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Precise mass detector based on "W needle – C nanowire" nanomechanical system

S Y Lukashenko^{1,2,*}, F E Komissarenko^{1,3}, I S Mukhin³, V V Lysak¹, D A Averkiev⁴, I D Sapozhnikov² and A O Golubok^{1,2}

¹ Department of Nanotechnology and Material Science, ITMO University, Kronverskiy av. 49, 197101, St. Petersburg, Russia

² Institute for Analytical Instrumentation of RAS, Rizhsky pr 26, St. Petersburg,

190103. Russia

³ Academic University, Russian Academy of Sciences, ul. Khlopina 8/3, 194021, St. Petersburg, Russia

⁴ Department of computer photonics and digital video processing, ITMO University, Kronverskiy av. 49, 197101, St. Petersburg, Russia

Abstract. Nanomechanical system (NMS) based on amorphous carbon nanowhiskers localized on the top of tungsten tip were fabricated and investigated. The whiskers were grown in the scanning electron microscope (SEM) chamber using focused electron beam technique. The manipulation of SiO₂ and TiO₂ nanospheres was provided in SEM by means of dielectrophoretic force. Oscillation trajectories and amplitude-frequency characteristic of the oscillator were visualized at low pressure using a scanning electron microscope. The estimation of mass sensitivity of NMS was conducted.

1. Introduction

Nanomechanical oscillators (NMO) with small mass and volume are perspective sensitive elements for ultralow physical values sensors [1-3]. NMO's are particularly interesting as they show quantum properties at low temperatures and high resonance frequencies, though having macro- sizes in comparison with atoms [4].

NMO is a perspective mass sensor, which can be used for weightening of single cell or detection of biochemical reactions [5]. For using NMOs at atmospheric pressure, it is important to keep their Qfactor on high enough level. Works [6,7] were dedicated to studying NMO behaviour at atmospheric pressure.

State-of-the-art NMOs though having a number of advantages, have significantly lower Q-factor in comparison with macroscopic mechanical oscillators. It is important to find optimal construction of nanomechanical system (NMS).

This work presents the investigation of NMS "W needle - C nanowire". This system consists of single amorphous carbon nanowire, localized on a tungsten tip. This system can be also used for

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creation of specialized sensors based on local interaction probe sensors of scanning probe microscopes.

2. Experiment

We grow the amorphous carbon nanowire (CNW) on the top of sharp W tip with ~100 nm radius. CNW was made from precursor gas using focused electron beam induced deposition (FEBID) technique [8] inside the chamber of scanning electron microscope (SEM) Inspect S50 (FEI, USA). The oscillation were excited by applying of the sinusoidal signal from high-frequency generator to a piezoactuator 'Figure 1'. Measuring the amplitude-frequency characteristics (AFC) of as-grown structures was provided by using the SEM trajectory visualization method [6]. NMO with diameter (50-200) nm and length (1-3) μ m 'Figure 2a' were created depend on growing parameters. W tips were fabricated from polycrystalline tungsten wire with base diameter of 150 μ m using the electrochemical etching method in 5% alkali solution. Using W tip provides simple and convenient manipulation of the single CNW to excite and register the resonant frequencies.



Figure 1. Experimental setup for the C nanowire growth and AFC measurement at low pressure. (1) piezotube, (2) W tip, (3) C nanowire, (4) micromanipulator, (5) base, (6) electron beam, (7) focused lens, (8) secondary electron detector, (9) high-frequency generator.

Ultrasonic wave propagates through W wire and excites CNW oscillations perpendicularly to electron beam axis. In this case, the sharp W needle concentrates the energy to the fixed end of the oscillator. SEM images of the CNW oscillations are obtained by means of secondary electron detector. The accelerating voltage of electron column was 20 kV. Mechanical fluctuations of the CNW blur the image on the SEM monitor. The amplitude of the blurred image has maximum on the resonance frequency of CNW.



Figure 2. SEM images of carbon nanowire (CNW) on tungsten tip (a); the resonance of this CNW at 3,68 MHz.

The resonant frequency of grown C nanowire was 3,68 MHz, on the 'Figure 2b' one can see the blurred image carbon nanowire on the tungsten tip at resonance.

