Basic R&D for High gradient ILC SC cavity in KEK-STF

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Gradient limitation in Superconducting Cavities;

(1)Sudden thermal quench

seems to be by defects which have magnetic field enhancement.
detection tools: T-map, Inspection camera
counter-measure (repair): more surface removal (CP, EP, etc),
or barrel polish/local grinding, …

(2) Field emission and slow thermal quench

seems to be by contaminations which will be a field emitter.
detection tools: T-map, X-ray map?, ???
counter-measure : ethanol rinse, degreaser rinse,
or sponge wipe, …

To understand what is residuals on EP'd Nb surface is important.
When we have a tool to observe its residuals,
then development of counter-measure will be easy and scientifically validated.

High Gradient basic R&D in STF

1. Electro-chemical Polishing Facility in STF

1. Surface Analysis of EP'd Nb
2. Sulfur removal
3. Sponge wipe

contaminations

1. Inspection Camera (upgrade)
2. Local grinding

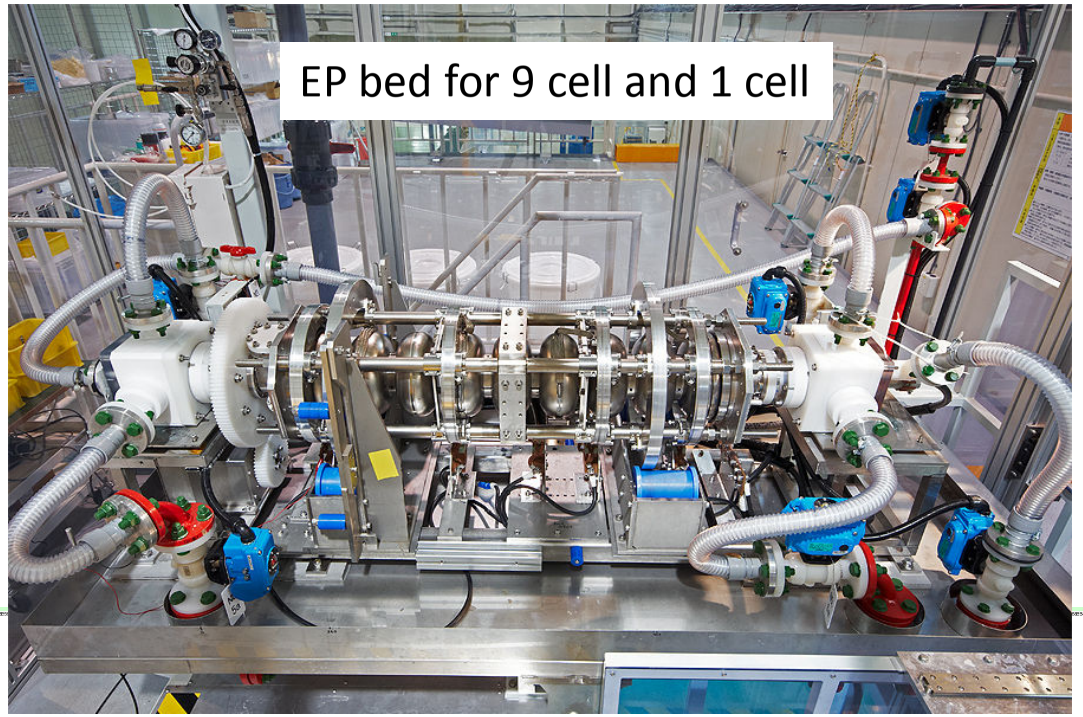
defects

New EP facility at STF

- Introduction -

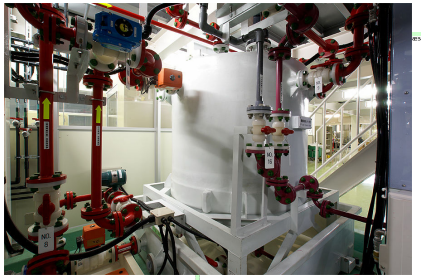
STF EP system

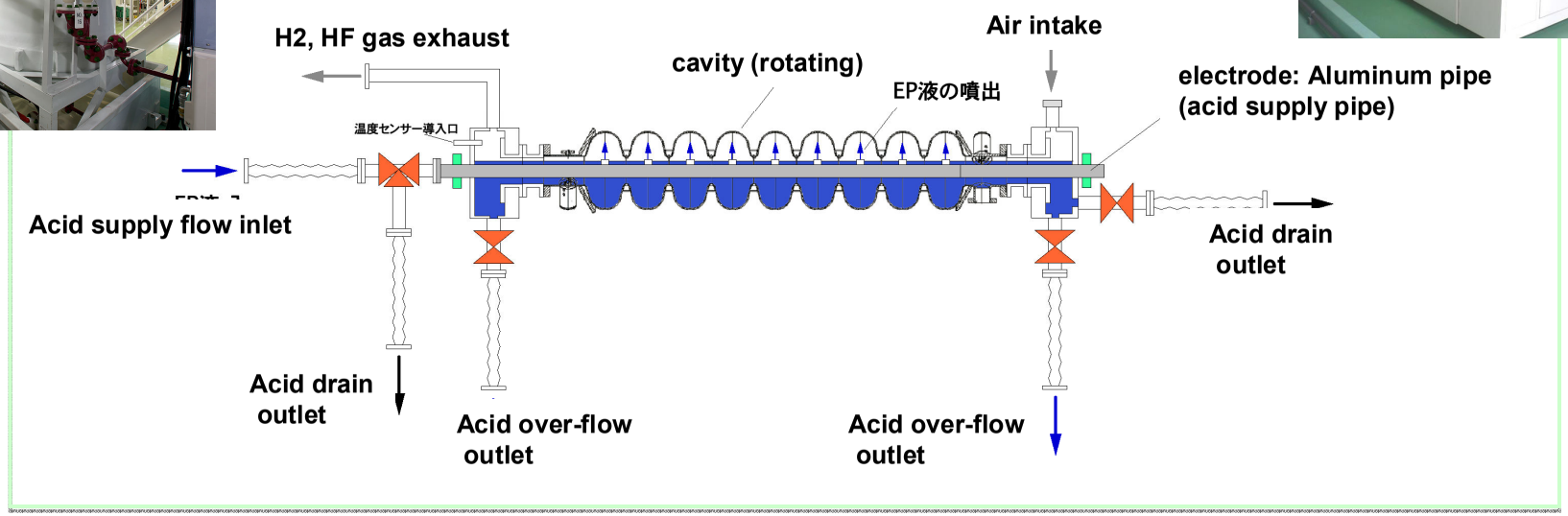
EP acid:
$HF + H_2SO_4$



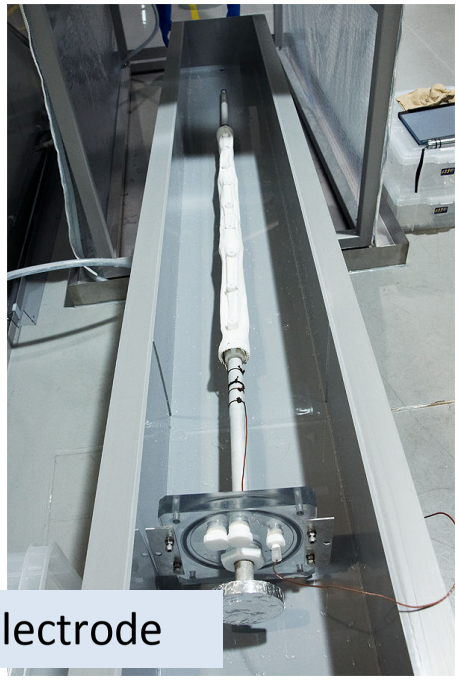
UPW system

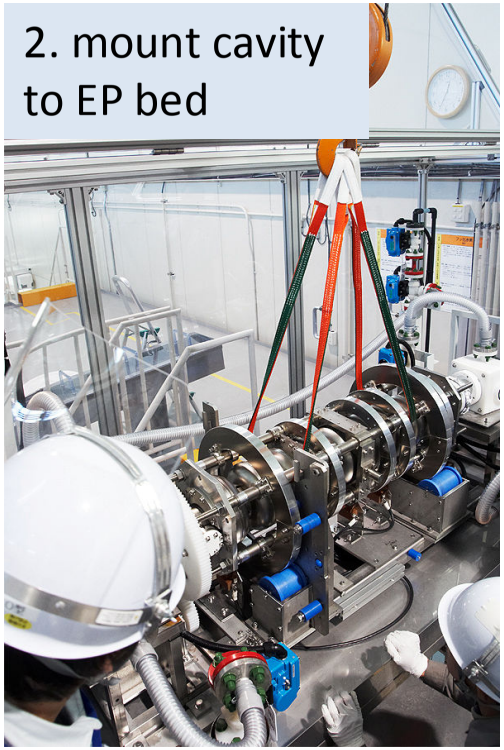
1000L acid tank

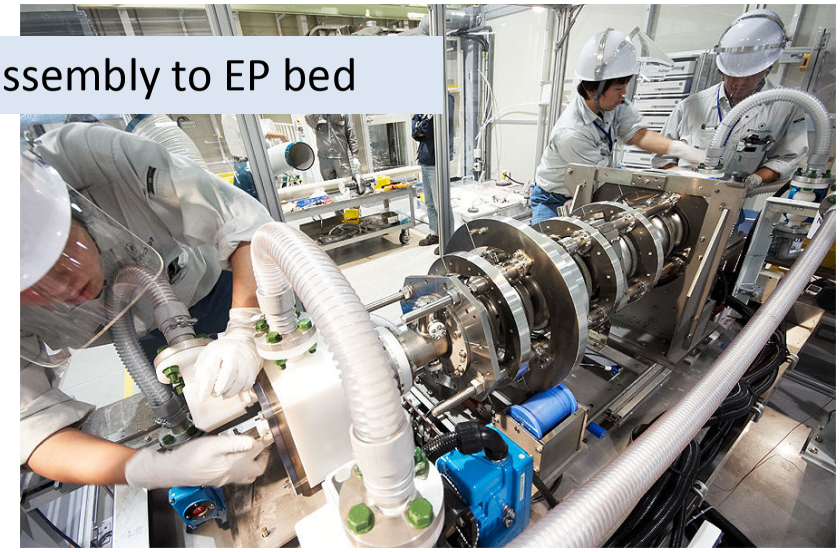




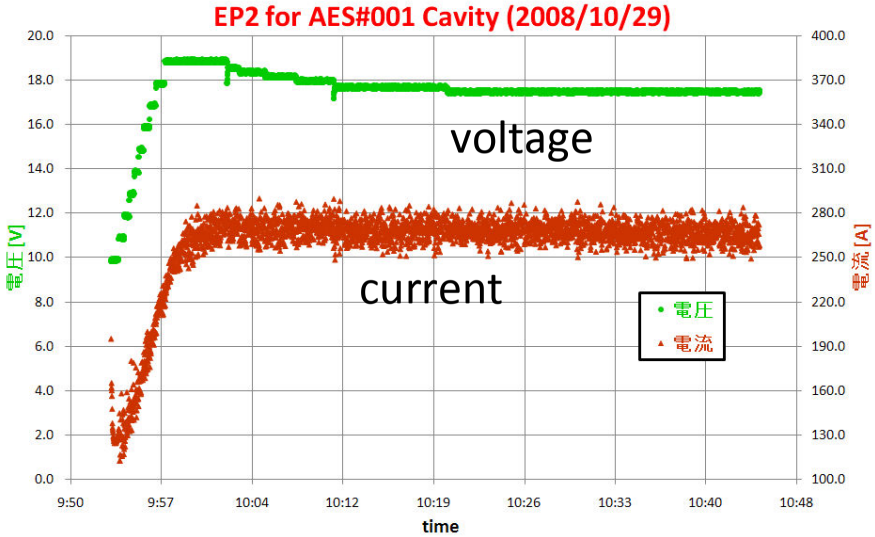
