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Liquid phase exfoliated black phosphorus and reduced graphene oxide polymer-based saturable absorbers fabrication using the droplet method for mode-locking applications

Rodrigo M. Gerosa [a], David Steinberg [a], Danilo A. Nagaoka [a], Juan D. Zapata [a,b], Sergio H. Domingues [a], Eunézio A. Thoroh de Souza [a], Lúcia A.M. Saito [a,*]

[a] MackGraphe – Graphene, Nanomaterials and Nanotechnology Research Center, Mackenzie Presbyterian University, Rua da Consolação, 896, São Paulo, SP 01302-907, Brazil
[b] Faculty of Engineering, Universidad de Antioquia, Cl. 67 #53 – 108, Medellin, Colombia

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ABSTRACT

We present the droplet method for liquid phase exfoliated reduced graphene oxide (r-GO) and black phosphorus (BP) saturable absorbers fabrication. By using this fast and powerful technique, nanomaterials/polymer-based films were directly transferred to optical fiber end-face and used as all-fiber saturable absorbers in an Erbium-doped fiber laser. As results, spectrum bandwidth of 7.30 nm and pulses as short as 570 fs could be generated, proving the reliability, practicability and robustness of this method for mode-locking applications.

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1. Introduction

The active field of mode-locked fiber lasers development for generating ultrashort pulses in the mid-infrared range has been currently a wide area for potential applications in research areas on industry, military, ultrafast science and optical telecommunications, because of their low cost, compactness and robustness. For this purpose, passively mode-locked (ML) fiber lasers have been one of the attractive technologies for ultrashort pulse generation through the nanomaterials saturable absorber (SA) incorporation.

During the past years, the SESAM (semiconductor saturable absorber mirror) had been the most common SA, widely used in industrial systems because of stable and self-starting mode-locking [1,2]. However, due to the main drawback of complicated and expensive manufacturing technology based on molecular beam epitaxy (MBE), which limits its optical bandwidth, the photonics community has searched for novel broadband nanomaterials suitable to use as SA. These include carbon-based nanomaterials such carbon nanotubes [3–6], two-dimensional (2D) graphene [4–12] and graphene derivate like graphene oxide (GO) and reduced graphene oxide (r-GO) [13–16], 2D-layered transition metal dichalcogenides (TMDs) [17–20], topological insulators (TI) [21,22] and black phosphorus (BP) [23–27].

There are three main fabrication procedures for obtaining 2D layered nanomaterials: mechanical exfoliation by using scotch-tape method [10], chemical vapor deposition (CVD) [9,11] epitaxy and liquid phase exfoliation (LPE) [8]. The first one involves micro mechanically cleavage of a bulk crystal for obtaining monolayer or few-layer flakes, although the number of 2D layered nanomaterial is statistical and the method is small scale production. The LPE and CVD are currently the most potentials methods for large-scale production, which is desirable for developing saturable absorbers and photonic devices commercially. Despite the CVD typically produces high-quality large-area flakes of some 2D-nanomaterials as graphene [12] and MoS_2 [17], for our knowledge, LPE is a universal and practical method, in which is capable to yield any nanomaterials with different sizes, areas and thicknesses coupled to a wide variety of solvents and polymers nanocomposites [8,13,14,17–19,22–27].

* Corresponding author.
E-mail address: lucia.saito@mackenzie.br (L.A.M. Saito).

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For fabricating 2D-nanomaterials SA devices and make them suitable for mode-locked fiber lasers, photonic crystal fiber, tapered fibers, D-shaped fibers and the most popular of them, optical fiber connectors, are the usual technology approaches, in which utilize the optical beam and evanescent field of the light to interact with the 2D nanomaterial SA. Unlike the CVD and mechanically 2D-layered nanomaterials SA which are fabricated by using fiber connectors [9,10] and evanescent field-based optical fibers [11], LPE is the only method that allows the large scale fabrication and deposition of any nanomaterials-based polymers/solvents into any type of fiber [8,13,14,17–19,22–27].

Although LPE is often reported as the main 2D-nanomaterial large-scale method for SA fabrication, all reported works presented numerous steps and longer time production of the nanomaterials with their complex transfer processes to the fiber. Therefore, in this work, we present the droplet method as a practical and reliable method for thin-films 2D nanomaterials SA fabrication [3]. Faster and robust, this requires simple steps of film production and direct transference into optical fiber connectors end-faces with any type of nanomaterials dispersions embedded in polymers. As all-fiber SA, liquid phase exfoliated black phosphorus (BP) and reduced graphene oxide (r-GO) were dispersed in an N-methyl pyrrolidone (NMP) solution, suspended with a urethane-based polymer and transferred directly to the optical fiber end-face connectors. When incorporated as SA in an EDFL cavity, spectrum bandwidths of 4.50 (BP) and 7.30 nm (r-GO) could be generated, resulting in pulse durations as shorter as 650 (BP) and 570 fs (r-GO), proving the simply, robustness and efficiency of this method in manufacturing any 2D-nanomaterial polymer-based samples for mode-locking applications.

