



## Double line waveguide amplifiers written by femtosecond laser irradiation in rare-earth doped germanate glasses

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### ARTICLE INFO

#### Keywords:

Optical materials  
Optical spectroscopy  
Amorphous materials  
Laser processing

### ABSTRACT

We report the production of active double waveguides in Er/Yb doped GeO<sub>2</sub>-PbO glasses, by direct femtosecond laser writing. The glasses were produced using the melt-quenching technique and the active waveguides were written using 30 fs laser pulses, at 800 nm, with writing speed of 0.06 mm/s and pulse energy of 32 μJ. The photo-induced negative refractive index change was of  $-7.4 \times 10^{-3}$ . The Er/Yb doped sample showed a relative gain (signal enhancement of 7.5 dB/cm, for 105 mW of 980 nm pump power. The relative gain compensates both, the propagation losses and the absorption losses, and a positive maximum internal gain of 4.6 dB/cm can be obtained at the signal wavelength of 1550 nm. The results obtained in present work demonstrate that Er/Yb glasses are promising materials for the fabrication of integrated amplifiers, lossless components and lasers based on germanate glasses.

### 1. Introduction

Femtosecond (fs) laser processing of transparent dielectric materials is finding numerous promising applications [1] such as integrated optics [2] and integrated microsystems with optofluidic and mechanical characteristics in a single substrate (lab-on-a-chip) [3]. Fused silica has been the most widely used material in this sort of application due to its properties such as high stability, broad optical transmission, low self-fluorescence and the possibility to fabricate devices such as waveguides [4]. Channel waveguides written by using ultrafast lasers in rare-earth (RE) doped materials have been demonstrated in a number of hosts, including heavy metal oxide doped glasses [5,6]. Depending on the material properties as well as on the characteristics of the laser used for inscribing, two types of waveguides can be obtained in practice. The first type consists of a single line, where the structural modification of the material causes a positive refractive index increase, leading to light confinement [7]. The second type consists of stress-induced negative refractive index change in the laser focal region. In the latter case, the light is guided in between two or more lines of writing. The methodology used in this work is based on the second type of writing, where double line waveguides demonstrated good results, after previous

experiments testing both types of waveguides [8].

Among several materials that can be used for photonic applications, germanate glasses are particularly interesting due to their mechanical and chemical stability, wide compositional range and high optical nonlinearity [9,10]. The GeO<sub>2</sub>-PbO (GPO) glassy matrix was chosen due the prior experience and successful results the authors have obtained with this system in spectroscopic and structural studies [11,12], thin film waveguides [13] and femtosecond-laser written double line waveguides in bulk glass [8]. Nonlinear optical studies of GeO<sub>2</sub>-PbO glasses in the fs regime with and without silver nanoparticles have been reported and demonstrated that these glasses are good candidates for the development of all-optical switching in the wavelength range of 700–1400 nm [14].

The aim of this work is to verify the feasibility of direct double line fs laser writing of waveguides in GeO<sub>2</sub>-PbO (GPO) glasses codoped with Er<sup>3+</sup> and Yb<sup>3+</sup> and to investigate optical amplification around 1550 nm under 980 nm excitation since there is a need for low cost amplifier devices and alternative approaches for semiconductor and Raman fiber amplifiers in the 2nd and 3rd telecommunication windows that has stimulated research activities using rare-earth doped glasses [15–17].

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## 2. Experimental apparatus and procedure

### 2.1. Preparation of the glasses

A relative amount of 1.0 wt% of  $\text{Er}_2\text{O}_3$  and 2.0 wt% of  $\text{Yb}_2\text{O}_3$  were added to the basic glass composition of GPO (40.3GeO<sub>2</sub> – 59.7PbO wt. %), resulting in GPO:Er/Yb. The samples were prepared by a conventional melt-quenching method. Batches with 12 g of high purity (99.999%) compounds were fully mixed in an alumina crucible and melted at 1200 °C for 1 h under mechanical agitation by a quartz rod, to avoid bubbles and striae in the glassy matrix. The melts were then poured into pre-heated brass molds, in air, and annealed at 420 °C for 1 h to reduce internal stress. After cooling, the samples were polished to acquire optical quality surface.

