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REFRACTION MODULATION *Z*-SCAN FOR MEASUREMENT OF NONLINEAR REFRACTIVE INDEX

We present a novel single-beam refraction-modulation Z-scan technique that combines elements of the standard Z-scan and loss-modulation methods to enable sensitive measurement of third-order refractive nonlinearities in the closed-aperture configuration. Compared with the original Z-scan technique, the proposed method provides an almost background-free signal. An analytical expression relating the nonlinear refractive index n_2 to the powers of the incident beam and the transmitted beam components at the first and second modulation harmonics is derived and experimentally verified using SiO_2 and LiF samples.

Keywords: *Z*-scan technique, nonlinear refractive index, two-photon absorption coefficient.

1. Introduction

Femtosecond lasers are widely used nowadays to fabricate micro-optical elements both in the bulk and on the surface of optoelectronic materials through laser-induced micromodification of their optical properties. However, the propagation of intense femtosecond laser pulses is strongly influenced by material's nonlinearity. The absorption of laser light – and thus the precision of point modifications and energy deposition inside the bulk of material – depends on two-photon absorption (2PA) coefficient β, while the beam spatial transformation in transparent materials is governed by nonlinear refractivity (or Kerr) index n_2. Thus, to understand the physics of laser propagation and interaction with optical materials – as well as to develop advanced laser-based microfabrication techniques, we need highly sensitive and accurate methods for measuring nonlinear optical parameters.

The well-known *Z*-scan technique, first proposed in [1], involves moving a thin sample through the focus of a laser beam and measuring the transmitted light to determine the sample's nonlinear optical properties. Both refractive nonlinearities, governed by the real part of the nonlinear susceptibility tensor, and absorptive nonlinearities, governed by the imaginary part of the nonlinear susceptibility tensor, can be tested in this way (Fig. 1). If we measure two-photon absorption coefficient β, open aperture configuration collecting all the transmitted power is used (Fig. 1, *a*). In this case *Z*-scan trace features a dip at the focal position of the sample due to the 2PA. Using the *Z*-scan model in [1], β can be calculated from the *Z*-scan trace. In case of measuring nonlinear refractive index n_2, closed aperture configuration is used (Fig. 1, *b*), and only the light, which passes a small diaphragm is recorded. A *Z*-scan trace has an oscillating shape in this case due to the Kerr lens induced

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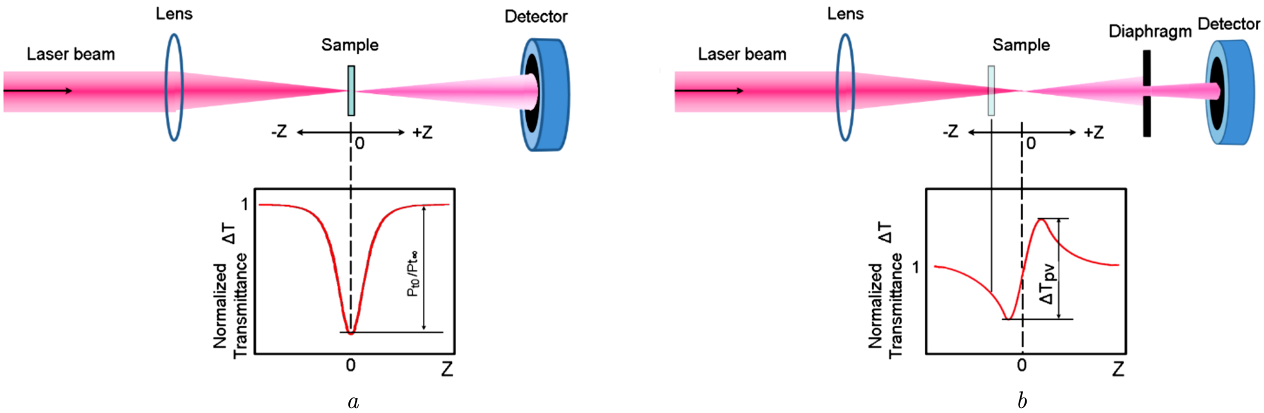

Fig. 1. *Z*-scan open-aperture configuration for measuring the two-photon absorption coefficient β (*a*). *Z*-scan closed-aperture configuration for measuring the nonlinear refractive index n_2 (*b*)

in the illuminated area of the sample, and n_2 can be calculated from this trace.

