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Brief Intro to Vamsi & Daniel's presentation: Motivation, hypothesis, and appreciation

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Motivation

- **What determines MW diameters remaining almost the same in the process of longitudinal growth? *(Note: MW thickness in micron range, much greater than the nano-sizes of the typical nuclei in solids)**
- **What determines their statistical distributions? ****
- * Thank you Steve Smith pointing to me at the importance of that questions.
- **Thank you Lyudmyla Panashchenko & Mike Osterman and Steph Meschter & Polina Snugovsky for making their data available.

Motivation (continued)

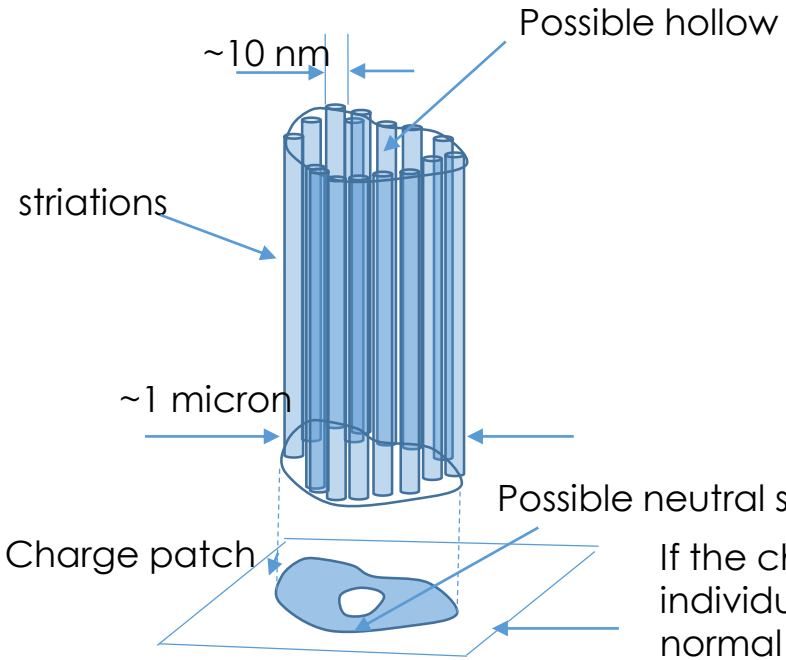
- **Can MW have some internal structure at nano-scale?**

  Motivated by

  - **The well known longitudinal striations on MW surfaces**
  - **Asymmetric random cross-sections strongly deviating from the circular**
  - **Voids inside MW (hollow whiskers) reported**
  - **Thermal stability of Sn MW depends on the film deposition, evaporated vs. electro-deposited:**
    - **Under a focused ion beam, local melting of comparable MW is easier on the evaporated than on electroplated structure pointing at some internal structure inherited from the film ***
  - **The electrostatic theory does not describe the facts as is.**

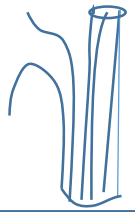
*Thank you Vamsi and Daniel for sharing the observation

Hypothesis: massive nucleation and co-growth of multiple thin filaments = nanostructure of whiskers



If the charge patch is related to an individual grain, then the well-known log-normal distribution of grain sizes translates into that of MW diameters

One anticipated consequence: the multi-filament structures can occasionally split: Observed and related by Steve Smith and Bill Rollins



Thank you Vamsi and Daniel for your interesting experiments and effort!
Thank you Bill for inviting this talk.

Part – 1: Whisker characterization

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Outline

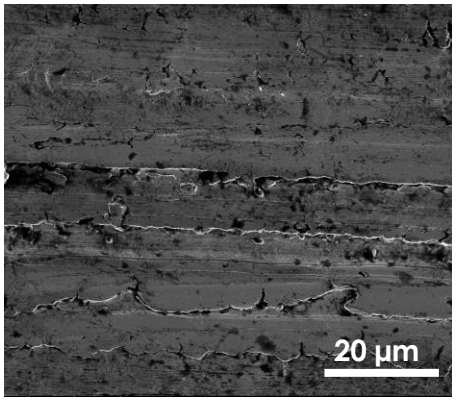
- Samples
- Characterization
- Tools
- Whisker FIB and TEM analysis (Part 1)
  - Whisker cross-section study
  - TEM sample preparation

Characterization

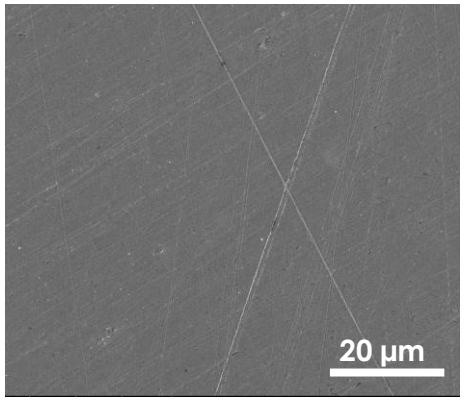
Samples preparation

Electroplating:

- Cu coupons were mechanically polished with silicon carbide sand paper and machine polished using alumina slurry (50nm).
- Cleaned and then electroplated from sodium stannate bath.



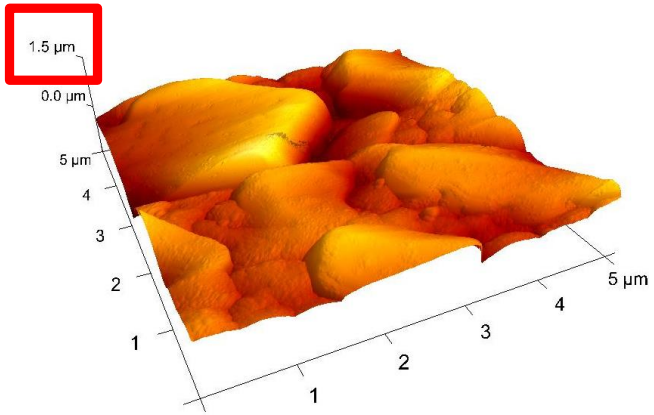
Before polishing



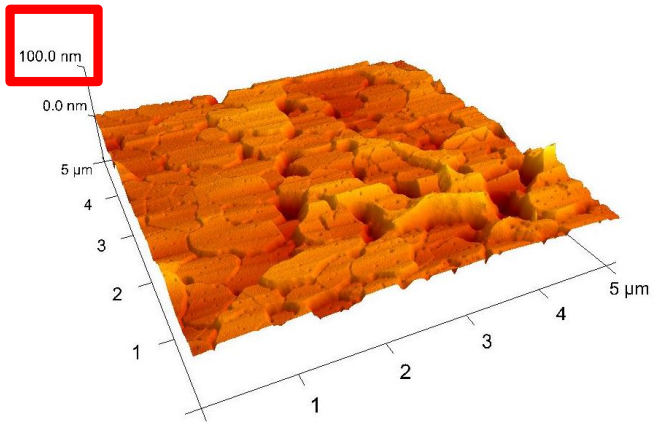
After polishing

Evaporation:

- Sn samples were prepared by evaporating Sn pellets from tungsten boats onto the Cu coated glass substrate.