STF EP system commissioning

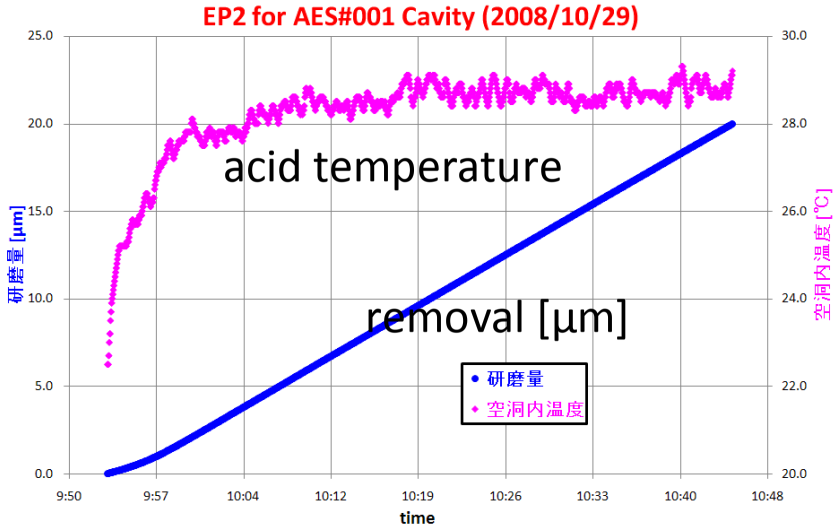


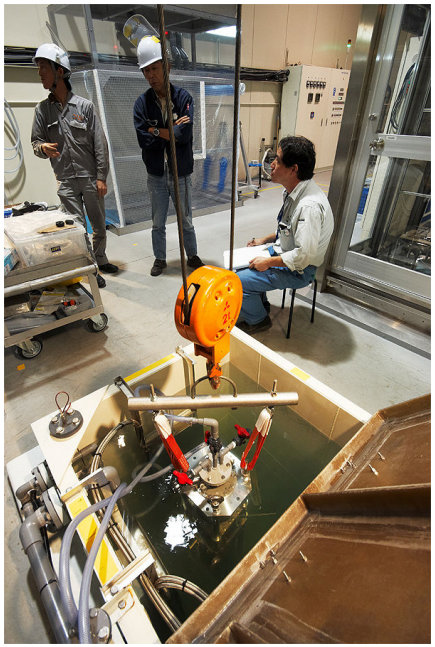




STF EP system commissioning



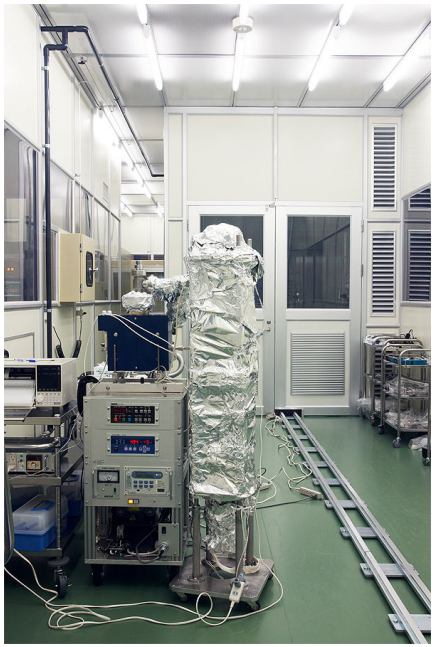




ultra-sonic cleaning

HPR installation

HPR dismounting

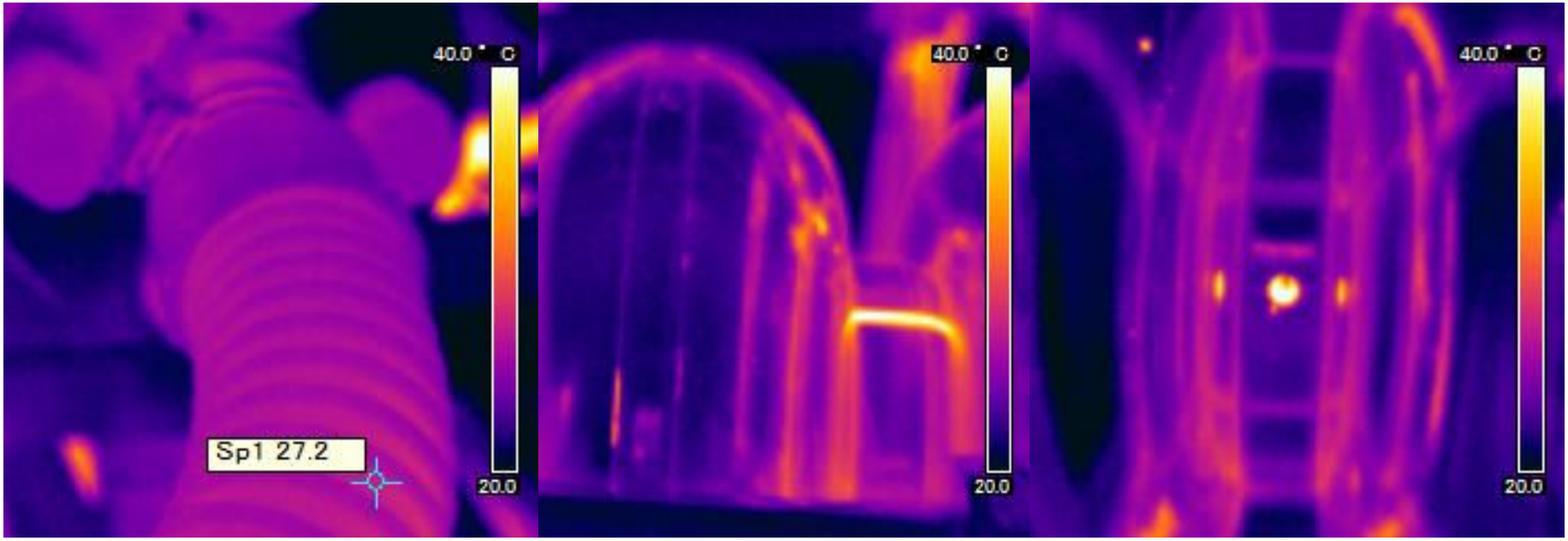


120C bake

Monitoring cavity surface temperature

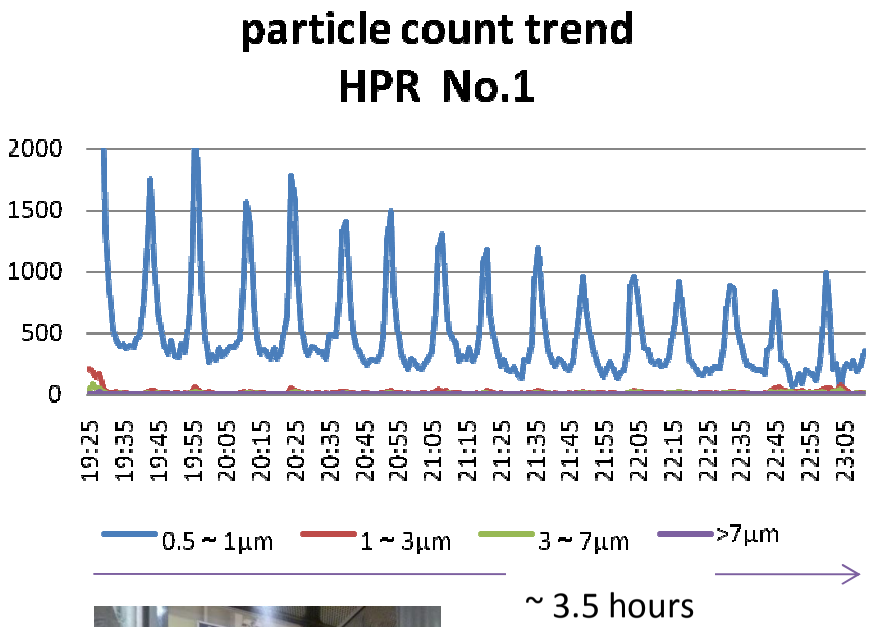
Use of infra-red viewer during EP

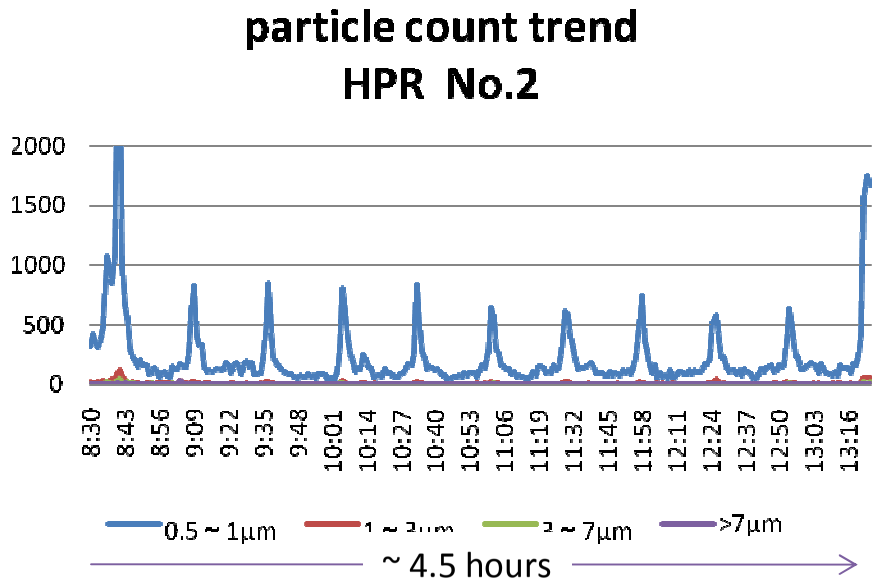
EP acid over-flow pipe **No.7 Cell** **No.2 ~ 3 Iris**



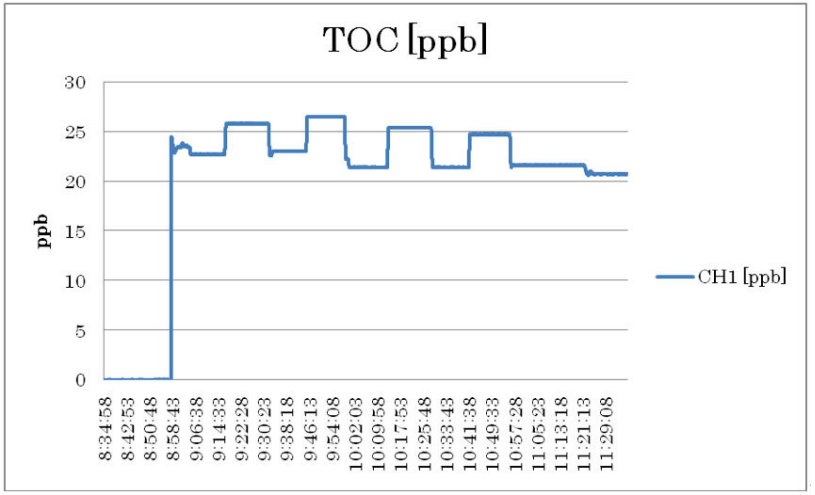
Monitoring particles and TOC in the wash-out water of HPR

- HPR with port open 4Hr. (No.1) 2009/02/18 BL#5
- HPR with port close 4Hr. (No.2) 2009/02/19 BL#5





