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Wavelength-Dependent Nonlinear Absorption in Platinum Nanoparticles at Off-Resonant Wavelength

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Abstract: In order to obtain optical nonlinear materials with high transparency and low propagation loss in the visible and infrared range, noble metal materials in the off-resonant band have become a hot spot in the optical field in recent years. Therefore, the nonlinear absorption characteristics of platinum nanoparticles (PtNPs) with the surface plasmon resonance (SPR) in the ultraviolet band were investigated with multi-wavelength (500–700 nm) nanosecond Z-scan technology. The measurement results showed that the SPR wavelength of PtNPs was far away from the excitation wavelength, but there were still the saturated absorption (SA) and the reverse saturated absorption (RSA) phenomena, and the size of nonlinear absorption was related to the excitation wavelength and the excitation energy. When the excitation wavelength was constant, with the increase in excitation energy, PtNPs converted from SA to RSA. When the excitation energy was constant, with the excitation wavelength approaching SPR, PtNPs also converted from SA to RSA. The SA and RSA phenomena in the off-resonant region were complementary to the systematic study of the nonlinearity of PtNPs.

Keywords: nonlinear absorption; platinum nanoparticles; wavelength-dependent; Z-scan



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1. Introduction

Under the action of external electromagnetic fields, surface plasmon resonance (SPR) is a collective oscillation phenomenon of free electrons on the surface of metal nanoparticles. The local electric field enhancement of SPR affects the optical Kerr polarizability $\chi^{(3)}$, which is the main index of nonlinear optics [1–3]. In the visible region, some metal nanoparticles have optical nonlinearity owing to SPR, among which gold nanoparticles, silver nanoparticles, and copper nanoparticles are typical representatives [1–11]. However, it is worth noting that although the SPR of some metal nanoparticles is in the ultraviolet region. They still show good optical nonlinearity in the visible region, such as platinum nanoparticles and palladium nanoparticles.

In the past, the nonlinearity of platinum nanomaterials has also been studied and applied in limiters [12,13], mode-locking [14], and saturable absorbers [15,16]. In 2002, S. Qu et al. [12] studied the nonlinearity of PtNPs with a pulse width of 8 ns and a wavelength of 532 nm. The optical property of incident energy attenuation was applied to the optical limiter. The limiter mechanism was the interband transition of PtNPs excited by a nanosecond pulse. In 2007, B. Karthikeyan et al. [13] prepared Pt poval thin films and studied the nonlinear optical transmission behavior in the interband absorption region by using ultrafast laser pulses. The experimental results showed that the film had a strong optical power limitation. In 2009, R. A. Ganeev et al. [14] used a pulse with a width of 50 ps and a wavelength of 1064 nm to measure PtNPs with a Z-scan under different excitation intensities. At high excitation intensities, the saturated absorption (SA) and the reverse saturated absorption (RSA) occurred simultaneously. The suspension of PtNPs was

used as a saturable absorber to realize the mode-locking of the Nd: glass laser. In 2018, J. Yuan et al. [15] measured the nonlinear optical properties of PtSe₂ thin films. The results showed that the PtSe₂ thin films had a larger modulation depth (26%) and a lower saturated strength at 1064 nm. Using a fiber-doped ytterbium as an optical gain medium, and PtSe₂ film sandwiched between two fiber cores and saturable absorbers, an all-fiber ring cavity was established. In 2021, Y. R. Yuzaile et al. [16] prepared a platinum saturable absorber by plasma sputtering, and studied the influence of platinum saturable absorber parameters on the performance of a Q-switched erbium-doped fiber laser. Platinum saturable absorbers prepared under five different sputtering times (50~400 s) were deposited directly on the end surface of the optical fiber connector. The morphology, surface roughness, and linear and nonlinear optical parameters of each saturable absorber were characterized. The experimental results showed that the optimal bandwidth and pulse width were obtained at 50 s sputtering times. It was the first report on controlling saturable absorber parameters by changing platinum sputtering time. In addition, Y. Gao et al. [17] conducted a Z-scan with a 532 nm nanosecond pulse laser in 2005 to study the nonlinear optical absorption of platinum nanospheres protected by PVP in an aqueous solution, and observed the conversion from SA to RSA. The reason for the conversion was that the d-band of PtNPs was very close to the s-p band, which made it easy for the interband electron transition to occur under the excitation of a laser.

