Phase tuned, highly conductive graphene by ultra-short laser irradiation of PEEK

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Laser-induced graphene (LIG) is an emerging technique for direct writing of 3D porous conductive patterns on selected polymer materials when exposed by a laser beam. An ultrashort laser system can trigger remarkable phenomena in materials due to its extremely short pulse duration. This work reports interesting findings in direct laser writing of conductive patterns during femtosecond laser-polymer interactions. Two different interaction regimes, one with an sp^3 carbon dominant phase (regime 1) and one with a sp^2 dominant phase (regime 2), both of which are electrically conductive, are observed. During Regime I, ultra-short laser pulses create a highly conductive layer on 50 µm thick flexible polyether ether ketone (PEEK) sheet with strong adhesion to the PEEK sheet; the sheet resistance of laser treated PEEK surface reduced to 9.60 Ω/□ at 270 $mJcm^{-2}$ laser fluence. In this regime I, for all investigated fluences, we could not obtain the characteristic Raman peaks of graphene. High resolution XPS analysis confirms that PEEK has been modified to carbon phase with sp^3 being more dominant than sp^2. Even though the sp^2 bonding component was not dominant, this regime reported the lowest sheet resistance corresponding to excellent electrical conductivity.

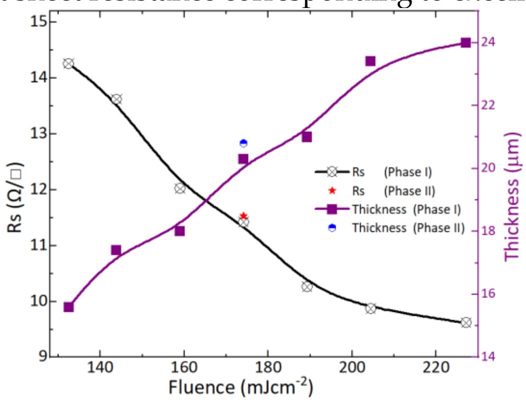

Figure 1: Variation in sheet resistance and thickness of laser generated carbon/graphene on the PEEK surface with applied laser fluences.

At a higher fluence than applied in regime I, the PEEK surface can be converted to sp^2 at the expense of surface damage which is less useful for practical applications. Therefore, in regime II, PEEK surface was laser scanned multiple times at a low fluence of 131 $mJcm^{-2}$ first and then at a fluence of 172 $mJcm^{-2}$ to convert it into sp^2 graphene without causing any damage and affect to adhesion with the underlying PEEK surface. The sp^2 dominated phase was confirmed from Raman and XPS in this fluence regime. The corresponding sheet resistance of phase II was 11.53 Ω/□ which corresponds to 4.17×10^3 S/m electrical conductivity. The sheet resistance is slightly higher compared to that obtained in regime I, but here in regime II, a conversion of sp^3 to sp^2 carbon and an increase in C−C content is a confirmation of laser graphitization of the PEEK film. Additionally, for Regime II, at lower investigated fluence (131 $mJcm^{-2}$), we collected the ablated nanoparticles. SEM analysis suggests that in ultra-short laser interaction with PEEK, non-melted spherical nanoparticles were emitted during the laser irradiation. The light brown color of emitted nanoparticles is the same as that of PEEK sheet revealing that these nanoparticles were produced in a non-thermal process. It is important to highlight that the first high conductive regime with such high conductivity and dominant sp^3 is not observed with longer pulse durations like CO_2 laser graphene writing. This reported low fluence femtosecond laser process is highly relevant for direct writing of various conductive structures for electrical and biomedical sensing applications.