#### Nanotube Mechanical Resonators Péter Balla 3. May 2013

- Intro
- SWNT resonators
- Mass sensing with SWNT resonators
- Performance
- yg=10<sup>-24</sup>g, nucleon mass=1,7yg
- zg=10^-21g

# Intro

- Goal: precise mass detection/measurement
- Build a small, light and stiff resonator
- Capture a load to measure
- Kick it to resonate
- Detect the resonance shift
- And a few technicalities...
- "What is this thing, anyway?" said the Dean, inspecting the implement in his hands. "It's called a shovel," said the Senior Wrangler. "I've seen the gardeners use them. You stick the sharp end in the ground. Then it gets a bit technical."

(Terry Pratchett, Reaper Man)

### Resonators

 $f_0 = \frac{1}{2\pi} \left(\frac{k}{m_{\rm eff}}\right)^{1/2}$ 

δn

- The stiffer the better (large k)
- The lighter the better (small m\_eff)
- Use nanotubes or graphene (high R, mass responsivity)
- Memo: quality factor (Q): the bigger the better (Q=1/2 crit. damp.)

$$Q = 2\pi \times \frac{\text{Energy Stored}}{\text{Energy dissipated per cycle}}$$

$$\delta_z = \frac{1}{(2\pi)^2} \frac{F_{\text{el}}}{m_{\text{eff}}} \frac{1}{\sqrt{(f^2 - f_0^2)^2 + \frac{f^2 f_0^2}{Q^2}}} \xrightarrow{\text{Spring force}} \frac{f_{\text{force}}}{\sqrt{(f^2 - f_0^2)^2 + \frac{f^2 f_0^2}{Q^2}}} \xrightarrow{\text{Velocity}} \frac{f_{\text{force}}}{\sqrt{(f^2 - f_0^2)^2 + \frac{f^2 f_0^2}{Q^2}}}$$

### **SWNT Resonators**





tacts, displays fourfold shell filling of holes. (**D**) (Inset) The mechanical resonance induces a corresponding resonance in the dc current which can have a narrow linewidth with *Q* up to 150,000. (Main plot) The resonance frequency can be tuned using a tensioning force from the dc voltage on the gate.

#### Strong Coupling Between Single-Electron Tunneling and Nanomechanical Motion

G. A. Steele,\* A. K. Hüttel,† B. Witkamp, M. Poot, H. B. Meerwaldt, L. P. Kouwenhoven, H. S. J. van der Zant Fig. 1. (A) Schematic of the mechanical resonator. The moveable part is a SWNT (oscillations indicated by a red arrow). The SWNT length is  $1 \mu m$  and the diameter is ~1.1 nm. The motion is capacitively driven by the application of an oscillating voltage on the gate. The nanotube position is detected by applying an oscillating voltage on the source electrode and measuring the current from the drain electrode.  $\delta f = 10$  kHz. (**B**) Measured



mechanical resonance (mixing current as a function of driving frequency). The red curve is a fit of the resonance. (**C** and **D**) Schematic of the charge on the SWNT and its conductance as a function of the control charge ( $q_c = -C_g V_g^{DC}$ ) in the Coulomb-blockade regime. Vibrations cause the charge on the SWNT to oscillate ( $\delta q_{dot}$  and  $\Delta q_{dot}$ ).

