Direct writing of low-loss birefringent laser waveguides in Nd^{3+}, Y^{3+} codoped SrF_2 crystal by ultrafast laser

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Abstract: High refractive index contrast, birefringence and luminescence properties of written channel waveguides inside fluoride crystals written by femtosecond (fs) laser have been studied. Herein, fs laser-induced stress affected zone is an efficient tool to create birefringence and high refractive index contrast in Nd^{3+}, Y^{3+} codoped SrF_2 crystal for wide range of pulse energies and repetition rates. In fabricating the waveguides at high repetition rate (typ. 500 kHz), we avoided too much heat accumulation, and thus they exhibit lower propagation loss in codoped SrF_2 crystal (1.63 ± 0.21 dB/cm for TM-polarization). The measured retardance can be interpreted as related to stress-induced birefringence in response to free of stress volume expansion photo-induced in the non-spherical irradiated zone. The photoluminescence and lifetime measurements are also carried out in order to understand local changes of the network in and around femtosecond laser induced waveguides written in codoped SrF_2 crystal.

Keywords: SrF_2, CaF_2 crystal; Femtosecond laser processing; Birefringence; Photoluminescence

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1. Introduction

The femtosecond (fs) laser is a revolutionized material processing opening new avenues of the three dimensional (3D) photonic devices [1,2]. Fs laser driven self-organized nanostructures and nanovoids initially observed in fused silica related structural and optical properties that is studied extensively over one decade ago [1,2]. Subsequently, these sort of nanostructures are commonly known as "Nanogratings" consisting of sheet like cavities with a sub-wavelength periodicity and is successfully harnessed to scalable photonic applications including optofluidics and lab-on-a-chip devices [1-6]. In particular, these nanogratings, hitherto, are observed inside volume of a few specialized optical materials merely [1,2]. On the contrary, the stress-induced birefringent waveguides (no nanogratings) are observed in bulk transparent materials, which is arisen by the coupling of elasto-optical and the mechanical stress induced at vicinity of the laser track [1-6,26]. Noticeably, the high symmetric fabricated type-II waveguides inside materials can preserve non-destructive material properties and high resistance by so called "double line" technique [7-9]. This direct writing gives rise to high index parallel lines as a core that can be fenced by low index strained lines (i.e., it is commonly known as depressed cladding waveguides) [9]. In fact, there are coexisting of different phases in written waveguides inevitable to allow wide tunability in the refractive index contrast [9]. This can facilitate light confinement and guiding structure accompanied by micro- and sub- micron dimension inside bulk materials becoming one of the key building blocks in photonic devices. However, the high refractive index contrast fabricated waveguides make control of birefringence, which is one of the fundamental challenging task.

To improve the communication bottleneck and waveguide lasers in Nd^{3+} doped disordered crystals relative to Nd^{3+} doped transparent glass and crystals with unique properties of high

thermal conductivity, inhomogeneous broadening and large stimulated emission cross-section [7-11]. In particular, the SrF_2 crystal is multifunctional optical raw material and could be widely disseminated in many fields. Importantly, the low phonon energy, high rare earth solubility, low non-radiative transition probability, long lifetime metastable state and good transparency spanning up to 11 μm from 0.2 μm of SrF_2 crystal enable to offer high performance birefringent waveguide lasers by ultrafast laser processing [12]. Recently, it has been reported by Xu et al. that high content of Nd^{3+}, Y^{3+} codoped SrF_2 crystal can accomplish negative refractive index due to the stress formation only with high propagation losses of about 3.1 dB/cm at 632.8 nm using Ti-Sapphire laser with a typical repetition rate of 1 kHz [13]. One boundary criteria in rare earth spectroscopy is concentration quenching that led to degradation of device performance above threshold value of neodymium content. By codoping buffer ions (i.e., it is yttrium and lutetium) into RE doped alkali crystals give rise to minimize concentration quenching effects, progressive improvement in material properties and laser action markedly as well as full-fill these criteria [14]. When exposed materials to high energy irradiation, there are structural defects driven by the high pressure and temperature and needs to be understand for photonic applications [15]. Thus, the Nd^{3+} doped SrF_2 disordered crystals are more attractive and applicable to femtosecond laser operation and is barely reported in literature [16]. Keeping in view of this, we make them to be developed low-loss birefringent buried waveguide lasers and might be opened a new gate to diversify the field of numerous photonic applications.