Electroplated sample

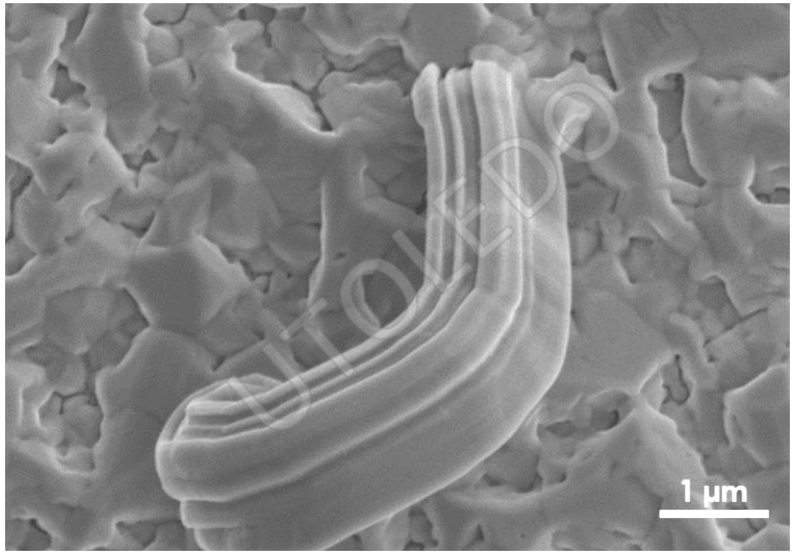


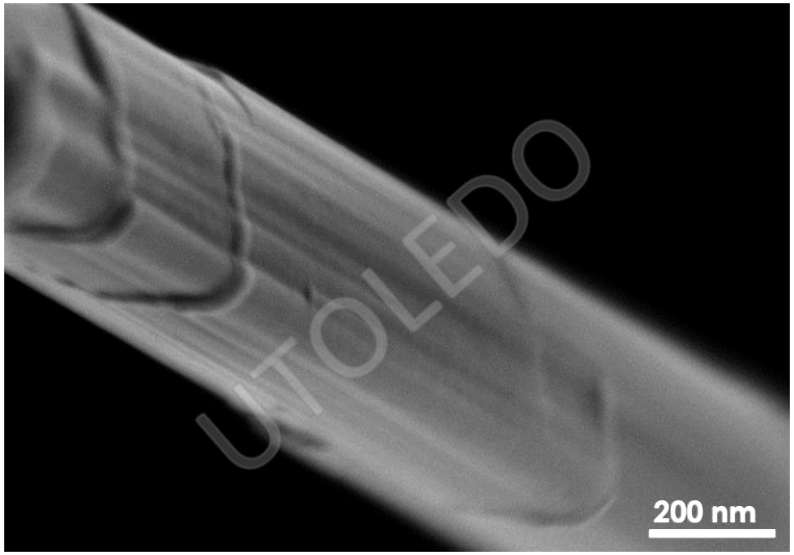
Evaporated sample

Characterization

Techniques employed:

- **Scanning electron microscopy (SEM)**
- **Energy-dispersive x-ray spectroscopy (EDS)**
- **Focused ion beam (FIB)**
- **Transmission electron microscopy (TEM)**





Characterization

Tools:

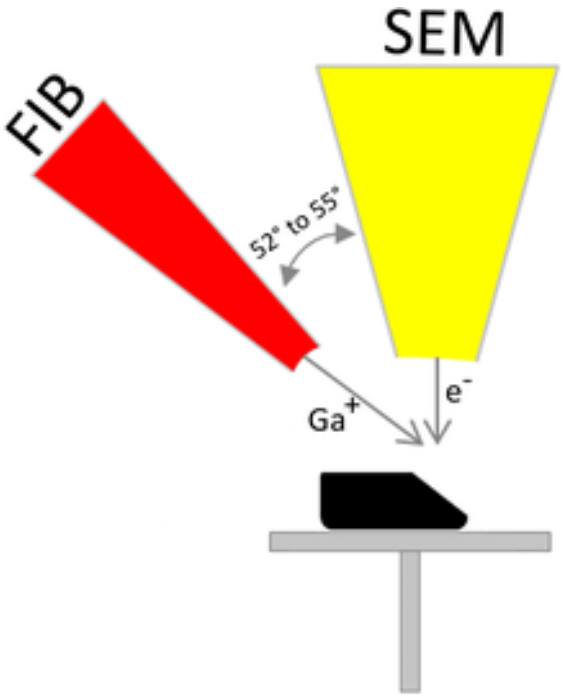
- **SEM – 2nm @ 1kV**

- **FIB (Dual beam)**
  - **Ion beam:10nm @ 30 kV (1pA), Ga source**
  - **Electron beam : 1.2 nm @ 30kV (HV)**

- **TEM – 200 kV – 0.203 nm – TE, ZC, SE**
  - *Each instrument is equipped with its own EDS detectors.*

Whisker FIB-TEM analysis

FIB-milling:



