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Preparation and photoluminescence of Er^{3+}-doped Al_2O_3 films by sol–gel method

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Abstract

The 0–1.5 mol% Er^{3+}-doped Al_2O_3 films have been prepared on the thermally oxidized SiO_2/Si(100) substrate in the dip-coating process by the sol–gel method, using the aluminium isopropoxide $[Al(OC_3H_7)_3]$-derived γ-AlOOH sols with the addition of erbium nitrate $[Er(NO_3)_3 \cdot 5H_2O]$. The continuous Er^{3+}-doped Al_2O_3 films with the thickness of about 1.2 μm were obtained for nine coating cycles at a sintering temperature of 900 °C. The aggregate size for the Er^{3+}-doped Al_2O_3 films increased with increasing the Er^{3+} doping concentration from 0 to 1.5 mol%. The root-mean-square roughness of the films was independent on the Er^{3+} doping, which was about 1.8 nm for the 0–1.5 mol% Er^{3+}-doped Al_2O_3 films. The γ-Al_2O_3 phase with a (110) preferred orientation was produced for the Al_2O_3 film. The photoluminescence (PL) spectra of 0.1–1.5 mol% Er^{3+}-doped Al_2O_3 films were observed at the measurement temperature of 10 K. There was no significant change for the PL peak intensity with the increase of Er^{3+} doping concentration from 0.1 to 1.5 mol%, and similar full width at half maximum of about 40 nm was detected for the 0.1–1.5 mol% Er^{3+}-doped Al_2O_3 thin films. The Er^{3+}-doped Al_2O_3 films possess the available PL properties for use in planar optical waveguides.

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Keywords: Al_2O_3 films; Sol–gel method; Er^{3+} doping; Photoluminescence

1. Introduction

The sol–gel technique is a low-temperature route widely employed to prepare thin films for use in the different fields, because it can offer the homogeneous thin films at molecular scale and control of chemical purity. In recent years, great interest was devoted to the preparation of thin films for optical applications [1,2], and especially elaboration of planar waveguides doped with the active elements such as rare-earth ions [3,4]. Among the rare-earth ion-doped planar waveguides, Er^{3+} doping in different matrix materials prepared by the sol–gel method has attracted much attention following the success of the Er^{3+}-doped fiber amplifier, because Er^{3+} processes the intra-4f transition around 1.53 μm, coinciding with the low-loss window of standard silica-based optical fiber [5]. The oxides of groups III and IV elements have been used as the Er^{3+}-doped planar waveguides matrix material by the sol–gel method, including SiO_2 [6], Al_2O_3 [7], TiO_2 [8], In_2O_3 [7], etc.

Al_2O_3 is an interesting matrix material used in the planar waveguides, because the relatively high refraction index of Al_2O_3 (n=1.64) waveguides cladded with SiO_2 (n=1.45) results in high confinement of the optical mode in the waveguides, leading to efficient pumping and amplification. In addition, the high contrast of refraction index allows for the use of small device structures possible. Therefore, the Er^{3+}-doped Al_2O_3 planar waveguides films have been successfully prepared by the different techniques, including ion implantation [9], pulsed laser deposition [10], plasma-enhanced chemical vapor deposition [11], etc. Gaponenko et al. [7] first fabricated the 1.4 mol% Er^{3+}-doped Al_2O_3 films by the

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sol–gel method, using the aluminium hydroxide-derived boehmite (γ-AlOOH) sols with the addition of erbium nitrate [$Er(NO_3)_3 \cdot 5H_2O$], and the photoluminescence (PL) spectrum at 1.53 μm was observed in the excitation with the 514.5 nm line of an argon ion laser. However, the characterization of the Er^{3+}-doped Al_2O_3 films and the effect of Er^{3+} doping concentration on PL properties of the Er^{3+}-doped Al_2O_3 films were not reported systemically. The luminescence mechanism of the Er^{3+}-doped Al_2O_3 material by the sol–gel method is not clear so far. In this paper, we present the experimental results systemically about the surface morphology, phase structure and photoluminescence properties of Er^{3+}-doped Al_2O_3 films on the thermally oxidized SiO_2/Si(100) substrates by the sol–gel method, in order to estimate the possibility and further spread to their application in the planar waveguides.

2. Experiment

The sol–gel technique was performed by hydrolyzing the aluminium isopropoxide [$Al(OC_3H_7)_3$] at 90 °C under reflux and strong agitation for 2 h with a molar ratio of 110:1 for distilled water and $Al(OC_3H_7)_3$ [12]. The reaction container was open in order to remove the isopropanol [$(CH_3)_2CHOH$] produced by hydrolysis reaction under strong agitation at 90 °C. Nitric acid (HNO_3) was added as catalyst to a molar ratio of 0.15:1 for H^+/Al^{3+}. After the reaction continued under reflux and strong agitation at 90 °C for 16 h, a clear γ-AlOOH sol was obtained. Finally, Er^{3+} was introduced by the addition of $Er(NO_3)_3 \cdot 5H_2O$ with the different molar ratio of (0–0.015):1 for Er^{3+}/Al^{3+}, the different Er^{3+}-doped γ-AlOOH sols were produced.

