Microelectronics Journal 45 (2014) 1648–1655

Contents lists available at ScienceDirect

Microelectronics Journal

journal homepage: www.elsevier.com/locate/mejo

A novel approach to high-speed high-resolution on-chip mass sensing

C. Kauth *, M. Pastre, M. Kayal

STI-IEL Electronics Laboratory, Ecole Polytechnique Fédérale de Lausanne, Switzerland

ARTICLE INFO

Article history:
Received 26 December 2013
Accepted 14 July 2014
Available online 2 August 2014

Keywords:
Analytical models
Carbon nanotubes
Closed loop systems
Nanoelectromechanical systems
Nonlinear systems
Oscillators
Phase locked loops
System analysis and design

ABSTRACT

The state-of-the-art mass sensing so far has been rather developed along the resolution axis, reaching atomic-scale detection, than into the direction of high-speed. This paper reports a novel self-calibrating technique, making high-speed inertial mass sensors capable of instant high-resolution particle detection and weighing. The sensing nanoelectromechanical resonator is embedded into a phase-locked loop and the sensor-inherent nonlinear phase–frequency relation is exploited for auto-calibration. A tunable on-chip carbon nanotube based mass balance serves as a case study of small-size and low-cost environmental and healthcare applications. Tunability and a phase-locked loop topology make the system widely universal and invariant to nanotube characteristics. Operational for tube eigenfrequencies up to 385 MHz, the circuit integration in a 180 nm technology achieves instantaneous zeptogram resolution, while yoctogram precision is obtained within the tenth of a second. These figures of merit range at the physical limits of carbon nanotube resonators, in both mass- and time-resolution.

1. Introduction

The state-of-the-art mass sensing is predicated on the detection of a shift in a resonator's eigenfrequency when an additional mass binds to its surface. Evaluation of the theoretical ultimate limits to such inertial mass sensing [1] suggests that single proton weighing is feasible at room temperature. This yoctogram mass resolution has been achieved [2] at liquid-helium temperature with an ultrasensitive carbon nanotube resonator, operated in a mixer setup [3]. The equivalent room-temperature record ranges at a hundredfold worse resolution [4]. These high-resolution sensors find applications in atomic physics, biology and life science as molecule identifiers, gas detectors and cell weighing to name but a few.

In contrast to this impressive mass resolution, the temporal resolution of such sensors appears unspectacular. Frequency sweeps that last several seconds [5] and feedback loops of tens of milliseconds [2] set the benchmark hitherto. By comparing this sensing speed to the quasi-gigahertz NEMS eigenfrequency, it becomes apparent that there is immense potential to improve sensing speed by several orders of magnitude.

A novel approach to simultaneous high-speed and high-resolution nanomechanical mass sensing is presented in this contribution. Today's state-of-the-art mass sensors do not combine these two desirable properties yet: high-resolution methods [2] rely on frequency sweeps or mixing, which makes them inherently slow, while high-speed methods [6] present non-quantifiable offsets, which lead to erroneous mass inference. An auto-calibrating system is shown to compensate offsets and hence enables high-resolution high-speed sensing. Such precise and fast mass detection allows observation of phenomena that so far are not observable because they are either too subtle (insufficient sensor resolution) or happen too fast (insufficient sensor speed), like chemical and biochemical reactions.

The concept condensates into an implementable topology and suspended carbon nanotubes (CNT) are the resonator of choice, favoured by their low effective mass and acceptable quality factor. Their high elasticity allows us to tune the CNT for maximal compatibility with the interface circuitry via mechanical strain. Feedback electronics, implemented in a 180 nm technology, form a phase-locked loop (PLL) around the CNT-NEMS resonator in order to drive and sustain the latter's motion at resonance. Metamorphosed into small-size and low-cost sensor nodes for large-scale healthcare or environmental applications, this on-chip sensor circuit presents unprecedented speed, and mass resolution down to the physical limits of nanoelectromechanical systems.

