Title: Printing of tunable diffractive optical elements on graphene oxide thin-film using femtosecond laser induced photoreduction

Author(s): Lim, Joel Chin Huat; Low, Mun Ji; Murukeshan, Vadakke Matham; Kim, Young-Jin


Date: 2018

URL: http://hdl.handle.net/10220/45686

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PRINTING OF TUNABLE DIFFRACTIVE OPTICAL ELEMENTS ON GRAPHENE OXIDE THIN-FILM USING FEMTOSECOND LASER INDUCED PHOTOREDUCTION

CHIN HUAT JOEL LIM, MUN JI LOW, VADAKKE MATHAM MURUKESHAN, YOUNG-JIN KIM
Singapore Centre for 3D Printing, School of Mechanical and Aerospace Engineering, Nanyang Technological University, 50 Nanyang Avenue, 639798, Singapore

ABSTRACT: We present diffractive optical elements printed on graphene oxide (GO) thin film using femtosecond (fs) laser induced photoreduction process. Graphene oxide is an interesting advanced material as its optical and electrical properties change after laser induced photoreduction to become reduced graphene oxide (rGO). rGO is opaque with good electrical conductivity. Femtosecond laser enables printing of sub-micrometer diffractive optical structure on rGO films which can be used transmission grating and Fresnel lens. The physical dimensions of diffractive optical elements can be tuned by transferring the rGO-GO patterns to a dielectric elastomer actuator (DEA). rGO played a dual role being a compliant electrode to the DEA and as an optical element with low transmittance. Highly transparent DEA substrate ~ tens of μm thickness was used for printing of the desired structures. The diffractive optical elements undergo compression or expansion varied to the designed rGO pattern as the DEA substrate stretched when a voltage is applied. The ease of the printing process and the arbitrary patterning capability of Fs laser induce photoreduction allows the fabrication of highly efficient ultrathin tunable optical components.

KEYWORDS: Direct laser writing, reduced graphene oxide, photoreduction, micro-optics, dielectric elastomer actuator (DEA)

INTRODUCTION

Femtosecond (fs) laser induced photoreduction is a new and emerging process in the nanofabrication field due to its high precision and accuracy in the nanometer scale. The technology involving this process can be grouped Femtosecond Laser Direct Writing (FsLDW). The printing of arbitrary pattern enables a large range of electrical, optical, and optoelectronic applications (An et al. (2017), Zhang et al. (2014)) It does not require masking which is being used in conventional process such as lithography (Zhang et al. (2010)) or specialized grating elements as in interferometric lithography (Sreekanth et al. (2010), Murukeshan et al. (2008)). The usage of a femtosecond laser source as the light source in manufacturing is becoming a common sight due to its high peak power allowing it to process many materials which were not possible before using conventional lasers (Dantus (2017), Malinauskas et al. (2016)).

Graphene Oxide (GO) is an interesting transparent nanomaterial due to its ability to change its property to that of graphene through a variety of reduction mechanisms. The reduction form of GO is called reduced graphene oxide (rGO). Using a laser source, the photon energy of the laser pulse removes the oxygen-containing group by photoreduction through both photothermal and photochemical process. GO is a non-conductive material whereas rGO is highly electrically conductive. GO’s transmittance is also drastically reduced during the photoreduction process. GO
can be easily coated for large area fabrication through simple drop casting. By using FsLDW to pattern and design rGO area to be of low transmittance, many interesting diffractive optical applications such as Fresnel lens or transmission grating can be patterned using rGO-GO or rGO-substrate interface. Due to the ultrathin thickness of rGO, ultrathin 2D optics element can be produced and future tuned by adjusting the parameters of FsLDW to change the properties of rGO.

Dimensions of the fabricated optical elements pattern can be further tuned by transferring to a dielectric elastomer actuator (DEA). DEA is a compliant capacitor in which a soft electroactive material is sandwiched between two compliant electrodes and can deform in under electric field due to an electromechanical pressure between the electrodes. Silicon-based polymers such as polydimethylsiloxane (PDMS) and acrylic based polymers such as VHB tape (from 3M) are commonly used electroactive materials in DEA applications (O’Halloran et al. (2008)). Incidentally, these materials are also transparent which are used in optical applications. rGO plays a dual role due to its high electrical conductivity property as it can be used as the electrodes’ material in DEA application. By controlling the voltage, the grating period can be tuned.