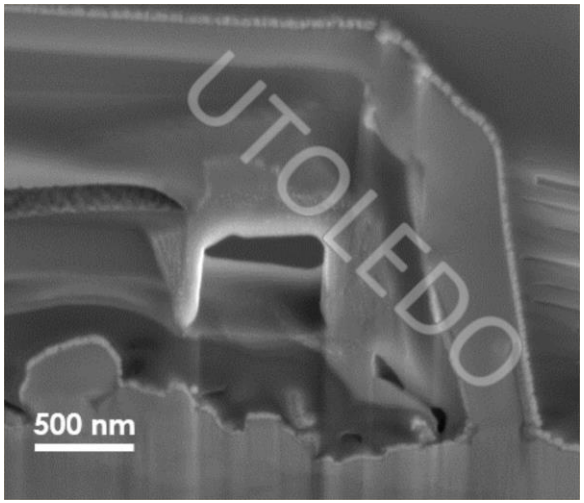
Milling (ion-beam) and imaging (electron beam) done simultaneously

Ion-beam can be used to take images (gives more details)

Two goals:

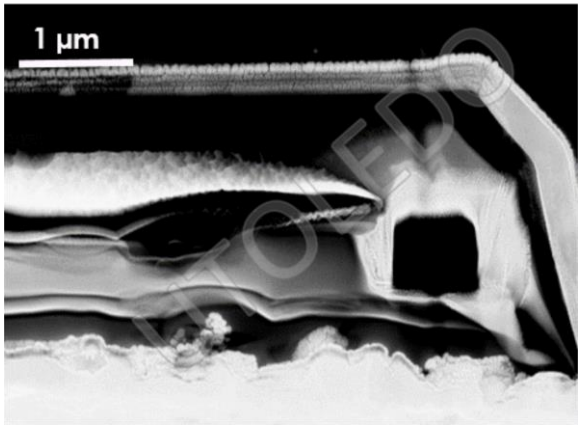
1. **Whisker cross-section analysis.**

2. **TEM sample preparation.**



Whisker cross-section

Unpublished



Whisker TEM image

Unpublished

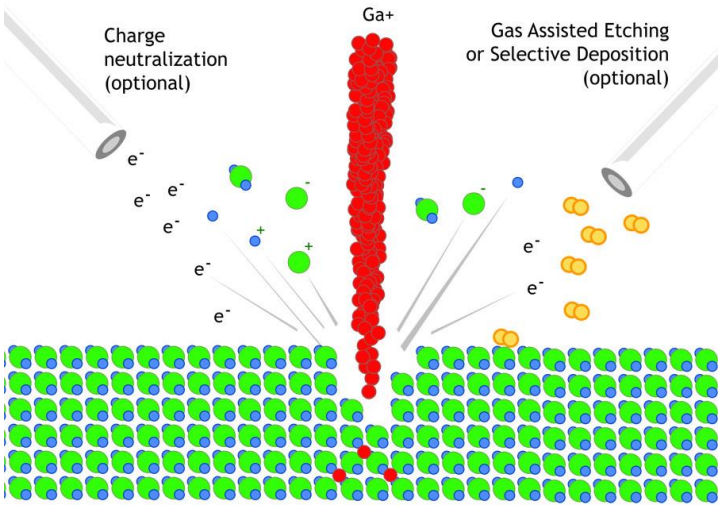
FIB illustration: C. Kizilyaprak et al "Focused ion beam scanning electron microscopy in biology," J. Microsc., vol. 254, no. 3, pp. 109–114, Jun. 2014.

Whisker FIB-TEM analysis

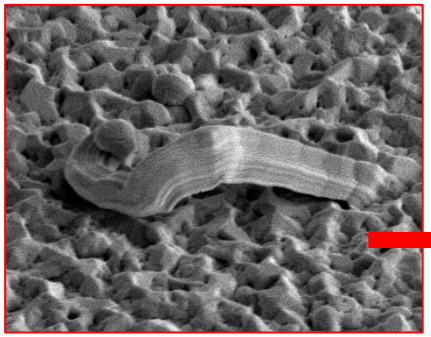
FIB-milling: cross-section

Imp. notes:

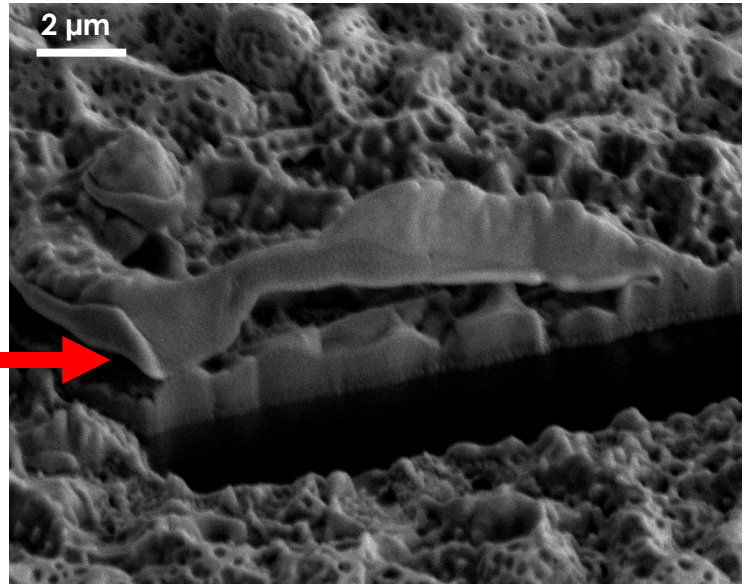
1. **Au-Pd coating to guard sample and whisker from *Platinum*.**
2. ***Platinum* deposition to protect whisker from ion source.**

