



# Atmospheric pressure plasma treatment on graphene grown by chemical vapor deposition



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## ARTICLE INFO

### Article history:

Received 8 January 2015

Received in revised form

2 February 2015

Accepted 9 February 2015

Available online 16 February 2015

### Keywords:

Atmospheric pressure

Plasma

Graphene

Doping

Chemical vapor deposition

## ABSTRACT

We demonstrate the surface treatment of graphene using an atmospheric pressure plasma jet (APPJ) system. The graphene was synthesized by a thermal chemical vapor deposition with methane gas. A Mo foil and a SiO<sub>2</sub> wafer covered by Ni films were employed to synthesize monolayer and mixed-layered graphene, respectively. The home-built APPJ system was ignited using nitrogen gas to functionalize the graphene surface, and we studied the effect of different treatment times and interdistance between the plasma jet and the graphene surface. After the APPJ treatment, the hydrophobic character of graphene surface changed to hydrophilic. We found that the change is due to the formation of functionalities such as hydroxyl and carboxyl groups. Furthermore, it is worth noting that the nitrogen plasma treatment induced charge doping on graphene, and the pyridinic nitrogen component in the X-ray photoelectron spectroscopy spectrum was significantly enhanced. We conclude that the atmospheric pressure plasma treatment enables controlling the graphene properties without introducing surface defects.

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## 1. Introduction

The 2-D carbon nanomaterial, graphene, has attracted much attention due to its remarkable physical properties and potential applications in many fields [1–3]. In particular, the properties of graphene are governed and can be controlled by the number of layers, edge structures, and chemical doping [4–7].

There have been many efforts to control or enhance the graphene properties by structural modifications or chemical doping with foreign elements achieved through thermal treatment [8–11]. The controllable etching of graphene by thermal oxidation at 750–800 °C was reported as a suitable way to fabricate graphene nanoribbons (GNRs) of few nanometers [8]. Campos-Delgado et al. demonstrated the formation of a defect-free GNRs through graphitization by post-annealing at 1500–2800 °C [9]. Chemical doping by thermal treatment at relatively high temperatures was also employed to control the electrical properties of graphene [10,11]. However, it is very difficult to use thermal treatments to control with high precision both the nanoscale structure and the doping level of graphene. Furthermore, the inherent high

temperature and time-consuming character of these approaches limit their applicability on an industrial scale.

The plasma treatment was introduced as an alternative to overcome these issues, since the plasma process can be operated at room temperature, and the treatment time can be shortened owing to the highly active species present in the plasma. For these reasons, control and enhancement of the graphene properties by plasma treatment have recently been reported [4,12–16]. The fabrication of GNRs [12] and layer-by-layer etching of multi-layered graphene by sequential plasma and thermal treatments were also reported [4,13]. In the case of plasma doping of graphene, substitutional doping by foreign atoms such as nitrogen or oxygen was achieved by plasma treatment using various gases at room temperature for a short time [14–16]. However, plasmas were easily ignited and had to be maintained at low pressure due to the requirement of a long mean free path of electrons [17]. This means that, in spite of the advantages of the plasma treatment such as short processing times and low temperatures, the necessity of a vacuum chamber and a pumping system represents a drawback which limits large-scale applications and continuous processing.

Recently, the atmospheric pressure plasma treatment has attracted great attention as one of the most promising alternatives to address these problems [18–23]. Since this treatment is

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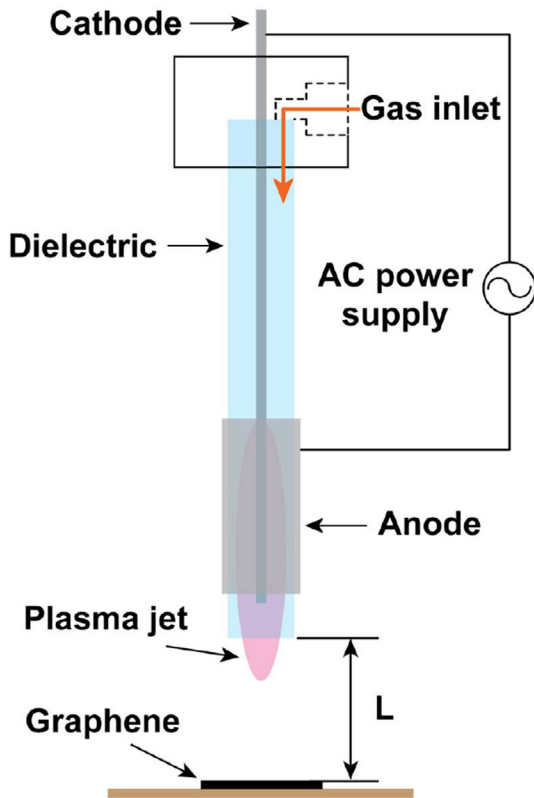


