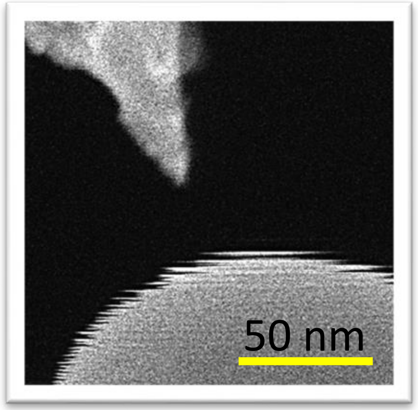
UNIVERSITY OF TARTU
Institute of Physics

INSTITUTE OF SOLID STATE PHYSICS
UNIVERSITY OF LATVIA

UNIVERSITY OF TARTU
Institute of Technology

Nanoscale experiments inside SEM

(*Real-time observation of strong electric-field-induced surface modification*)

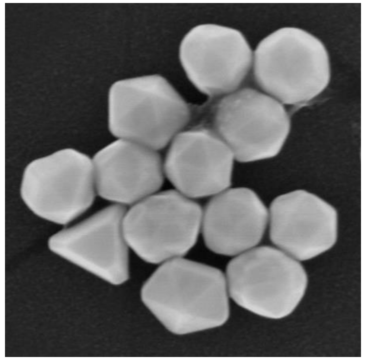
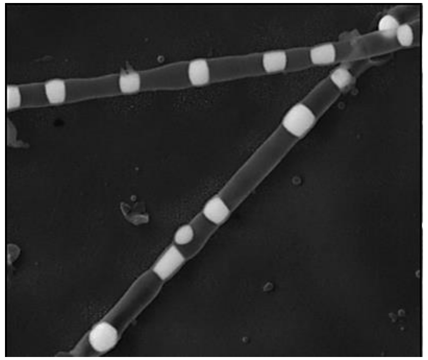


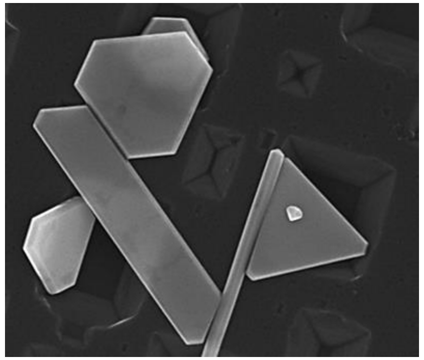
Sergei Vlassov
Institute of Physics, University of Tartu, Estonia

MATTER

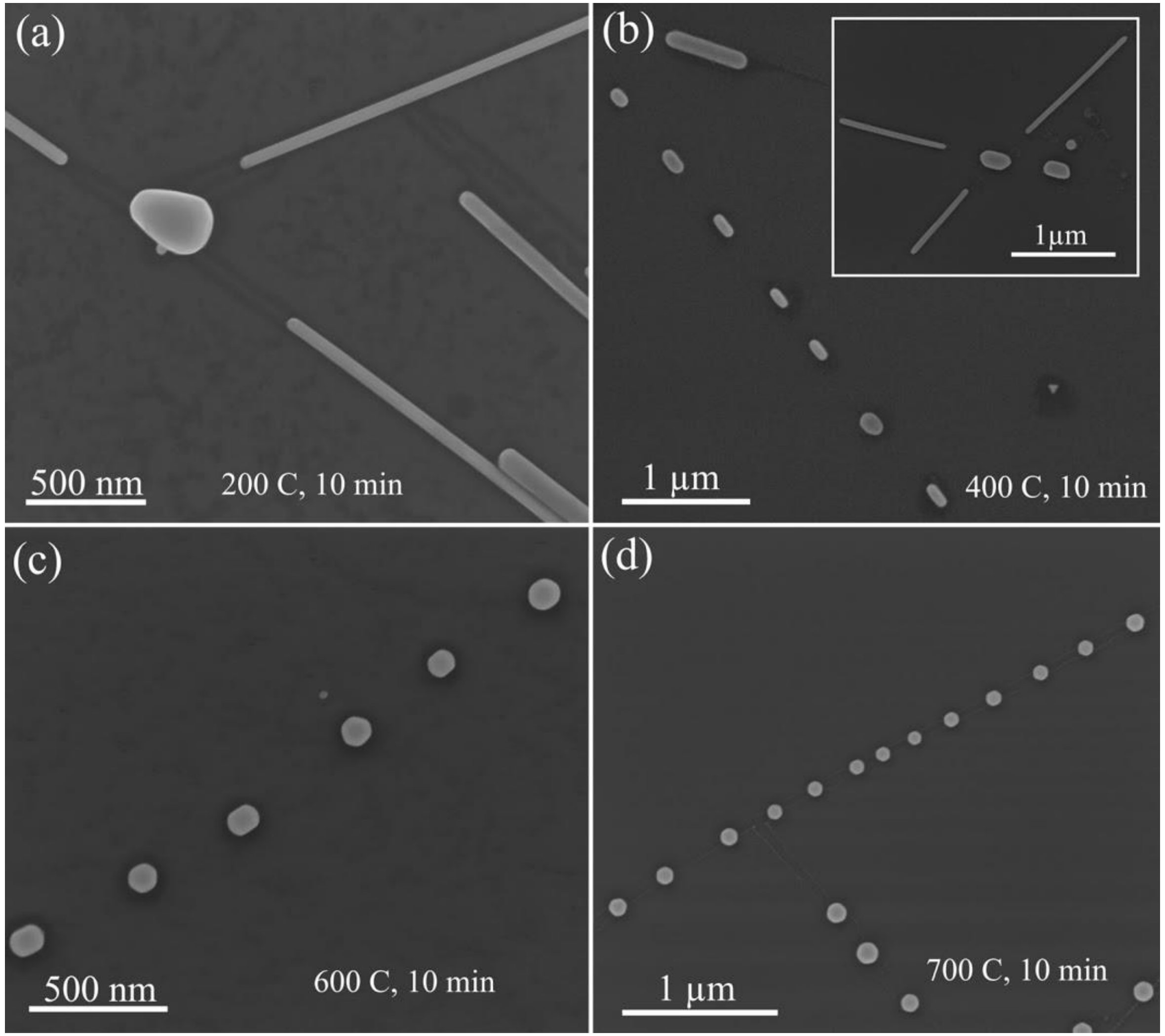
Size effect in nanostructures

- Specific properties (melting temperature, mechanical strength, thermal and electrical conductivity etc) of nanostructures depend on size.
- Reasons
  - Quantum confinement
    - The size effects arise in structures if its size *d* is reduced down to a critical value where the scale length of physical phenomenon (free path of electrons or phonons, exciton radius etc) becomes to be comparable with characteristic size (length, thickness, diameter) of building blocks of structures.
  - Surface-to-volume ratio

Example:
Melting temperature of nanostructures



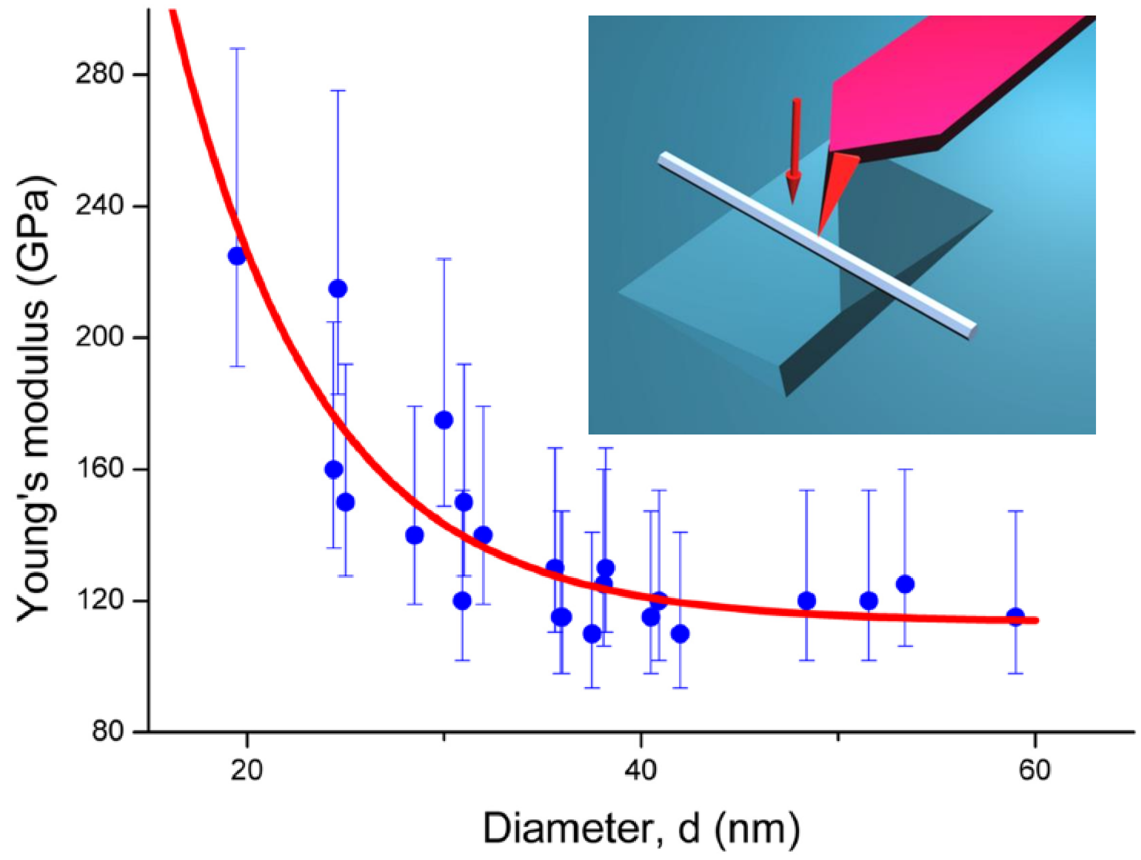
Thermal stability of Au nanowires:
effect of annealing at different temperatures.

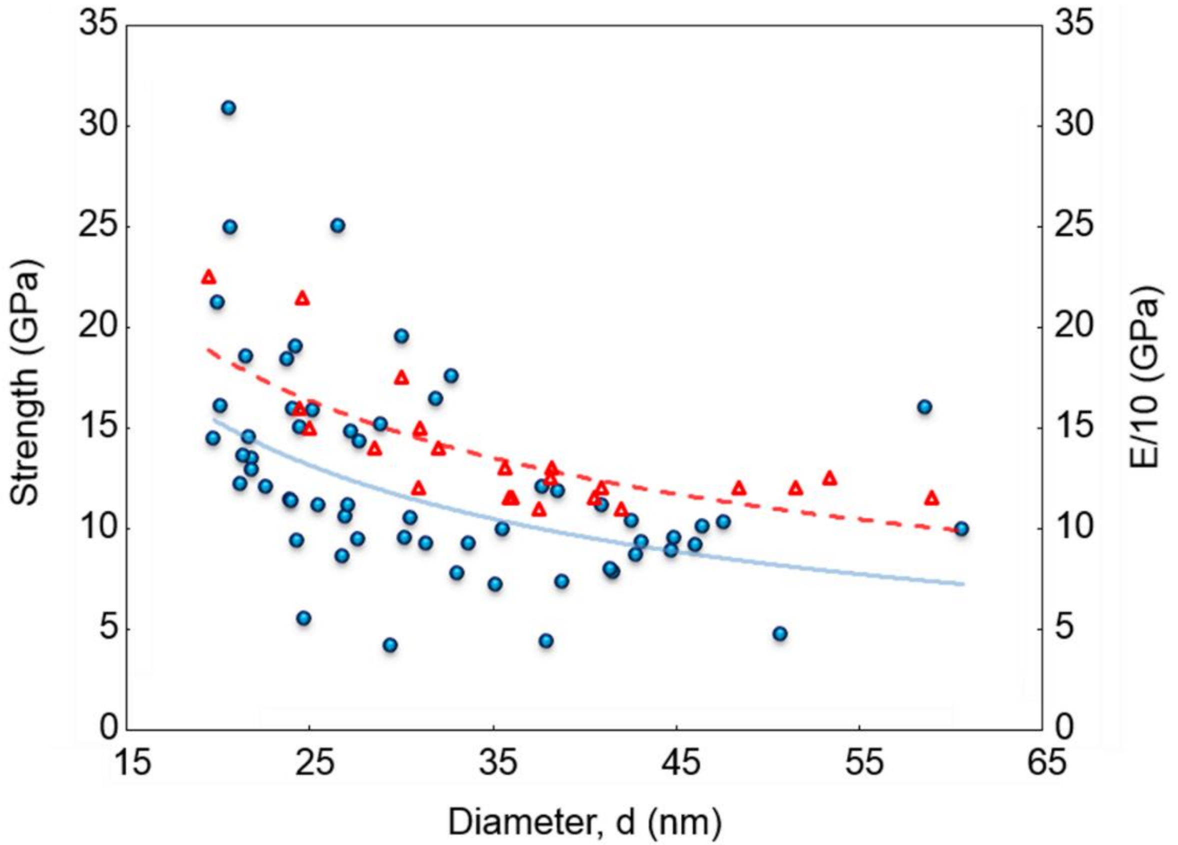
First, nanowires „melt“ at interceptions.

