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Part – 2: Whisker characterization

Vamsi Borra
Daniel G. Georgiev

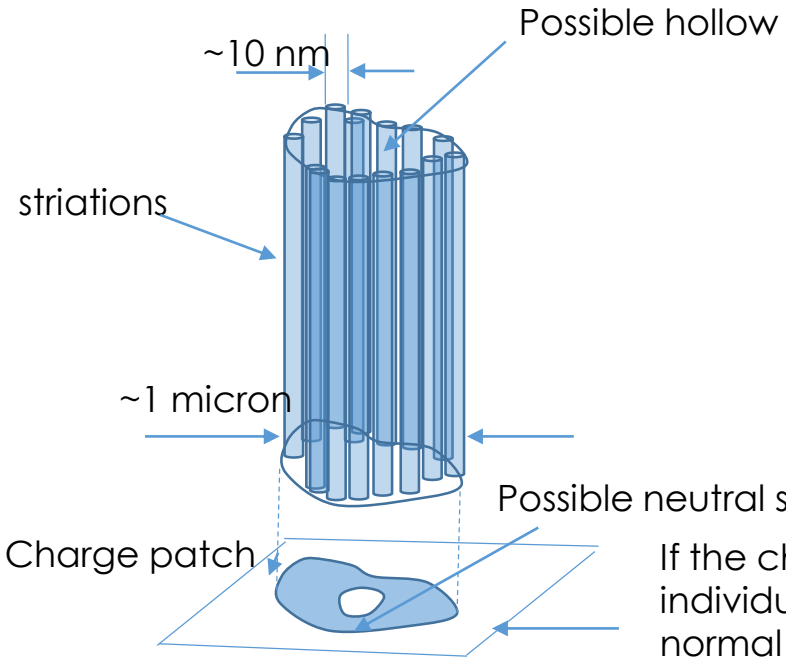
Dept. of Electrical Engineering and Computer Science,

The University of Toledo, Toledo, OH, USA.

Outline

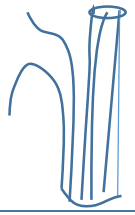
- Review part 1
- TEM characterization
  - EDS
  - SADP
- Conclusions

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



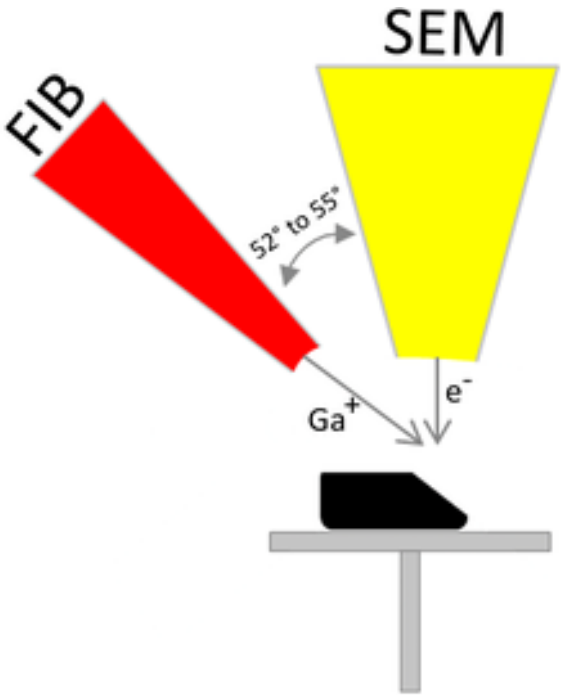
If the charge patch is related to an individual grain, then the well-known log-normal distribution of grain sizes translates into that of MW diameters

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



Whisker FIB-TEM analysis

FIB-milling:



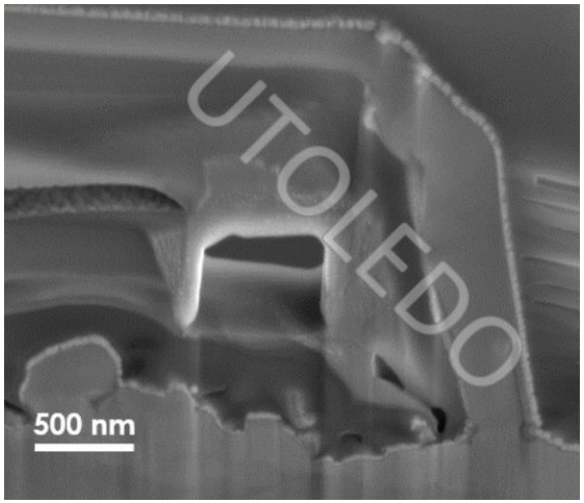
Milling (ion-beam) and imaging (electron beam) done simultaneously

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

Two goals:

