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Energy transfer upconversion determination by thermal-lens and Z-scan techniques in Nd$^{3+}$-doped laser materials

C. Jacinto,1 D. N. Messias,2,* A. A. Andrade,2 and T. Catunda3

1Instituto de Física, Universidade Federal de Alagoas (UFAL), Maceió-AL, Brasil
2Instituto de Física, Universidade Federal de Uberlândia (UFU), Uberlândia-MG, Brasil
3Instituto de Física de São Carlos, Universidade de São Paulo (USP), São Carlos-SP, Brasil
*Corresponding author: dnmessias@infis.ufu.br

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The Z-scan and thermal-lens techniques have been used to obtain the energy transfer upconversion parameter in Nd$^{3+}$-doped materials. A comparison between these methods is done, showing that they are independent and provide similar results. Moreover, the advantages and applicability of each one are also discussed. The results point to these approaches as valuable alternative methods because of their sensitivity, which allows measurements to be performed in a pump-power regime without causing damage to the investigated material. © 2009 Optical Society of America

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1. INTRODUCTION

Nowadays, high-power solid-state lasers can be achieved through direct diode-laser pumping. Nd$^{3+}$-doped materials are appropriate systems used to produce such efficient power level. For instance, power scaling as high as 2.0 kW has been found in Nd:YAG ceramics [1]. However, in such a high-power regime, some detrimental mechanisms, e.g., energy transfer upconversion (ETU), also known as Auger upconversion, may become very important factors that, in principle, change the system parameters required for optimum performance [2–5]. The ETU process has two main consequences: a decrease of the excited-state population and, at the same time, an increase in the thermal loading of the material [6–10]. Both processes, related to ETU, induce phase shift variation in the excitation beam, namely, an electronic and a thermal one. These contributions add up to create a total phase shift variation, $\Delta \varphi$. Nevertheless, when the electronic and thermal response times are some orders of magnitude different, it is possible to temporally discriminate them [11]. In this work we have used this property to access the ETU parameter in two distinct ways. To this end the Z-scan and thermal-lens (TL) techniques have been applied to obtain the ETU parameter separately from the electronic and thermal contributions, respectively. In addition, a comparison between these techniques was done, and the advantages of each one are discussed.

2. THEORETICAL BACKGROUND

In ion-doped solid-state materials, the Auger upconversion process occurs when two neighbor ions, both at the same metastable level, interact nonradiatively so that one of them is transferred to a higher excited state while the other goes to a lower one (e.g., the ground state). For Nd$^{3+}$-doped samples these levels are $^4F_{3/2}$, $^2G_{9/2}$ (and/or $^2G_{7/2}$) and $^4I_{11/2}$ (and/or $^4I_{13/2}$), respectively. The form as the ETU occurs is well described through standard rate equations and, under cw excitation, it can be represented by [2,3,6,8]

$$\frac{dN_e}{dt} = R_p N_g \frac{N_e}{\tau} - \gamma N_e^2,$$

(1)

where $N_e$ ($N_g$) is the excited-(ground-) state ion concentration, $\gamma$ is the ETU parameter, and $\tau$ is the unsaturated level lifetime; $R_p$ is the pump rate given by $R_p = \sigma I / h \nu_{exc}$, with $\sigma$ being the absorption cross section at the pump photon energy ($h \nu_{exc}$), and $I$ is the pump intensity. The steady-state solution of this rate equation can be written as

$$N_e = N_i \left( (1 + S)^2 + 4\beta S - (1 + S) \right) / 2\beta,$$

(2)

