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At the 70th Anniversary of Professor Vlad, one team in nonlinear optics

MEASUREMENT OF THE NONLINEAR REFRACTIVE INDEX OF SOME SILICON NANOSTRUCTURES USING REFLECTION INTENSITY SCAN METHOD

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Abstract. In this paper, we study the magnitude and the sign of optical refractive index of Si nanostructures (nanoporous silicon and 1D periodic silicon-on-insulator nanostructures) using the reflection intensity scan method, which was developed in our laboratory. Our experimental results were reproducible and in good agreement with theoretical predictions, proving the efficiency of this method in the characterization of nonlinear properties of inhomogeneous media and of small areas containing patterns of micro- and nano-structures.

Key words: reflection intensity scan, third-order nonlinearities, inhomogeneous nanostructures, 1D periodically nanostructures, nano-porous silicon, silicon-on-insulator.

1. INTRODUCTION

In nonlinear photonics, the investigation of the optical nonlinear refractive index of nonlinear materials, such as semiconductors (bulk and nanostructured silicon) [1-9], isolators [1,10], quantum dots [1,11,12] and organic materials [13], provides information about interaction of light with these media and this is important for basic research and for applications in photonics, materials science, information technology etc.

Silicon (Si) is an important nonlinear material in photonics [1–9], this being used in realization of optical modulators, amplifiers, photodetectors etc. [14, 15]. In time, special methods were developed in order to achieve Si nanostructures, such as: nano-porous silicon [16], periodic Si nanostructures, silicon-on-insulator (SOI) etc. [17], these being very important in realization of waveguides, optical switches, lasers, photodetectors, solar cells, sensors etc [14, 15, 18–24]. Many applications of

Si nanostructures depend on the values of their third-order optical nonlinearities, so these properties were studied for various Si composite materials (such as: nano-porous Si and periodic 1D nanostructures on SOI) by using many characterization methods, among them the Z-scan technique [1, 4–9, 25–29].

The Z-scan is a simple and sensitive optical method for measuring the magnitude and sign of nonlinear refractive index and of nonlinear absorption coefficient of transparent materials. This method was introduced by M. Sheik-Bahae, A. A. Said and E. W. Van Stryland [30], in 1989. In Z-scan, the sample is moved along the laser beam propagation direction (z-axis), in front and back of the focal plane of a focusing lens and this action lead to the changes of the excitation intensity with the distance from the focus. These changes modify the phase and / or the amplitude of the transmitted beam, which are monitored in the far field [1-3, 10-13]. Since 1989, further experimental variations and theoretical models of this method were developed, in order to investigate different types of nonlinear materials [1, 4-9, 31-53].

In order to investigate the optical nonlinearities of the highly absorbing and reflecting materials, or the optical nonlinearities at the interfaces of materials, D.V. Petrov [44] and later M. Martinelli et al [45, 46] developed the reflection Z-scan (RZ-scan) method [1, 4, 5, 8, 44–52].

Since the transmission Z-scan method presented some drawbacks in the optical nonlinear characterization of a large range of materials including those with a low damage threshold, such as polymers, absorbing glasses and nanoparticles immersed in different solvents or glasses, Taheri et al. [53] have introduced a new single beam method, named transmission intensity scan (TI-scan) for the measurement of magnitude and sign of the nonlinear optical response of materials using intensity dependence of the complex refractive index.

Starting from a proposal of V. I. Vlad, we developed the theory and the first experimental setup for reflection I-scan (RI-scan) method (to the best of our knowledge) for the investigation of optical nonlinearities of the highly absorbing and reflecting inhomogeneous structured thin film (such as nanoporous silicon layers on Si wafers) and of small areas containing patterns of micro- and nano-structures (such as 1D periodically nanostructures on small areas of silicon-on-insulator (SOI) wafer) [6-9].

Below, we present a description of RI-scan method and our experimental results obtained with this method in investigation of optical nonlinearities of nano-porous Si and of 1D periodic nanostructure on SOI.

