Gold Nanocages as Saturable Absorbers for Passively Q-Switched Nd:YVO₄ Lasers with Optimized Performance

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Received: 9 September 2020; Accepted: 4 October 2020; Published: 6 October 2020

Abstract: Based on a gold nanocage saturable absorber (GNC-SA) with the surface plasmon resonance (SPR) absorption peak located at 1.06 µm, passively Q-switched Nd:YVO₄ lasers with a center wavelength of 1064.1 nm were demonstrated. Q-switched pulses with the shortest pulse duration of 143 ns and a pulse repetition rate of 467 kHz were achieved at transmittance T = 4% under a pump power of 5.98 W. Under a pump power of 3.95 W, the maximum average output power of 372 mW was obtained at a laser with transmittance of 10.8%, corresponding to an optical conversion efficiency of 9.4% and a slope efficiency of 14.1%. Our results reveal that for passively Q-switched lasers at a given wavelength, we are able to synthesize the most suitable GNC-SA to obtain the best output characteristics of lasers.

Keywords: gold nanocages; saturable absorber; passively Q-switched; Nd:YVO₄ laser

1. Introduction

Diode-end-pumped all-solid-state Q-switched Nd-doped lasers operating around 1 µm have been extensively used in the realms of biomedicine, mechanical processing, laser marking, and remote sensing due to their superiorities in terms of compact structure and high-efficiency method of generating high-performance pulses [1–3]. In addition, some researchers have focused on the application of lasers in an industrial environment, such as laser cladding and hardening technology [4,5]. Passive Q-switching technology stands out from all Q-switched methods and has attracted widespread attention in recent decades because of its simple structure and compactness. For these passively Q-switching lasers, the quality of the output pulse mainly depends on properties of the saturable absorber (SA), which always sparks a wide range of research interests. The traditional SAs, such as Cr⁴⁺:YAG [6], GaAs [7], and semiconductor saturable absorber mirrors (SESAMs) [8], have gradually matured and been widely used in passively Q-switched lasers. But they have limitations in terms of a narrow absorption band and complicated preparation process [9]. By contrast, novel two-dimensional (2D) materials with the characteristics of a wide working band, fast recovery time, and convenient preparation have more advantages for obtaining Q-switched pulses. Up to now, in passively Q-switched lasers the SA properties of topological insulators (TIs) [9], graphene [10,11], semiconducting transition metal dichalcogenides (s-TMDs) [12], black phosphorus (BPs) [13], and gold nanoparticles (GNPs) [14,15] have been demonstrated.
In these 2D materials, the existence of localized surface plasmon resonance (SPR) peaks has made GNPs become the research focus in recent years [16,17]. SPR is a phenomenon caused by the vibration of GNPs’ free electrons resonating with input photonics on the surface of GNPs [18]. Through controlling the metal composition and particle shape and size, the electron charge density on the particle surface could be tuned. Thus, GNPs have tunable SPR peaks from visible to mid-infrared (IR) regions [17,19]. All of these properties indicate that GNPs can be use as effective SAs. In 2013, Kang et al. used gold nanorods (GNRs) as SAs and reported a 4.8 µs passively Q-switched erbium-doped fiber (EDF) laser [20]. Using a gold-nanosphere (GNS) as a SA, Fan et al. demonstrated an all-fiber passively Q-switched EDF laser at 1562 nm with the pulse width of 1.78 µs and the pulse repetition frequency (PRF) of 58.1 kHz in 2014 [21]. In 2016, Zhang et al. investigated a passively Q-switched laser at 1064.1 nm using gold nanobipyramids (GNBPs) as SAs, in which the pulse width of 396 ns and the PRF of 90.6 kHz were achieved [22]. Gold nanotriangles (GNTs) and GNRs as SAs for a passively Q-switched Nd:YAG ceramic laser at 1 µm was demonstrated by our team in 2017 [23,24]. With lasers using GNRs as SAs, the pulse width of 223 ns and PRF of 300 kHz at 1064.3 nm and the pulse width of 504 ns and PRF of 120 kHz at 1112 nm were obtained, respectively [23]. While with lasers using GNTs as SAs, the pulse width of 179 ns and PRF of 320 kHz at 1064 nm and the pulse width of 231 ns and PRF of 457 kHz at 1123 nm were obtained, respectively [24]. In 2018, based on GNCs and GNCs/SiO₂ as SA, a passively Q-switched Nd:YVO₄ laser with the shortest pulse duration of 154.2 ns and PRF of 279 kHz operating at 1064.3 nm, a passively Q-switched Yb-doped fiber laser with the pulse width of 1.4 µs and the PRF of 126.9 kHz operating at 1060.5 nm, and another Yb-doped fiber laser with the dual wavelength at 1059.9 and 1060.5 nm were demonstrated by our team, respectively [25–27].