where $N_i = N_g + N_e$ is the total ion concentration, $S = I / I_S$ is the saturation parameter with $I$ and $I_S$ being the incident and saturation intensities, respectively. $\beta = \gamma N_i$ is a dimensionless parameter related to the strength of the ETU processes, which is proportional to the ratio between upconversion rate ($\gamma N_e$) and total transition rate in the absence of upconversion ($\tau^{-1}$). The well-known expression for $N_e$, without ETU, can be readily obtained taking $\beta \rightarrow 0$ in Eq. (2). Since the upconversion rate depends on the excited-state population, and therefore on the ion concentration in the material, in the low-intensity-pump regime ($\gamma N_e \ll \tau^{-1}$) the fluorescence decay is basically exponential. On the other hand, at high intensities the total decay leads to a nonexponential fluorescence character, which...
can be fitted to obtain $\gamma$. Indeed, the majority of data in the literature for the ETU parameter were obtained using a fluorescence decay study [6–9]. Nevertheless, this method applies to the initial decay time (typically a few tens of $\mu$s), and it needs high concentration or very high pump intensity for the measure to be accurate. Not uncommonly, the used intensity is close to damage threshold [9]. Recently, the Z-scan and TL techniques have been extended to determine the ETU parameter [3,4,12]. In the present study, these two approaches were applied to Nd$^{3+}$-doped glasses and a crystal to obtain $\gamma$, and a comparison of them is performed. The fluorescence quantum efficiency behavior was also obtained as a function of the incident intensity.

3. EXPERIMENTAL

Nd$^{3+}$-doped glasses such as phosphate (Q-98, Kingre Inc.), fluorozirconate (ZBLAN) and fluorindate (PGIZCa), as well as Nd$^{3+}$-modified yttrium lithium fluoride (YLF) crystal, were all investigated by using the Z-scan and thermal lens (TL) experimental techniques, based on the pump-induced refractive index ($\Delta n$) variation phenomenon. The TL relies on the refractive-index variation caused by the pump-induced heating that modifies the optical path length. The pump-induced temperature profile and, therefore, the refractive-index one, follows the incident Gaussian beam profile. In this way, at a first-order approximation, this induced profile can be considered as a lens, indeed, a TL. Besides the thermal effect, there is an important electronic contribution to $\Delta n$, due to population redistribution. This effect arises from the susceptibility difference between ground and excited states. Therefore, the pump-induced population redistribution changes the overall susceptibility of the ions, promoting a change in the refractive index. Since the pump beam usually has a Gaussian intensity profile, the excited-state population will follow the same radial profile to that of the pump beam. This characteristic makes the induced refractive-index variation, $\Delta n$, to have an approximate lens-like shape. Consequently, this phenomenon is known as the population lens (PL) effect [11], and it will be studied by the Z-scan technique.

A. Thermal-Lens Technique

Thermal lens spectrometry (TLS) has shown to be a useful tool for measuring thermo-optical properties of materials with more diverse characteristics. For instance, it has been used to measure very low optical absorption coefficients, trace analyses, quantum yields, heat generation of solid-state lasers when working [13], chemical reaction kinetics, etc. [14]. The TL effect is caused by heat deposition, via nonradiative decay processes, after the laser energy has been absorbed by the sample. This effect can be treated through the calculation of temporal evolution of the sample temperature profile $\Delta T(r, t)$. Among the several experimental configurations for the TLS, the two-beam mode-mismatched arrangement has shown to be the most sensitive one [15]. The theoretical modeling for the TL in this configuration has been developed and an analytical expression to treat the TL quantitatively has been derived [14]. The TL transient signal amplitude is proportional to its phase shift $\theta$, which is given by

$$\theta = -\frac{P_{\text{abs}}}{K\lambda_p} \frac{\phi}{dT},$$

where $P_{\text{abs}}$ is the absorbed pump power, $\lambda_p$ is the probe beam wavelength, $ds/dT$ is the temperature coefficient of the optical path length, $\phi$ is the fraction of absorbed energy converted into heat, and $K$ is the thermal conductivity. In fluorescent samples, the fraction of absorbed energy converted into heat, $\phi$, which is also known as absolute nonradiative quantum efficiency or fractional thermal loading, is not equal to unit. For systems with only one emitting state, $\phi$ is given by

$$\phi = 1 - \eta_{\text{ex}}(\lambda_{\text{em}}^{-1}),$$

where $\eta$ is the fluorescence quantum efficiency, $\lambda_{\text{ex}}$ is the excitation beam wavelength, and $\lambda_{\text{em}}$ is the inverse of the average emission wavelength. The term $ds/dT$ in Eq. (3) describes the thermally induced distortion of a laser beam during its passage through the sample. In solids, it has contributions due to the temperature coefficient of the refractive index, $dn/dT$, the sample thermal expansion, and the photoelastic effect [14].