2. DESCRIPTION OF THE RI-SCAN METHOD

The experimental setup for RI-scan resembles with that for RZ-scan, up to the particularity of keeping the sample fixed at approximately a Rayleigh length behind or in front of the focal plane of the focusing lens and varying the laser intensity by a variable attenuator. In comparison with RZ-scan, the RI-scan method presents several advantages in measuring the optical nonlinear response of those materials described above [6–9, 53]:

– the same area of the sample is illuminated during the experiments and not continuously varying as in the case of Z-scan, this being very important for investigation of small areas or of inhomogeneous nanostructures;

– the sample is not passing through the focal plane of the focusing lens, thus avoiding the damaging of the nanostructures;

– the total exposure time of the sample is reduced, so other sample distortions are smaller;

– the lack of moving elements in the experimental setup leads to a considerable minimization of the misalignment or other errors introduced by optical components.

In RI-scan experimental setup can be used laser beams in continuous wave (c.w.) regime or in pulsed regime, having different wavelengths and various repetition rate (nanosecond, picosecond, femtosecond), with the condition to adapt the optical components according to the laser beam utilized in the experiment. In experiments using lasers beams in c.w. regime, the slow response of the sample is investigated. By using laser beams in pulsed regime, the fast third-order nonlinear response or high-order nonlinearities are obtained. Below, we present the theoretical formulae useful in analysis of the slow and fast nonlinear responses of the nonlinear highly absorbing materials by RI-scan method.

Starting from the theoretical proposal by D. V. Petrov for RZ-scan [44] and the implementations of Martinelli et al [45, 46], we have derived the dependence of nonlinear normalized reflection (the ratio between the power reflected by the sample with and without the nonlinear effect) on total refractive index change, Δn, as [1, 4, 6–9]:

$$R = 1 + \frac{2 \cdot \Delta n}{n_0^2 - 1} \cdot \frac{1}{1 + \left(z / z_R \right)^2}, \tag{1}$$

where: $z_R = \pi w_0^2 / \lambda$ is the Rayleigh length of the beam, z is the distance between the focus and the investigated sample, w_0 is the beam waist, n_0 is the linear refractive index of the sample. Eq. (1) holds for Gaussian beams and $n_0 >> k_0$ (k_0 is the linear extinction coefficient; for Si, $n_{0Si} = 3.7$, $k_0 = k_{Si} = 0.006$).

In order to study the nonlinear response of the investigated materials, we have to discriminate the slow and the fast nonlinearities. The slow nonlinearities, n_{2slow}, are present both in the case of continuous wave (c.w.) and high-repetition-rate fast excitation (when are produced by the average incident intensity), leading to the nonlinear induced refractive index change [1, 4]:

$$\Delta n_{c.w.} = n_{2slow} I_{0c.w.}, \tag{2}$$

where: $I_{0c.w.}$ is the on-axis c.w. laser beam intensity at distance z_R from the focus.
In the case of high-repetition-rate fs excitation, following Vlad's hypothesis, the slow and the fast nonlinear induced refractive index changes are additive (as they are very small, in steady-state, and one can take the first term only in a development in power series) [1, 4]:

$$\Delta n_{pulse} = n_{2slow} I_{average} + n_{2fast} I_{peak}, \tag{3}$$

where $I_{average}$ is the laser beam intensity calculated with the average power of the beam ($I_{average} = I_0$), n_{2fast} is the fast nonlinear refractive index of the sample from pulsed experiments, I_{peak} is the peak intensity of the femtosecond pulses. $I_{peak} = (T/\tau)\cdot I_0$, $T = 1/\nu_{laser}$, ν_{laser} is the repetition rate of the laser, τ is the pulse duration. Using (2) and (3), one can deduce n_{2fast}.

We introduced this method for measurement of slow (thermal and electronic) nonlinearity and fast (eventually electronic) nonlinearity of SOI nanostructures, using a c.w. laser and a fast femto-laser and named it double reflection I-scan [9].