Section 2 brings forward the key elements for high-speed and high-resolution mass sensing, and presents a novel topology combining both features. The physical phenomenon at the basis of this self-calibrating sensor topology is investigated in Section 3. Section 4 demonstrates that this nonlinear phenomenon can indeed be exploited in practice for calibration and the operation principle of the resulting high-resolution high-speed mass sensor is explained in Section 5. Section 6 starts a case study illustrating how suspended carbon nanotubes can fulfill the role of the sensing NEMS. The motional information processing system and its integration are the topics of Section 7. Section 8 identifies which CNTs are adequate for the proposed chip and translates the latter's jitter into sensor performance in terms of resolution and speed.

* Corresponding author.
E-mail address: christian.kauth@epfl.ch (C. Kauth).

http://dx.doi.org/10.1016/j.mejo.2014.07.004
0026-2692/

2. Motivation and novelty

Fast and precise identification of the NEMS eigenfrequency ω_0 may be modelled as a search problem. A questioner interrogates an oracle

$$O:\omega\mapsto\begin{cases}-1 & \text{if } \omega<\omega_0\\ 0 & \text{if } \omega=\omega_0\\ +1 & \text{if } \omega>\omega_0\end{cases} \tag{1}$$

on whether the unknown eigenfrequency ω_0 equals an arbitrary guess ω. The oracle answers with -1, 0 or $+1$ as a function of the relative position of ω and ω_0. Two key parameters to high-speed high-resolution mass sensing crystallize from this formal description of the eigenfrequency search. First must the oracle provide fast and trustworthy responses, and second shall the questioner interpret the answer quickly and rapidly to improve the quality of the guess.

The fastest way to determine a periodic signal's frequency is to measure exactly one period. Hence high NEMS eigenfrequencies are beneficial for the first criterion and suspended carbon nanotubes are a neat choice, as shown in Section 6. Noise may distort the oracle's answer, but can be countervailed by averaging over several periods. The resulting speed-precision trade-off is assessed in Section 8. The current section's emphasis lies on the inspection of techniques to rapidly observe the oracle's answer and formulate a precise guess on ω_0. The following observations are independent of the exact NEMS nature and averaging. A generic dynamic NEMS may be modelled as a normalized damped harmonic oscillator

$$H(s)=\frac{\frac{\omega_0}{Q}\left(1-\frac{1}{4Q^2}\right)}{s^2+\frac{\omega_0}{Q}s+\omega_0^2},\qquad \begin{cases}|H(s)|=A\\ \arg(H(s))=\phi\end{cases} \tag{2}$$

with eigenfrequency ω_0 and quality factor Q. The eigenfrequency is observable via detection of the amplitude A peak at ω_p or the phase ϕ inflection at ω_i:

$$\omega_p=\omega_0\sqrt{1-\frac{1}{2Q^2}},\qquad \omega_i=\omega_0\sqrt{\sqrt{4-\frac{1}{Q^2}}-1} \tag{3}$$

$\omega_p\approx\omega_i\approx\omega_0$ for sufficiently large Q. Adsorption of a particle onto the oscillating NEMS causes a relative change $\Delta m_{eff}/m_{eff}$ in the oscillator's effective mass, defined by the particle's weight and the binding position [7], and shifts the resonance characteristics as $(\omega_0,Q)\rightarrow(\omega_0/\sqrt{\alpha},\ \sqrt{\alpha}Q)$, with $\alpha=1+(\Delta m/m)$. This shift in eigenfrequency is to be detected as fast and precise as possible.