B. Determination of ETU by TL

In the very low-pump-power regime (absence of ETU) the fluorescence quantum efficiency is given by $\eta = \pi \tau_{\text{rad}}$, in which $\tau_{\text{rad}}$ is the radiative lifetime. As the pump rate increases and, consequently, so does $N_e$, ETU becomes significant and then the total decay must be written as $W_t = \tau^{-1} + \gamma N_e$, where $\tau$ is the fluorescence lifetime without ETU. In this case $\eta$ is given by [3,4]

$$\eta = \frac{\eta_0}{1 + \beta n_e},$$

in which $\eta_0$ is the intrinsic quantum efficiency (in the absence of ETU), $n_e = N_e/N$, is the fraction of ions in the excited state $|4F_{9/2}\rangle$ [Eq. (2)]. Thus, considering the ETU effect on $\eta$, from Eq. (5) the expression for $\theta$ normalized by the absorbed pump power becomes

$$\Theta = \frac{\theta}{P_{\text{abs}}} = C \left[ 1 - \left( \frac{\eta_0}{1 + \beta n_e} \right) \lambda_{\text{exc}}(\lambda_{\text{em}}^{-1}) \right],$$

where $C = (K\lambda_p)^{-1} ds/dT$ is a constant that depends only on the host characteristics and the probe beam wavelength. Therefore, the fit of the experimental data of $\Theta$ against $S$ with Eq. (6) gives the parameter $\beta$ and the constant $C$, and thus the upconversion parameter ($\gamma = \beta N_e \tau$) [16,17]. Note that $\Theta$ depends upon $S$ through $n_e(S)$. Moreover, the probe wavelength was provided by a 632.8 nm He–Ne laser for all samples.

C. Z-Scan Technique

The Z-scan technique was developed in 1989 by Sheik-Bahae et al. [18] to study degenerate susceptibilities, and it has been largely used since then. The main advantage of this technique is that the signal and magnitude of the nonlinearity is straightforwardly obtained from a simple scan along the beam axis. This makes the experimental
apparatus and data analysis very simple. Many variants have since been developed to improve its sensitivity and/or applicability such as implementation for thick samples [19]. For materials with slow nonlinearities ($\tau \geq 100 \mu s$, e.g., population redistribution), a new approach called time resolved Z-scan procedure was developed [20]. In this case a chopper modulates the beam, and the transient behavior of the transmitted beam carries the nonlinear information. The transmittance is defined as the ratio between the intensity at a time $t_i \ll \tau$, where there are only linear effects, and at a time $t_f > \tau$, where both linear and nonlinear effects are present together. Therefore, it is possible to eliminate linear parasitic effects due to parallelism, bad polish, etc., thereby enhancing its sensitivity.

D. Determination of ETU by Z-Scan
The pump-induced population redistribution changes the absorption coefficient of ion-doped materials. For nonresonant excitation (pumping a level different of $4F_{3/2}$) the Nd$^{3+}$ absorption coefficient can be written as $\alpha = \alpha_L + N_e \Delta \sigma$ with $\alpha_L = \sigma g N_L$ being the linear absorption and $\Delta \sigma = \sigma_e - \sigma_g$, where $\sigma_e(\sigma_g)$ is the excited- (ground-) state absorption cross section. As the intensity falls off as $\exp(-\alpha L)$, with $L$ being the sample thickness, the Beer law can be applied to the beam crossing the sample, and we suppose that $N_g \Delta \sigma L \ll 1$ (as usually is the case). Therefore, it can be shown that the transmittance can be written as $T = T_0 (1 - \Delta T)$, with $T_0 = \exp(-\alpha_0 L)$ as the linear transmittance

$$\Delta T = \Delta \sigma L N_e.$$  \hspace{1cm} (7)

Experimentally all the transmitted light is collected by a lens into the detector, but only $\Delta T$ depends on the incident intensity. Hence, just the open-aperture Z-scan curve is needed in order to obtain $\gamma$. The dependence of $\Delta T$ with $\gamma$ (and $S$) comes from the excited-state population, given by Eq. (2), through the parameter $\beta$. Thus, monitoring the transmittance variation of the open aperture, it is possible to find $\gamma$.

These two techniques were then applied to Nd$^{3+}$-doped samples, excited at the $4F_{5/2}$ level, and the results were compared. The advantages and particularities of each one are also discussed.

