Materials Research Express

OPEN ACCESS

Near-infrared emission in Er:ZnO thin films with embedded Si nanoparticles synthesized by ion implantation

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Keywords: silicon nanoparticles, ion implantation, photoluminescence, zinc oxide, thin film

Abstract

Er-doped ZnO thin films on a SiO$_2$/Si substrate were fabricated by radio frequency magnetron sputtering, in which embedded Si nanoparticles (NPs) were formed by ion implantation and subsequent thermal annealing. The effects of Si NPs on the Er photoluminescence (PL) at 1.54 $\mu$m were investigated. In addition to the typical emission at 1.54 $\mu$m from Er$^{3+}$, a new 1.16-$\mu$m emission peak was also observed after a thermal treatment. Further annealing resulted in shift of emission intensity between the 1.16- and 1.54-$\mu$m luminescence features. The observed Si nanoparticles (NPs) were ~4 nm in diameter. The formation of new components Zn$_2$SiO$_4$ and Er$_2$Si$_2$O$_7$ was also presented in this study. The 1.16-$\mu$m luminescence is attributed to the Si NPs, and the suppression of Si NPs related emission is caused by consumption of Si in the formation of Er silicate and zinc silicide and the energy transfer between Si NPs and Er$^{3+}$. The intensity of Er$^{3+}$ related 1.54-$\mu$m PL can be modulated by the Si NPs fabricated by implantation and optimizing the annealing condition.

1. Introduction

Rare-earth (RE) doped semiconductors have attracted considerable interest in recent years owing to their wide range of applications in the field of optoelectronic devices [1]. The doping of Er ions into semiconductor materials is an effective approach for making them efficient light emitters. Erbium-doped semiconductor materials can be used in optical communication technology as active elements in photonic devices, because Er$^{3+}$ ions emit at 1.54 $\mu$m, which falls within the minimum transmission loss range of silica optical fibers [2]. The planar integrated optical devices, in which all the optical components are fabricated and integrated on a single chip directly, is of the essence to build all-optical communication networks. In many cases, silicon as a substrate allows the integration of optoelectronic devices with other types. Among various semiconductors, ZnO is a good candidate host material for Er doping, for the wide bandgap of ZnO (3.37 eV) reduces energy back-transfer from the emission center to the host which might occur in the case of a small energy difference, and hence gives a high efficiency [3, 4]. Additionally, ZnO has a large exciton binding energy of 60 meV at room temperature, which results in more efficient excitonic emission at room temperature [5, 6]. It has been investigated extensively in the optoelectronic field, such as surface acoustic wave devices, ultraviolet light-emitting diodes, photo detectors and laser diodes, and so on. Moreover, owing to its low phonon frequencies, the multi-phonon de-excitation processes of Er are suppressed leading to a higher relative probability for radiative decay [7]. Therefore, ZnO provides a matrix which supports the coexistence of Er and O, which might induce the formation of a Er$_2$O$_6$ complex that can optically activate Er$^{3+}$ ions. However, the relatively low solubility of Er atoms in ZnO leads to low efficiency of emission. Co-doping of Si, Li, and Yb with Er has been reported as a promising method for achieving higher Er PL efficiency [8–10]. An increase of the excitation is possible in Er:ZnO systems by doping those elements in the form of very small aggregates and/or nanoclusters. The dopant atoms as sensitzers for Er luminescence increases the excitation cross-section owing to the broad band excitation at wavelengths not absorbed by Er. Sub-nanometer-sized aggregations of Si atoms (Si NPs) seem to be the best sensitization of the
RE although the mechanisms of energy-transfer between the sensitizing atoms and the RE atoms are still not completely understood [11].