In Fig. 1, a scheme of RI-scan experimental setup is presented. The laser beam passes through a variable attenuator, A, in order to modify the laser intensity. Then, the beam is focalized with lens L_1 and the sample is placed at approximately a Rayleigh length ($z = z_R$) behind of the focal plane of L_1, this leading to the simplification of Eq. (1):

$$R = 1 + \frac{\Delta n}{n_0^2 - 1}. \tag{4}$$

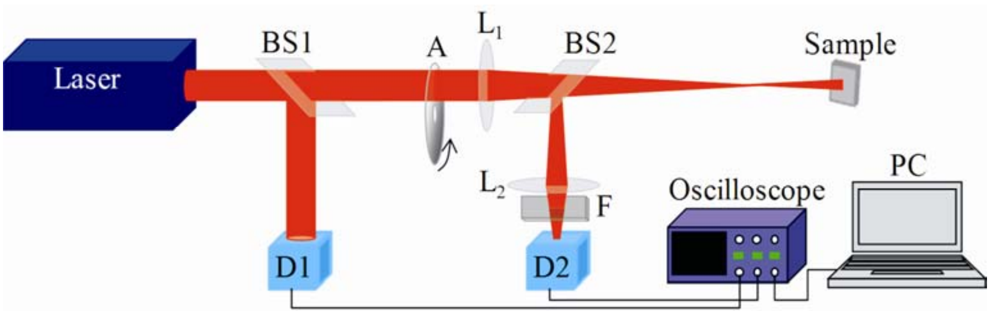


Fig. 1 – RI-scan experimental setup: laser, A – variable attenuator, L_1, L_2- lenses, BS1, BS2 – beam-splitters, D1, D2 – detectors, F – neutral filter.

The magnitude and sign of the optical nonlinear response can be obtained by analyzing the reflected signal from the sample, in the far field. A beam splitter, BS2, directs the reflected signal on a detector (D2) and the experimental data are sent, for processing, to an oscilloscope connected to a computer. The lens L_2 has the role to focalize the reflected signal on the detector matrix and the filter F has the role to attenuate the intensity of that signal, in order not to saturate the detector. Using the beam-splitter BS1 and the detector D1, the monitoring of the excitation laser beam is realized.

We have to mention that for measuring the values of nonlinear refractive index of the investigated samples, the open-aperture configuration is used (when there is no aperture in the front of D2). Instead, for measuring the nonlinear absorption coefficient, in front of the D2 is placed a small aperture (close-aperture configuration) and the sample is placed in the focal plane of the focusing lens, L_1.

3. RI-SCAN EXPERIMENTAL DATA ON NANOSTRUCTURED NONLINEAR MATERIALS

3.1. NANO-POROUS SILICON

The nonlinear refractive responses of nano-porous silicon (np-Si) were investigated by many researchers. Due to its convenient production processing, emission, nonlinear optical properties and infiltration possibilities, the np-Si is a nano-composite material with many applications in photonics. This inhomogeneous material shows controllable linear and nonlinear optical properties, which are interesting for photonic devices [14, 15, 54]. The np-Si is formed by two randomly intermixed components (silicon and air) and the sizes of silicon (Si) structures (in the order of tens nanometers) are much smaller than the excitation wavelength, so that this composite can be considered as an effective material with refractive index between that of air and Si [5–8]. In order to calculate rapidly and accurate the effective optical linear refractive index and third-order nonlinearity of np-Si layers, in function of Si volume fill fraction (f_{Si}) and of excitation wavelength (λ), Vlad proposed a simplified formalism starting from Bruggeman model and Boyd equations [5–8, 55, 56]. The full procedure, through which these simplified relations were obtained, for samples having $f_{Si} \leq 0.5$ and for excitation wavelength $\lambda \leq 1$ μm, is described in our previous work presented in Ref. [7, 8]. Our simple approximate formulae are [7, 8]:

$$n_{eff} \approx 3.07 \cdot f_{Si} - 0.148 \cdot \lambda + 0.812\,, \tag{5}$$

$$\frac{\chi_{eff}^{(3)}}{\chi_{Si}^{(3)}} \approx 1.8 \cdot f_{Si}^{2} - 0.735 \cdot f_{Si} + 0.007 \cdot \lambda + 0.072\,, \tag{6}$$

where n_{eff} is the effective linear refractive index of np-Si, $\chi_{eff}^{(3)}$ and $\chi_{Si}^{(3)}$ are the third-order nonlinear susceptibilities of np-Si layers and bulk Si, respectively.

The third-order nonlinear optical susceptibility $\chi^{(3)}\ [\text{esu}] \approx 12.7 \cdot n_{eff}^2 \cdot n_2$ [cm^2/W] is proportional [7] to the nonlinear refractive index of the investigated material, n_2, where n_{eff} can be expressed as in our simplified formula (5). Replacing the third-order nonlinear optical susceptibilities in Eq. (1), we derived the equation that describes the dependence of effective nonlinear refractive index on the measured reflection in RI-scan, for np-Si layers with different f_{Si} and for various excitation wavelengths:

$$\frac{n_{2\,np-Si}}{n_{2\,Si}} \approx \frac{R_{np-Si}-1}{R_{Si}-1} \cdot \frac{\left[\left(3.07 \cdot f_{Si} - 0.148 \cdot \lambda + 0.812\right)^2 - 1\right]}{\left(n_{Si}^2 - 1\right)}, \tag{7}$$

where $R_{np\text{-}Si}$ and R_{Si} are normalized nonlinear reflections of np-Si layers and bulk Si, respectively, $n_{2\ np\text{-}Si}$ and n_{2Si} are the nonlinear refractive indices of np-Si and bulk Si, respectively.

In Eq. (7), n_{Si} dependence on wavelength is described by Sellmeier dispersion formula (T = 300 K) [57]:

$$n_{Si}^2 = 1 + \frac{A_1\lambda^2}{\lambda^2 - B_1^2} + \frac{A_2\lambda^2}{\lambda^2 - B_2^2} + \frac{A_3\lambda^2}{\lambda^2 - B_3^2} \tag{8}$$

where λ is the light wavelength (in µm) and the Sellmeier coefficients for Si, valid in the range 0.36–2.33 µm, are [57]:

$$A_1 = 1.06684293 \cdot 10,\ A_2 = 3.04347484 \cdot 10^{-3},\ A_3 = 1.54133408,$$
$$B_1 = 3.01516485 \cdot 10^{-1},\ B_2 = 1.13475115,\ B_3 = 1.104 \cdot 10^{3}.$$

Our experimental study of np-Si layers were made by using low intensity lasers, in c.w. regime, with wavelengths at 633 nm, 664 nm and 808 nm. We have to mention that our np-Si layers are not removed from the substrate of Si bulk and have a large reflectivity, these being the reasons for using the RI-scan method. The RI-scan measurements were done in more different areas of np-Si samples and an averaged value for every sample was calculated. The results obtained for nano-porous Si samples with different f_{Si} were compared with those obtained in RI-scan experiments on bulk Si, at the same wavelengths, in order to demonstrate that there are differences between the optical nonlinearities of these np-Si layers with different f_{Si}.

The RI-scan experimental data obtained in investigation of nonlinear refractive responses of bulk Si and np-Si samples with different f_{Si}, at the excitation wavelength of 664 nm, are presented in Fig. 2. The experimental data were fitted with Eq. (4).

