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The effect of excitation intensity variation and silver nanoparticle codoping on nonlinear optical properties of mixed tellurite and zinc oxide glass doped with Nd_2O_3 studied through ultrafast z-scan spectroscopy

L. Moreira [a], R.F. Falci [a], H. Darabian [a], V. Anjos [a], M.J.V. Bell [a,*], L.R.P. Kassab [b], C.D.S. Bordon [c], J.L. Doualan [d], P. Camy [d], R. Moncorgé [d]

[a] Laboratório de Espectroscopia de Materiais, Departamento de Física, Universidade Federal de Juiz de Fora, Juiz de Fora-MG, Brazil
[b] Laboratório de Tecnologia em Materiais Fotônicos e Optoeletrônicos, Faculdade de Tecnologia de São Paulo, CEETEPS/UNESP, São Paulo, Brazil
[c] Departamento de Engenharia de Sistemas Eletrônicos, Escola Politécnica da USP, São Paulo, SP, Brazil
[d] Centre de recherche sur les Ions, les Matériaux et la Photonique (CIMAP), UMR, CNRS-CEA-Ensicaen, Universite de Caen, 6 Boulevard Maréchal Juin, F-14050, Caen, France

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ABSTRACT

The research on Nd^{3+} doped new solid-state laser hosts with specific thermo-mechanical and optical properties is very active. Nd^{3+} doped tellurite glasses are suitable for these applications. They have high linear and nonlinear refraction index, wide transmittance range. The TeO_2-ZnO (TZO) glass considered in the present work combines all those features and the nonlinear optical properties can be used for the development of Kerr-lens mode-locked sub picosecond lasers. Recently the laser performance of Nd^{3+} doped TZO glass and was reported and laser slope efficiency of 21% was observed. We investigate how the intensity variation and the silver nanoparticles codoping affects the nonlinear optical properties of Nd^{3+} doped TZO glasses. Intensity dependent nonlinear refraction indices coefficients at 750, 800 and 850 nm were observed. The nonlinear optical features were obtained through ultrafast single beam z-scan technique with excitations at 750, 800 and 850 nm and are up to two orders of magnitude higher than those reported in the literature.

1. Introduction

Nd^{3+} doped laser materials have a wide variety of applications such as short pulse with high peak power laser systems. Thus, they are very attractive and extensively studied. Also, they have a very interesting ensemble of features, namely, easier 4-levels laser operation mode and usually higher gain cross sections if compared to Yb^{3+} doped laser materials [1]. Even though laser action of Nd^{3+} has been observed in a many solid media such as Nd:YAG systems, the research on Nd^{3+} doped new solid-state laser hosts with specific thermo-mechanical and optical properties is very active. This is the case of some Nd doped nonlinear tellurite glasses [2–8]. They have a conjunction of good thermo-mechanical properties, typical of crystals, and broad-band spectral properties, typical of glasses. Also, a very interesting combination of large nonlinear refraction index (25 times larger than that of silica), wide transmittance range [9].

Usually, crystalline laser hosts lead to higher absorption and emission cross sections, while glasses are produced in larger volumes with optimal optical quality at lower cost. In order to minimize the non-radiative multiphonon relaxations and to optimize the quantum efficiency of the $^4F_{3/2} \rightarrow {}^4I_{11/2}$ emission of Nd^{3+}, it is also suitable to work with Nd^{3+} doped host materials with low contents of OH impurities. In that sense, laser emission of Nd^{3+} in glasses has been reported in fluorides [10–12], chalcogenides [13], aluminosilicates [14], germinates [15], and, as just mentioned, in tellurite glasses [2–6]. Among oxi-tellurites, the TeO_2-ZnO glass which is considered here in the present article combines good mechanical stability, chemical durability, high linear refraction index together with a wide transmission window (0.4–6 μm) and a

* Corresponding author.
E-mail addresses: mjbell@fisica.ufjf.br, mjbell06@gmail.com (M.J.V. Bell).

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high rare-earth solubility [10,16,17]. The large linear refraction index (1.97) [18] of this tellurite glass imply large stimulated emission cross-sections, sometimes larger than for phosphate glasses [19]. These glasses also have high nonlinear optical properties, which can be used advantageously for the development of Kerr-lens mode-locked sub picosecond lasers.

