely due to the increase in the electron scattering center density. The resistivities and mobilities of ZnO films decreased vs. increasing annealing temperatures, and the carrier densities increased with respect to increasing

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