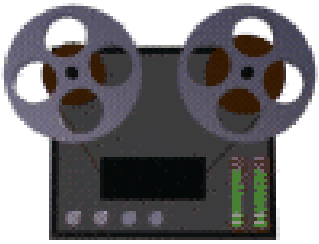
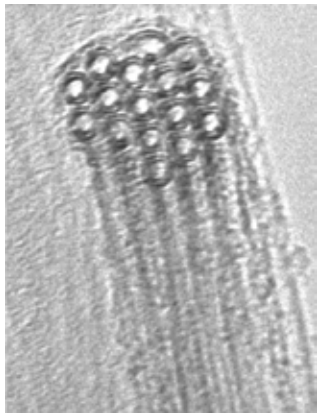
Instrumentation and Metrology

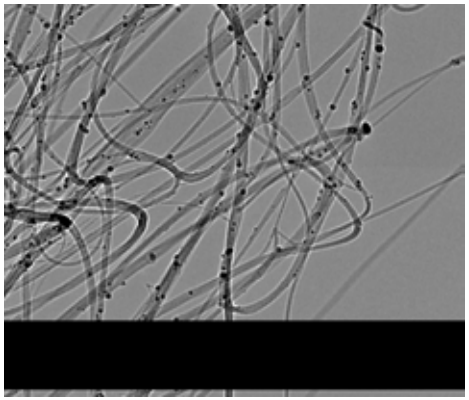
Horacio Espinosa
Department of Mechanical Engineering
Northwestern University

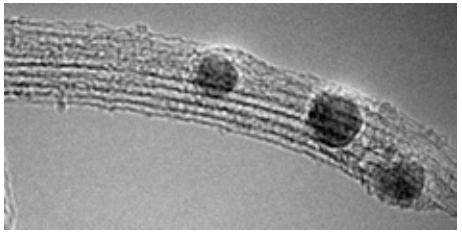
Problem Definition: Manufacturing of CNTs



