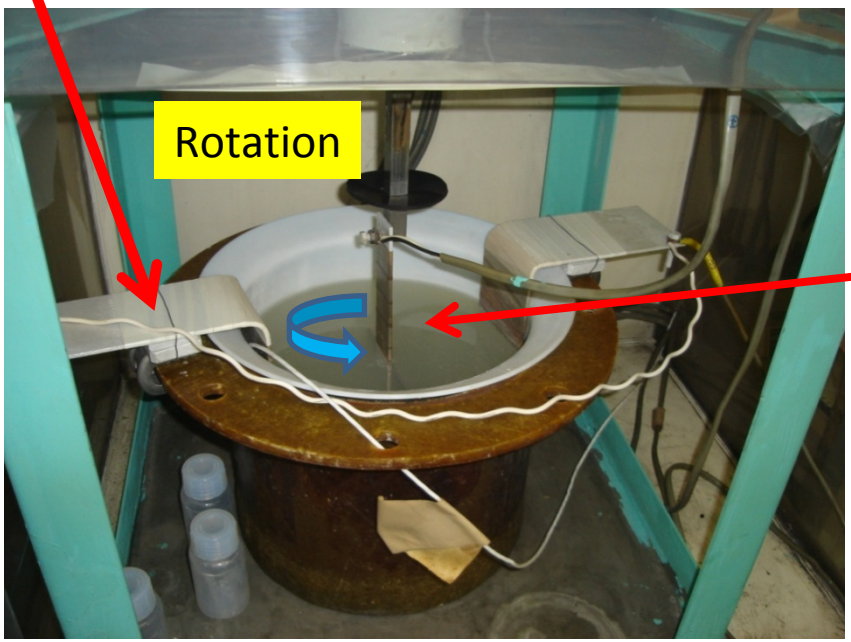
Sponge cleaning study with Nb samples

- Prepare two Nb samples, sample-1 and sample-2.
- Sample-1: BCP(30um)+EP(70um) + U.P.W. rinse.
- Sample-2: BCP(30um)+EP(70um) + U.P.W. rinse + sponge cleaning.
- Compare sample-1 and sample-2 with FS-SEM at Jlab.
- Sample-1': BCP(30um))+EP(70um) + U.P.W. rinse. And analyzed by XPS at KEK.

EP(70 um) of Nb samples at Nomura Plating

Al cathode



Jlab-type Nb sample

Repetition of EP (2 min) + Rotation of sample w/o EP (2 min)

V = 8 ~ 13 (V), I = 3 ~ 23 (A) with oscillation, T= 25 – 50 ^{0}C w/o T control.
[Nb] in EP acid = 4.0 ~ 6.2 g/L during the EP of two samples.
We expected sulfur contamination by aged EP acid.

U.P.W. rinse and sponge cleaning

Moving into a clean-room (class-1000) after EP(70 um)

Ultra Pure Water rinse (20 min)

sponge cleaning with U.P.W.

+ Ultra Pure Water rinse (5 min)

Wipe 10 times for each arrow = 40-times wipes in total

Nb-sample-2

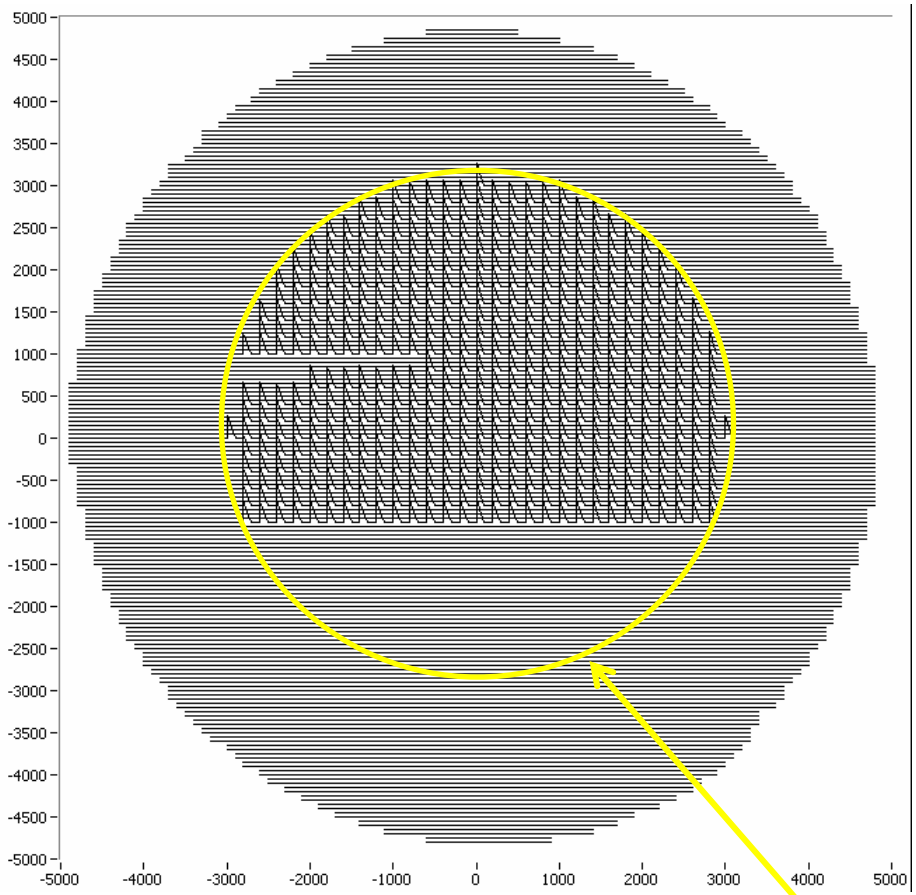
Packing under U.P.W.