Recently, GNCs have stood out from all these GNPs due to their advantages of excellent thermal effects, such as high heat resistance, fast thermal recovery time on the scale of tens of picoseconds, and high photothermal stability [28]. GNCs have a large surface/volume ratio benefit due to their cubic structure with porous walls and hollow interiors, which promise their extraordinarily large optical absorption cross-sections [29]. Meanwhile, the cubic structure is also conducive to heat dissipation, which profits from expanding the area contacting with air. Thus, GNCs have excellent heat dissipation performance and a high damage threshold, which indicate that it can be used as a SA in high-power lasers to achieve stable pulse output [27]. In addition, the SPR peak mainly depends on the difference between inner cavities and external walls. Scientific research has found that as the difference decreases, the SPR peak will red-shift [30]. Therefore, GNCs have tunable SPR absorption peaks from visible to mid-IR regions, which can be achieved by adjusting the size and thickness of the external walls [31]. In other words, when we use GNCs as SAs in lasers, in order to obtain the best performance, we can customize the desired particles to meet our needs by controlling the growth of GNCs with diverse morphologies.

In this letter, GNC-SAs with an SPR peak located at 1.06 µm were successfully fabricated and their characteristics demonstrated in a passively Q-switched Nd:YVO₄ laser with a center wavelength of 1064.1 nm using two output couplers with transmittances of 4.0 and 10.8%. In a laser with an output mirror transmittance of 4.0%, the shortest pulse width of 143 ns and PRF of 467 kHz were achieved at a pump power of 5.98 W. In a laser with a transmittance of 10.8%, the maximum average output power was 372 mW, which was obtained under 3.95 W pumping power, corresponding to an optical conversion efficiency of 9.4% and a slope efficiency of 14.1%.

2. Preparation and the Characterization of the GNCs

In the experiment, we prepared GNCs by the seed-mediated method [32]. First, the Ag nanocube solution was prefabricated by the polyol method, serving as sacrificial templates; its transmission electron microscope (TEM) image is shown in Figure 1a with a scale bar of 2.0 µm. Then, based on the as-prepared Ag nanocubes, GNCs were synthesized by the galvanic replacement reaction [33].
Figure 1b shown the TEM image of the GNCs with a scale bar of 0.1 μm. Gold nanocage is a hollow nanocrystal, and its crystal structure is single crystalline, which can be regarded as a kind of inorganic crystalline material [33–35]. It can be seen that the GNCs have a cubic shape with a hollow interior and show strong variation in size. The size distribution histogram of 30 GNCs is shown in Figure 1c. As we can see, the size of the GNCs ranges from 33 nm to 103 nm, and the range of 40 nm–70 nm occupies a large proportion of about 65%. The average size of GNCs is calculated to be about 62.53 nm. In fact, “gold” nanocages are essentially Au/Ag alloyed structures, which means that the composition of GNCs contains Au and Ag, probably as an alloy [33–35]. Lastly, the GNCs solution is mixed with polyvinyl alcohol (PVA) and then the mixed solution is applied to a K9 glass sheet by spin coating. Finally, it is slowly dried at room temperature to obtain the final GNC/PVA film SA. The films contained a few layers of GNCs, and so the GNC-SAs were regarded as 2D materials. The minutiae of the synthesis process of the GNCs can be found in our previous work [27].

