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Er-doped oxidised porous silicon waveguides

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Abstract

The present work reports Er-doped channel oxidized porous silicon waveguides (OPSWG) formed from n⁺-type Si by the two-step anodisation process. Er has been introduced into porous silicon before oxidation by a cathodic treatment in 0.1 M Er $(NO_3)_3$ aqueous solution. A correlation between Er concentration and refractive index profiles has shown dominant core doping with Er relative to cladding regions. Reported Er concentration of 0.8 at.% in the OPSWG is large enough to attain the amplification effect. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

Recently, integrated optical waveguide (WG) amplifiers are growing in interest [1]. If these amplifiers are to be fabricated, WG should be doped with a specific dopant, typical of which is Er. It is essential that the amplifier is: (i) transparent to the both Er exciting wavelength and to the wavelength of the travelling signal; (ii) contains a large amount of optically active Er; and (iii) can be made into a channel WG using a technology compatible with standard Si processing [1]. Integrated WGs based on oxidised porous silicon (OPS) are capable of meeting these criteria [2-4]. First, broad range of light transmission has been found to occur in OPSWGs. Second, porous structure promotes Er introduction into porous silicon (PS) in abundance. A subsequent oxidation of PS makes Er to be optically active. Third, technology of Er-doped channel OPSWGs is compatible with standard silicon microelectronic tech-

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nology. Previously, we reported Er-doped OPSWGs formed from p^+ -type Si by one-step anodisation with electrochemical Er introduction and Er-related photoluminescence from the waveguide formed [5]. In the present work, n^+ -type Si and two-step anodisation were employed to produce Er-doped OPSWGs with advanced optical parameters suitable for optical amplifiers.

2. Experimental

The process sequence to produce a channel Er-doped OPSWG is shown in Fig. 1. The starting (100) n⁺-type Si wafers of 0.01-Ohm \cdot cm resistivity were oxidised to form a 0.7-µm thick masking SiO₂ layer. Using photolithography, the SiO₂ layer was patterned to define layout of waveguides, which were designed as straight lines of width 10 µm and of length 3 cm (Fig. 1a). Selective anodization of Si within openings in the mask was performed in the electrolyte consisted of 48% HF and ethylene glycol in the ratio of 2:3. To provide WG with core and cladding regions of controlled porosities

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Fig. 1. The process sequence to produce an Er-doped channel OP-SWG.

and thicknesses, two-step anodisation process was elaborated. The first anodization step was performed at the current density of 40 mA/cm² for 4 min providing a 7-µm thick PS 1 layer (Fig. 1b). On further oxidation, this layer is expected to form compact OPS for the core region. Then the current density was raised to 80 mA/cm^2 and the samples were anodized for 45 s providing a waveguide periphery with a $3-\mu m$ thick PS 2 layer (Fig. 1c). On the following oxidation step, this layer is expected to form a cladding layer. After the second anodisation step, the remainder of the SiO₂ mask was of $\sim 0.15 \,\mu$ m in thickness. Next, the samples were immediately placed into a 0.1 M $Er(NO_3)_3$ solution and a cathodic treatment was performed for 8 min at the cathodic current density of 50 mA/cm². An Er-containing layer of Er(OH)₃ composition was electrochemically deposited into PS (Fig. 1d). A subsequent etching in 20% HCl removed the Er-containing layer from the surface while the layer within the PS layer remained intact (Fig. 1e). Next, three-step thermal oxidation was performed: (i) at 300°C for 60 min in dry O_2 ; (ii) at 1000°C for 25 min in wet O_2 ; and (iii) at 1150°C for 60 min in dry O_2 (Fig. 1f).