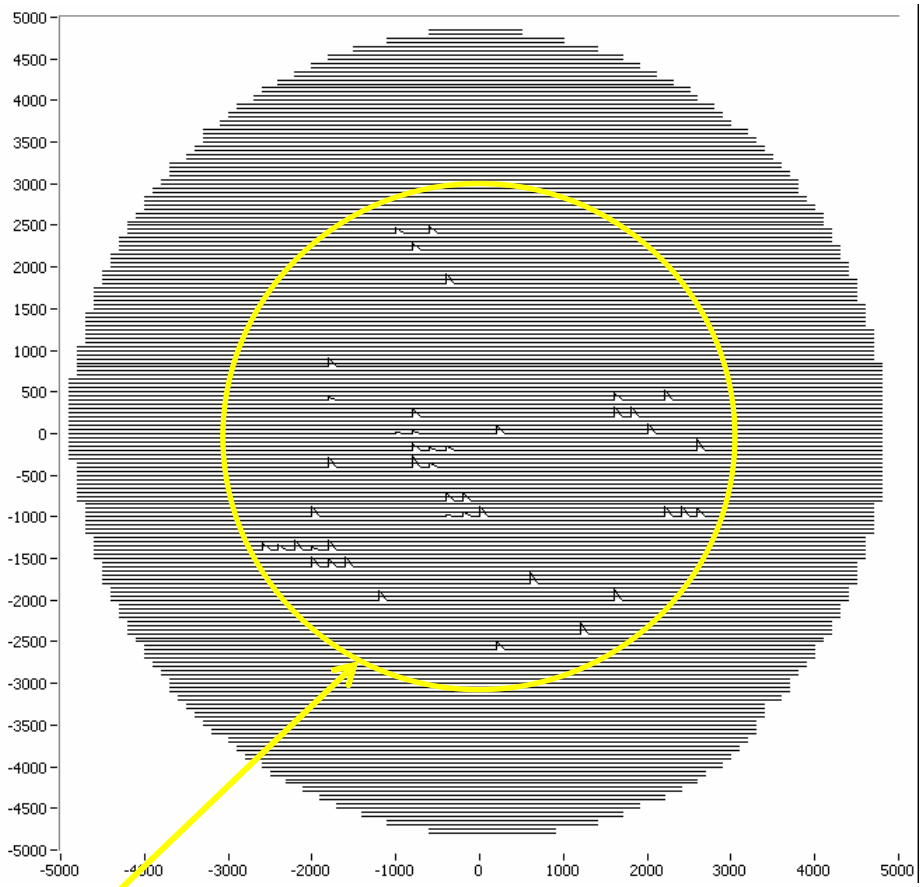
Nb-sample-1

Packing done

Sample#1: many field emitters
Low FE onset ~ 10 MV/m

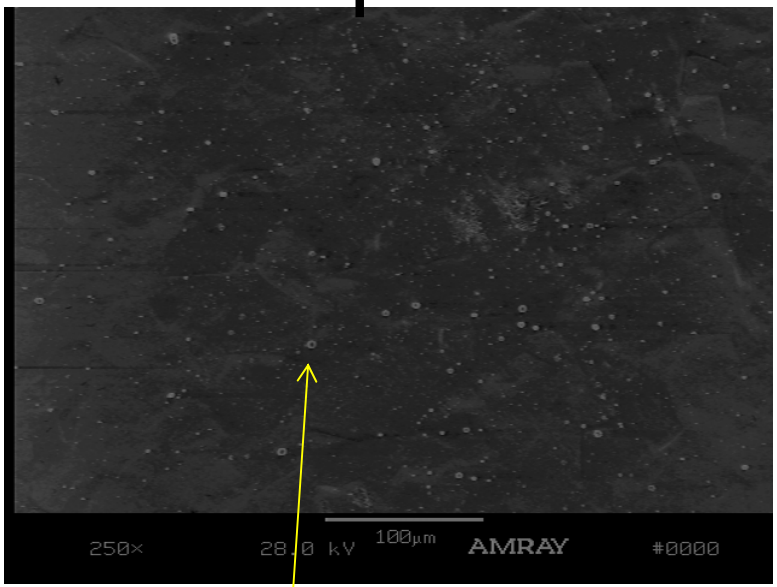
Sample#2: much less field emitters
Higher FE onset