2. Materials and methods

2.1. 2D-nanomaterials preparation

The r-GO powder were prepared by a modified Hummers method and dispersed in NMP. The final product in this step is a multilayer r-GO. In the same method, BP was exfoliated in NMP under an inert environment, N_2 (g) flow, using an ultrasound bath. Both dispersions had a final concentration of 2 mg/mL. These dispersions were suspended with a commercial UV curable optical adhesive based on polydimethylsiloxane (PDMS) (NOA73TH; refractive index of 1.56 and 90% of transmittance @ 1550 nm) at 1:3 proportion. For homogeneous dispersion, the suspensions were placed into an ultrasonic bath for 10 min.

For 2D-nanomaterial SA all-fiber sample fabrication, the droplet method was employed [4], as shown in Fig. 1. It consists of collecting a droplet of the 2D-nanomaterial/polymer suspension with a micro-tip and transferring it to the surface of optical fiber connector end-face. A 1-μm diameter micro-tip was dipped into the 2D-nanomaterial/polymer suspension to induce the formation of a micro-droplet and fixed to one triaxial translation stage orthogonally to a second triaxial stage attaching an optical fiber connector. As the optical fiber connector end-face touched the micro-tip, the micro-droplet was directly transferred, resulting in the ~20 μm thick-film formation [4], which was subsequently cured with UV radiation. By attaching the fiber connector containing the deposited film in-line with another one, the 2D-nanomaterials SA were obtained.

The main advantages of this technique are the shorter time manufacturing and its practicability. The total time of all method steps, including the 2D-nanomaterial/polymer suspension preparation (about 10 min), the micro-droplet collection and its deposition into the optical fiber connector end-face (about 1 min) and the film curing by UV radiation exposure (about 10 min) was less than 30 min, which results in a fast all-fiber SA sample fabrication without any complex film transference process. Due to the direct deposition and thin film thickness, there is no need of its manipulation or transference, and prevents the film mechanical damage from back-to-back fiber connector end-faces.

2.2. Optical images, Raman spectroscopy and nonlinear characterization

In Fig. 2a and b are shown the optical microscopes images of the deposited 2D-nanomaterial/polymer film into the fiber connector ferrule and the film distribution on the cladding/core areas, respectively, with some agglomerates of 2D-nanomaterials around the illuminated core area of the fiber. By using an optical backscatter reflectometer equipment, we measured the film thicknesses about ~20 μm for both r-GO and BP samples, very close to our previous reports [4,6].

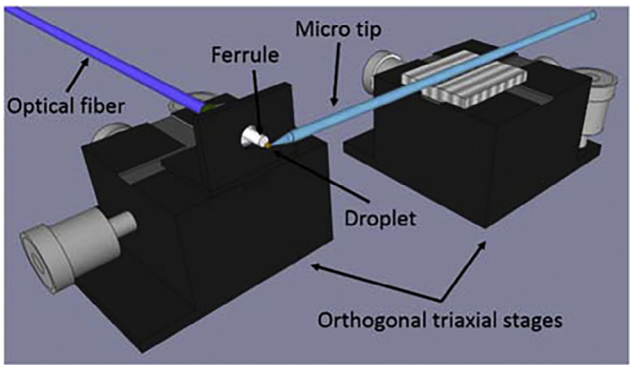

Fig. 1. Droplet method experimental setup.

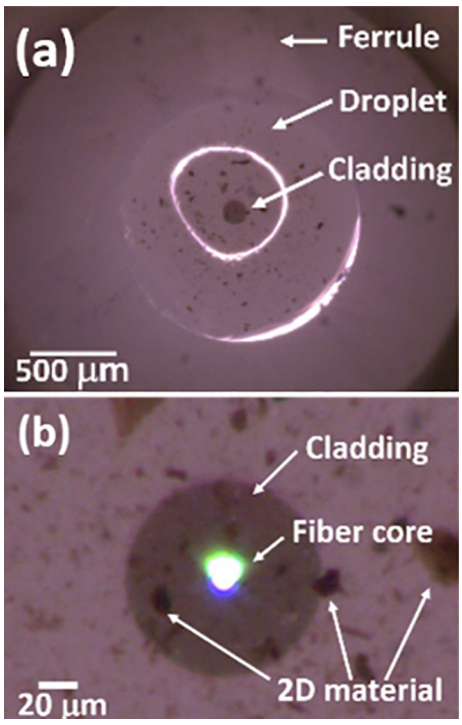

Fig. 2. Optical images of 2D-nanomaterial/polymer droplet at (a) ferrule and (b) cladding/core areas.

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