

Doctoral Dissertation Doctoral Program in Physics (31.th cycle)

Microbridge resonators with embedded nanochannels for attogram resolution in liquid

Abstract

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Abstract

The purpose of this thesis is to investigate the realization of microbridge resonators with embedded nanochannels used as mass sensors with attogram resolution in liquid. The attogram resolution in liquid has been taken as target because the aim of this work is to fabricate a sensor able to perform real-time detection of buoyant nanoparticles (NPs).

Natural or engineered nanoparticles, with diameters below 100 nm and so with mass of the order of some femtogram (1 fg = 1×10^{-15} g), are extremely used in many fields, such as medicine [1], energy [2], environment [3], cosmetics [4] and food [5]. A tool for real-time and accurate identification of cell-secreted nano-vesicles (such as exosomes) in liquid media would for sure open new perspectives in comprehension and treatments of cancer and neurodegenerative diseases. Similarly, an instrument able to routinely identify and quantify the number and particle size distribution of objects in the 1 nm–100 nm range would be essential to accurately monitor the degradation and fate of nanoparticles after their use, a topic that every day is growing in interest, since evidence of toxicology of many nano-objects ("nanotoxicology") [6].

Determination of nanoparticles in liquid is, in fact, a major challenge for the scientific community, because it presents extremely serious analytical difficulties for definition of identity (whether a certain substance is in the sample, i.e. size and chemical composition), as well as quantification (how much of the substance is in the sample, i.e. mass). Currently used techniques such as Transmission Electron Microscope (TEM) [7], Dynamic Light Scattering (DLS) [8] and Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) [9] pose severe limitations in sample preparation, cost and data interpretation.

The here proposed sensing platform is based on a resonating microbeam on which a nanochannel is integrated, such kind of sensor is called suspended nanochannel resonator (SNR) [10]. When a NP is passing through the vibrating nanochannel it will produce a variation of the effective mass of the device, changing its resonating frequency [11]. Thanks to this approach (proposed in [12] for suspended microchannel resonator - SMR), it is possible to circumvent the main drawback of cantilever-based mass sensors that consists of the complexity to operate directly in liquid phase, where the viscous friction decreases the frequency and the quality factor, making the sensor unstable and poorly sensitive.

Chapter number one reports the state of the art of SMR/SNR devices, chapter number two explains the working principles and the theory of the mechanical resonators as mass sensors and the third one deals with the design of the devices

in order to reach the target sensitivity. In this thesis three different devices are presented: nanoslit, in which the only nanometric dimension is the height of the hollow channel; nanochannel, in which both height and width are at the nanoscale; and the so called "two-step" nanoslit in which the height of the hollow channel is not constant along the extension of the device. The fabrication, done in the Centre of Micro and Nanotechnologies at the École polytechnique fédérale de Lausanne (CMi@EPFL), is deeply described in the fourth chapter. The process flow was based on a sacrificial layer approach that allows to easily tailor the dimensions of the channel by changing the thickness of the sacrificial layer or its patterning during the lithographic step. Then the fabrication process was applicable to the three types of devices designed only carrying punctual modification at some step. With this approach, devices with channel dimension of 50 nm was successfully fabricated. In the fifth chapter it is illustrated the experimental setup used to get the result analyzed in the sixth chapter. The characterization activity has been focused on the nanoslit devices with the mass sensitivity of the resonators assessed to be up to 256.4 ag/Hz. Finally, in the seventh chapter a resume of the work is disclosed as conclusion of the thesis. To the best of our knowledge, the devices presented here are the first SNR reported with a channel height as low as 50 nm that are fabricated with a top-down approach.

Bibliography

- 1. Salata, O. V. Applications of nanoparticles in biology and medicine. *J. Nanobiotechnology* **2**, 3. ISSN: 1477-3155 (Apr. 2004).
- 2. Lan, Y., Lu, Y. & Ren, Z. Mini review on photocatalysis of titanium dioxide nanoparticles and their solar applications. *Nano Energy* **2**, 1031–1045. ISSN: 2211-2855 (2013).
- 3. Zhang, W.-x. Nanoscale Iron Particles for Environmental Remediation: An Overview. J. Nanoparticle Res. 5, 323–332. ISSN: 1572-896X (2003).
- 4. Lu, P.-J., Huang, S.-C., Chen, Y.-P., Chiueh, L.-C. & Shih, D. Y.-C. Analysis of titanium dioxide and zinc oxide nanoparticles in cosmetics. *J. Food Drug Anal.* 23, 587–594. ISSN: 1021-9498 (2015).
- 5. Dickinson, E. Use of nanoparticles and microparticles in the formation and stabilization of food emulsions. *Trends Food Sci. Technol.* **24**, 4–12. ISSN: 0924-2244 (2012).
- Suh, W. H., Suslick, K. S., Stucky, G. D. & Suh, Y.-H. Nanotechnology, nanotoxicology, and neuroscience. *Prog. Neurobiol.* 87, 133–170. ISSN: 0301-0082 (2009).
- 7. Michen, B. *et al.* Avoiding drying-artifacts in transmission electron microscopy: Characterizing the size and colloidal state of nanoparticles. *Sci. Rep.* **5**, 9793 (May 2015).
- 8. Hoo, C. M., Starostin, N., West, P. & Mecartney, M. L. A comparison of atomic force microscopy (AFM) and dynamic light scattering (DLS) methods to characterize nanoparticle size distributions. *J. Nanoparticle Res.* **10**, 89–96. ISSN: 1572-896X (2008).
- 9. Donovan, A. R. *et al.* Single particle ICP-MS characterization of titanium dioxide, silver, and gold nanoparticles during drinking water treatment. *Chemosphere* **144**, 148–153. ISSN: 0045-6535 (2016).
- Lee, J., Shen, W., Payer, K., Burg, T. P. & Manalis, S. R. Toward attogram mass measurements in solution with suspended nanochannel resonators. *Nano Lett.* 10, 2537–2542. ISSN: 1530-6984 (2010).

- 11. Schmid, S., Villanueva, L. G. & Roukes, M. L. Fundamentals of nanomechanical resonators 1–175. ISBN: 9783319286914. doi:10.1007/978-3-319-28691-4 (2016).
- 12. Burg, T. P. & Manalis, S. R. Suspended microchannel resonators for biomolecular detection. *Appl. Phys. Lett.* **83**, 2698–2700. ISSN: 0003-6951 (2003).

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