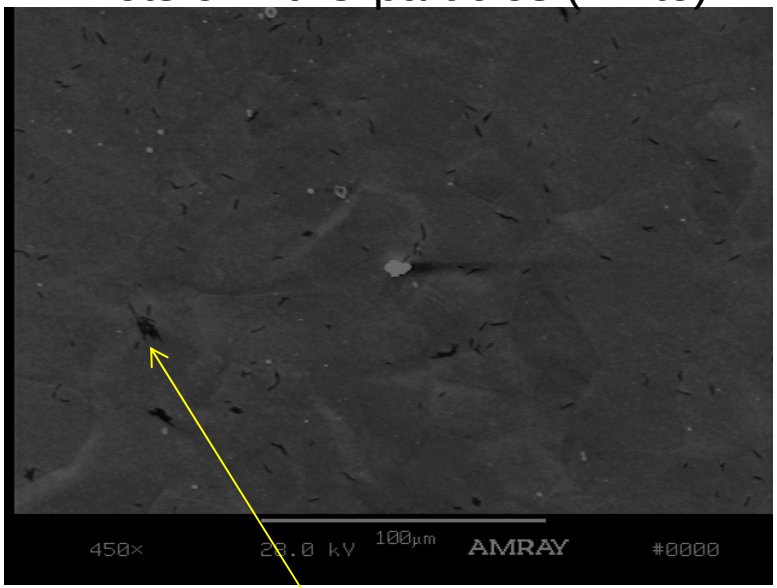


Area inside circle is scanned

Sample#1

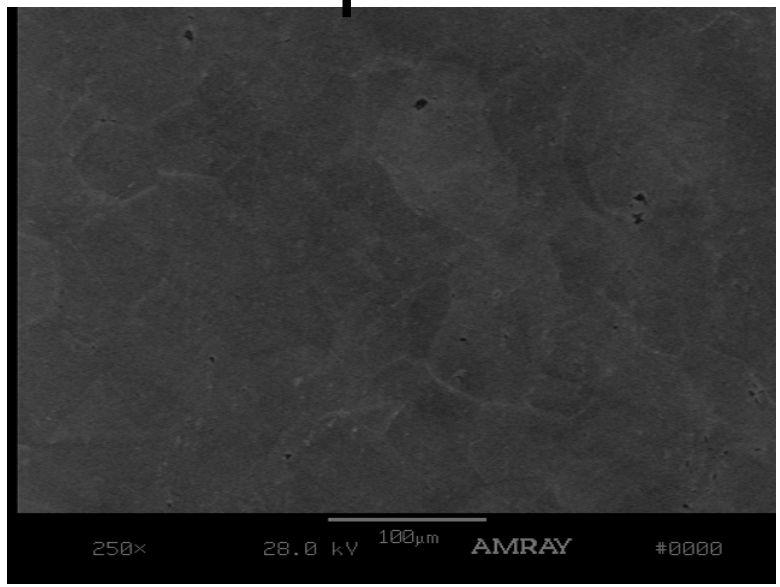


Lots of Nb-O particles (white)

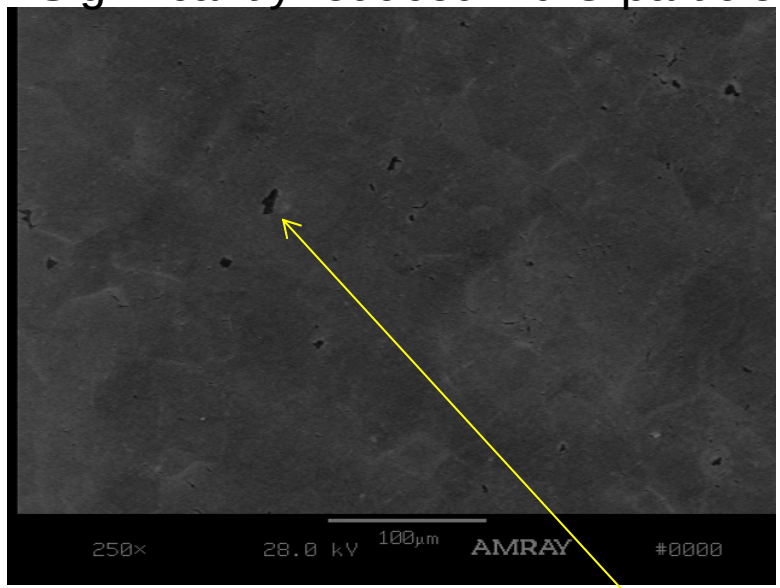


Suspected bacteria (black worm)

Sample#2

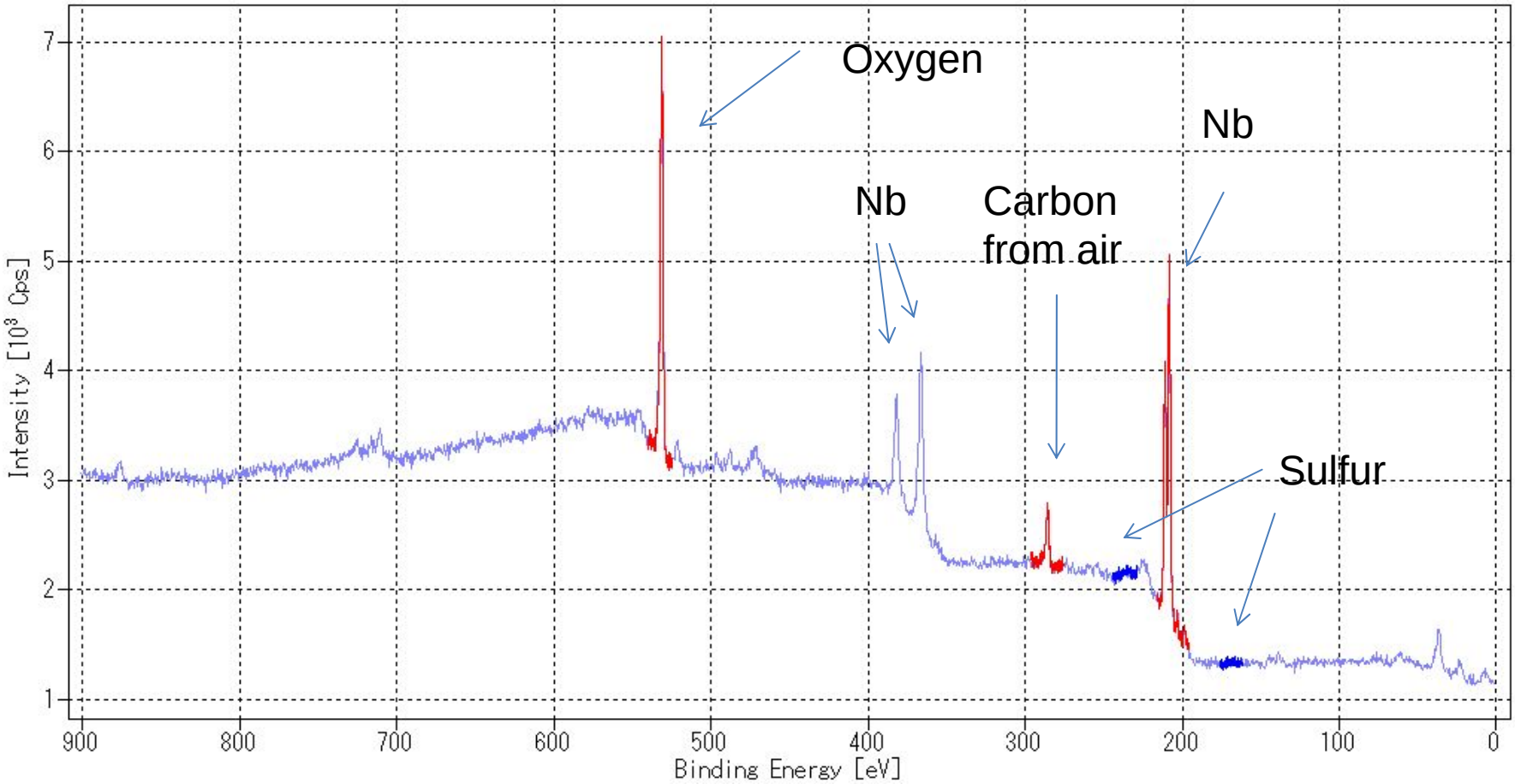


Significantly reduced Nb-O particles



Somehow less bacteria growth, but new type of Contamination (black particulate)

