Effects of rapid thermal annealing on structural, magnetic and optical properties of Ni-doped ZnO thin films

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XPS depth profiles were used to investigate the effects of rapid thermal annealing under varying conditions on the structural, magnetic and optical properties of Ni-doped ZnO thin films. Oxidization of metallic Ni from its metallic state to two-valence oxidation state occurred in the film annealed in air at 600 °C, while reduction of Ni2+ from its two-valence oxidation state to metallic state occurred in the film annealed in Ar at 600 and 800 °C. In addition, there appeared to be significant diffusion of Ni from the bottom to the top surface of the film during annealing in Ar at 800 °C. Both as-deposited and annealed thin films displayed obvious room temperature ferromagnetism (RTFM) which was from metallic Ni, Ni2+ or both with two distinct mechanisms. Furthermore, a significant improvement in saturation magnetization (M_s) in the films was observed after annealing in air (M_s = 0.036 μB/Ni) or Ar (M_s = 0.033 μB/Ni) at 600 °C compared to that in as-deposited film (M_s = 0.017 μB/Ni). An even higher M_s value was observed in the film annealed in Ar at 800 °C (M_s = 0.055 μB/Ni) compared to that at 600 °C mainly due to the diffusion of Ni. The ultraviolet emission of the Ni-doped ZnO thin film was restored during annealing in Ar at 800 °C, which was also attributed to the diffusion of Ni.

1. Introduction

Spintronics has emerged as a new technology, which is based on electron spins rather than electron charges to carry information, and has offered an opportunity for a new generation of devices combining standard microelectronics with spin-dependent effects that arise from interactions between spins of charge carriers and magnetic properties of materials [1]. For spintronic applications, diluted magnetic semiconductors (DMS) have attracted a lot of attention [1], especially ZnO-based DMS, due to the prediction of the existence of room temperature ferromagnetism (RTFM) in transition metals (TMs) doped ZnO by Dietl et al. [2] and Sato et al. [3]. However, contradictory experimental results have been reported on TM-doped ZnO, i.e., RTFM was observed in some studies [4–7] while no RTFM was detected in some other studies [8–11]. Interestingly, the RTFM in pure ZnO was also reported recently [12–17]. Although the RTFM in some ZnO-based DMS materials was observed, the origin of such RTFM still could not be satisfactorily interpreted. For TM-doped ZnO, in some cases, the RTFM was suggested to be a consequence of the precipitation of magnetic clusters or the formation of secondary magnetic phases [18–20] and in some other cases, RTFM was suggested to be intrinsic in origin [21–24]. A free carrier mediated exchange mechanism was proposed to induce the intrinsic RTFM in TM-doped ZnO [2,21,22]. However, TM-doped ZnO materials that are ferromagnetic are often poorly electrically conductive or even highly insulating, which cannot be explained by the carrier-mediated model. The origin of RTFM for insulating oxide DMS has been widely explained by a bound magnetic polaron (BMP) model [25,26], suggesting that oxygen vacancies [23,27–30] or metallic interstitials [31,32] in oxide DMS materials locally trap electrons to occupy orbitals interacting with the d subshells of neighboring TM dopants, resulting in the ferromagnetism. For pure ZnO, the origin of the RTFM has been attributed to the defects at Zn sites in ZnO thin films [12]. Zn nanoclusters embedded in ZnO nanowire matrices [13], interstitial Zn at the surface of ZnO nanorods [14], intrinsic defects in ZnO films [15], defects that cause a 469 nm emission [16], and vacancies present in grain boundaries [17], etc. It is still a challenge to properly understand the mechanisms governing the ferromagnetic ordering in ZnO-based DMS, e.g., Ni-doped ZnO [33–40]. The depth distribution of Ni in Ni-doped ZnO thin films is expected to influence the structural, magnetic and optical properties of the
films, especially when the films are annealed at elevated temperatures, which has seldom been reported in the literature. For optical property of TM-doped ZnO, the ultraviolet (UV) emission is always weakened by the TM doping which can strengthen magnetic property of ZnO [40–43]. For instance, Ni doping in ZnO may result in a weaker peak and a red shift of UV emission [41].