Using a visible-ultraviolet (UV) spectrophotometer, we measured the absorption spectrum of GNCs, which is shown in Figure 2a. As the picture shows, the absorption spectrum of GNCs features two peak values of 528 nm (the transverse SPR absorption peak) and 1.06 μm (the longitudinal SPR absorption peak), which is consistent with the emission spectrum of Nd atoms at 1064 nm. In order to evaluate the nonlinear absorption property of GNCs, the dependence of transmittance on the input intensity was measured by a Nd:YVO4 laser operating at 1064 nm with a pulse width of 15 ps and PRF of 41.5 MHz using open-aperture Z-scan technology [36]. As described in Figure 2b, by fitting the data with the formula \( y = A_1 \times \exp(-x/t_1) + y_0 \), the modulation depth \( A_1 \) is 5.3% and the saturable optical intensity \( I_1 \) is 0.16 MW/cm², further indicating that the GNCs can be used as SAs to trigger the lasers to enter the Q-switched state.

Figure 1. TEM image of the (a) Ag nanocubes and (b) gold nanocages (GNCs). (c) Size distribution histogram of the GNCs.
We wrapped the Nd:YVO$_4$ crystals with indium foil and clamped them in a copper block, which is cooled with water at 20 °C to aid heat dissipation. In order to compare the performance of lasers, the output coupling (OC) mirror adopted two plane mirrors M2 with output rates of 4.0% and 10.8% at 1064 nm. The gain medium was a c-cut Nd:YVO$_4$ crystal with the dimensions of $4 \times 4 \times 18$ mm$^3$ and the Nd-doping density of 0.5 at%, which was AR for 808 nm and 1064 nm (R < 0.2%) on both sides. We wrapped the Nd:YVO$_4$ crystals with indium foil and clamped them in a copper block, which is cooled with water at 20 °C to aid heat dissipation.

3. Experimental Setup

The experimental device of the passively Q-switched Nd:YVO$_4$ laser using GNCs as a SA is shown in Figure 3. A plane-concave cavity with a length of 30 mm was composed of a plane-concave mirror (M1) and a plane mirror (M2). The pump source was a commercial 808 nm fiber-coupled laser diode (LD, numerical aperture: 0.22, beam radius: 400 μm). A 1:1 coupling optical system was used to focus the light into the laser crystal. A plane-concave mirror with a radius of curvature of 300 mm was used as the input mirror, which was anti-reflected (AR) to 808 nm and high-reflected (HR) to 1064 nm. The gain medium was a c-cut Nd:YVO$_4$ crystal with the dimensions of $4 \times 4 \times 18$ mm$^3$ and the Nd-doping density of 0.5 at%, which was AR for 808 nm and 1064 nm (R < 0.2%) on both sides. A 1 GHz InGaAs detector produced by New Focus (Santa Clara, CA, USA) with the rising time of 400 ps was employed to detect the laser pulses. A digital storage oscilloscope (DSO90804A Infiniium, Santa Clara, CA, USA) with an 8 GHz bandwidth and 40 G samples/s was employed to record the pulse width and repetition rate. An optical spectrum analyzer (Yokogawa AQ6315A, 350–1750 nm, Tokyo, Japan) was used to measure the emission spectrum.