Among various doping methods, ion beam technology offers an alternative approach for the doping of Er:ZnO. The direct implantation of Si ions can be used to synthesize NPs with spherical geometries in dielectrics. By the use of swift heavy ion irradiation, the nanostructures can be reshaped, thus elongating the spherical NPs in the thin films [12–15]. The concentration of NPs can be controlled by the ion dose. Ion implantation offers precise control over both lateral and depth concentration profiles; however, many defects are incorporated into the material, which dominate the material properties after the implantation process. The majority are Frenkel pair vacancy and interstitial defects of both zinc and oxygen created by the elastic knock-off processes in the implantation cascade. Such defects are usually removed by annealing procedures. Many studies have also shown that annealing treatments can greatly influence the PL properties of Er:ZnO film [16, 17]. Most studies on silicon NPs have focused on glass substrates, whereas, the investigation of their synthesis and applications in Er:ZnO has been limited. In this work, we systematically study the morphology and emission properties of the Er, Si co-doped ZnO thin films on SiO2/Si substrates prepared by magnetron sputtering and ion implantation. This study reveals that annealing conditions greatly affect the Er3+ ions PL properties. An interplay between the emission features at 1.16 and 1.54-μm during the post synthesis annealing process has been observed. The mechanism that controls the intensities of these PL features is also discussed in detail.

2. Experimental

300-nm SiO2 film on n-type Si (100) grown by Plasma-enhanced chemical vapor deposition (PECVD) were used as substrates. Er:ZnO thin films were deposited on SiO2/Si by radio frequency magnetron sputtering. The target was ZnO:Er2O3 with 0.6 at% Er2O3. Part of the sample surface is covered by aluminium foil before ZnO film deposition. The thickness of the thin films is determined by measuring the step height between the deposited and covered part using Profilometer. The vacuum pressure in the chamber was ~10−4 Pa. During the deposition, the substrate temperature and the gas pressure in the chamber were held at room temperature (RT) and 0.5 Pa, respectively. The ratio of argon to oxygen was maintained at 10:4 (sccm) and the deposition time was 5 h. The Si doping was performed by ion implantation at RT with a 7°-tilt to normal direction of substrate to avoid channel effects. The implantation energy was 120 keV corresponding to a depth of ~100 nm simulated by the stopping and range of ions in matter (SRIM) [18, 19]. The implantation doses were 4 × 1015 ions cm−2, 1.4 × 1016 ions cm−2, and 2.5 × 1016 ions cm−2 (samples implanted with doses from low to high are labeled as 1, 2, and 3). The samples were then respectively annealed in a furnace at 600, 800, 900, and 1000 °C in an O2 atmosphere.

The surface roughness was measured by atomic force microscope (AFM). The crystal structures were examined by X-ray diffraction (XRD) with Cu Kα radiation (λ = 0.15406 nm). The XRD peak assignments were determined according PDF cards in MDI jade software. The ultraviolet (UV)/visible and near-infrared (NIR) emissions were measured at RT with the use of a 325-nm He-Cd laser and 532-nm laser as the excitation sources. The laser beam was chopped through a modulator at a frequency of 400 Hz. The infrared emission was detected by a thermoelectrically cooled InGaAs detector and all the spectra were recorded by a lock-in amplifier where the chopping frequency was used as a reference. The structural characteristics of the films were studied by high-resolution transmission electron microscopy (HR-TEM; Tecnai G2 F20 S-Twin microscope, FEI, Eindhoven, Netherlands) in cross-sectional images.

3. Results and discussion

The film thickness is 575 nm after 5 h of deposition. The concentration of Er ions is around 0.5 at%, which is close to the component of target (Er ∼0.6 at%). To study the effect of annealing on the film surface morphology, we analyze the AFM images of the as-deposited Er:ZnO film and films implanted by Si with doses from 4 × 1015 to 2.5 × 1016 ions cm−2 after annealing at 1000 °C. Sample 1 annealed at 900 °C is also shown for comparison. As shown in figure 1, considerable variation of the surface roughness can be observed in these films. The root-mean-square (RMS) surface roughness values of the samples are 15.6, 33.8, 26.4, 44.7, and 34.9 nm, as shown in figures 1(b) to (f). The values of RMS for the Si implanted samples are higher compared with those of the as-deposited films. The surface roughness is approximately increased with ions dose. The high RMS surface roughness of the implanted and annealed samples is attributed to the formation of ZnO grains and Si nanoclusters, as shown in the following TEM images. The increased surface roughness also originates from the residual stress induced by ions implantation.