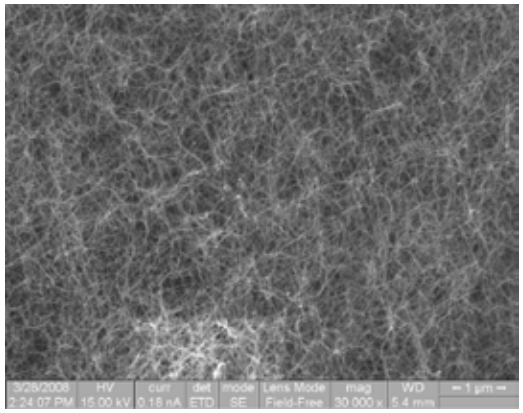


Several
10s of nm





Submicron to μm

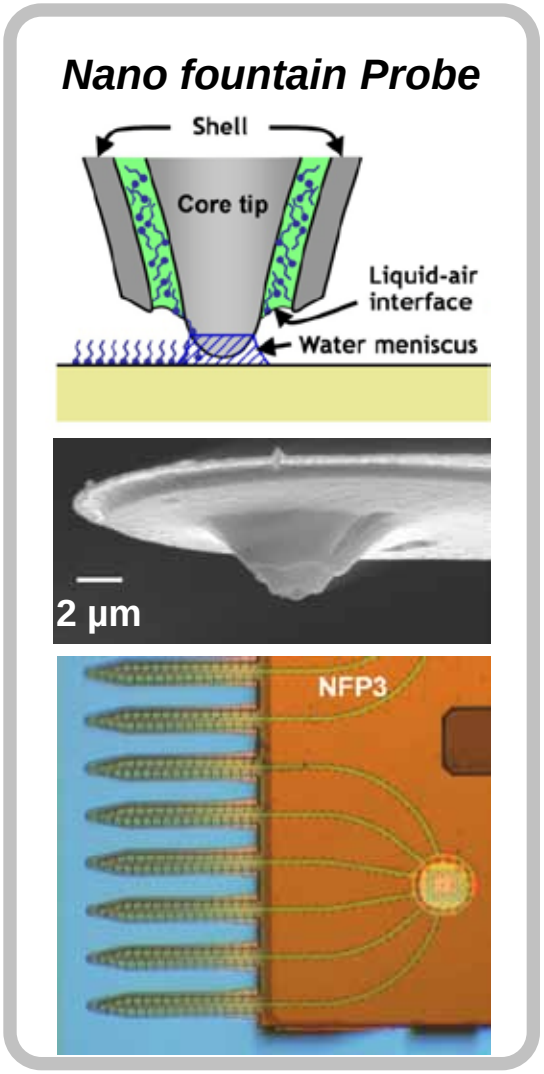


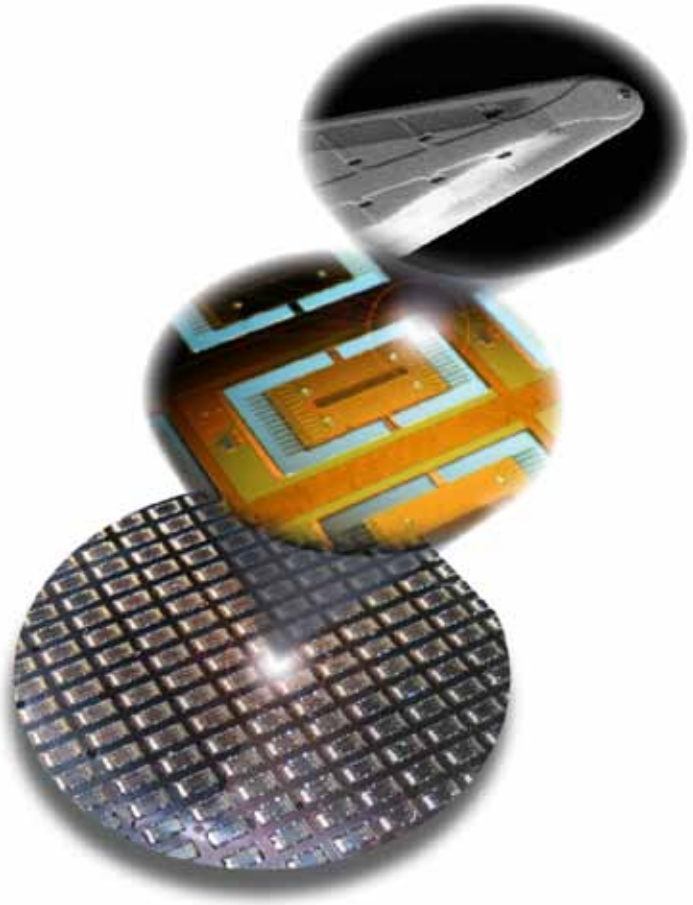
Entangled network
of CNT bundles

Nanotechnology Tools:

- **Characterize Effects of NTs and NPs**
- **Chemical Composition with nm spatial resolution**

Nanofauntain Probes





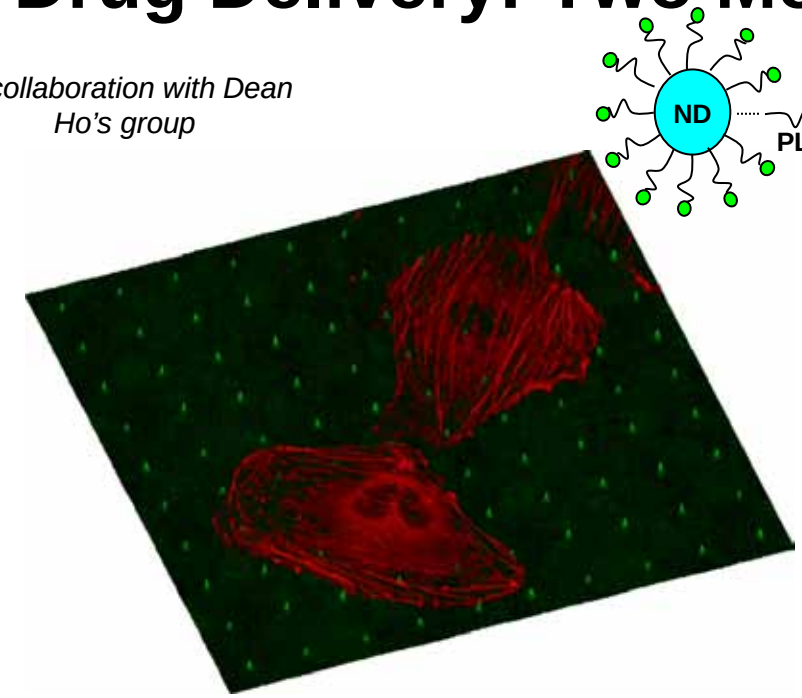
Develop a microsystem platform for mass production of nanoscale devices, sensors and structures using chemicals, biomolecules, nanoparticles, nanotubes and nanowires.

