Nanoobject mass measurement using the node displacement of the second mode of the nanomechanical resonator

N A Solomonov1, K N Novikova1, I V Nadoyan1, A M Mozharov1, V A Shkoldin1,2, Y S Berdnikov3 and I S Mukhin1,2

1 Alferov University, Khlopina 8/3, 194021, St. Petersburg, Russia
2 ITMO University, Kronverkskii 49, 197101, St. Petersburg, Russia
3 St. Petersburg State University, Universitetskaya Emb. 13B, 199034, St. Petersburg, Russia

imukhin@spbau.ru, nadoyan@spbau.ru

Abstract. This work suggests a new approach to weighting the nanoscale objects placed at the tip of cantilever vibrating inside the camera of scanning electron microscope. In contrast to traditional approach to mass determination, we suggest tracing the shift of the node of the second vibration mode as an alternative to frequency shift measurement. We demonstrate the applicability of our approach to carbon nanowhisker cantilevers grown on tungsten needles by focused electron beam induced deposition. We compare experimentally the performance of the suggested approach with the traditional frequency shift-based method.

1. Introduction
Nanomechanical resonators are used in design of new class of mass sensors with exceptional precision and fast response [1–3]. Focused electron beam induced deposition (FEBID) is fast versatile technique enabling fabrication of carbon nanowhisker (CNW) cantilevers for nanomass sensing inside a scanning electron microscope (SEM) [4,5]. Typically, the mass measurement mechanism implies determination of the resonant frequency shift induced by a nanoparticle placed on cantilever. This approach inevitably requires recording an amplitude frequency response (AFR) for a specific resonant mode. The amplitude is measured from SEM visualization of the cantilever vibration during frequency scanning. In this process requires long exposure of the CNW cantilever to electron beam and unavoidably leads to parasitic deposition, which limits the precision of the mass sensing [5,6]. This work suggests tracing the shift of the node as an alternative to frequency shift measurement. We demonstrate the applicability of the new approach and compare its performance with the AFR-based method.

2. Fabrication of the mass sensor
The resonant mass sensor consists of vibration generator and CNW cantilever grown on the tip of a tungsten needle. As a generator of vibrations, we used the tubular piezoelectric transducer with generator of signals of special form «АКИП - 3413/3». The sharpened tungsten needle was prepared using the electrochemical etching in a 5% KOH solution as schematically illustrated in Figure 1 (a). The optimal tip sharpness lies in the range of 300-800 nm. Figure 1 (b) show the schematic for FEBID growth of CNW in FEI Quanta Inspect SEM. In our experiments the FEBID process employs the hydrocarbon groups from the residual atmosphere of the SEM camera. Decomposition of hydrocarbon
precursors is driven by secondary electrons in the area exposed to primary electron beam [7]. Therefore, the trajectory of the electron beam defines the shape of the deposited carbon nanostructure, thus allowing to control the CNW geometry. Figure 1 (c) sketches the mass sensor consisting of W-needle with the grown CNW placed in piezotube of the vibration generator.

![Figure 1](image)

**Figure 1.** (a) Electrochemical etching of the W-needle; (b) FEBID process in the SEM camera; (c) Mass sensor geometry.

For obtaining the AFR of the CNW cantilever we used the most easy-to-implement method based on visualization of vibrations at the investigated frequency band by means of SEM. When the oscillator frequencies match the resonant frequencies of the nanoresonator, the image of whisker is blurred on the screen and takes the form of a fan or figure-of-eight for the first and second resonances, respectively, as sketched in Figure 2 (a) and (b).

![Figure 2](image)

**Figure 2.** (a) Visualization of the first resonance; (b) Visualization of the second resonance.

Generally, the placement of a nanoobject at the tip of the whisker can be performed using the developed "pick-and-place" method, which consists of picking up a nanoparticle with the tip of a nanomanipulator located in the SEM chamber and transferring it to the target place. The particle can be
securely fixed by carbon welding or special nanotraps [5]. However, in this work we consider the test structure grown at the tip of the CNW via the FEBID process to demonstrate the applicability of the suggested mass sensing mechanism.