At higher temperatures nanowires decompose into smaller fragments due to phenomena known as **Rayleigh instability**.

Vigonski et al., (2017) ***Nanotechnology*** *29 (1), 015704*

Mechanical properties of ZnO nanowires





*Vlassov et al, **Nanotechnology**, 2016, Volume 27, Number 33*

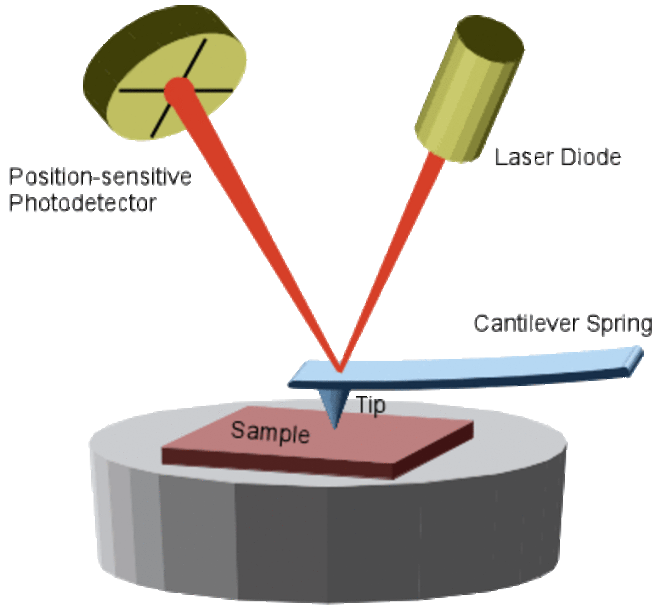
Experimentator Challenge

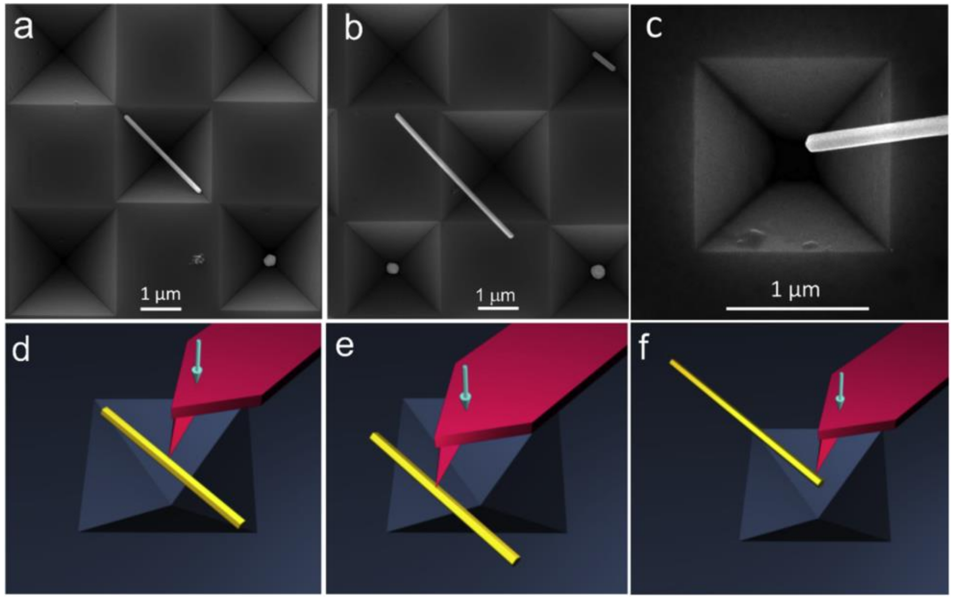
How to measure properties of <u>**individual**</u> nanostructures?

Atomic Force Microscope (AFM)

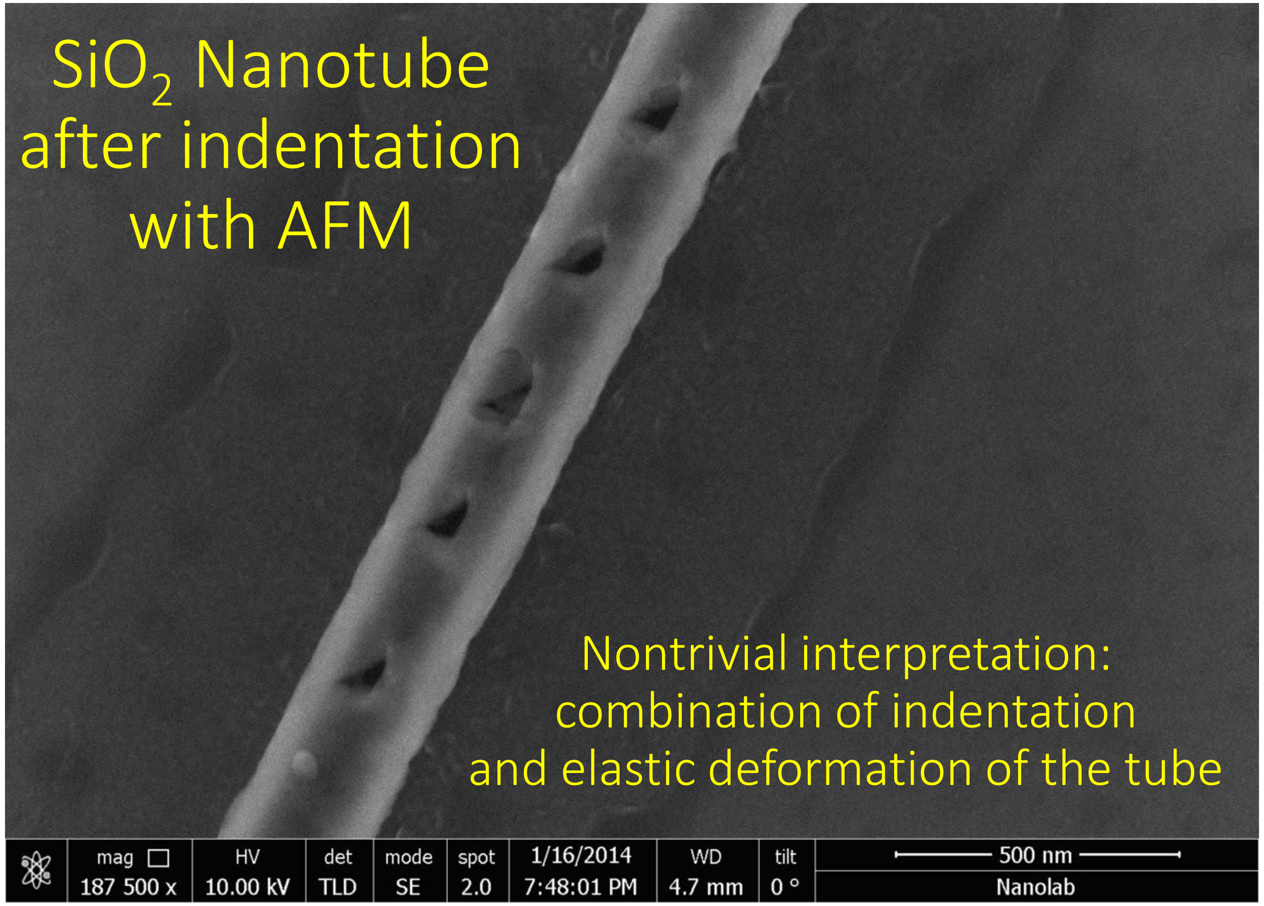
- Atomic resolution (in z)
- Extremely high force sensitivity (below nN)

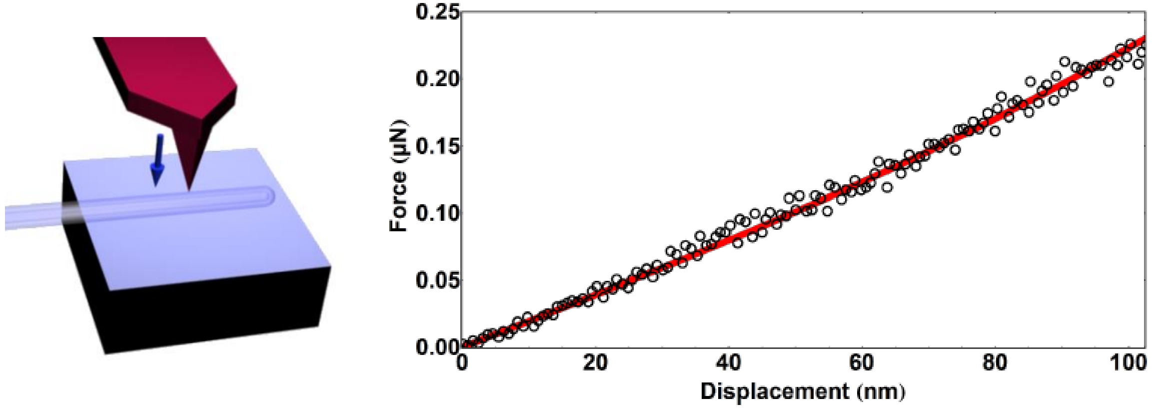
- Blind manipulation
  - Probe can be used either for visualization or manipulation, but not simultaneously
- Time-consuming
  - Scan large – scan small – measure – scan
- Only flat samples



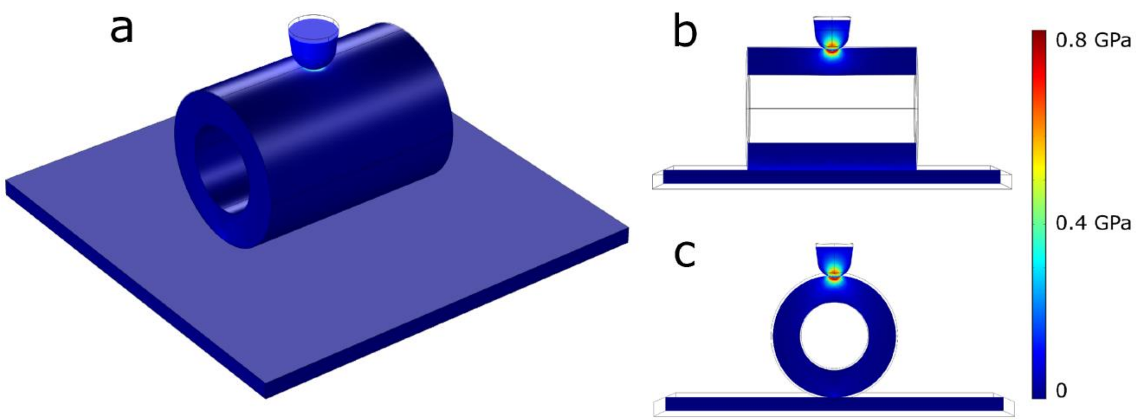


“Blind” nanoindentation with AFM





Finite Element Method to account for elastic deformation of the tube

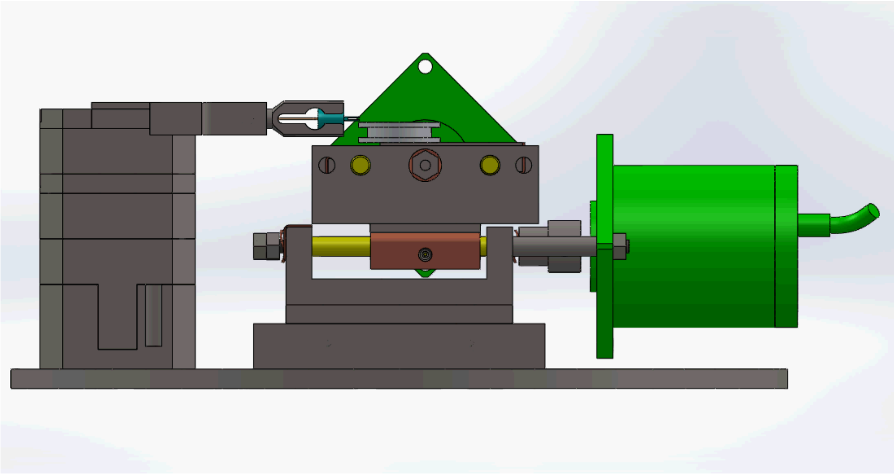


Beilstein J. Nanotechnol. **2014,** *5,* 1808–1814

Nanomanipulation platforms for in situ SEM experiments.