4. Experimental Results and Discussion

Firstly, without the GNCs, we investigated the continuous wave (CW) Nd:YVO$_4$ lasers employing two OCs with $T = 4.0\%$ and 10.8%. Figure 4 shows the dependence of the CW laser output powers on the pump powers, and the threshold powers of the lasers were 0.264 W and 0.497 W for $T = 4.0\%$ and 10.8%.
on the pump powers, and the threshold powers of the lasers were 0.264 W and 0.497 W for T = 4.0% and 10.8%, respectively. The average output power increases linearly as the pump power raises, with slope efficiencies of 32.0% and 42.3% at T = 4.0% and 10.8%, corresponding to the optical conversion efficiencies of 30.3% and 38.8%, respectively.

![Figure 4](image4.jpg)

**Figure 4.** Average output powers vs. pump powers of the continuous wave (CW) laser at T = 4.0% and 10.8%. Toc: Transmittance of output coupling mirror

Then, we realized the typical passive Q-switching operation by inserting the GNC-SA into the cavity. Figure 5a describes the relationship of the incident pump powers and the output powers. For the Q-switched lasers with T = 4.0% and 10.8%, the Q-switched pulses output was obtained when the pump power reached 1.54 W and 1.26 W. The large insert loss of the GNC-SA led to the thresholds of the Q-switched lasers being higher than the CW lasers. For lasers with T = 4.0% OC under a pump powers of 5.98 W, the maximum average output powers of 211 mW were received, corresponding to optical-to-optical conversion efficiencies of 3.5% and slope efficiencies of 4.9%. For lasers at T = 10.8%, under a pump power of 3.95 W, a total of 372 mW average output powers were received, corresponding to optical-to-optical conversion efficiencies of 9.4% and slope efficiencies of 14.1%, which are remarkable breakthroughs compared with previous works on other reported Q-switched lasers based on GNP's [20–27]. When pump powers are further increased, the passive Q-switching operation becomes unstable. Figure 5b describes the emission spectrum of the Q-switched laser at T = 4.0%, and the spectrum of the laser with 10.8% OC was the same as that of 4.0% OC. It was evident that the central wavelength is 1064.1 nm, which is in line with the SPR absorption peaks of the GNC-SA.

![Figure 5](image5.jpg)

**Figure 5.** (a) Average output powers vs. pump powers of the Q-switched laser at T = 4.0% and 10.8%. (b) Emission spectrum of the Q-switched laser at T = 4.0%.
Figure 6 depicts the pulse width and the PRF of the Q-switched lasers at $T = 4.0\%$ and $10.8\%$ OCs versus the pump power. When the pump power gradually increases from 2.0 W to 5.98 W, the pulse width decreases from 700 ns to 143 ns and the PRF increases from 50 kHz to 467 kHz for lasers with 4.0\% OC. Under a pump power of 5.98 W, the waveforms with the maximum PRF of 467 kHz and the narrowest pulse width of 143 ns are shown in Figure 7a. As far as we know, 143 ns is the narrowest pulse width and 467 kHz is the highest PRF obtained within GNP-based passively Q-switched lasers. For the Q-switched laser at $T = 10.8\%$, the pulse width decreased from 446 ns to 240 ns and the PRF increased from 79 kHz to 440 kHz as the pump power changed from 1.3 W to 3.95 W. Under a pump power of 3.95 W, the waveforms with the PRF of 440 kHz and the pulse width of 240 ns are shown in Figure 7b.

![Figure 6](image1.png)

**Figure 6.** Pulse width and pulse repetition frequency vs. pump powers of lasers with (a) $T = 4.0\%$ and (b) $T = 10.8\%$.

![Figure 7](image2.png)

**Figure 7.** Single pulse contour and pulse sequences of GNC Q-switched lasers with (a) $T = 4.0\%$ and (b) $T = 10.8\%$.

Finally, in order to further highlight the outstanding characteristics of GNCs, passively Q-switched lasers operating at near-IR regions using GNP-SAs are presented in Table 1. As is shown, compared with previous works, GNC-based Q-switched lasers at $T = 4.0\%$ obtained the narrowest pulse width and highest PRF. Meanwhile, GNC-based Q-switched lasers obtained the highest slope efficiency with a transmittance of 10.8\%. All of the mentioned results indicate that GNCs have significant advantages
in Q-switched lasers over the GNP-SAs reported before, which might be attributed to the high heat resistance, fast thermal recovery time, and high photothermal stability of GNCs.