### 2.2. Waveguide writing

The femtosecond laser system (Ti:sapphire, model PRO 400, Femtolasers GmbH) with emission wavelength centered at 800 nm had a pulse length of 30 fs, maximum energy per pulse of 200 μJ and 10 kHz repetition rate. The system uses a stationary focal point while the sample is positioned by a three-axis translation stage (Aerotech ANT130 series) with accuracy of ±2 nm.

To focus the laser beam, a doublet lens with focal length  $f = 20$  mm and numerical aperture  $\text{NA} = 0,2$  was used, which produced a focal point with a calculated diameter of 3.6 μm in air. The beam was incident perpendicular to the polished surface with its linear polarization tilted 45° with respect to the direction of movement and the focal point was positioned 0.75 mm below the surface. Pairs of parallel lines were written closely spaced by 10 μm using the parameters shown in Table 1. The set-up for the waveguide writing is presented in Fig. 1. The writing speeds used in Table 1 were previously optimized in terms of small waveguide losses and good overlap of confined pump and signal mode [8]. Absorption was present in the GPO:Er/Yb sample, as evidenced by the characteristic green upconversion emission of  $\text{Er}^{3+}$  observed during the writing and shown in the inset of Fig. 1 [18,19].

After waveguide writing, the input and output facets of the samples were re-polished to eliminate the surface damage suffered during femtosecond writing [8]. The final length of the obtained waveguide was of 0.8 cm.

### 2.3. Characterization

The visible to near-infrared optical absorption spectrum was obtained at room temperature from 350 to 1600 nm, using a commercial spectrophotometer, to verify the incorporation of rare earth ions. To estimate the refractive index change, the waveguide output beam diameter was measured at several distances and the numerical aperture of the waveguides was calculated from the ratio between the distance and the mode radius. The refractive index change can be estimated by the measured N.A. (numerical aperture) of the waveguide, as described in equation (1) where  $n_1$  and  $n_2$  represent the refractive index of the core and the cladding, respectively [20].

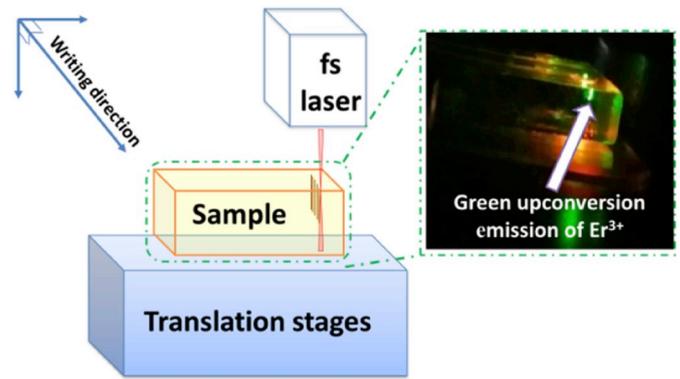
$$\text{N.A.} = \sqrt{n_1^2 - n_2^2} \approx \sqrt{2n_2\Delta n} \quad (1)$$

For waveguide alignment and preliminary characterization, a fiber

**Table 1**

Parameters used in the writing process.

Sample	GPO:Er/Yb
Writing speed (mm/sec)	0.06
Pulse energy (μJ)	32
Fluence (J/cm <sup>2</sup> )	140
Number of overlap pulses	90



**Fig. 1.** Set-up for waveguide writing. The inset shows the green upconversion emission observed during writing in GPO:Er/Yb.

coupled HeNe laser beam was focused into the waveguides to capture the near field profiles at the output with a CCD (Newport Inc.).

Propagation losses were determined at 1300 nm using the cut back method [21,22]. The gain properties of the waveguides were characterized as follows: the relative gain (signal enhancement) was obtained by using cw laser diodes operating at 980 nm and 1550 nm for excitation pump and signal wavelengths, respectively. Pump and signal beam were multiplexed by a 980 nm/1550 nm WDM and sent into the waveguide by a lensed fiber tip. During the measurements, the power of the signal was kept constant at 700 nW to avoid gain saturation. The setup is shown in Fig. 2.