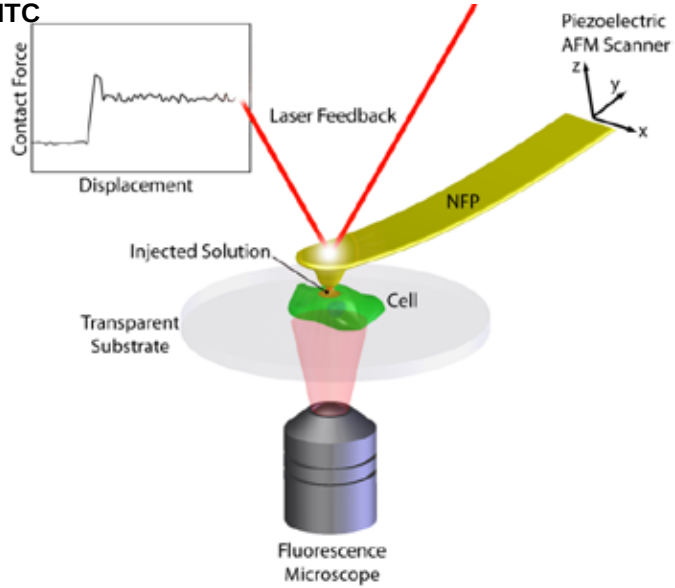
Continuous Ink Feeding

High Throughput

Studying Nanomaterial-Mediated Cancer Drug Delivery: Two Methodologies

In collaboration with Dean Ho's group



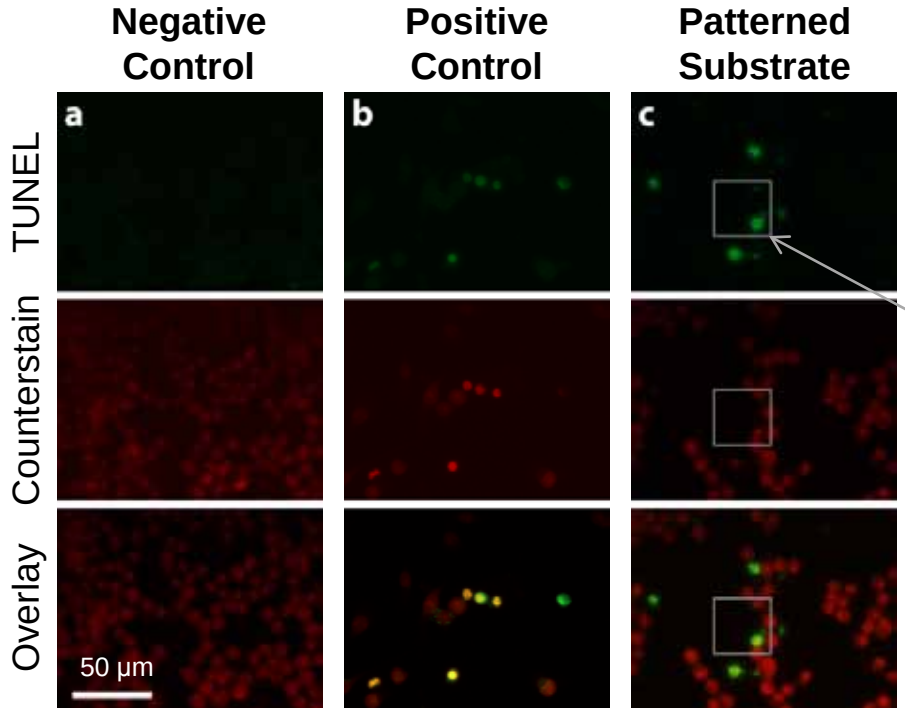


1. Direct-Write Nanopatterning:
Cells cultured on substrates patterned with drug-coated nanoparticles (10^{-24} gram dosing resolution) and their response observed

2. Single Cell In Vitro Injection:
Functional nanoparticles injected directly into individual cells

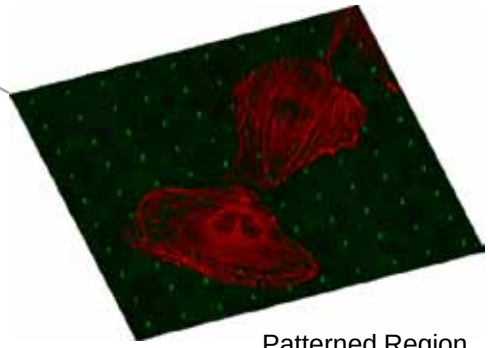
[Loh, Espinosa, *et al. Small*, 5(14), 2009]

Direct Deposition of Drug-Conjugated Nanoparticles – *Preserved Drug Activity*



In collaboration with D. Ho's Group

[Loh, *et al. Small*, 5(14), 2009]