 SiO_2 and TiO_2 nanospheres (350 nm diameter and 100 nm, respectively) were manipulated with piezomanipulator (Kleindiek, Germany) in SEM chamber. First of all SiO_2 and TiO_2 nanospheres were landed on a silicon substrate. Then nanospheres were captured with the tip, installed on piezomanipulator with the help of dielectrophoretical force as described in [10]:

$$F_D = \int_V (D - \varepsilon_0 E) \nabla E \, \mathrm{dV},$$

where D – electrical induction, E – electric field, ϵ – the dialectric constant.

Influenced by the electron beam, the ungrounded metal tip was negatively charged. This metal tip produced the field, so dielectric nanosphere placed near this tip was polarized and attracted to the negatively charged tip. In particular, when the W needle approaches at a distance of ~ 300 nm to the nanosphere it jumps from substrate on the top of the needle. On the second stage, the nanospheres were moved to the apex of nanowire 'Figure 3a'. Attaching the nanospheres on the CNW was produced by a short (about 1 sec) local deposition of carbon at the point of contact between nanosphere and the C nanowire 'Figure 3b'. After that, the manipulator moved aside and nanosphere kept at the top of the NMO 'Figure 3c'.





The resonant frequency of "W needle - C nanowire" nanomechanical system was measured again after fixing of the particle on the CNW 'Figure 4'.



Figure 4. Electronic images of "W needle – C nanowire – SiO₂ nanospere" system (a); the resonance of CNW wih attached mass at 2,58 MHz (b).

Estimation of Nanospheres Mass

In order to estimate the attached mass, we use a simple equation that describes the relation of the increased oscillator mass ΔM and resonant frequency shift $\Delta \omega$ [11]:

 $\frac{\Delta\omega}{\omega} = \frac{\Delta M}{2M_{eff}}$, where M_{eff} - effective mass (for cantilevered beams $M_{eff} = \frac{33}{140}M_{NMO}$), ω - resonant frequency of initial nanowire. $M_{NMO} = \pi r_{NMO}^2 * l * \rho_C$, where $\rho_C = 2.2 g/cm^3$ - density of amorphous carbon, r_{NMO} - radius of the NMO, *l*- length of the NMO. The second formula for estimation of the attached mass is general relation between density and volume $\Delta M = V\rho = \frac{4}{3}\pi r^3\rho$, where r – radius of nanospheres and the density is $\rho_{SiO_2} = 2.4 g/cm^3$, $\rho_{TiO_2} = 3.9 g/cm^3$ for SiO₂ and TiO₂ nanospheres accordingly.

All SiO₂ spheres were the same sizes with diameter of 350 nm, the mass was about $\Delta M = V * \rho = 31 * 10^{-15}(g)$ using density and volume. The estimation via relation between frequency shift and attached mass is $M_{SiO_2} = 26fg$.

The mass of quartz nanospheres is too big for accurate estimation of mass sensitivity for our nanomechanical system. Therefore, we also manipulated with TiO_2 spheres with diameter of 100 - 120 nm. In this case, we measured resonant frequencies after fixing four TiO_2 nanospheres sequentially on the apex of CNW 'Figure 5'.



Figure 5. Series of electronic images of CNW resonances after sequentially addition of four TiO₂ nanoparticles.

The average mass of TiO₂ nanosheres $M_{TiO_2} = 3.5 \pm 0.5 fg$. The approximation error of calculation is 5-15%. Figure 6' shows response of nanomechanical system on attaching four TiO₂ nanoparticles. The initial resonance frequency of the nanooscillator was 13,42 MHz.



Figure 6. Graph of nanooscillator resonant frequency versus attached mass.

The results show good correlation between calculation of attached mass via classic linear approximation using the resonant frequency shift and calculation using volume and density of nanosheres. These results show a high sensitivity for the mass measurement. In recent work [9], we showed that at low pressure the Q-factor of our system is about 100. In particular, by setting for the smallest shift frequency ~ 50 kHz we get value about ~ 10^{-16} g that is the minimum detectable mass of concerned "W needle – C nanowire" nanomechanical system.

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