To calculate the relative gain first the output signal is recorded ( $P_{\text{signal}}$ ) without pump, then the pump is switched on and the output is recorded together with the amplified spontaneous emission ( $P_{\text{signal+ASE}}$ ) and last, ASE is measured by turning the signal off and leaving the pump on. The relative gain was determined by the following expression, where  $d$  is the length of the waveguide [22,23]:

$$G \left[ \frac{\text{dB}}{\text{cm}} \right] = \frac{10 \times \log \left( \frac{P_{\text{signal+ASE}} - P_{\text{ASE}}}{P_{\text{signal}}} \right)}{d} \quad (2)$$

Fourier-transform infrared spectra (FTIR) of the studied glasses were registered at room temperature in the range of 400  $\text{cm}^{-1}$  to 2000  $\text{cm}^{-1}$  with a resolution of 4  $\text{cm}^{-1}$  (JASCO FTIR 6200) and using the KBr pellet technique where the samples (piece of bulk glass unaffected by the laser and piece of glass containing the waveguides) were grinded, mixed with potassium bromide (KBr) and pressed in order to obtain a pellet for the measurements.

## 3. Results and discussion

Linear absorption measurements of the samples after the fabrication process demonstrated transparent and homogeneous glasses (Fig. 3). The observed pattern highlights the typical absorption spectra of GPO:Er/Yb glasses and demonstrates the presence of the rare-earth ions in trivalent form, which are responsible for the active behaviour [22]. Visible to NIR transitions associated to  $\text{Er}^{3+}$  and  $\text{Yb}^{3+}$  are shown. The inset presents the absorption cross section spectrum of the  $^4\text{I}_{13/2} \rightarrow ^4\text{I}_{15/2}$  transition; the absorption cross section at 1550 nm is  $3.0 \times 10^{-21} \text{ cm}^2$ .

The refractive index change at 632 nm was determined by equation (1) and the obtained value is  $\Delta n = -7.4 \times 10^{-3}$ . The near field beam profile at the output of the GPO:Er/Yb waveguide is shown in Fig. 4 and shows a confined beam, consistent with single transverse mode.

Fig. 5a shows a pair of double line waveguides in the GPO:Er/Yb sample. The waveguide at the left is responsible for the GPO:Er/Yb results reported in this work. Fig. 5b shows the characteristic green upconversion emission of  $\text{Er}^{3+}$  as a result of 980 nm pumping during the gain measurements in the same sample (pumping occurs from the right).

Propagation losses of  $(0.87 \pm 0.28) \text{ dB/cm}$  at 1300 nm were obtained

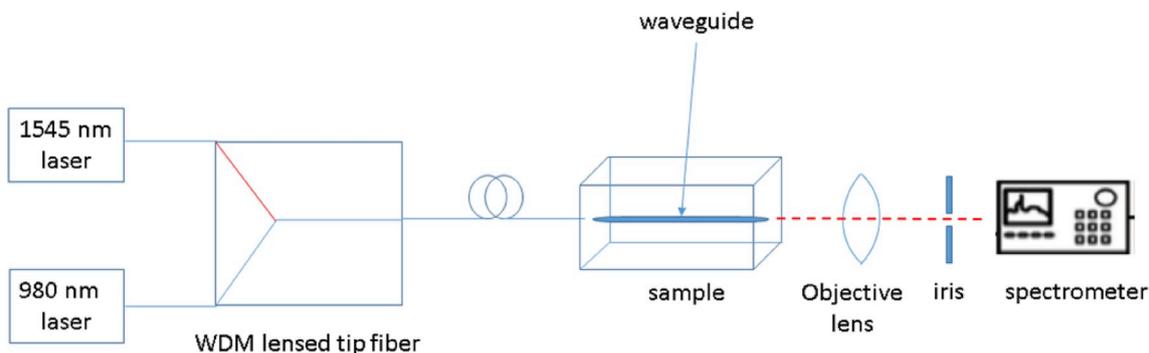


Fig. 2. Experimental setup used for relative gain measurements of the GPO:Er/Yb waveguide.