![Figure 1. (a) Schematic illustration for the operational principle of DEA (b) Example of an rGO diaphragm shaped dielectric elastomer actuator](image)

**EXPERIMENTAL SETUPS AND CHARACTERIZATION**

**Experimental Configuration for FsLDW**

The GO films were photo-reduced with femtosecond laser pulses of 515 nm wavelength generated at a Yb-doped fiber femtosecond laser (Amplitude Systèmes, Satsuma HP). A pulse picker was used to control the pulse’s repetition rate. The output repetition rate of laser was set to 500 kHz with pulse duration of 220 fs. FsLDW was carried out using a galvano scanner with an f-theta lens. The scan speed was fixed to 100 mm/s and hatch spacing of 10 μm. The set-up of the fabrication space is shown in Figure 2.
Sample Preparation

Acrylic-based VHB 4905 was stretched by 300% and a supporting frame was used to maintain the stretch. 0.5 mg/ml DI water-based GO solution was drop-casted on the substrate and the water solvent was evaporated leaving a thin GO film. Optical pattern structures such as Fresnel lens, diffraction grating or electrode pad for DEA were fabricated using FsLDW. Excess GO can be removed by soaking the sample in DI water and through light brushing. For the patterning on the back surface, the same process can be applied or transparent conductive polymer such as PEDOT:PSS can be spin-coated to form a compliant electrode. In DEA application, the electrodes are connected to a high voltage supply.

Figure 3. Schematic illustration of sample preparation of rGO-VHB grating through drop-casting method and FsLDW
RESULTS AND DISCUSSIONS

rGO as a Compliant Electrode Material

PEDOT:PSS and rGO were investigated for their suitability as compliant electrode materials. The maximum radial strains of a diaphragm-shaped actuator with electrodes fabricated using the two different materials were measured. Also, Carbon grease also is investigated as it is a well-tested compliant electrode material and is usually used in DEA applications. A maximum strain of 44.6% was achieved using carbon grease–carbon grease electrodes. PEDOT:PSS is a transparent conductive polymer which has diffractive optical applications. PEDOT:PSS–rGO is able to achieve 16.7% strain.

![Figure 4. Maximum radial stain achieved for various electrode combination on VHB 4905 (pre-stretch 300%) of a diaphragm shape actuator.](image)

Fabrication of rGO Diffractive Optical Components using FsLDW

FsLDW was used to fabricate various diffractive optical components for different applications. rGO-GO Fresnel lens of 1 mm diameter in size was fabricated. Minimum width of the ring can be as low as 10 µm. A rGO-VHB 1D transmission diffraction grating was also fabricated. It has a grating period of 20 µm with an individual grating linewidth of around 8 µm. The rGO grating pattern was fabricated on VHB to allow the period of grating to be tunable using DEA. Using 638 nm wavelength laser as the light source (laser diode fiber coupled from Thorlabs), the diffraction of light could be observed up to the 12th order. The diffracted beams maintained its Gaussian shape as seen in the 3D intensity profile of the 0th and 1st order beam in Figure 5e.
Figure 5. (a) Optical image of an rGO-GO Fresnel lens of diameter 1mm (b) Zoom-in optical image of rGO-GO Fresnel lens (c) Optical image of an rGO-VHB 1D transmission diffractive grating with period 20 μm (d) Camera captured image of the maximum order when 638 nm light pass through rGO-VHB grating (e) 3D intensity profile of 0th and 1st order diffraction beams when 638 nm light pass through rGO-VHB grating.

CONCLUSIONS

This paper proposed an illustration of a femtosecond laser induced photoreduction process of GO, which is used to fabricate different 2D diffractive optical components such as Fresnel lens and diffractive grating. It is demonstrated that DEA could be used in conjunction to tune the grating period. These tunable diffractive optics have various applications such as phase or frequency modulation and focal length control. Furthermore, rGO has demonstrated its usages in the fabrication of optical patterns and as a compliant electrode for DEA. It is envisaged that the result presented in this paper can lead to the realization of 3D printed grating structure such as waveguides.

ACKNOWLEDGMENTS

This study was supported under the research collaboration agreement by Panasonic Factory Solutions Asia Pacific (PFSAP) and Singapore Centre for 3D Printing (SC3DP) (RCA-15/027).

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