IN-SITU LASER REDUCTION OF GRAPHENE OXIDE ON TEXTILES: FROM LAB-SCALE OPTIMIZATION TO ROLL-TO-ROLL MANUFACTURING

Natalia Kantouni,[1,2*] Chrysanthi Paparouni[3], Despoina I. Batsouli,[3] and Spyros N. Yannopoulos [1,2*]

[1] Foundation for Research and Technology – Hellas, Institute of Chemical Engineering Sciences (FORTH/ICE–HT), GR-26504, Rio-Patras, Greece

[2] Department of Chemistry, University of Patras, GR-26504, Rio-Patras, Greece

[3] Adamant Composites Ltd., Aghias Lavras and Stadiou St., GR-26504, Rio-Patras, Greece

* Correspondence at: kantouni@iceht.forth.gr & sny@iceht.forth.gr

BACKGROUND

- The integration of graphene into textiles is a vital component for developing next-generation wearable electronics, energy storage, and smart fabrics.
- The laser-assisted method for graphene oxide reduction is considered a green and versatile technique to acquire graphene-like structures with high quality and enhanced conductivity.
- The ease of fabrication, cost efficiency and environmental sustainability of this method, aligns perfectly with the needs of the market. Offering a great alternative to traditional synthesis routes, that lack on the scalability aspect.

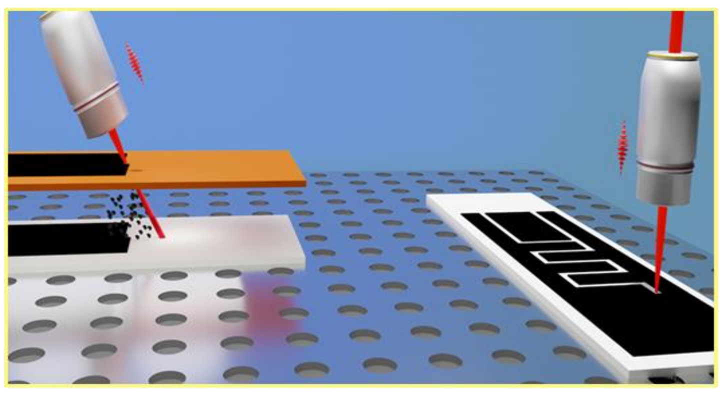
OBJECTIVES

- To establish an eco-friendly laser-assisted method for **in-situ graphene deposition** onto flexible substrates.
- Investigation of the effect of varying GO loadings on the conductivity of the laser irradiated samples.
- Comprehensive characterization of the graphene embedded textiles employing **Raman Spectroscopy**, **Scanning Electron Microscopy** and **Sheet Resistance** measurements to assess the structural, morphological and electrical changes of the prepared samples, in respect to different laser power treatments.
- Extend the study to diverse textile substrates and utilize an Nd:YAG laser of the **roll-to-roll pilot line**, to further assess the scalability and versatility of the proposed manufacturing procedure.

LASER-ASSISTED GRAPHENE REDUCTION ON TEXTILE

Methodology

Laser- assisted synthesis of reduced Graphene Oxide (GO)



A simple spraying and a brushing method were employed to evenly coat the graphene oxide dispersion on a thin polyester textile substrate.

Followingly, various samples with GO loadings ranging from 0.5 to 3.1 mg cm^{-2} were prepared to evaluate the effect of loading on the conductivity of the laser irradiated samples.

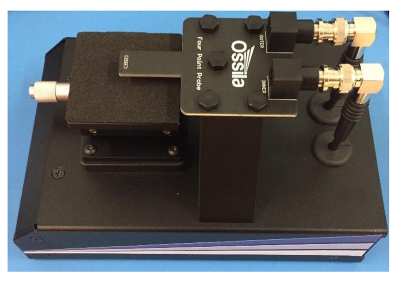
- A CO_2 (10.6 μm) industrial type laser was employed to irradiate and reduce the GO coating.
- The produced materials were thoroughly characterized to identify the optimum loading and laser conditions to attain the lowest sheet resistance (R_s).