The present work mainly deals with the fabrication of birefringent, low-loss buried channel waveguides inside 0.5 mol. % Nd^{3+}, 5.0 mol. % Y^{3+}:SrF_2 crystals. For comparison, we also took 0.5 mol. % Nd^{3+}, 10 mol. % Y^{3+}: CaF_2 (NYCa) crystal. From our experimental results, the Nd^{3+},

Y^{3+} codoped SrF_2 single crystal demonstrates superior optical properties over codoped CaF_2 crystal and promising materials for the developing new 3D-photonic device based applications.

2.0 Experimental details

Transparent Nd^{3+} (0.5 mol. %), Y^{3+} (5.0 mol. %) codoped SrF_2 single crystal had grown by Bridgman-Stockbarger method. There were labeled hereafter as NYSr. NYSr crystal exhibit typical dimension of 10 (x-axis) × 10 (y-axis) × 1 (z-axis) mm^3 and were then polished to an optical grade. For 3D laser direct writing, we have used an ultrafast laser (Satsuma, Amplitude Systems Ltd.) that operates at 1030 nm with a linearly polarized beam and delivering pulses of 250 fs with varying repetition rate from 10 to 500 kHz. Then, 20 × aspheric lens with a numerical aperture (NA) of 0.6 was accustomed to focus the laser beam about 150 μm below the front face of NYSr crystal plates. Consequently, we can able to minimize spherical aberration inside it. Ultimately, a computer controlled program (GOL3D from GBC&S) can be used to translate NYSr crystals using a three dimensional XYZ-motorized stage. 3D waveguides writing using the twin lines technique with a typical separation of 12 μm was carried out using different pulse energies in the range of 0.1-5 μJ at two different repetition rates (10 kHz & 500 kHz). In addition, single laser lines were inscribed in a similar range of pulse energy and repetition rates in order to examine retardance and refractive index contrast related to the formation of single laser track. For the following experiments, an end-facet of the crystal waveguides were polished to diminish scattering losses, after laser material processing.

After laser inscription, the waveguide "striations" were probed not only using an optical microscope, but end-face coupling alignment was also carried out to distinguish type of mode formation and to study the propagation losses. In addition, optical retardance measurements related to the occurrence of linear birefringence was measured using Olympus BX51 polarizing

optical microscope equipped with a Senarmont compensator. The quantative phase microscopy (QPm) was applied to determine average refractive index (Δn) changes along the laser written waveguide. The laser tracks were imaged using white light through a 20 × microscopic objective. Thanks to a piezo-mounted objective a set of 3 images were performed in the focus and out of focus with a step of +/-3 μm defocusing. The resulting intensity 3D-images were computed using QPm algorithm to determine the quantitative phase changes across the laser track. The optical absorption spectra were also performed by using a double-beam spectrophotometer (TU-1900, PG Instruments Co., Ltd.) in the spectral range of 200-900 nm with a spectral resolution of 1.0 nm. Micro-photoluminescence spectroscopy was performed using 532 nm green laser, as an excitation source that is focused on waveguides directly by the help of 50 × microscopic objective. The backscattered light was detected by a spectrometer through the same microscopic objective and finally signals are captured by charge-coupled device (CCD) Andor camera. In addition, PL spectra were performed using a spectrofluorometer (FLS920 EDINBURGH,) of 450 W Xe lamp as a source. The lifetime measurement was also performed by μF920 microsecond flash lamp. All measurements were post-mortem and carried out at room temperature.