A number of derivative techniques have been developed to advance the standard *Z*-scan method. Among them, several variations of the eclipsing *Z*-scan technique enhance sensitivity by blocking a large portion of the transmitted beam power near the optical axis [2–4]. The *Z*-scan technique for thick samples was discussed in [5–8]. The two-color *Z*-scan uses two lasers with different wavelengths [9, 10], while the white-light continuum *Z*-scan investigates nonlinear properties over a wide spectral range [11, 12]. The time-resolved pump-probe *Z*-scan combines time-resolved spectroscopy with *Z*-scan to study the dynamics of nonlinear optical processes [13, 14]. The top-hat beam *Z*-scan uses a flat on-axis intensity distribution [15, 16], and the polarization *Z*-scan examines the influence of different polarization states of the probe light on nonlinear optical properties [17, 18]. Sensitivity was increased with the differential *Z*-scan technique, which provides a background-free signal dithering the sample's longitudinal position [19]. The F-scan technique uses a tunable focus instead of a fixed-focus lens [20, 21]. A modification named reflection *Z*-scan studies the nonlinear optical properties of material surfaces using a reflectivity signal [22, 23].

The above techniques have been useful in characterizing the nonlinear optical properties of a wide range of advanced functional materials, including semiconductors, nanomaterials, organic compounds, and metamaterials, which find applications in science and technology, e.g. photonics, lasers, quantum optics, material science, optical switching, 3D memory, telecommunications, bio-imaging, *etc.*

The loss modulation technique for the measurement of two-photon absorption (2PA) was first introduced in [24] and later refined by our group in [27]. In this method, the intensity of the laser beam is modulated with a pure sinusoidal waveform at frequency F. The modulated beam is then normally focused into the bulk of the sample, and the transmitted light is detected using a lock-in amplifier at the second harmonic ($2F$) of the modulation frequency. The open-aperture configuration is used, where the entire transmitted beam is collected by the detector. The name of the technique reflects the fact that the modulated beam induces a corresponding modulation in the sample's optical losses due to the nonlinear absorption effect. The underlying principle of the loss modulation technique is that 2PA preferentially attenuates the peaks of the sinusoidal intensity modulation, thereby distorting the waveform. This distortion generates second harmonic (SH) components at $2F$ in the frequency spectrum of the transmitted signal. Due to the sensitive lock-in detection of the small $2F$ component in the wide spectrum of the transmitted power, the loss modulation technique provides significantly higher sensitivity ($\beta \sim 10^{-6}$ cm/GW) compared to the conventional *Z*-scan method.

In the original implementation [24] of the method, a rather sophisticated experimental setup was used to achieve sinusoidal modulation. It involved combining two beams of slightly different frequencies derived from the same femtosecond laser source. These beams are frequency-shifted using two acousto-optic modulators and then precisely recombined in space and time with the help of a delay line. Moreover, the original loss modulation method does not involve longitudinal Z-scanning of the sample under test. Consequently, its sensitivity is limited by a background spurious signal at the doubled modulation frequency ($2F$), which is unrelated to 2PA and remains independent of the Z-position.

In the improved loss modulation technique [27], we not only significantly simplified the optical setup but also integrated the Z-scan and loss modulation methods into a single configuration. The second harmonic (SH) signal at $2F$ was recorded while scanning the sample along the Z-axis. This approach enabled the separation of the useful $2F$ signal from a z-independent high spurious $2F$ background by comparing the total $2F$ signal in the focused and defocused positions.

Here we present a novel single-beam technique that combines the principles of the standard Z-scan and loss modulation methods for the sensitive measurement of third-order refractive nonlinearities. Unlike in [24, 27], a closed-aperture configuration was used. We refer to this method as refraction modulation Z-scan, highlighting the fact that the measurement procedure involves nonlinear modulation of the sample's refractive index by the intensity-modulated laser beam. Both the experimental setup and the mathematical model for the processing of measurement results are presented.