In this work, X-ray photoelectron spectroscopy (XPS) depth profiles were used to investigate the effect of rapid thermal annealing under varying conditions on the structural, magnetic and optical properties of Ni-doped ZnO thin films prepared via magnetron sputtering deposition.

2. Experiment

Ni-doped ZnO thin films were grown on quartz substrates by magnetron co-sputtering a pure Ni target and a pure ZnO target (Kurt J. Lesker) with an RF power of 350 W applied to the ZnO target and a DC power of 11 W applied to the Ni target. An oxygen non-stoichiometry can appear in the Ni-doped ZnO thin films grown in pure Ar ambient due to the loss of oxygen during the sputtering process. Thus, the gas used during the film depositions was a mixture of 40 sccm Ar and 4 sccm O₂ and the working gas pressure was about 10 mTorr. The function of O₂ is to improve the oxygen stoichiometry in the films. The deposition rate of the films was about 33.2 nm/min. The films were annealed in 1 atm of air at 600 °C or in 1 atm of Ar gas environment at 600 °C and 800 °C using rapid thermal processing for 10 min. The structure of the films was characterized by means of high resolution X-ray diffraction (HR-XRD, Xpert PRO) with a Cu Kα anode (i ~ 0.154 nm). X-ray photoelectron spectroscopy (XPS) with a monochromatic Al Kα X-ray source (1489.7 eV) was used to determine the chemical states of elements in the films. The XPS depth profiles of the sample surfaces were measured down to about 90 nm using low-energy Ar⁺ sputtering. The magnetic properties of the films were measured with an alternating gradient magnetometer (AGM, MicroMag™, 2900) at room temperature. The optical properties of the films were measured using photoluminescence (PL) with an excitation wavelength of 325 nm at room temperature.

3. Result and discussion

Fig. 1 shows the XRD spectra for the Ni-doped ZnO thin films both as-deposited and annealed under different conditions. Only the peak corresponding to ZnO(002) is observed in the as-deposited film, indicating that a polycrystalline wurtzite phase with c-axis preferred orientation is formed in the film. No diffraction peaks attributed to Ni-related secondary phases are observed, implying that the Ni atoms might substitute for the Zn atoms or any occurrence of Ni should be below the resolution of the XRD measurements. After annealing in Ar or air at 600 °C, the ZnO(002) peak still exists but shifting toward right. Moreover, the standard peak position of ZnO powders (34.45°) locates between those of the as-deposited (34.20°) and annealed (34.59°) thin films, which means that the lattice constant c is elongated in the as-deposited film and shortened in the annealed films. The stress σ in the plane of the film can be calculated using the estimated lattice constant c using the following expression [44],

\[ \sigma = 4.5 \times 10^{11} \left( c_0 - c \right) / c_0, \]

where \( c_0 \) is the lattice constant of bulk ZnO. An elongated c indicates that the as-deposited film is in a state of compressive stress parallel to the (002) plane and tensile stress parallel to the c-axis. However, a shortened c in the annealed films reveals that the films after annealing are in a state of tensile stress parallel to the (002) plane and compressive stress parallel to the c-axis. Similar results have been reported by Gupta et al. [45] and Liu et al. [44]. The change in stress in the films after annealing was attributed to interstitial oxygen by Gupta et al. However, the interstitial oxygen is not the primary defect in ZnO crystal [46,47]. Thus, Liu et al. developed Gupta’s theory and attributed it to oxygen vacancies and interstitial zinc atoms.

The change in stress in the films after annealing is primarily due to the defects produced during the film growth process. As the deposition rate of the films is high during the sputtering deposition, the zinc atoms are prone to enter the octahedral interstices [44,46,47]. Simultaneously, the oxygen atoms have a tendency to deviate from their positions in the ZnO crystal lattice, so there are also a large number of oxygen vacancies in the films. On one hand, the interstitial zinc atoms and the zinc atoms at the normal sites in the ZnO crystal lattice exclude each other, which leads to the expansion of the film lattice. On the other hand, the existence of oxygen vacancies destroys the balance of attraction and repulsion between neighboring crystal planes, which leads to the contraction of the film lattice. Therefore, the lattice deviation of the Ni-doped ZnO films from that of the stress free ZnO powders may be a result of the formations of oxygen vacancies and interstitial Zn. There are relatively fewer oxygen vacancies but more Zn interstices in the as-grown Ni-doped ZnO film, which shows a state of compressive stress parallel to the (002) plane and tensile stress parallel to the c-axis, thus causing the elongation of the lattice constant c. After annealing at 600 °C in Ar or air, the oxygen vacancies increase in the films. The thermal energy supplied in annealing motivates the migration of the zinc interstices to the normal crystal lattice sites, which reduces the number of interstitial zinc atoms. Consequently, the films after annealing at 600 °C in Ar or air are in a state of tensile stress parallel to the (002) plane and compressive stress parallel to the c-axis, which causes a contraction of lattice constant c.