3.0 Results

At first, the fs laser-induced waveguides written inside SrF_2 crystal with different pulse energies at two different repetition rates are observed using an optical microscope (Axio Imager, Carl Zeiss) in a transmission mode. Optical microscopic cross-sectional images of the written waveguides are depicted in Fig. 1 with respect to pulse energies at a fixed repetition rate of 10 kHz. In the pictures shown in Fig. 1 for TM polarization together with the normalized intensity scale bar.

As shown in Fig.2, it is noteworthy that there is significant decrement in propagation losses with respect to pulse energies indicating a better confinement of the light. However, losses are higher for waveguides made at 10 kHz as a consequence of heat-accumulation phenomena at 500 kHz leading the bleaching of point defects and also reducing scattering losses [17]. The lowest propagation loss of NYSr crystal is estimated to be 1.63 ± 0.21 dB/cm for TM polarization at 632.8 nm for 500 kHz repetition rate waveguides at typical pulse energy of 0.3 μJ. Red- and black-spots in Fig. 1 at 0.5 μJ indicate the location of μ-PL measurements that was carried out in the laser-induced cladding and in unmodified area, respectively.

The absorption spectra of NYSr crystal are recorded in the range of 200-900 nm, before (0 μJ, black line) and after femtosecond laser (3 mm × 3 mm square region with a pulse energy of 0.3 μJ, red line) irradiation, as shown in Fig. 3. It is observed that there is an overall increase (typ. +0.05 abs unit) of the optical absorption, which could be likely attributed to the point defects in the UV and Vis range. In addition, we observe two new feeble absorption bands at 262 nm and 612 nm might be related to fluorine defects. Another significant feature of optical absorption band at around 796 nm is mainly originating from the yttrium ions and is in agreement with previous work [21]. Interestingly, 796 nm yttrium (Y^{3+}) absorption band seems to be unvaried with femtosecond laser irradiation at this particular energy (i.e., there is no modifications in the yttrium environment).

Fig. 4 portrayed the optical retardance (proportional to the linear birefringence) of NYSr crystal subjected to varying pulse energies in the range of 0.1-5 μJ and for two different repetition rates (10 & 500 kHz). The fabricated optical waveguides were written with an orientation of the laser polarization (E) perpendicular to the scanning direction (v). For waveguides fabricated at low repetition rate of 10 kHz, there is no detection of optical retardance

until the pulse energy of 0.1 μJ and then retardance is monotonically increasing with pulse energy to reach a maximum retardance about 46 nm for a pulse energy of 5 μJ [18]. For high repetition rate waveguides, no optical retardance could be detected below 0.1 μJ like 10 kHz waveguides. Then, we observed a growing retardance approaching a maximum value of 40 nm followed by a significant decrease beyond a pulse energy of 1 μJ. Above 2 μJ, laser tracks are too damaged to probe the photo-induced optical properties due to a too heat accumulation phenomena for 500 kHz repetition rate waveguides, as shown in Fig. 4. The lowering in the magnitude of the retardance could be likely attributed to the onset of apparition of the heat accumulation process. This leads to a temperature around the melting temperature, which partly relax the stress-induced birefringence within the laser tracks. Finally, we add similar measurements made in the NYCa crystal waveguides for the sake of comparison. It is clear that the highest optical retardance has been achieved in NYSr crystal especially at low-repetition rate waveguides.