These tellurite glasses have been also studied recently for the possibility of using thin films for the fabrication of rib waveguides [20]. The possibility of increasing the luminescent quantum yield of rare-earth ions by codoping TeO_2-ZnO glasses with silver nanoparticles [21,22] showed that they are potential materials for photonic devices applications. Recently it was demonstrated the reversible memory phenomena in Au-nanoparticles-incorporated TeO_2–ZnO films [23]. Also, encouraging improvements have been reported regarding the laser performance of a Nd^{3+} doped TZO (TeO2-ZnO) glass [24,25]. In this work, laser slope efficiency of 21% was observed. Thermo-optical properties of tellurite glasses codoped with rare Earth ions and metallic nanoparticles have also been reported [26–28].

These results motivated the present study that reports how the incident intensity variation and the silver nanoparticles codoping affects the nonlinear optical properties of Nd^{3+} doped TZO glasses.

Measurements were carried out with the Z-scan technique. The results displayed high intensity dependent nonlinear refraction indices at the wavelength range of 750–850 nm, at 80 MHz repetition rate and 100 fs pulses.

2. Experimental details

Glasses with the composition 85%TeO2-15%ZnO (wt.) (TZO) were obtained with the addition of 1% Nd_2O_3 and 1% $AgNO_3$ (wt.) (TZO:*1%*Nd:*1%*Ag). Also a sample without $AgNO_3$ (*TZO:1%Nd*) was prepared to be used as reference Reagents were melted at 800 °C in an platinum crucible for 20 min, quenched in a pre-heated brass mold, annealed at 325 °C for 2 h, and cooled down to room temperature during 2 h to avoid internal stresses. At the end of the fabrication process, an additional heat treatment was performed for 24 h, to the sample produced with $AgNO_3$ to thermally reduce the Ag^+ ions to Ag^0 and nucleate silver nanoparticles, following the procedure already reported [21,22]. Through this procedure it was possible to obtain high quality samples, with even distribution of the dopants within the glass matrix (TZO).

Absorption spectra were measured in a Perkin-Elmer LAMBDA 9 spectrophotometer in wavelength range from 350 to 1000 nm.

A 200 kV transmission electron microscope (TEM) was employed to investigate the presence of nanoparticles in the samples.

The nonlinear optical features were obtained through ultrafast single beam z-scan setup displayed in Fig. 1. The excitation beam is a Mai Tai HP, Ti:Sapphire NIR, 100 fs, 80 MHz pulsed Gaussian beam linearly polarized. A Glan-Laser linear polarizer (GL5) positioned at the laser output controls the output intensity. Just after the GL5, a lens focuses the excitation beam in the sample posed on a displacement stage. After crossing the sample the beam goes through the aperture that is set either open or partially (50%) closed depending on the experiment needs – nonlinear refraction demands closed aperture, while nonlinear absorption demands open aperture. Then, the beam crosses a lens that collimates the signal into a silicon detector connected to a computer, by which the data acquisition is performed.

3. Absorption spectra and TEM imaging

The UV-VIS-NIR absorption (absorption coefficient) spectrum of the samples, registered between 350 nm and 950 nm is shown in Fig. 2. The features corresponding to the main absorption transitions of Nd^{3+} from $^4I_{9/2}$ fundamental level to excited levels $^4F_{3/2}$(890 nm), $^4F_{5/2}+^2H_{9/2}$(808 nm), $^4F_{7/2}+^4S_{3/2}$(750 nm), $^4F_{9/2}$(690 nm), $^4G_{5/2}+^2G_{7/2}$(580 nm) and $^4G_{7/2}+^4G_{9/2}+^2K_{13/2}$ have been identified and highlighted in the figure. As expected for a glass, the absorption features appear as broad bands. Also it is interesting to notice that the addition of silver nanoparticles (TZO:1%Nd:1%Ag) to the initial composition (TZO:1%Nd) have resulted in a reasonable enhancement of the absorption. This effect may be attributed to the