In addition, the grain growth can also induce the change in stress. For the as-deposited film, the film grows with a columnar structure along the c-axis due to a (002) preferential orientation during the sputtering deposition [44]. The columnar grains squeeze...
each other in a direction parallel to the (002) plane, which leads to a restraint of grain growth and a compressive stress along the direction. After annealing, the zinc atoms released from the interstitial positions result in a grain growth along the c-axis due to the lowest binding energy in the [002] direction of ZnO when they revert to the crystal lattice sites. However, the grain growth is simultaneously restrained along the c-axis because of the existing film, which causes a compressive stress along the c-axis.

After annealing in Ar at 800 °C, the ZnO(002) peak disappears and no other obvious diffraction peaks are observed, which means that the ZnO matrix in the film has been degraded. During the annealing in Ar at 800 °C, the decomposition of the ZnO matrix occurs substantially because of the inert environment and high temperature. As a result, the grains are damaged and the grain size becomes smaller, which have caused the diminished diffraction peaks. The decomposition of the ZnO matrix is also confirmed by the loss of oxygen from the film during the annealing in the inert gas through XPS.

XPS is performed to determine the depth profile of atomic concentration for each element in the Ni-doped ZnO thin films, as shown in Fig. 2. The atomic concentrations of Zn, Ni, and O in the as-deposited film are uniform along the depth profile, which are about 47.26, 2.25, and 50.49%, respectively (Fig. 2(a)). After the annealing in air at 600 °C, no significant differences have been observed (Fig. 2(b)). After the annealing in Ar at 600 °C, the atomic concentrations of each element in the film are still uniform along the depth profile, while the O decreases to about 44.55% (Fig. 2(c)), which means that the O in the film diffuses into the inert gas because of the concentration gradient of O in the film and the ambient during the annealing in Ar. The oxygen decomposed from the film matrix diffuses onto the film surface, and then departs the film surface and enters the Ar gas environment. It is more interesting to notice that after the annealing in Ar at 800 °C, the atomic concentrations of each element in the film become not uniform any more along the depth profile (Fig. 2(d)). The O is from about 31.56 to 41.88% at the etching depth from 10 to 90 nm in the film, which is much lower than 44.55% in the film annealing in Ar at 600 °C. The annealing in Ar at higher temperature of 800 °C causes a more severe loss of oxygen from the film compared to the annealing in Ar at 600 °C. The more severe loss of oxygen confirms the decomposition of the ZnO matrix through XRD. Apart from the diffusion of O, a significant diffusion of Ni from the bottom to the top surface of the film during annealing in Ar at 800 °C is also observed from Fig. 2(d). Ni becomes 23.97% at the etching depth of 10 nm, while 0% at the etching depth of 90 nm in the film. As a result of the diffusion of Ni, the concentration of Ni increases at the surface, while the pure ZnO is left at the bottom of the film.

The x value is the mole fraction of Ni in the Ni-doped ZnO (Zn_{1-x}Ni_xO) film. Fig. 3 shows the x values of the Zn_{1-x}Ni_xO films before and after annealing as functions of the etching depth. The values of x in the as-deposited film are uniform along the depth profile, which are around 0.045 (Fig. 3(a)). No significant differences have been observed after annealing in air (Fig. 3(b)) or Ar (Fig. 3(c)) at 600 °C, which indicates that there is no diffusion of Ni during the annealing. However, after the annealing in Ar at 800 °C, the values of x become not uniform any more along the depth profile (Fig. 3(d)). x becomes about 0.350 at the etching depth of 10 nm, while 0 at the etching depth of 90 nm in the film, which shows a significant diffusion of Ni from the bottom to the top surface of the film during annealing in Ar at 800 °C.