2. Theory

To derive an equation relating the SH component in the transmitted modulated laser beam probe to the nonlinear refractive index n_2, we start with the well-established theoretical framework of the traditional Z-scan technique [1, 25], which involves probing the sample using a laser beam. In contrast to the conventional approach [1, 25], where the laser beam average power P_0 is assumed to be constant, we introduce a slow sinusoidal modulation of the beam power at the frequency F (with $F < f_0$, where f_0 is the repetition rate of the laser pulses). Specifically, we consider the incident power to vary in time as

$$P(t) = P_0(1 + \cos(2\pi Ft)). \quad (1)$$

Applying straightforward trigonometric analysis, we derive an expression that links the second harmonic (SH) component (at frequency $2F$) of the detected signal to the nonlinear refractive index n_2 of the sample material.

However, to validate this approach, we first apply it to the Z-scan analysis for determining not the nonlinear refractive index, but the two-photon absorption (2PA) coefficient [1, 25] and compare the resulting expression with the well-established formula for the loss modulation technique [24]. It is shown in [1, 25] that in open aperture mode, assuming thin samples (sample thickness $L \ll Z_0$, where Z_0 is Rayleigh length in air), small linear losses ($\alpha L \ll 1$, where α is a linear absorption coefficient, small 2PA losses ($\beta I_0 L \ll 1$, where β is a two-photon absorption coefficient, I_0 is an on-axis intensity of the laser beam within the sample) and Gaussian time shape of the laser pulse, β can be found by fitting the experimental shape $\Delta T(z)$ in the change of the transmitted beam power $P_t(z)$ against z, normalized to the linearly transmitted power $P_{t\infty}$ at the defocused position ($|z| \gg Z_0$, where Z_0 is Rayleigh length):

$$\Delta T(z) = -\frac{\beta I_0 L}{2\sqrt{2}} \frac{1}{[1 + z^2/Z_0^2]}. \quad (2)$$

Here $\Delta T(z) = (P_t(z) - P_{t\infty})/P_{t\infty}$.

However, if we limit ourselves to measuring $P_t(z)$ only at two positions – at focus (P_{t0} at $z = 0$), where the transmitted power is minimal, and far from focus ($P_{t\infty}$), where the nonlinear absorption does not affect the transmitted power – then Eq. (2) takes the form

$$P_{t0}/P_{t\infty} = 1 - \beta I_0 L/2\sqrt{2}. \quad (3)$$

For a Gaussian beam with the Rayleigh length in air Z_0 formed by the train of the pulses with Gaussian temporal shape of duration τ, and repetition rate f_0, $I_0 = 2P/(Z_0\lambda\tau f_0)$, where P is the average beam power within the sample. Thus, formula (3) takes the form

$$P_{t0}/P_{t\infty} = 1 - \beta PL/\sqrt{2}(Z_0\lambda\tau f_0). \quad (4)$$

Assume now that the average power of the incident pulse train experiences a slow purely sinusoidal modulation (1). Hence, Equation (4) becomes

$$P_{t0}(t)/(P_{t\infty}(1 + \cos(2\pi Ft)) =$$
$$= 1 - \beta P_0(1 + \cos(2\pi Ft))L/\sqrt{2}(Z_0\lambda\tau f_0). \quad (5)$$

Here the normalized transmitted power, measured at the sample position far from focus, remains modulated by a pure sinusoid. We neglect second-order smallness effects in the term $\beta P_0(1+\cos(2\pi Ft))L/\sqrt{2}(Z_0\lambda\tau f_0)$, assuming that the beam power inside the sample is unaffected by 2PA and can be approximated by a pure sinusoid, given the previously stated condition $\beta I_0 L \ll 1$.

Fresnel reflections do not influence the ratio on the left-hand side of the equation. However, they should be accounted for when determining the power inside the sample $P_0(1+\cos(2\pi Ft))$.