As shown in figure 2(a), the peak of Si distribution is at a depth of 100 nm below the surface when samples are implanted with 120 keV Si ions. The concentration peaks of Si range from 0.44 to 2.75 at% for samples 1–3 at
different implantation doses. The full width at half maximum (FWHM) of the plot is approximately 110 nm. Therefore, the implanted Si is mainly located in this narrow-buried region compared with the whole thickness of the films (575 nm). The films grown at RT without ion implantation show poor crystallinity in the XRD patterns [figure 2(b)]. After implantation the quality of crystallinity increases with the ion dose. Films with the highest dose of ion implantation (sample 3) show the best crystallinity. Beam heating occurs during the Si implantation process and the kinetic energy of implanted ions is mostly turned into heat [20]. As a result, high implantation doses increase the sample temperature, which improve the crystallinity.

Figure 3 shows the PL spectrum of as-deposited and implanted samples prior to annealing. Although the as-deposited film has poor crystallinity in the XRD pattern, the sample shows the strongest intensity of the 1.54-μm emission. We attribute the decreased emission of as-implanted samples to the introduction of Si atoms by ion implantation in this study. Elastic collisions between the incident Si ions and the target atoms occurs during ion implantation. As a result, many defects/defect clusters are created in the implanted layer and the film is re-

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**Figure 1.** (a) Schematic diagram of the Si ions implantation process; (b) AFM images of as-deposited Er:ZnO film; Si-implanted films with different dose and annealing at 1000 °C (c) sample with 4 × 10^{15} ions cm^{-2}, (d) 1.4 × 10^{16} ions cm^{-2} and (e) 2.5 × 10^{16} ions cm^{-2} Si; (f) sample with 4 × 10^{15} ions cm^{-2} annealed at 900 °C.

**Figure 2.** (a) Distribution of incident Si ions calculated using SRIM for sample implanted with dose of 2.5 × 10^{16} ions cm^{-2} at 120 keV and (b) XRD patterns of as-deposited and Si ions implanted films prior to annealing.
structured in this process. Hence, the intensity of 1.54-μm emission decreases and that of band edge and defect-related emission increase (as discussed below). This result implies that the efficiency of energy transfer from ZnO to Er is suppressed by Si ions implantation and more detailed illustration is shown in the UV/visible PL spectra part [21, 22].

When Er ions are indirectly excited, there are three processes leading to the 1.54-μm emission, which are the absorption of excitation energy by the Si ions implanted ZnO host, the energy transfer from Si ions implanted ZnO to the Er ions, and the radiative relaxation in the Er ions. To analyze the relationship between annealing temperature and NIR PL intensity, the PL spectra of samples firstly annealed at 600 °C and then further annealed at 800 °C are given in figure 4. A new 1.16-μm emission was detected after the 600 °C annealing treatment. After 30 min annealing at 600 °C under an oxygen atmosphere, the intensity of 1.54-μm emission markedly increased compared with that of the as-implanted film for sample 2 and 3. The increased NIR PL intensity is owing to the reduction of defect density, which dissipates the Er pump efficiency [23]. Further thermal treatment at 800 °C decreases the emission at 1.16 μm and increases the Er 1.54-μm emission. Figure 4 illustrates the balance between the 1.16 and 1.54 μm emission in the annealing process. The 1.16-μm emission is attributed to Si NPs [24–26]. This Si NPs related PL is inhibited after 800 °C annealing owing to Si combining with other elements to form zinc silicide [27]. Furthermore, when Si NPs grow to an appropriate size, the 1.16-μm emission is suppressed and energy transfer between Si NPs and Er^{3+} ions occurs [28, 29]. These observations are consistent with our earlier suggestions that the 1.54-μm PL depends on the size distribution of the formed Si-NPs, and on their coupling with Er ions. In our previous and other studies, a 1.16-μm emission has also been observed from a sample without Si implantation [30, 31]. In those cases the 1.16-μm emission derives from Si diffusion from the substrate into the ZnO film. In this study the Si related 1.16-μm emission derives from both the implantation and diffusion of Si from the substrate.

