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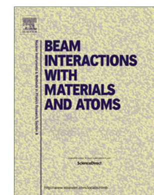

Irradiation of graphene field effect transistors with highly charged ions

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ABSTRACT

In this work, graphene field-effect transistors are used to detect defects due to irradiation with slow, highly charged ions. In order to avoid contamination effects, a dedicated ultra-high vacuum set up has been designed and installed for the in situ cleaning and electrical characterization of graphene field-effect transistors during irradiation. To investigate the electrical and structural modifications of irradiated graphene field-effect transistors, their transfer characteristics as well as the corresponding Raman spectra are analyzed as a function of ion fluence for two different charge states. The irradiation experiments show a decreasing mobility with increasing fluences. The mobility reduction scales with the potential energy of the ions. In comparison to Raman spectroscopy, the transport properties of graphene show an extremely high sensitivity with respect to ion irradiation: a significant drop of the mobility is observed already at fluences below 15 ions/μm^2, which is more than one order of magnitude lower than what is required for Raman spectroscopy.

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1. Introduction

The irradiation with energetic ions may be used to obtain permanent modifications of surfaces [1–6,5,7–11] or so-called 2D materials such as graphene, carbon nanomembranes, MoS_2, and hexagonal BN [4,12–19]. For 2D materials in particular, slow highly charged ion (HCI) beams seem to be the appropriate tool as these projectiles deposit their potential energy, i.e. the sum of the ionization energies of the missing electrons, in a very small volume close to the surface. However, despite ongoing research in this field [1,20,2,21,17], the underlying interaction mechanisms are still not very well understood, in particular with respect to 2D materials. In the case of HCI it is rather difficult to distinguish between the influence of the key parameters, i.e. the kinetic energy E_{kin} and the potential energy E_{pot}, as a complete deceleration of the ions is not feasible [22]. One method is to keep one parameter fixed and vary the other to find a threshold value for a given modification. With respect to graphene, such a threshold has already been determined by atomic force microscopy (AFM) [17], while this has yet to be done with Raman spectroscopy. Both methods are very powerful tools to investigate irradiation effects in graphene but have their disadvantages as well. The AFM may detect individual impact sites on the order of one per μm^2, provided they are large enough. Raman spectroscopy on the other hand is sensitive to the smallest defects [23–25], but is only effective at relatively high fluences, starting from 10^{10} ions/cm^2 (corresponding to 100 ions/μm^2) onwards. In addition, most experiments are performed ex situ under ambient conditions, i.e. the samples have to be taken out of the irradiation chamber and as a consequence undergo additional, uncontrolled changes. In this paper we present an alternative method to estimate ion-induced radiation damage, which is sensitive in a fluence regime that bridges the two aforementioned methods. We use field-effect transistors (FET) made from single layer graphene [26,27]. We show that by analyzing the transfer characteristics of graphene FETs (G-FETs) before and after irradiation, conclusions with respect to ion-induced defects in graphene may be drawn.

2. Experiment

2.1. Experiment

For this experiment, single layer graphene was mechanically exfoliated onto oxidized (285 nm SiO_2), degenerately p-doped Si wafers. Metallic contacts were provided by photolithography and vacuum evaporation of a 5 nm thick Ti adhesion layer and a 100 nm thick gold layer. The graphene quality is checked by µ-Raman spectroscopy (ReniShaw, $\lambda = 532$ nm, $P = 1$ mW) before and after photolithography, and after irradiation as well (see Section 3.1). Fig. 1 shows a schematic sketch of our devices with the typical channel length and width of $L = 5$ µm and $W = 5$ µm, respectively. In this configuration, the degenerately doped Si works as a global backgate (U_{GS}), whereas the gold contacts are used as

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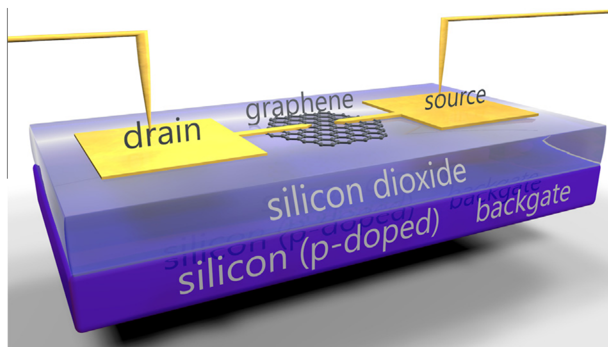

Fig. 1. Schematic sketch of a graphene field-effect transistor (G-FET).

both the drain and the source contact. The transfer characteristic $I_{DS}(U_{GS})$ reflecting the electrical properties of the G-FETs is measured before, during and after irradiation with HCIs. The current I_{DS} between drain and source is driven by a constant potential difference U_{DS}. In order to calculate the electron and hole mobility, respectively, the following equation can be used [28]

$$\mu_{\pm} = \sigma \frac{L}{W C_i U_{DS}}. \quad (1)$$

The differential conductance is given by $\sigma = \frac{\partial I_{DS}}{\partial U_{GS}}$ and the sheet capacitance $C_i = \frac{\epsilon_0 \epsilon}{d} = 1.212 \cdot 10^{-8}$ F/cm^2, with $\epsilon = 3.9$ [28] being the dielectric constant of SiO_2 with a thickness of $d = 285$ nm. Additionally, the charge carrier density can be calculated by [29]

$$n = \frac{C_i}{e}(U_{Dirac} - U_{GS}) \quad (2)$$

where e is the elementary charge and U_{Dirac} is the voltage, at which the Dirac point (characterized by $n \approx p \approx 0$, i.e. minimum conductivity) is observed.