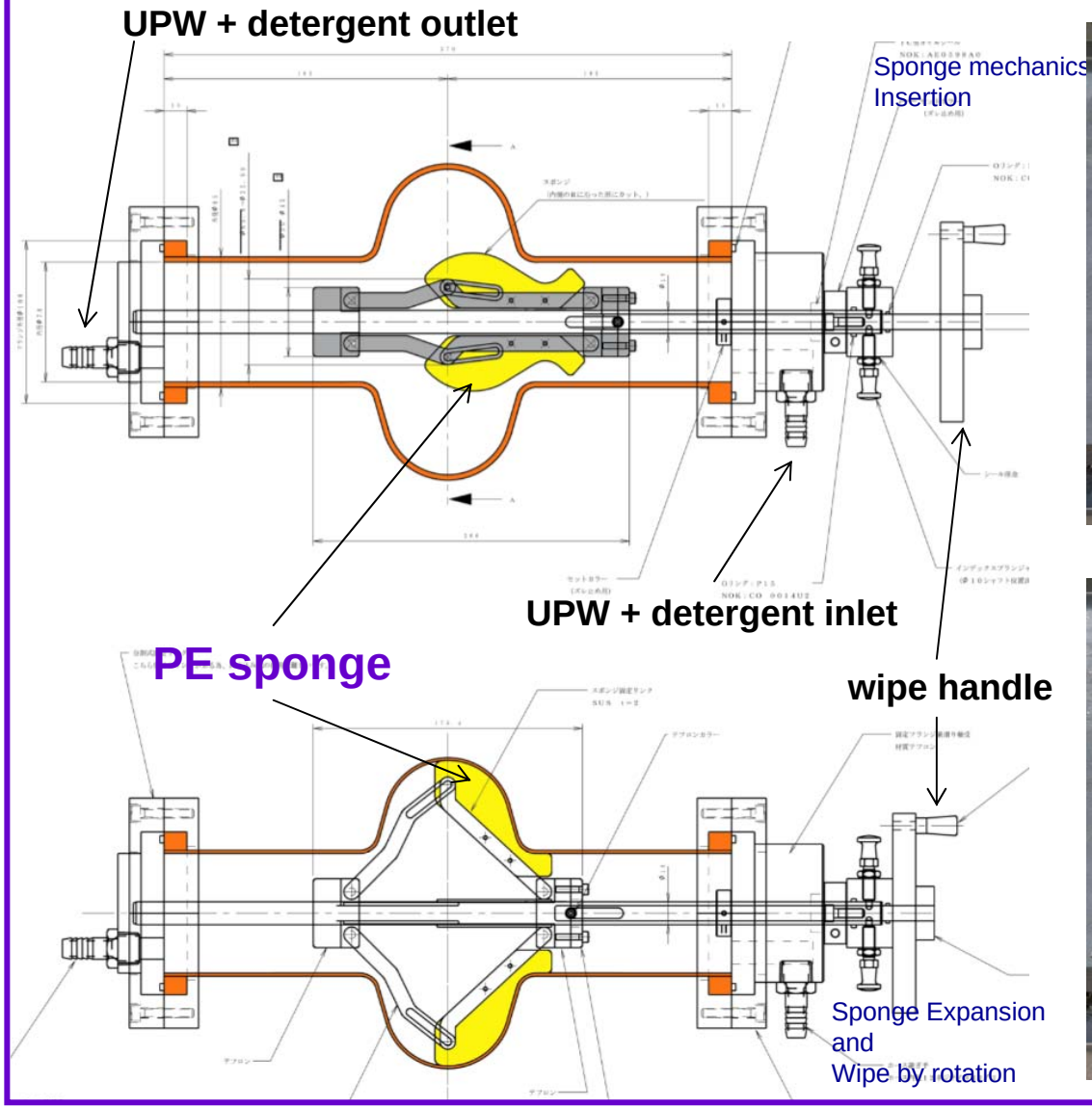
XPS Analysis of KEK-type Nb-sample-1 (BCP+EP w/o sponge cleaning) at KEK