Figure 3. PL spectra of as-deposited and Si ions implanted Er:ZnO films in NIR region excited with 532 nm laser lines.

Figure 4. (a) NIR PL spectra of samples annealed at 600 °C; (b) PL spectra of samples annealed at 800 °C excited with 532 nm laser lines.
The XRD patterns of the samples are presented in figure 5(a). After annealing at 1000 °C, all the films exhibit similar XRD spectra, whereas the (002) peak is dominant. Several peaks appear, which are related to Er compounds. As shown in this figure, all the samples show improved crystalline properties with a hexagonal wurtzite phase ZnO (002). The crystalline orientations of (100) and (101) are also detected in sample 3 implanted with a high dose of Si. This result can be explained by the ion implantation induced lattice distortion and strain. The insertion of a high concentration Si and aggregation of Si after annealing results in lattice distortion. The polycrystalline of ZnO layer is confirmed by the electron diffraction pattern (shown in TEM part). A new component with a Er$_2$Si$_2$O$_7$ phase (122) reveals the formation of a Er silicate; however, there is a decline in the 1.54-μm PL intensity of the 1000 °C annealed sample. Previous studies have indicated that a mixed crystal of ZnO and Zn$_2$SiO$_4$ forms in films sputtered on silicon even at post annealing temperatures higher than 800 °C. Short time annealing at 1000 °C markedly suppresses the 1.16 μm PL, which can be attributed to both Si depletion in the formation of Zn$_2$SiO$_4$ and Er$_2$Si$_2$O$_7$ and energy transfer between Si NPs and Er$^{3+}$ [32]. The decrease of the 1.16-μm emission does not result in an increase of the 1.54 μm PL intensity. The degraded optical activity of the Er$^{3+}$ ions induced by the high temperature treatment is consistent with previous studies [33]. Annealing at 1000 °C for 5 min also changes the local environment around Er. An intense Stark splitting was detected for the 1.54 μm PL, as shown in figure 5(b).

Figure 6 shows the UV and visible PL spectra of as-deposited samples and samples with Si ion implantation annealed at 1000 °C excited with a 325 nm laser lines.

The XRD patterns of the samples are presented in figure 5(a). After annealing at 1000 °C, all the films exhibit similar XRD spectra, whereas the (002) peak is dominant. Several peaks appear, which are related to Er compounds. As shown in this figure, all the samples show improved crystalline properties with a hexagonal wurtzite phase ZnO (002). The crystalline orientations of (100) and (101) are also detected in sample 3 implanted with a high dose of Si. This result can be explained by the ion implantation induced lattice distortion and strain. The insertion of a high concentration Si and aggregation of Si after annealing results in lattice distortion. The polycrystalline of ZnO layer is confirmed by the electron diffraction pattern (shown in TEM part). A new component with a Er$_2$Si$_2$O$_7$ phase (122) reveals the formation of a Er silicate; however, there is a decline in the 1.54-μm PL intensity of the 1000 °C annealed sample. Previous studies have indicated that a mixed crystal of ZnO and Zn$_2$SiO$_4$ forms in films sputtered on silicon even at post annealing temperatures higher than 800 °C. Short time annealing at 1000 °C markedly suppresses the 1.16 μm PL, which can be attributed to both Si depletion in the formation of Zn$_2$SiO$_4$ and Er$_2$Si$_2$O$_7$ and energy transfer between Si NPs and Er$^{3+}$ [32]. The decrease of the 1.16-μm emission does not result in an increase of the 1.54 μm PL intensity. The degraded optical activity of the Er$^{3+}$ ions induced by the high temperature treatment is consistent with previous studies [33]. Annealing at 1000 °C for 5 min also changes the local environment around Er. An intense Stark splitting was detected for the 1.54 μm PL, as shown in figure 5(b).