X-Ray microanalysis using an EDX-Spectrometer AN-10000 was performed to study Er distribution within the OPSWG. Photoluminescence (PL) spectroscopy was performed by direct end-fire pumping the waveguides with focused light from an Xe lamp. The PL signal was coupled out by a prism and the transmitted spectrum was analyzed with a grating spectrometer MDR-23 equipped with a liquid nitrogen-cooled Ge detector. The attenuation spectrum was determined from a set of WG transmission spectra recorded at various prism positions. Refractive index of OPS was determined at $0.63 \mu m$ by refractive near field measurement.

3. Results and discussion

Fig. 2 shows the Er concentration and refractive index as functions of a distance from the surface deep into the OPSWG. The Er concentration increases initially with depth from 0.4 at.% at the WG surface to ~ 0.8 at.% at the depth of 3–5 μ m, decreases to ~ 0.2 at.% at the depth of 7-9 μ m and then decreases to under 0.1 at.% at the depth of over 10 µm. Refractive index increases with a depth from 1.41 at the surface to 1.458 at a depth of 3 µm. On further moving into the PS layer, it decreases to 1.428 at a depth of 7 μ m. Then the refractive index increases sharply to values higher than 1.5. Referring to Fig. 2, refractive index profile displays boundaries of core and cladding regions. With the whole WG thickness of 10 μ m, the core is in the range of $1.5-7.5 \,\mu$ m. The cladding region has an estimated thickness of 1.5 μ m at the surface and 2.5 μ m at the WG bottom. The refractive index step between the core and the cladding at the WG bottom approximates 0.03, twice as much as for OPSWGs formed from p⁺-type Si by one-step anodisation [6]. These results are due to the two-step anodisation process in which the controlled variation of the current density was used to form PS layers of varying porosity, with appropriate refractive index and thickness.

Fig. 3 gives Er concentration and refractive index against the WG width on scanning over the WG crosssection at the depth of 3 μ m. Referring to Fig. 3, Er concentration profile correlates with refractive index profile. A correlation between Er concentration and refractive index profiles discloses that Er is introduced generally into the WG core. Cladding regions were doped with Er to a lesser extent. Dominant core doping with Er relative to cladding regions is of great significance for WG amplifiers wherein pumping and signal modes propagate only through the core.



Fig. 2. The Er concentration (solid line) and the refractive index (dashed line) as functions of the WG depth.



Fig. 3. The Er concentration (solid line) and the refractive index (dashed line) as functions of the WG width.



Fig. 4. The attenuation spectrum of the Er-doped OPSWG.

The attenuation spectrum of the Er-doped OPSWG is shown in Fig. 4. Absorption bands at 650, 720, 810, 940, 980, 1240 and 1530 nm may be observed from the spectrum, among which 650, 810, 980 and 1530 nm bands are caused by optically active Er, while the rest of them seem to be from OH⁻ hydroxyl groups. The total losses involving Er-related absorption at 1530 nm and background loss are ~ 7 dB/cm. After the hightemperature annealing, the optical loss at 1300 nm was approximately 1 dB/cm. The losses obtained are lower than the losses measured for the OPSWG in p⁺-type Si substrates (5-15 dB/cm) [2-4]. This improvement is due to: (i) the use of two-step anodization process for n⁺-type Si provides a refractive index jump between the core and the cladding at the WG bottom of approximately 0.03, that is three times higher than the one

obtained in p^+ -type Si WG, realized by one-step anodization process [2,6]; and (ii) core of WG made of oxidized porous silicon material in n^+ -type Si has a much more dense structure than the p^+ -type Si as shown by the high resolution TEM study.

4. Conclusion

Er-doped channel OPSWGs formed from n^+ -type Si by the two-step anodization process and electrochemical cathodic treatment in 0.1 M Er(NO₃)₃ solution have been demonstrated. The two-step anodization process allows a formation of PS layers with controlled refractive index and thickness to form waveguide core and cladding layers, correspondingly. A correlation between Er concentration and refractive index profiles has shown dominant core doping with Er relative to cladding regions. Reported Er concentration in the OPSWG is large enough to attain the amplification effect. A more detailed study of the Er-doped OP-SWGs will be conducted in the future.

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