Based on the completed nonlinear research of PtNPs, it can be concluded that the Z-scan is carried out under a single wavelength, and the novel nonlinear optical characteristics under multi-wavelength and multi-excitation are unknown. The size and position of the SPR are affected by the size, shape, material, and surrounding medium environment of the particles. The nonlinear absorption characteristics are affected by the SPR and experimental conditions [18,19]. In this paper, the PtNPs prepared by the ablation method have good stability and good size controllability. The SPR can be clearly observed in the absorption spectrum, which ensures the smooth progress of the Z-scan experiment. Compared with the previous investigations, we used a multi-wavelength and multi-excitation nanosecond laser to conduct a Z-scan of the PtNPs. The excitation wavelengths are off-resonant wavelengths. The nonlinear absorption characteristics are observed from the distance between the excitation wavelength and the SPR wavelength. The excitation wavelength ranges from 500 nm to 700 nm, and each wavelength corresponds to three excitation energies of 752 μ J, 1088 μ J, and 1388 μ J. The transmission curves of different energies are measured. With the change of wavelengths, we need to observe whether the changing trend of the transmission curve of the different wavelengths is consistent. As a result, the multi-wavelength and multi-excitation measurement method complements the nonlinear absorption characteristics of PtNPs at broadband off-resonant wavelengths effectively, and provides more possibilities for the improvement of optical device performance.

2. Materials and Methods

A mode-locked coherent laser system (mira 900) combined with a coherent regenerative amplifier (legend-f) was used to generate laser pulses with a repetition rate of 1 kHz, wavelength of 800 nm, and pulse width of 130 fs. PtNPs were prepared with laser ablation [20].

The experiment was carried out at a room temperature of 24 °C. After cleaning a culture plate with ultrasonic wave, 5 mL absolute ethanol was added. Additionally, 1 mg poly (N-vinyl-2-pyrrolidone) (PVP) was dissolved, which is a macromolecular polymer with an average molecular weight of 40,000, to prevent the oxidation and agglomeration of PtNPs and to obtain PtNPs with small size and narrow size distribution. After the platinum target with a radius of 10 mm and a thickness of 0.5 mm was cleaned with ultrasonic wave, it was put into the solution contained in the culture plate, and needed to be immersed completely. The experimental setup is shown in Figure 1a. The height of the lens, with a focal length of 150 mm, was adjusted to focus the light on the platinum target, whose platinum content was 99.99%. When the target surface was ablated, sparks accompanied by

light, sound, and smoke were generated, and many bubbles were generated simultaneously. In order to avoid the attenuation of light strength caused by bubbles, the bubbles were removed with an absorbent ball, and the ethanol layer with a thickness of 2–3 mm was kept on the target surface. More than ten minutes later, a small amount of PtNPs colloid appeared, which were pink. With the increase in the amount of colloid generated by ablation, the colloid color became darker and darker. After approximately 30 min, a large number of PtNPs colloids were obtained, as shown in Figure 1b. The schematic diagram of the experimental device is shown in Figure 1c.

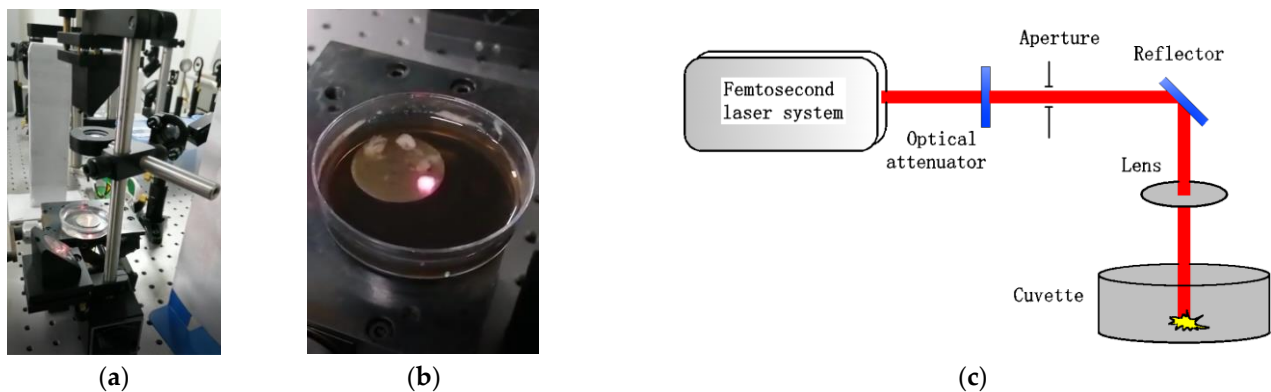


Figure 1. Experimental setup and schematic diagram of PtNPs prepared with ablation method. (a) Experimental setup. (b) PtNPs colloids in preparation. (c) Schematic diagram.

