Journal of the Korean Vacuum Science & Technology; JKVST
Vol.6, No.2, 2002, pp.101 ~ 104

Microstructure and Properties of Er-SiOX Films Synthesized by Ion Beam Assisted Deposition

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(Received)

Abstract

Er doped SiO_x films have been synthesized by ion beam assisted deposition (IBAD). The morphology and microstructure of films and their annealing behaviors have been examined by using scanning electron microscopy and x-ray diffraction. The composition and properties of films have been systematically investigated.

1. Introduction

Er-doped films have attracted much attention after the success of the Er-doped fiber amplifier [1-3] because of their potential applications in integrated optics. Erbium is used as optical dopant because the transition of trivalent erbium from the first excited state to the ground state in Er^{3+} occurs at the energy of 0.8 eV, corresponding to a wavelength of 1.54 μm, where standard silica-based optical fibers have their maximum transparency. Er-doped SiO_x films can be possibly applied in silicon-based optoelectronics. Planar optical devices, such as optical amplifiers, waveguide lasers or more complicated optical integrated circuits might be fully compatible with silicon technology and are of particular interest.

In order to optimize Er^{3+}-doped planar optical waveguides, an extensive range of film materials has been used as the host for the dopant, including silica glasses, Al_2O_3, Y_2O_3, SiO_2, $BaTiO_3$ and $LiNbO_3$. In the mean time, various syntheses or doping methods, such as high-energy ion implantation, plasma-enhanced chemical vapor deposition, metal organic chemical vapor deposition, and sol-gel method [4-6], have been used to fabricate Er-doped planar optical waveguides. In the present report, we synthesized Er-doped SiO_x films by Ion beam assisted deposition (IBAD). We have studied the microstructures of the films and their annealing behaviors by using x-ray diffraction (XRD). The photo luminescence of Er-doped SiO_x thin films and their annealing behaviors, have also been investigated.

2. Experimental Details

2.1 Technology of film Deposition

The deposition of Er-doped SiO_x films was carried out in an IBAD system as shown in Fig. 1, where the films can be synthesized by sputter deposition of silicon in an oxygen atmosphere and bombarded simultaneously by low energy oxygen ions. Er was doped in SiO_x thin film by using metal erbium rods embedded into aluminum slices. The number of erbium rods embedded in the target controlled the concentration of erbium in the film. In this work, the diameter of erbium rods was 2 mm and the sputter area of erbium

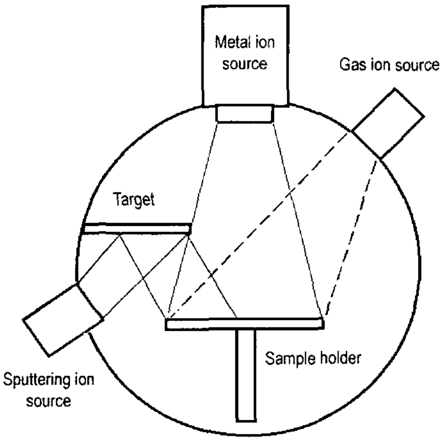

Fig. 1. Schematic drawing of IBAD facility used.

is <5 % of target area. This combining target can control the concentration of erbium lower than 1.0% in the SiO_x films.

Er-SiO_x thin films were deposited on Si (100) substrates and optical glasses. The silicon wafers were cleaned by the standard cleaning method. The sputtering ions were Ar ions with energy of 3 keV and beam current of 50 mA. The bombardment of oxygen ions was 500 eV and 50 mA. The base vacuum of the system was in the order of 10^{-3} Pa. Before deposition, Ar ion sputtering was used to clean the surfaces of samples for 15 minutes. During deposition, the ratio of oxygen flows to argon flow was kept at 2.2 and the working press was kept at 3.0×10^{-2}Pa. The substrate temperature was controlled at 500℃ by using a heater.