The chemical states of nickel in the films are studied also using XPS. Fig. 4 shows the Ni 2p depth profiles measured from the Ni-

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doped ZnO films before and after annealing. In the XPS spectra, the peaks at about 852.5 and 855 eV are characteristic of metallic Ni $^{2+}$ and Ni$^{2+}$ 2p$_{3/2}$ states, respectively. Although the noises in the Ni 2p spectra are relatively high due to the low nickel contents in the films, they are not obstructing the effective information. The Ni 2p spectra measured from the top surfaces of the films due to the contamination of the top surfaces. Fig. 4(a) is the Ni 2p depth profile measured from the as-deposited film. The metallic Ni 2p$_{3/2}$ peak at 852.2 eV and the Ni$^{2+}$ 2p$_{3/2}$ peak at 854.4 eV illustrate that the nickel is in its metallic and two-valence oxidation states simultaneously in the as-deposited film. It is expected that Ni$^{2+}$ has been effectively doped into the ZnO wurtzite lattice at the Zn$^{2+}$ sites, while the metallic Ni exists in the interstices of the ZnO wurtzite lattice or the grain boundaries of the ZnO matrix.

Fig. 4(b) shows the Ni 2p depth profile measured from the film annealed in air at 600 °C. The unique Ni$^{2+}$ 2p$_{3/2}$ peak at 854.6 eV indicates that the nickel is only in its two-valence oxidation state in the film. The metallic Ni is oxidized to Ni$^{2+}$ in the film after annealing in the air. The XRD results in Fig. 1 have indicated that the interstitial Zn atoms revert to the crystal lattice sites during annealing in air at 600 °C. The metallic Ni as well as the interstitial Zn atoms can diffuse into the Zn$^{2+}$ sites in the ZnO wurtzite lattice because of the small diffusion barriers. As a result, the metallic Ni in the film is oxidized to Ni$^{2+}$ by the oxygen in the lattice, and the concentration of Ni$^{2+}$ at the Zn$^{2+}$ sites in the ZnO wurtzite lattice increases. However, the activation energy for oxygen diffusion is much larger than that for the diffusion of the interstitial Zn atoms and metallic Ni, and no significant oxygen diffusion from air into the film has been observed in Fig. 2(b), so no enough oxygen atoms from air diffuse in the lattice to fill up the new oxygen vacancies caused by the increased Zn$^{2+}$ and Ni$^{2+}$, oxygen vacancies increase. Thus, after annealing in air at 600 °C, the concentration of oxygen vacancies is increased in the film, which is consistent with the XRD results.

On the contrary, the unique metallic Ni 2p$_{3/2}$ peak at 852.0 eV indicates that the nickel is only in its metallic state in the film annealed in Ar at 600 °C, as shown in Fig. 4(c). Ni$^{2+}$ is reduced to the metallic Ni in the film, which is caused by the loss of oxygen from the film during the annealing in the inert gas (Fig. 2(c)). Ni$^{2+}$ at the Zn$^{2+}$ sites in the ZnO wurtzite lattice is easier to lose oxygen and then be reduced to metallic form than Zn$^{2+}$ in the film. Thus, Ni$^{2+}$ is reduced to the metallic Ni in the film. The metallic Ni reduced locates at the grain boundaries rather than in the interstices of the ZnO wurtzite lattice, which can be explained by the XRD results as shown in Fig. 1. The interstitial Ni can elongate the lattice constant $c$ of the ZnO like interstitial Zn, but the lattice constant $c$ is shortened with annealing in Ar at 600 °C as depicted by the XRD results, so the metallic Ni exists at the grain boundaries of the ZnO matrix.