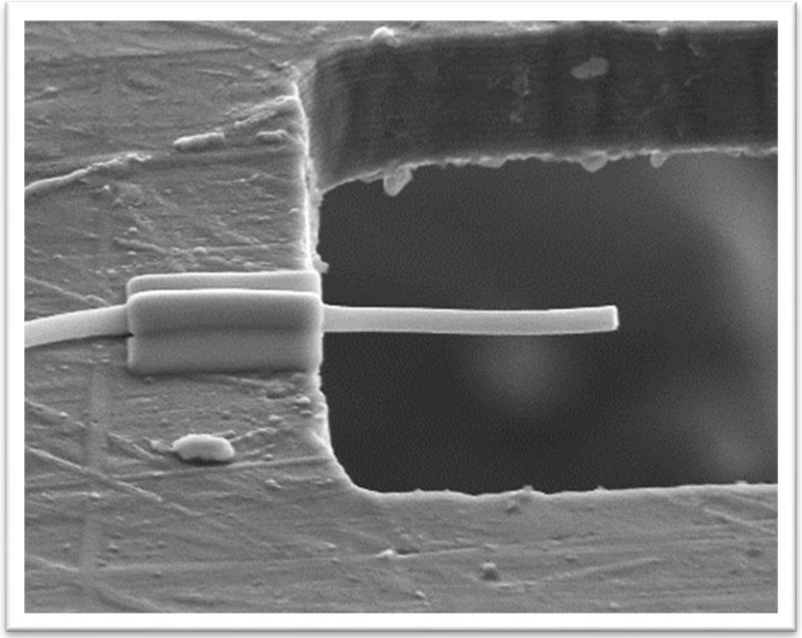


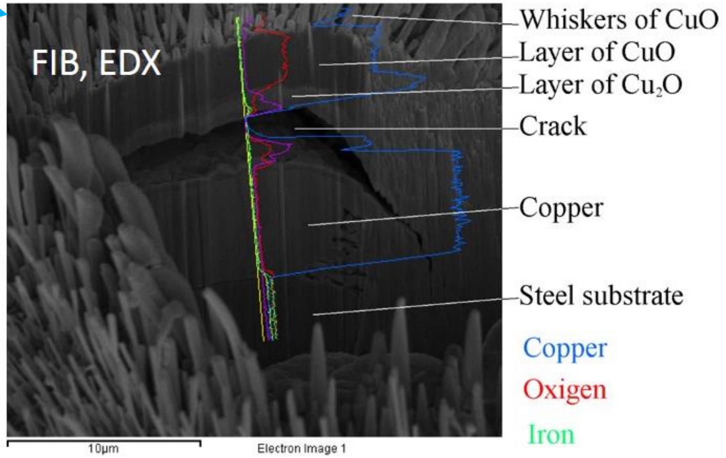

Nanomanipulation platforms based on piezo-positioners (partially commercial, partially home-built) are installed inside scanning electron microscopes allowing to perform very fine experiments with individual nanostructures



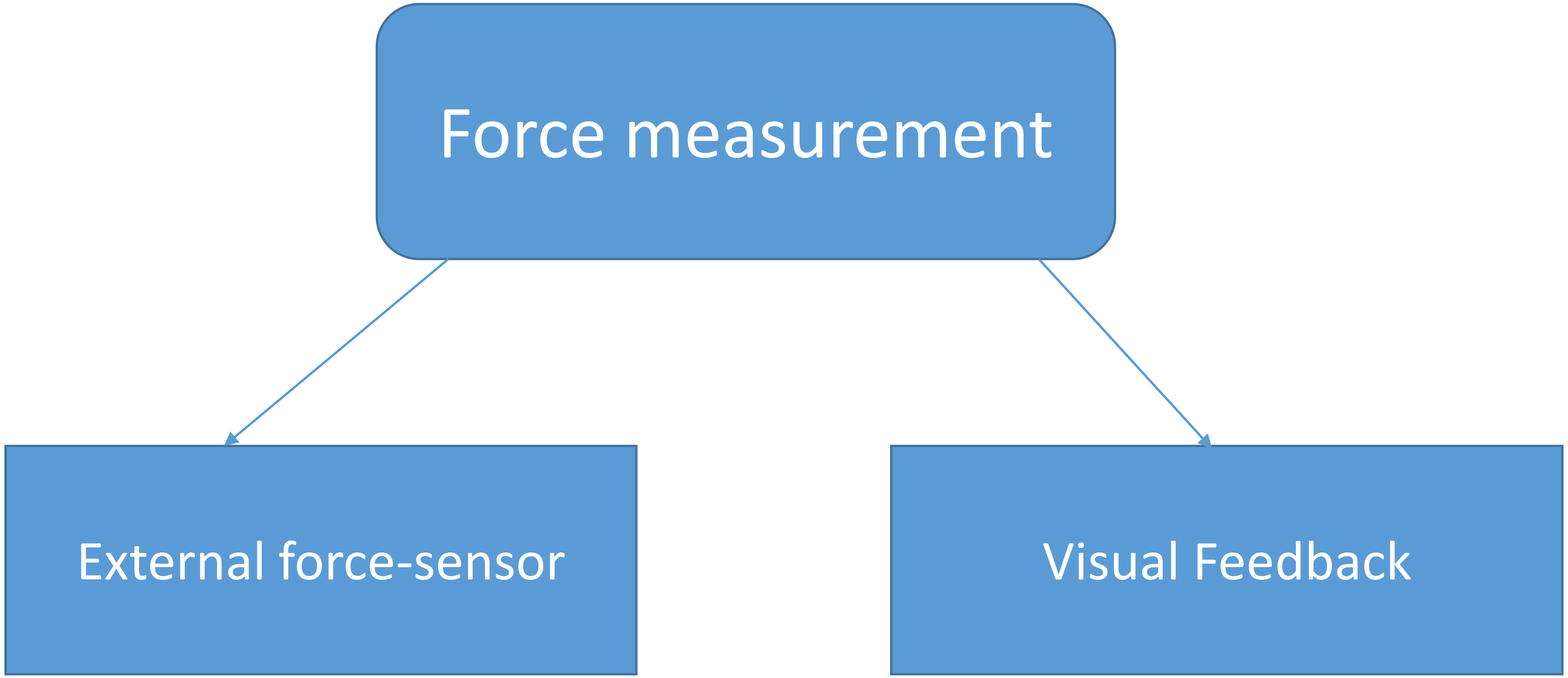
Peculiarities of inside-SEM experiments

- **Visual guidance**
- Vacuum conditions
- **Focused Ion Beam (FIB) capabilities**
  - **In situ welding**
  - **In situ cutting**
- **Elemental analysis**
- Electrical conductivity
  - (choice of materials is limited to metals and semiconductors)
- Electron-beam induced effect
  - Some materials may be sensitive to e-beam
  - Carbon deposition can occur on irradiated areas





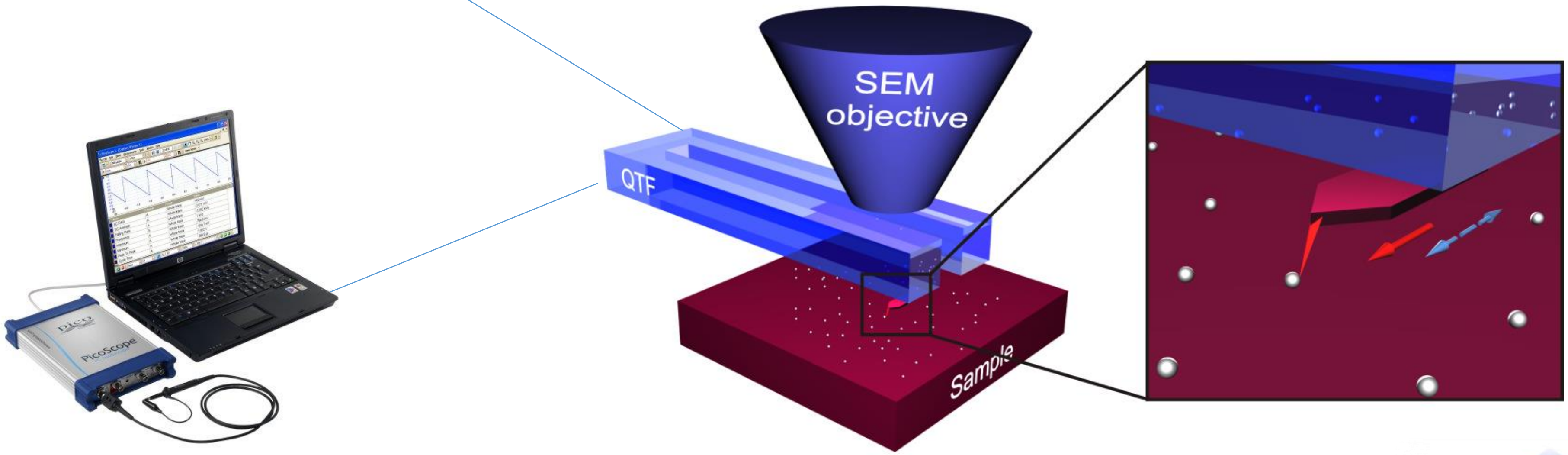
How we measure forces?