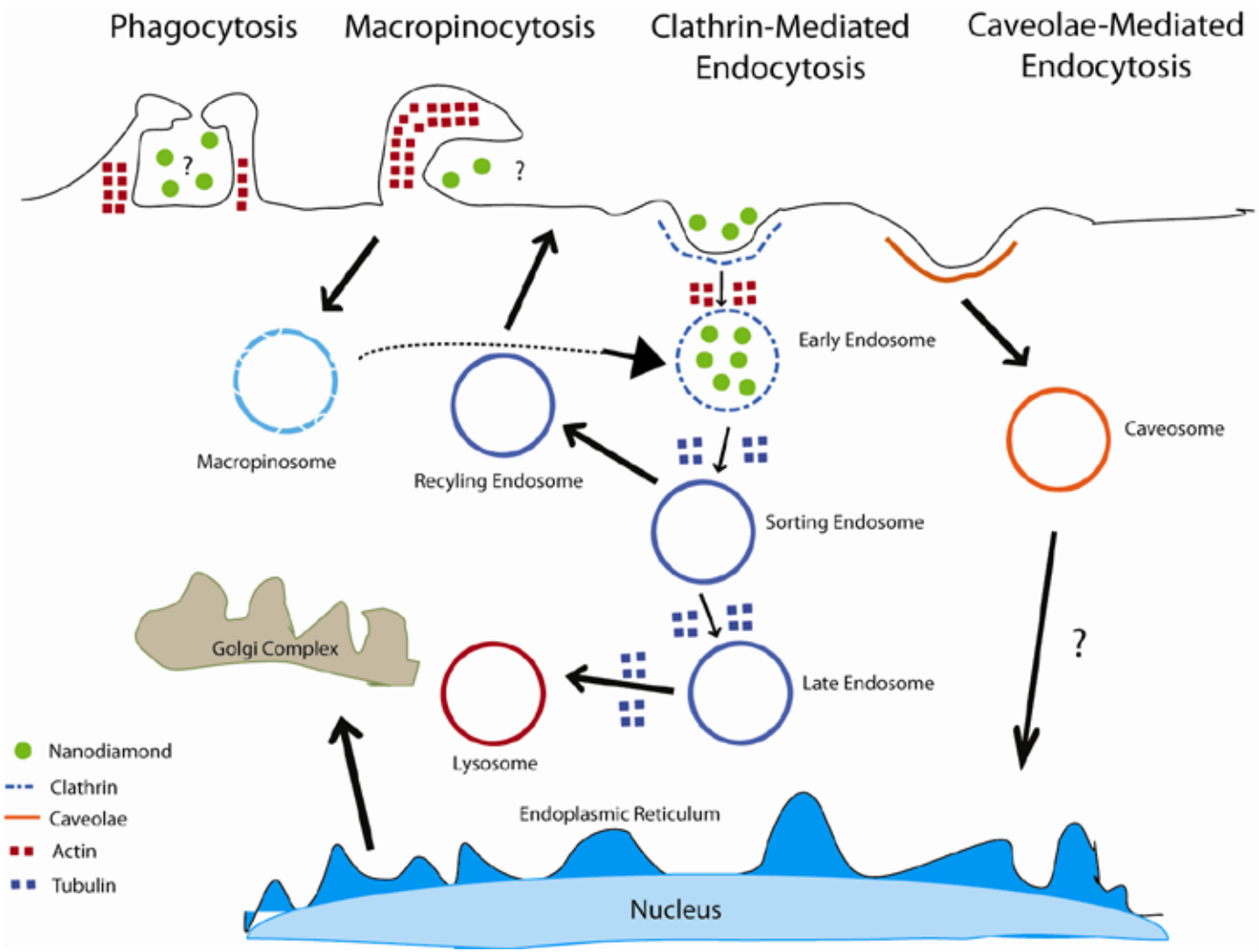


Patterned Region

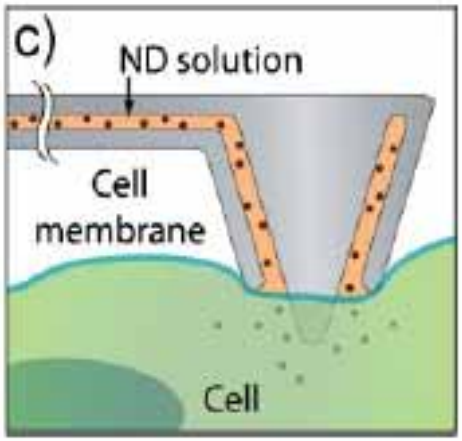
Murine macrophages incubated 24 hrs

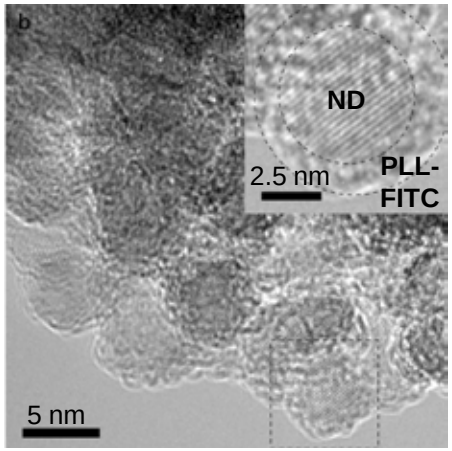
- Doxorubicin HCl: commonly-used apoptosis-inducing chemotherapy drug
- Tested preserved activity of the drug after nanopatterning (*TUNEL assay to detect DNA fragmentation and apoptosis)*