The different Er^{3+}-doped γ-AlOOH gel films were prepared on the thermally oxidized SiO_2/Si(100) (30×20×0.5 mm) substrates by a dip-coating technique with a uniform withdraw rate of 100 mm/min. The deposited films were firstly dried at room temperature for 1 h and then dried at 100 °C for 10 min. After every third dipping time, the Er^{3+}-doped Al_2O_3 films were obtained at the sintering temperature of 900 °C with a heating rate of 4 °C/min and maintaining at the temperature for 1 h, and cooled down to room temperature in the furnace. Finally, the thickness of the thin films was built up by repeating the cycles (one cycle is composed of dipping, drying and heating process).

The surface morphology and thickness of the Er^{3+}-doped Al_2O_3 films were analyzed using a JEOL JSM-5600LV scanning electron microscope (SEM), operating at 20 kV. The surface morphology was further detected by a Digital Instruments Nanoscope IIIa atomic force microscope (AFM). The AFM images were captured in the Contact Mode using an e-scanner with a maximum scan dimension of 16 μm in the atmosphere. Silicon nitride tips were used, which have a radius of curvature of about 40 nm. The contact force was minimized during scanning by using a set point just above the disengagement point. The height images were captured at a scan rate of 1–5 Hz. The root-mean-square (RMS) roughness values were determined from the AFM images using the Nanoscope IIIa software. The phase structure of the Er^{3+}-doped Al_2O_3 films was investigated using a Rigaku D/Max-3A X-ray diffractometer (XRD) with CuKα radiation. The photoluminescence was performed with a Nicolet 760 Fourier transform spectrophotometer with a resolution of 0.1 cm^{-1}. In the spectrophotometer, the Ar^+ laser line at 514.5 nm with an 80 mW was utilized as the excitation source, the diameter of light spot at the samples was about 100 μm. A liquid nitrogen-cooled Ge detector was used to receive the luminescence response. The PL spectral structure in the 1.400–1.700-μm region was resolved at liquid helium temperature (10 K).

3. Results and discussion

The surface and cross-sectional SEM photographs of Al_2O_3 film for nine coating cycles on the thermally oxidized SiO_2/Si(100) substrate at the sintering temperature of 900 °C are shown in Fig. 1. The flat and smooth surface is

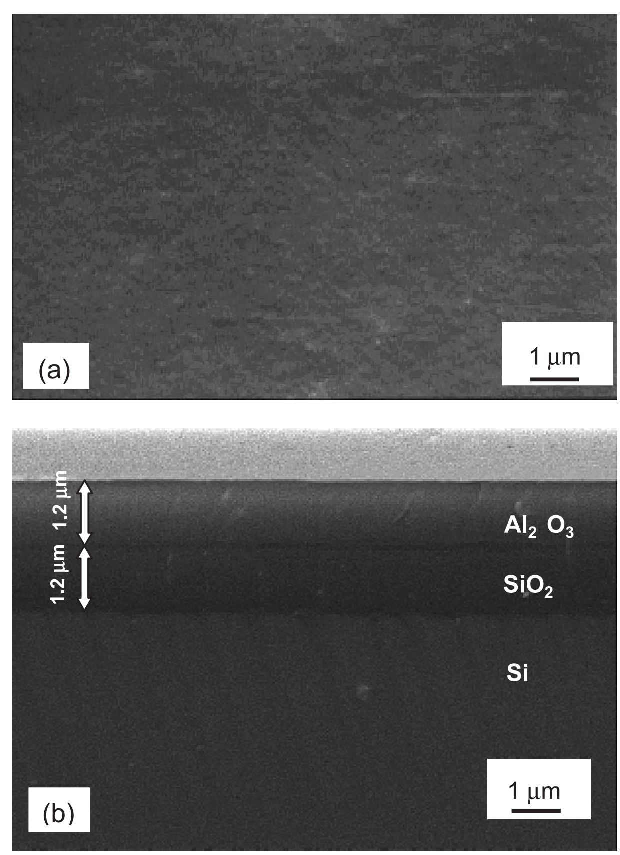

Fig. 1. The surface and cross-sectional SEM photographs of the Al_2O_3 film for nine coating cycles on the SiO_2/Si(100) substrate sintered at 900 °C.