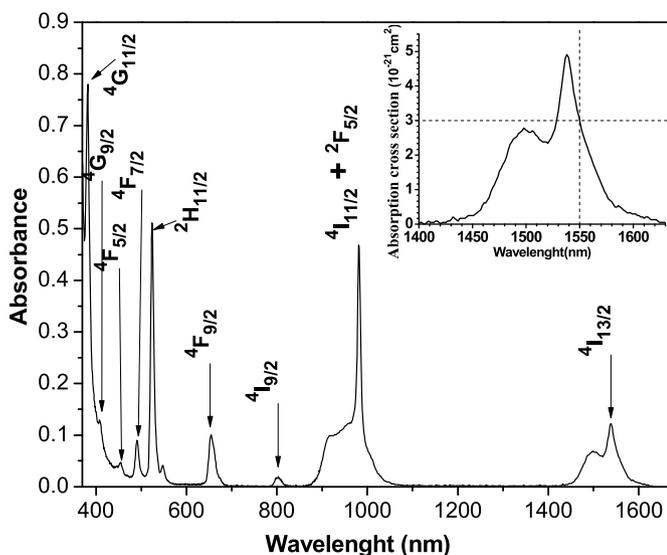


Fig. 3. Absorption spectrum of the GPO:Er/Yb waveguide. The inset shows the absorption cross section spectrum of the  ${}^4I_{13/2} \rightarrow {}^4I_{15/2}$  transition (absorption cross-section at 1550 nm is  $3.0 \cdot 10^{-21} \text{ cm}^2$ ).

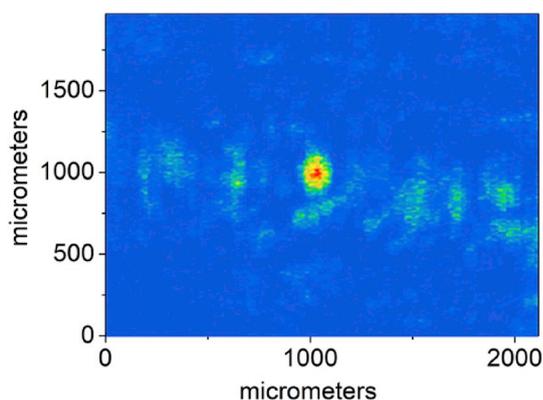


Fig. 4. Near-field mode profile of the waveguides obtained with a 632 nm laser.

using the cut back method. Fig. 6a presents the ASE spectrum without signal for 105 mW of pump power at 980 nm and the signal at the waveguide output after ASE removal for two cases: using 700 nW and 10  $\mu$ W of input signal strength at 1550 nm. The variation of the relative gain at 1550 nm as a function of the pump power for 700 nW of input signal power, determined using equation (2), is shown in Fig. 6b. The relative gain at 1550 nm, reached 7.5 dB/cm, for 105 mW of 980 nm pumping. The gain did not saturate but rather a second and lower slope

efficiency can be observed for pump powers beyond 65 mW.

If we take into account propagation losses and absorption losses, the internal gain can be calculated as in Ref. [22]. Considering the propagation losses,  $\alpha_p = (0.87 \pm 0.28) \text{ dB/cm}$  at 1300 nm, obtained from the cut back method, and the absorption losses,  $\alpha_A = (2.0 \pm 0.1) \text{ dB/cm}$  at 1550 nm, estimated from the absorption cross section spectra (as in Ref. [21]) of the GPO: Er/Yb, shown in the inset of Fig. 3, we can calculate the internal gain for 105 mW of pump power, as follows  $G_{INT} = G_R - \alpha_p - \alpha_A = 4.6 \text{ dB/cm}$ . The relative gain,  $G_R$ , compensates both, the propagations losses and the absorption losses, and a positive maximum internal gain of 4.6 dB/cm can be obtained at 1550 nm for 105 mW of pump power.