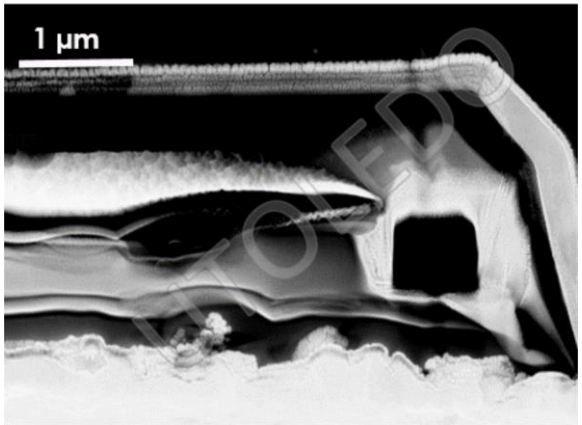
1. **Whisker cross-section analysis.**

2. **TEM sample preparation.**



Whisker cross-section

Unpublished



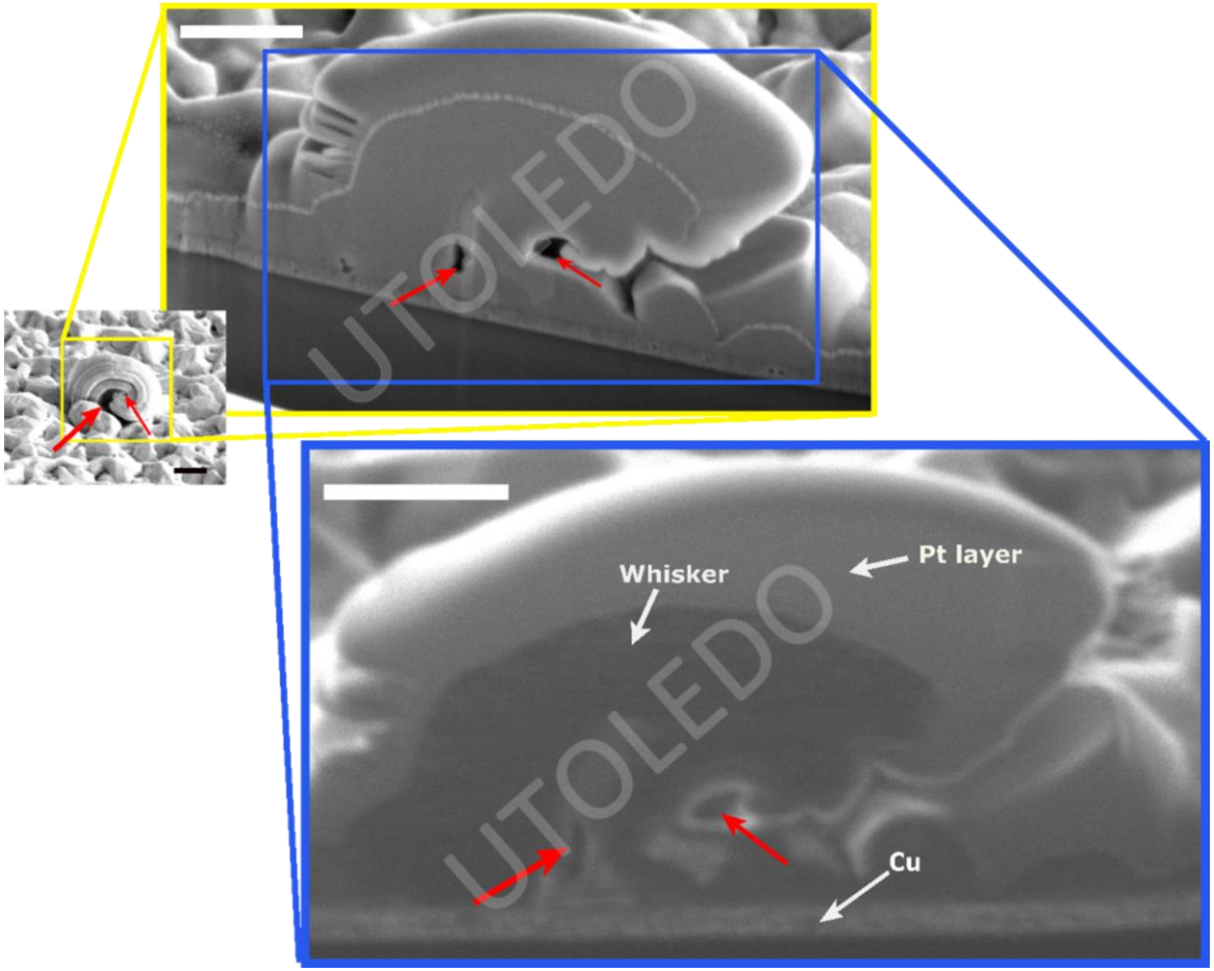
Whisker TEM image

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

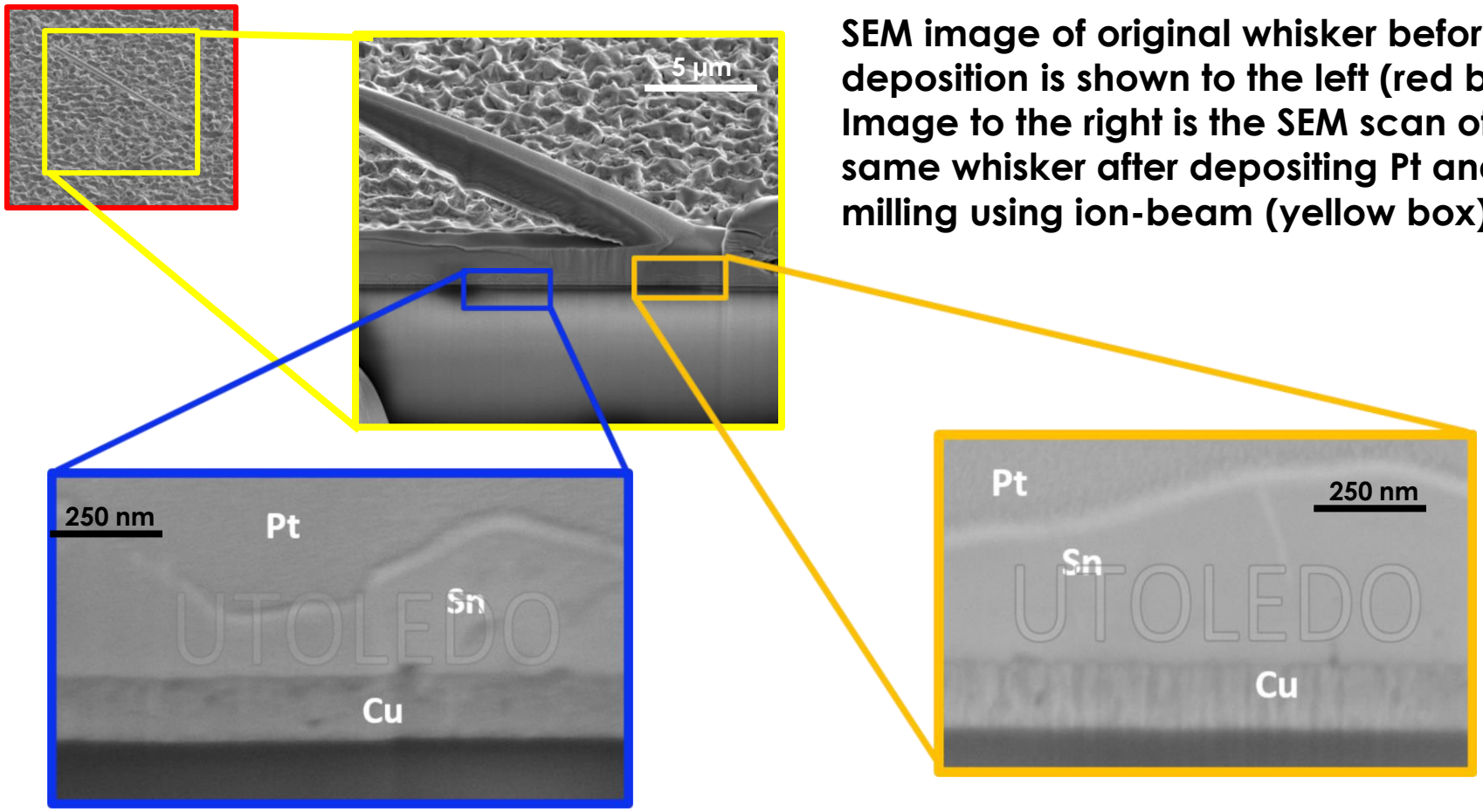
Cross-section analysis (Evaporated sample)



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)