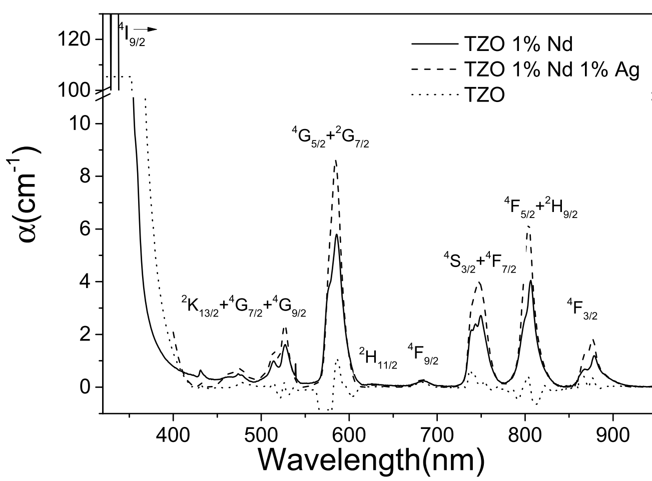

Fig. 2. UV–Vis–NIR absorption spectra for TZO doped and undoped samples.

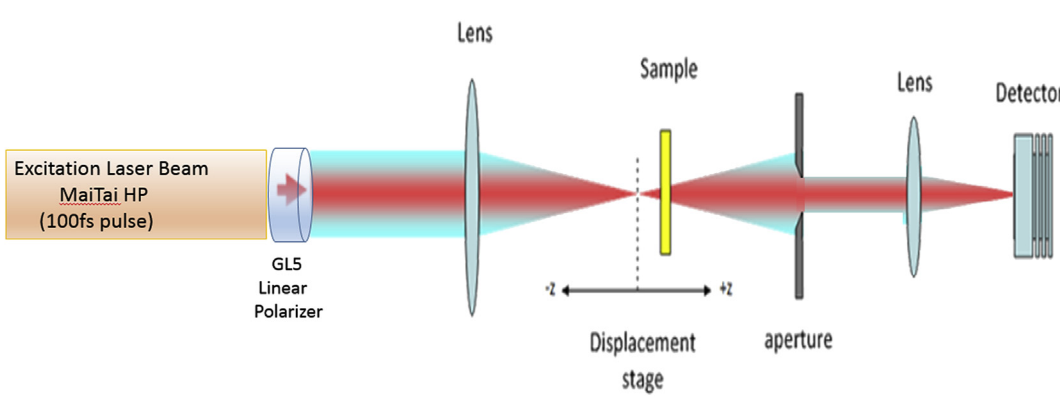

Fig. 1. Z-scan experimental setup [28].

Table 1
TZO samples parameters.

Sample	Thickness l(mm)	Linear absorption coefficient α_0(cm^{-1})	Wavelength (nm)
TZO:1%Nd	1,65	2,64	750
		2,61	800
		0	850
TZO:1%Nd:1%Ag	2,98	3,91	750
		4,94	800
		0	850

modification of the Nd^{3+} ion environment due to the presence of Ag nanoparticles. 29 Furthermore, when the TZO:1%Nd and TZO:1% Nd:1%Ag samples are compared with the matrix (TZO) it is possible to notice the absence of optical absorption above 500 nm. The linear absorption coefficients (α) correspondent to the chosen excitation wavelength for each sample can be found at Table 1.

Fig. 3 (left) presents TEM images of TZO:1%Nd:1%Ag sample that assures the existence of the silver nanoparticles. We observe isolated silver nanoparticles with average size of 20 nm. Fig. 3 (right) shows the simulated absorption spectra for 20 nm silver nanoparticles embedded TZO glass, where two resonant plasmons at 425 and 500 nm are evidenced [30].

4. Nonlinear refraction

Nonlinear refraction of the samples was examined with the aid of the z-scan setup depicted on Fig. 1. To evaluate the nonlinear refraction of a transparent medium the aperture should be set partially closed, thus known as closed aperture (C.A.) experiment. Namely, the aperture is set open by 40%, to let only the center of the spot at far field pass to the detector.

Figs. 4–6 display the normalized transmittance for the samples TZO:1%Nd and TZO:1%Nd1%Ag. Since the TZO sample has not shown any optical nonlinear behavior for the wavelengths analyzed here, there is no available data. The experiments were carried for the excitation intensities within the range 0.05–0.45 GW/cm^2. No higher intensity was available due to limitations of the system, including the damage limit threshold for the samples TZO:1%Nd and TZO:1%Nd1%Ag. The samples were excited at 750, 800 and 850 nm. The different wavelengths used for excitation and presented in Figs. 4–6 have been based on the nonlinear optical refraction responses detected. The TZO:1%Nd sample showed higher sensitivity at 750 and 800 nm, whereas TZO:1%Nd:1%Ag at 800 and 850 nm. This difference can be attributed to the silver nanoparticles interaction with the matrix TZO.