For simplicity, let us designate $\beta P_0 L/\sqrt{2}(Z_0\lambda\tau f_0)$ as K. Then

$$P_{t0}(t)/P_{t\infty} = 1+\cos(2\pi Ft)-K(1+\cos(2\pi Ft))^2, \quad (6)$$

$$P_{t0}(t)/P_{t\infty} = (1-1{,}5K)+$$

$$+(1-2K)\cos(2\pi Ft)-0{,}5K\cos(2\pi 2Ft). \quad (7)$$

It follows from Eq. (7) that the ratio $P_{t0}(t)/P_{t\infty}$ apart from a DC term and a sinusoidal oscillation at the modulation frequency F, acquires a zero-offset oscillating term at the doubled frequency $2F$ that is proportional to the 2PA coefficient β.

Equation (7) can be rearranged for the case of measuring the powers of oscillating components of the transmitted beam with a photodiode and a lock-in amplifier. Considering 100% modulation, we replace the ratio of the transmitted powers $P_{t0}(t)/P_{t\infty}$ with the ratio of the corresponding lock-in signals V. In this case, absolute power calibration of the lock-in measurements is not required. If $|z| \gg Z_0$, the power signal $P_{t\infty}$ can be replaced with the lock-in signal V_{1F} at the reference frequency $1F$. The signals at doubled frequency can be measured separately at doubled reference frequency $2F$. Thus, when measuring the transmitted power signals V_{2F} at the second harmonic of the modulation frequency ($z = 0$) the DC and $1F$ terms are irrelevant, and equation (7) reduces to the following form:

$$V_{2F}/V_{1F} = 0.35\beta P_0 L/(Z_0\lambda\tau f_0). \quad (8)$$

Here the power P_0 inside the sample should be measured separately before the sample in absolute units with a power meter and corrected for Fresnel reflection. To determine β from Eq. (8) we must – aside from knowing P_0 – also measure the ratio between the signal $V_{2F}(0)$ at the focal position ($z = 0$) and the signal V_{1F} at $|z| \gg Z_0$, both obtained using the lock-in amplifier.

To validate the above analysis, we compare Eq. (8), that we derived based on the results of the standard Z-scan technique for thin samples [1, 25], with a corresponding equation for the loss modulation technique presented in [24] for the samples of arbitrary thickness. This comparison reveals that, in the limit of thin samples, apart from the difference in designations, the only discrepancy between the two equations lies in the numerical coefficient, which is 0.35 in our case and 0.33 in [24].

Thus, having validated our approach, we apply a similar method to the equation relating the nonlinear refractive index n_2 to the difference between peak and valley transmittance, ΔT_{pv}, (see Fig. 1, *b*) for closed-aperture Z-scan measurements [1, 25]:

$$\Delta T_{pv} \cong 0.406(1-S)^{0.27}\frac{2\pi}{\lambda}|n_2|\, I_0 L, \quad (9)$$

where S represents the transmittance of the aperture in the absence of the sample. It is assumed here that both linear and two-photon absorption are negligibly small, and $\Delta T_{pv} \ll 1$.

Considering the slow modulation of the laser beam power under similar assumptions as for open-aperture Z-scan, we derive the following equation from Eq. (9):

$$\frac{\Delta V_{2Fpv}}{V_{1F}} \cong 2.55(1-S)^{0.27} n_2 \frac{P_0}{Z_0\lambda^2\tau f_0} L. \quad (10)$$

Equation (10) relates n_2 to the beam power P_0 inside the sample, and to the ratio of the lock-in signal V_{1F} at $|z| \gg Z_0$ to the peak-value difference of the lock-in signal $\Delta V_{2F_{pv}}$.

3. Experiment and Results

To further validate this concept, we verified Eq. (10) experimentally, having conducted experiment on refraction modulation Z-scan using 1-mm-thick samples of fused silica and LiF crystals.

The experimental setup is shown in Fig. 2.

A femtosecond regenerative amplifier (Legend, Coherent) used in the experiment operated at a central wavelength of 800 nm, photon energy of 1.55 eV with a pulse repetition rate $f_0 = 1$ kHz, pulse duration $\tau = 150$ fs, and an average output power of 1 W. The power of the beam transmitted through the first polarizing cube (PC1) was manually controlled by adjusting its polarization plane using a half-wave plate (HWP1) and varied between 90 μW