Figure 6 shows the UV and visible PL spectra of as-deposited samples and samples with Si ion implantation and annealing. The PL curves for implanted Er:ZnO films show an intense near-band-edge (NBE) and defect-related deep level emission. The strong UV emission centered at 375 nm is attributed to recombination of excitons. The ZnO films also exhibit broadband emission in the visible range, centered around the green and extending to yellow and orange regions. This visible emission originates from transitions of deep-level states into the gap of
ZnO, which are associated with the defects such as Zn and O vacancies, and are thus denoted as deep-level emission (DLE). For films with Si ion implantation, the strong emission in the UV and visible region is observed in figure 6. After implantation, these non-radiative-related defects could be reduced and re-structured. However, the implantation would also reduce the traps for non-radiative transition. Consequently, the visible light emission associated with structural defect is enhanced. Such reducing and re-structuring made the film structure more perfect supported by the results of the XRD characterization shown in figure 2(b) and lead to a decrease in the non-radiative transition probability. Accordingly, the NBE and DLE light emission is enhanced markedly by the implantation. The intensity of the UV emission band to the DLE band is regarded as an indicator of the crystallinity of ZnO materials [34]. Therefore the strong UV/visible emission for implanted samples is resulted from defects induced by the implantation. Even after annealing at 1000 °C, the defects still remain in the ion implanted samples. Generally, better crystal quality helps to reduce exciton scattering, thereby increasing the exciton emission probability. Compared with the implanted sample spectra, the spectrum of as-deposited ZnO:Er has slight band edge emission and defect related emission, but higher 1.54-µm emission intensity. Several papers have reported that the ZnO crystallinity correlates with PL emission intensity, because ZnO can absorb excitation energy from pumping light and transfer energy to Er ions [35]. When the energy transfer has taken place, we can observe the reduction of the host emission followed by an increase of the Er emission. Thus the low intensity of the band edge and defect emissions but high 1.54-µm emission (see figure 3) in as-deposited samples is due to highly efficient energy transfer from ZnO to the Er dopants [36, 37].

The structure of a film subjected to 1.4 × 1016 ions cm−2 Si ion implantation and then annealed at 1000 °C is shown in the TEM images of figure 7. The ZnO layer can be divided into two parts [figure 7(a)], namely the implantation region and the bulk. The near surface region has a loose structure, which is induced by high temperature annealing. In the implantation region a mixed polycrystalline and amorphous state is observed [figures 7(b)–(d)]. Figures 7(b)–(d) are HR-TEM images of selected region marked as ① and (e), (f) are HR-TEM images of region marked as ② in figure 7(a). The Si NPs with a size ∼4 nm can be observed in figure 7(c) and (d). A ∼200 nm polycrystalline ZnO bulk layer near the SiO2 buffer film is shown in figures 7(e) and (f). The polycrystalline state of the interface is confirmed by the electron diffraction pattern, as shown in figure 7(g). In figure 7(h) nanocrystals with a size ∼3–5 nm are observed in the SiO2 layer. Previous reports have shown that the diffusion of Zn from the ZnO film into SiO2/Si substrate occurs even at 400 °C annealing [32]. It is reasonable to attribute these nanocrystals in the SiO2 layer to Zn2SiO4 crystals.