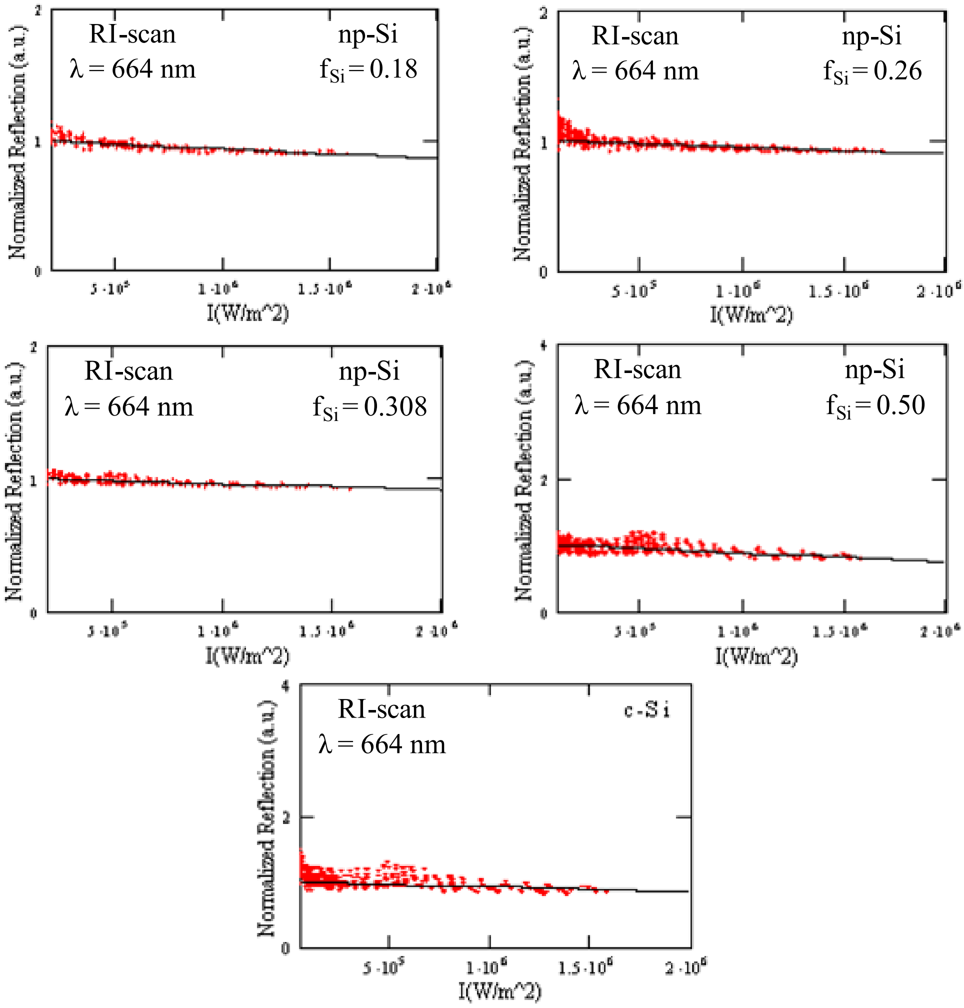


Fig. 2 – RI-Scan experimental data and for nano-porous silicon layers with different f_{Si} and for bulk Si (c-Si), at 664 nm excitation wavelength.

In Table 1, we present the average experimental values obtained with RI-scan technique for $n_{2np\text{-}Si}$ /n_{2Si} dependencies on different f_{Si} and on laser wavelength.

In Fig 3, a comparison between our experimental results and the theoretical predictions of our simplified formula (7) is presented. As we presented above, these theoretical formulae are available for samples having $f_{Si} \leq 0.5$ and for the interval of the excitation wavelength: 600 nm ≤ λ ≤ 1 µm.