The platinum target was taken out and the mixture of the solution and colloid in the culture plate was placed until the temperature dropped to room temperature, 24 °C. The cooled mixture was poured into a sample bottle and an ultrasonic dispersion was conducted to obtain a uniformly distributed suspension of PtNPs. The average size of PtNPs measured with a scanning electron microscope (SEM) was approximately 25 nm. The characteristics of PtNPs shown in Figure 2a were the sample, dark brown, which was the result of light selective absorption. Figure 2b is the SEM image. The SEM image showed that PtNPs were spherical, whose diameters were 25 ± 2 nm. Figure 2c is the linear absorption spectrum of PtNPs, which was measured with the ultraviolet visible spectro-photometer usb4000. There was an obvious absorption peak at 226 nm caused by SPR.

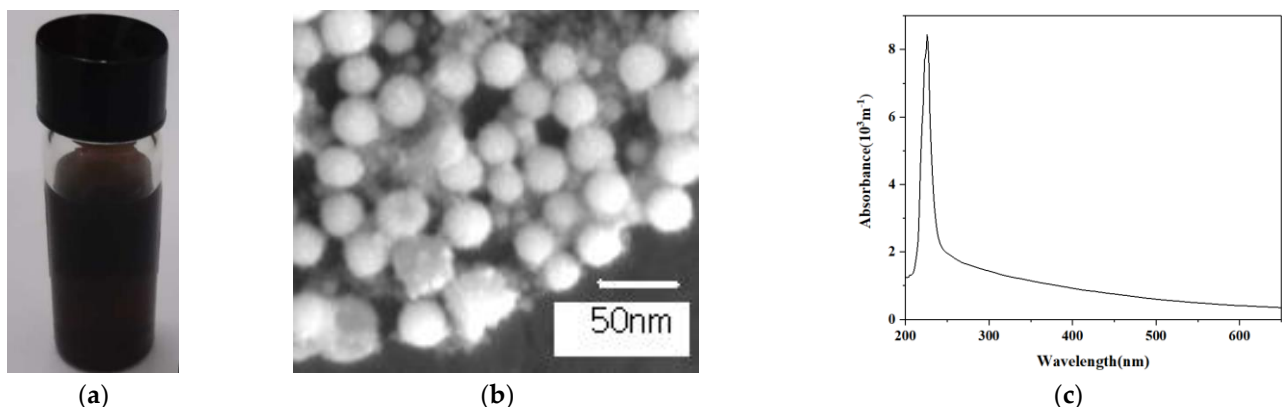


Figure 2. Absorption spectrum of PtNPs. (a) Colloid of PtNPs. (b) SEM image. (c) Linear absorption spectrum.

The nonlinear absorption properties of PtNPs were studied by using a typical open aperture Z-scan technique. In the Z-scan measurement, nanosecond Nd: YAG laser with a pulse width of 6 ns, a repetition rate of 10 Hz, and an optical parametric oscillator (OPO) were used to generate tunable laser pulses, whose spatial distribution was close to the

Gaussian distribution. The suspension of PtNPs was put into a cuvette with a thickness of 2 mm, and the laser pulse was focused on the cuvette. The linear transmission of the sample at 500 nm was 30%. In the Z-scan experiment, the waist radius ω_0 of the laser beam was 50 μm , which was measured with the blade method. Additionally, the focal length of the laser beam was 20 cm. The cuvette containing PtNPs was placed on a mobile platform and was moved along the z -axis. The computer could accurately control the moving step of the platform and record the laser pulse energy received and transmitted at each point when the sample passed through the focus area of the laser beam.

3. Result and Discussion

The nonlinear absorption characteristics of PtNPs in the wavelength range of 500–700 nm were studied by the open aperture Z-scan. Five representative wavelengths of 500 nm, 550 nm, 600 nm, 650 nm, and 700 nm were selected and measured at the excitation energies of 752 μJ , 1088 μJ , and 1388 μJ . The incident light strength at the focus was $3 \times 10^{13} \text{ W/m}^2$, $4.34 \times 10^{13} \text{ W/m}^2$, and $5.54 \times 10^{13} \text{ W/m}^2$, respectively.

Figure 3 shows the characteristic curve of normalized transmission changing with the z position under different excitation energies at a certain wavelength. Figure 3a–e were the normalized transmission curves at the excitation energies of 752 μJ , 1088 μJ , and 1388 μJ at the wavelengths of 500 nm, 550 nm, 600 nm, 650 nm, and 700 nm, respectively. The dotted lines were the experimental data, and the solid lines were the theoretical fitting data. Normalized transmission was the measured transmission divided by the linear transmission of 30%.

Figure 3a shows that when the wavelength was 500 nm, the samples showed the conversion from SA to RSA under three excitation energies. With the increase in excitation energy and incident light strength, the degree of conversion increased. Specifically, when the sample moved to the focus, the transmission of the sample first increased and then decreased, and the transmission had a valley at the focus. When the sample moved away from the focus, the transmission of the sample first increased and then decreased with the moving sample. The transformation degree was the lowest at 752 μJ and the highest at 1388 μJ . With the increase in excitation energy, the conversion degree caused by two-photon absorption deepened.

