CMOS INTEGRATED NANOMECHANICAL MASS SENSORS: DETERMINATION OF EVAPORATION RATE OF FEMTOLITER DROPLETS

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During recent years, the need of methods to manipulate very small liquid quantities has emerged, leading to the development of nanofluidics or nanodispensing [1] techniques. At these scales, evaporation processes become important. However, previous comprehensive studies of evaporation are limited to microliter droplets [2]. We address here the question of the validity of the macroscopic models at micro and nano scales (sub-femtoliter range). We present the use of CMOS integrated nanomechanical resonators [3] to determine with high precision the change in mass of liquid droplets with time. We have studied [4] the evaporation of droplets with diameters in the one micron range, what corresponds to volumes of femtoliters, nine orders of magnitude smaller than previously published data.

The nanomechanical resonator is a polysilicon quad-beam resonator (QBR) defined by electron-beam lithography on pre-fabricated CMOS substrates. It provides high mass sensitivity together with large active area for convenient droplet deposition. Because its resonance spectrum is detected using a capacitive scheme, QBR are monolithically integrated with CMOS circuitry (fig.1) for signal amplification and parasitic capacitance reduction. Prior to the evaporation experiments, QBR are calibrated by successive loading of silica beads using a micro-manipulator. The obtained mass resolution is three orders of magnitude better than commercial quartz crystal microbalances

Dispensing of droplets is based on the NADIS technique described in [1]. Commercial AFM tips are modified by FIB milling to allow loading liquid and dispensing of droplets through a hole of 300 nm in diameter milled at the tip apex. Droplets with diameters ranging from 1 to 5 µm were reproducibly deposited on the resonators (fig.2).

During evaporation, we monitored the resonance frequency shift of the QBR (fig.3). Using a calibration curve, the temporal evolution of the droplets mass was determined down to 10 fg (10 attoliters volume) for glycerol droplets with initial volumes ranging from 0.2 fL to 20 fL. The droplet mass decreases nonlinearly, with a slowing down of the evaporation rate with time. This behaviour, is well described by macroscopic model which predicts, in constant contact angle mode, a decrease of the droplet mass with evaporation time with a power law 2/3 [2]. The linear decrease of $m^{2/3}$ as a function of time (figure 4) together with the fact that the total evaporation time depends on the initial weight m_0 at power 2/3 (inset of figure 4), indicates that the macroscopic laws remains valid down to these scales.

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Figures:

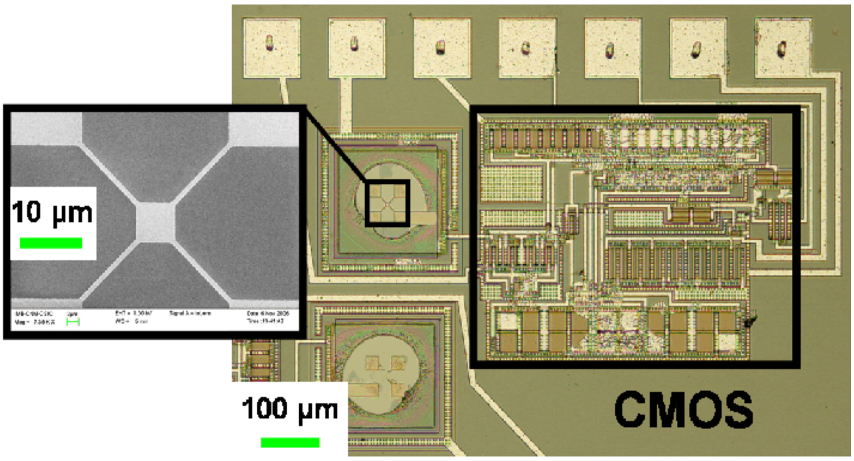

Figure 1. Optical image of a Quad-Beam resonator (QBR) monolithically integrated into CMOS circuitry. The polySi structural layer is electrically connected to the input of the circuit that acts as a trans-impedance amplifier. The inset shows a SEM image of a QBR defined after CMOS fabrication by e-beam lithography and subsequent Al lift-off, pattern transfer to polysilicon by RIE and release wet etching. The device has the following dimensions: central plate (6*6 μm^2), beams width and length: 600 nm and 13.5 µm, thickness: 500 nm.

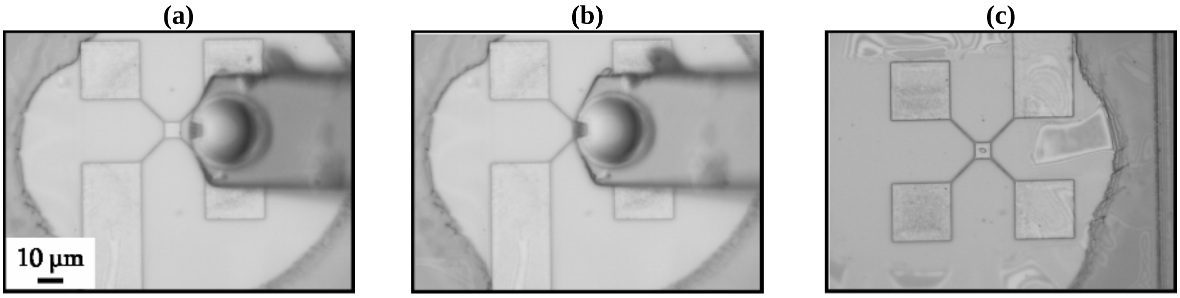

Figure 2. Top view of a nanodispensing probe. (a) the tip, pre-loaded with a liquid, is approached; (b) it is aligned with a QBR adjusting its position with the control table; (c) final image of the deposited droplet on the QBR central plate.

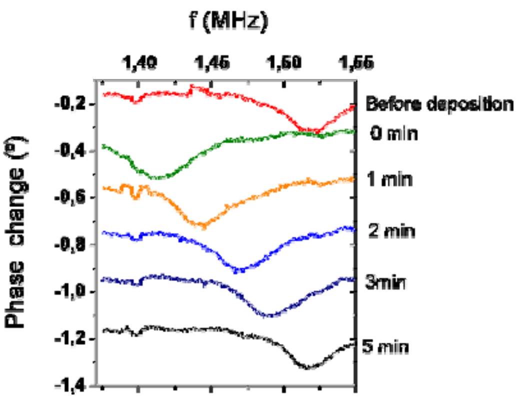

Figure 3. Evolution of the resonance frequency of a QBR as a function of the evaporation time (unloaded resonance frequency ≈ 1.52 MHz) during the evaporation of a micron-sized droplet. After complete evaporation, the resonance frequency turns back to its unloaded value. A phase offset has been added to each curve to make easier the visualization of the temporal evolution

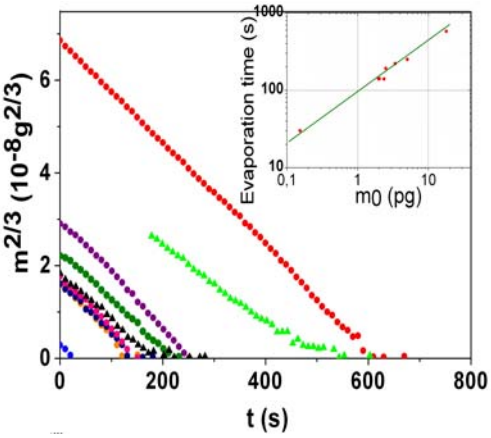

Figure 4. Temporal evolution of the droplet mass at a power 2/3 for the same data as in fig. 5. All the droplets exhibit a linear trend with the same slope. The inset is a plot of the total evaporation time of as a function of the initial mass