Fig. 1. Schematic illustration of the atmospheric pressure plasma jet system.

performed at atmospheric pressure and room temperature, and is not affected by the chamber size, it enables large-scale and continuous surface treatment of various materials. The atmospheric pressure plasma treatment has been mainly applied for the sterilization of viruses or bacteria [18,19]. It has also been utilized for surface modification of heat-sensitive materials such as polymers, plastic films, and textiles [20,21]. Surface modification of carbon nanomaterials is being actively investigated. For example, the reduction of graphene oxide and the formation of surface functional groups on carbon nanotubes (CNT) using atmospheric pressure plasmas were reported [22,23].

Here, we present the surface modification of graphene using an atmospheric pressure plasma jet (APPJ) system. The graphene was prepared using a thermal chemical vapor deposition (TCVD) with methane feedstock. The home-built APPJ system was constructed with a high-voltage AC power supply. After the nitrogen plasma treatment, we observed the formation of functional groups on the graphene surface, as well as charge doping on graphene.

## 2. Experimental section

Mono-layered and mixed films of mono-, few-, and multi-layered graphene were synthesized to comparatively investigate the effect of the plasma treatment. As catalyst layers, a 200 nm-thick Ni layer deposited on Mo foil and a 300 nm-thick Ni layer deposited on SiO<sub>2</sub> wafer were employed for the synthesis of monolayer and mixed-layered graphene, respectively. The graphene synthesis was carried out at 1000 °C for 5 min using mixtures of CH<sub>4</sub> (5 standard cubic centimeters per minute, sccm), Ar (500 sccm), and H<sub>2</sub> (500 sccm) gases. The synthesized graphene films were transferred by an etching method onto a 300 nm-thick SiO<sub>2</sub>-covered Si wafer for plasma treatment and characterization.

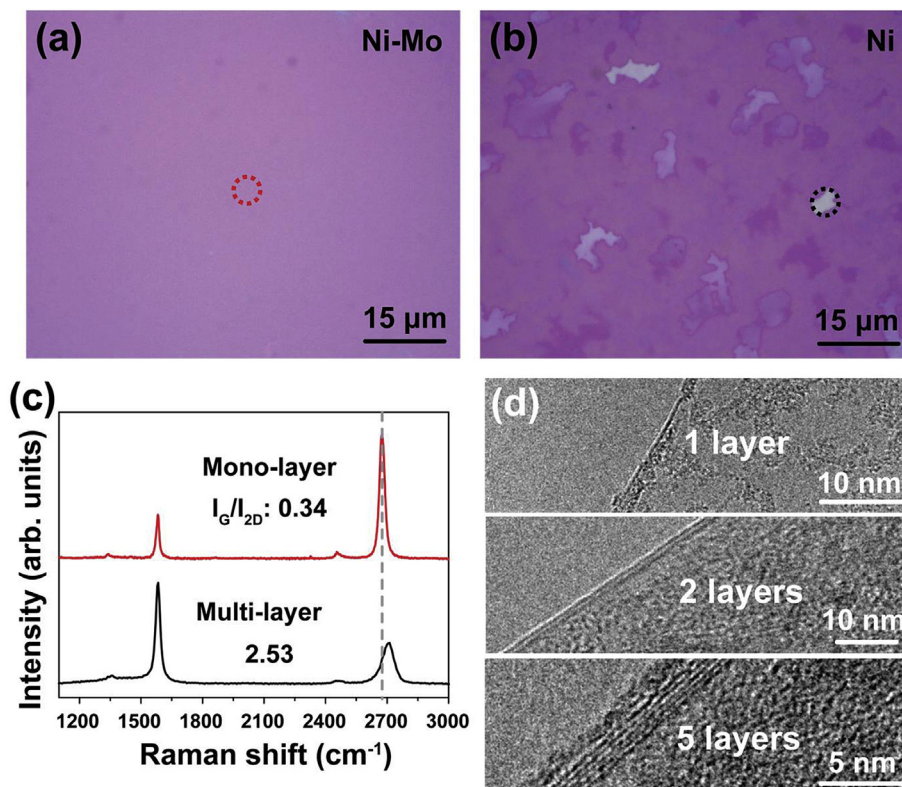


Fig. 2. Optical microscope images of transferred (a) mono-layered graphene and (b) mixed graphene films on SiO<sub>2</sub>-covered Si wafer. (c) Raman spectra recorded in the regions indicated as dotted circles in panels (a) and (b). (d) TEM images of mono-, bi-, and five-layered graphenes.

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