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Luminescence at 2.8 μm: Er^{3+}-doped chalcogenide micro-waveguide

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ABSTRACT

This paper reports the fabrication of luminescent optical rib/ridge waveguides made of erbium doped Ga-Ge-Sb-S films deposited by RF magnetron sputtering. Several fluorescence emissions of Er^{3+} ions from the visible to the middle infrared spectral domain were clearly observed within the films. The study of the $^4I_{13/2}$ level lifetime enabled development of a suitable annealing treatment of the films to reach the value of the bulk counterpart while the variation in surface roughness was limited, thus ensuring reasonable optical losses (0.7–0.9 dB/cm). Amplification experiments were carried out at 1.54 μm leading to complete characterization of the erbium-doped micro-waveguide with ~3.4 dB/cm on/off gain. A demonstration of mid-IR photoluminescence from Er^{3+}-doped chalcogenide micro-waveguide was recorded at ~2.76 μm. The multi-luminescence from the visible to mid-IR generated using erbium doped chalcogenide waveguiding micro-structures might find easy-to-use applications concerning telecommunication technologies or on-chip optical sensors for which luminescent sources or amplifiers operating at different wavelengths are required.

1. Introduction

The fabrication of amplifiers and light sources emitting in various spectral regions is of a growing interest for the development of more complex integrated optical devices in the field of modern telecommunication technologies and optical sensing. These active optical components can be applied to overcome optical losses issues along the propagation, the coupling or beam-splitting in waveguide structure. Such devices could be used to fabricate integrated laser sources or to provide frequency conversion operating in the visible, telecommunication bands or the mid-IR as well. In this perspective, chalcogenide glasses (ChG) appear as a complementary alternative to the capacities offered by oxide glasses [1]. While the transmission of the latter is limited for wavelengths above 3 μm, that of ChG extends up to 10–20 μm according to their composition, which allows to cover a large part of the mid-IR making them suitable as optical micro-sensor or integrated optics components for interferometric facilities for instance [2–5]. In the field of active optics, these amorphous materials show interesting characteristics such as high linear and non-linear refractive index, non-linear second order susceptibility, photorefractive effects and low phonon energies. All described properties are attractive for photonic micro-devices based on nonlinear optical or photoluminescence phenomena [6–17].

In optical integrated circuits, light is confined in a thin film that is deposited on the surface or buried in a substrate. Several methods have been described for the fabrication of amorphous chalcogenides waveguides like chemical vapor deposition (CVD) [18], thermal evaporation [19,20], RF sputtering [21], and pulsed laser deposition (PLD) [22,23]. To date, channel waveguides have been frequently produced by laser-writing. Indeed, ChG can exhibit a photodarkening effect when exposed to above band-gap light [24]. Using this irradiation writing approach, a laser effect was only reported once with a maximum laser output of 8.6 mW (at 1075 nm, by doping with Nd^{3+}) for an absorbed laser pump power of 89 mW [11]. An alternative way for the fabrication of luminescent waveguides is required as a result of possible relaxation of photoinduced effects during the pumping leading to slight

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instability of the refractive index. Despite the fact that new interest to study ChG doped with rare earth lies mainly in mid-IR applications as proposed in theoretical studies [25,26], no comprehensive study has presented so far the photoluminescence of amorphous chalcogenide films from visible to mid-IR. The emissions of the films reported in the literature were focused on the visible and near infrared spectral domain [21,27–36] like in the case of microwaveguides [10,11,21,37,38]. Lately, the $^4I_{11/2} \rightarrow {}^4I_{13/2}$ transition emitting at 2.7 μm was measured using annealing process in GaLaS sputtered strip waveguide [39].

In this paper, we report the fabrication of erbium doped sulfide rib waveguides by RF magnetron sputtering combined with classical dry etching technique, aiming at an efficient transfer for industrial purposes. The sputtering offers a complex composition control, adequate homogeneity, sufficient thickness and adhesion on different substrates resulting in high-quality films. For the fabrication of such devices, an accurate tailoring of rib waveguide shape can be obtained by dry etching; a specific treatment was developed here for studied sulfide films containing gallium. The potential degradation of optical properties in the waveguide regions during the different steps of the photolithography and etching processes was controlled leading to the fabrication of a photoluminescent sulfide waveguide. The optimization of photoluminescence and lifetime characteristics was also reported for these waveguides, obtained by a specific annealing treatment while the roughness variation was controlled. Apart from the observation of mid-IR generation and propagation at ~2.8 μm in rib ChG waveguide, amplification tests at 1.54 μm were carried out leading to an accurate characterization of the erbium doped sulfide waveguide.