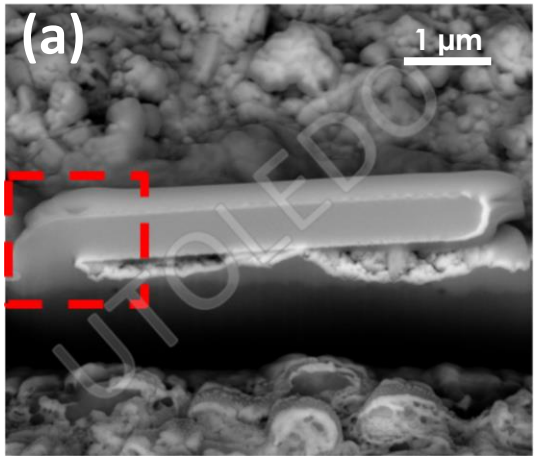


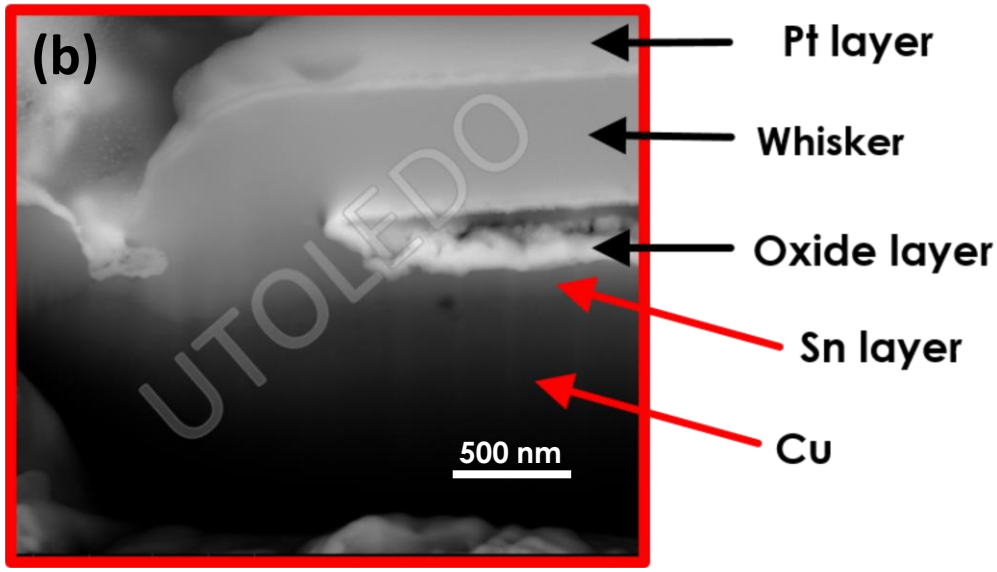
SEM image of original whisker before Pt deposition is shown to the left (red box). Image to the right is the SEM scan of the same whisker after depositing Pt and milling using ion-beam (yellow box).

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

Whisker FIB-TEM analysis

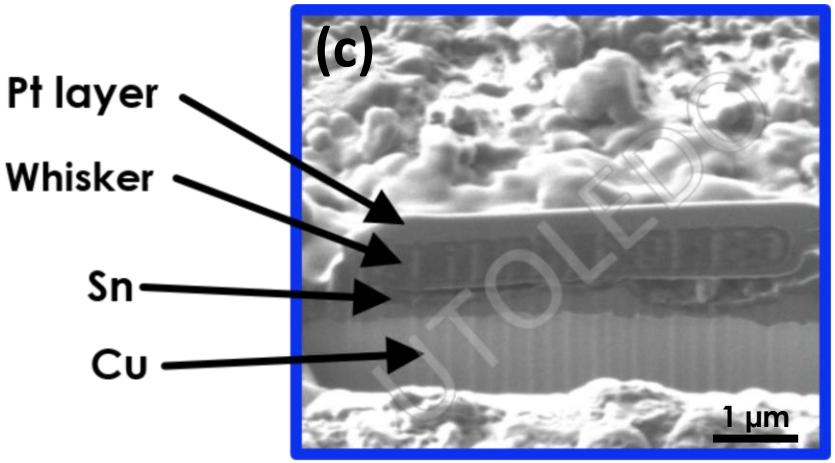
Cross-section analysis (Electroplated sample)





Z-contrast images (BSED)

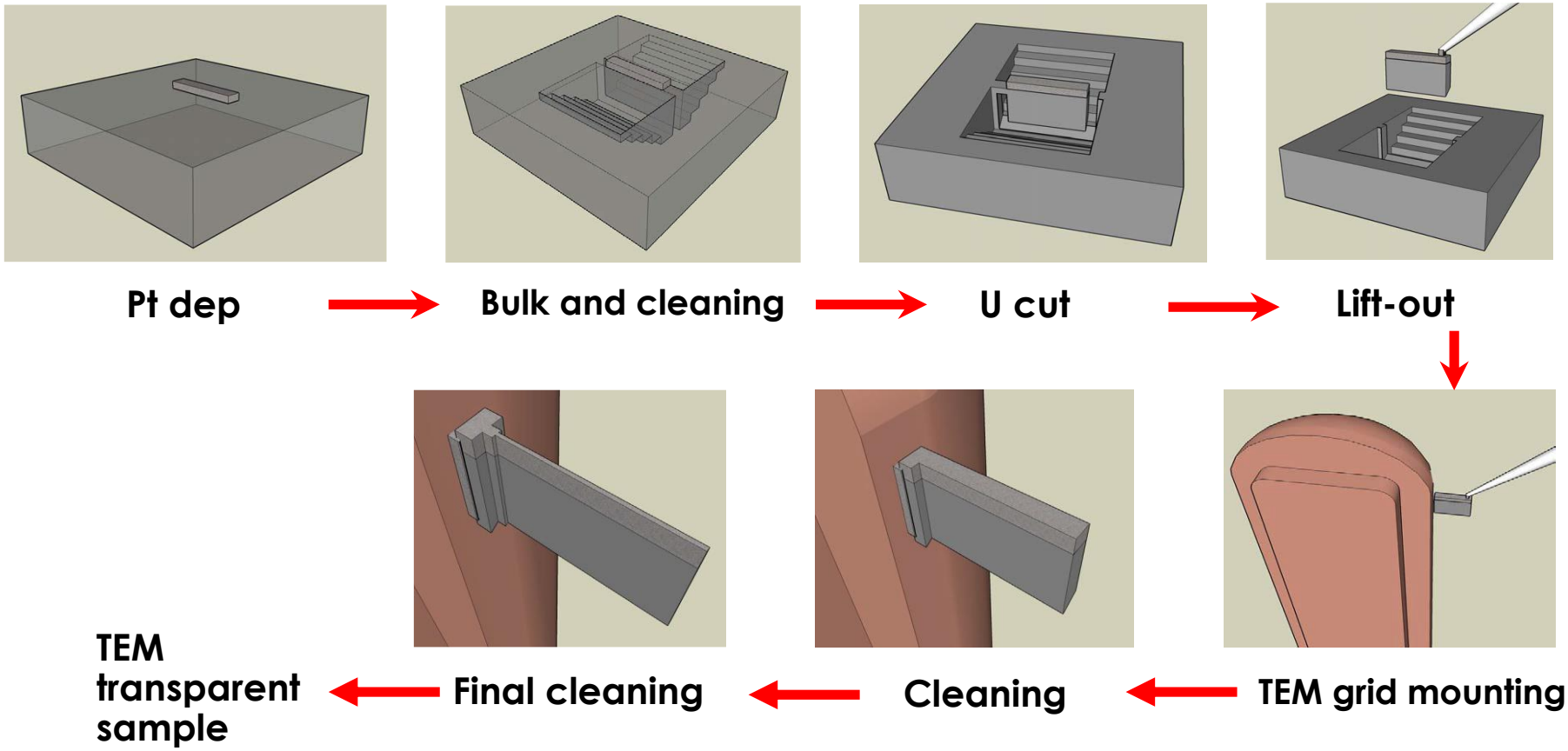
- Z-contrast and ion images showing more details of the layers.
- Cross-section reveals no signs of IMCs.



