



A new type of graphene oxide and its application in laser devices



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ARTICLE INFO

Article history:

Received 14 December 2016

Received in revised form

22 January 2017

Accepted 3 February 2017

Available online 16 February 2017

Keywords:

Graphene oxide

Preparation

Laser materials

Saturable absorber

ABSTRACT

Laser materials are very important in development of efficient laser devices. In this paper, a new type of graphene oxide (GO) was synthesized for developing pulse laser devices. The GO was characterized by X-ray diffraction, scanning electron microscopy, Fourier transform infrared, elemental analysis. Experimental data show that percentage of oxygen (46.85 wt%) of the GO is higher than that (27.97 wt%) of GO which was prepared by a Hummers' method. Main oxygen-containing groups at the GO are hydroxyl groups. The GO could be potential optoelectronic materials, because the increase of oxygen percentage would improve electrochemical pseudocapacitor. Optoelectronic properties of the GO were demonstrated by a Q-switching laser which has an optical-optical conversion efficiency of 11.3%. The GO shows very high stability under 6 W pump power over 1 h. Experimental results suggested that this kind of GO would be potential pulse laser materials.

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1. Introduction

Laser materials are very important in development of efficient laser devices. Popular laser materials include crystal, films and so on [1–6]. In recent years, a lot of laser materials were developed for improving properties of laser devices [1–4,7–10]. Ko et al. [1] and Nattermann et al. [7] reported that crystals were used as laser materials to improve parameters of solid laser devices, because organic crystals exhibited ultrafast intermolecular charge transfer [11–14] and good transparency [15]. Nano particles are usually employed to develop efficient lasers [2,4,8]. Metal salts and oxides were employed to develop efficient laser devices [9,16]. However, it is still an important investigation that an economic and efficient materials would be developed for improving laser devices.

Graphene is a good optoelectronic material which may be applied in ultrafast laser, superconductor and heat storage [17,18]. However, it is expensive due to its complex production procedures [19–22]. Graphene oxide is a kind of 2-dimension materials with oxygen-containing groups. It may be produced by several simple

methods [23–25], therefore, graphene oxide is a cheap materials compared with graphene. It would be interesting that graphene were replaced by graphene oxide for developing solid laser devices.

In this paper, a new type of graphene oxide (GO) was synthesized for developing pulse laser devices. The GO was characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier transform infrared (FT-IR), elemental analysis (EA). Optoelectronic properties of the GO were demonstrated by a Q-switching laser. Experimental results suggested that this kind of GO would be a potential pulse laser materials.

2. Experimental

2.1. Materials and equipment

Natural flake graphite (400 Mesh, 99.0%) was bought from Qingdao Guangyao Graphite Co. Ltd. Other chemicals were bought from Sigma-Aldrich. All chemicals were used without further purification. IR spectrum was recorded on a Thermo Fisher Nicolet IS 5 spectrometer (USA), in which samples were embedded in KBr thin films. Elemental analysis was performed by a Vario EL III element analyzer (Germany). Surface area was measured by an Autosorb-iQ-AG-MP gas sorption analyzer (USA). Scanning electron microscopy (SEM) images were taken using a HITACHI SU1510 scanning electron microscopy (Japan) with gold coating. Thermogravimetric analyses (TGA) were carried out on a Mettler-Toledo TGA/DSC 1/

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1100 SF instrument (Switzerland). Powder X-ray diffraction (XRD) was performed by a XRD-6100 (Japan).

2.2. Synthesis of GO

GO was prepared according to a literature [26]. IR spectrum is in accordance with that in the literature [21]. The FT-IR (KBr): $\nu = 3380$ (O–H), 1729 (C=O), 1592 (C=C), 1228 and 1067 (C–O) cm^{-1} . Elem. Anal. (GO) Found: C, 46.75; H, 2.80; O, 50.45.

2.3. Preparation of saturable absorber

GO (0.1 g) was added into deionized water (100 mL) to give a suspension. The suspension was dispersed using an ultrasonic apparatus for 6 h. Then, the dispersed suspension (0.4 mL) was dropped on a quartz sheet (2×2 cm). At 80 °C, the quartz coated by GO was dried under vacuum for 6 h.

2.4. Experimental setup of GO based Q-switching laser

The experimental setup of the GO based Q-switching laser is shown in Fig. 1. A fiber-coupled laser diode (LD) with a central wavelength of 808 nm was employed as the pump source. A Nd:YVO₄ crystal with the dimensions of $3 \times 3 \times 5$ mm (width \times height \times length) was used as the laser gain medium, which was cut along its a-axis. The laser crystal was wrapped in a piece of indium foil and mounted in a copper block cooled to 25 °C by a water cooling system. The pump light was focused in the crystal by using a matched achromatic doublet pair with the beam compression ratio of 1:1. The resonant cavity comprises a plane-concave mirror (M1) and a flat mirror (M2). The input mirror (M1) was high transmission (HT) at the pump wavelength and high reflection (HR) at around 1064 nm. The output mirror (M2) has a transmission of 5% at the lasing wavelength. The GO saturable absorber (SA) was inserted in the laser cavity close to M2.