To further investigate refractive index changes across the written laser tracks we used quantitative phase microscopy (QPM) in natural light. QPM is directly employed to compute phase change (directly proportional to the average change in refractive index) across the written waveguides. The resulting phase change was estimated from two-dimensional (2D) mapping of refractive index profile in the xy plane as shown in Fig 5. For low repetition rate (typ. 10 kHz), we observe uniform modifications with the absence of voids formation. On the contrary to low repetition rate fabricated waveguides, high repetition rate waveguides clearly reveal inhomogeneous refractive index due to void formation, when increasing the pule energy above 0.6μJ. It is scrutinized that there is a strong occurrence of black line indicating a characteristic features of negative index changes attributed to volume expansion both at low and high

repetition rates according to the pulse energies. It was found that the diameter of waveguides (black color) follows obvious increment with pulse energies. In addition, there are some microscopic disruptions especially at high repetition rate waveguides, where voids formation or inhomogeneity's took place. The white line (Fig. 5) represents features of elastic strain occurring in and out laser exposed region as a result of the permanent strain photo-induced within irradiated area.

From a quantitative view, phase change (Δφ) can be expressed in radians that is plotted against pulse energies for both repetition rates in Fig. 5. At first there is no change in the amplitude of phase change (kinetic behavior) until pulse energy of 0.3 μJ. For low repetition rate, we observe an increase of the negative phase change until a pulse energy of 1.5 μJ down to –3.5 radians. Thereafter refractive index is slightly increasing and stabilized around -2.5 radians whereas the diameter of the waveguides exhibits a monotonous increase from 4.7 up to 8.3 μm. On the other hand, a similar kinetic behavior could be observed at high repetition rate (500 kHz) up to 1.0 μJ and then the phase change dramatically approached almost to zero phase shift as the pulse energy typically reached to 2.0 μJ where catastrophic damage can be observed. It discloses clearly heat accumulation process in agreement with a width increase that reach 12 μm at 2 μJ. It is in good agreement with the measured kinetics of optical retardance shown in Fig 4.

After femtosecond laser modification, the change in the refractive index (Δφ) can be quantified using the relation $\Delta\phi = 2\pi\Delta n(\frac{d}{\lambda})$ (here, $\Delta\varphi_{max}$ = -3.5 rad, d = 50 μm) and the maximum obtained value is about -6×10^{-3}. When compared to NYCa crystal, higher amplitude of negative phase change (i.e., lower refractive index) was taken place in the NYSr crystal under similar irradiation conditions. This can be explained due to higher refractive index *n* of SrF_2

(1.488 for SrF_2 and 1.434 for CaF_2 at 550 nm) leading to a higher index changes Δn_{ii}^{p} for a similar free-of-stress strain ε_0 following $\Delta n_{ii}^{p} = -\frac{(n^2-1)(n^2+2)}{2n}(1+\Omega)\varepsilon_0$

To further insight the influence of femtosecond laser irradiation on NYSr crystal, thereby, we have performed photoluminescence (PL) of NYSr crystal under 532 nm excitation before and after femtosecond laser irradiation. Resulting PL spectra of NYSr crystal waveguides (500 kHz) with different pulse energies are shown in Fig. 6. As a result, we can observe the main PL bands located at ~867 nm (R_1-Z_1) and ~900 nm ($^4F_{3/2}$-$^4I_{11/2}$) of Nd^{3+} ions [14,16]. Besides, a broad emission band consisting of peaks at 1055 nm (Y^{3+}) and two small humps (1047 nm and 1066 nm - Nd^{3+}) were recognized [14,16]. Our results suggest that PL is found to increase marginally with pulse energy in the investigated spectral regime until 1.5 µJ. Above 1.5 µJ, there is a reverse trend in the PL intensity that slightly decrease. Similar observations were done in 10 kHz repetition rate waveguides, but it is lower than for high repetition rate, whatever the pulse energy employed here. In addition, there is slight increment in the full-width at half maximum in codoped SrF_2 crystal. For the sake of comparison we compared to PL results from Nd^{3+}, Y^{3+}-codoped CaF_2 crystal where higher luminescence (nearly two times) of Nd^{3+},Y^{3+}:SrF_2 crystal has been assessed as shown in the inset of Fig. 6. Close view of PL results reveal that there is no appreciable shift in the Y^{3+} luminescence for all emission bands. Compared to unirradiated PL, PL spectra look very similar to each other and well-preserved properties of the materials after femtosecond laser irradiation.