The 3rd order nonlinear refraction effect could be observed just for the TZO:1%Nd and TZO:1%Nd1%Ag samples. Additionally, no relevant nonlinear absorption was observed at any of the tested samples. Thus, the data obtained from these measurements were fitted to the following expression [31,32]:

$$\Delta T_{PV} \cong 0,406(1-S)^{0,27}|\Delta\Phi_0| \quad (1)$$

$$\Delta\Phi_0 = \left(\frac{2\pi}{\lambda}\right) Z_0 I_0 n_2 l_{eff}, \;\; l_{eff} = \frac{1-e^{-\alpha l}}{\alpha}, \; Z_0 = \frac{\pi\omega_0^2}{\lambda} \quad (2)$$

where $\Delta T_{PV} = T_P - T_V$ is the change in transmittance between the peak and valley, and T_P and T_V are the normalized peak and valley transmittances, $\Delta\Phi_0$ is the nonlinear phase with the sample at the focus, Z_0 is the Rayleigh length, Z the sample position relative to the focus (Z = 0 mm), λ is the excitation laser wavelength, n_2 is the 3rd order nonlinear refraction index and I_0 is the peak intensity, ω_0 is the beam waist at the focus, S is the transmittance of the aperture in the absence of a sample, here $S = 0{,}4$.

Figs. 4–6 show a similar nonlinear behavior of the samples, self-focusing Kerr lenses in all cases studied. The amplitude of the signal denotes the value $\Delta\Phi_0$. The TZO:1%Nd results at 750 nm (Fig. 6A) and 800 nm (Fig. 5A), evidence that $\Delta\Phi_0$ increases with rise of the excitation intensity, but for 850 nm (Fig. 4A) the reverse situation is observed. The TZO:1%Nd1%Ag sample exhibits the same nonlinear optical behavior for the three excitation wavelengths but with higher amplitudes, indicating that the presence of the silver nanoparticles in the TZO:1%Nd 1%Ag enhanced the nonlinear optical features.

The results of the 3rd order nonlinear refraction index (n_2) as function of the excitation intensity, are shown in Fig. 7. It is possible to notice that n_2 shows an exponential decay behavior with the rise of I_0. We observe high values for n_2 that reaches 4×10^{-12} cm^2/W, at 850 nm (0.05 GW/cm^2), for the sample with silver nanoparticles whereas in the absence of silver nanoparticles n_2 is significantly lower, around 2×10^{-13} cm^2/W. It was observed enhancement of about 50%, in the presence of silver nanoparticles, at 800 nm, for the same excitation intensity (0.3 GW/cm^2). Furthermore, a fitting

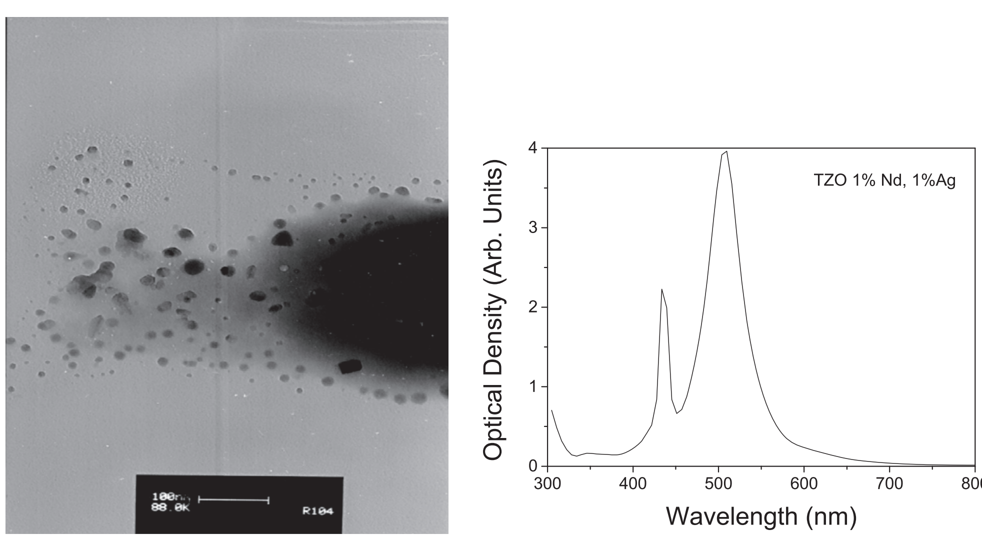

Fig. 3. TEM Image of the TZO:1%Nd:1%Ag (left) and the Simulated absorption spectra for 20 nm Silver Nanoparticles embedded at TZO glass [29](right).