Figure 3b shows that when the wavelength was 550 nm, the samples showed the conversion from SA to RSA under three excitation energies. With the increase in excitation energy and incident light strength, the conversion degree was deepened due to two-photon absorption at 752 μJ and 1088 μJ . At 1388 μJ , compared with 752 μJ and 1088 μJ , the conversion degree was weaker, because the number of electrons that could meet the two-photon absorption was decreasing.

Figure 3c shows that when the wavelength was 600 nm, at 752 μJ and 1088 μJ , the sample showed SA, which was due to the ground state plasma bleaching. With the increase in excitation energy and incident light strength, the degree of SA was deepened. At 1388 μJ , the sample showed the conversion from SA to RSA, because of the increase in excitation energy, two-photon absorption occurred.

Figure 3d shows that when the wavelength was 650 nm, the samples showed the conversion from SA to RSA at 752 μJ and 1088 μJ excitation energies, and with the increase in excitation energy and incident light strength, the conversion degree was deepened due to two-photon absorption. Compared with 752 μJ and 1088 μJ , 1388 μJ showed SA, which may be due to three-photon absorption and multi-photon absorption.

Figure 3e shows that when the wavelength was 700 nm and the excitation energy was 752 μJ and 1088 μJ , the sample showed SA. Due to the increase in the excitation energy, the incident light strength increased, and the degree of SA deepened. When the excitation energy was 1388 μJ , the sample showed the conversion from SA to RSA. This happened because two-photon absorption occurred with the increase in excitation energy.