Effect of GO loading on conductivity

Material	R_s (Ohm sq^{-1})	Ref.
GO on Nylon/spandex	87.6 ± 36.2	1
GO on Nylon filtration membrane	51 ± 2	2
GO on Cellulose Acetate membrane	58 ± 3	2
GO on Cellulose Acetate membrane Nitrocellulose	620 ± 40	2
GO on PET/textile	45	3
GO on PES (loading 1 mg cm^{-2})	8.1 ± 0.8	Our work

Table 1 : Comparison of recent literature R_s values with our work.

Four Point Probe for Sheet Resistance measurements



Raman Spectrometer

Conductivity optimization: Influence of the laser power

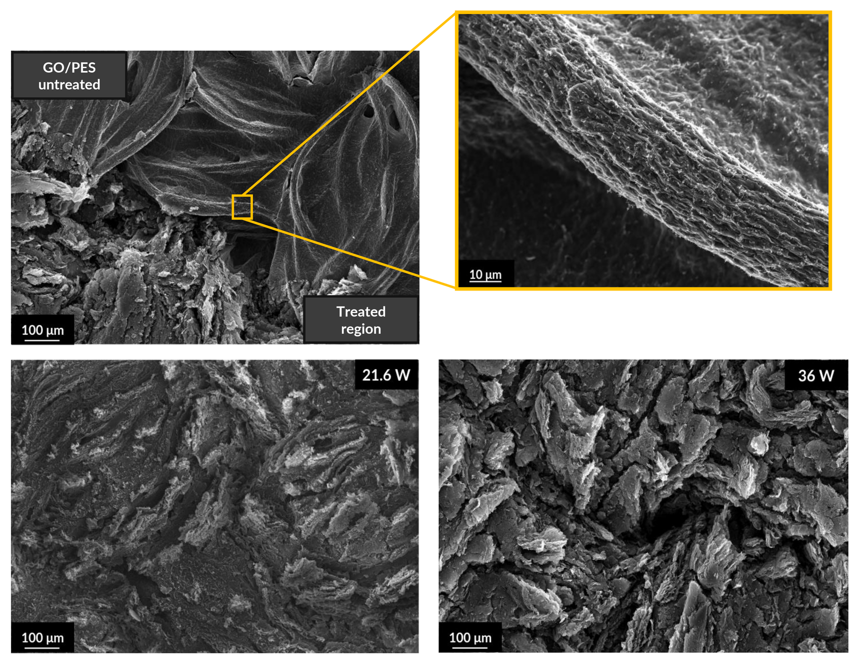


Fig. 1: SEM images of the bare GO and the LrGO coated textiles, laser treated with powers of 21.6 and 36 W respectively.

Laser Power (W)	Sheet resistance (Ohm sq^{-1})
18	52.1 ± 1.3
21.6	35 ±0.7
25.2	13.8 ± 1.0
28.8	10.1 ±0.1
32.4	9.9 ±0.4
36	12.3 ± 3.5
39.6	10.7 ± 2.6
43.2	18.1 ±1.0

Table 2: R_s values in respect to the laser power employed to reduce GO films on PES.

