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Multi-MHz micro-electro-mechanical sensors for atomic force microscopy

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ABSTRACT

Silicon ring-shaped micro-electro-mechanical resonators have been fabricated and used as probes for dynamic atomic force microscopy (AFM) experiments. They offer resotnance frequency above 10 MHz, which is notably greater than that of usual cantilevers and quartz-based AFM probes. On-chip electrical actuation and readout of the tip oscillation are obtained by means of built-in capacitive transducers. Displacement and force resolutions have been determined from noise analysis at 1.5 fm/√Hz and 0.4 pN/√Hz, respectively. Despite the high effective stiffness of the probes, the tip-surface interaction force is kept below 1 nN by using vibration amplitude significantly below 100 pm and setpoint close to the free vibration conditions. Imaging capabilities in amplitude- and frequency-modulation AFM modes have been demonstrated on block copolymer surfaces. Z-spectroscopy experiments revealed that the tip is vibrating in permanent contact with the viscoelastic material, with a pinned contact line. Results are compared to those obtained with commercial AFM cantilevers driven at large amplitudes (> 10 nm).

1. Introduction

Scanning probe microscopy (SPM) has been one of the most important instrumental discoveries during the last quarter of the last century [1]. In particular, atomic force microscopy (AFM) is a cross-disciplinary technique able to provide sample morphology down to the atomic scale [2]. This has been at the origin of, and constantly supports the development of nano-sciences, information technologies, micro-nanotechnologies and nano-biology. Dynamic mode AFM has a unique capability of characterizing soft and biological materials like molecular structures in native-like and functional conditions [3–6]. Interaction forces between the AFM oscillating tip and the sample surface can be kept in the 10 pN range, ensuring non-damaging observations. Beyond providing topography images by scanning XY axes, AFM through Z spectroscopy gives access to advanced analysis of tip-sample interactions and to rheological information like material elasticity and mechanical dissipation [7–9]. Increasing the AFM probe resonance frequency is desirable for such applications. First, it contributes to increase the measurement bandwidth of the AFM, one of the key parameters for time-resolved experiments and high-speed imaging, which remains a huge expectation in the field. Second, it allows the investigation of the dynamic behavior of material viscoelasticity over an extended frequency range. The mainstream option to achieve this goal consists in miniaturizing the conventional AFM probe based on the flexural mode cantilever to raise the resonance frequency. Downscaled cantilevers are now commercially available resonating in the range 1–5 MHz [10]. During the last decade, they have been employed successfully in high-speed AFM instruments [11], leading to impressive video rate images and to direct observations of the dynamic behavior of nano-biological systems [4,5]. The cantilever technology faces however difficulties to push further the frequency: the beam size of the laser used for the detection as well as the fabrication of the tip out of the plane of the cantilever limit its lateral dimensions to a few micrometers. For these practical considerations, the resonance frequency of AFM cantilevers is hard to exceed 5 MHz in air. Alternative strategies have been deployed since the 1990's to circumvent these limitations. In particular, numerous studies have been devoted to the integration of self-sensing and self-driving methods thanks to the progress in micro/nano fabrication and electromechanical transduction schemes. Integrated detection methods such as capacitive [12], piezoresistive [13] and piezoelectric [13,14] sensing schemes have been developed in purpose of overcoming the constraints of optical detecting method.

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Despite outstanding results in terms of resonance frequency up to 100 MHz [15], moderate success among AFM users is obtained because of the difficulty and the cost of fabrication of such small probes.

Leaving the cantilever concept aside, another paradigm appeared 15 years ago taking advantage of quartz resonators. AFM probes based on quartz tuning forks [16] and length-extension resonators [17] with self-driving and self-sensing capabilities have been developed successfully. Considering that the force resolution that is essential for the AFM sensitivity does not depend on the stiffness only but also on the resonance frequency, the quality factor and the resonator stability, it is possible to relax the constraint on the probe stiffness and thus on the resonator size when increasing the resonance frequency. Such AFM probes can achieve atomic resolution images and can be operated at sub-nanometer vibration amplitudes thanks to their high stiffness preventing the tip from snapping into the surface [17,18]. A drawback of the current AFM probe quartz technologies lies in the millimetric dimension of the resonators that limits the resonance frequency to 1 MHz [19].

In this paper, AFM probes are implemented using micro-electro-mechanical systems (MEMS) resonators, which allows raising the probe resonance frequency above 10 MHz. In-plane vibrating silicon micro-resonators that were primarily developed for electronic filters or time reference applications [20] are indeed promising candidates for AFM applications. Integrated capacitive electromechanical transducers bring self-driving and self-sensing capabilities; resonance frequencies can exceed 100 MHz, enabling large measurement bandwidth and time-resolved experiments at the microsecond scale or below; in-plane vibration modes suffer less from damping and energy losses than flexural modes, and quality factors can exceed 1,000 in air. In the light of the application of MEMS resonators in the field of surface imaging, the authors previously presented in 2007 the concept of AFM probes taking advantages of the elliptic vibration of a ring-shaped micromechanical resonator [21,22]. Further works demonstrated that such probes are sensitive to mechanical interactions with surface forces [23] and can be successfully used for AFM imaging [24]. Recent advances in signal processing gave access to the detection the thermomechanical displacement noise of the probes, paving the way for exquisite sensitivity and force resolution [25,26]. In the present study, AFM results obtained with 13.6 MHz MEMS AFM probes are presented. Firstly, the probes and the experimental set-up are described. Probes are characterized in terms of short- and long-term noise, resolution and stability. The results are discussed and operating conditions for AFM imaging are deduced for these high frequency probes. In a second part, the probes are used for amplitude-modulation (AM) and frequency-modulation (FM) AFM imaging modes. Topographic images and force spectroscopy results obtained with block copolymers surfaces are shown and compared to measurements done with a commercial equipment and cantilever probes.