Under 808 nm Xenon lamp excitation, the spectra shown in Fig. 7 reveal three prominent emission bands in the near infrared peaking at about 896 nm, ~1056 nm and ~1336 nm corresponding to $^4F_{3/2}$-^4I_J (J = 9/2, 11/2 & 13/2) [14,16]. It is important to underline that Nd^{3+}

emission in the irradiated area (0.3 μJ) exhibits a slightly lower integrated emission intensity when compared to the pristine NYSr sample. Further, note that the branching ratio (β_{exp}) of $^4F_{3/2}$-$^4I_{11/2}$ transition did not follow any appreciable change before (0.6141) and after (0.6129) femtosecond laser irradiation. Additional lifetime measurements using μF920 microsecond flash lamp were thus carried out to record the luminescence decay curves of NYSr crystal (λ_{ex}=808 nm; λ_{em}=1056 nm) before and after femtosecond laser irradiation as shown in Fig. 8. All luminescence decay curves can be well represented with a single exponential. The estimated lifetime of $^4F_{3/2}$-$^4I_{11/2}$ is around 398.37 μs after fs laser irradiation that is almost the same as the pristine one (399.46 μs). This small discrimination in lifetime and branching ratio might be caused by the fluorine defects and potential distortion of Nd^{3+} environment.

4.0 Discussion

For pure silica glass materials, there are type-I waveguides formation that took place by means of a permanent densification at focusing laser regime composed of positive index change leading to guiding of light. In contrast for most other multicomponent silicate glasses, we observed the formation of type II waveguides consisting of a free-of-stress expansion (and thus negative index changes) accompanied by a compressive stress providing a positive index changes that can be exploited to guide the light either along a single laser track or by writing the waveguides cladding. Following the same trend, the laser-induced modifications in crystals (SrF_2 & CaF_2) indeed facilitate "decreasing order" (i.e., partial amorphisation or polycrystalline, permanent strain) in the laser-irradiated zone resulting in declining the refractive index i.e., no guidance in the irradiated area [22]. However the observed birefringence reveals the formation of a compressive stress field, which provides the way to guide the light in between written laser tracks. Within the last years, there are many reported results of stress-induced birefringence that

was observed in transparent bulk materials namely YAG, Nd^{3+}: YAG and $LiNbO_3$ crystals, borosilicate glass (BK7), doped phosphate and tellurite oxide, where seemingly the cohesion energy of the bonding strength play a vital role [23,26]. Polarimetric techniques are applied to compute birefringence's that are arisen by the not only stress, but also potentially through non-cubic phase transformation to new formation of crystalline phase in the YAG waveguides under ultrafast laser irradiation [5].

In this work, we could not observe the formation of permanent nanogratings in fluoride crystals whatever the pulse energies and repetition rates may be. Although, we saw a significant birefringence, which is primary caused by the presence of a stress-field inside but also surrounding the laser track. At low repetition rate and for low energy at high repetition rate, this is likely due to the introduction of free-of-stress strain that produces elastic strain (thus dislocations and distortion of the crystal lattice) rather. The free-of-stress strain is produced by melting and recrystallization of the crystal [23,26]. It is noteworthy that more laser heat energy is necessary to heat up the SrF_2 crystal over CaF_2 crystal due to their different melting temperatures in order to know the dissociation of network [5], but thermal diffusivity is 5 times for SrF_2 faster for CaF_2 crystal. Optical retardance of NYSr crystal reaches 46 nm, which is two-fold higher than NYCa crystal retardance at 10 kHz. The corresponding optical birefringence in NYSr crystal is around 10^{-3}. This is partly due to a higher Young modulus and thus a higher stress for a similar strain, but there is likely also a higher free-of-stress strain imprinted and higher rate of crystal network decomposition in SrF_2. For high repetition rate waveguides, the time taking between successive laser pulses is too short when compared to thermal diffusion time. As a result, the heat accumulation leads to melting of the SrF_2 crystal for high pulse energy. This

partly or fully relaxes the stress –resulting in a much lower or even no birefringence for high pulse energy.