3. Sensing mechanism
Figure 3 (a) illustrates the standard method of weighing nanoobjects using mechanical nanoresonators consists of determining the shift of the first resonance frequency when the mass under study is localized at the cantilever tip. The AFR of the system is studied by visualization of the vibration shape in a scanning electron microscope camera and requires a long exposure of the sample to the electron beam, which leads to an uncontrolled increase in the whisker mass and an undesirable shift of the resonance frequency. As a solution to this problem, we propose an alternative approach to measure the mass of a nano-object by tracing the displacement of the second resonance node as shown in Figure 3 (b). In this case, it is sufficient to find the resonance with a “package” of frequencies and measure the distance between the needle tip and the resonance node, which significantly reduces the mass measurement time. In addition, the node position depends only on the linear distribution of mass along the length of the whisker [3], and therefore is weakly sensitive to parasitic deposition homogeneous along the CNW length.

\[
M_f = \frac{3E(d/2)^4}{16\pi \cdot f_{\text{res},W+M} \cdot L^3} - 0.227 \cdot m_1, \tag{1}
\]

where \(E\) is the Young’s modulus, \(d\) is the whisker diameter, \(L\) is the whisker length, \(m_1\) is the whisker mass. The frequency shift from 0.95 to 0.8 MHz, according to the eq. (1), corresponds to the additional
mass of $5.2\cdot10^{-17}$ kg. Note that this value is close to the value of $2.5\cdot10^{-17}$ kg, calculated using the density of FEBID grown carbon structures of 2200 kg/m$^3$ [5] and the measured in SEM sizes of the grown cylindric mass (118 nm in diameter and 650 nm in length). The difference in the results may be due to the fact that the studied object was not a point mass.

The node shift after adding the point mass at the CNW tip can be estimated as [8]

$$M_z \approx \frac{m_1 \cdot \Delta Z}{z_1}, \quad (2)$$

where $m_1$ is the mass of the whisker, $z_1$ is the position of the tug without mass, $\Delta Z$ is the node shift when the mass is added. The shift of the second resonance node position of ~300 nm was experimentally measured (Figure 4 b, c), which, according to eq. (2), corresponds to a localized mass of $1.04 \cdot 10^{-17}$ kg. Thus, the mass measured from node shift gives the value close to the mass values calculated from the frequency shift and the nanoobject size.

![Figure 4](image)

**Figure 4.** (a) CNW with the added mass at the tip; CNW vibrations at the second resonance before (b) and after (c) additional mass deposition.

5. **Conclusions**

This study presents a new method of mass measurement based on tracing the node shift at the second resonance of CNW vibrations. This method does not require a long exposure in SEM and excludes errors in determining the whisker geometry size and the influence of uncontrolled frequency drift on the weighing accuracy. We performed weighing of a test carbon mass localized at the tip of the whisker. We observe the first resonance frequency shift of ~150 kHz which corresponds to a localized mass of $5.2\cdot10^{-17}$ kg. For the same system experimentally obtained ~300 nm shift of the node position at second resonance, which corresponds to a localized mass of $1.04\cdot10^{-17}$ kg. Mass of investigated object determined by geometry is equal to $2.5\cdot10^{-17}$ kg. Thus, the suggested method has the accuracy in mass sensing similar to traditional approach but allowing much shorter exposure of the weighting object to the electron beam.

**Acknowledgments**

The work was financially supported by the Ministry of Science and Higher Education of the Russian Federation (FSRM-2020-0005). YB acknowledges St. Petersburg State University for the research grant 75746688.
References

[1] Yamaguchi H 2017 GaAs-based micro/nanomechanical resonators Semicond Sci. Technol. 32 103003

[2] Chaste J, Eichler A, Moser J, Ceballos G, Rurali R and Bachtold A 2012 A nanomechanical mass sensor with yoctogram resolution Nat. Nanotechnol. 7 301–4

[3] Malvar O, Ruz J J, Kosaka P M, Dominguez C M, Gil-Santos E, Calleja M and Tamayo J 2016 Mass and stiffness spectrometry of nanoparticles and whole intact bacteria by multimode nanomechanical resonators Nat. Commun. 7 1–8

[4] Gruber G, Urgell C, Tavernarakis A, Stavrinadis A, Tepsic S, Magén C, Sangiao S, De Teresa J M, Verlot P and Bachtold A 2019 Mass Sensing for the Advanced Fabrication of Nanomechanical Resonators Nano Lett. 19 6987–92

[5] Lukashenko S Y, Mukhin I S, Komissarenko F E, Gorbunov O M, Sapozhnikov I D, Felshtyn M L, Uskov A V. and Golubok A O 2018 Resonant Mass Detector Based on Carbon Nanowhiskers with Traps for Nanoobjects Weighing Phys. Status Solidi Appl. Mater. Sci. 215 1–5

[6] Lukashenko S Y, Mukhin I S, Veniaminov A V., Sapozhnikov I D, Lysak V V and Golubok A O 2016 Q-factor study of nanomechanical system “metal tip – carbon nanowhisker” at low and ambient pressure Phys. Status Solidi Appl. Mater. Sci. 213 2375–79

[7] Mukhin I S, Fadeev I V, Zhukov M V, Dubrovskii V G and Golubok A O 2015 Framed carbon nanostructures: Synthesis and applications in functional SPM tips Ultramicroscopy 148 151–7

[8] Tymoshenko S P, Yang D Kh and Weaver W 1985 Fluctuations in engineering Mashinostroenie 472