Ion-beam image

Whisker FIB-TEM analysis

FIB-milling: general TEM sample preparation steps

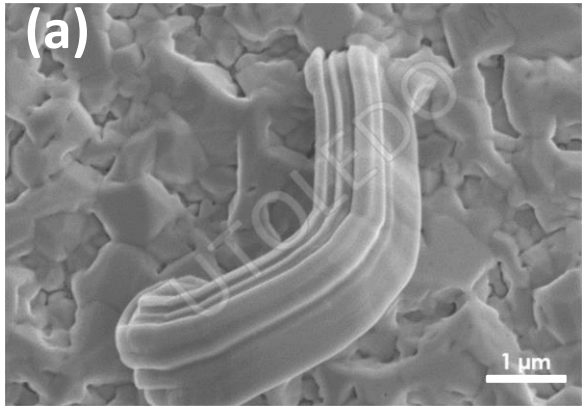


- **Sample lift-out was carried using a in-situ nano-manipulator.**
- **After final cleaning, sample is transferred to TEM.**

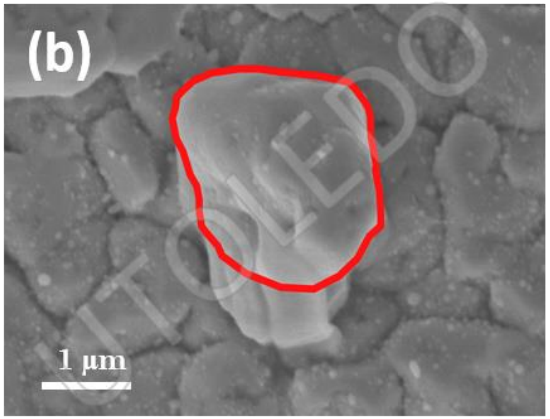
FIB steps: TEM Sample Preparation with FIB – Ben Myers – (NUANCE center)

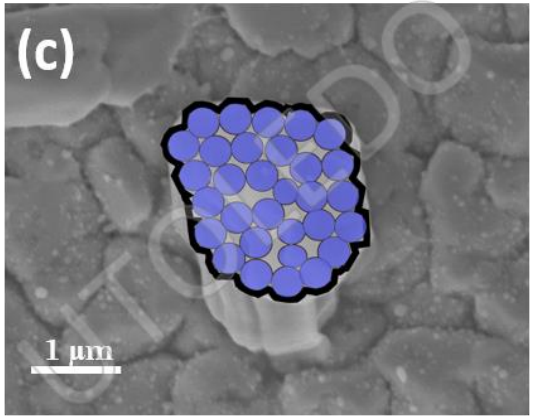
Whisker FIB-TEM analysis

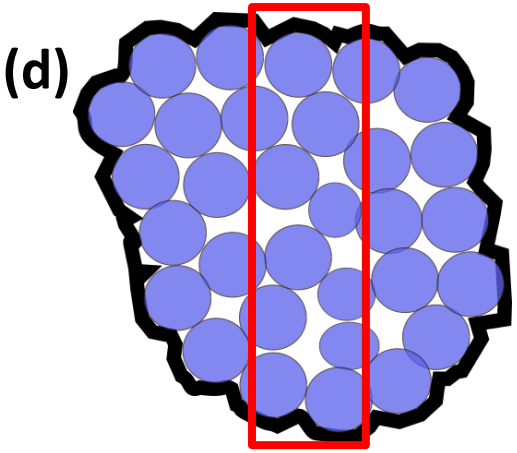
FIB-milling: Whisker TEM sample



- Longitudinal ridges or striations on the whiskers are quite common regardless of their diameter





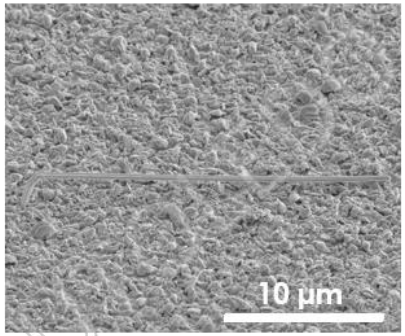


A single whisker → Possible multiple whiskers → Slice of a whisker

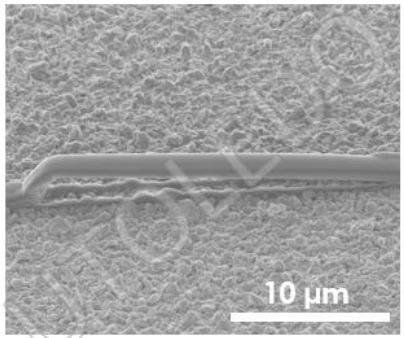
- Almost all whisker nucleation diameters appear to be in the micrometer range, which is substantially greater than crystalline nuclei sizes in solids
- We used FIB-TEM to verify this.