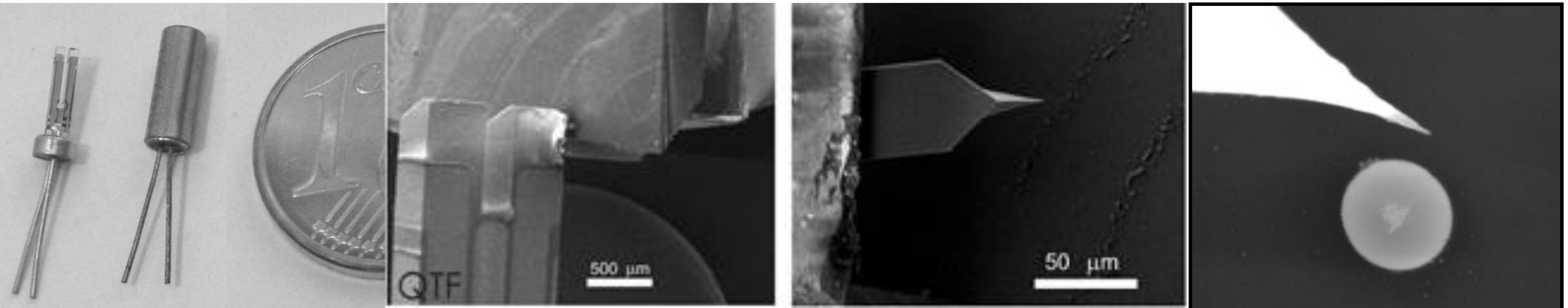


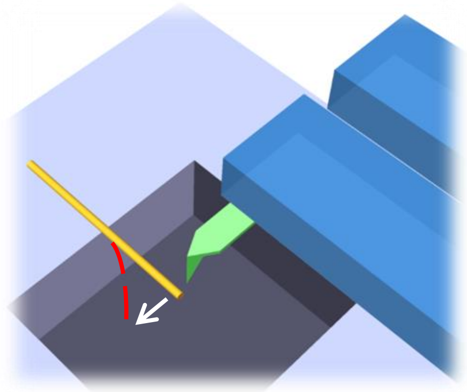
Method 1: QTF based **force sensor**

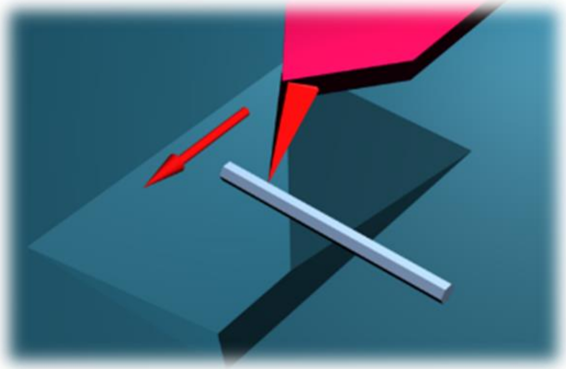
- AFM Tip glued to one prong of Quartz Tuning Fork (**QTF**).

- QTF is electricaly excited on its resonant frequency
- High sensitivity: nN range

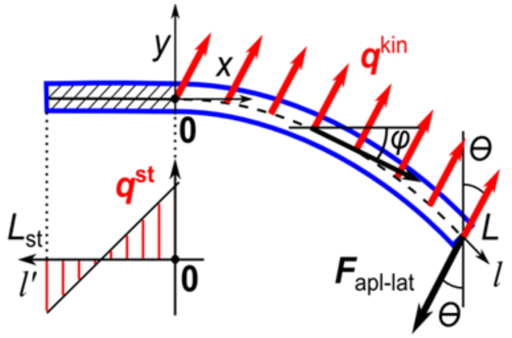




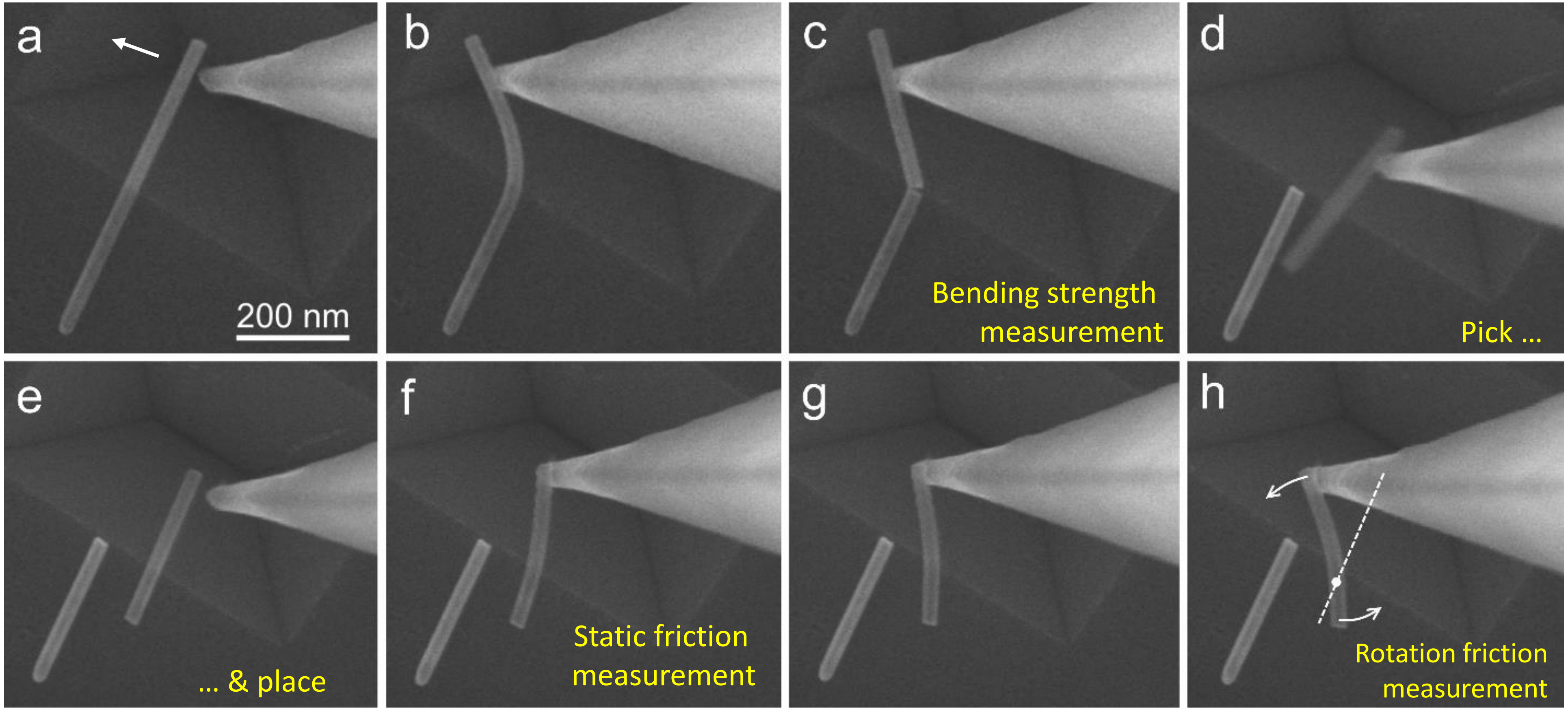




Method 2: **Visual feedback** from the deformation

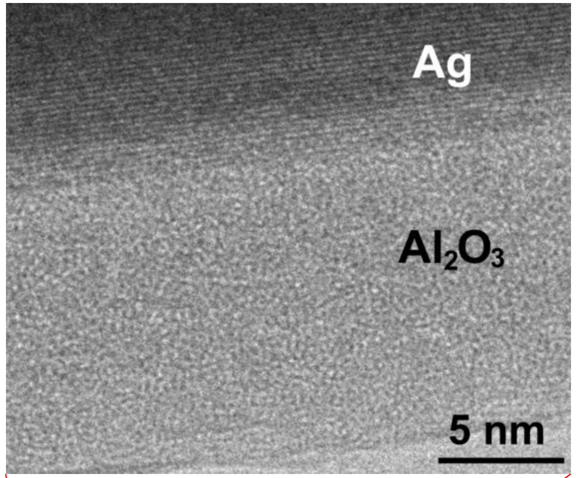


Example: Pick and place manipulation of ZnO nanowire for tribomechanical characterization

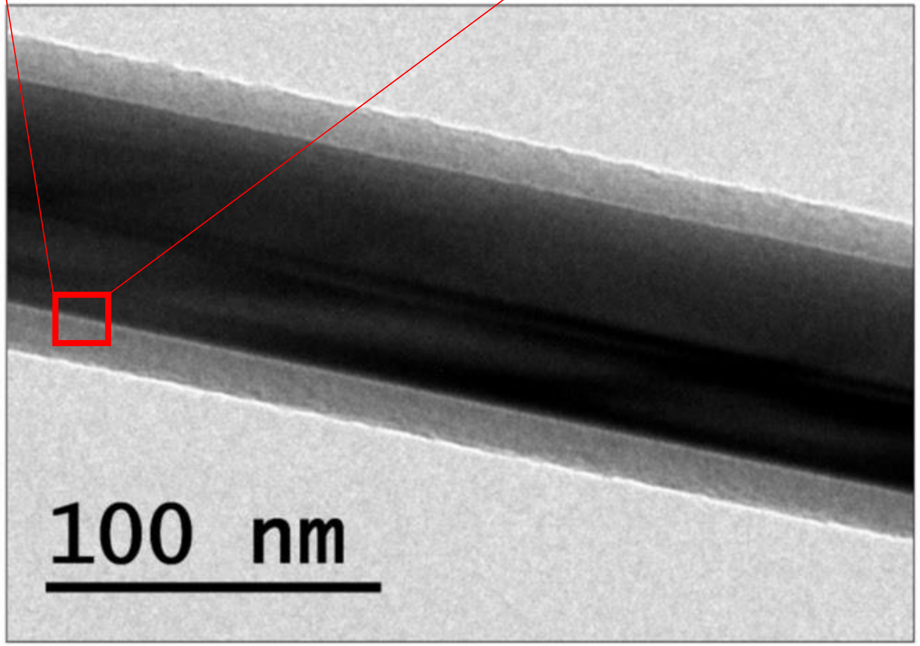


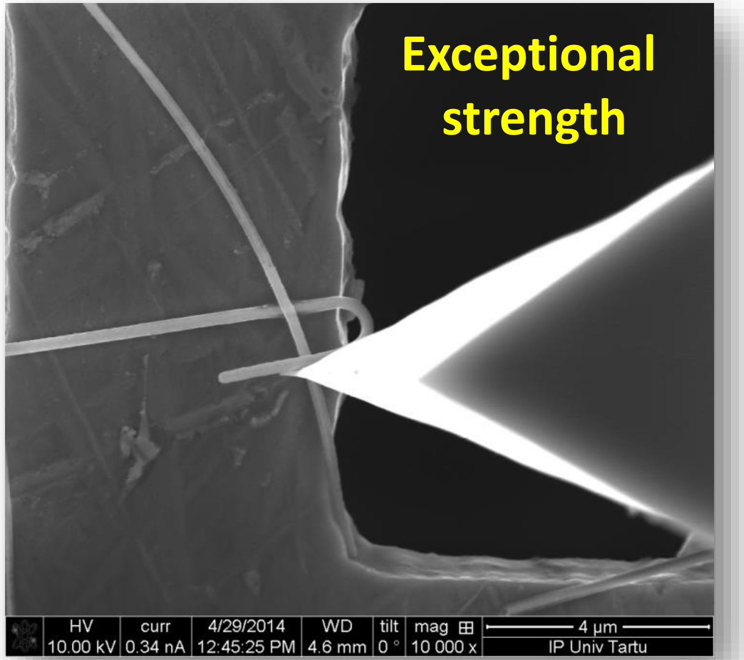
Vlassov et al., 2016, ***Nanotechnology*** *27 (33), 335701*

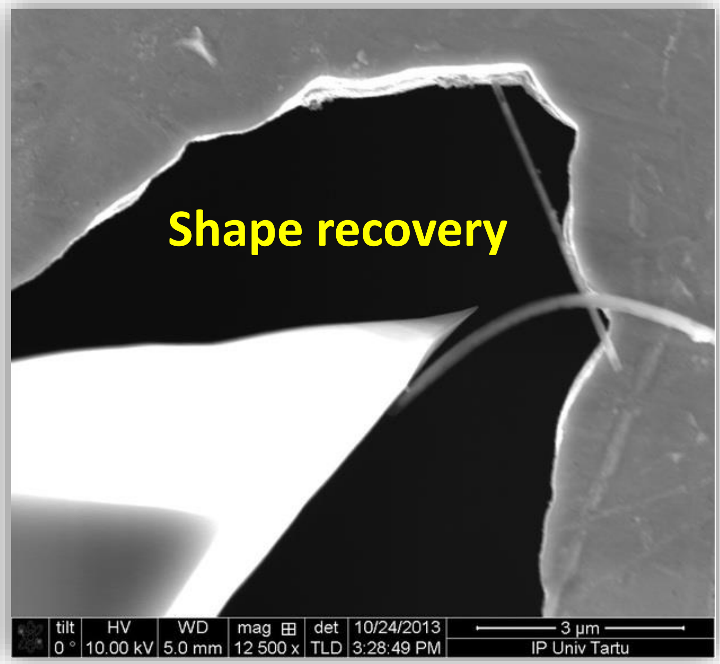
Core-shell nanowires



- Metallic core (Au, Ag) and amorphous oxide shell (SiO_2, Al_2O_3)
- Greatly enhanced mechanical strength
- E-beam activated REVERSIBLE elastic to plastic transition in amorphous SiO_2 (soft and plastic under e-beam, but hard and elastic when e-beam is off)
  - Shape recovery