3. Results and discussion

3.1. Structure of GO

3.1.1. Surface images and distances between two adjacent GO sheets

The SEM image and XRD spectrum of GO were shown in Fig. 2. GO exhibited an ordered layered structure (Fig. 2(a)). The surfaces of GO sheets were smooth. In XRD spectrum, there was a sharp diffraction peak located at $2\theta = 11.5^\circ$, corresponding to the interlayer distance of 0.77 nm. The peak of graphite located at $2\theta = 26.6^\circ$ was disappeared after it was oxidized. It suggested that graphite was already oxidized to form GO [27].

3.1.2. Thermodynamic stability

TGA curve of GO was shown in Fig. 3.

When temperature increased from 25 to 100 °C, GO lost 5.4 wt%

of the weight. It was attributed to evaporation of adsorbed water [28]. When temperature increased from 100 to 160 °C, GO lost 4.7 wt% of the weight. It was attributed to intramolecular dehydration. When temperature increased from 160 to 245 °C, GO lost 26.1 wt% of the weight. It was attributed to decarboxylation and dehydration [29]. When temperature increased from 245 to 800 °C, GO lost 17.7 wt% of the weight. It was attributed to breaking of carbon skeleton [30].

EA data of the GO and that in literature [25] were collected in Table 1.

In Table 1, percentage of oxygen (46.85 wt%) of the GO is higher than that (27.97 wt%) of GO which was prepared by a Hummers' method. Molar ratio of carbon to oxygen (1.33) of the GO is lower than that (2.25) in Hummers' paper. They would be attributed to the facts that H₃PO₄ and HNO₃ were used in the procedure of preparation of the GO to replace NaNO₃ and H₂SO₄ in the Hummers' method. Therefore, in this work, main oxygen-containing groups in the GO would be hydroxyl groups. The increase of oxygen percentage would improve electrochemical pseudocapacitor [31]. Therefore, the GO could be potential optoelectronic materials. Moreover, hydroxyl groups may improve hydrophilicity of materials, therefore, the GO would easily be dispersed in water to prepare laser devices.

3.1.3. FT-IR spectrum

The FT-IR spectrum of GO was shown in Fig. 4.

In Fig. 4, a strong and broad absorption peak located at 3380 cm^{-1} is O–H stretching vibration mode due to hydroxyl groups in alcohol. The peak located at 1729 cm^{-1} is stretching vibration of C=O in carboxyl group. The peak located at 1592 cm^{-1} is stretching vibration of C=C. The peaks located at 1228 and 1067 cm^{-1} are O–H in carboxyl group bending vibration and C–O–C absorption. It shows that hydroxyl groups are main oxygen-containing groups. The IR spectrum is in accordance with EA data.

3.2. Optical property

Using GO as the saturable absorber, we demonstrated a laser diode (LD) pumped passively Q-switched solid state laser. Typical characteristics of Q-switching pulses were obtained from the GO-based solid state laser at the pump power of 6 W (Fig. 5).

Without GO SA, the laser operated in a continuous wave (CW) regime. By optimizing the parameters of the resonant cavity, 1064 nm TEM₀₀ mode laser was obtained with power instability better than 5%. Then, the GO SA was placed close to the output mirror. The position of the GO SA was carefully adjusted to achieve mode matching with laser and resonator. The output characteristics in time domain were recorded with a fast response photodetector (Newport Model 818-BB-21) and a 500 MHz oscilloscope (Agilent Technologies MS07052B). The experimental results show that, when the pump power was increased to 3 W, the laser started to appear Q-switching laser operation. As the pump power increased,

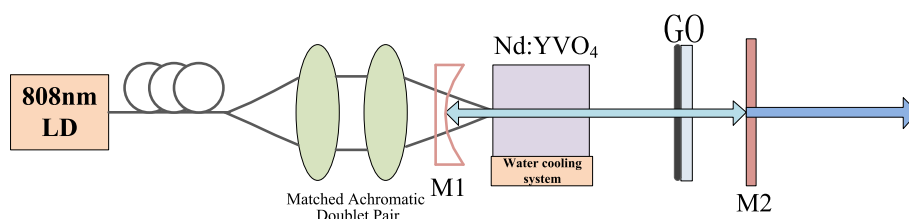


Fig. 1. Experimental setup of the GO based Q-switching laser.

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