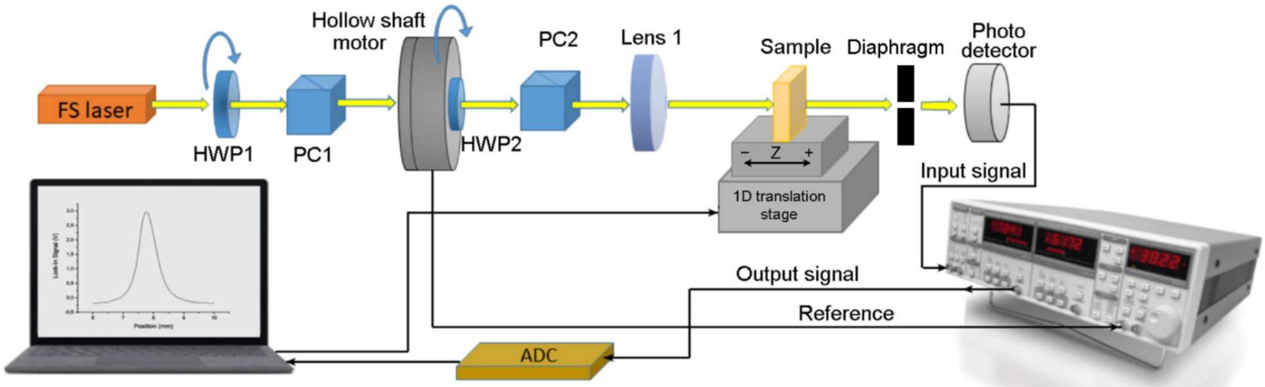

Fig. 2. Experimental setup

and 400 μW in the experiment. The beam's polarization was further rotated by a second half-wave plate (HWP2), mounted on a hollow-shaft motor rotating at 75 s^{-1}. As a result, after passing through the second polarizing cube (PC2), the output beam acquired intensity-modulation at a frequency $F = 300$ Hz with nearly 100% modulation depth. The beam of 2 mm diameter was focused onto the sample using lens $L1$ with a focal length of 61 mm. The Rayleigh length Z_0 at the beam waist under these conditions is 1.15 mm. The sample was translated along the Z-axis using a motorized linear stage. In contrast to our earlier work [27], where an open-aperture configuration was used, here we employed a closed-aperture configuration. In this arrangement, only a small axial portion of the beam transmitted through the sample passes through an approximately 0.5 mm diameter pinhole aperture diaphragm before being detected by a wide-area silicon photodiode, and the resulting signal is then fed to the input of a lock-in amplifier. Experimentally, the diaphragm transmittivity S was found to be 0.05 when the beam diameter was 2 mm. The reference signal for the lock-in detection of the signals at 300 and 600 Hz was provided by an IR LED–photodiode pair in combination with a four-blade chopper mounted on the hollow-shaft motor. The output of the lock-in amplifier was digitized using a 16-bit ADC for recording as a function of the Z-position.

As stated in [25], the Rayleigh length of the incident beam can be directly found from the peak-valley distance ΔZ_{pv} of the standard Z-scan trace using the following expression:

$$|\Delta Z_{pv}| \approx 1.7 Z_0. \tag{11}$$

Applying (11) to the refraction modulation Z-scan traces in Fig. 3, *a, d*, we find that $Z_0 = 1.1$ mm in our experiment. At first glance, this appears to contradict the requirement that the sample thickness L must be much smaller than Z_0. However, as shown experimentally in [1], this requirement can be relaxed to $L < Z_0$. We believe, that this requirement relaxation is at least partially justified by the fact that the Rayleigh length increases by a factor of n inside a refractive medium. Thus, the higher the refractive index n, the thicker the sample can be. This consideration is particularly relevant for IR semiconductors such as silicon, germanium, GaAs, and others with refractive indices close to 3. However, it should be noted that the simplified closed-aperture approach – where the nonlinear refractive index is extracted from the Z-scan peak-valley difference – is valid only if the two-photon absorption can be neglected. Therefore, the laser photon energy is restricted to the values less than half of the semiconductor bang gap.