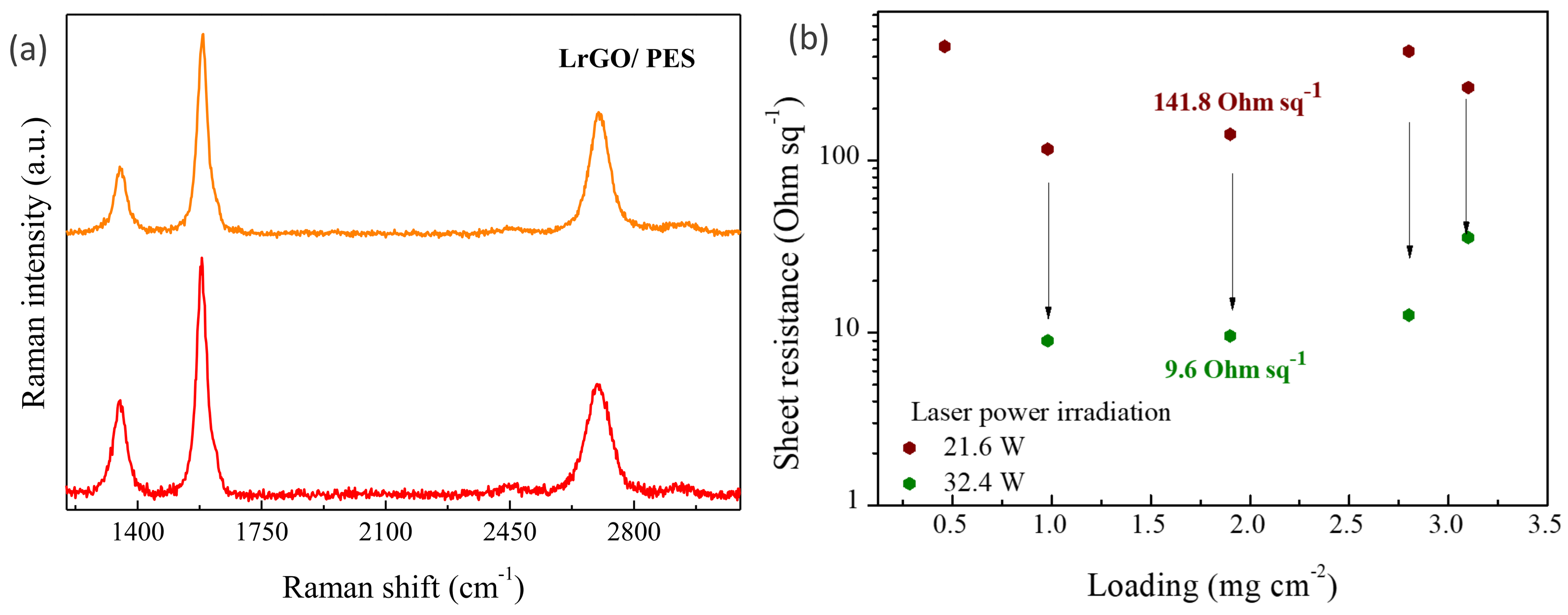


Fig. 2: (a) Representative Raman spectra of the LrGO, produced by different laser powers (red: 18 W and orange: 23.1W) (b) Sheet resistance versus loading plot of the various samples.

UPSCALING TO A ROLL-TO-ROLL (R2R) PROCESS

Methodology

- An airbrush method was used to uniformly coat GO on polyester (PES) ,polyamide (PA) and prepreg textiles.
- A fiber-optic-coupled Nd:YAG laser integrated within an R2R pilot line (ADA) was utilized to produce LrGO at large scale.

Fig. 3: (a) Image of the R2R pilot line (ADA), LrGO spots employing 4,6 and 8 J on (b) PA and (c)PES textiles.

Raman Spectroscopy

- All GO coatings were successfully reduced, independent of the substrate employed.
- In all cases, high quality graphene material with high crystallinity was acquired.
- Among all energies, the 6 J value seems to provide the best reduction across all samples.