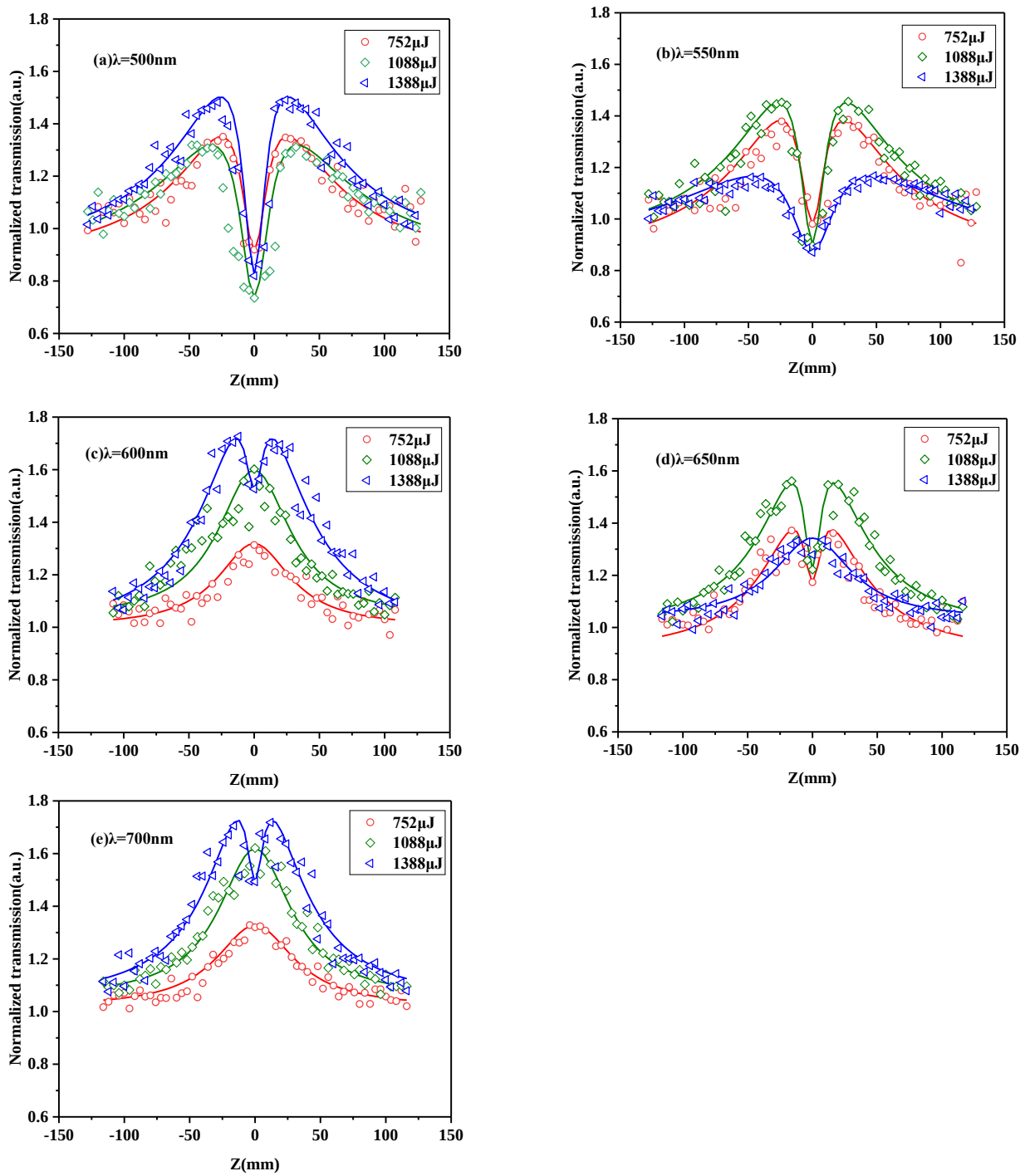


Figure 3. Normalized transmission of PtNPs for open aperture Z-scan measurements under different wavelengths of (a) 500 nm, (b) 550 nm, (c) 600 nm, (d) 650 nm, and (e) 700 nm. The dotted lines were experimental data, and the solid lines were theoretical fitting data.

In order to better understand the above results, corresponding to Figures 3a–e and 4a–e show the relationship between the normalized transmission of PtNPs and the energy density of the incident laser pulse. The dotted lines were experimental data.