2. Materials and methods

2.1. Glass target synthesis and film elaboration

The targets used for thin films fabrication were bulk ChG belonging to the Ga-Ge-Sb-S system. For a good solubility of rare earth ions in the host matrix, gallium was incorporated into the Ge-Sb-S system in order to obtain consequently rare earth doped films. Three compositions of the glasses ($Ga_5Ge_{20}Sb_{10}S_{65}$, $Ga_5Ge_{23}Sb_5S_{67}$, $Ga_3Ge_{22}Sb_{10}S_{65}$) were selected considering mainly their thermo-mechanical, optical, and spectroscopic properties. Glass targets of 50 mm in diameter were synthesized in silica tubes under vacuum following a procedure described elsewhere [40]. The rare earth choice dwelled on Er^{3+} for its many possible transitions in near- and mid-IR range. Thus, $Ga_5Ge_{20}Sb_{10}S_{65}$ glass matrix was doped with erbium in the range of 0.3–1.5 w.%. Ga-Ge-Sb-S films were deposited by RF magnetron sputtering, on cleaned microscope glass or silicon wafer covered with SiO_2. The optimal thickness of the oxide layer was determined considering the thickness and refractive index of the sulfide films in order to optically isolate the waveguide from the silicon substrate and thus reduce losses due to substrate leaking modes. For a Ga-Ge-Sb-S film of 4 μm thickness, a thickness of the SiO_2 confinement layer higher than 1.5 μm is required to ensure radiative losses lower than 0.01 dB/cm at 1.54 μm. For RF magnetron sputtering, argon pressure in the range of 5.10^{-3}–5.10^{-2} mbar was used. A low RF power (10–50 W) was required which is related to the dielectric character of the chalcogenide glass targets. The sulfide films were deposited onto substrates located parallel to the target surface at a distance varying from 5 to 10 cm. The deposition rate was about 10–15 nm min^{-1} with variation in thickness from 0.5 to 20 μm. The annealing of the fabricated films was performed at temperatures close to glass transition temperature of corresponding bulk ChG under argon atmosphere.

2.2. Dry etching

A classical photolithography process was used with positive Shipley S1813 photoresist. After soft baking, the photoresist was UV-exposed by means of a Cr-mask using a Suss Microtech MJB3 i-line mask aligner. The exposed bands were dissolved with a commercial developer. After hard baking, different parameters were varied in the CF_4 plasma etch process: gas flow from 1 to 30 sccm, gas pressure between 0.3 and 30 mTorr, and RF power in the range of 30–300 W [41]. The etch rates were determined by scanning electron microscope (SEM) observations of films' cross-sections.

2.3. Films and waveguide characterization

The compositional analysis of the thin films was performed using an energy dispersive X-ray spectrometer (EDS). Analyses using a NanoSIMS 50 (CAMECA) device were performed with two ion sources (Cs^+ source: analysis of S, Sb, and Ge elements; O^- source: analysis of Ga element). The surface of Ga-Ge-Sb-S multilayers and films were analyzed in a 20 × 20 μm area and the elements were collected from a depth of about 5–10 nm. The topography and morphology of the layers were analyzed with an atomic force microscope (AFM) in tapping mode and a SEM.

UV-vis-nIR and FT-IR spectrophotometers were used to study the optical transmission window of fabricated films. The effective refractive indices of the propagation modes in the planar waveguides were measured via prism coupling technique (Metricon-2010 instrument). Different laser beam wavelengths (633, 1302, and 1540 nm) for both TE and TM polarizations were employed. Optical functions and layers' thicknesses were controlled also through the analysis of spectroscopic ellipsometry data measured using an ellipsometer with automatic rotating analyzer (VASE, J. A. Woollam Co., Inc.). The data were recorded in the spectral region of 300–2300 nm with wavelengths steps of 10 nm (20 nm in case of target glasses); angles of incidence were 50°, 60°, and 70°.

The optical losses were measured by studying the scattered light intensity out of the surface of the waveguides (length from 2 to 2.5 cm). The laser input oncomes from a single-mode fiber and then it is coupled into the waveguide. The intensity of the scattered light was recorded by a digital camera placed above the sample. In this study, the attenuation values are taken as the average of several measurements performed on different samples. In addition, near field profiles of guided modes at 1550 nm were observed at the output of the waveguides.

2.4. Photoluminescence

For photoluminescence experiments within Er^{3+}-doped films and targets, the excitation beam was provided by a continuous wave Ti: Sapphire laser tuned at 804 nm. A monochromator and a thermoelectrically cooled extended InGaAs and a nitrogen cooled InSb photodiodes, combined with a lock-in amplifier triggered by a mechanical chopper were used to detect the fluorescence signal in the 850–1700 nm and 2.5–3 μm spectral regions. Fluorescence decays were recorded using a Nd: YAG pumped OPO laser (GWU C-355) delivering 6 ns pulses with a 10 Hz repetition rate.

Pump-probe experiments were also carried out to demonstrate the 1540 nm amplification in Er^{3+}-doped chalcogenide films under 975 nm pumping. A WDM was used to combine the 975 and 1550 nm beams; the fiber end was then butt-coupled to the waveguide. The output signal was collected in a multimode fiber and passed to an optical spectrum analyzer.