Whisker FIB-TEM analysis

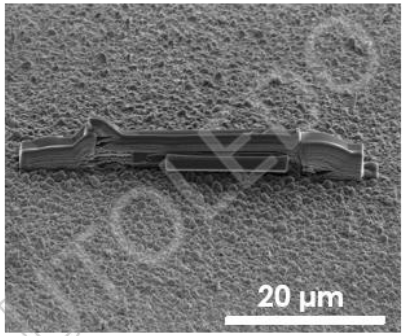
FIB-milling: Whisker TEM sample



Whisker

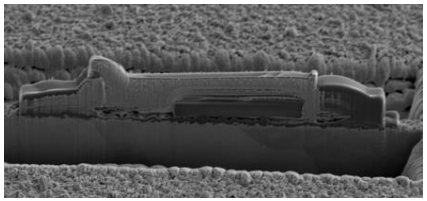


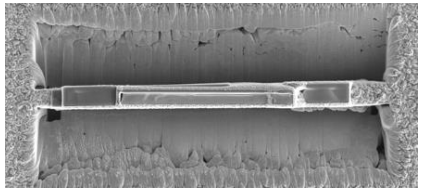
During Pt deposition



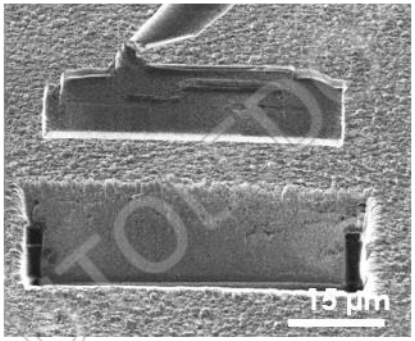
Pt deposited

Trenching

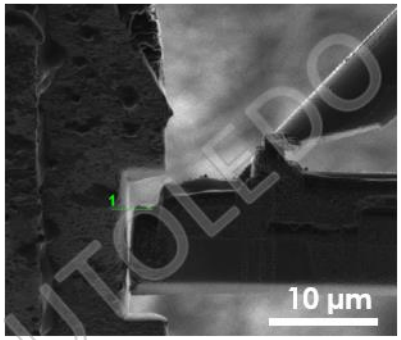




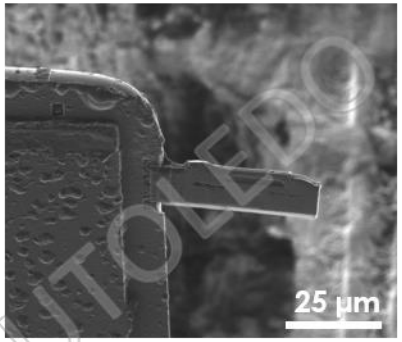
Top-view (trench)