HPR



Surface analysis of EP'd Nb

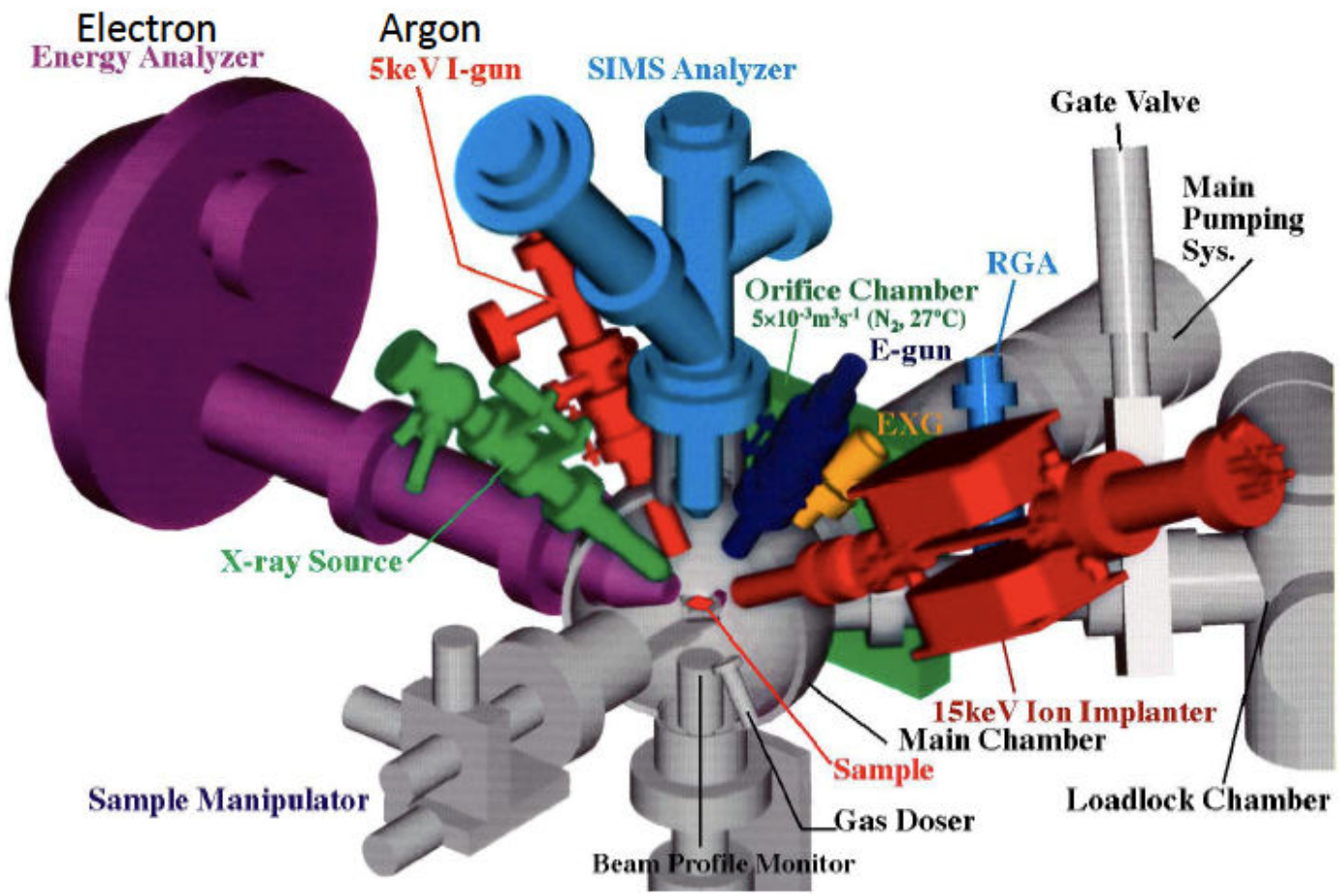
1. using Lab-EP samples

2. using sample tips in the single cell with EP process

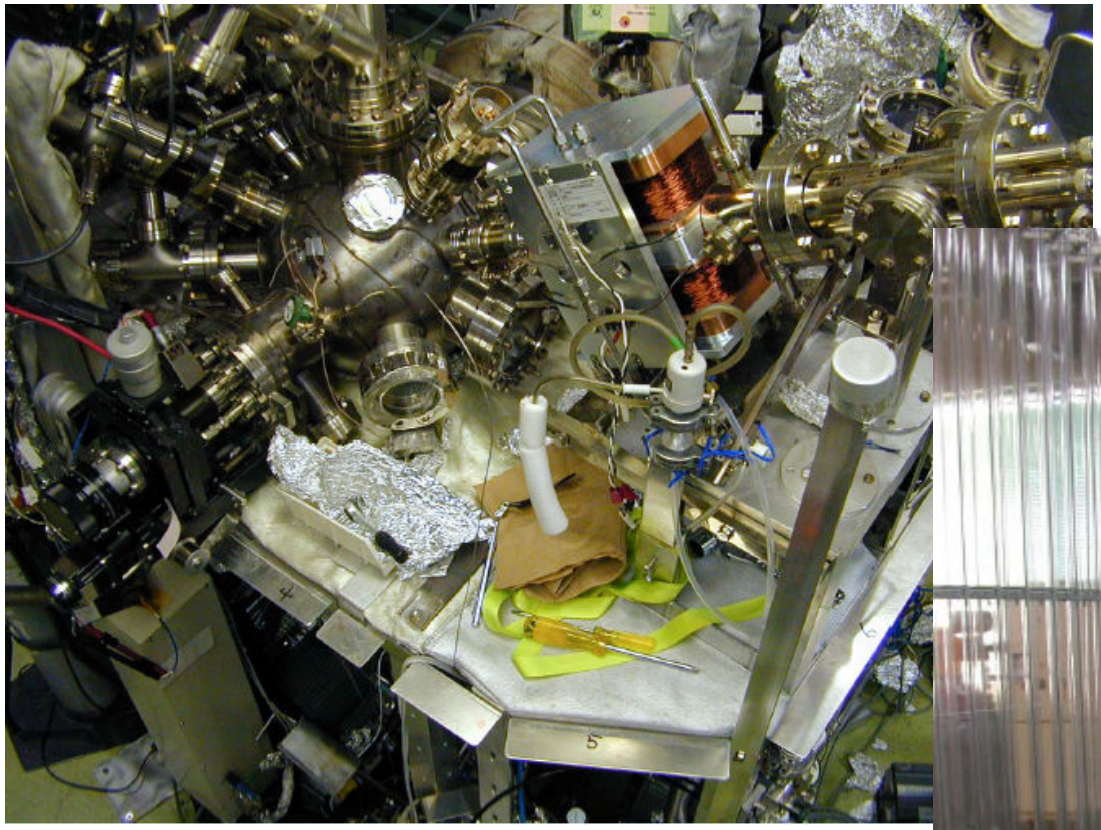
To understand surface contamination after EP process

KEK Analysis Tools

Surface Analyses (XPS) elemental analysis (>He), chemical analysis
Surface Analyses (Auger/AES) elemental analysis (>He)
Surface Analyses (SIMS) elemental analysis (all)
Surface Conditioning (Electron Irradiation, Sputtering, Heating)



KEK Analysis Tools



XPS, AES, SIMS, surface conditioners

SEM, EDX

Nb samples preparation

- Prepare four Nb samples, sample-1, -1’, -2, -2’.
- Sample-1, -1’: BCP(30um)+EP(70um) + U.P.W. rinse.
- Sample-2, -2’: BCP(30um)+EP(70um) + U.P.W. rinse + sponge wipe cleaning.
- Compare sample-1 and sample-2 by FS-SEM, AES at Jlab.
- Compare sample-1’ and sample-2’ by XPS, AES, SIMS, SEM,EDX at KEK.

* Sponge wipe is a pioneer study of new surface treatment.

This is a proposed physical/mechanical cleaning method for EP treated Nb surface.

Lab-EP at Nomura-plating co.

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

Lab-EP of Nb-plate sample

Nb sample + Teflon fixture
on Nb base-plate (10cm x 10cm)

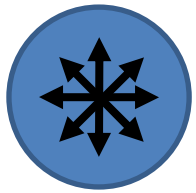
U.P.W. rinse and Sponge wipe

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

Ultra Pure Water rinse (20 min)

sponge wipe with U.P.W.