2.2 Structure and properties characterization

The crystal structure of films and their annealing behaviors were determined by XRD-6000. JOEL JSM-5600LV scanning electron microscopy (SEM) with energy dispersion x-ray spectrum (EDX) and EPMA-1600 were used to determine the thickness and composition of the films. The photo luminescence (PL) spectrum of Er-doped SiO_x films pumped by 980 nm laser were measured at room temperature.

3. Results and Discussion

3.1 Microstructures and annealing behaviors of Er-SiO_x films

The photoluminescence spectrum of Er-doped materials is sensitive to the thermal annealing processing. Especially for ceramic oxides and semiconductors, annealing treatment is always necessary to obtain optically active Er^{3+} [4]. Apparently, the microstructures and bonding nature of optical waveguides influence the environment and the active configuration of the Er. In this work, we investigated the influence of annealing temperature on the microstructures of Er-doped SiO_x.

Figure 2 shows the SEM cross-section image and

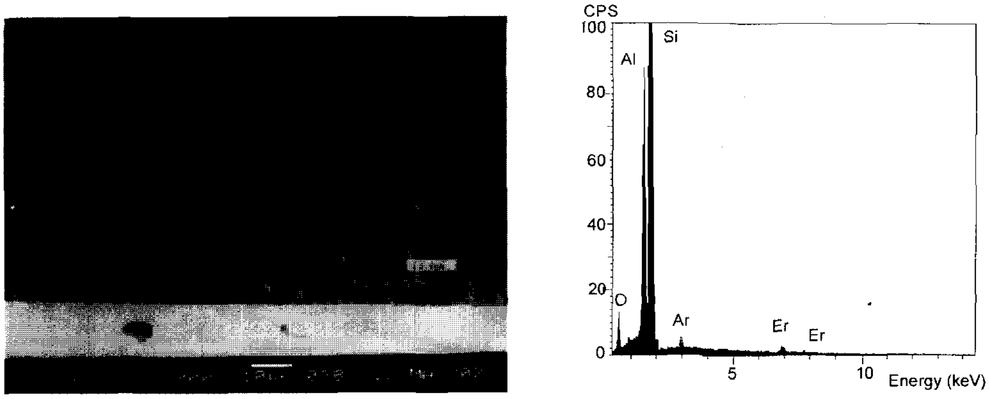

Fig. 2. SEM cross-section image and EDX spectrum of Er-doped SiOx film as deposited for 6 hours.

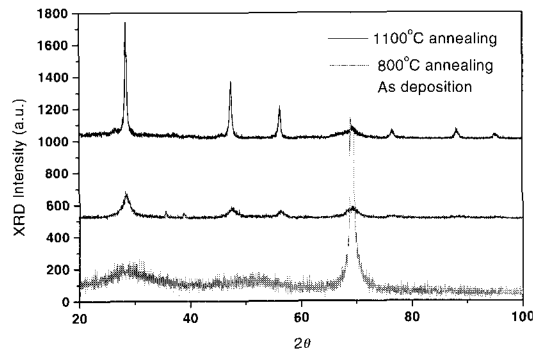

Fig. 3. XRD spectra of Er-doped SiO_x films as deposited and annealed at different temperatures.

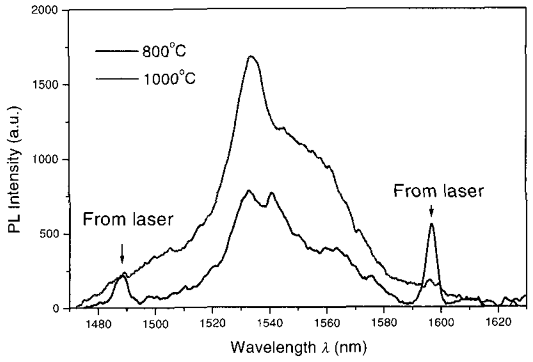

Fig. 4. PL spectra of Er-doped SiO_x films pumped with 980 nm laser.