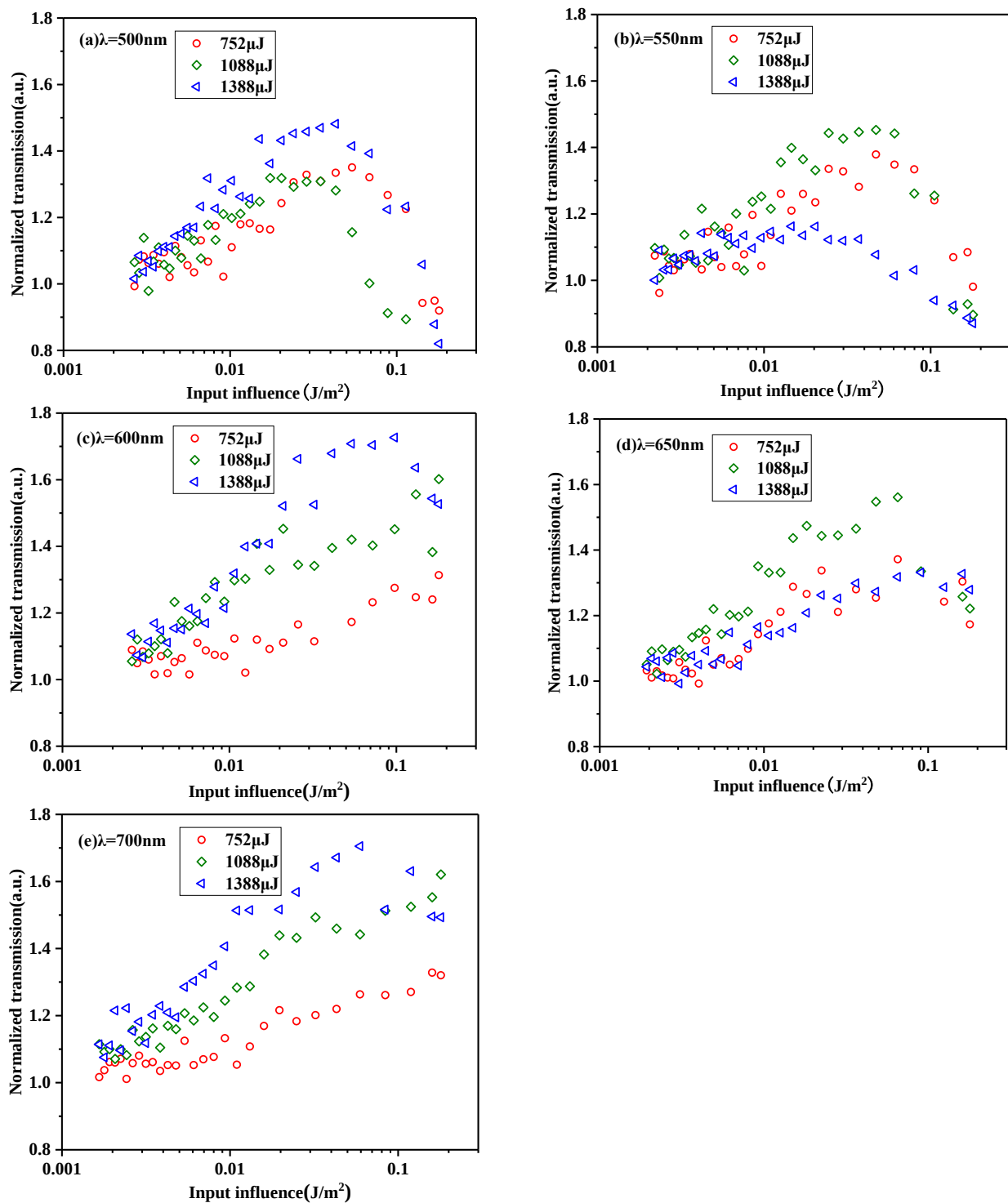


Figure 4. The relationship curve between the normalized transmission of PtNPs and the incident laser pulse energy density under different wavelengths of (a) 500 nm, (b) 550 nm, (c) 600 nm, (d) 650 nm, and (e) 700 nm. The dotted lines were experimental data.

Figure 4a,b show that the normalized transmission of the sample first increased and then decreased with the increase in energy density, which showed the conversion from SA to RSA. Figure 4c,e show that when the laser energy was 752 μJ and 1088 μJ , the normalized transmission of the sample increased with the increase in the energy density, which showed only SA. At 1388 μJ , the normalized transmission first increased and then decreased with the increase in energy density, which showed the conversion from SA to RSA. Figure 4d

shows that when the laser energy was 752 μJ and 1088 μJ , the normalized transmission of the sample first increased and then decreased with the increase in the energy density, which showed the conversion from SA to RSA. At 1388 μJ , the normalized transmission increased with the increase in the energy density, which showed only SA.

Combined with the energy level diagram of PtNPs in Figure 5, the reason for the results of the open aperture Z-scan experiment was analyzed. Under the excitation of a light pulse, the external field caused the collective oscillation of electrons in the conduction band. The light strength increased as the sample approached the focus, which caused nonlinear absorption. Most of the outermost electrons in the d-band of PtNPs and the quasi electrons in the s-p band near the Fermi level transitioned from the ground state to the excited state, which led to the decrease in ground state free electrons and ground state plasma bleaching. Additionally, at the focus, the transmission was the largest, and SA was formed. With the increase in excitation energy, the electrons in the d-band absorbed two, three, or more photons simultaneously and transitioned to the s-p band. Alternatively, the electrons in the ground-state absorbed two, three, or more photons simultaneously and transitioned to the excited state. These processes were two-photon, three-photon, and multi-photon absorption processes.

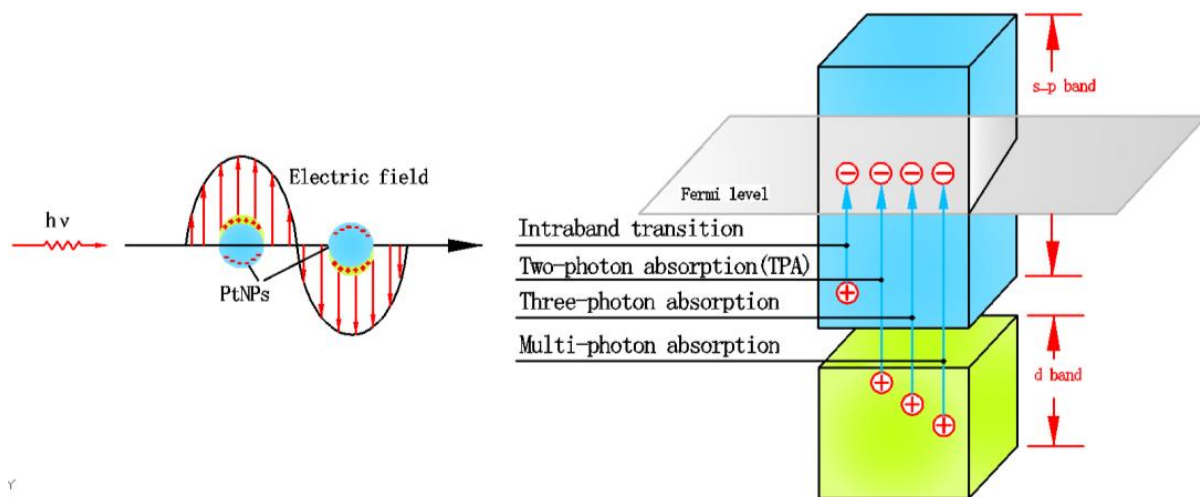


Figure 5. Schematic diagram of energy level transition of PtNPs.