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



Nb-sample-2

+ Ultra Pure Water rinse (5 min)

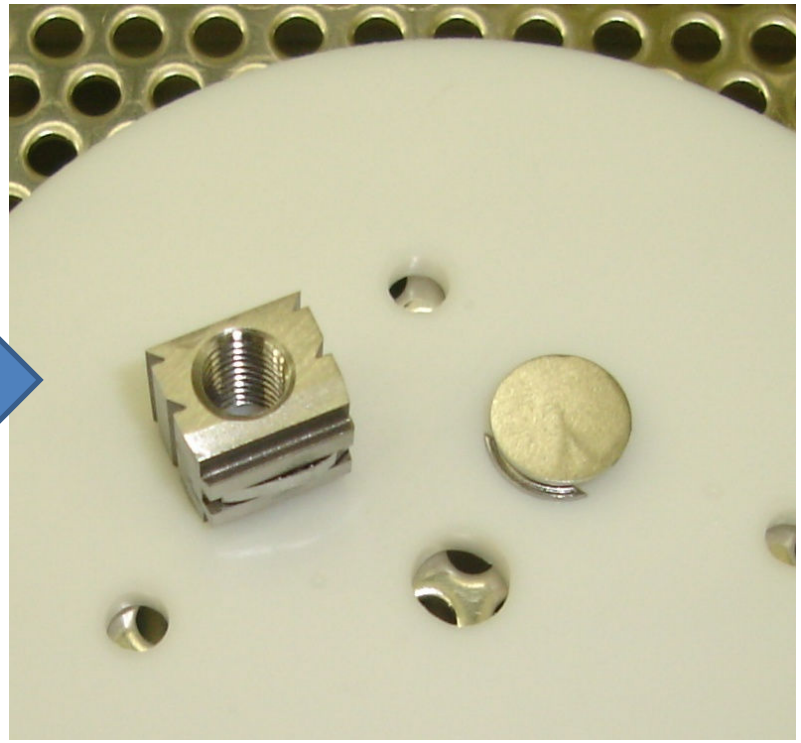
Packing under U.P.W.

Nb-sample-1

Packing in transportation container

KEK-type Nb-sample 1

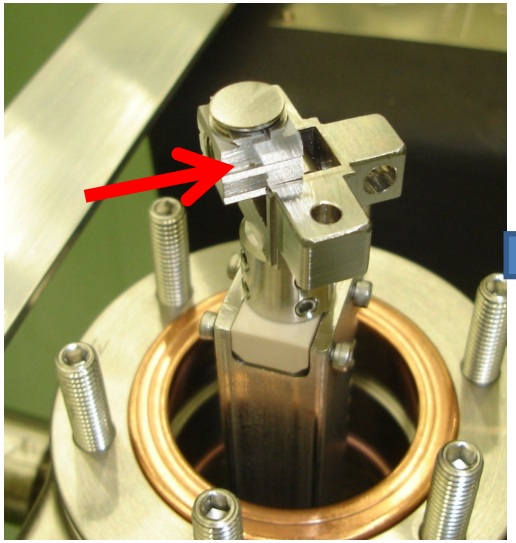
10 Sept. 2008

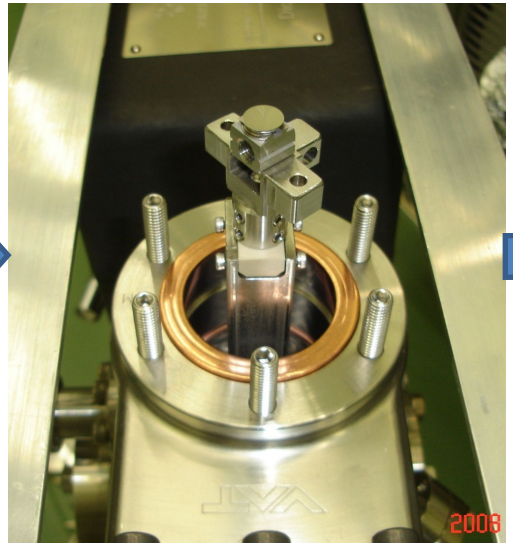


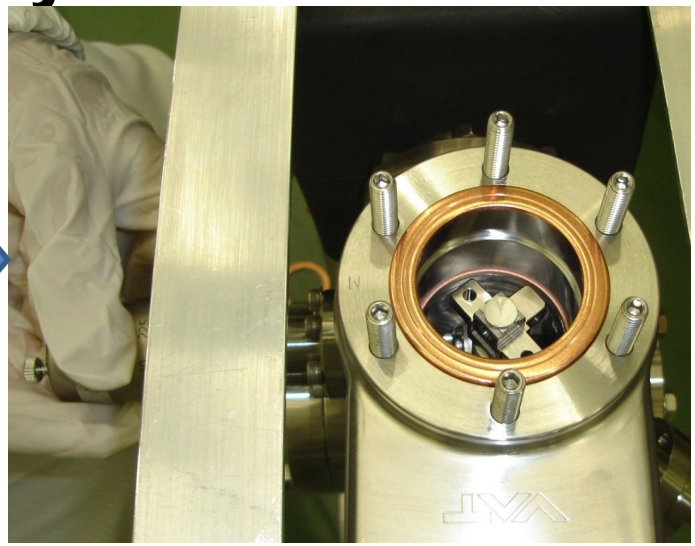
Nb-sample-1 kept in UPW container for 22 days.

Move out from the UPW container
Dry Nb-sample 1 in Class-10 C.R.

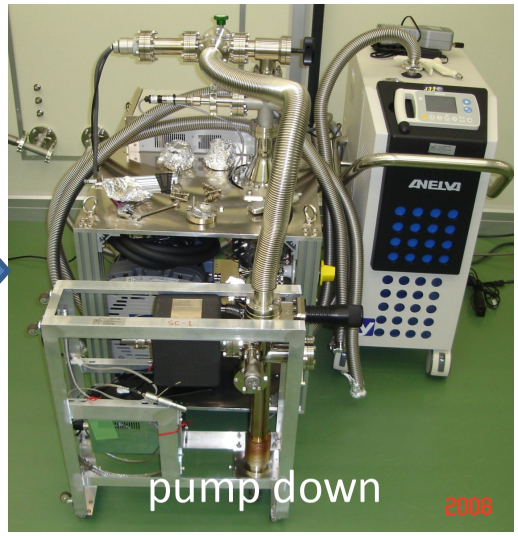
UPW container to vacuum container, then stored in the surface analysis device







store samples in clean-room

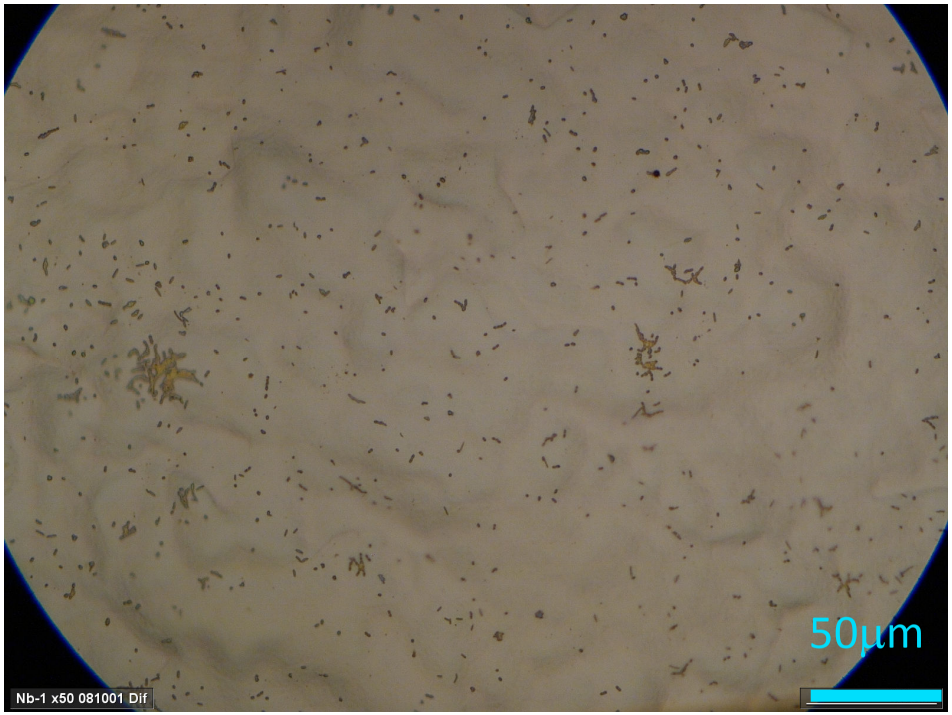


Surface analysis of EP'd Nb (Lab-EP sample)