4. RESULTS
Figure 1 shows a plot of the TL signal amplitude as a function of the saturation parameter $S$, for the 1 wt% Q-98 Nd-doped sample. As can be seen, a nonlinear enhancement of $\theta$ with $S$ is observed. This behavior can be better noted in Fig. 2, where it is plotted $\theta$ versus $S$. The $C = (4.1 \pm 0.1)$ parameter and $\eta_0 = (0.91 \pm 0.08)$ were obtained for low intensities in TL experiments performed elsewhere [2]. They were used together with Eq. (6) in Fig. 2 to fit the experimental data. For the other studied samples the curves are quite similar. The used parameters and found results can be seen in Tables 1 and 2, respectively.

Furthermore, several Z-scan experiments have been also performed at different intensities, in order to obtain the $\gamma$ parameter. Figure 3 depicts the open-aperture pump-induced transmittance variation, $\Delta T = T_p - 1$, as a function of the saturation parameter $S$, where $T_p$ is the transmittance at the peak position. As for TL experiments, results for the other Nd$^{3+}$-doped samples are quite similar and will not be shown. In the $\gamma$ determination by Z-scan, knowledge of $\Delta \sigma$ is required. This data was obtained through a simple open-aperture Z-scan experiment at low-intensity regime to avoid saturation effects. The achieved values for the 1 wt% Q-98 was $\Delta \sigma = 3.07 \times 10^{-20}$ $\text{cm}^2$, and it was used with Eq. (7) to fit the experimental data points. The values obtained for all samples are listed in Table 1.

From the data in Fig. 2, the dependence of the fluorescence quantum efficiency and thermal loading with $S$ parameter have been also determined [Eqs. (3) and (4)]. The results are presented in Fig. 4. As already expected, the

![Fig. 1. TL signal amplitude ($\theta$) as a function of the saturation parameter ($S$). The nonlinear behavior indicates extra heating. The dashed curve is only a guide showing the nonlinearity.](image)

![Fig. 2. TL normalized signal $\theta$ versus $S$ at $\lambda_{ex}=801.6$ nm. For the $1.1 \times 10^{20}$ ions/cm$^3$, the fit gave us $\beta=1.54$.](image)

<table>
<thead>
<tr>
<th>Sample</th>
<th>$\Delta \sigma \times 10^{-21}$ $\text{cm}^2$</th>
<th>$\eta_0$</th>
<th>$C$ (W$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Q-98 (Kigre)</td>
<td>$-30.7$</td>
<td>0.91</td>
<td>4.1</td>
</tr>
<tr>
<td>ZBLAN</td>
<td>1.6</td>
<td>1.02</td>
<td>12.8</td>
</tr>
<tr>
<td>PGIZCa</td>
<td>0.9</td>
<td>0.97</td>
<td>3.63</td>
</tr>
<tr>
<td>YLF</td>
<td>$-4.8$</td>
<td>$1^*$</td>
<td>2.4</td>
</tr>
</tbody>
</table>

$^*$estimated
These results can be easier understood as follows: from Eq. (2) one can see that, at the low-saturation regime, \( n_e = S[1 - (1 + \gamma S)] \) and \( \eta = \eta_0[1 - \beta S + (1 + 2\beta)\beta S^2] \). Hence, \( \eta \) always decreases in the presence of ETU, but initially (for \( \beta S < 1 \)) it goes linearly, and for higher intensities the behavior becomes parabolic. Indeed, in the presence of ETU the total decay rate is now given by \( W = 1/\tau + \eta N_e \), so that at high pump power it is an efficient nonradiative channel to depopulate the upper laser level, increasing \( \delta \) and decreasing \( \eta \).

Equation (3) indicates a linear dependence of \( \theta \) with \( P \) (excitation power), which should be true if \( \delta \) is a constant. However, as can be seen above, \( \delta \) also depends on \( P \), and the behavior of \( \theta \) becomes nonlinear. This deviation of the expected linear behavior indicates an increase in the heat generation due to the presence of ETU. This influence could be better taken into account in Fig. 2, from where the ETU parameter has been found.