In order to be able to measure the transfer characteristic during irradiation with HCI, a dedicated ultra high vacuum (UHV) set up for the G-FETs was built. To this end, the G-FETs were bonded onto a custom-made, UHV compatible chip carrier. Furthermore, the G-FETs were cleaned by heating at around $T = 150$ °C before irradiation to partly remove intercalated water and residual photoresist (see Section 4.1) stemming from the preparation procedure [30,31]. All samples were irradiated using the Duisburg HCI beamline HICS [32], which is based on an electron beam ion trap (EBIT) that can produce ^{129}Xe ions with a charge state q from 1+ to 45+ and a corresponding maximum potential energy of about e.g. $E_{pot} = 58.8\ keV$. The kinetic energy $E_{kin} = q(U_{acc} - U_{dec})$ can be controlled via a deceleration stage and is given by the charge state of the ion q, the acceleration voltage U_{acc}, and the deceleration voltage U_{dec}. The charge state selection is done with a dipole magnet, whereas a multi-stage lens system ensures a focused ion beam with a spot size of less than 1 mm^2 diameter. A Faraday-Cup is used to measure the beam current.

In previous experiments, the threshold charge state for ion-induced modifications of graphene was found to be $^{129}Xe^{30+}$ (corresponding to a potential energy of $E_{pot} \approx 15$ keV) at a kinetic energy of $E_{kin} \approx 260$ keV [17]. Therefore, we have chosen two charge states, one below and one above this threshold, namely $^{129}Xe^{25+}$ ($E_{pot} \approx 8$ keV) and $^{129}Xe^{32+}$ ($E_{pot} \approx 19$ keV), while keeping the kinetic energy fixed at $E_{kin} \approx 200$ keV. For both charge states, the transfer characteristic is measured as a function of the ion fluence in the range of 0 to about 2500 ions/cm^2. The back-gate-voltage is varied from $U_{GSmin} = -20$ V to $U_{GSmax} = +20$ V, while the drain-source-voltage is held constant at $U_{DS} = 50$ mV for all measurements.

3. Results

3.1. Raman-spectra

Raman spectroscopy can be used as a non-destructive method to analyze the structural quality of graphene and in particular, the existence, density and type of defects [33–36]. A typical Raman spectrum of exfoliated, high-quality graphene shows two prominent peaks [24,25]: The in-plane vibrational G mode at $\approx$1580 cm^{-1} and the 2D mode at $\approx$2690 cm^{-1}. The full width at half maximum (FWHM) of the 2D peak can be used to determine the layer thickness. A FWHM $\approx$ 25 cm^{-1} indicates single layer graphene, whereas a FWHM $\approx$ 52 cm^{-1} is typically found for bilayer graphene [37]. The 2D mode is sometimes also called the second-order in-plane overtone of the D mode vibration. The presence of the first order D mode at $\approx$ 1350 cm^{-1} is indicative for lattice defects or disorder, respectively [38]. The D mode as well as the D' mode at $\approx$1620 cm^{-1} can be used for defect analysis [39,33,34]. In Fig. 2 Raman spectra from our samples are shown, the first taken from pristine graphene (black line), the second from graphene after photolithography (blue line), and finally from irradiated graphene (red line). The FWHM of the 2D mode of pristine graphene (black line) is about $\approx$ 27 cm^{-1} and thus indicates single layer graphene. The absence of the D-Peak (black line) proves the high quality of the graphene flakes after exfoliation [40].

For a quantitative comparison of irradiation-induced disorder, we analyze the area ratios of the D and G mode, A_D/A_G [41,33]. Already due to the photolithography process (blue line) this ratio typically increases up to $A_D/A_G = 0.3$. After irradiation with $^{129}Xe^{32+}$ ions (>2500 ions/μm^2), an area ratio of $A_D/A_G = 3.5$ is measured (red line). Note, that in order to guarantee a sufficient signal/noise ratio of the relevant Raman peaks, graphene has to be irradiated with a fluence of at least 100–500 ions/μm^2, depending on the charge state.

4. Electrical characterization before irradiation

Without cleaning, the G-FETs fabricated as described above, are typically p-doped [26]. This becomes apparent in the transfer characteristic $I_{DS}(U_{GS})$ measurements, shown in Fig. 3a, where the Dirac-point is shifted to a high, positive backgate voltage. After the cleaning process, which consists of heating the sample in

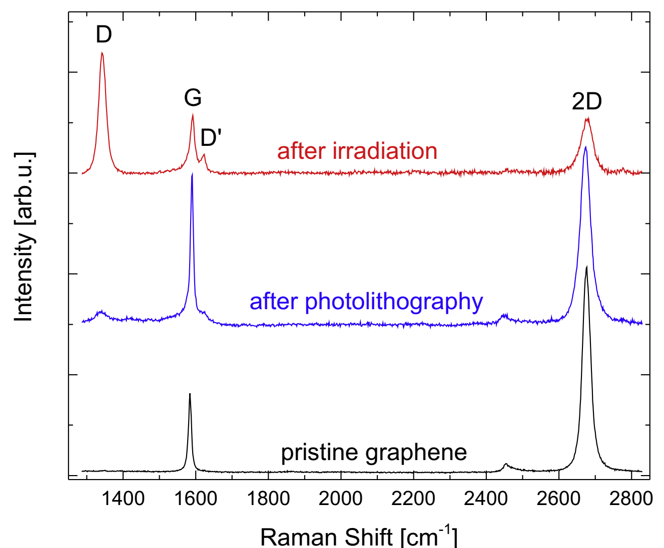

Fig. 2. Typical Raman-spectra from pristine graphene (black line), after photolithography (blue line) and after irradiation with HCIs (red Line). The different Raman modes are marked. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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