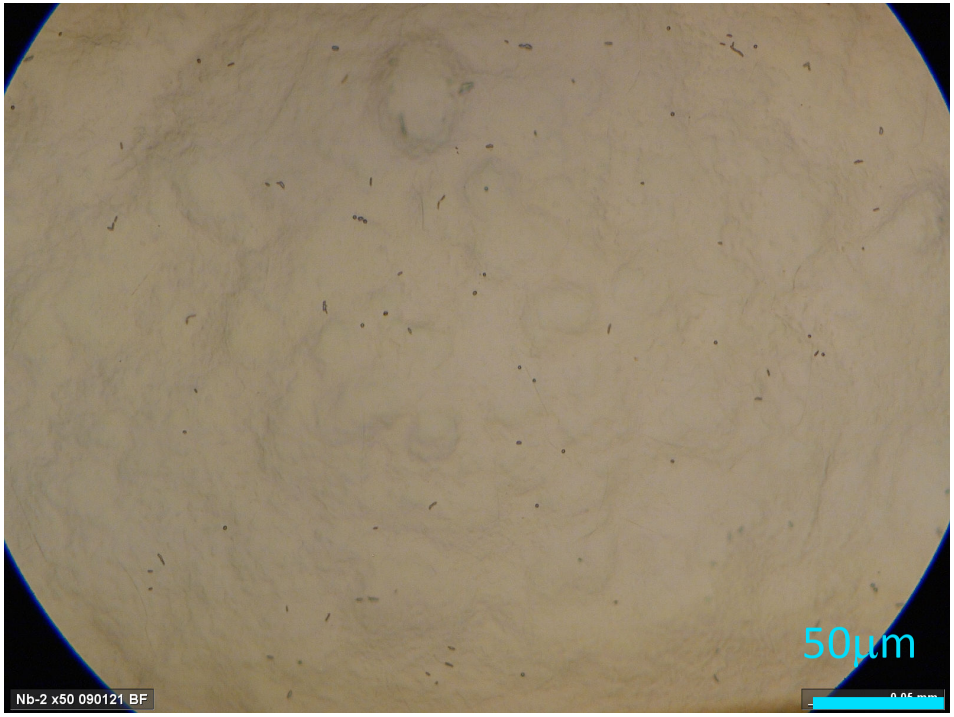
Lab-EP samples with UPW storage

(22 days)

Optical Microscope



Without Wipe

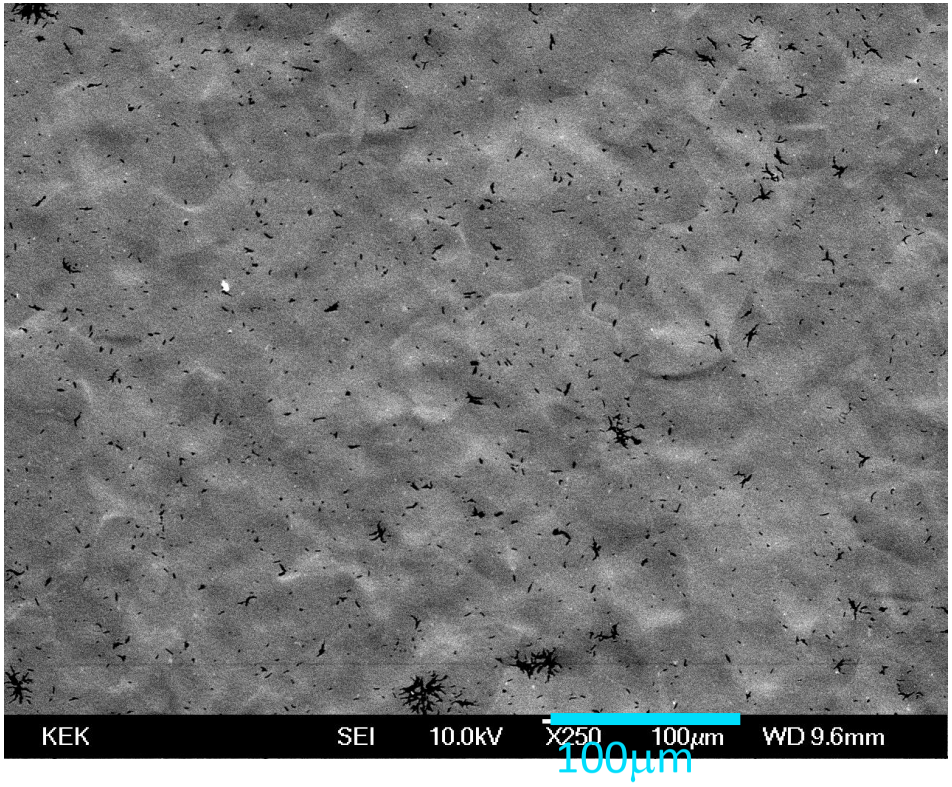


With Sponge Wipe

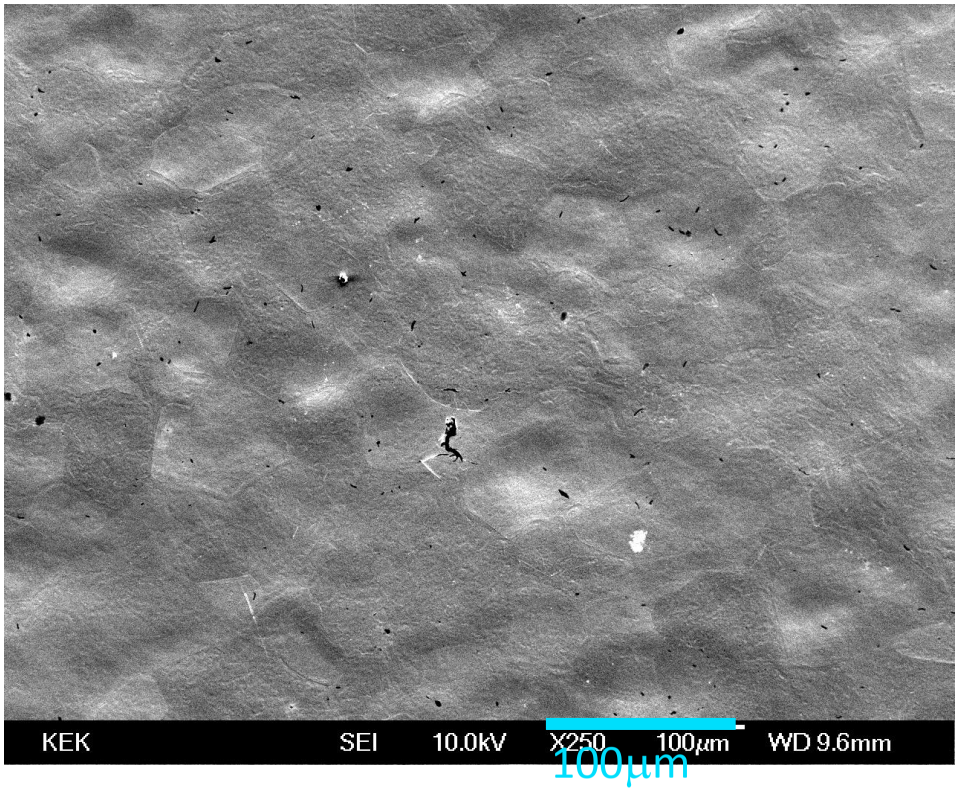
Bacteria? like objects were found, but it is few in sponge wipe sample.
The reason is not yet understood.