Table 1

RI-scan average experimental values for $n_{2\,np-Si}/n_{2\,Si}$ for np-Si samples with different f_{Si}

f_{Si}	$n_{2\,np-Si}/n_{2\,Si}$		
	λ = 633 nm	λ = 664 nm	λ = 808 nm
0.18	$(3.67 \pm 1.04)\cdot 10^{-2}$	$(3.73 \pm 0.08)\cdot 10^{-2}$	
0.26	$(7.44 \pm 0.33)\cdot 10^{-2}$	$(5.04 \pm 0.30)\cdot 10^{-2}$	$(5.59 \pm 0.28)\cdot 10^{-2}$
0.308	$(9.91 \pm 1.93)\cdot 10^{-2}$	$(9.60 \pm 0.20)\cdot 10^{-2}$	$(10.37 \pm 0.83)\cdot 10^{-2}$
0.5	$(3.93 \pm 0.63)\cdot 10^{-1}$	$(3.97 \pm 0.06)\cdot 10^{-1}$	$(3.82 \pm 0.03)\cdot 10^{-1}$

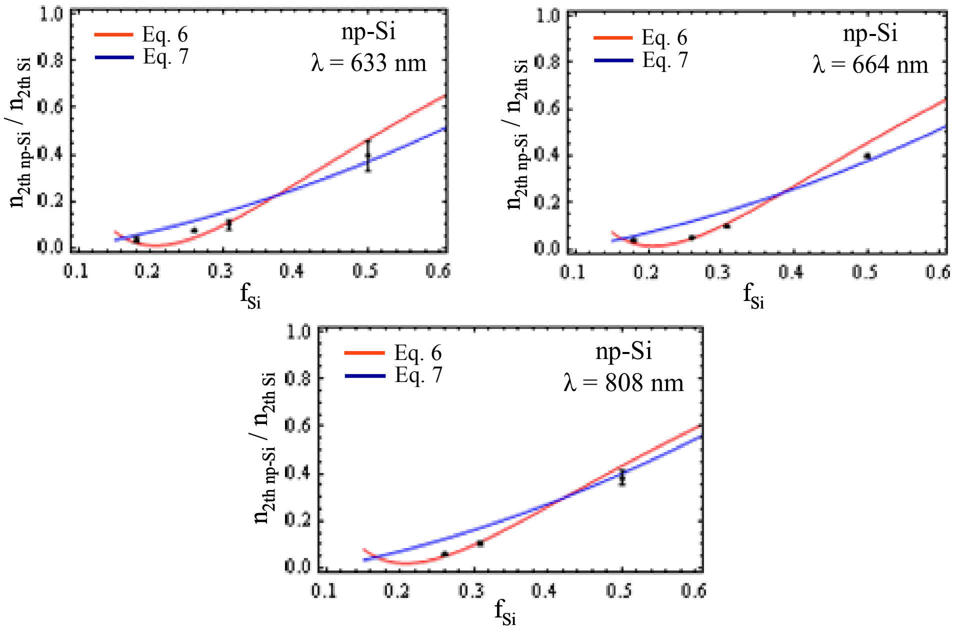


Fig. 3 – Dependences of $n_{2\,np-Si}/n_{2\,Si}$ on the Si volume fill fraction (at fixed wavelengths, in c.w. regime)

The differences between values provided by these relations for $n_{2\,np-Si}/n_{2\,Si}$ are reasonable, taking in account the random structure of np-Si layers and the variations of fabrications parameters.

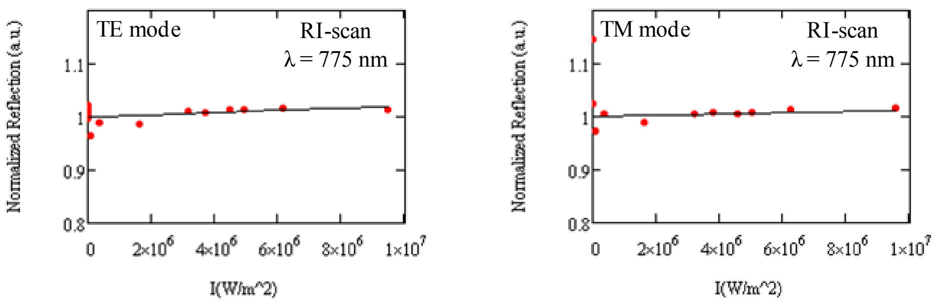
3.2. 1D PERIODICALLY NANOSTRUCTURED SILICON-ON-INSULATOR

We have investigated also the third-order nonlinearities of other nano-composite material, which is silicon-on-insulator (SOI), by using RI-scan method.