Results from the FTIR spectroscopic measurements of the femto-second laser focal region in the waveguides and also in the bulk region (unaffected by the laser) are shown in Fig. 7 for the undoped GPO host.

Table 2 shows the FTIR peak positions and the corresponding assignment to the  $\text{GeO}_2 - \text{PbO}$  glass system. In previous experiments we demonstrated that the Raman results in the region between the double lines are similar to those of the bulk glass, which suggests that no fundamental structural change is caused by the fs laser writing technique at the locus of wave guiding [8]. Comparing the results of the bulk area with those of the writing area we observe almost no shift of the peaks in the spectra, however, a small intensity decrease for most of them is noticeable for the sample originating from the laser focal region. The majority of these peaks correspond to vibrations of different kinds of linkages in  $\text{GeO}_4$  units. A decrease of these structural units causes a decrease in density of the  $\text{GeO}_4$  units and may give an indication of why the waveguiding occurs between the two written lines, since the refractive index is proportional to the electronic density. These observations corroborate with previous results where, using a different fs laser system, we have already shown clear evidences of a decrease of the refractive index at the center of the written line for this kind of glass [8]. Despite the FTIR measurements shown in the present work are related to the pure GPO matrix (not rare earth doped), their results should be similar to the rare earth doped GPO samples. Cuela et al. demonstrated by FTIR experiments that no dramatic changes occur with a  $\text{Nd}_2\text{O}_3$  content increase in their samples [24].

#### 4. Conclusion

This work has addressed the feasibility of producing double line waveguide amplifiers in doped germanate glasses by direct fs laser writing. Samples were prepared using a standard melt-quenching technique, that allowed the production of optical-quality glasses that were characterized from a spectroscopic viewpoint. Absorption measurements confirmed the trivalent form incorporation of the rare earth. The waveguides were written by 30 fs laser pulses at 800 nm. The best condition for amplification was achieved by waveguides written with 0.06 mm/s and 32  $\mu$ J. The photo-induced, negative refractive index change was  $-7.4 \cdot 10^{-3}$  and the observed near-field pattern image

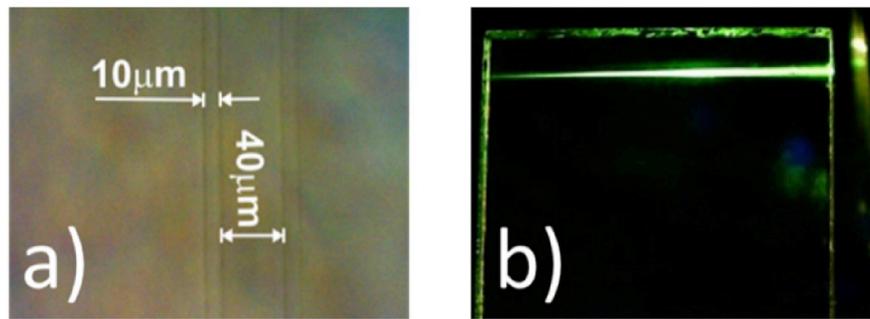


Fig. 5. Top view microscope images of double line waveguides in the GPO: Er/Yb. a) Two double line waveguides captured by using a 200x magnification and polarized light. b) Top view camera image of a single double line waveguide in the GPO: Er/Yb pumped at 980 nm.