Internalization Pathways: Endocytosis

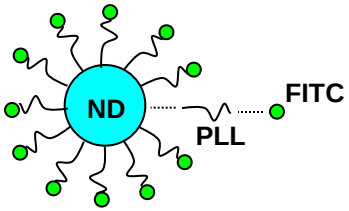


In Vitro Direct Injection – *Single Cell Studies*



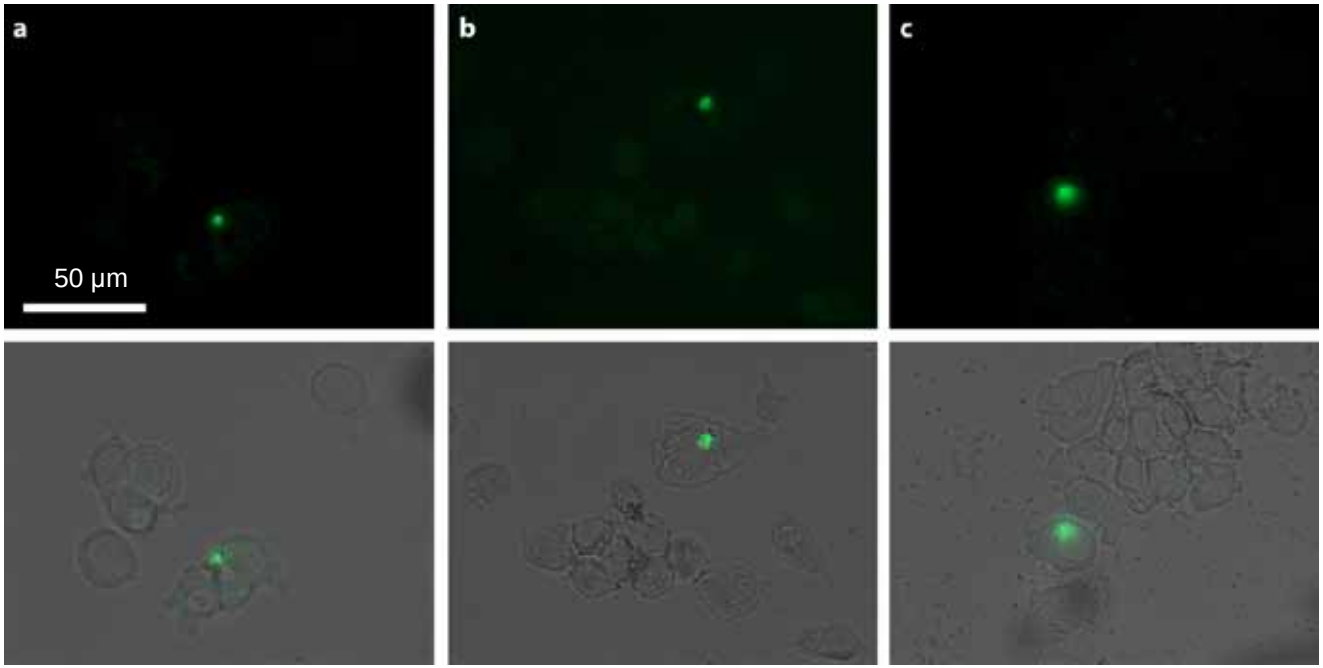


TEM of coated nanodiamonds



FITC-Labeled Nanodiamond Injection

Single Cell Injection



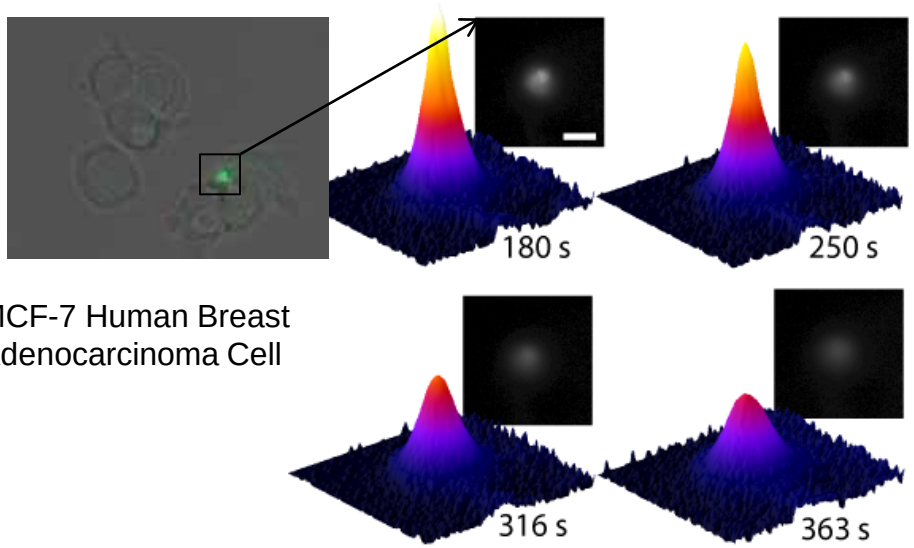
MCF-7 Human Breast Adenocarcinoma Cell (5 sec injection)

RAW 264.7 Murine Macrophage (10 sec injection)

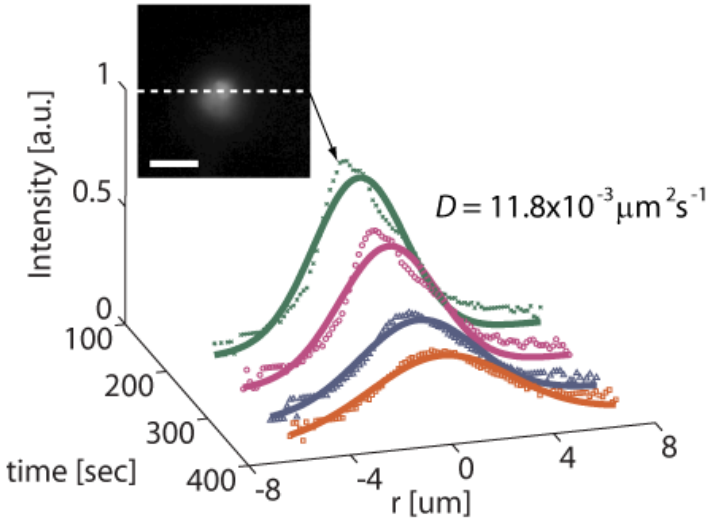
RKO Colorectal Carcinoma Cell (15 sec injection)

[Loh, Espinosa, *et al. Small*, 5(14), 2009]

In vitro Injection: Diffusion of Diamond Nanoparticles in Cells



MCF-7 Human Breast Adenocarcinoma Cell



[Loh, Espinosa, *et al. Small*, 5(14), 2009]

Calculating diffusion coefficient:

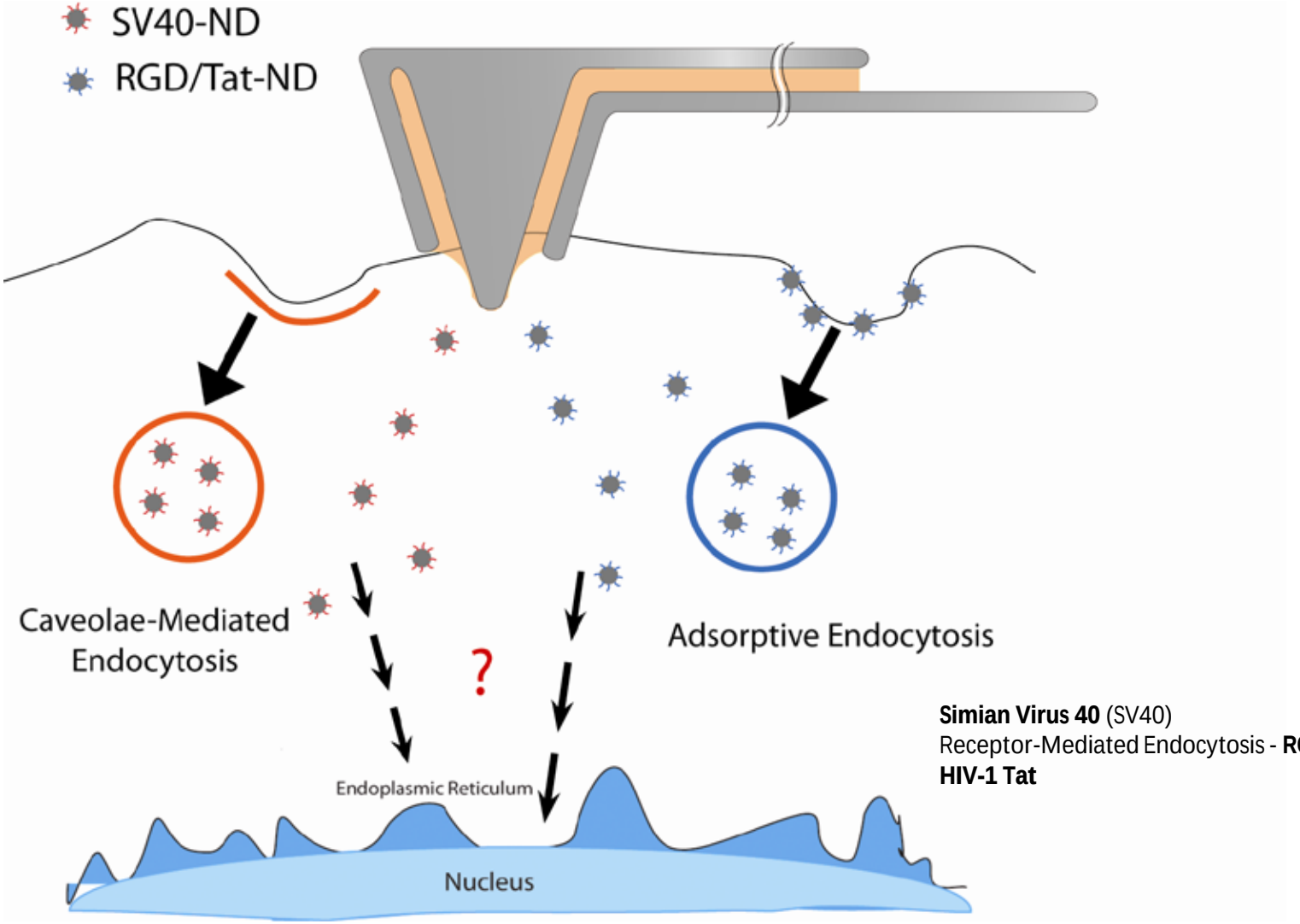
Fit Gaussian at each time t_i, $\exp\left[-\left(r / g_i\right)^2\right]$

This yields a series of estimates for γ_i.

Linear regression yields D: $g_i^2 = 4Dt_i$

D = 11.8x10^{-3} ± 0.2 μm^{2}s^{-1} for 4 to 8-nm NDs (versus D = 3.1x10^{-3} μm^{2}s^{-1} for 35-nm NDs, Chang *et al.* 2008)

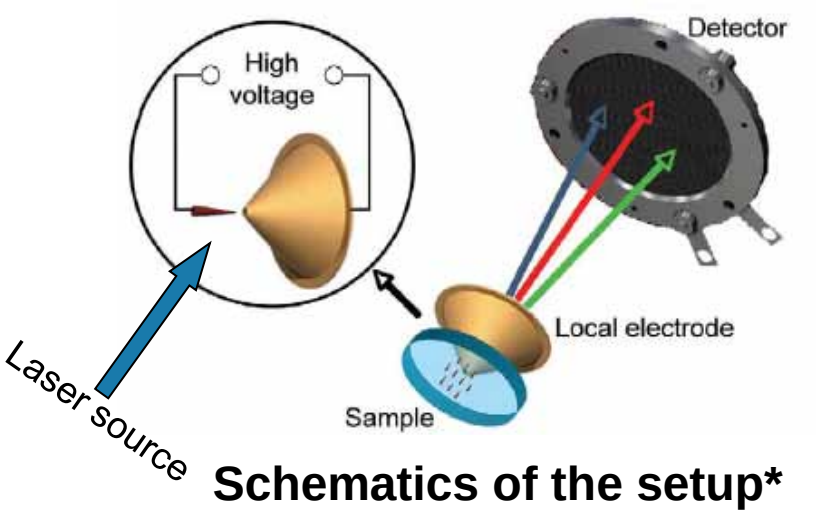
NFP-Mediated Studies



Simian Virus 40 (SV40)
Receptor-Mediated Endocytosis - **RGD**
HIV-1 Tat

Chemical Composition of NWs

Local Electrode Atom Probe (LEAP)