As mentioned above, the 1.54-µm PL intensity decreases rather than increases indicating thermal quenching. Previous studies on annealing of Er:ZnO have also reported such thermal quenching behavior [31, 38]. Gourbilleau et al reported that the intensity of the main peak at 1.54 µm increased with annealing temperature, reaching a maximum at approximately 900 °C, and then decreased at higher temperatures [39]. When the temperature exceeds 1000 °C, the 1.54-µm emission becomes weak owing to the precipitation of Er and the growth of Si NPs. Thus, the decrease of optically activated Er concentration and the number of Si NPs in the Er:ZnO layer results in the decreased intensity of the 1.54-µm emission. Although energy transfer between Si NPs and Er3+ ions might enhance the 1.54-µm emission, the increase in its intensity can be neglected considering that the Si NPs are located at a narrow region compared with the whole thickness of the film and the low coupling efficiency between them. Si NPs larger than 5 nm appear ineffective for resonant excitation of Er reported in previous study [38]. The direct no-phonon transition probability between Si and Er is closely correlated to the energy transfer rate, decreasing when Si-NPs size increases. For large Si-NPs with a bandgap lower than 1.26 eV, the energy transfer is no longer possible, leading to the observed decrease of the 1.54-µm emission. Therefore increasing the Er, Si content and implantation under high temperature are possible pathways to fulfill the energy transfer between the Si and Er for enhancing 1.54-µm emission. As is known, Si nanocluster size and the interaction distance of carrier-Er are very important to efficient energy transfer and enhancing Er emission. High concentration of Er or Si can reduce the distance between them. When implanted at high temperature Si NPs can be formed directly in the implantation process. High temperature annealing and new phases can be prevented. More work will be done afterwards. Although no enhancing of Er ions’ PL at 1.54 µm is observed in this study, embedded Si NPs in Si ions implanted samples and suppression of the Si NPs’ 1.16-µm emission by annealing is achieved. This result offers valuable reference for Si-based film growth and device fabrication, especially for those where Er ions’ PL (1.54 µm) is needed.

4. Conclusions

The Si doped Er:ZnO films have been synthesized by ions implantation. The emission and morphology properties of Si ions implanted Er:ZnO thin films is studied. A new 1.16-µm PL peaks is observed in implanted samples. Si NPs with a size of ∼4 nm in diameter are formed in the implantation region. This 1.16-µm PL band is attributed to the Si NPs. It was found that the correlation between 1.16-µm and the Er3+ emission. 1.54-µm PL
bands are competing with each other. A comparison of the Er:ZnO films between 600 and 800 °C showed that the 1.54-μm emission of Er$^{3+}$ increased at the expense of Si related emission. Considerable suppression of the 1.16-μm emission was induced by annealing at high temperature owing to the formation of Er silicate, zinc

Figure 7. Cross-sectional TEM images of Er:ZnO film implanted by Si with a dose of $1.4 \times 10^{16}$ ions cm$^{-2}$ and annealed at 1000 °C; (a) Structure of the whole layers; (b)-(d) HRTEM micrograph of the selected regions around implantation range; (e) and (f) selected region near the SiO$_2$ buffer layer; and (g) corresponding electron diffraction pattern of Er:ZnO films at the interface; (h) HRTEM from SiO$_2$ layer.
silicide, and energy transfer between NPs and Er$^{3+}$. The new component Zn$_2$SiO$_4$ and Er$_2$Si$_2$O$_7$ are also formed in high temperature annealed samples. The decrease of the 1.54-µm emission after high temperature treatment is mainly related to the detrimental effects induced by the higher annealing temperature, such as the aggregation of Er and the degenerated coupling between Er$^{3+}$ and Si NPs.

### Funding

This work was supported by the National Nature Science Foundation of China (Grant No. 6197504, 81974467, 81800156), Natural Science Foundation of Shandong Province (Grant No. ZR2019MH136, ZR2017BH024) and Postdoctoral Research Foundation of China (Grant No. 2019M652356).

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