### **Coupling Mechanics to Charge Transport in Carbon Nanotube Mechanical Resonators**

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Excite and Read



Figure 2. Diagram of the measurement circuitry and characteristics of nanotube resonators. (a) Diagram of the experimental setup. A voltage  $V_{g}^{ac}$  oscillating at frequency f is applied to the Si backgate to actuate the motion. The motion is detected using a mixing technique, which consists of applying an oscillating voltage  $V_{SD}$  at  $f + \delta f$  on the source and measuring the current at  $\delta f$  from the drain.  $\delta f$  is 10 kHz, and the lock-in time constant is 100 ms. (b) Mixing current as a function of gate voltage and frequency at 300 K and  $\sim 10^{-6}$  mbar.  $V_{\rm SD} = V_{\rm g}^{\rm ac} = 25$  mV. (c) Mixing current as a function of frequency for  $V_{g}^{d\bar{c}} = 2.2 \text{ V}$  and  $V_{SD} = V_{g}^{ac} = 1 \text{ mV}$  at 20 K and  $\sim$ 1 mbar. The resonance peak shape is fitted using eqs 3 and 4 (blue curve). Equations 3 and 4 can be written as  $I_{\text{mix}} = A$  $+ B \cos(\theta_{\rm m} - \alpha)/[(f^2 - f_0^2)^2 + f^2 f_0^2/Q^2]^{1/2}$  with  $\theta_{\rm m} = \arctan(ff_0/Q^2)$  $Q(f^2 - f_0^2)$ ). The parameter A corresponds to the background current signal, which is extracted from the experimental data far from the resonance. B,  $\alpha$ ,  $f_0$ , and Q are fitting parameters. The result does not depend on the starting value of the fitting parameters.

phase between  $F_{el}$  and the mechanical motion

$$\delta_{z} = \frac{1}{(2\pi)^{2}} \frac{F_{\text{el}}}{m_{\text{eff}}} \frac{1}{\sqrt{(f^{2} - f_{0}^{2})^{2} + \frac{f^{2} f_{0}^{2}}{Q^{2}}}}$$

Fig. 1. (A) Schematic of the mechanical resonator. The moveable part is a SWNT (oscillations indicated by a red arrow). The SWNT length is 1 µm and the diameter is ~1.1 nm. The motion is capacitively driven by the application of an oscillating voltage on the gate. The nanotube position is detected by applying an oscillating voltage on the source electrode and measuring the current from the drain electrode.  $\delta f = 10$  kHz. (**B**) Measured



mechanical resonance (mixing current as a function of driving frequency). The red curve is a fit of the resonance. (**C** and **D**) Schematic of the charge on the SWNT and its conductance as a function of the control charge ( $q_c = -C_g V_g^{DC}$ ) in the Coulomb-blockade regime. Vibrations cause the charge on the SWNT to oscillate ( $\delta q_{dot}$  and  $\Delta q_{dot}$ ).

Fig. 2. Electronic and mechanical properties of the device at 4 K. (A) The SWNT differential conductance as a function of source-drain voltage and gate voltage. The measurements are consistent with Coulomb blockade. Blue corresponds to low conductance, and red corresponds to high conductance. (B) Mixing current from the drain as a function of driving frequency and gate voltage. The frequency of the red lobes (related to the me-



chanical resonance) goes up and down when increasing  $V_g^{DC}$  with the same period as for the SWNT conductance. This shows that the mechanics and the charge transport in the device are correlated. In addition, the frequency of the red lobes shifts continuously over the full  $V_g^{DC}$  sweep caused by mechanical tension (the static electrostatic force bends the nanotube).  $V_g^{AC}$  is 0.5 mV and  $V_{SD}^{AC}$  is 0.1 mV ( $f_0$  and Q remain the same when decreasing  $V_a^{AC}$  to 0.15 mV at 4K). Blue corresponds to low current, and red corresponds to high current. T=4.2K

Coulomb-blockade

$$\delta C_g = C_g' \delta z$$

- Strong electro-mechanic coupling
- Charge-discharge: (current-Joule-heat on interfaces) ->Q
- Ch. fluct.->electronic force fluct.  $\rightarrow$  f\_0 depends on V\_g^DC

### Oscillating f\_0 and Q

Fig. 3. Electronic and mechanical properties of the device at 4 K. (A) Conductance of the SWNT as a function of gate voltage. (B) Schematic of the dissipation process. The mechanical oscillation charges and discharges the SWNT by the amount  $\delta q_{dot}$ , which results in a current  $\delta I_{dot}$ flowing through the resistance at the nanotubeelectrode interface. (C and D) Measured and calculated resonance frequency as a function of gate voltage. (E and F) Measured and calculated quality factor as a function of gate voltage.