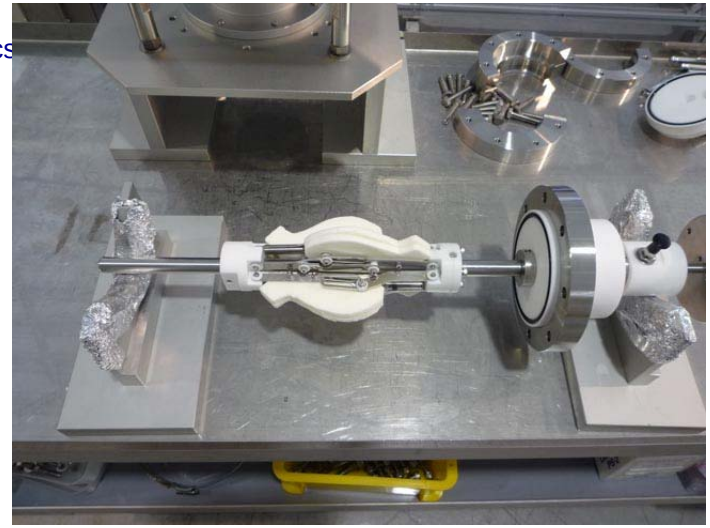


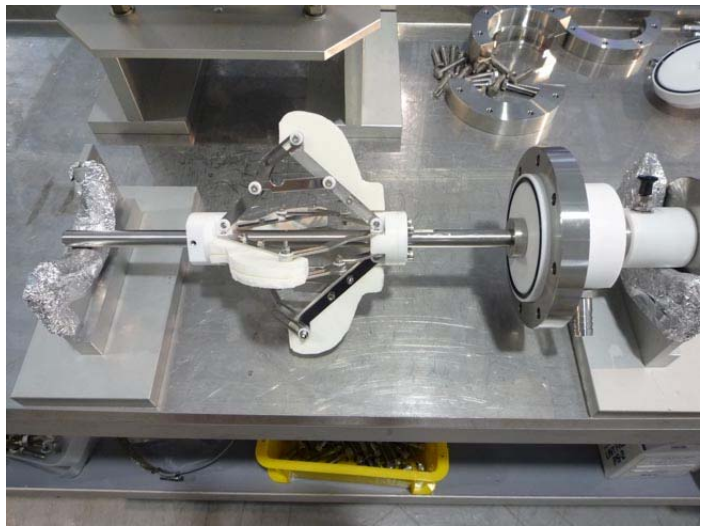
Atomic percentage = Nb:19%, O:55%, C:25%
(Red peaks were used for the estimation)
Depth of analysis ~ 0.5 nm

Sulfur: ~ 0%

Prototype sponge-cleaner for single-cell cavity
First wipe/fitting test was done for single-cell cavity



