OBTAINING OF GRAPHENE BY CHEMICAL VAPOR DEPOSITION

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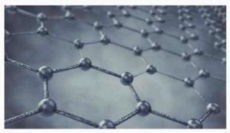


A new stage of graphene development began with obtaining of single-layer and two-layer samples in 2004, when the scientists, by multiple using of an adhesive tape, separated a monolayer of graphene from the bulk graphite and transferred it to the silicon substrate with oxide of 300 nm thickness. After demonstration of the unique electronic properties of graphene in these works, a rapid development of research of this material and the development of new methods for its production began. We use the chemical vapor deposition

Methods for obtaining graphene

The method of obtaining graphene is divided into several categories: *detached graphene; chemical graphene; epitaxial graphene*on metals or on SiC; chemical vapor deposition (CVD) graphene on nickel or on copper.

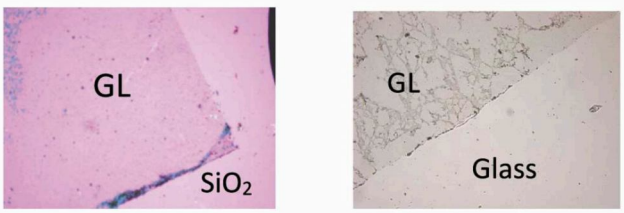
In order to obtain **detached graphene** from a plate of bulk well-oriented pyrolytic graphite by means of an adhesive tape multiple using, it is possible to separate the film into a single monolayer, which can be transferred to another substrate.

Chemically graphene is obtained either by restoration of graphite oxide or by liquid-phase stratification of graphite.

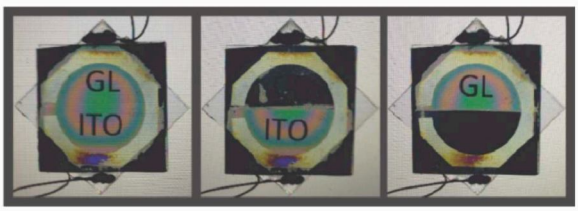
When **graphene is grown epitaxially** on the metal surfaceat a temperature exceeding 1000 °C, the metal saturates as a result of the chemical deposition of carbon from the gas phase. In high or ultrahigh vacuum, when the substrate temperature decreases, the solubility of carbon in metal significantly decreases, and due to the thermal compression of crystal grating, carbon appears on the surface, forming graphene domains of large area.

At 900 °C, a graphite film with thickness of ~400Å can be formed on metal substrate by **Chemical Vapor Deposition method**. In a mixture of carbon-containing gas, hydrogen and argon at various pressures, gas decomposition takes place during heating. With increase in temperature starting from 650 °C, the carbon atoms are deposited on a nickel substrate and at temperatures above 800 °C they begin to diffuse into the volume of nickel. The heating stops at temperatures 950-1000 °C and then, when the sample is cooled to room temperature, the crystal grating of the metal squeezes out carbon atoms to the surface where they form a graphite-like structure.

The formation of agraphene film on the surface of copper polycrystalline substrate is different. Since the solubility of carbon in copper is about 1000 times less than in nickel, then after the decomposition of carbon-containing gas and the deposition of carbon on the copper surface, diffusion into volume does not occur. As the temperature of the copper substrate increases, both the probability of graphene film formation and the covered area increase. In this case, the formation of multilayer graphene sheets is impossible on copper, since copper is a catalyst in the deposition of carbon. When the graphene monolayer is coated with a copper surface, the formation of subsequent layers becomes very unlikely. Today this method is used for a large-scale production of graphene.



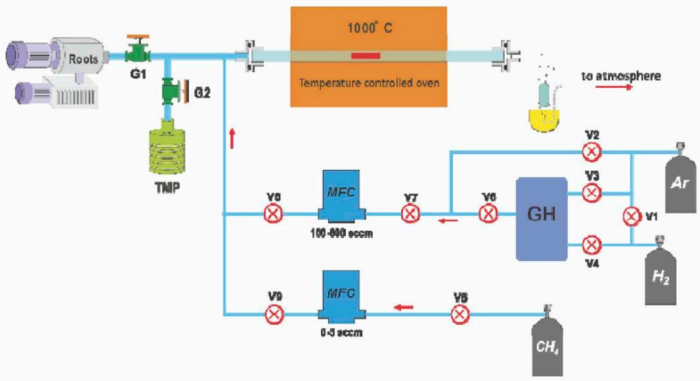
Micro-images of the graphene-layer



Images of the Graphene-ITO hybrid LC cell, obtained between crossed polarizers: voltage not applied (a),voltage applied to LC cell with graphene electrodes (b), voltage applied to LC cell with ITO electrodes (c).

The device description and experiment

The installation consists of three main parts - a thermal reactor, a vacuum system and a gas distribution system.



The reactor is a quartz tube of 22 mm diameter, placed in a heat furnace. The furnace is installed on the base, which smoothly moves along the rails for withdrawal of the substrate- catalyst from the hot zone (synthesis zone) in order to cool the substrate after the end of the process. The temperature of the hot zone is controlled by FU-72 temperature controller in the range of 10-1200 °C with an accuracy of ±0.5°C. At the outlet of the reactor, a bubbler is installed to exclude backflow from the atmosphere into the quartz tube and neutralize the exhausted gas mixture before discharge to the atmosphere.

The vacuum system consists of a Roots pump and a turbomolecular pump designed to clean the quartz tube and provide vacuum in the reactor.

The gas distribution system consists of a tank for gases mixing with a reducer, gas pipes, a valve system and two programmable gas flow regulators. The control of gas flow regulators and vacuum valves (V1-V9) is carried out by specially created software in the LabVIEW medium.

A technological process for graphene obtaining on copper foil has been also developed. After chemical cleaning and electropolishing, the copper foil is placed in a quartz tube and dried in a stream of argon. At that the chamber is heated uniformly to a synthesis temperature of 1000 °C at a rate 20 °C/min in a stream of argon at a flow rate 300 N/cm3/min. When the temperature reaches 1000 °C, the copper foil is annealed within 10 minutes.

After the copper foil annealing, the **process of graphene deposition** begins, which lasts for 20 minutes. For this mixture of argon in hydrogen Ar:H2 (80sccm) and methane CH4 (1sccm) is supplied into the chamber through MFCs. The heat anneals the copper increasing its domain size. The hydrogen catalyzes a reaction between methane and the surface of the Cu substrate.

Then the furnace is abruptly moved along the rails in order to remove copper foil from the hot reaction zone.

Once the graphene/copper foil has been removed from the furnace and cooled the graphene layer can be transferred an arbitrary substrate.

Conclusion

A device for graphene obtaining by CVD method is developed and assembled. A liquid crystal cell consisting of two sections with various transparent electrodes is made: graphene and ITO. The work of the obtained cell as an optical valve is shown, both sections of the cell function identically. Graphene can be successfully used as a transparent electrode in optical elements based on LC.

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