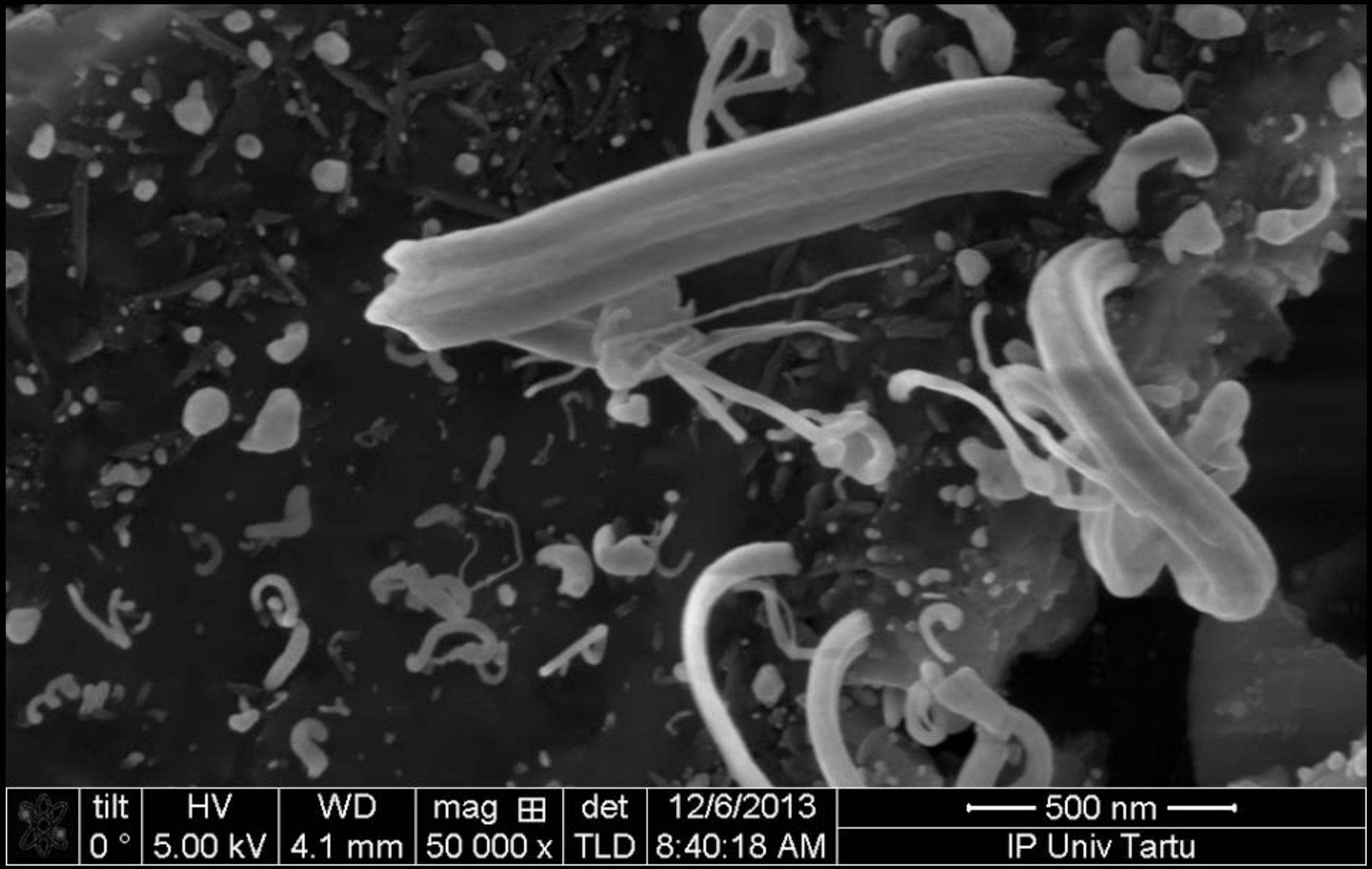




*Vlassov et. al, **Nano Lett**. 2014, 14, 5201−5205*

Vlassov et. al, 2017, Nanotechnology 28 (50), 505707

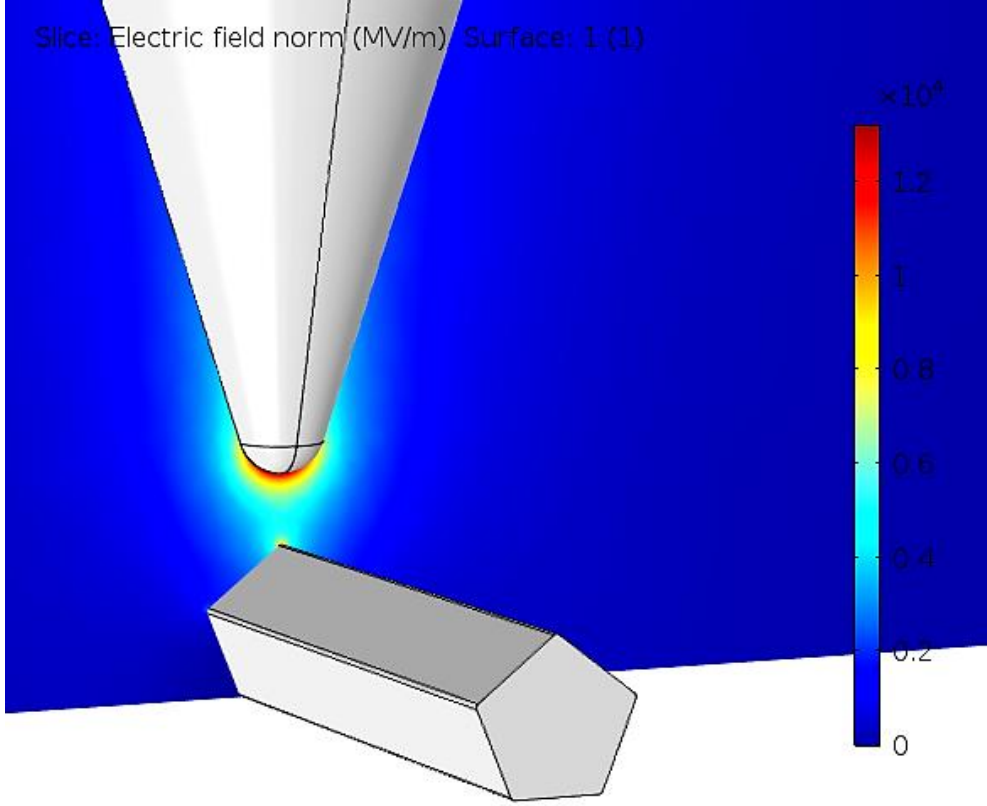
E-beam induced growth of silver nanowhiskers



https://photos.app.goo.gl/37y61kXKuvJsnkGo8

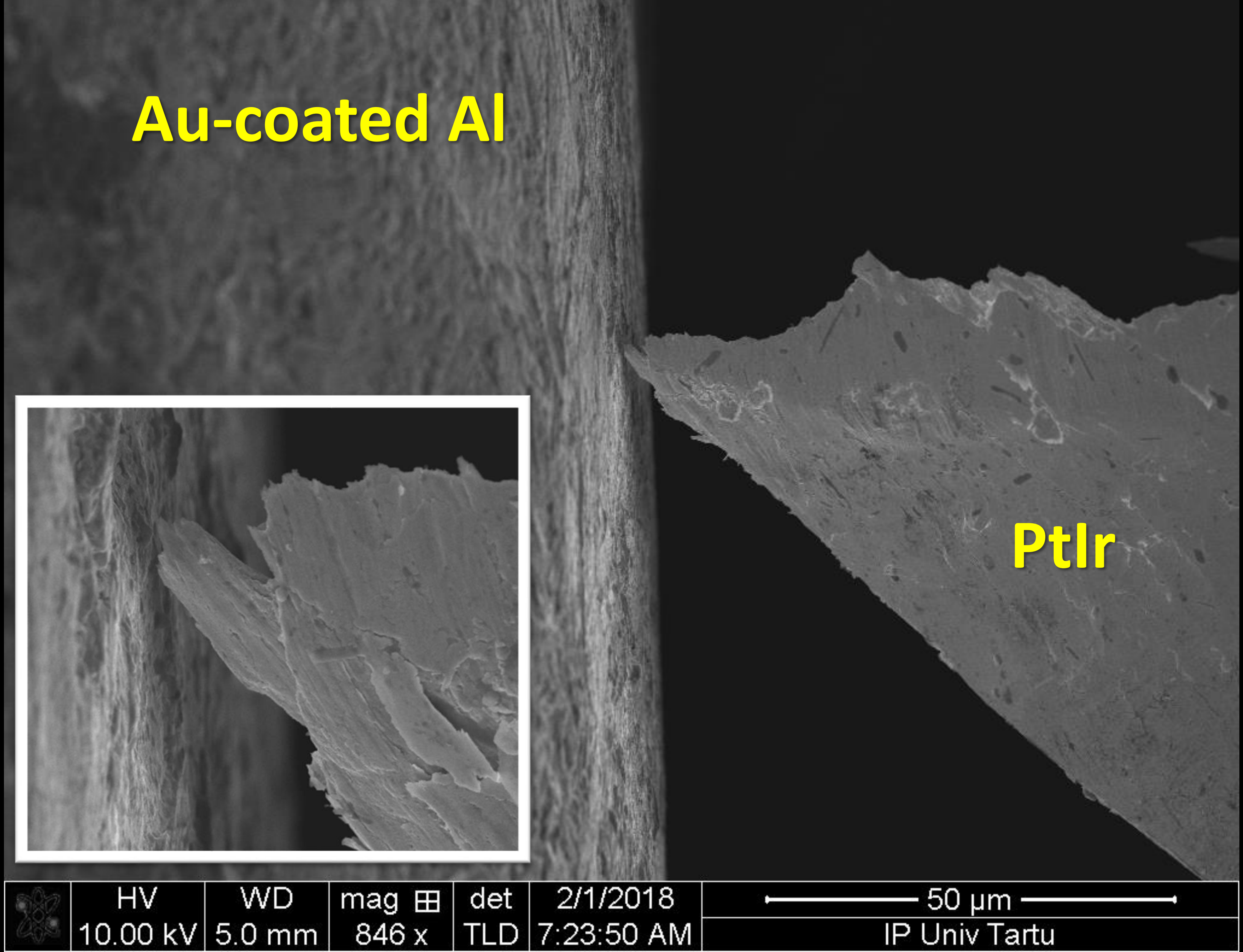
- System: Ag nanowires network on Si substrate and coated with few hundreds of nm of TiO_2