For the Z-scan experiments, the behavior of \( \Delta T \) in Fig. 3 follows that of the excited-state population. Therefore, one can see a decrease in \( N_e \) when compared with the \( \beta = 0 \) case (dashed curve). So, the main effect of ETU on the electronic nonlinearity is to lessen the population lens with the increase of the pump power. As the transmittance variation is proportional to the excited-state population, the determination of \( \beta \), and thus \( \gamma \), is straightforward in this experiment.

Table 2 shows an excellent agreement between the \( \gamma \) values obtained with the two techniques for the used concentrations. These results show the power of these techniques for characterization of losses in laser materials. It should be noticed that the approaches used here are independent. Moreover the obtained values for \( \gamma \), shown in Table 2, are close to those found by Payne et al. [9] for other phosphate glasses, in the case of Q-98 glasses. It is worth noting that, in order to obtain reasonable accuracy (\( \sim 30\% \)), the measurements of [9] were performed at high excitation levels near the damage threshold of the samples. In our experiments, \( \gamma \) was determined with higher accuracy (error \( \sim 10\% \)) with much lower excitation levels, avoiding damage to the investigated materials. This also shows the high sensitivity of these methods.

It is also important to note that the experiments were performed under very different experimental conditions, which were chosen so that there were no dual contributions to the acquired signal. Besides the open Z-scan aperture, to see only electronic nonlinearity (thermal lensing is not detected) the chopper frequency has been used.

### Table 2. Upconversion Parameters Found for Studied Samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>( N_e^a ) (( 10^{20} ) cm(^{-3} ))</th>
<th>( \phi ) (( \mu s ))</th>
<th>( \beta )</th>
<th>( \gamma ) (10(^{-17}) cm(^3)/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Q-98 (Kigre)</td>
<td>1.1</td>
<td>346</td>
<td>1.54</td>
<td>1.60</td>
</tr>
<tr>
<td>ZBLAN</td>
<td>5.2</td>
<td>260</td>
<td>15.93</td>
<td>15.0</td>
</tr>
<tr>
<td>PGIZCa</td>
<td>6.5</td>
<td>164</td>
<td>20.70</td>
<td>21.3</td>
</tr>
<tr>
<td>YLF</td>
<td>1.7</td>
<td>520</td>
<td>16.80</td>
<td>15.7</td>
</tr>
</tbody>
</table>

\( ^a \)\( N_e \) is the total ion density.

\( ^b \)\( \phi \) is the fluorescence lifetime.

\( ^c \)\( \beta \) is proportional to the ratio between upconversion and total transition rates.

\( ^d \)\( \gamma \) is the ETU parameter.

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### 5. DISCUSSION

The increase of \( \Theta \) with \( S \), seen in Fig. 2, can be explained as an enhancement in the factor \( \phi \), or equivalently a decrease in the fluorescence quantum efficiency, \( \eta \), as shown in Fig. 4. These results can be easier understood as follows: from Eq. (2) one can see that, at the low-saturation regime, \( (S \ll 1) \), \( n_e = S[1 - (1 + \beta S)] \) and \( \eta = \eta_0[1 - \beta S + (1 + 2\beta)\beta S^2] \). Hence, \( \eta \) always decreases in the presence of ETU, but initially (for \( \beta S < 1 \)) it goes linearly, and for higher intensities the behavior becomes parabolic. Indeed, in the presence of ETU the total decay rate is now given by \( W = 1/\tau + \eta N_e \), so that at high pump power it is an efficient nonradiative channel to depopulate the upper laser level, increasing \( \delta \) and decreasing \( \eta \).

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It is also important to note that the experiments were performed under very different experimental conditions, which were chosen so that there were no dual contributions to the acquired signal. Besides the open Z-scan aperture, to see only electronic nonlinearity (thermal lensing is not detected) the chopper frequency has been used.

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Fig. 3. Z-scan open-aperture transmittance variation \( \Delta T \) versus \( S \). For the \( 1.1 \times 10^{20} \) ions/cm\(^3\), we found \( \beta = 1.60 \). The dashed curve represents the case \( \beta = 0 \) for comparison.

The presence of ETU leads to a decrease of \( \eta \) and a correspondent increase of \( \phi \).

