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Dry cleaning of polymeric residues from graphene with high density hydrogen plasma: the issue of plasma purity

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Graphene consists of two exposed sp2-hybridized carbon surfaces and has no bulk. Therefore, graphene surface contamination by adsorbed polymer residues have a critical influence on its electrical properties and can drastically hamper its widespread use in device fabrication. Therefore, graphene-based technology requires "soft" and selective surface cleaning process to suppress this surface contamination. However, polymeric contamination is resistant to cleaning due to p-stacking and is problematic because it originates from typical technological processes used to fabricate graphene devices. Since solvents are not efficient to clean these residues, other strategies based on reactive plasmas have been proposed. Here, we investigated a high density H2 plasma cleaning process of graphene monolayer in an industrial ICP plasma reactor designed to etch 300mm diameter wafers. Firstly, we show that there is a considerable issue associated with the use of H2 plasmas to treat graphene and other 2D materials: H atoms and H3+ ions reduce the surface of all the materials exposed to the plasma, which include the reactor walls and the substrate holder (i.e. the 300-mm diameter wafer on which the graphene sample is stuck). As a result, metallic and O atoms are released in the H2 plasma, resulting respectively in graphene metallic contamination and damages, Si stick on graphene while O atoms etch it spontaneously. We investigated various coating of the reactor walls to prevent this phenomenon. We concluded that the only solution to get rid of parasitic O is to use a wafer holder made of Aluminum and to fully fluorinated the reactor walls and the wafer with a F rich plasma prior the H2 process. Under such controlled conditions, we show that H2 plasmas can provide an infinite etching selectivity between sp2 and sp3 hybridized form of carbon, i.e. H2 plasma can clean polymer residues from graphene. The quality of the cleaning is characterized by various surface diagnostic techniques, including k-PEEM to measure its band structure. We show that the cleaned graphene lattice remains undamaged by H2 high density ICP plasma. This dry-cleaning has the advantage to be an industrially mature technology adapted to large area substrates and to other 2D materials.

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