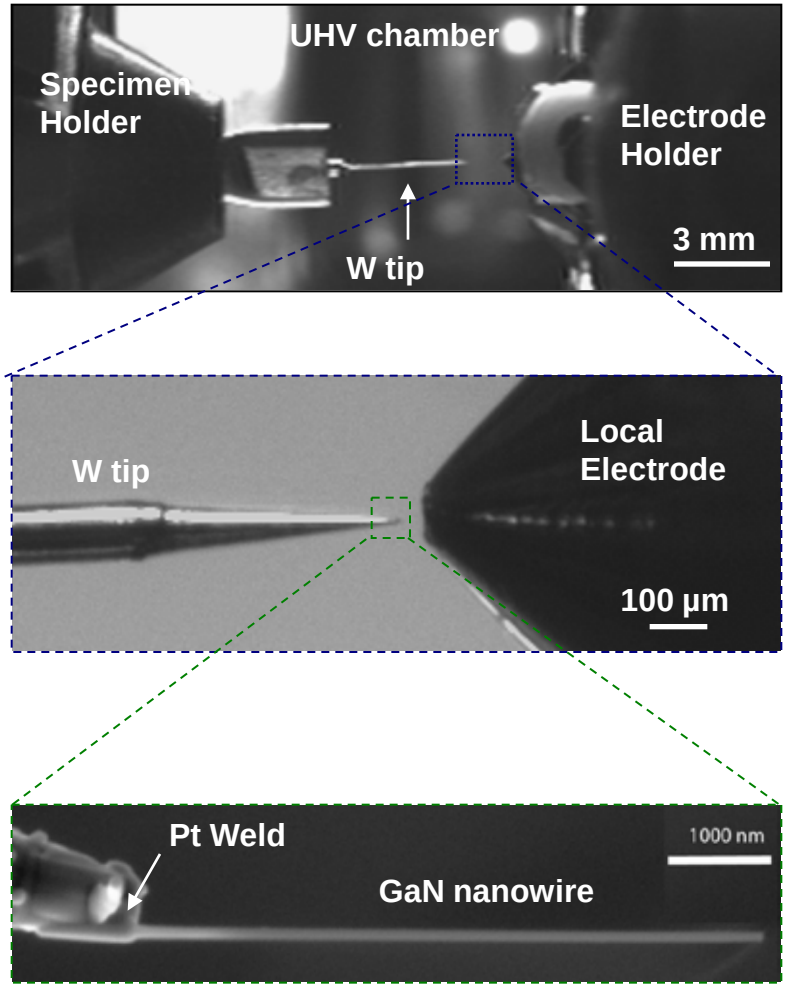
Schematics of the setup*

- A technique to get atomic composition in nanomaterials with ppm resolution
- A sharp tip (<200 nm in diameter) is aligned in front of a local electrode
- Analysis chamber is maintained at cryogenic temperatures (20-60K) at ultrahigh vacuum (~10^{-11} torr)
- A bias (~3-10 kV) is applied between the tip and the electrode to provide directionality to the evaporated atoms
- Atoms are evaporated by exciting them with a laser of given energy (0.1 – 100 pJ) and frequency (50 kHz – 1 MHz)
- Evaporated atoms then hit the detector and are identified based on their time of flight
- The 3D reconstruction is done based on their position on the position-sensitive detector