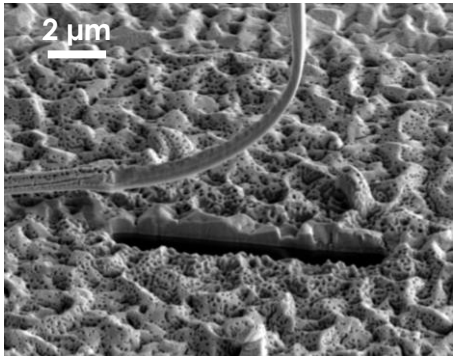


Ga+ sputtering out sample



Whisker without Pt protection



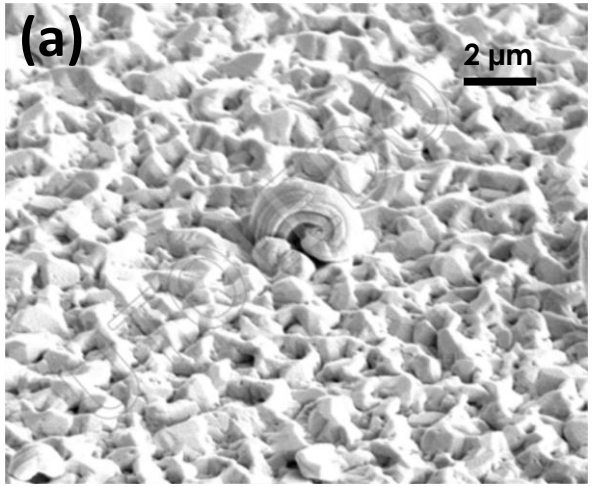


Whisker starting to bend

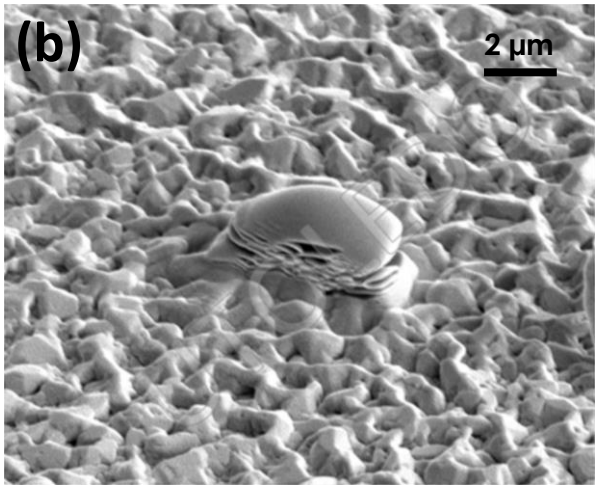
FIB illustration: http://www.fibics.com/

Whisker FIB-TEM analysis

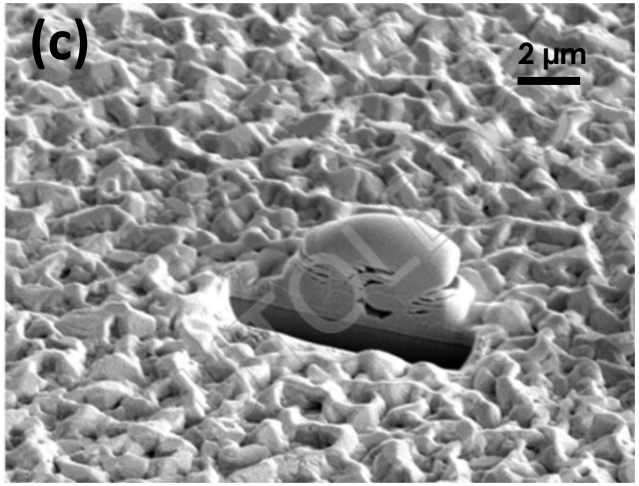
FIB-milling: cross-section (Evaporated sample)



Select a whisker



Dep Pt (low current)

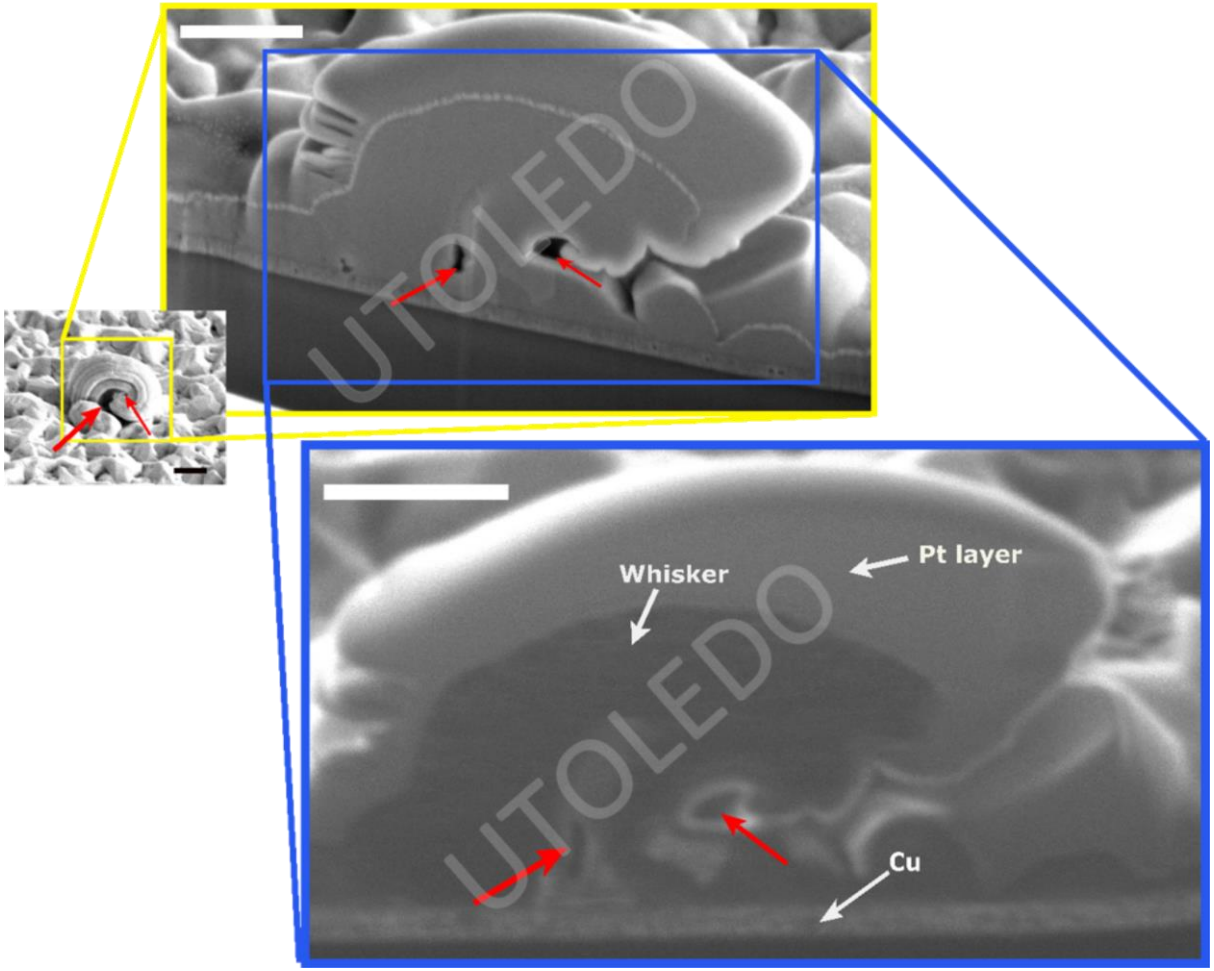


Start milling (again low current)

- Small whiskers are better to work with.
- Optimized beam current is used during Pt deposition.
- Milling process is done with low beam current as well.
- Above SE images are taken at an angle (52 deg).