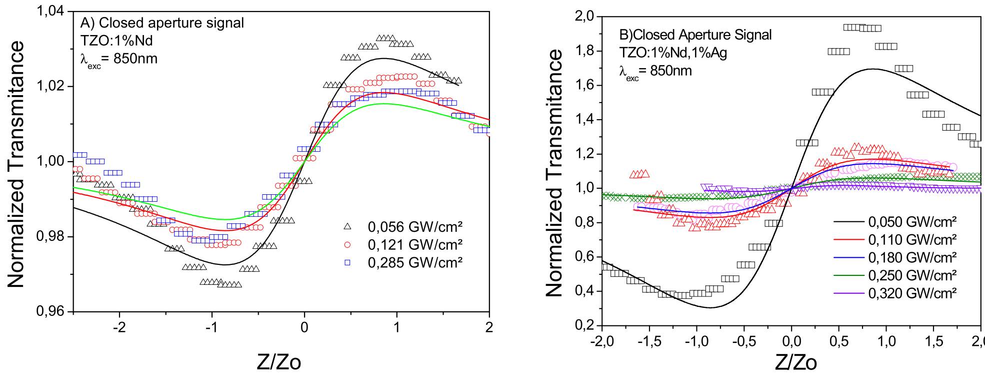

Fig. 4. Closed Aperture (C.A.) Z-scan signal at 850 nm **for different excitation intensities.**

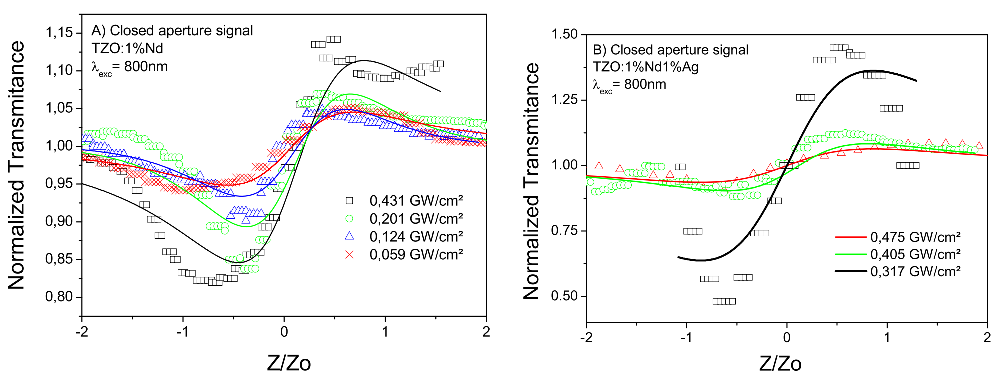

Fig. 5. Closed Aperture (C.A.) Z-scan signals at 800 nm **for different excitation intensities**.

was performed using the empirical expression (3) in order to obtain the 3rd order nonlinear refraction indices for high intensity excitation, $n_{2,\infty}$. The results are displayed in Table 2.

$$n_2(I_0) = n_{2,I}\exp(-I_0/I_{n2}) + n_{2,\infty} \tag{3}$$

In the equation above $n_2(I_0)$ is the 3rd order nonlinear refraction index as function of the excitation intensity I_0, $n_{2,I}$ is the amplitude, I_{n2} is the decay constant and $n_{2,\infty}$ is the 3rd order nonlinear refraction index at high intensity excitation.

Regarding the $n_{2,\infty}$, it is possible to observe a significant rise at 850 nm with addition of silver nanoparticles. However, for the lower excitation wavelengths, $n_{2,\infty}$ is diminished roughly by half. Even though this result is unexpected, we should consider the fact that the addition of metallic nanoparticles to an emission media, should favor radiative transitions over other energy transfer processes. In fact, 750 nm and 800 nm lay among pumping wavelengths for the Nd^{3+} ion, as can be seen in Fig. 1. Thus it is possible to expect a significant enhancement in emission, when silver nanoparticles are added.