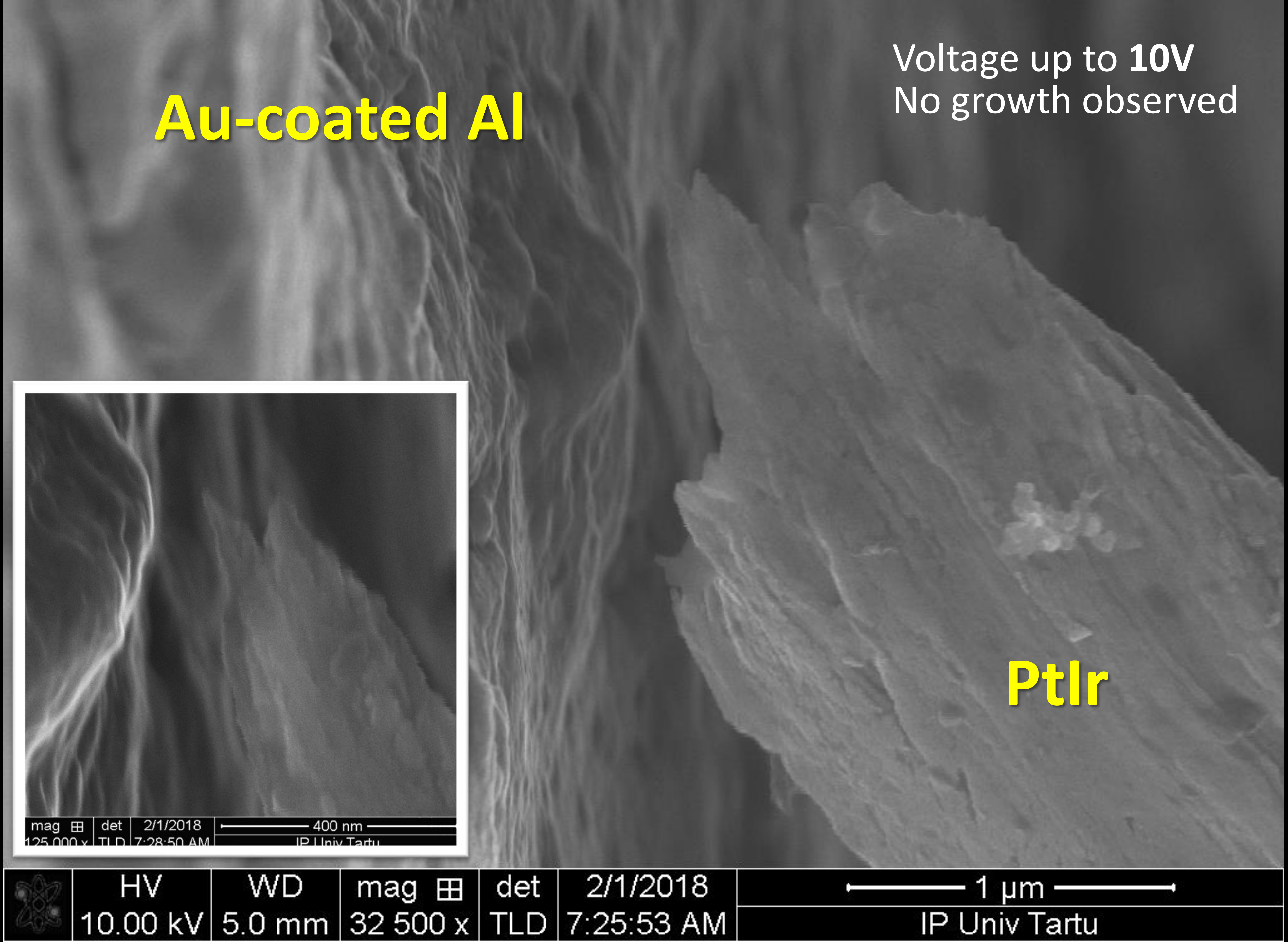
Strong electric fields inside SEM



- Our intent is to create conditions for **controlled emitter formation and growth** under a strong electric field, in predetermined locations and with an immediate visual feedback.

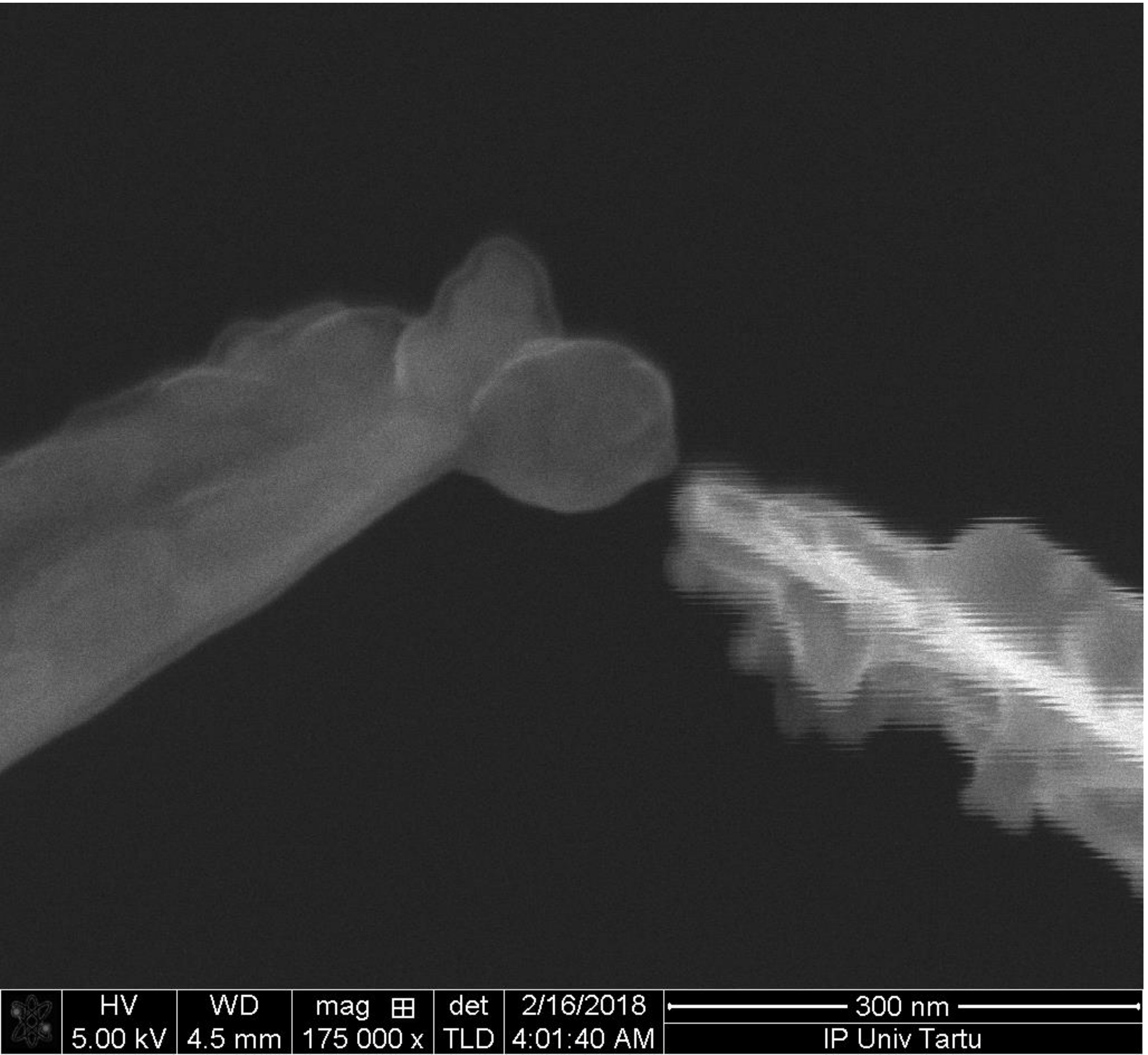
- The strong electric field will be created by applying voltages between the investigated surfaces and a **sharp probe** controlled by piezo-positioner that will be brought to a **close proximity** to the sample.
- The use of **sharp probes** (or even individual nanowires) will allow to reach **extreme local electric field gradients at low voltages** due to the curvature effect.
- Local heating by laser through optical fiber will be utilized for temperature ramping to boost surface diffusion.

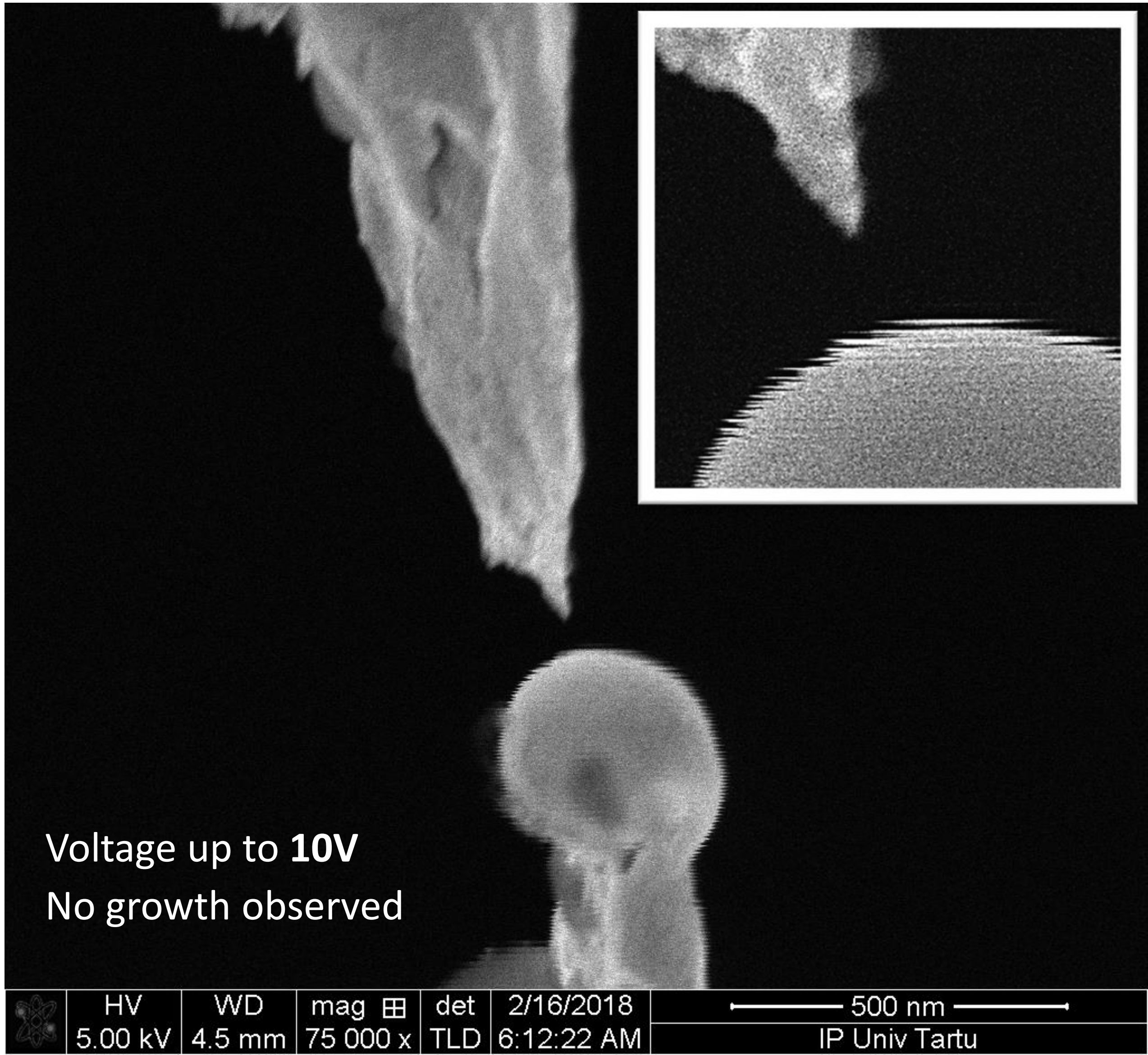

Au-coated Al
PtIr
HV
10.00 kV
WD
5.0 mm
mag ⊞
846 x
det
TLD
2/1/2018
7:23:50 AM
50 µm
IP Univ Tartu