Whisker FIB-TEM analysis

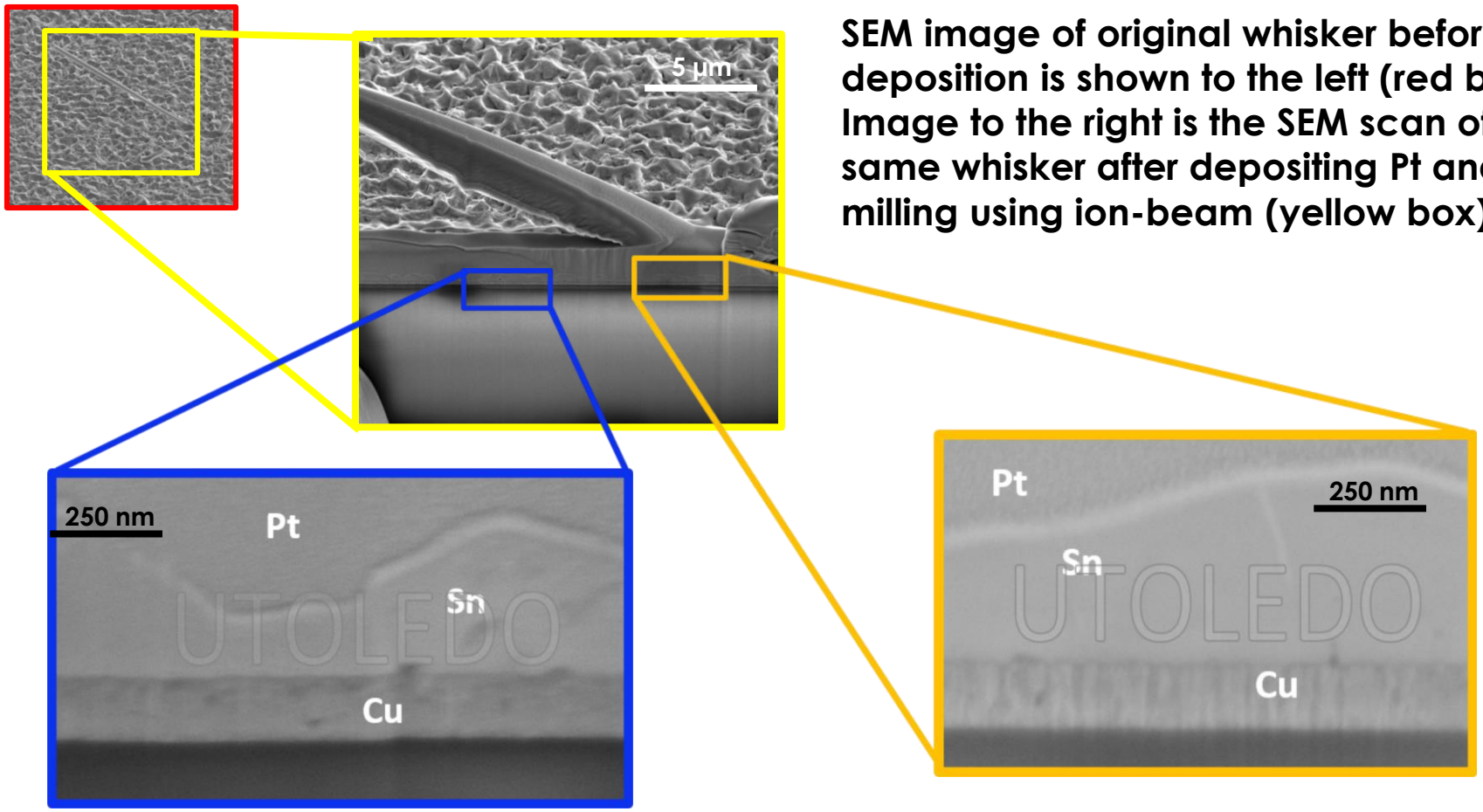
Cross-section analysis (Evaporated sample)



The smaller SEM image to the left is the original whisker before the FIB work. Red arrows in all the 3 figures point to voids, which should not be confused with IMCs. The bottom one is an ion beam picture (blue box) of the same whisker and it clearly shows the contrast difference between different materials, uniform Cu layer, inherent voids and the layers with no signs of IMCs. The scale bar in all the three images is 1μm.

Whisker FIB-TEM analysis

Cross-section analysis (Evaporated sample)

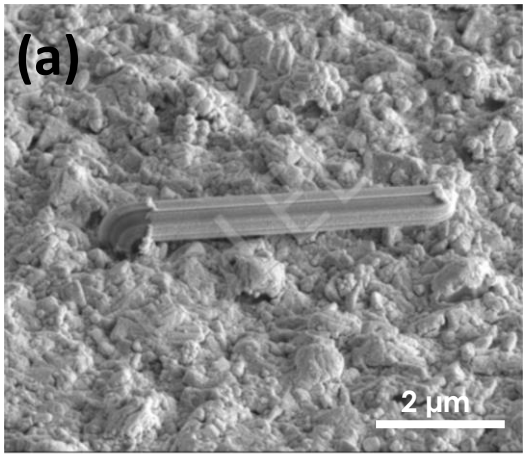


SEM image of original whisker before Pt deposition is shown to the left (red box). Image to the right is the SEM scan of the same whisker after depositing Pt and milling using ion-beam (yellow box).