Due to the high-index contrast between silicon and silica (SiO_2), the silicon-on-insulator is characterized by strong optical confinement, leading to an enhancement of optical nonlinear effects. If the Si thin layer from the top of SOI wafer is periodically nanostructured, SOI waveguides with a higher nonlinear optical response in comparison with bulk Si or unstructured SOI are obtained [1, 4, 8, 9, 28, 29].

In this section, we present our experimental measurement of the nonlinear refractive properties of 1D periodical SOI nanostructures, by using double RI-scan [9] in c.w. regime (808 nm wavelength) and in femtosecond pulsed regime (775 nm wavelength and 140 fs pulse duration). The experimental results were compared with the data obtained in the same experimental conditions on bulk silicon and unstructured SOI. By separating the thermal effects (and other slow effects), we show that ultrafast electronic third-order optical nonlinearities are larger than those obtained on bulk silicon and unstructured SOI [9]. The RI-scan experiments were realized in more different areas of our 1D periodical SOI nanostructured sample, for the cases when: (a) Si stripes are parallel with laser beam polarization (TE mode) and (b) Si stripes are perpendicular on laser beam polarization (TM mode).

The nanostructure period of our investigated sample is Λ = 543 nm and the effective linear refractive indices of our sample measured by reflectivity experiments (at 808 nm wavelength) are: $n_{eff\parallel}$ = 2.62, for TE mode, and $n_{eff\perp}$ = 2.49, for TM mode; for bulk Si, $n_{eff} = n_{0\ Si}$ = 3.7. The investigated area, containing the 1D periodic nanostructures, has 1 mm^2.



(a) (b)

Fig. 4 – RI-Scan experimental data for 1D periodic SOI nanostructures, at λ = 775 nm in fs pulsed regime, for TE mode (a) and TM mode (b).

In Fig. 4, are presented our experimental RI-scan data for 1D periodic SOI nanostructures, in pulsed regime at 775 nm, for TE and TM polarizations and their fit with the Eq. (2) [9]. The experimental data from the RI-scan in c.w. regime were fitted with Eq. (1) and de value of n_{2th} was replaced in Eq. (2), in order to calculate n_{2el} of our 1D periodic nanostructure.

The thermal and electronic nonlinear responses of 1D periodic nanostructure on SOI show pronounced enhancements with respect to the nonlinear responses of bulk Si or on unstructured SOI (Table 2).

Table 2

The ratios between thermal and electronic nonlinearities of 1D periodic SOI nanostructure and those obtained on bulk Si and on unstructured SOI, for TE and TM modes

		$n_{2\ nanostr}/n_{2\ Si}$	$n_{2\ nanostr}/n_{2\ SOI}$
Thermal nonlinearity	*TE mode*	2.5	2.5
	TM mode	2	2
Electronic nonlinearity	*TE mode*	5	4
	TM mode	3.3	2.7

These enhancements of the thermal and electronic nonlinear responses of 1D periodic nanostructure on SOI in comparison with those obtained for bulk Si and unstructured SOI wafer can be explained by the increase of the electric field at the walls of 1D periodic SOI nanostructures and by the vertical confinement in unstructured SOI [1, 4, 9, 29].

4. CONCLUSIONS

In this paper, we have presented the experimental investigation of third-order nonlinearities of nano-porous silicon (with different Si volume fill fractions) and of 1D periodic SOI nanostructures by using the reflection intensity scan, a method that was proposed and tested firstly by us. We have shown controllable nonlinear properties for nano-porous silicon (with different Si volume fill fractions) and for 1D periodic SOI nanostructures, in some cases, pronounced and size-controllable enhancement of the optical nonlinearity.

The experimental data were reproducible proving the efficiency of this method in the characterization of nonlinear properties of inhomogeneous thin films and of small areas containing patterns of micro- and nano-structures.

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