EDX spectrum of Er-doped SiO_x film as deposited for 6 hours by IBAD. The film thickness is about 10-20 μm for different deposition position. In the figure, Er-doped SiO_x film deposited by IBAD is dense and flat. The composition of Er-doped SiO_x film determined by electron probe is O: 18.89 at.%, Al: 12.37 at.%, Si: 68.44 at.% Er: 0.30 at.%. Fig. 3 shows the XRD spectra of Er-doped SiO_x films annealed at different temperatures. In the figure, we can see that the films as deposited by IBAD at 500℃ are typical amorphous. After annealing at 800 for 2 hours, the films become to polycrystal films. By indexing the XRD spectrum, we find the films are pure silicon in the grain size of nanometer scale. For the case of samples annealing at 1100℃ for 2 hours, the microstructure of the films is still dominated by pure silicon and the grains grow up considerably.

3.2 PL luminescence of Er-SiOx films

Figure 4 is the PL spectra of Er-doped SiO_x films annealed at 800℃ and 1100℃. The measurement of PL spectra was carried out with reflection mode and pumped by 980 nm laser. In the figure, we can see that the PL spectra are typical Er^{3+} luminescence corresponding to the $^4I_{13/2} \rightarrow {}^4I_{13/2}$ transition in the 4f-shell of erbium ions for Er-doped SiO_x films annealed at 800℃ and 1100℃. However, no PL spectrum was detected for Er-doped SiO_x films as deposited at 500℃. In the PL spectrum of Er-doped SiO_x films annealed at 800℃ with reflection mode, two narrow lines from 980 nm laser were also detected. In the comparison between the PL spectra of Er-doped SiO_x films annealed at 800℃ and 1100℃, we found the PL intensity of Er-doped SiO_x films annealed at 800℃ is much lower than that annealed at. 1100℃. The result means that there are more active Er^{3+} in Er-doped SiO_x films annealed at 1100℃ than that annealed at 800℃.

Figure 5 shows the change of PL intensity of Er-doped SiO_x films with laser power after the background subtraction. As can be seen in the figure, The PL

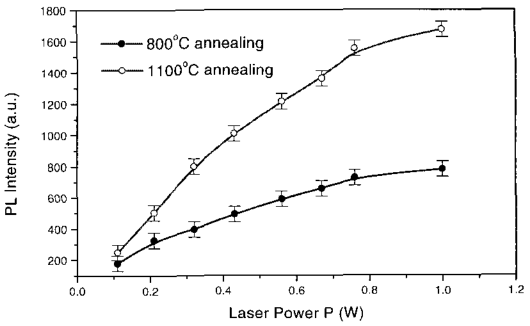

Fig. 5. Change of PL intensity of Er-doped SiO_x films with laser power.

intensities of Er-doped SiO_x films annealed at 800℃ and 1100℃ increase with the increase of pump laser power. No obvious power saturation was observed up to 1 W for both films annealed at 800℃ and 1100℃.

4. Conclusions

According to investigation on Er-doped SiO_x films synthesized by IBAD, the conclusions can be given as following:

(1) Er-doped SiO_x films are amorphous as deposited at 500℃, and change into nano-crystalline after 800℃ annealing and ploycrstalline after 1100℃ annealing;
(2) No PL spectrum has been observed in Er-doped SiO_x films as deposited at 500℃. The PL intensity of Er-doped SiO_x films annealed at 1100℃ is stronger than that annealed at 800℃;
(3) No obvious saturation of PL intensity was observed in Er-doped SiO_x films pumped by 980 laser with the power up to 1 W.

Acknowledgements

This work is supported by the National Natural Science Foundation of China under grant no. 69878002 and by the Education Ministry of China. The authors thank Prof. C.L. Song and his students in Dept. of physics, Dalian University of Technology, for the helps in the measurements of PL spectra.

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