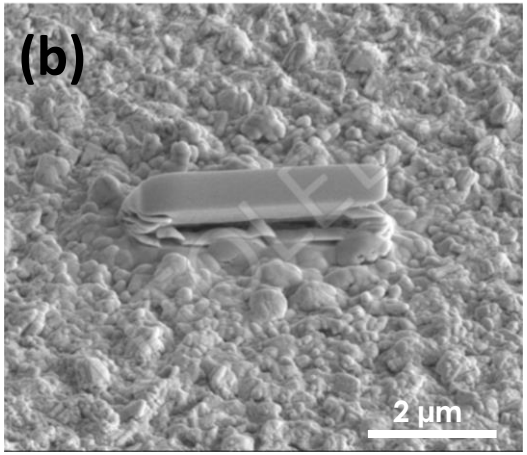
Above are the close-up scans of the cross-section clearly showing the layer separation, no signs of IMCs.

Whisker FIB-TEM analysis

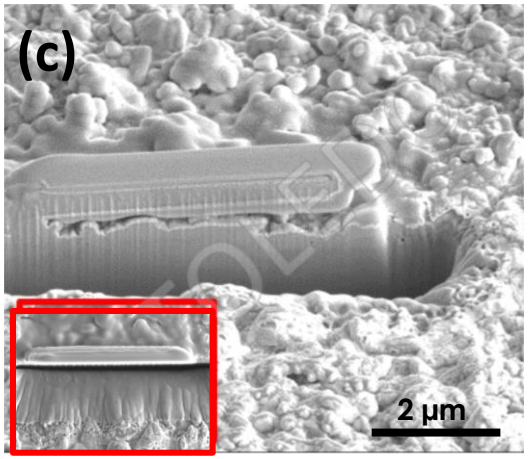
FIB-milling: cross-section (Electroplated sample)



Whisker before milling



Pt deposition

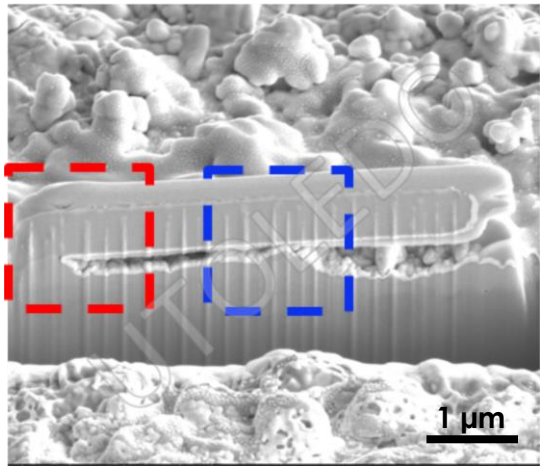


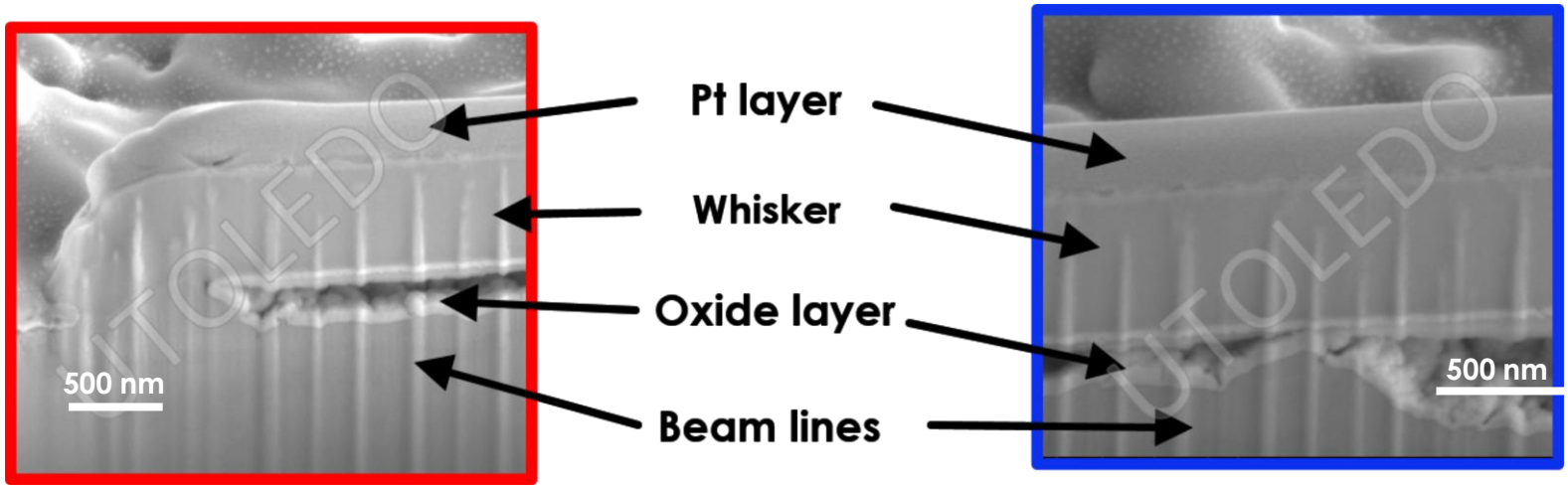
Milled whisker

- **Whiskers in this case sustained a little higher beam currents than whiskers that grew on evaporated sample . This may be due to the oxide layer on the whisker's surface or any other reasons.**

Whisker FIB-TEM analysis

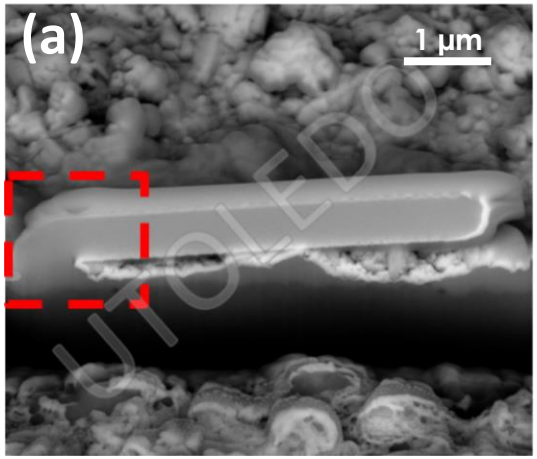
Cross-section analysis (Electroplated sample)