Image of the entire setup from IMAGO

Cerezo et al., Materials Today, 10, 12, 2007

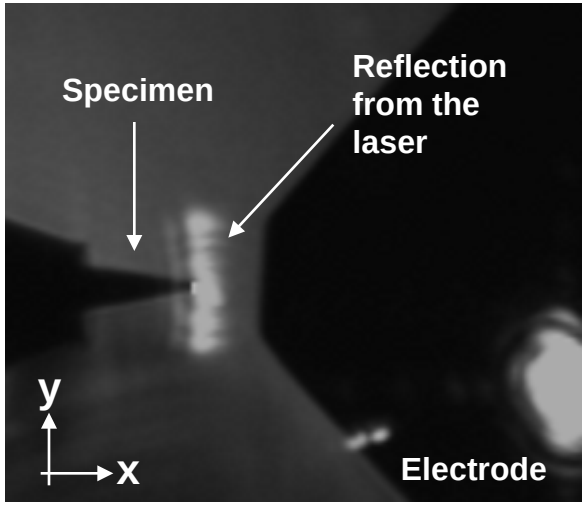
Sample Preparation and Mounting

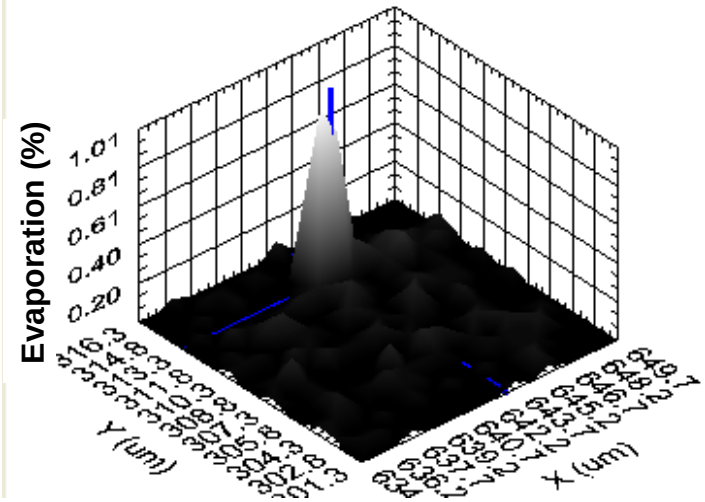


Customized set-up for nanowires

- Sharp needle like tips with diameter less than 200 nm
- Typical methods for specimen preparation
  - Electropolishing for metallic specimens
  - Focused-ion beam (FIB) sharpening
- For nanowires
  - Epitaxial growth on the microposts
  - Nanomanipulation, applicable to all materials

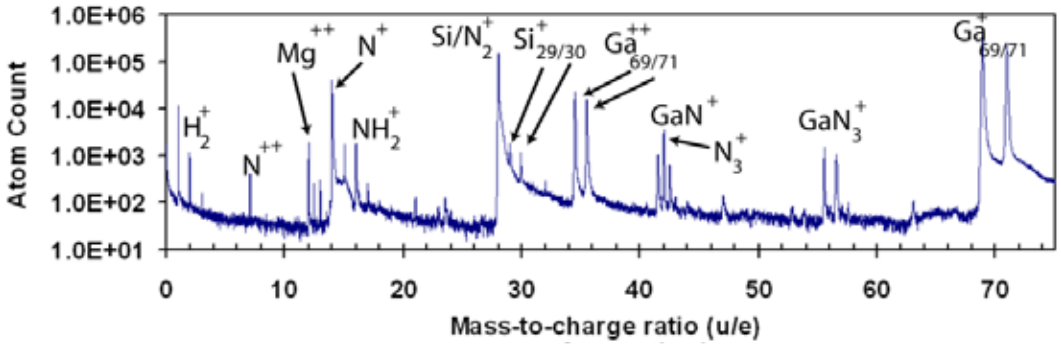
Laser alignment on the tip

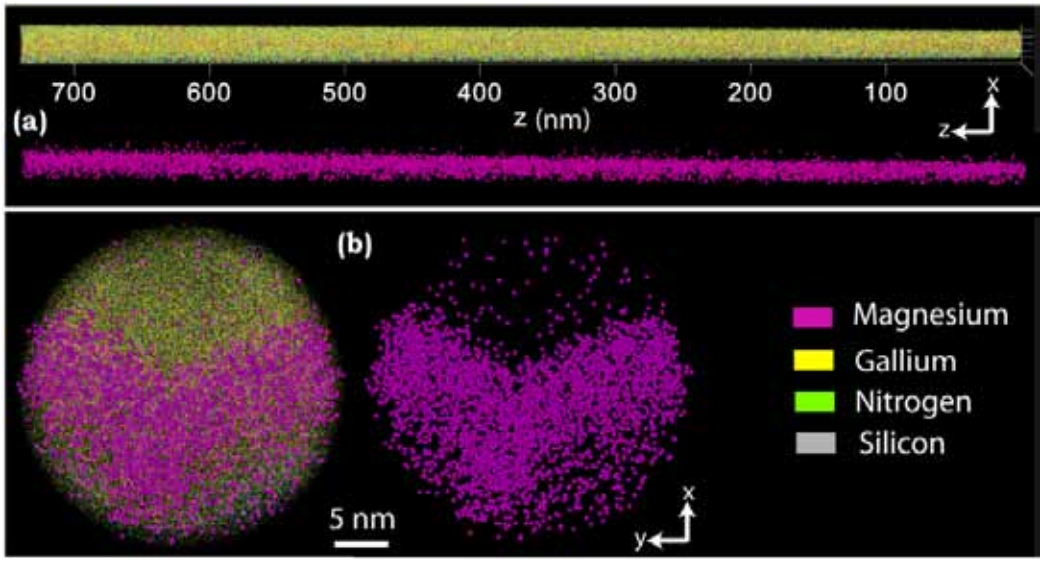




- A laser is scanned along x and y directions
- Peak evaporation is monitored to align the laser on to the tip
- Once the peak is detected, a focus scan (in z-direction) is performed to maximize the evaporation
- Frequency of the laser determines the rate of evaporation
- Atoms evaporate at each pulse of the laser and hit the position-sensitive detector (PSD)

Dopants in GaN nanowires





- Atomic species identified based on their time of flights
- Mass resolution is < 0.1 atomic mass units
- Concentrations as low as 10 ppm are identified
- Position on PSD and time of hit identifies s used to backtrack the atomic positions in the specimen for 3-dimensional reconstructions

Applicable to other materials for identifying harmful elements , for e.g. carcinogens

Questions