It is known that the concentration quenching phenomena play a vital role in the device fabrication and working functionality. For instance, Jiang et al., investigated more deeply change in the absorption and luminescence properties by the addition of Y^{3+} ions, which is driven by the [Nd^{3+}-nY^{3+}] non-quenching clusters [14]. According to the previous work [14], it is clear that luminescence band at about 1054 nm is answerable for site-I, while there is two bands at around 1047 nm and 1066 nm responsible for site-II especially in Y^{3+} doped materials. On the basis of an electron paramagnetic resonance technique, it is concluded that there exists color centers such as F-defect centers occurs in the rare earth doped alkali fluoride (CaF_2 & SrF_2) materials. In pure SrF_2 crystal, there is an observation of laser action obtained at 1.64 μm. By the addition of buffer ions, they led to the suppression of concentration quenching followed increasing rare and buffer ion bonding formation (Nd-Nd replaced by Nd-Y pair centers). Another possible reason might be due to the presence of cross-relaxation channels ($^4F_{3/2}$; $^4I_{9/2} \rightarrow {}^4I_{15/2}$; $^4I_{15/2}$) or ($^4F_{3/2}$; $^4I_{9/2} \rightarrow {}^4I_{13/2}$; $^4I_{15/2}$). Recently, Ma et al., demonstrate good performance in turn slope efficiency and higher photoluminescence properties of Er^{3+} doped SrF_2 single crystal and outperform to Er^{3+} doped CaF_2 single crystal [25]. Close observation from PL results, the site-II marginally shortened, while site-I is improved after femtosecond laser irradiation. However, this small assertion might be possible within the experimental error.

5.0 Conclusions

In conclusion, we have investigated laser-induced changes namely refractive index changes, birefringence, guidance, and PL studies in ultrafast laser written channel Nd^{3+},Y^{3+}:SrF_2 crystal

waveguides. Also, the obtained results are compared with Nd^{3+},Y^{3+}:CaF_2 waveguides. For low-repetition rate SrF_2 crystal waveguides, we found a higher birefringence (nearly two times) within laser-modified zone in contrast to CaF_2 crystal. In addition, higher Nd^{3+} luminescence (nearly twice) is assessed than the one observed in CaF_2 crystal. Our results emphasized here that the Nd^{3+}, Y^{3+}: SrF_2 crystal is revelatory and might be attractive for development of waveguide lasers, integrated photonic circuits and UV-Vis stress-birefringent based devices.

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Figure Captions

Fig. 1 End face cross-sectional images of the channel waveguides with respect to different pulse energies for 10 kHz repetition rate. Mode profile pictures for 10 kHz and 500 kHz repetition rate waveguides with different pulse energies for TM polarization.

Fig. 2 Propagation losses versus pulse energies at 632.8 nm for 10 kHz and 500 kHz repetition rates.

Fig. 3 Absorption spectra of NYSr crystal before and after femtosecond laser ionization in the range of 200-900 nm.

Fig. 4. Optical retardance of NYSr crystal as a function of pulse energies for 10 and 500 kHz repetition rates.

Fig. 5 Phase change ($\Delta\varphi$) of the laser written track as per pulse energies according to the repetition rates (10 and 500 kHz) associated QPM images of the NYSr crystal.

Fig. 6 PL spectra of NYSr crystal waveguides as a function of pulse energies. The inset shows the comparison study of the SrF_2 and CaF_2 crystals.

Fig. 7 NIR PL spectra of NYSr crystal before and after femtosecond laser irradiation under 808 nm Xenon-lamp excitation.

Fig. 8 Luminescence decay curves of NYSr crystal before and after femtosecond laser irradiation (λ_{ex}=808 nm; λ_{em}=1056 nm).