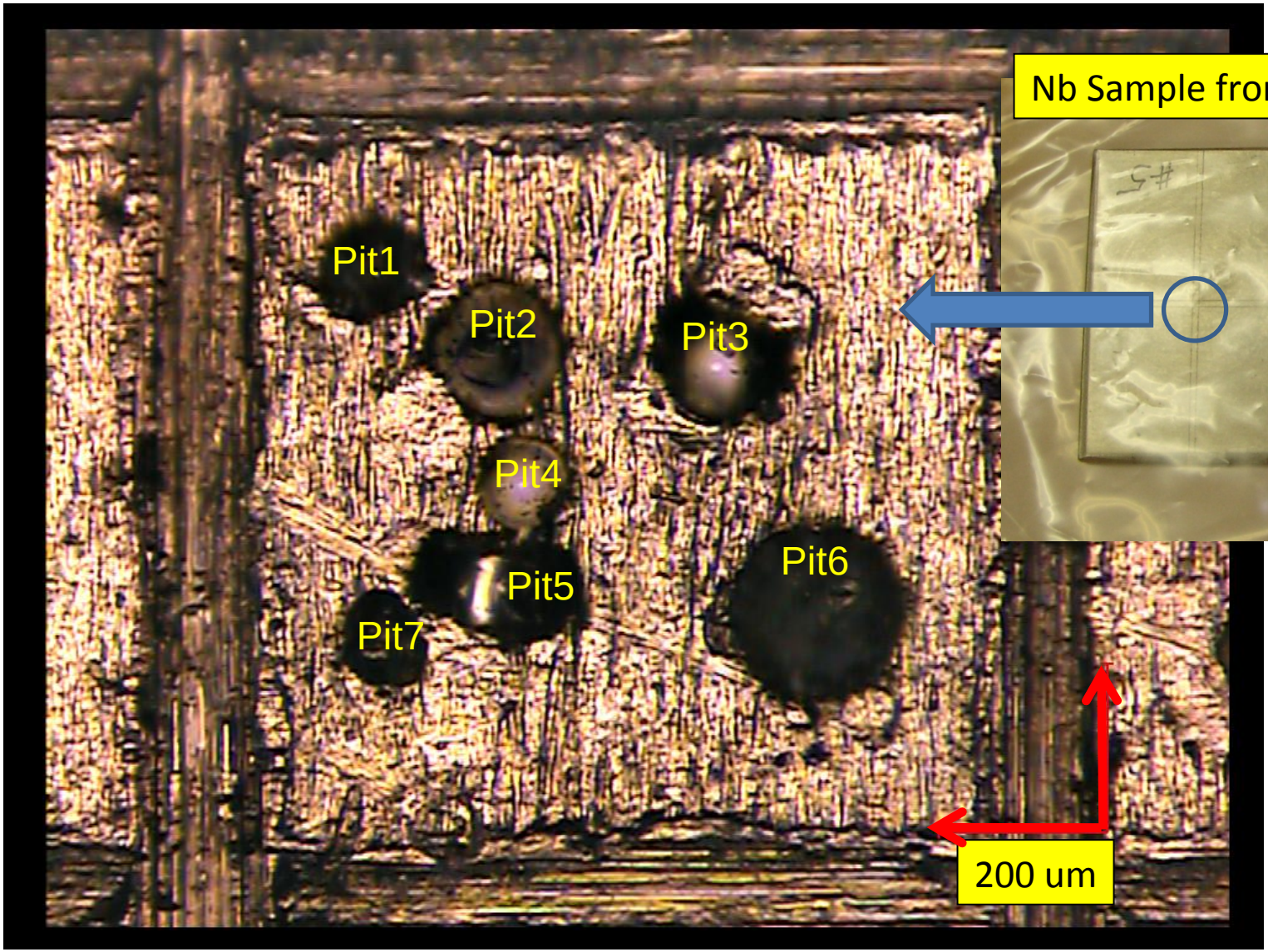




Summary of sponge cleaning study

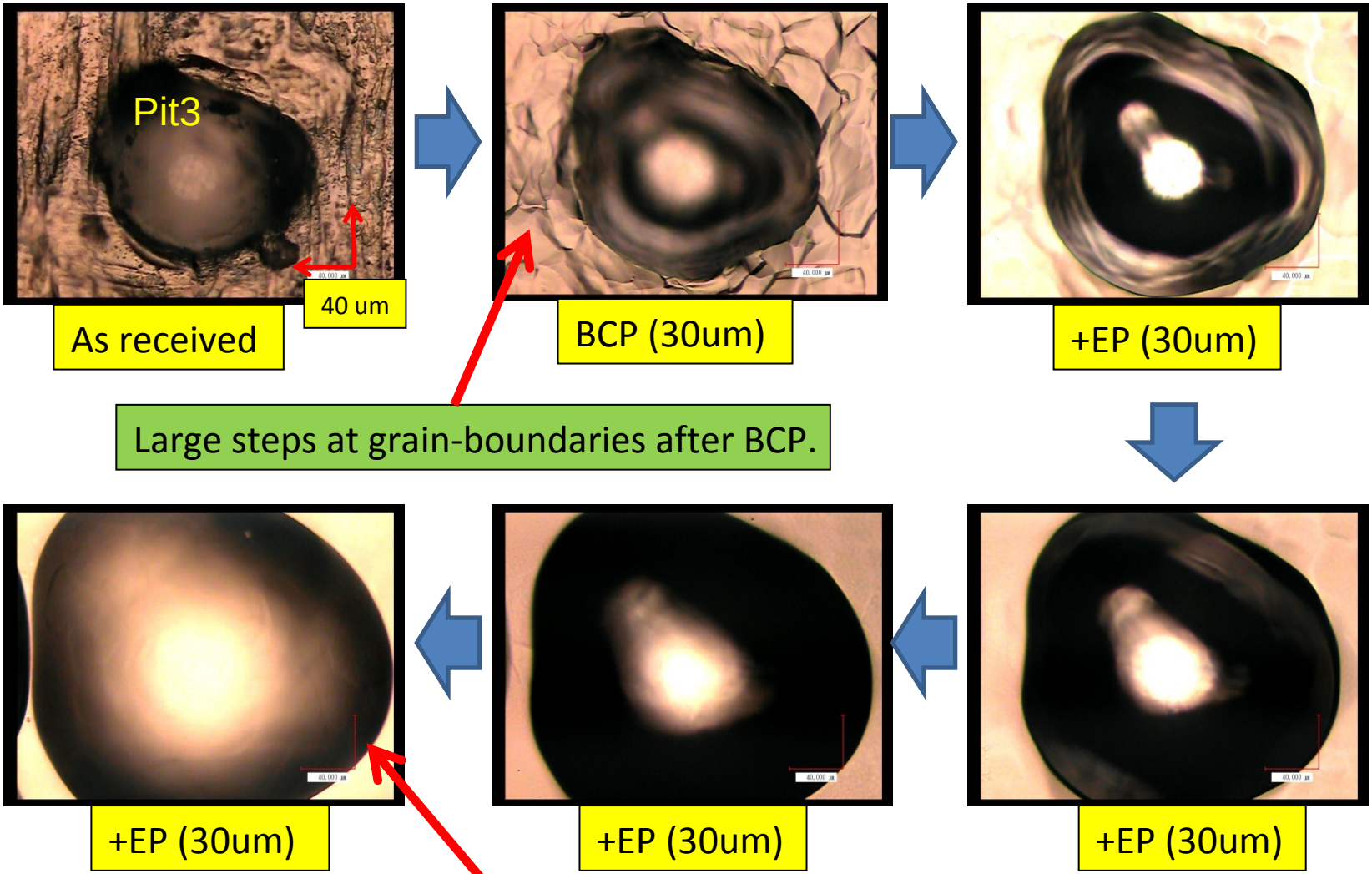
- Two samples w/ and w/o sponge cleaning after EP-process were analyzed by FS-SEM.
- lots of field emitters were found on the sample w/o sponge cleaning.
- Few field emitters were found on the sample w/ sponge cleaning.
- Sulfur was not found on the sample (w/o sponge cleaning).
- Lots of NbxOy particles (field-emitter) were found all over the surface of Nb after EP, but most of these particles can be removed by sponge cleaning.
- More detailed information can be found at http://ilc.kek.jp/JFK-S0/

Nb sample with artificial pits from JLab

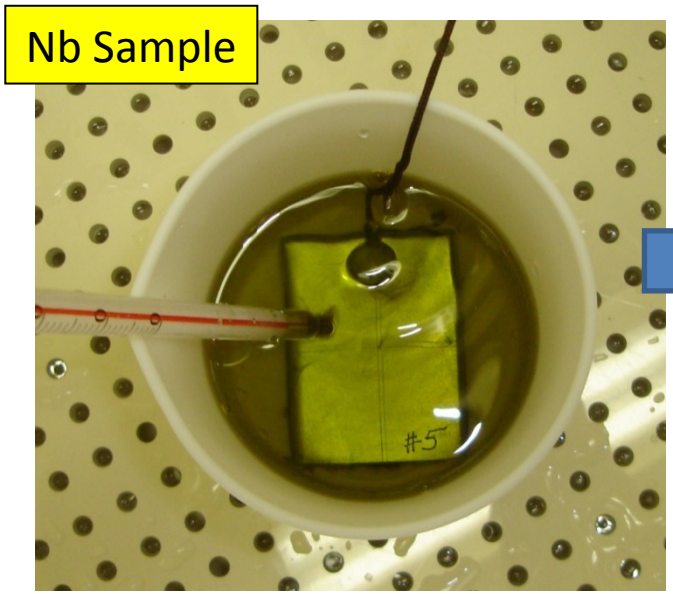


On interested surface of both samples, there are marked lines for purpose of location referencing.