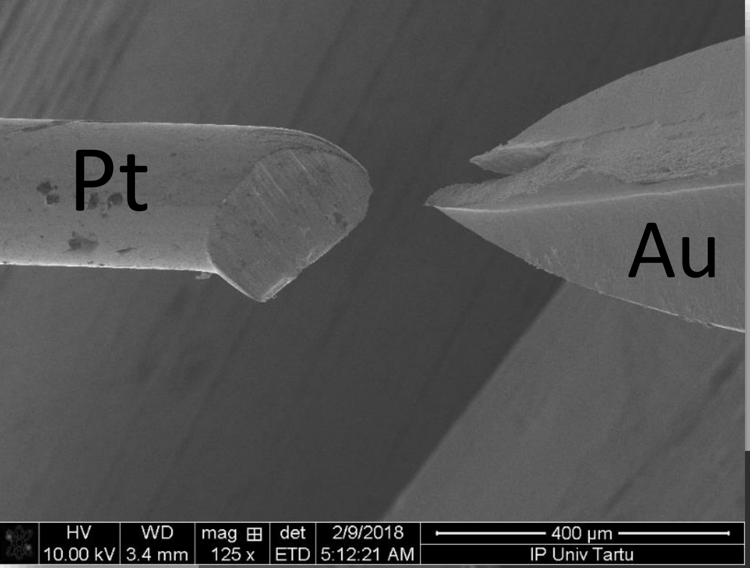
Voltage up to **10V**
No growth observed
Au-coated Al
PtIr
mag
det
2/1/2018
400 nm
HV
10.00 kV
WD
5.0 mm
mag
32 500 x
det
TLD
2/1/2018
7:25:53 AM
1 µm
IP Univ Tartu

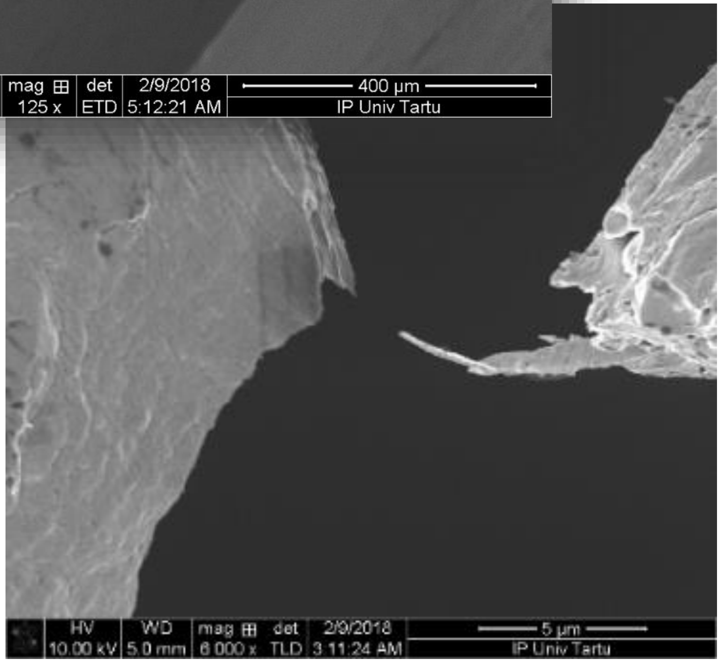
Two **PtIr** wires

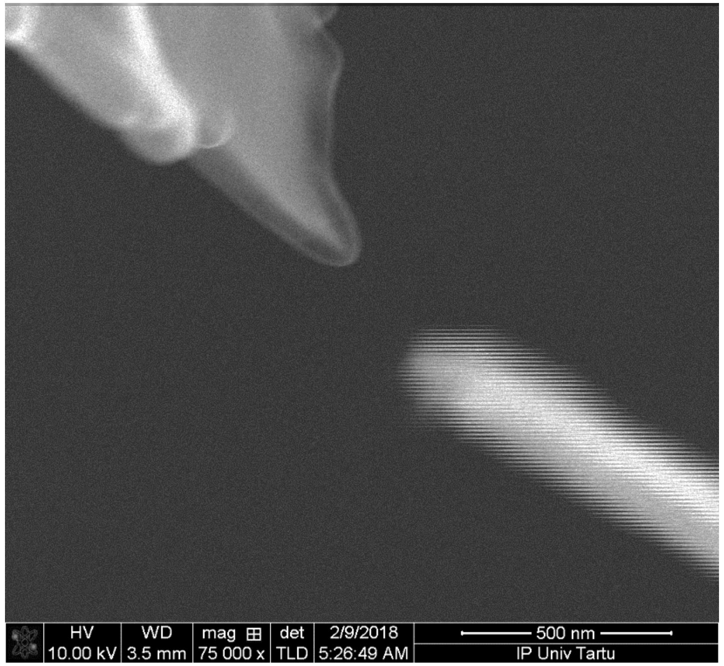


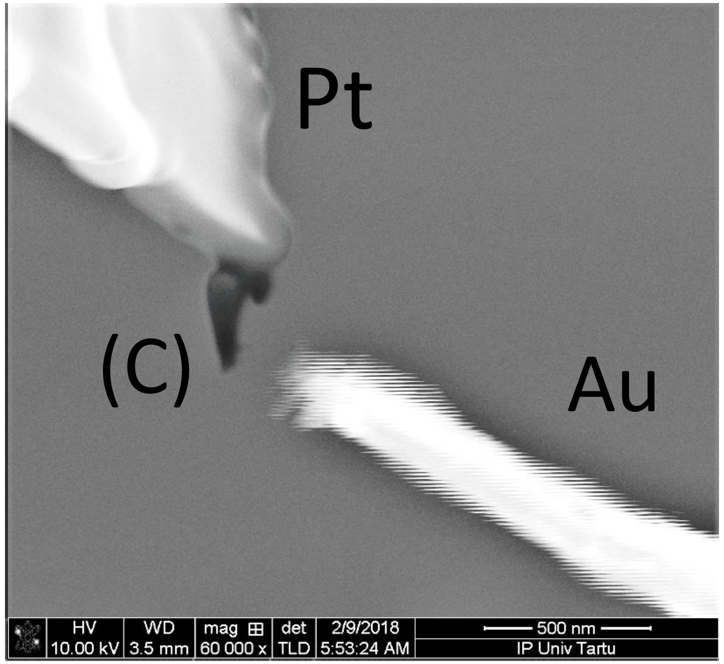


Non-uniform carbon deposition on Pt



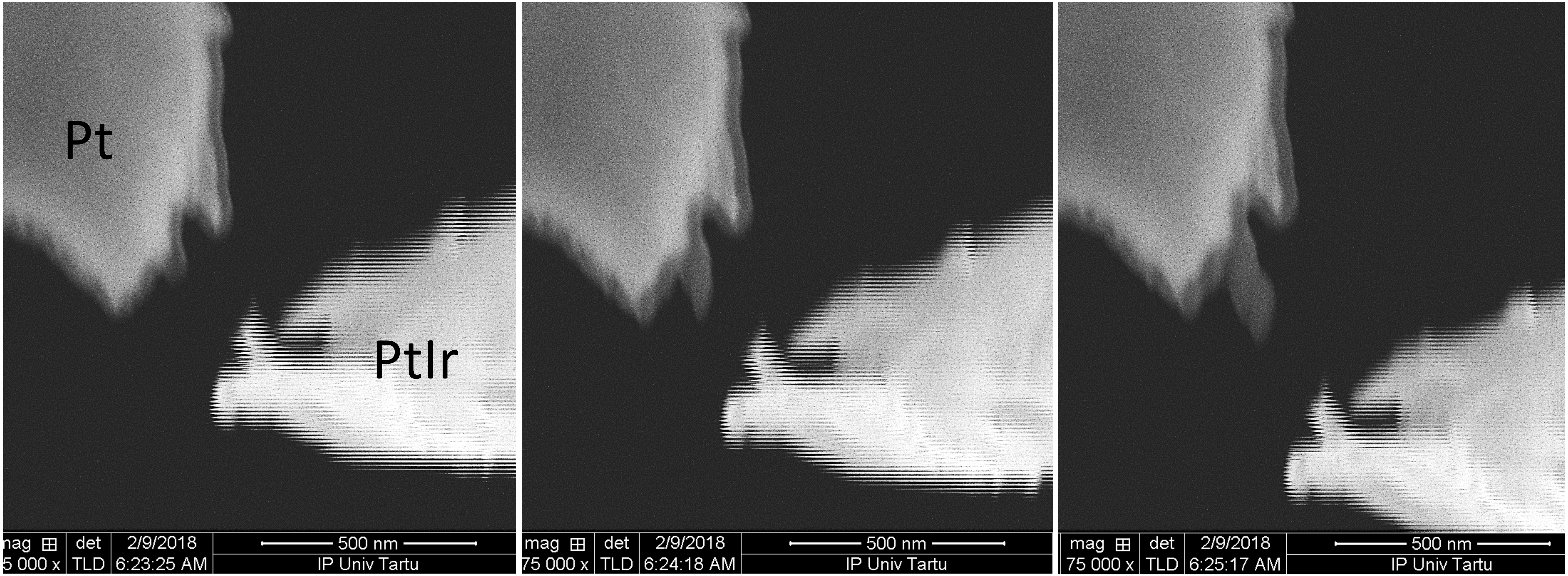






- Chamber pressure is 10^{-6} mbar (in order of **10^{15}** gas molecules in the SEM chamber)

Non-uniform carbon deposition

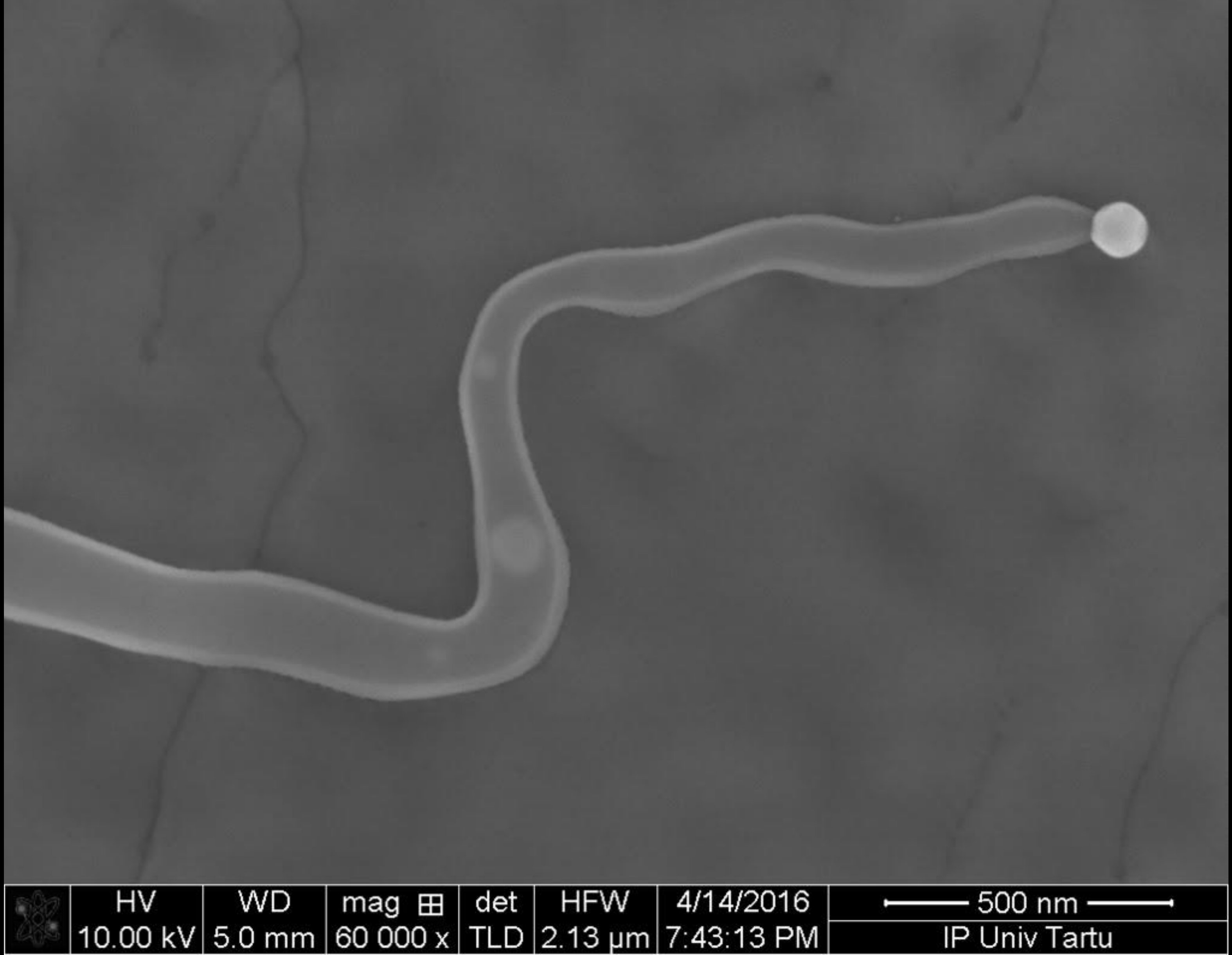


Note! No carbon deposition on PtIr

Problems and future plans

- Electric field disturbs the electron beam
  - Low voltage should be used (below 10 V)
  - E-beam should be switched off when voltage applied
- Carbon deposition on many materials
  - Choice of materials should be corresponding (Au, PtIr, W, …)
  - Blind experiments
- Drifts
  - Prevents “blind” experiments at low separation
  - More rigid system should be built with shorter parts.