The evolution of the nonlinear indices with the increasing intensity have been observed and discussed before, even if not analyzed mathematically. The behavior of n_2 variation due to increasing excitation intensities have been reported by A.S. Reyna and C. B. de Araujo [33] and also by R.A.Ganeev et al. [34]. They reported as the most likely reasons for the n_2 changes due to excitation rise:

(1) Interband transitions taking into account the possibility of two-photon process [34]. This process can be taken into

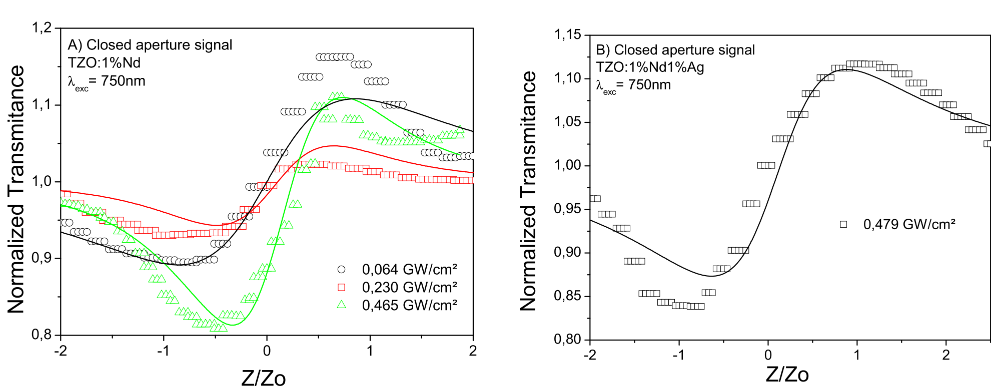

Fig. 6. Closed Aperture (C.A.) Z-scan signals at 750 nm **for different excitation intensities**.

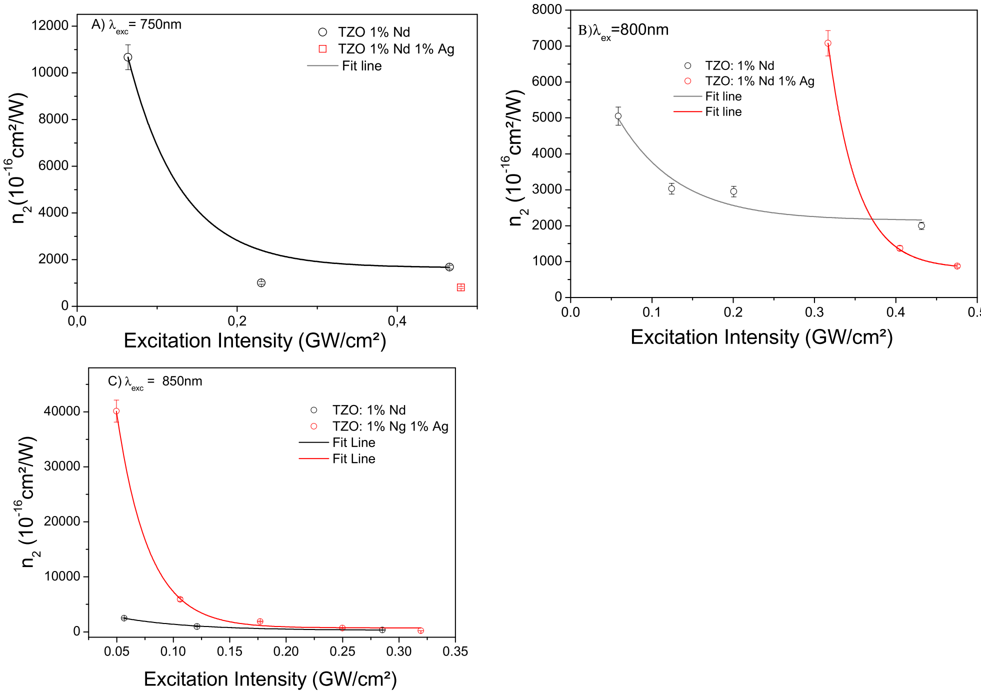

Fig. 7. Nonlinear Refraction Index (n_2) as Function of the Excitation Intensity at A) 750 nm, B) 800 nm, C) 850 nm.

Table 2
TZO nonlinear refraction indices (n_2) as function of the excitation intensity (I_0).