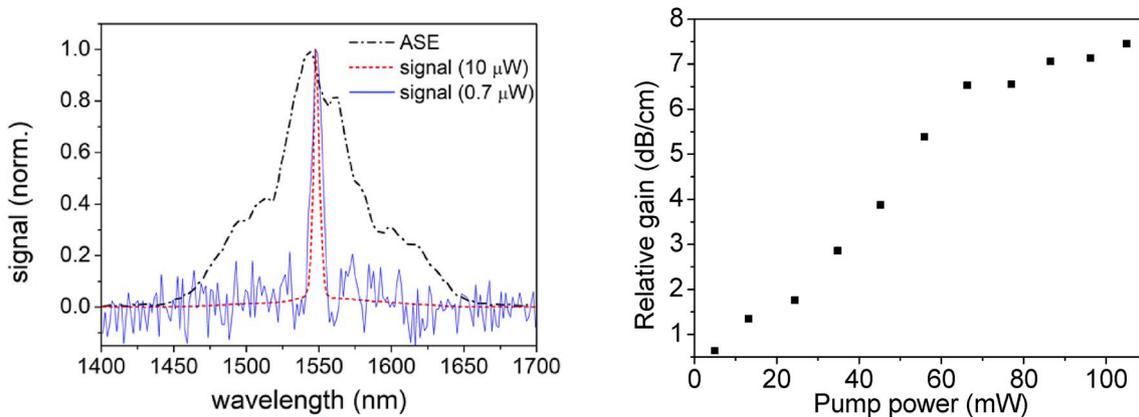


Fig. 6. a) ASE spectra for 105 mW of pump power (no signal, dash-dotted line); signal at output after ASE removal (equation (2)) for 105 mW of pump power at 980 nm for two cases: using 700 nW (line) and 10  $\mu$ W (dots) of input signal strength at 1550 nm. b) Relative gain at 1550 nm of the GPO:Er/Yb waveguide as a function of pump power for 700 nW of signal input power.

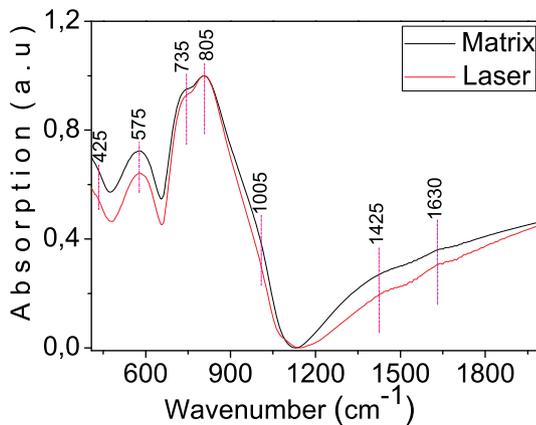


Fig. 7. FTIR spectra of the GPO matrix sample measured in the writing area and in the bulk area.

showed good waveguiding quality, consisting of a single, circular lobe. FTIR results showed that the main structural change caused by the writing process is the decrease of the  $\text{GeO}_4$  unit density corroborating with the observed negative index change at the femtosecond laser focal position. The GPO:Er/Yb waveguide achieved a relative gain of 7.5 dB/cm for 105 mW of pump power at 980 nm and a positive internal gain of 4.6 dB/cm. No saturation of the gain was observed, which suggests a potential for optimization of the amplifier. The results obtained in the present work demonstrate that Er/Yb glasses are promising materials for the fabrication of integrated amplifiers, lossless components and lasers based on germanate glasses.

Table 2

FTIR peak positions and assignment to  $\text{GeO}_2$  – PbO glass system.

Peak position ( $\text{cm}^{-1}$ )	Assignment
425 $\text{cm}^{-1}$	Symmetric stretching vibrations of Ge-O-Ge from $\text{GeO}_4$ units [25]
575 $\text{cm}^{-1}$	Symmetric stretching mode of Ge – O – Ge bonds [25]
735 $\text{cm}^{-1}$	Asymmetric bending vibration mode of Pb – O – Pb bonds [26]
1425 $\text{cm}^{-1}$	Asymmetric stretching vibrations of Ge-O-Ge in $\text{GeO}_4$ units [25]
1630 $\text{cm}^{-1}$	Asymmetric stretching vibration modes of Ge-O-Ge bonds [25]
	Bending mode of vibration [26]

## Acknowledgments

We acknowledge financial support from Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP) (2016/02326-9, 2017/10765-5 and 2013/26113-6) and from Conselho Nacional de Desenvolvimento Científico e Tecnológico - Grant: INCT/CNPq 465.763/2014 (Instituto Nacional de Ciência e Tecnologia de Fotônica).

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