Lift-out



Mounting on TEM grid

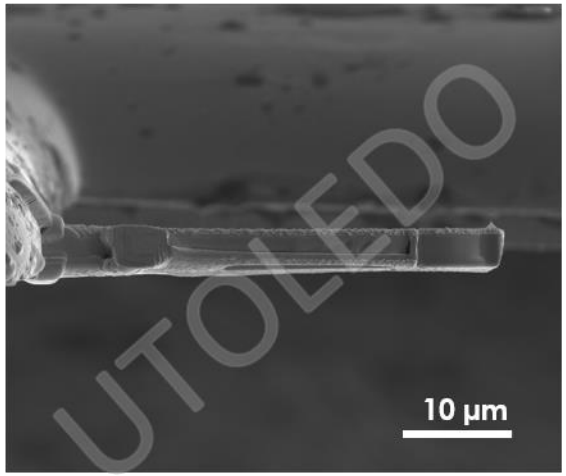


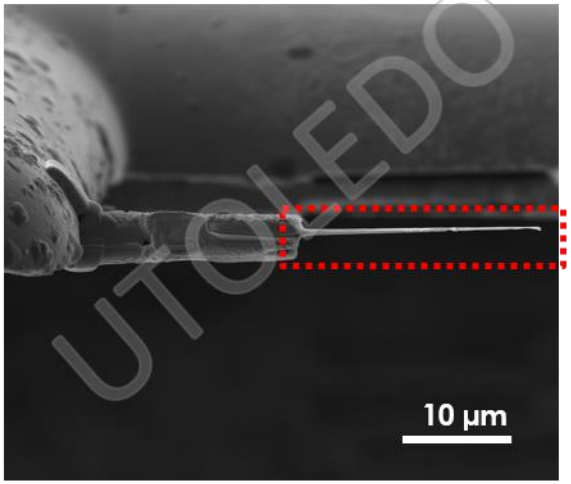
Whisker attached to TEM grid

Whisker FIB-TEM analysis

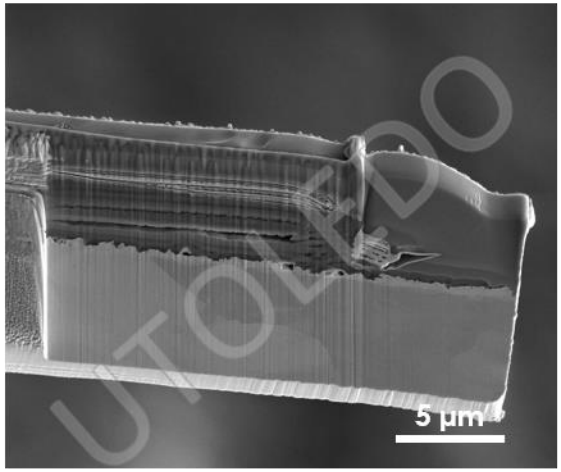
FIB-milling: Whisker TEM sample

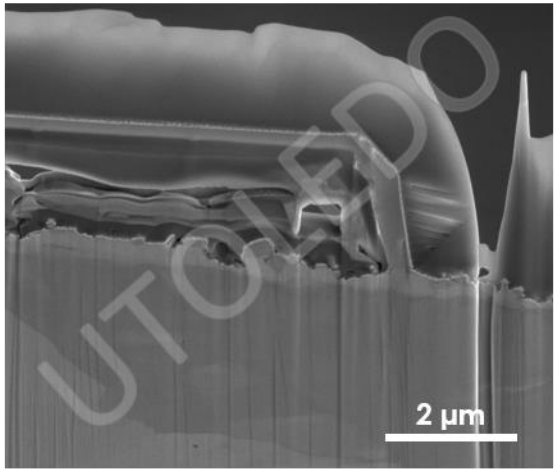
Top view





Side view



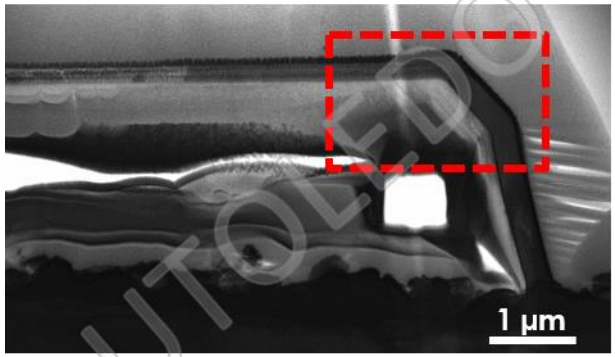


Before thinning

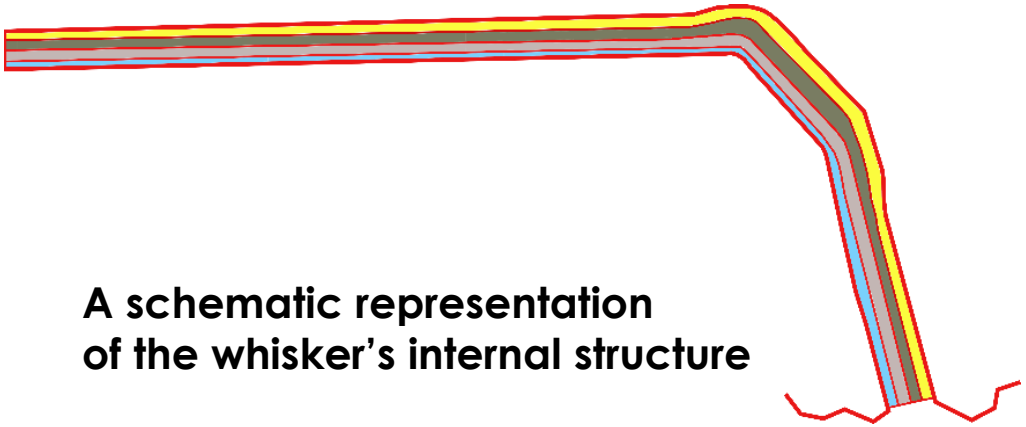
After thinning

Whisker FIB-TEM analysis

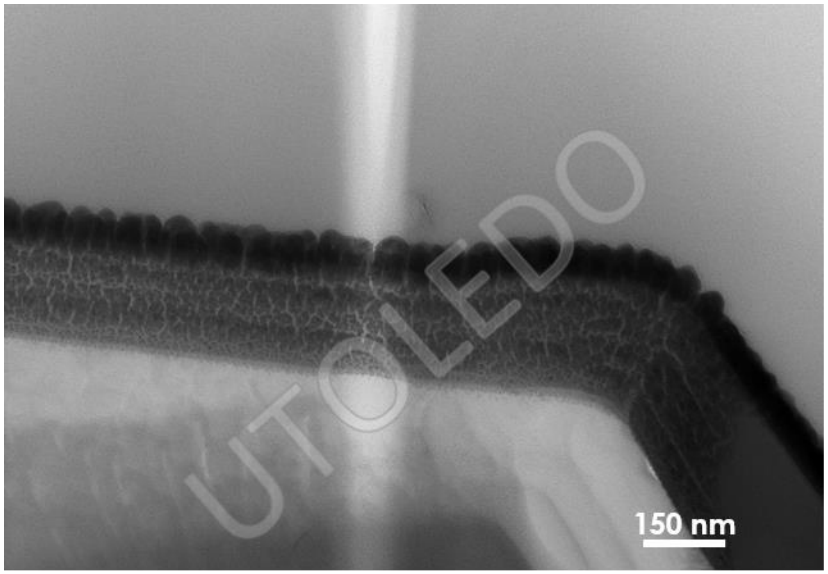
TEM analysis



Low magnification TE image



A schematic representation of the whisker's internal structure

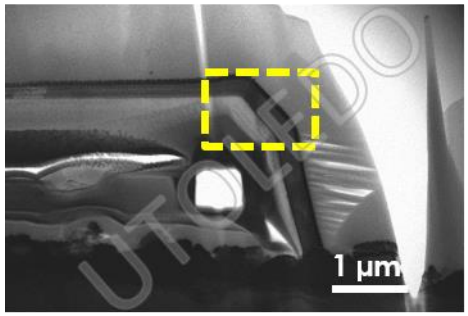


TE image

ZC image

Whisker FIB-TEM analysis

TEM analysis



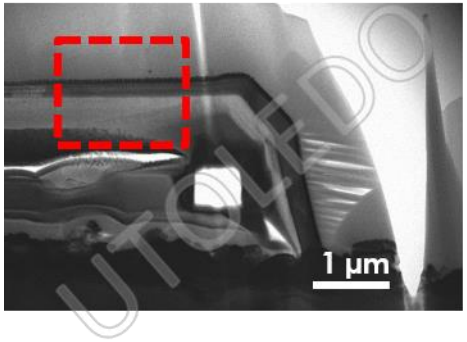
Low magnification TE image

ZC image

- The internal structure observed indicates alignment along the length of the whisker.
- Filament of size 20-50nm appear to comprise the volume of the whisker.