We conducted refraction modulation Z-scans varying the excitation power in a SiO_2 ($E_g = 8.9$ eV) and LiF ($E_g = 14$ eV) samples (Fig. 3). Fig. 3, *a* demonstrates a typical trace of refraction modulation Z-scan for a 1-mm-thick fused silica sample under an incident beam with an average power of $P_0 = 190$ μW and $V_{1F} = 2.9$ mV. The peak-valley difference signal $\Delta V_{2F_{pv}}$ is 185 μV for this measurement, resulting in the $\Delta V_{2F_{pv}}/V_{1F}$ ratio of 0.064. A notable advantage of the technique, contributing to its sensitivity, is that in contrast to the ordinary Z-scan, the z-independent background offset of the useful V_{2F} signal at sample positions z far from the focus is close to zero. Unlike the traditional Z-scan, the sign of the peak–valley os-

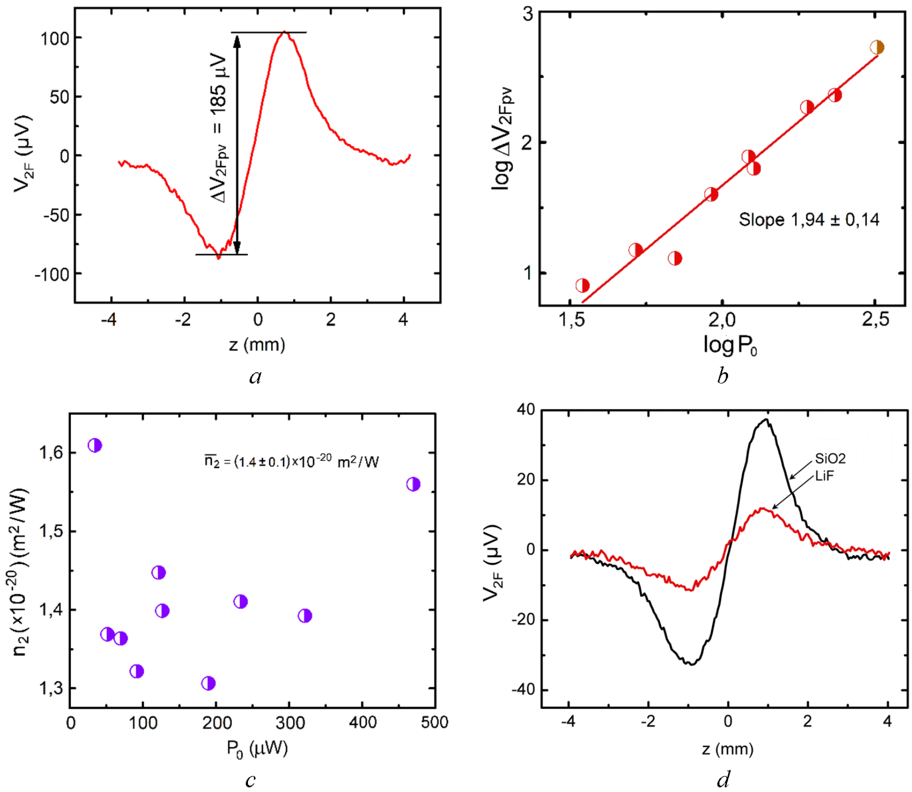

Fig. 3. Refraction modulation Z-scan trace obtained using a 1-mm-thick fused silica sample (a). $\log\Delta V_{2F_{pv}}$ versus $\log V_{1F}$ for an 1-mm-thick fused silica sample. Both signals are given in microvolts (b). Experimental n_2 values for SiO_2 (c). Refraction modulation Z-scans for 1-mm-thick samples of SiO_2 and LiF under identical excitation (d)

cillation here is determined not only by the sign of the nonlinear refraction coefficient of the medium under study, but also by the measurement phase set in the lock-in amplifier. A 180° change in the phase setting reverses the sign of the peak–valley oscillation trace without changing the value of $\Delta V_{2F_{pv}}$.

The logarithmic plot of the measured values of $\log\Delta V_{2F_{pv}}$ versus $\log P_0$ where $\Delta V_{2F_{pv}}$ is expressed in μV and P_0 – in μW, is shown in Fig. 3, b. The fitted slope of 1.94 ± 0.14 indicates a nearly quadratic dependence, consistent with the third-order nonlinearity associated with the n_2 nonlinear refraction index.