Moving towards the focus, the free carriers in the excited state continued to absorb photons, and the electrons transitioned from the excited state to a higher second excited state. Thus, the transmission before the focus increased, which formed SA. When the transmission increased to a certain extent, it began to decrease until the light strength reached the maximum. The transmission reached the minimum at the focus, which formed RSA. The SPR of PtNPs was at 226 nm, and the excitation wavelength increased from 500 nm to 700 nm. Under the same excitation energy, 500 nm near SPR was easier to achieve than the nonlinear absorption of PtNPs. At the same excitation wavelength, the higher the excitation energy was, the faster the electrons could obtain the energy of the interband transition, and the easier it was to realize the nonlinear absorption of PtNPs.

In addition to the experimental analysis of nonlinear absorption, it is also necessary to describe the nonlinear absorption characteristics of PtNPs quantitatively in order to obtain the saturation light strength and nonlinear absorption coefficient.

The sample transmission [17] can be expressed as

$$T = 1 - \left(\frac{\alpha_0}{1 + \frac{I_0}{(1+z^2/z_0^2)I_s}} + \frac{\beta I_0}{1 + z^2/z_0^2} \right) L \quad (1)$$

where α_0 is the linear absorption coefficient, I_0 is the light strength at the focus, I_s is the saturation light strength, β is the nonlinear absorption coefficient, z is the axial displacement

of the sample from the focus, z_0 is the Rayleigh diffraction length, and L is the thickness of the cuvette.

The experimental data were fitted by Equation (1), and the theoretical fitting results were shown in Figure 3, which showed that the theoretical fitting curves were in good agreement with the experimental data curves. The calculated values of saturation strength I_s and nonlinear absorption coefficient β are shown in Table 1.

Table 1. Nonlinear Optical Parameters of PtNPs.

$\lambda(\text{nm})$	$E(\mu\text{J})$	$I_0(\text{W/m}^2)$	$I_s(\text{W/m}^2)$	$\beta(\text{m/W})$
500	752	3×10^{13}	1.36×10^{12}	2.1×10^{-10}
	1088	4.34×10^{13}	1.97×10^{12}	1.81×10^{-10}
	1388	5.54×10^{13}	1.73×10^{12}	2.2×10^{-10}
550	752	3×10^{13}	1.2×10^{12}	2.08×10^{-10}
	1088	4.34×10^{13}	1.74×10^{12}	2.16×10^{-10}
	1388	5.54×10^{13}	2.21×10^{12}	5.37×10^{-11}
600	752	3×10^{13}	1.07×10^{12}	0
	1088	4.34×10^{13}	2.41×10^{12}	0
	1388	5.54×10^{13}	2.21×10^{12}	1.28×10^{-10}
650	752	3×10^{13}	1.36×10^{12}	1.02×10^{-10}
	1088	4.34×10^{13}	1.97×10^{12}	1.38×10^{-10}
	1388	5.54×10^{13}	3.07×10^{12}	0
700	752	3×10^{13}	1.67×10^{12}	0
	1088	4.34×10^{13}	2.41×10^{12}	0
	1388	5.54×10^{13}	2.41×10^{12}	1.23×10^{-10}

In order to directly reflect the influence of excitation wavelength and excitation energy on the nonlinear absorption of PtNPs, Figure 6 was plotted according to Table 1.

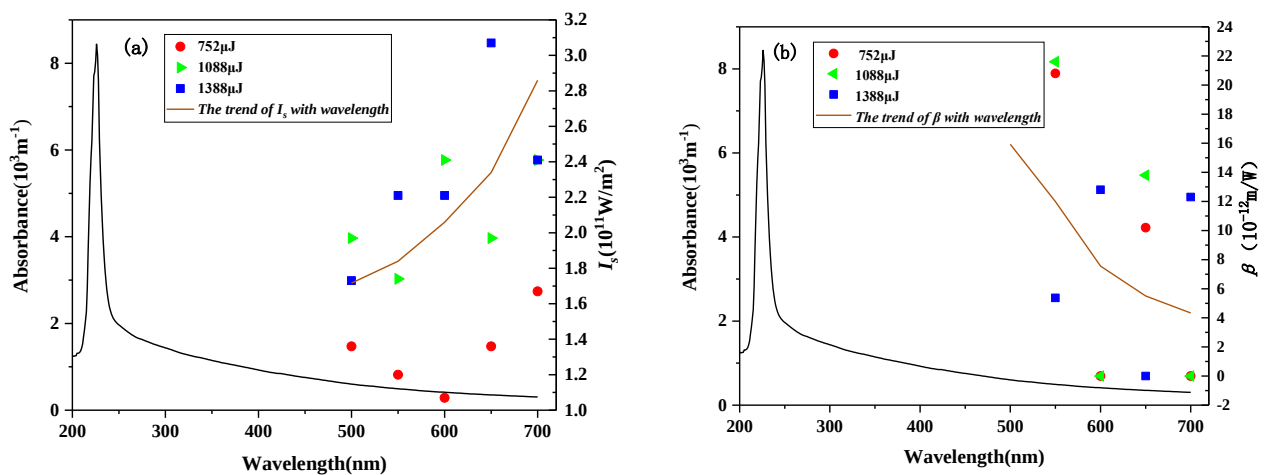


Figure 6. Nonlinear absorption characteristic curves of PtNPs depending on excitation wavelengths and the excitation energy. (a) The relationship curve of the saturation strength I_s . (b) The relationship curve of the nonlinear absorption coefficient β .