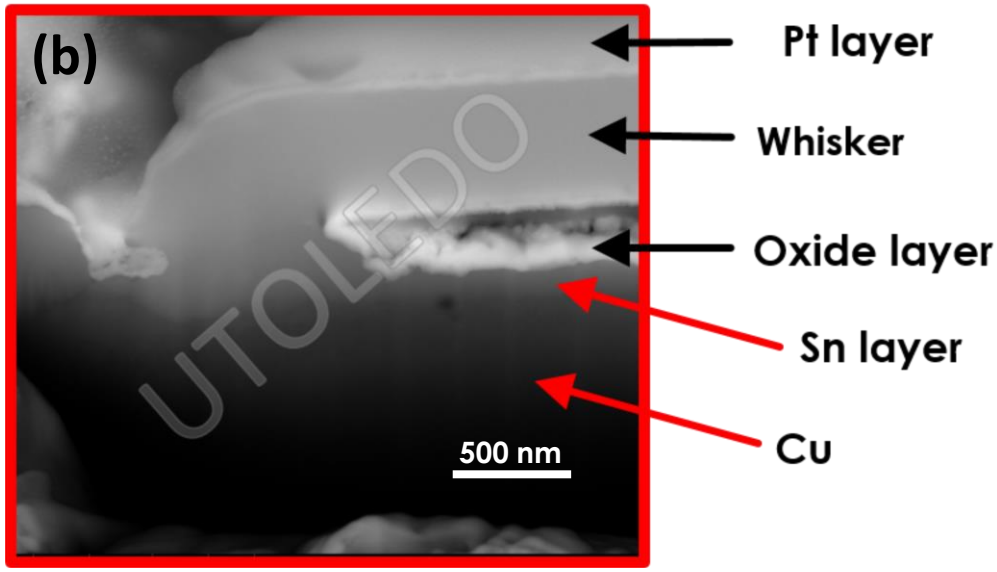




Whisker FIB-TEM analysis

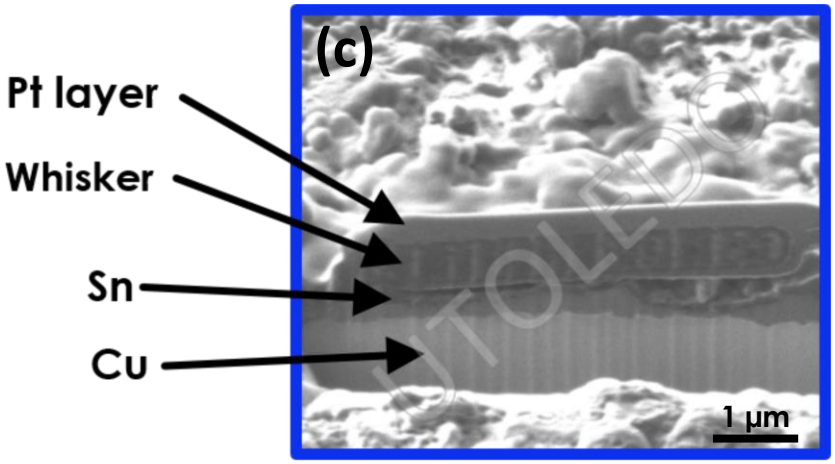
Cross-section analysis (Electroplated sample)





Z-contrast images (BSED)

- Z-contrast and ion images showing more details of the layers.
- Cross-section reveals no signs of IMCs.

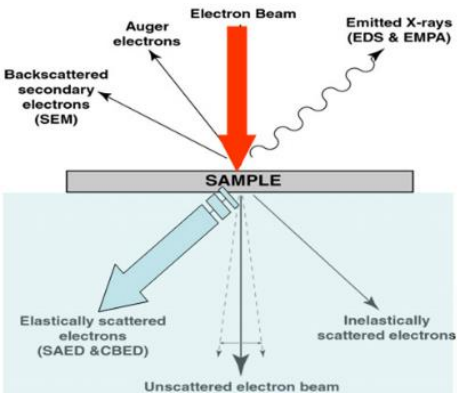


Ion-beam image

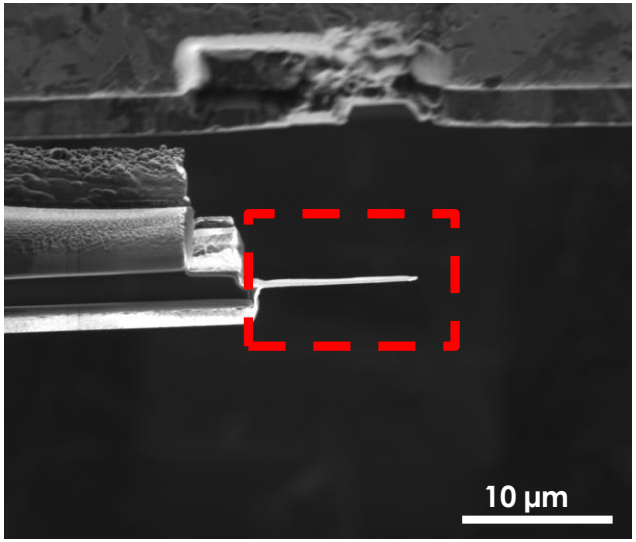
Whisker FIB-TEM analysis

FIB-milling: TEM sample (whisker)

- **Transmitted Electrons (TE) are required for our micro-structural examination.**
- **TEMs need <u>electron transparent</u> samples.**
- **Typical thickness values ~50nm to ~200nm.**
- **So, we have to mill our whisker to electron transparent thickness.**