During the annealing in Ar at 800 °C, the reduction of Ni$^{2+}$ and the diffusion of Ni atoms occur in the film, as shown in Fig. 4(d). Below 10 nm in depth in the film, the metallic Ni 2p$_{3/2}$ peaks at 852.5 eV indicate that the nickel mainly exists in the form of metallic Ni due to the reduction of Ni$^{2+}$ in the inert Ar. From the XRD (Fig. 1) and XPS (Fig. 2(d)) measurements for the film annealed in Ar at 800 °C, it has been found that the decomposition of the ZnO matrix and the more severe loss of oxygen from the film obviously occur during the annealing. The more severe loss of oxygen from the film causes a more complete reduction of Ni$^{2+}$. Usually, only the reduction of Ni$^{2+}$ occurs in the inert gas, but at the top surface of the film, only strong Ni$^{2+}$ 2p$_{3/2}$ peak at 855 eV is observed, which
means that the metallic Ni is fully oxidized to Ni$^{2+}$. The reason is that the reduced metallic Ni at the top surface of the film is oxidized again by the oxygen when the sample is stored in the air environment. The diffusion of Ni from the bottom to the top surface of the film during annealing in Ar at 800°C is also observed from Fig. 4(d). At the film surface, the peaks corresponding to nickel are very strong, but no peak for nickel is observed near the bottom of the film. The diffusion of Ni has also been confirmed by the depth profile of atomic concentration for Ni using XPS in Fig. 2(d). Thus, there is metallic Ni with higher concentration near the surface of the film annealed in Ar at 800°C, which causes the aggregation of more metallic Ni and the formation of more and larger metallic Ni clusters at the grain boundaries near the film surface.

Fig. 5 shows the magnetization of the Ni-doped ZnO thin films both as-deposited and annealed measured as a function of magnetic field at room temperature. Both the as-deposited and annealed films can display obvious room temperature ferromagnetism (RTFM). Furthermore, a significant improvement in saturation magnetization ($M_s$) in the films is observed after annealing in air ($M_s = 0.036 \mu B/\text{Ni}$) or Ar ($M_s = 0.033 \mu B/\text{Ni}$) at 600°C compared to that in as-deposited film ($M_s = 0.017 \mu B/\text{Ni}$). An even stronger $M_s$ is observed after annealing in Ar at 800°C ($M_s = 0.055 \mu B/\text{Ni}$) compared to that at 600°C.

The XPS spectra in Fig. 4(a) have illustrated that nickel is in its metallic and two-valence oxidation (Ni$^{2+}$) states simultaneously in the as-deposited film, so the RTFM of the as-deposited film is from the metallic and ionized nickel at the same time with two distinct mechanisms. The metallic Ni nanoclusters should exist at the grain boundaries, which may have certain RTFM but cannot be detected within the sensitivity of the XRD system used. The RTFM from Ni$^{2+}$ can be explained using a bound magnetic polaron (BMP) model. The defects at the grain boundaries, such as oxygen vacancies, locally trap electrons to occupy the orbitals interacting with the d subshells of the neighboring Ni dopants, leading to the formation of a ferromagnetic grain-boundary network. There are more Ni dopants and defects at the grain boundaries than in the volume of grains, so the RTFM induced by Ni$^{2+}$ is mainly from the grain boundaries rather than the volume of grains [17]. The BMP effect may also appear within the volume of grains, but the RTFM in the volume of grains is very weak, so the volume of grains displays a diamagnetic behavior coming from the ZnO matrix.

It was reported that the $M_s$ of TM-doped ZnO films declined after annealing in air due to the decreased oxygen vacancies in ZnO [23,44]. However, in this study, an enhanced $M_s$ is observed in the film annealed in air at 600°C, which is attributed to a stronger BMP effect. The XRD and XPS measurements in Figs. 1 and 4(b) illustrate that the oxygen vacancies increase after annealing in air at 600°C. In addition, the XPS spectra indicate that the concentration of Ni$^{2+}$ at the Zn$^{2+}$ sites in the ZnO wurtzite lattice also increases after annealing in air at 600°C. With the BMP model, higher densities of

![Fig. 4. XPS Ni 2p depth profiles of Ni-doped ZnO films: (a) as-deposited, (b) annealed in air at 600°C, (c) annealed in Ar at 600°C, and (d) annealed in Ar at 800°C.](image-url)
oxygen vacancies and Ni$^{2+}$ species yield a greater overall area occupied by the BMPs, thus activating more Ni$^{2+}$ and oxygen vacancies to migrate into the ferromagnetic domains and enhancing the RTFM of the film [23].