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Fig. 4. Normalized fluorescence quantum efficiency \( \eta/\eta_0 \) and thermal loading \( \phi \) as a function of the saturation parameter \( S \).
to separate the thermal and electronic contribution to $\Delta n$, as already done for other materials [11]. In the Z-scan experiment, the chopper was put at 800 Hz so that $\tau < t_f < t_c$ (where $t_f$ is the TL formation time), in order to avoid thermal contributions. The TL experiment was carried out at low frequency, 3 Hz, so that at this time scale $t_f > \tau$, and the electronic contribution has already reached its stationary value and was eliminated through the normalizing procedure. Furthermore, the main advantage of the mode-mismatched configuration is the notable enhancement in the sensitivity when the probe beam waist $(\omega_p)$ is much larger than the excitation one $(\omega_e)$. This sensitivity enhancement is a consequence of heat diffusion that makes the refractive-index profile $\Delta n(r)$ wider than the excitation intensity profile. On the other hand, in the PL effect there is no diffusion mechanism, so the PL signal decreases when the probe beam waist increases [21]. From the definition of the $m = (\omega_p/\omega_e)^2$ TL parameter [14], it was shown that, for $m \gg 1$, the PL signal decreases as $m^{-1}$ [11]. We have used the probe beam area at the sample about 10 times greater than the excitation beam, so that the electronic contribution to the acquired signal can be neglected. As a matter of fact, the possibility of eliminating the PL effect is an additional advantage of the mode-mismatched configuration for the study of thermal nonlinearity and determination of the thermo-optical parameters such as $D$, $ds/dT$, and Auger upconversion parameter. Also, the existence of only one effect or the dominance of one of them may be tested by measuring the distances separating the peak and valley, $\Delta z_{p-v}$, which is $\sim 1.7Z$ and $\sim 3.4Z$ for PL and TL effects alone, respectively. The Z-scan technique is the simplest one, and it requires neither previous knowledge of the photothermal parameters nor about the fluorescence quantum efficiency. The only required parameter is $\Delta \tau$, which can be found in a simple low-intensity measurement. On the other hand, the parameters necessary for TL can be obtained, also, in a low-intensity TL measurement. Thus, in addition to $\gamma$, the fluorescence quantum efficiency $\eta$ (that is a hard parameter to be obtained) can be obtained in a single experiment.

In the case of YLF crystal, the measurements were performed both with the pump beam parallel and perpendicular to the optical axis. In this TL experiment, the probe was unpolarized, so the shown results are an average value for the two crystal directions. We have used this procedure, since there is no specific treatment for anisotropic samples using TL and Z-Scan techniques.

Finally, the data of Fig. 2 have been also used to understand the effect of upconversion processes on the fluorescence quantum efficiency and on the thermal loading in the Q-98 glasses. These results are shown in Fig. 4, where both normalized fluorescence quantum efficiency $(\eta/\eta_0)$ and the fractional thermal loading $(\phi)$ as a function of the excitation parameter $S$ are presented. From this figure, it is clear that the influence of ETU in the heat generation processes takes place in strongly pumped laser materials, and it cannot be neglected. As a matter of fact, a reduction of $\sim 10\%$ in the fluorescence quantum efficiency due to ETU has been found for a saturation parameter $S = 0.1$. For this saturation parameter we have predicted an enhancement of the thermal load of $\sim 20\%$ (for a sample with 1 wt% of Nd). Therefore, when a Q-98 phosphate glass laser is strongly pumped, any estimation of the thermal loading should include the effect of ETU processes. Obviously, these discussions should be extended for all other studied samples.

6. CONCLUSION

In conclusion, the ETU parameter has been determined using two different techniques: Z-scan and TL. A comparison between the found results shows that they present almost the same values. Moreover, these values are close to those found in the literature for other commercial Nd$^{3+}$-doped materials. The advantages and applicability of each method were discussed, and it was shown that they provide independent and complementary results, so they could be used separately depending on the experimental conditions. It is also possible to conclude that both the fluorescence quantum efficiency and the fractional thermal loading are, as a consequence of ETU processes, strongly dependent on the pumping intensity. Consequently, ETU constitutes a non-negligible heating channel, which should be included in future modeling of pump-induced thermal loading in Nd$^{3+}$-doped solid-state lasers.

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12. D. N. Messias, A. A. Andrade, and T. Catunda are preparing a paper to be called “Determination of Auger upconversion parameter by Z-scan measurements.”