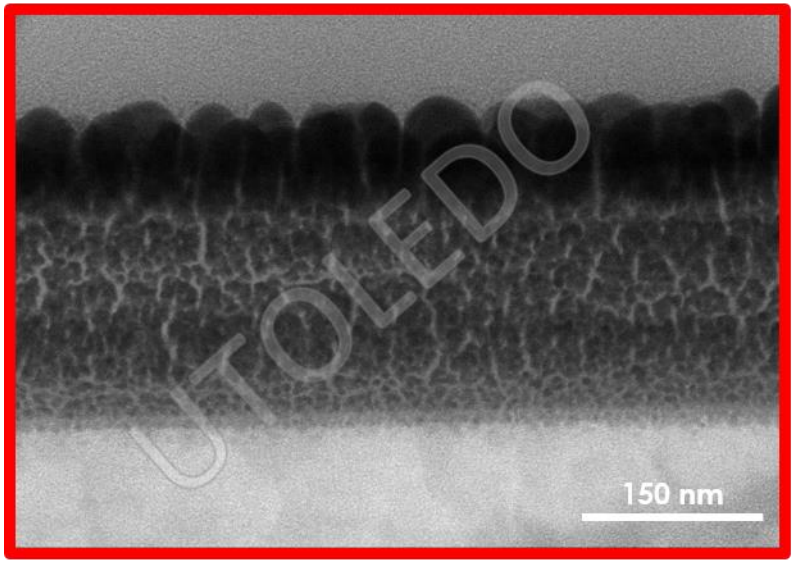
Whisker FIB-TEM analysis

TEM analysis

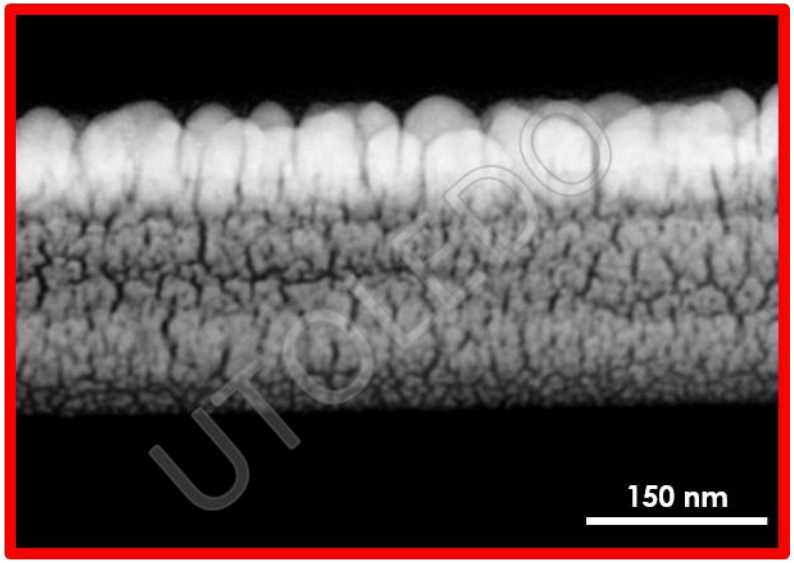


Low magnification TE image

- **Longitudinal internal structure is consistent along whisker's length.**



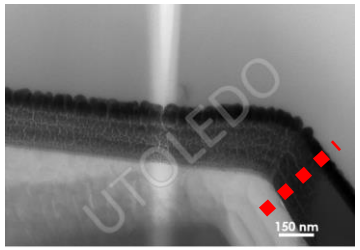
TE image



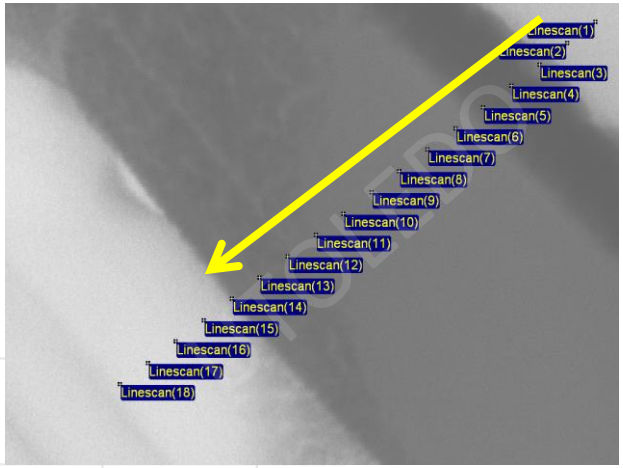
ZC image

Whisker FIB-TEM analysis

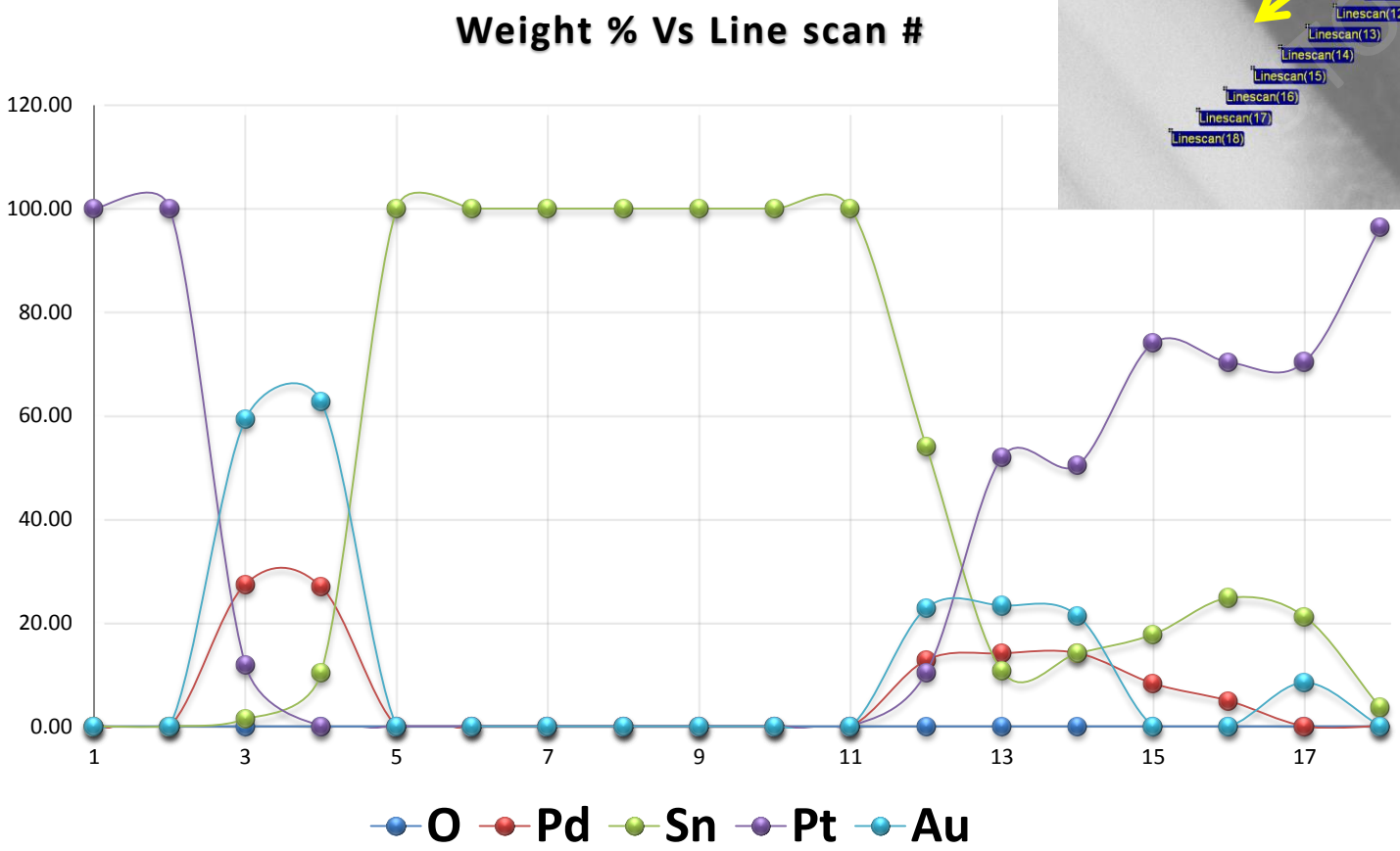
EDS line analysis



EDS line scan

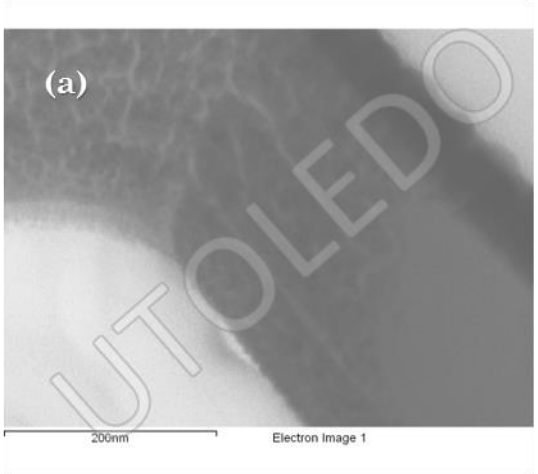


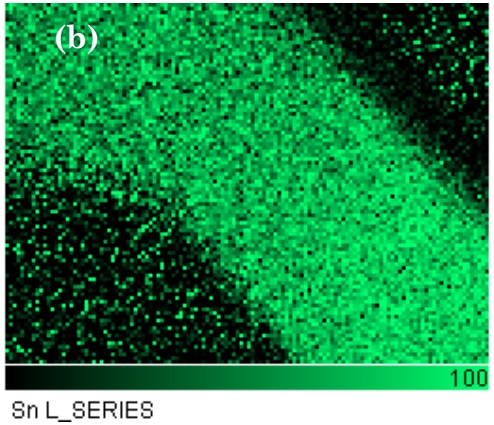
Chemical composition analysis as a function of the location across the whisker