### Mass sensing

## Ultrasensitive Mass Sensing with a Nanotube Electromechanical Resonator

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- Two articles: 2008/2012, Bachtold's group
- Params: mass responsivity:  $\mathscr{R}$ Q, mass resolution:  $\delta m_{\min}$
- Clean, low pressure, low temp.

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# A nanomechanical mass sensor with yoctogram resolution

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# 2008



**Figure 1.** Experimental setup for mass sensing. (a) Schematic of the setup. Chromium atoms are deposited onto the nanotube resonator in a Joule evaporator and the mass of the atoms adsorbed on the nanotube is measured. (b) Scanning electron microscopy image of the nanotube resonator.

- Several devices, Q=50...200, (300K) // Q=800...2000 (20K), unknown reason
- Evaporizing Cr (high bindig energy), as slowly as poss.
- Evap. step: (1,5s), 160 zg Cr=1860 Cr atoms
- T=300K, vacuum

### 2008. mass responsivity



- Theor. Estimate is the same order of magn.
- Exceptionally stabil regions

**Figure 3.** Mass sensing experiment. (a) Resonance frequency measured sequentially at 300 K and  $\sim 5 \times 10^{-6}$  mbar.  $f_0$  shifts down stepwise each time  $\sim 160$  zg of chromium is evaporated onto the nanotube.  $f_0$  is obtained by measuring  $I_{\text{mix}}$  as a function of frequency, such as in Figure 2c. The time between two points is 40 s except when chromium is evaporated (5 min). We notice that the resonance frequency does not drift in time between two evaporations, so it is not a thermal effect. (b) Resonance frequency as a function of the mass evaporated on the nanotube.

### 2008, mass resolution



**Figure 4.** Mass resolution. (a) Resonance frequency measured sequentially at 300 K and  $\sim 5 \times 10^{-6}$  mbar in the evaporator. Each  $f_0$  is obtained in 40 s by measuring  $I_{\text{mix}}$  as a function of frequency, such as in Figure 2c. (b) Resonance frequency measured sequentially at 5 K and  $\sim 1$  mbar in helium gas in a cryostat. We estimate the motion amplitude to be  $\sim 10$  nm by comparing on- and off-resonance  $I_{\text{mix}}$  signals in conjunction with eq  $3.^{12} V_{\text{g}}^{\text{ac}} = V_{\text{SD}} = 7.5$  mV and  $V_{\text{g}}^{\text{dc}} = 6.8$  V. Notice that  $f_0$  changes with temperature; we speculate that this is due to temperature-induced tension. Q is 865 at 5 K and 50 at 300 K.

standard deviation of the resonance frequency  $\sigma_{f_0} = \langle (f_0{}^i - \langle f_0 \rangle)^2 \rangle^{1/2}$  is 280 kHz (Figure 4a). By use of the above  $\mathcal{R}$ , this corresponds to a mass resolution of 25 zg. When the nanotube resonator is placed in a cryostat (without the possibility of evaporating a metal),  $\sigma_{f_0}$  is reduced to 15.7 kHz, which corresponds to a resultion of 1.4 zg or about 15 atoms of Cr (Figure 4b).

- Mass resolution: 0.1 zg (1Cr atom!)
- Reason: charge fluct. in readout circuit, stabil plateau: non-moving charge centers
- In situ reset: few microampers for a few mins (no loss in sensitivity)

### Ways to improve

• Theoretical max. (mass of a n!): 1yg, with B=10Hz

$$\left\langle z_{\rm th}^2 \right\rangle = \frac{k_{\rm B}T}{m_{\rm eff}(2\pi f_0)^2}$$

Cooler, cleaner, without charge fluctuation, low pressure

$$\sigma_{f_0}^{2} \simeq \frac{\langle z_{\rm th}^{2} \rangle}{z_0^{2}} \frac{f_0}{Q} \frac{B}{2\pi}$$