Lab-EP samples with UPW storage

SEM



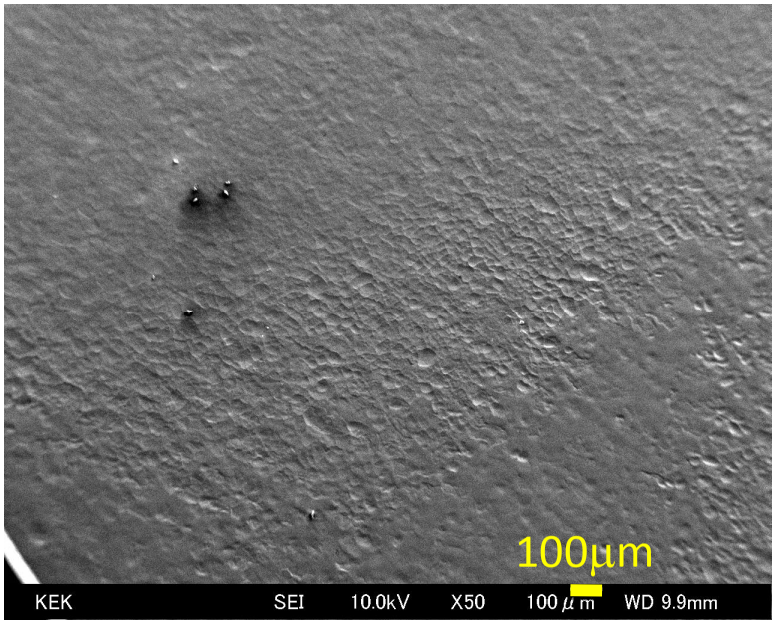
Without Sponge Wipe

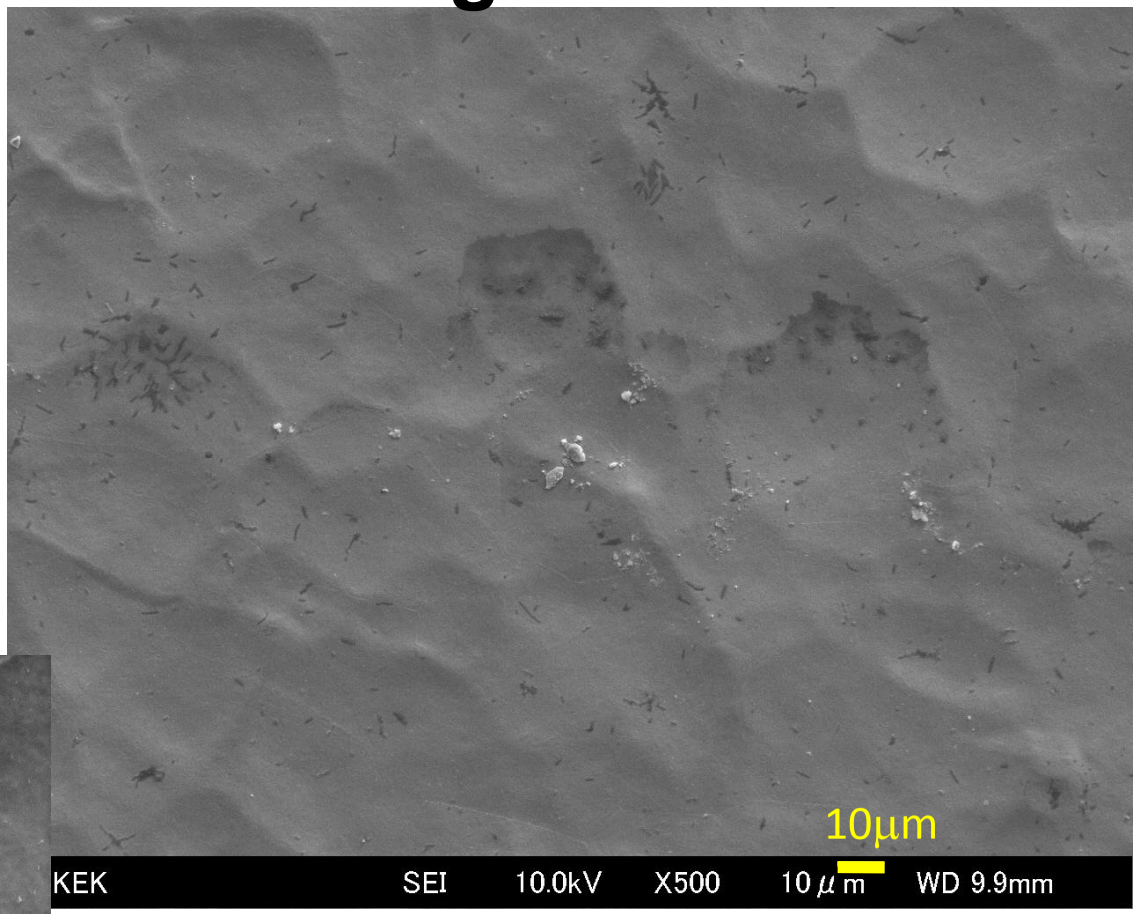


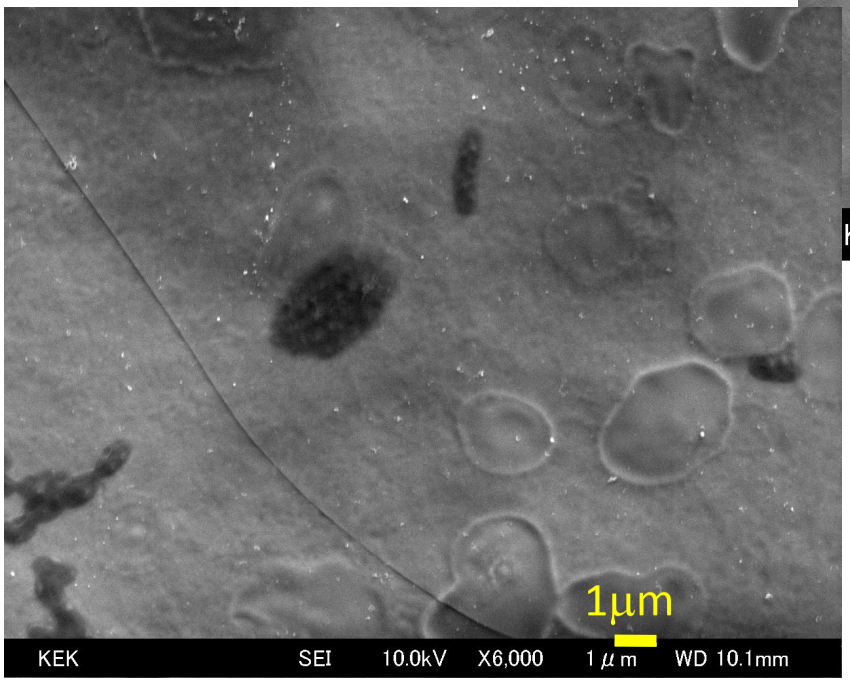
With Sponge Wipe

Bacteria? like black objects were found, but it is few in sponge wipe sample.
The reason is not yet understood.
White objects (Nb oxide?) are seen in both.