### Table 1. Performance comparison based on the gold nanoparticle superabsorber (GNP-SA) passively Q-switched lasers at the near-IR regions. GNRs: Gold nanorods; GNS: Gold-nanosphere; GNBPs: Gold nanobipyramids; GNTs: Gold nanotriangles

| Type of GNP | Wavelength /nm | Maximum Output Power /mW | Conversion Efficiency /% | Slope Efficiency /% | Narrowest Pulse Width /µs | Repetition Frequency /kHz | Ref. |
|-------------|----------------|--------------------------|--------------------------|-------------------|-----------------------------|---------------------------|------|
| GNRs        | 1560           | 12.5                     | -4.5                     | -4.4              | 4.8 ns                      | 39.9                      | [20] |
| GNS          | 1562           | 7.7                      | 3.5                      | 1.78 µs           | 58.1                        | 21]                      |
| GNBPs       | 1064.1         | 151                      | 1.26                     | 2.82              | 396 ns                      | 90.6                      | [22] |
| GNRs        | 1064.3         | 101                      | 1.24                     | 2.22              | 223 ns                      | 300                      |      |
| GNTs        | 1064.3         | 226                      | 5.4                      | 10.7              | 179 ns                      | 320                      | [23] |
| GNCs/SiO₂   | 1064.3         | 150.2                    | 6.21                     | 15.4              | 280                         |                          | [24] |
| GNCs/SiO₂   | 1060.5         | 10.6                     | 2.41                     | 4.0               | 14.2 µs                     | 136.9                     | [25] |
| GNCs        | 1059.9&1060.5  | 6.03                     | 1.57                     | 2.75              | 2.06 µs                     | 134.9                     | [26] |
| GNCs        | 1064.1         | 372                      | 9.4                      | 14.1              | 143 ns                      | 467 This work            |      |

5. Conclusions

In conclusion, using GNCs as SAs, passive Q-switching operation was realized successfully in Nd:YVO₄ lasers, which have the optimized performance. The two OCs with transmittances of 4.0% and 10.8% were used. For the laser at T = 4.0%, the maximum output power of 211 mW was received under the pump power of 5.98 W, with the shortest pulse width of 143 ns and the PRF of 467 kHz, which are both the best capability compared with previously reported passively Q-switched lasers using GNPs as SAs. For the laser at T = 10.8%, the pulse width of 240 ns, the PRF of 440 kHz, and the maximum output power of 372 mW were obtained at a pump power of 3.95 W, corresponding to optical-to-optical conversion efficiencies of 9.4% and slope efficiencies of 14.1%. As far as we know, in comparison with the other reported GNPs based on passively Q-switching lasers, optical conversion efficiencies (9.4%) and slope efficiencies (14.1%) obtained in our experiment have been significantly enhanced. Our experimental results reveal that we can synthesize the appropriate GNC-SAs by transforming their properties to obtain the best output characteristics of the lasers at a given wavelength.

### Author Contributions

Conceptualization, P.L.; Methodology, all authors; Software, B.Z., H.L.; Validation, X.C. and P.L.; Investigation, B.Z., L.X., and B.L.; Resources, X.C. and P.L.; Writing—original draft preparation, B.Z. and X.C.; Writing—review and editing, all authors; Visualization, B.Z., H.L., L.X., and B.L.; Project administration, P.L.; Funding acquisition, X.C. and P.L. All authors have read and agreed to the published version of the manuscript.

### Funding

This research is supported by the Natural Science Foundation of Shandong Province (ZR2019MF043, ZR2019MF047).

### Conflicts of Interest

The authors declare no conflict of interest.

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