Graphite Oxide Reduction to Graphene Applying Ultrashort Laser Pulses

Romualdas Trusovas[1], Gediminas Račiukaitis[1], Jurgis Barkauskas[2], Regina Mažeikienė[3]

[1]Center for Physical Sciences and Technology, Savanoriu 231, LT-02300 Vilnius, Lithuania
romualdas.trusovas@ar.fi.lt
[2]Faculty of Chemistry, Vilnius University, Naugarduko 24, LT-03225 Vilnius, Lithuania
[3]Institute of Chemistry, Center for Physical Sciences and Technology, A. Gostauto 9, LT-01108 Vilnius, Lithuania

Graphene can be produced by mechanical exfoliation, chemical exfoliation, chemical synthesis, pyrolysis, epitaxial growth, CVD and other methods [1]. There is still need in novel graphene production methods which are suitable for mass production and can avoid usage of harmful chemical materials. Chemical, thermal and light reduction methods are implied for graphite oxide (GO) reduction. Recently there were described several methods of producing graphene based on laser-induced GO reduction [2-5].

Samples prepared, using modified Hummers-Otieman method were used in our experiments. Congo red (CR) dye was used as an additive. The GO coatings were prepared on the polycarbonate membrane filters via slow filtration into alkaline media. The samples were treated using a picosecond laser (Atlantic, 10 ps, 100 kHz, Ekspla) with the scanner setup working at 1064 nm wavelength. During the tests, the average laser power and scanning speed was varied. Experiments were conducted in air, nitrogen and argon atmospheres. Laser treated samples were investigated with scanning electron microscope and Raman spectroscopy (Fig. 1 a,b). Morphology modifications appeared in laser irradiated areas. Raman spectra revealed formation of graphene by emerging of the 2D-line. Ratios of the Raman line intensities (I_D/I_G, I_{2D}/I_G) were found to be dependent on the CR concentration. Variation in the intensity ratios related to quality of the resulting graphene film indicates importance of the CR concentration on linkage of graphene sheets together and formation larger blocks with increase in stacking order. Simulation of transient temperature inside the GO film during and after pulsed laser irradiation was performed using COMSOL Multiphysics software. Simulation revealed that the temperature above 1000 ^{0}C, which is necessary to remove most carboxyl, hydroxyl and epoxy groups from GO [6] was achieved at the optimal laser beam scanning conditions.

Irradiation of GO films with the picosecond laser created significant changes in material properties. We found that in our experiments the figure of merit of GO reduction to graphene was position and width of Raman lines as a function of a product of the pulse energy and the irradiation dose. Results of laser treatment depended on the Congo red dye concentration in GO. Optimal scanning irradiation dose – 50 μJ corresponds to the effective thermal reduction temperature of 1400 K.

References

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Figures

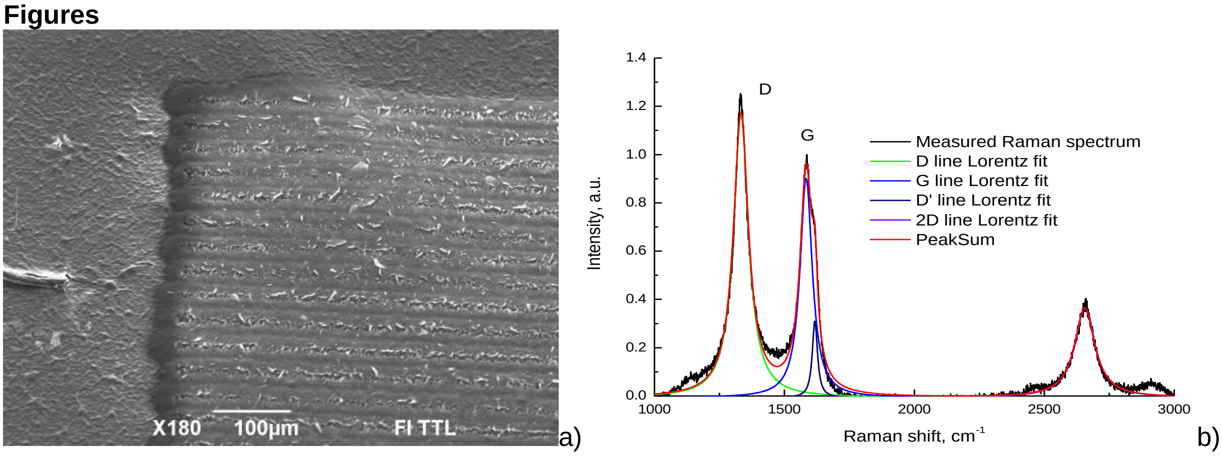

*Fig. 1 SEM picture of the laser scribed lines in GO film a); Raman spectrum of laser treated area in the GO film b) Process parameters: 50 μJ irradiation dose, laser treatment in nitrogen atmosphere; Sample: aqueous GO suspension, concentration. 1.5*10^{-4} g/ml; filtrated to 0.1 M KOH, PC substrate (pore d=0.4 µm) doped with Congo Red dye.*