$n_2(I_0) = n_{2,I}\exp(-I_0/I_{n2}) + n_{2,\infty}$

Sample	Wavelength(nm)	$n_{2,\infty}(10^{-14}\,cm^2/W)$	$n_{2,I}(10^{-13}\,cm^2/W)$	I_{n2}(GW)
TZO:Nd	750	16,584	23,356	0,067
	800	21,411	6275	0,045
	850	2655	5355	0,063
TZO:Nd:Ag	750	8073		
	800	8008	4.383,840	0,036
	850	7188	229,350	0,028

consideration for the TZO:1%Nd1%Ag sample since it has shown resonance plasmon at 425 nm and was excited at 850 nm.

(2) Thermal effect. Positive contribution to the nonlinear refractive index can be caused by thermal effect, which can be considered as a result of energy transfer from heated dopant to surrounding dielectric matrix. However, the time necessary for this process to be important corresponds to a few nanoseconds, whereas the pulse duration was five orders shorter (100 fs), which diminishes the influence of the thermal effect causing the acoustic-induced variation of density and refractive index of matrix [34].

(3) The irreversible change of n_2 caused by laser radiation. This effect was analyzed in several studies of composite materials doped with copper and silver nanoparticles. The mechanism responsible for the change was a photochemical reaction, which produced a silver-oxide layer on the surface of nano-clusters. The irreversible changes in both studies were caused by a thermal influence produced by high pulse repetition rate radiation [33]. In our case, the Z-scans had a good reproducibility in time, so the influence of irreversible changes can be easily excluded.

It also possible to evaluate the results of n_2 for similar materials. S.K.Mahajan reported $n_2 = 1.21 \times 10^{-15}$ cm^2/W, at 800 nm, 100 fs, 80 MHz, for a Er^{3+} doped TeO_2-Li_2O-WO_3 glass [35]. Nonlinear refraction indices of PbO-Nb_2O_5-TeO_2 glasses at 800 nm,90 fs, 1 kHz were studied recently; nonlinear refraction indices in the range 1.42-1.78 $\times 10^{-14}$ cm^2/W were obtained for different concentration of PbO-Nb_2O_5-TeO_2. Another interesting result has been obtained by K. Kato [36], who studied the optical nonlinearities of $20Ag_2O-80TeO_2$ glasses, their highest value was $n_2 = 8.75 \times 10^{-15}$ cm^2/W, at 800 nm,90 fs,1 kHz.

D.Linda observed the role of silver nanoparticles on nonlinear optical properties at $TeO_2-Ti_2O-Ag_2O$ and $TeO_2-ZnO-Ag_2O$ ternary systems [37]. The silver nanoparticles increased the energy transfer among the ions that build the matrix generating enhancement of n_2 ranging from 28 to 40×10^{-14} cm^2/W at 800 nm, 90 fs, 1 KHz. Goméz observed the nonlinearities of silver nanoparticles in colloidal media, and obtained n_2 around 10^{-15} cm^2/W(533 nm,8ns, 10 Hz) and linear absorption peak around 500 nm [38]. In the present study we have added 20 nm silver nanoparticles to the original composition of TZO:1%Nd. Through simulation [29] it was possible to obtain the nanoparticles absorption spectrum embedded in TZO:1%Nd medium, available at Fig. 6. We can observe two peaks, around 425 nm and 500 nm. This would justify the enhanced sensibility at 850 nm, for the sample prepared with silver nanoparticles.

Then comparing the results recently reported we conclude that the samples presented in this work have nonlinear refraction indices that are at the same order or up to two orders of magnitude higher than those reported to tellurite glasses in the 750–850 nm range.

5. Conclusion

The nonlinear optical properties of Nd^{3+} doped TZO glasses with and without silver nanoparticles is studied. The nonlinear optical features were obtained through ultrafast single beam z-scan technique. The samples were excited at 750, 800 and 850 nm. The 3rd order nonlinear refraction index (n_2) shows an exponential decay behavior with the rise of the excitation intensity. Then it reaches a constant value at high intensity values. In comparison to the glasses available in the literature, the samples presented in this work have nonlinear refraction indices that at the same order or up to two orders of magnitude higher than those reported to tellurite glasses in the 750–850 nm range. The enhanced nonlinear refraction index due to the increased local field that surrounds the silver nanoparticles could be observed. The high nonlinear properties of the samples presented here make them good candidates for subpico Kerr effect based lasers. Likewise, they demand further examinations such as recovery time and damage threshold to better define the potential for possible applications.

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