Figure 6a shows a double y-axis characteristic curve of PtNPs, which shows the change of the saturation strengths and the linear absorption spectrum, so as to visually display the change of saturation strengths under three excitation energies of 752 μJ , 1088 μJ , and 1388 μJ and the influence of SPR on the saturation strength. The red dots represent the saturation strengths of 500 nm, 550 nm, 600 nm, 650 nm, and 700 nm at 752 μJ . The green triangles represent the saturation strengths of 500 nm, 550 nm, 600 nm, 650 nm, and 700 nm at 1088 μJ . The blue squares represent the saturation strengths of 500 nm,

550 nm, 600 nm, 650 nm, and 700 nm at 1388 μJ . The brown line was the trend of the saturation strength changing with wavelength, and the black line is the linear absorption spectrum. Figure 6b also shows a double y-axis characteristic curve showing the nonlinear absorption coefficients and the linear absorption spectrum, which could show the trend of the nonlinear absorption coefficient under 752 μJ , 1088 μJ , and 1388 μJ excitation energies and the influence of SPR on the nonlinear absorption coefficient. The red dots represent the nonlinear absorption coefficients of 500 nm, 550 nm, 600 nm, 650 nm, and 700 nm at 752 μJ . The green triangles represent the nonlinear absorption coefficients of 500 nm, 550 nm, 600 nm, 650 nm, and 700 nm at 1088 μJ . The blue squares represent the nonlinear absorption coefficients of 500 nm, 550 nm, 600 nm, 650 nm, and 700 nm at 1388 μJ . The brown line is the trend of the nonlinear absorption coefficient changing with wavelength, and the black line is the linear absorption spectrum.

In Figure 6a, the brown line is the overall trend diagram of the red dot, the green triangle, and the blue square distribution, from which it could be concluded that when the excitation wavelength was far away from the resonance wavelength, the overall trend of the saturation strength of the PtNPs increased under the three different excitation energies. At the same excitation wavelength, the higher the excitation energy was, the greater the saturation strength of the PtNPs was. The reason why the saturation strength curve and the linear absorption spectrum were put together was to observe the influence of the distance between the excitation wavelength and the SPR on the saturation strength, which helps to visually observe how the saturation strength changes far away from the SPR.

In Figure 6b, the brown line is the overall trend diagram of the color dot distribution, from which it could be observed that the general trend of the nonlinear absorption coefficient was different from that of the saturation absorption strength. With the excitation wavelength away from the resonance region, the nonlinear absorption coefficient corresponding to higher excitation energy was smaller, and the nonlinear absorption coefficient corresponding to lower excitation energy was larger. The reason why the nonlinear absorption coefficient curve was plotted together with the linear absorption spectrum is similar to Figure 6a.

The increase in the saturation strength was due to the resonance decrease in nonlinear characteristics. When the excitation energy was appropriate, the bleaching of ground-state plasma would lead to SA. However, the number of electrons in the conduction band was different with different excitation wavelengths. The closer to SPR, the easier to realize the ground-state plasma bleaching. The larger the excitation energy was, the more electrons the conduction band could obtain, which could realize the bleaching easier. The results showed that the absorption decreased, and the saturation strength increased. The decrease in the nonlinear absorption coefficient far away from the SPR was due to the enhancement of free carrier absorption.

4. Conclusions

In order to study the nonlinear absorption of PtNPs, the open aperture Z-scan experiments were carried out at 500–700 nm broadband off-resonant wavelengths under multi-excitation energy. The results showed that the SPR of PtNPs was far away from the excitation wavelength, but there were still SA and RSA. The nonlinear absorption of PtNPs was related to the excitation wavelength and the excitation energy. At the same excitation wavelength, the higher the excitation energy was, the smaller the nonlinear absorption coefficient was, and the faster the energy of the interband transition could be obtained, which made it easier to realize the nonlinear absorption of PtNPs. With the same excitation energy, the wavelength near SPR was easier to achieve than the nonlinear absorption. PtNPs had high transparency and low propagation loss in the visible and infrared range, which could improve the performance of optical limiters and saturable absorbers.

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