# 2012

- Putting everything together:
- Short tube: 150 nm
- Low-noise motion detection
- He temperature
- 3×10<sup>^</sup>-11 mbar (3200 s between impinging events)
- Current annealing (8 microA, 300s), also used as a reset, for 9 months, only a minor loss in sensitivity
- Hopping on contaminating molecules between trapping sites

### 2012, 2yg mass resolution



**Figure 2 | Measuring mass resolution.** Standard error of the resonance frequency (left axis) and corresponding mass resolution (right axis) as a function of averaging time at 5.5 K. The red dashed line corresponds to the mass of one hydrogen atom. Inset: resonance frequency as a function of time. The amplitude of the applied FM voltage is 4 mV. The current-frequency conversion used in the computer-controlled feedback loop is  $0.29 \text{ kHz pA}^{-1}$ .

The current annealing cleaning process allows us to achieve a mass resolution of  $1.7(\pm 0.5)$  yg. The inset of Fig. 2 shows that the fluctuations in  $f_0$  are remarkably low. The standard deviation  $\delta f_0$  is a function of the averaging time  $\tau$ ,

$$\delta f_0 = \left[\frac{1}{N-1} \cdot \sum_{i=1}^N \left(\langle f_i^{\tau} \rangle - \langle f_0 \rangle\right)^2\right]^{1/2}$$

where  $\langle f_i^{\tau} \rangle$  is the resonance frequency averaged over the time interval *i* with duration  $\tau$ ,  $\langle f_0 \rangle$  is the resonance frequency averaged over the whole measurement and *N* is the number of time intervals (Fig. 2). Using  $\delta m = 2m_{\text{eff}} \delta f_0 / f_0$  together with the effective mass  $m_{\text{eff}} = 3(\pm 0.8) \times 10^{-19}$  g (estimated from the length and diameter of the nanotube; Supplementary Section B), we obtain a mass resolution of  $1.7(\pm 0.5)$  yg after 2 s averaging time. This ultralow value corresponds to approximately the mass of one proton (1.67 yg). The error in  $\delta m$  reflects the uncertainty in the estimation of the length and the diameter of the nanotube, obtained by atomic force microscopy (AFM).



Figure 3 | Adsorption of xenon atoms and naphthalene molecules.

**a**, The change in the resonance frequency  $\Delta f_{\Omega}$  as a function of time at 6 K as xenon atoms are being dosed onto the nanotube. Red arrows indicate some of the abrupt upward shifts discussed in the main text. The shaded area corresponds to the time when xenon atoms are dosed. Xenon atoms arrive directly from the microdoser onto the nanotube so the dosing rate cannot be estimated (Supplementary Section A). The resonance frequency is obtained by continuously measuring  $I_{mix}$  as a function of f. **b**,  $\Delta f_{\Omega}$  versus time at 4.3 K when naphthalene molecules are being dosed. Red arrows point to the shifts of the resonance frequency consistent with the adsorption of  $C_{10}H_8$  molecules. The resonance frequency is measured using the computer-controlled feedback loop; the current-frequency conversion is 0.4 kHz pA<sup>-1</sup>. Inset: expected shift in  $f_{\Omega}$  as a function of the position of the  $C_{10}H_{B}$  adsorption along the 150-nm-long nanotube. Xenon atoms and  $C_{10}H_{B}$ molecules are admitted from a gas reservoir at 300 K into the vacuum chamber through a pinhole microdoser. In the case of C10H8 dosing, unwanted contaminants are removed by freeze-pump cycles of the gas reservoir before adsorption experiments<sup>29</sup>.

- Xenon: upward shifts: single desorption events
- Naphtalene: downward shifts: single adsorption events (2 orders of magn. better than before)
- Trapping position dependent (en route to improve with a trapping site)

# Summary

- SWNT-s for mass sensing: stiff, light-> precise
- Resettable, last for months, easy to excite, easy to read
- Mass resolution of neutronmass