Today's techniques of doing so may be systematically categorized into

- ω*-controllable* techniques drive the NEMS at a given frequency ω and observe its amplitude A or phase ϕ response. Such approaches benefit from a reduced noise bandwidth when operating the NEMS as a mixer [5]. The mixed signal out of the NEMS, however, has a much lower frequency than ω_0. This makes mixing incompatible with the high-speed detection exigence, and feedback loops take tens of milliseconds [2] to improve the estimation of ω_0. High-resolution weighing is enabled by the simultaneous extraction of ω_p or ω_i and Q from a response plot around the resonance peak [2], which takes seconds. It is concluded that ω-controllable methods are precise, but slow.
- ϕ*-controllable* techniques embed the NEMS into a phase locked loop (PLL) and loop its output onto its input [4,8], with a controllable phase shift Φ, which forces the NEMS to oscillate at a unique frequency ω. An adsorption-related shift in ω is directly observable, enabling high-speed weighing, but the error in the observed $\Delta\omega_0$ scales as $1/Q\ \tan(\phi)$. If $\phi=\pi/2$, the error vanishes, but this ideal hypothesis is hard to satisfy in practice, where component delays play a role and are *a priori* hardly entirely corrigible. It is concluded that ϕ-controllable methods are fast, but have limited resolution.

As a matter of fact could the high-resolution and high-speed sensing hitherto be combined yet. An novel approach is shown in Fig. 1(a), where in situ calibration of the NEMS phase ϕ via a nonlinear technique adds precision to ϕ-controllable methods and opens the gate to high-speed high-resolution mass sensing. The NEMS is embedded into a phase locked loop, formed by a phase–frequency detector (PFD), a loop filter (F_{PLL}) and a voltage controlled oscillator (VCO). At steady state, the VCO oscillates at a frequency for which both feedback paths, one formed by the NEMS and the readout electronics [9], the other by a simple phase shifter, have exactly the same delay. Hence the NEMS is forced to oscillate at a precise frequency, at which ϕ satisfies the above constraint. This frequency depends consequently on the loop delay Φ. The additional feedback adjusts Φ so as to centre resonance at ω_i, quasi-equal to ω_0 for NEMS with decent Q. Fig. 1(b) illustrates how the auto-calibration circuit adjusts the loop phase Φ so as to bring the NEMS phase ϕ to $\pi/2$, which reflects oscillation at ω_0 and error-free sensing. Once the NEMS has been calibrated, Φ may simply be memorized and sensing happens precisely at the speed of ϕ-controllable methods. The underlying hypothesis for this memorization to work out is that the NEMS phase is far more sensitive to the oscillation frequency than any other circuit component. This condition is tacitly met in ϕ-controllable methods, as the PLL locks onto a frequency imposed by the circuit component with the highest quality factor, speak phase sensitivity, which must be the NEMS, if sensor operation is envisioned.

3. Physical phenomenon

The technique to enable calibration of the feedback loop phase shift Φ such that NEMS oscillation at ω_i is enforced roots in the

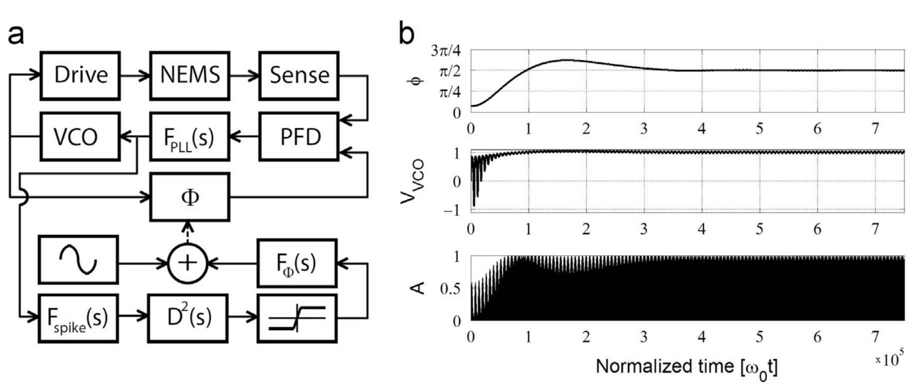

Fig. 1. Auto-calibrating mass balance. (a) System architecture. (b) Calibration process.