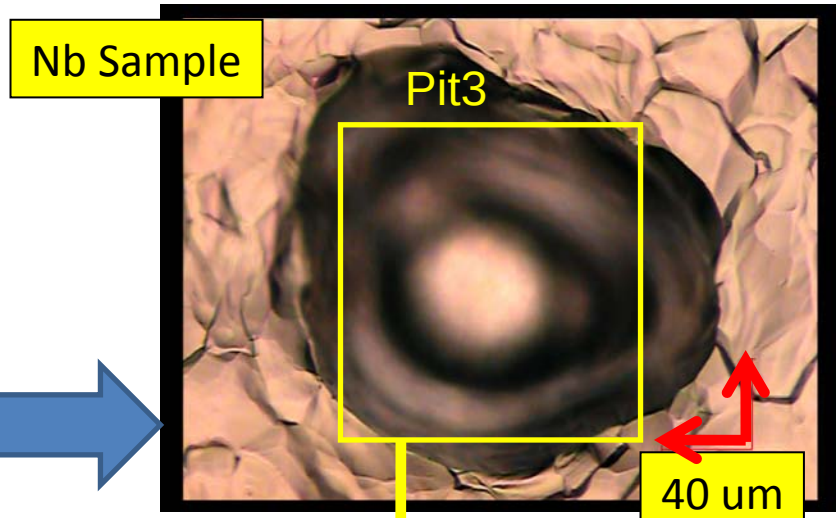
JLab Nb sample with artificial pits

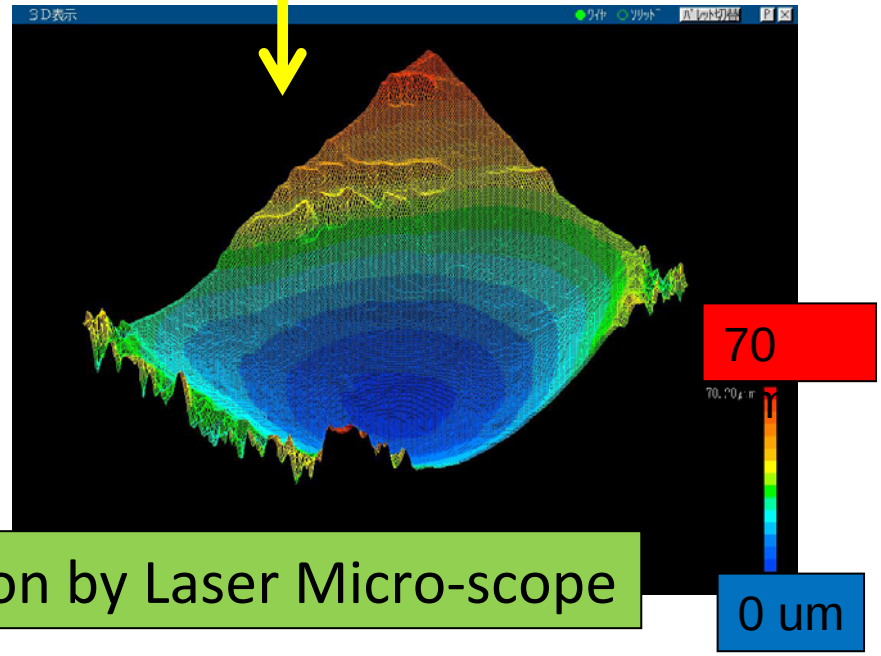


Jlab Nb sample with artificial pits



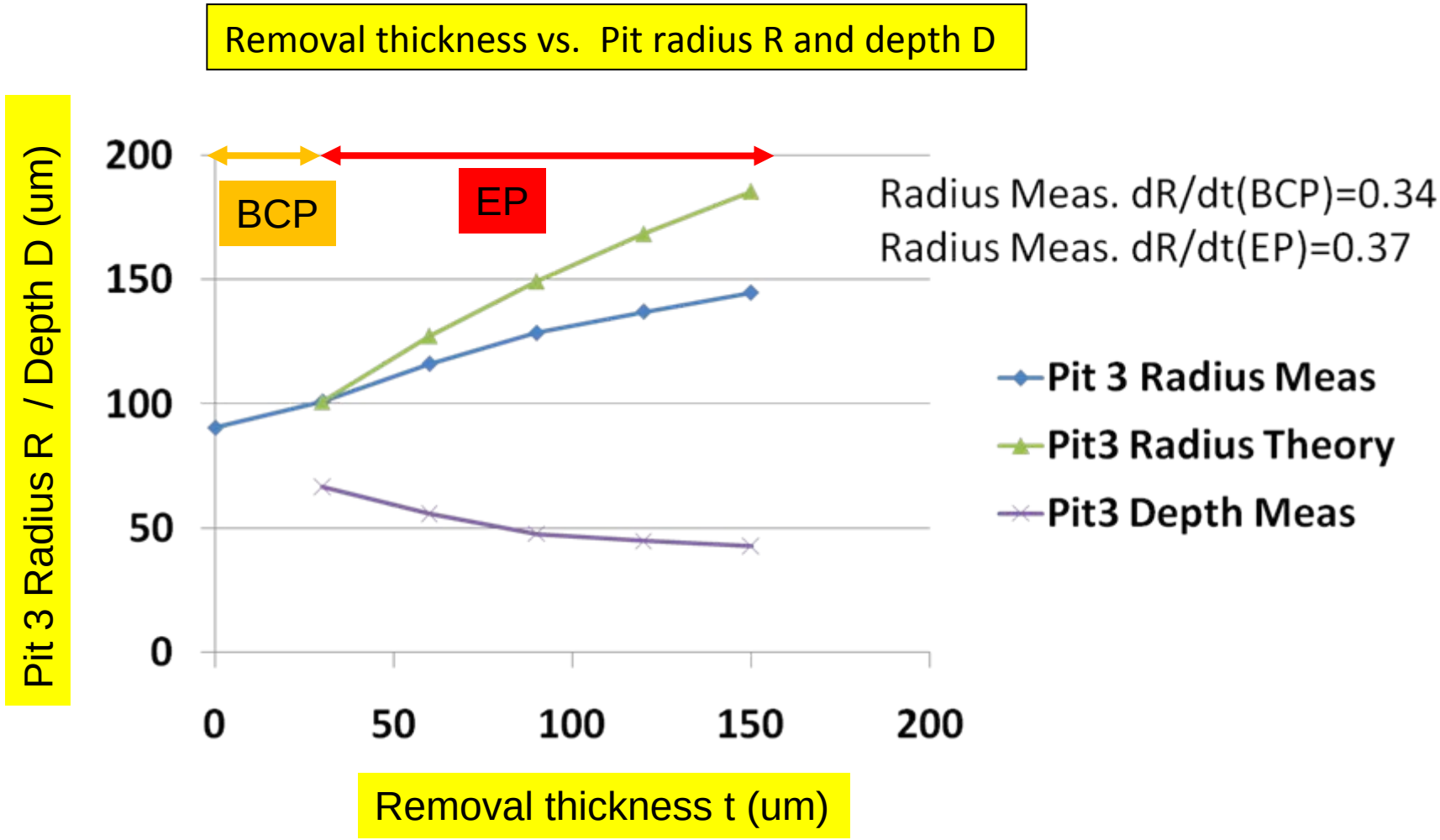
BCP (30um) at STF/KEK





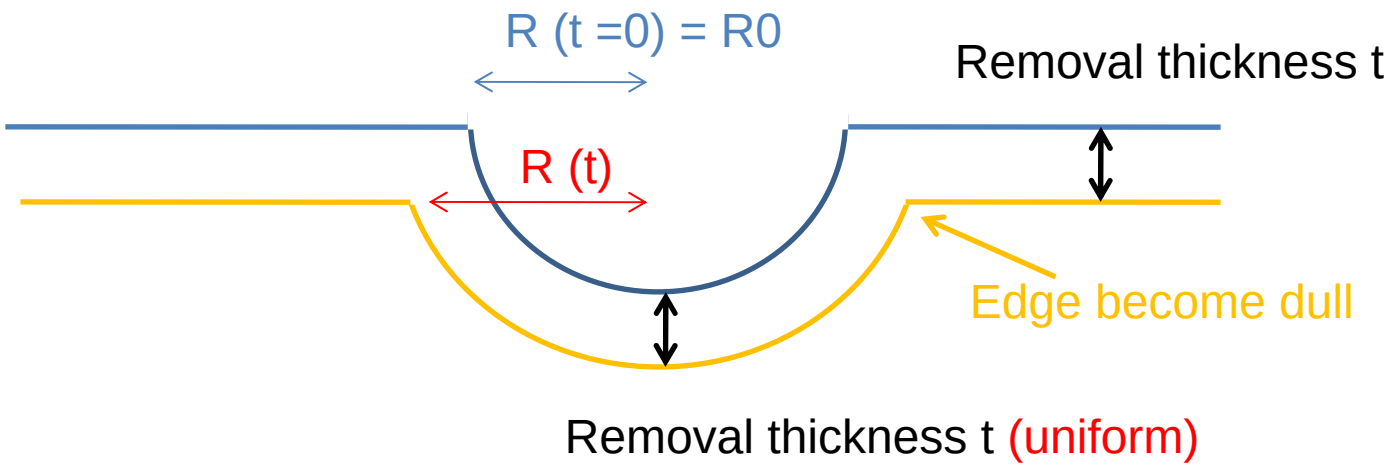
Observation by Laser Micro-scope

JLab Nb sample with artificial pits



Jlab Nb sample-1 with artificial pits

Radius for Uniform Removal (Theory)

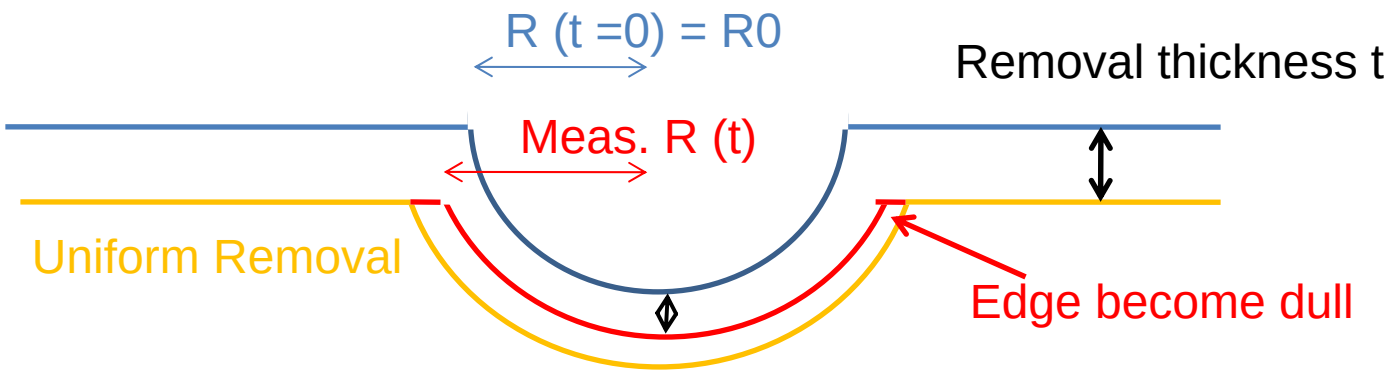


$$R(t) = SQRT (R0 \times R0 + 2 \times R0 \times t)$$

In this theory, the depth of pit is constant / no change.

Jlab Nb sample with artificial pits

Measured Radius and Depth



Removal rate is less in the pit

Radius increases slowly and the depth becomes shallower.

However, EP seems not to round the edge of pit very effectively / selectively.
EP is not all mighty. All pits should be removed before EP process.

Summary of artificial-pit EP-process

- Nb sample with artificial pits were EP-processed and the shape of pit was observed with laser / optical micro-scope.
- EP process makes the steps at grain-boundaries smooth.
- The removal rate in the pit was smaller than that on the flat surface.
- The radius of pit increase slowly and the depth of pit becomes shallower while EP develops.
- In a result, the edge of pit became relatively dull, but EP process did not remove the edges of pits effectively / selectively.
- EP process is not almighty. All pits should be removed before EP process.
- More detailed information can be found at http://ilc.kek.jp/JFK-S0/