Thank you for your attention!



Appendix

Effect of the electron beam

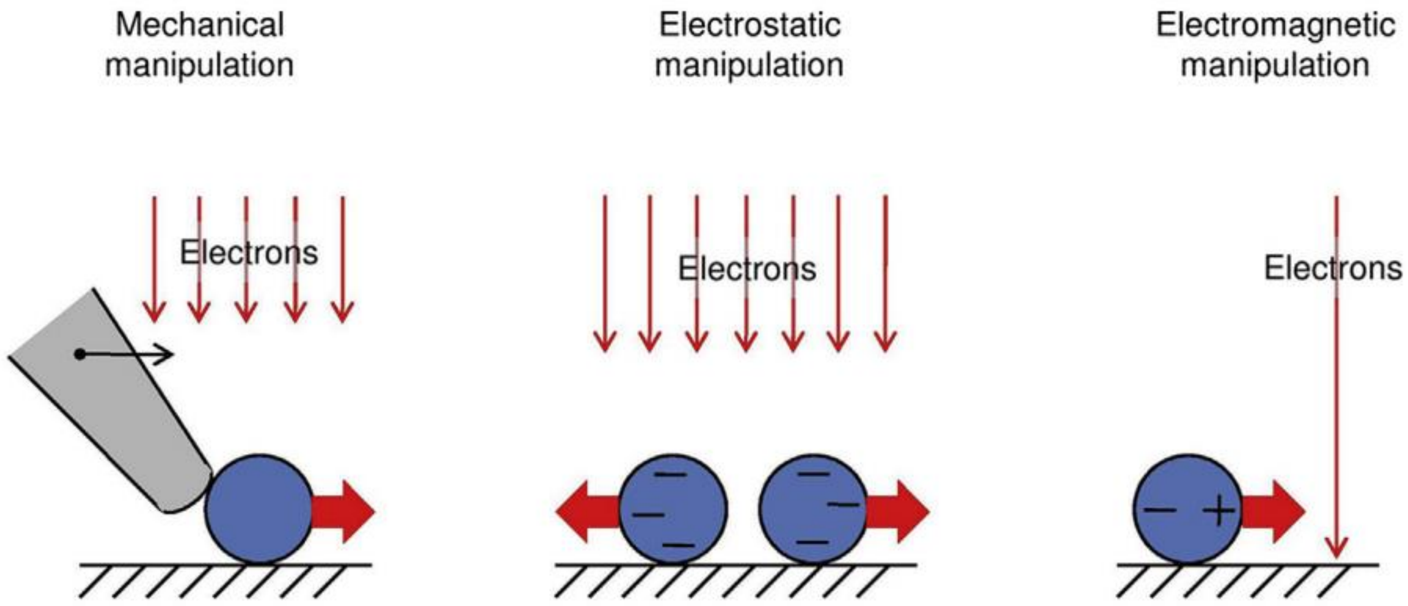


Figure 3.1 Schematic illustration of micromanipulation techniques in electron microscopes: (left) mechanical manipulation (where a micromanipulator is employed, while an electron microscope is used for imaging purposes); (center) electrostatic manipulation (where an electron beam charges particles, which results in attractive or repulsive forces); and (right) electromagnetic manipulation (where an electron beam induces an electromagnetic force exerted on the particle). (See the color plate.)

Effect of the electron beam

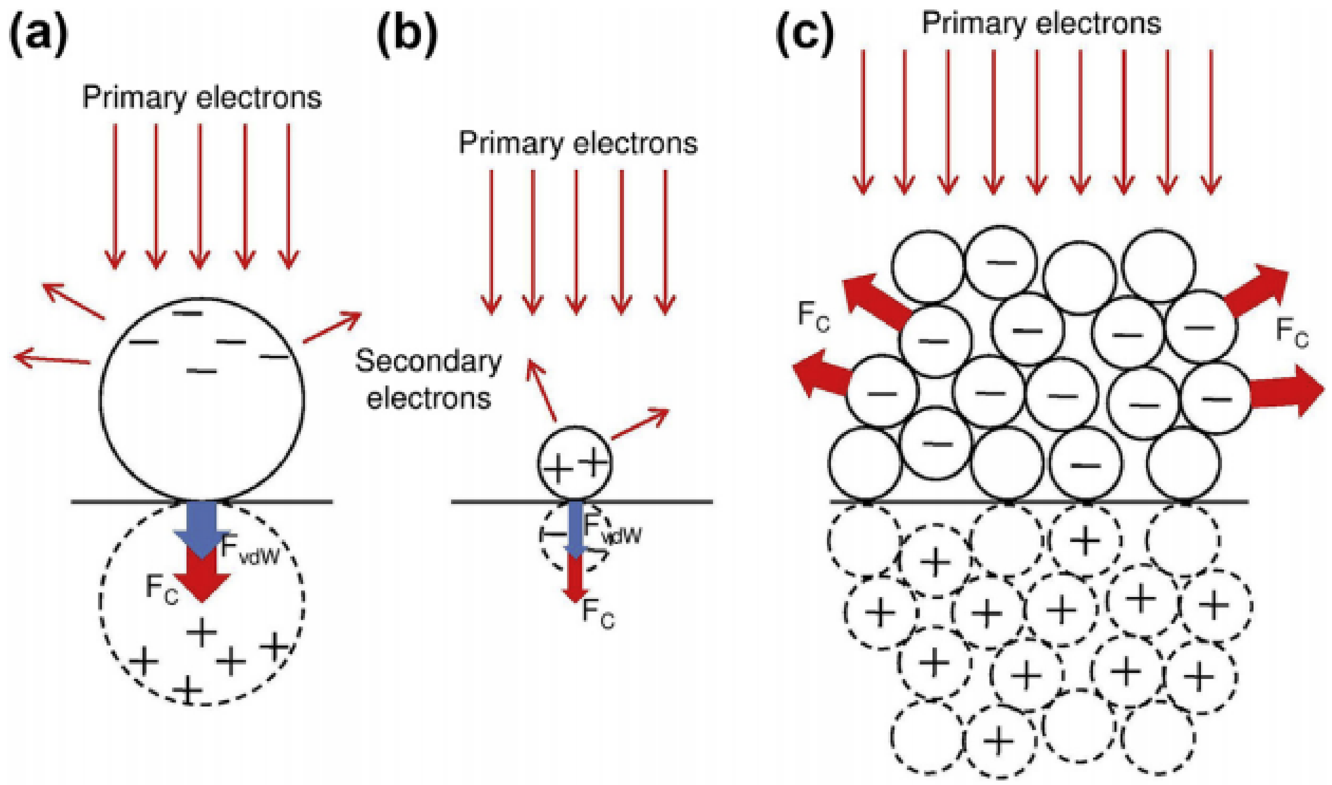


Figure 3.5 Different cases of charging under electron illumination and the resulting adhesion/repulsion for dielectric particles sitting on a conductive substrate (F_{vdW} = van der Waals force; F_C = Coulomb force). (a) A big particle will accumulate a negative charge, which causes increased adhesion to the substrate due to attraction between the charge and its mirror image; (b) a small particle most probably will accumulate a positive charge, and attraction and increased adhesion will take place; (c) small particles piled on top of each other will be charged negatively, which will cause repulsion between them. (See the color plate.)

Effect of the electron beam

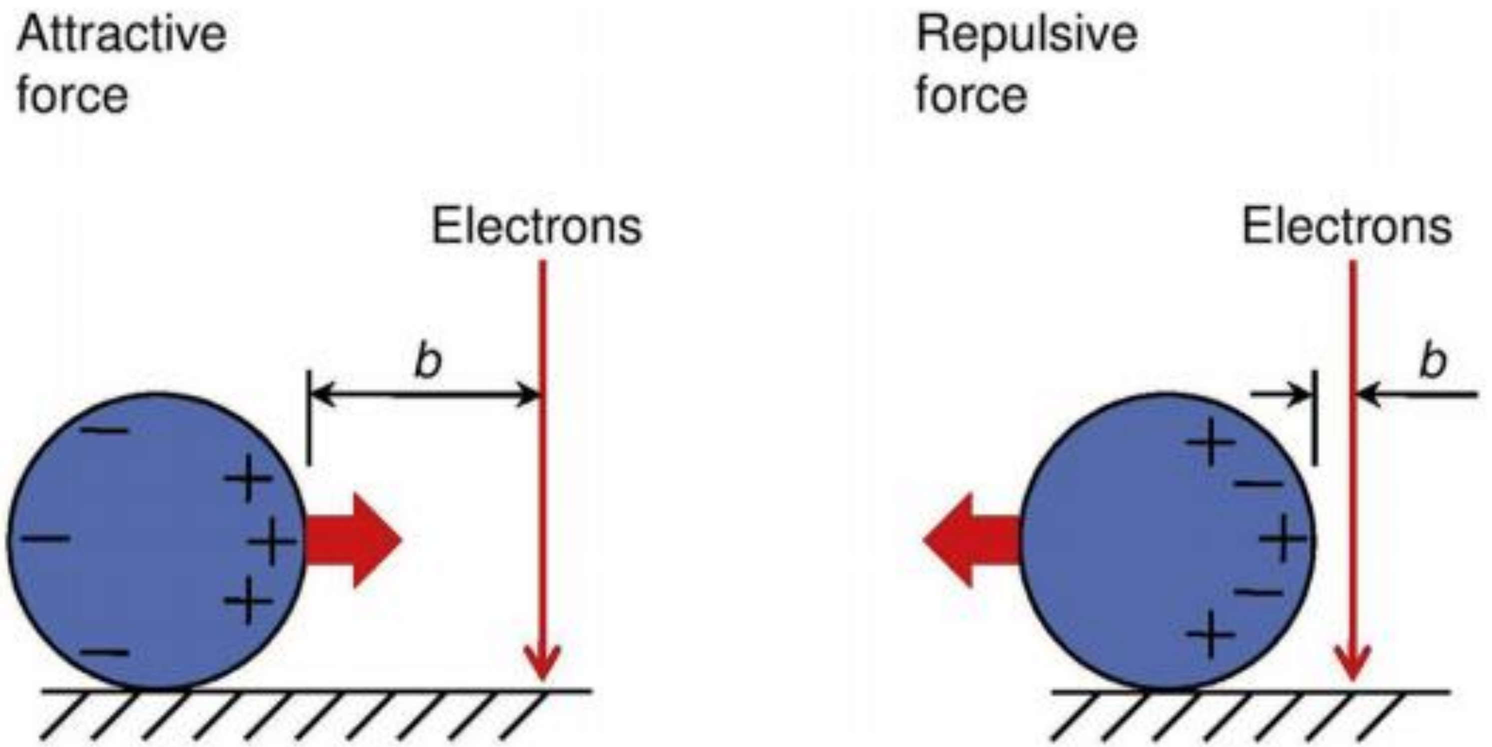


Figure 3.6 Schematic illustration of an electromagnetic force exerted on a particle by a fast-passing electron. The force can be attractive or repulsive, depending on the impact parameter *b* (following Batson *et al.*, 2011). (See the color plate.)