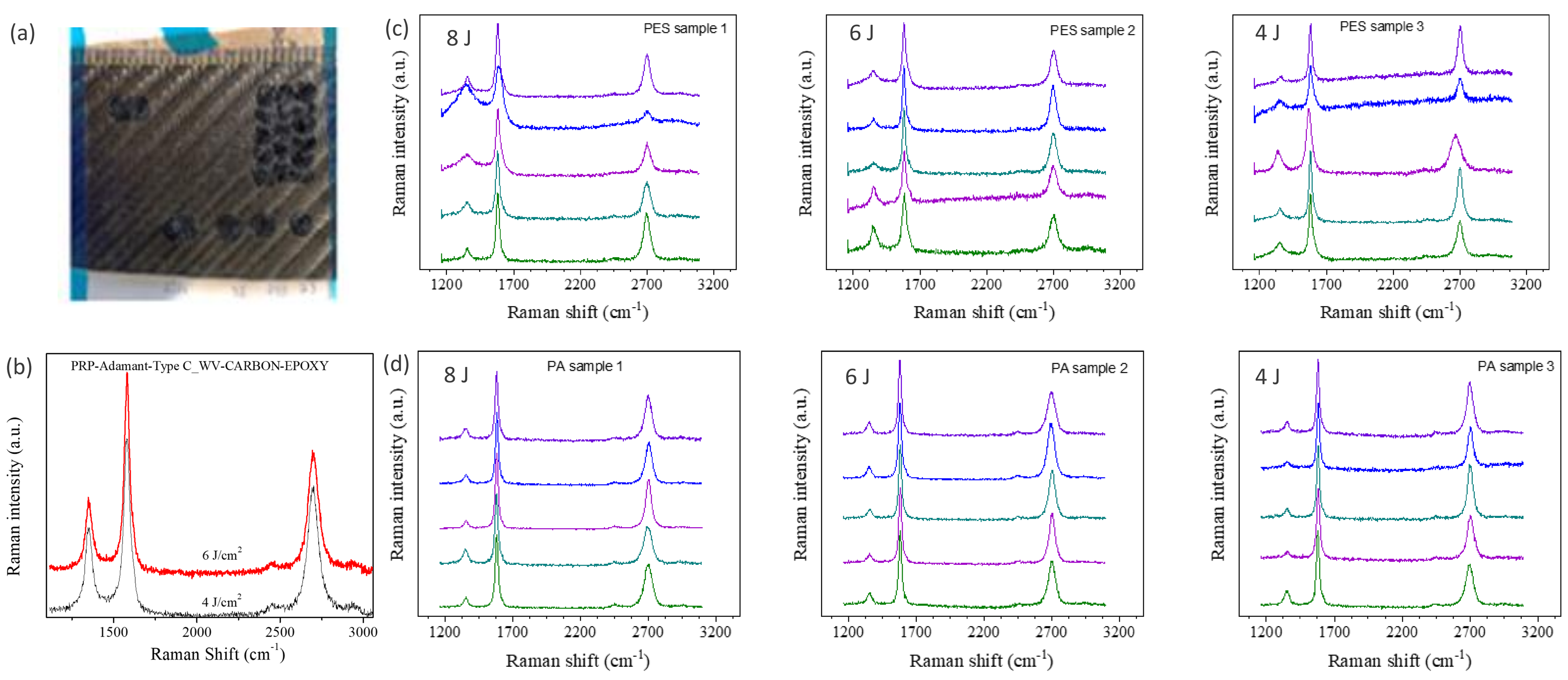


Fig. 4: (a) Image of the prepreg textile with LrGO spots, Raman spectra of the LrGO produced by diverse laser energies on (b) prepreg, (c) polyester and (d) polyamide textile substrates.

REFERENCES

1 Lipovka, A. *et al.* Textile Electronics with Laser-Induced Graphene/Polymer Hybrid Fibers. *ACS Appl. Mater. Interfaces* **15**, 38946–38955 (2023).
2 Bonando, M. G. *et al.* The impact of different flexible substrates on the photothermal reduction quality of graphene oxide. *Nanoscale Adv.* **6**, 4604–4610 (2024).
3 Fatkullin, M. *et al.* Smart Graphene Textiles for Biopotential Monitoring: Laser-Tailored Electrochemical Property Enhancement. *ACS Sens.* **9**, 1809–1819 (2024).

Funded by the European Union