Fig. 3, c shows the experimental n_2 values for SiO_2, calculated using Eq. (10), for P_0 ranging from 35 mW to 322 mW. The average n_2 value over the data points is $(1.4 \pm 0.1) \times 10^{-20} \mathrm{m}^2/\mathrm{W}$.

According to the refractiveindex.info database, the authors of [28] report $n_2 = 2.07 \times 10^{-20}$ m^2/W for SiO_2 at a wavelength of 772 nm. A value of $n_2 = 2.7 \times 10^{-20}$ m^2/W at 800 nm is given in [29]. At wavelengths near 1.06 μm, the reported reference values for SiO_2 range from 2.14×10^{-20} m^2/W to 2.74×10^{-20} m^2/W [28, 30–34]. A possible reason for the difference between the experimental n_2 values and the literature data can be in the imperfection of Gaussian profile of the incident beam. This discrepancy can be eliminated by replacing the coefficient 2.55 in Eq. (11) with 4.55.

The two refraction modulation Z-scans for 1-mm-thick fused silica and LiF samples, presented in Fig. 3, d were conducted under similar excitation parameters ($P_0 = 470$ μW, 800 nm wavelength, 150 fs pulse width, 300 Hz modulation frequency, 1 kHz pulse repetition rate, and a 61 mm focal length focusing lens). The only distinction from the measurements shown in Fig. 3, a is that a darker neutral density filter was placed in front of the photodetector to prevent saturation of the detected beam intensity. However, the optical density of the filter does not affect the calculated value of n_2 as the ratio $\Delta V_{2F_{pv}}/V_{1F}$ remains unchanged. According to these measurements, $n_2 = 1.56 \times 10^{-20}$ m^2/W for SiO_2 and $n_2 = 5.2 \times 10^{-21}$ m^2/W for LiF. For LiF, the refractiveindex.info database lists a single reference value of $n_2 = 8.8 \times 10^{-21}$ m^2/W at 800 nm [29]. Other available LiF measurements were obtained at 1.064 μm:

$n_2 = 7.9 \times 10^{-21}$ m^2/W [33], $n_2 = 7.55 \times 10^{-21}$ m^2/W [34], and $n_2 = 1.1 \times 10^{-20}$ m^2/W [32]. The ratio of the measured values $n_{2\mathrm{SiO2}}/\ n_{2\mathrm{LiF}} = 3$ is consistent with that for the reference values [29, 31]. Applying Eq. (11) with the corrected coefficient 4.55 instead of 2.55 we obtain corrected values of $n_2 = 2.78 \times \times 10^{-20}$ m^2/W for SiO_2 and $n_2 = 9.2 \times 10^{-21}$ m^2/W for LiF, which are in closer accordance with the literature data.

In summary, the refraction-modulation technique in the closed-aperture configuration for measuring nonlinear refractive index n_2 in closed-aperture configuration is presented and experimentally demonstrated for the first time. The method features a near-zero Z-independent background offset in the measured signal, making it significantly less susceptible to laser-power fluctuations and sample-surface imperfections. The introduction of a correction coefficient into the derived theoretical expression, which relates n_2 to the experimental parameters, results in experimental n_2 values that agree closely with reference data.

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Z-СКАНУВАННЯ З МОДУЛЯЦІЄЮ РЕФРАКЦІЇ ДЛЯ ВИМІРЮВАННЯ НЕЛІНІЙНОГО ПОКАЗНИКА ЗАЛОМЛЕННЯ

Представлено новий метод однопроменевого Z-сканування з модуляцією показника заломлення, який поєднує елементи стандартних методів Z-сканування і методу модуляції втрат для забезпечення чутливого вимірювання рефракційних нелінійностей третього порядку в конфігурації із закритою діафрагмою. Порівняно з оригінальним методом Z-сканування, запропонований метод забезпечує майже безфоновий сигнал. Отримано аналітичний вираз, який пов'язує нелінійний показник заломлення n_2 з потужностями падаючого променя й компонентів пропущеного променя на першій і другій гармоніках модуляції; вираз експериментально перевірено з використанням зразків SiO_2 та LiF.

Ключові слова: метод Z-сканування, нелінійний показник заломлення, коефіцієнт двофотонного поглинання.