TEM principle

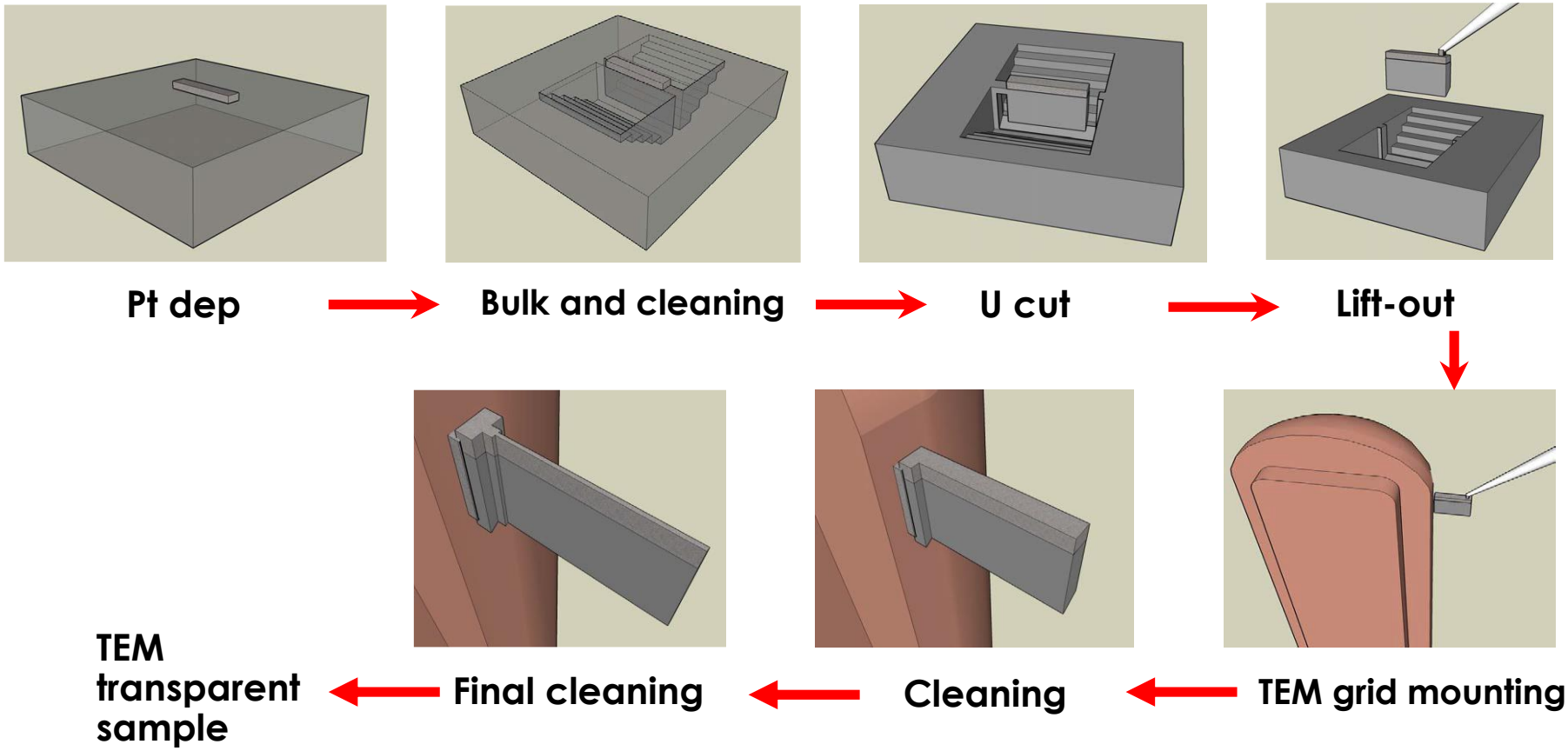


TEM transparent sample in red box

TEM schematic: www.ammrf.org

Whisker FIB-TEM analysis

FIB-milling: general TEM sample preparation steps

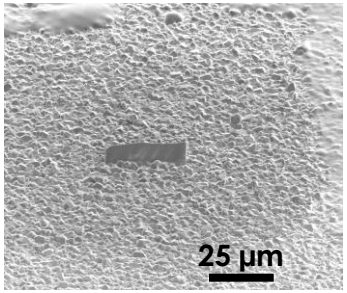


- **Sample lift-out was carried using a in-situ nano-manipulator.**
- **After final cleaning, sample is transferred to TEM.**

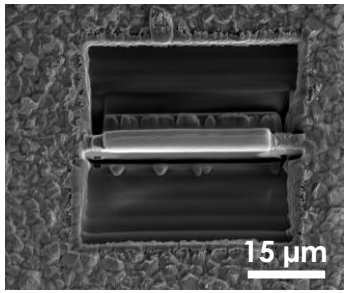
FIB steps: TEM Sample Preparation with FIB – Ben Myers – (NUANCE center)

Whisker FIB-TEM analysis

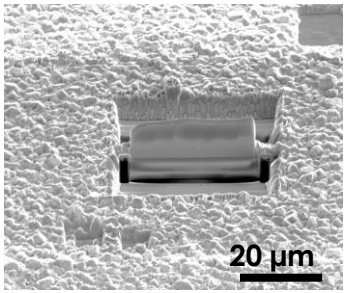
FIB-milling: Whisker TEM sample



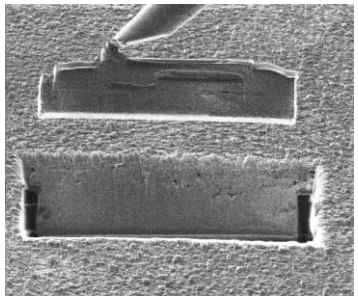
Pt dep



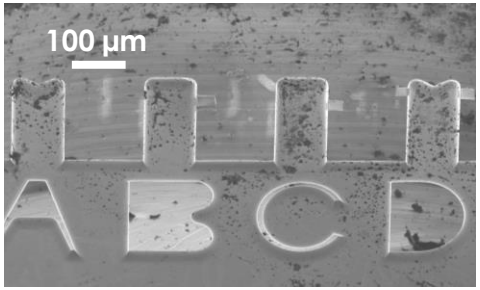
Bulk and cleaning



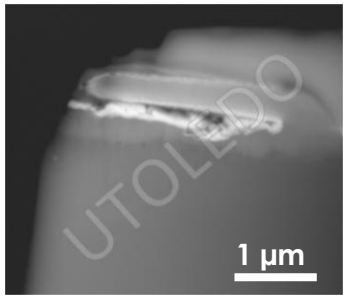
U cut



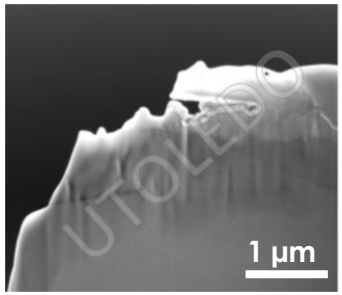
Lift-out



TEM grid mounting



Cleaning



Final cleaning

TEM transparent sample

- Success and failures

Whisker FIB-TEM analysis

Whisker TEM analysis:

Part -2: To be presented next week

- **TEM analysis**
  - **TE imaging**
  - **BS imaging**
- **EDS analysis**
- **SAPD**
- **Discussion**
- **Conclusions**