2. Material and methods

2.1. Probe description

The MEMS AFM probes exploit the elliptical mode of a single-crystal silicon ring as shown in Fig. 1. The tip is located at one of the maxima of displacement of the ring, the in-plane vibration of the ring inducing the radial oscillation of the tip [22,24]. Electrodes are located at the other maxima of displacement, separated from the ring by an air gap. They act as capacitive electromechanical transducers able to drive and sense the ring vibration. Consequently, no external actuator nor optical detection unit are required to use the probe. Four beams anchor the resonator to the substrate. The batch fabrication of the probes starts from a silicon-on-insulator (SOI) wafer with a 5-µm thick (100) silicon top layer, a 2-µm thick buried oxide layer and a 380-µm thick silicon handle layer. The probe body is first etched using a combination of photolithography and deep reactive ion etching (DRIE). The capacitive transduction air gap is defined by a thermally-grown sacrificial oxide layer. Polycrystalline silicon deposition and patterning are then performed to define the electrodes. Afterwards, the probe in-plane tip is fabricated using a process similar to the one developed by Ried *et al.* [27] that can yield curvature radius at the tip apex lower than 10 nm. Back-end steps consist of metallization for electrical contacts and backside etching (DRIE) of the probe holder to leave the tip apex prominent from its support. Finally, the devices are released using vapor hydrogen fluoride (HF) sacrificial etching of oxide. More details of the fabrication process can be found elsewhere [28].

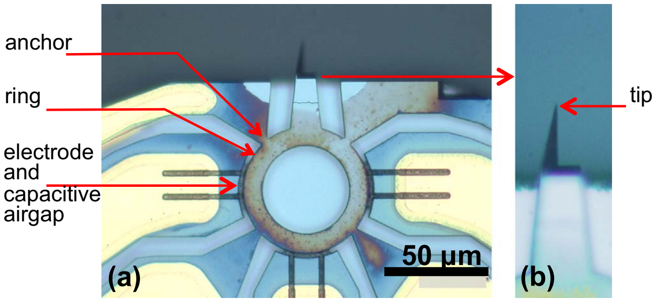

Fig. 1. (a) Optical microscopy image of a MEMS AFM probe. (b) Close view of the tip apex. Color variations of the surface of the device come from the fabrication process and do not affect the probe operation.

The resonance frequency f_O and stiffness K_{eff} of the MEMS AFM probe depend on the material properties and ring dimensions. Previous studies made use of 500-µm outer diameter rings that yielded resonance frequencies close to 1 MHz [24]. In the present work, a 60-µm outer diameter ring allows to reach resonance frequencies greater than 10 MHz. Table 1 summarizes the parameters and dimensions that are used in this study. Whereas analytical approaches can be used to predict the mode shape and eigen frequencies of the in-plane elliptical vibration of a free ring [24,29,30], finite element modeling (FEM) is required here to take into account the effect of the mass and stiffness added by the tip and the anchors, and to obtain an accurate description of the MEMS AFM probe vibration. Fig. 2 shows FEM modal analysis of the MEMS AFM probe displacement. It clearly shows the radial displacement of the probe tip. As a consequence, the probe chip will be mounted vertically in the AFM setup. For the parameters of Table 1, the resonance frequency is f_0=13.893 MHz and the effective stiffness at the tip location is K_{eff}=198 kN/m. One can note that the amplitudes of the 4 maxima of vibration of the ring are unbalanced, which is explained by the presence of the tip that breaks the symmetry of the ring resonator.

Table 1
MEMS AFM probe parameters and dimensions.

Parameter	**Signification**	**Value**
Material	Device structural material	Silicon
R_{ext}	Ring outer radius	30 µm
R_{int}	Ring inner radius	20 µm
t	Ring thickness	5 µm
L_{tip}	Tip length	15 µm
$\theta_{electrode}$	Angular width of the electrodes	π/4 rad
g	Transduction air gap	45 nm
f_0	Probe resonance frequency (FEM)	13.893 MHz
Q	Probe quality factor	*
K_{eff}	Probe effective stiffness at the tip location (FEM)	198 kN/m
Ψ_{tip}	Modal amplitude at tip location (FEM)	1
Ψ_{drive}	Modal amplitude at drive electrode location (FEM)	1.09
Ψ_{sense}	Modal amplitude at sense electrode location (FEM)	1.17

*Quality factor Q cannot be determined by FEM modal analysis. In the following, Q value is obtained from the experimental measurement of the probe resonance frequency response.