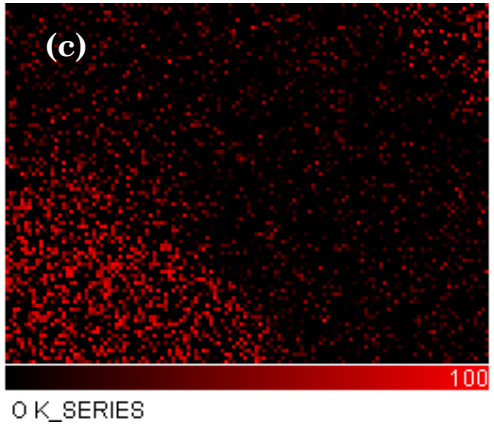
Whisker FIB-TEM analysis

EDS mapping

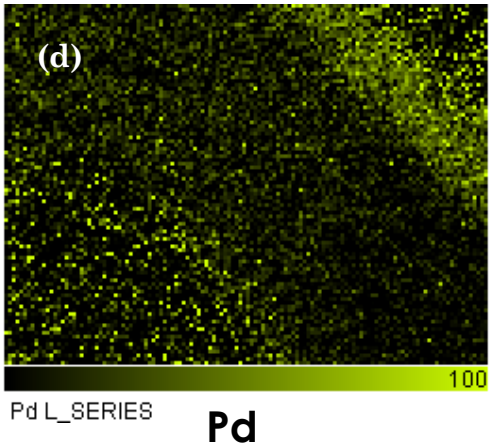




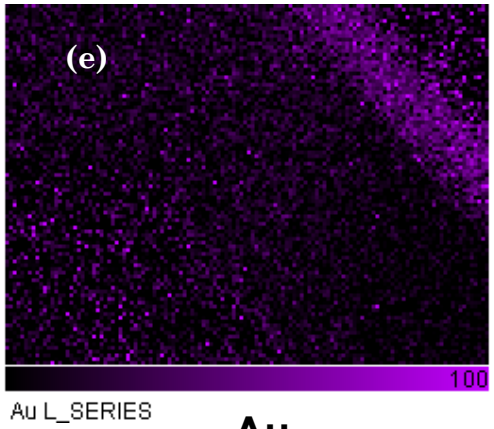
Sn



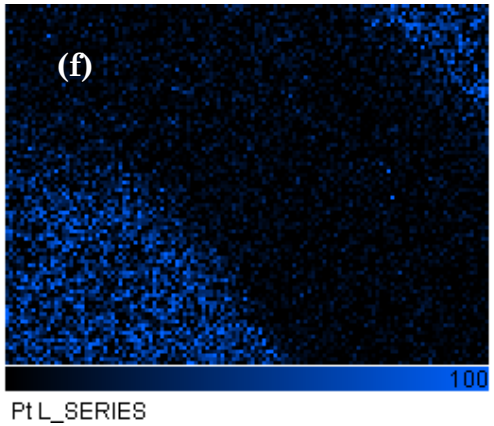
O



Pd



Au

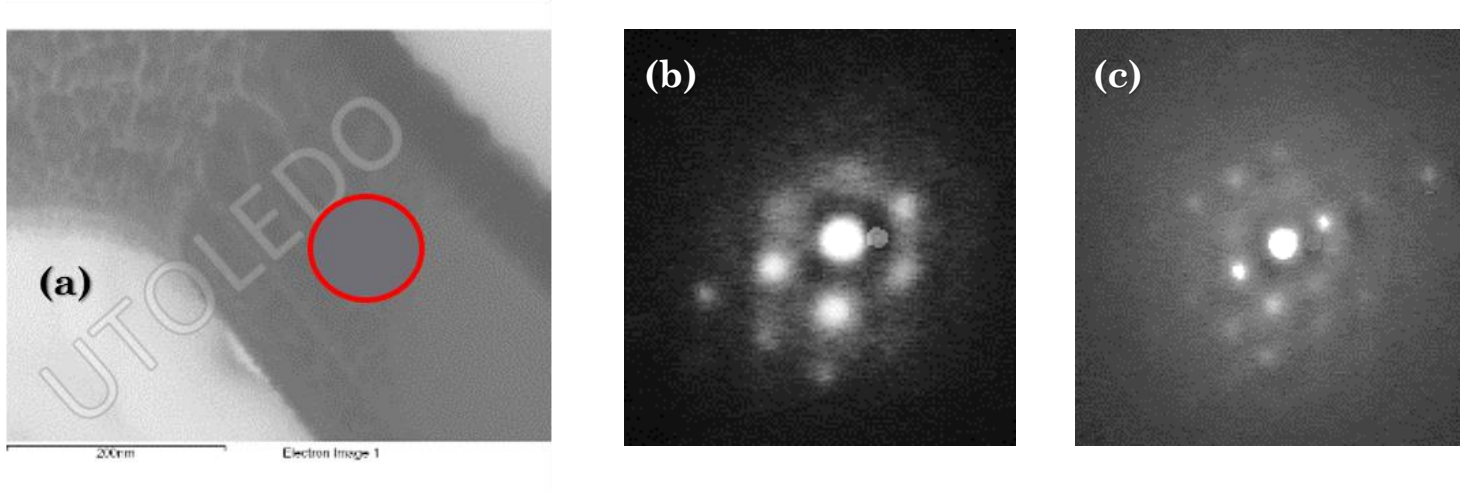


Pt

(b) to (f) show the relative chemical elements distribution over the area shown in (a).

Whisker FIB-TEM analysis

Selective-Area Electron Diffraction (SAED)



(b) & (c) SAED patterns taken from the whisker in (a).

The SAED spot patterns indicate well-aligned crystalline material, which is in agreement with the filaments internal structure interpretation of the TEM images.

Whisker FIB-TEM analysis

Conclusions:

- **FIB cross-sectioning is found to be an effective way of studying the internal structure of Sn whiskers (obtained from pure Sn films, deposited on Cu substrates) at the nanoscale.**
- **A combination of complementary imaging techniques (SEM, TEM, ion-beam imaging, backscattered electron imaging), chemical analysis (EDS), and structure (SAED) techniques were applied in this study.**
- **The results indicate that the whisker internal structure consists of aligned, highly oriented crystalline filaments that consist of pure Sn material.**
- **The results support the hypothesis that Sn whiskers (and metal whiskers, in general) can grow as result of massive nucleation and co-growth of thin filaments.**
- **No evidence of the existence of IMCs in material in the vicinity of the whiskers was found.**