Transfer and weighing of graphene flakes by using a nanowire mass sensor

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Abstract

This work demonstrates an experimental method for the transfer and characterization of individual graphene flakes *in situ*. The great advantage of this method is preventing of environmental impact during the preparation of samples for future experiments.
The experimental setup includes 13D SmarAct nanomanipulation system staged inside a Hitachi 4800 scanning electron microscope (SEM). The nanomanipulation system SmarAct preforms precise moving of its actuators with possible step size of *100nm*. The scanning electron microscope enables the visualization of the experiment. The externally connected signal generator *AGILENT N9310A* operates in the output frequency range of 9 kHz-3 GHz with resolution of 0.1 Hz.
Nanowires turned out to be suitable for shaping nanoelectromechanical mass sensor, because of their flexibility and high resonant frequencies. Ge nanowires, due to easy synthesis, monocrystalline structure and high Young's modulus already have been applied in nanoelectromechanical switches [1]. A germanium nanowire is integrated into the nanomanipulation system as an active element (Fig. 1a, top image). Germanium nanowires are glued onto sharp electrochemically etched Au tips with conductive epoxy. To prevent oxidation of Ge nanowires, they are etched with Ar ions for 120 s using etching/coating equipment *Gatan PECS 682*. The self-resonance frequency of the nanowire is pre-measured by applying AC field sweep to the nanowire and observing the nanowire's behavior in SEM. This frequency depends on nanowire's shape, mass and Young's modulus. When the frequency of AC field fits nanowire's self-resonance, rapid increase in nanowire's oscillation amplitude is observed in SEM images (Fig. 1a, bottom image). Exact resonant frequency and resonator quality factor are determined from the nanowire`s deflection amplitude distribution. The resonant frequencies of the unloaded nanowires are in the range of 100 kHz, typical quality factors are from 100 up to 500. The Young's modulus of Ge nanowires, calculated from resonant frequencies turned out to be about 70-120GPa that is close to the Young's modulus for bulk material [2].
After the self-resonance frequency of the nanowire is determined, the nanowire is used for graphene flake characterization and transferring to a substrate. Graphene samples are produced by splitting few layer thick flakes from the surface of highly oriented pyrolytic graphite.
The transfer of a graphene flake is based on balancing of the adhesion interaction between the surfaces involved in the process. Due to the high adhesion between the germanium nanowire and graphitic structures, partially detached graphene flake can be picked up from the graphite surface by the free end of a single-clamped nanowire (Figure 1b,c). The free surface energy of germanium (1.5 J/m^2 [3]) is higher than that of graphite (0.1-0.2 J/m^2 [4]). Therefore a graphene flake whose contact area with the graphite surface has been decreased might adhere to germanium nanowire on a single position and stay there. The nanowire-graphene contact area relates to the nanowire thickness. In comparison, for graphene flake with surface area of 1um^2, placed onto the end of the nanowire with radius of 50nm, the contact area is less than 1% of flake's surface area. Loading the nanowire with graphene sample causes its resonant frequency decrease. The mass of the loading graphene flake can be calculated from the resonant frequency shift between the not loaded and loaded nanowire as following [5]:

$$m = \frac{3m_0}{\beta_1^4}\left(\left(\frac{f_0}{f}\right)^2 - 1\right),$$

where m_0 is initial mass of nanowire, β=1.875 is an appropriate constant for the first harmonic frequency equation, f_0 and f are the resonant frequencies for the not loaded and loaded nanowire. When the mass of the flake is known, the number of graphene layers in it can be calculated as following:

$$N = \frac{m}{A \cdot \rho},$$

where *A* is the surface area of the flake, $\rho = 0.76 \cdot 10^{-6}$ kg/m^2 is the graphene mass density. Shape of the flake is determined from SEM pictures (Figure 1c,d).

After the weighing procedure the graphene flake with determined mass and number of layers is transferred to the desired position on the substrate (Figure 1d).The flake transfer from the nanowire to the substrate is performed by varying contact areas and contacting materials, thus balancing adhesion interaction. Other factors that facilitate the manipulations with graphene flakes are also investigated.
The nanowire mass sensor operation affecting factors are explored and discussed in this work. Presented controlled characterization transfer of graphene allows the fabrication of nanodevice prototypes under not changing laboratory conditions. This minimizes the chance of change of sample properties due to the environmental influence. The next step of our research is to realize controlled nanowire-based transport of a single layer graphene samples.

References

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Figures

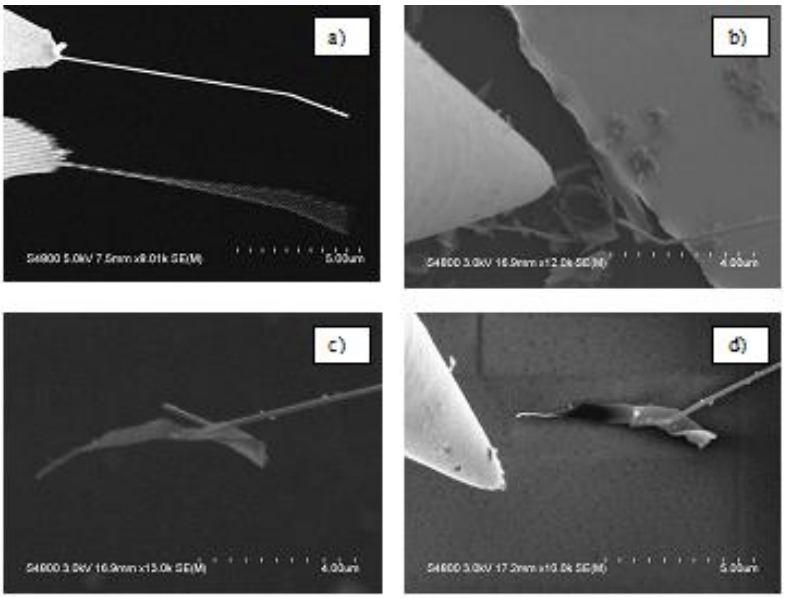

Figure 1: a) a single-clamped germanium nanowire (top image) and the nanowire at resonant frequency (bottom image); b) graphene flake in contact with the nanowire; c) graphene flake picked up by the end of the nanowire; d) transfer of the graphene flake to the Si substrate.