Annealing in the Ar environment at 600 °C has a similar effect on the $M_s$ of the film. It was reported that the increased structural defects after annealing in Ar, such as oxygen vacancies, were considered as a reason for the increased $M_s$ [23], but this work shows that the higher $M_s$ can be attributed to the increased metallic Ni nanoclusters. The metallic Ni at the grain boundaries is increased after annealing in Ar at 600 °C according to the XPS and XRD results. More metallic Ni nanoclusters are formed at the grain boundaries by the aggregation of the increased metallic Ni, which enhances $M_s$. The two distinct strengthening mechanisms on $M_s$ after annealing in air and Ar at 600 °C further identify that the RTFM is from metallic Ni, Ni$^{2+}$ or both with two distinct mechanisms in this work.

An even higher $M_s$ is observed in the film annealed in the Ar environment at 800 °C compared to that at 600 °C. On one hand, annealing at a higher temperature in Ar induces a more complete reduction of Ni$^{2+}$ and a more production of metallic Ni. On the other hand, according to the XPS measurement, Ni diffuses from the bottom to the surface of the film during annealing in Ar at 800 °C, leading to a higher concentration of metallic Ni near the film surface. Thus, more metallic Ni aggregates at the grain boundaries near the top surface of the film, which promotes the formation of more and larger metallic Ni clusters. The more and larger metallic Ni clusters enhance the $M_s$ of the film annealed at 800 °C compared to that at 600 °C.

Room temperature PL spectra of the Ni-doped ZnO thin films both as-deposited and annealed are shown in Fig. 6. The films as-deposited and annealed in air or Ar at 600 °C show very weak ultraviolet (UV) peaks centered at about 380 nm (3.26 eV). The UV emission is attributed to the near band-edge excitonic emission. Interestingly, a significant strengthening in the intensity of UV emission peak is observed in the film annealed in Ar at 800 °C. In addition, this UV emission peak is centered at about 377 nm, which exhibits a blue shift of 3 nm compared to the 380 nm observed from the others. Usually, Ni doping in ZnO may result in a weaker peak and a red shift of UV emission [41]. The stronger UV emission and its blue shift of the film annealed in Ar at 800 °C show the depletion of Ni in the ZnO matrix. However, the total Ni concentration in the film is constant, thus it is a local depletion of Ni in the ZnO matrix near the bottom of the film due to the diffusion of Ni from the bottom to the top surface of the film, which has been confirmed by the XPS measurement. As a result, the pure ZnO left at the bottom causes the stronger UV emission and its blue shift. Thus, the annealing in Ar at 800 °C simultaneously restores the optical property of the Ni-doped ZnO thin film when it enhances the magnetic property of the film.

4. Conclusion

XPS depth profiles were used to investigate the effect of rapid thermal annealing under varying conditions on the structural, magnetic and optical properties of Ni-doped ZnO thin films. Oxidation of metallic Ni atoms occurred in the film annealed in air at 600 °C, while reduction of Ni$^{2+}$ ions occurred in the film annealed in Ar at 600 and 800 °C. Significant diffusion of Ni from the bottom to the top surface of the film during annealing in Ar at 800 °C was observed. Both the as-deposited and annealed thin films displayed obvious room temperature ferromagnetism (RTFM) that originated from metallic Ni, Ni$^{2+}$ or both with two distinct mechanisms. Furthermore, a significant improvement in saturation magnetization ($M_s$) in the films was observed after annealing. The increase in $M_s$ in the film annealed in air at 600 °C was attributed to a stronger bound magnetic polaron (BMP) effect. A similar improvement in $M_s$ in the film annealed in Ar at 600 °C was attributed to increased metallic Ni nanoclusters. An even more significant $M_s$ was observed from the film annealed in Ar at 800 °C compared to that at 600 °C, which was attributed to more and larger metallic Ni clusters at the grain boundaries near the film surface mainly due to the diffusion of Ni in the film. A significant strengthening in the intensity and a blue shift of the ultraviolet emission peak were observed in the film annealed in Ar at 800 °C, which was attributed to the local depletion of Ni in the ZnO matrix near the bottom of the film due to the
diffusion of Ni in the film. Thus, the annealing of the Ni-doped ZnO thin film in Ar at 800 °C simultaneously restored the optical property of the film when it enhanced the magnetic property of the film.

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