More SEM images

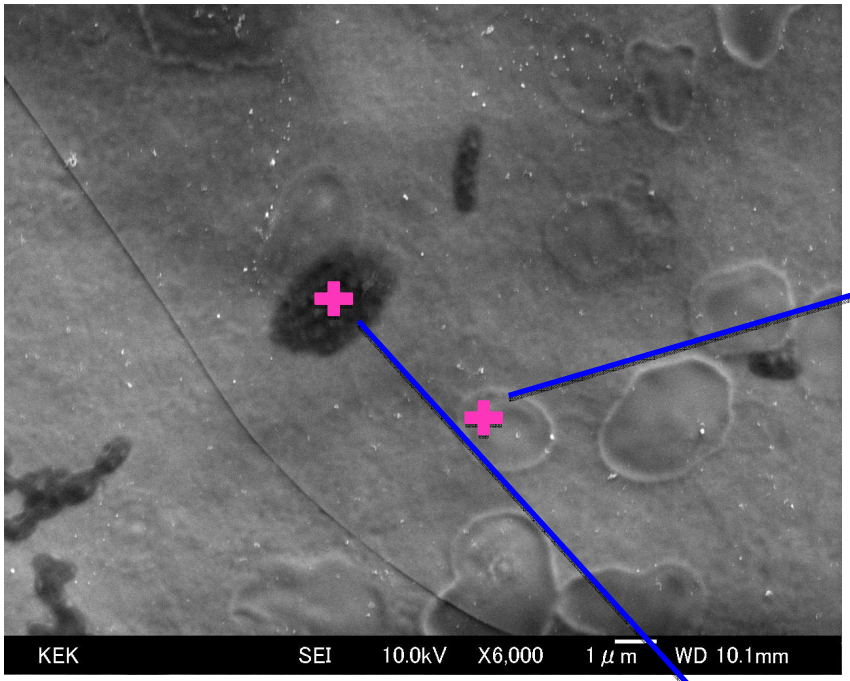


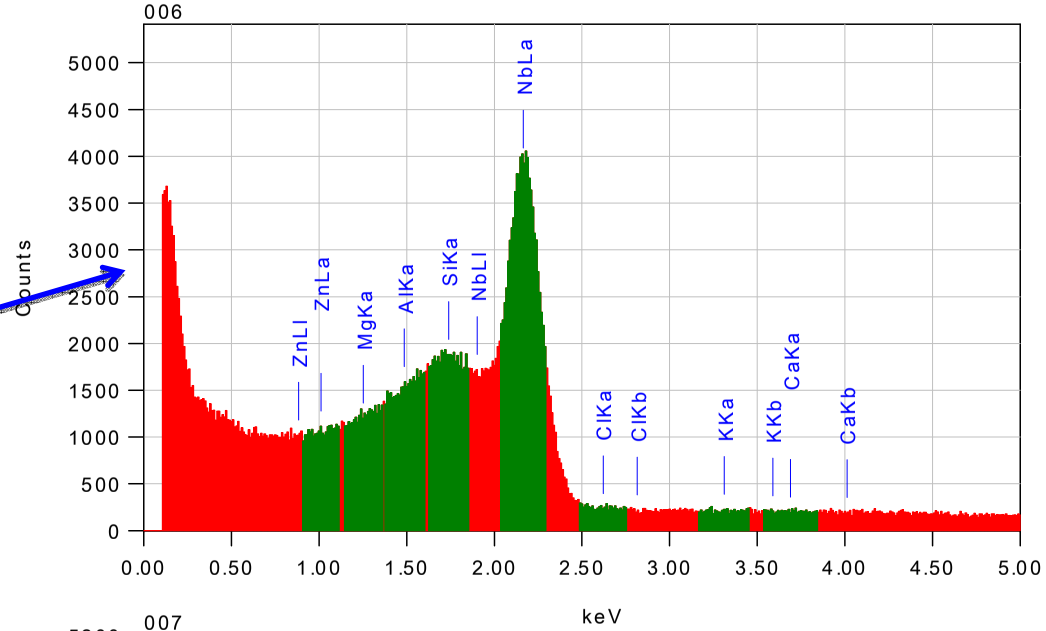


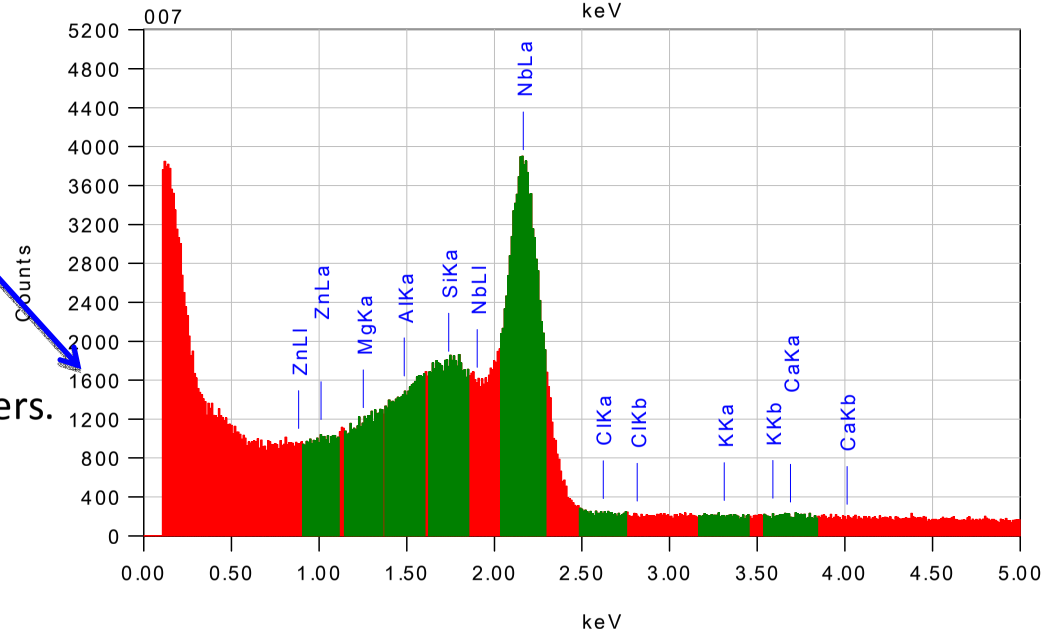


Black objects and white objects are seen.

EDX Analysis for black area

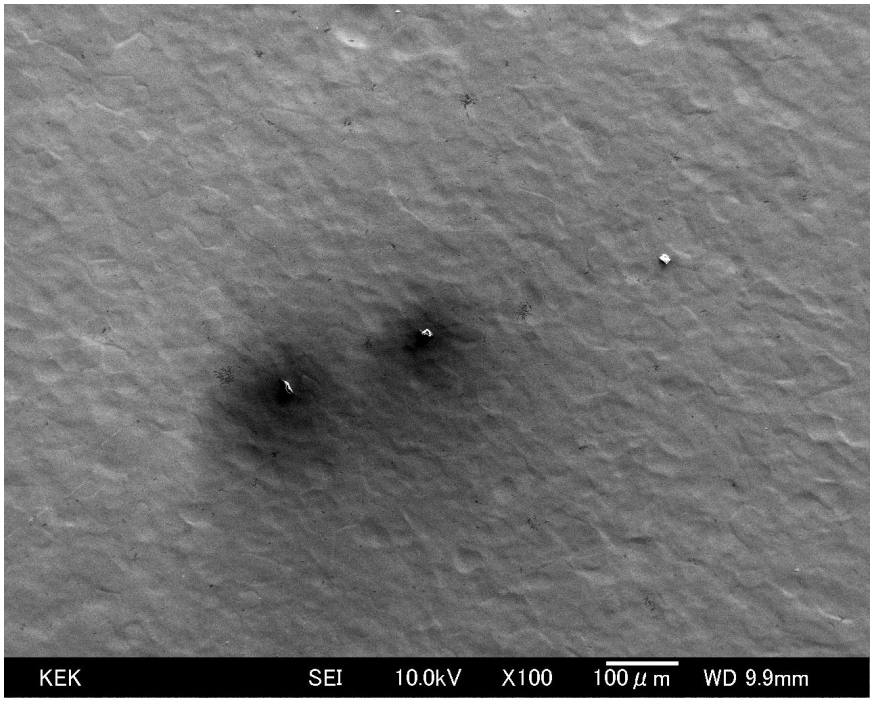


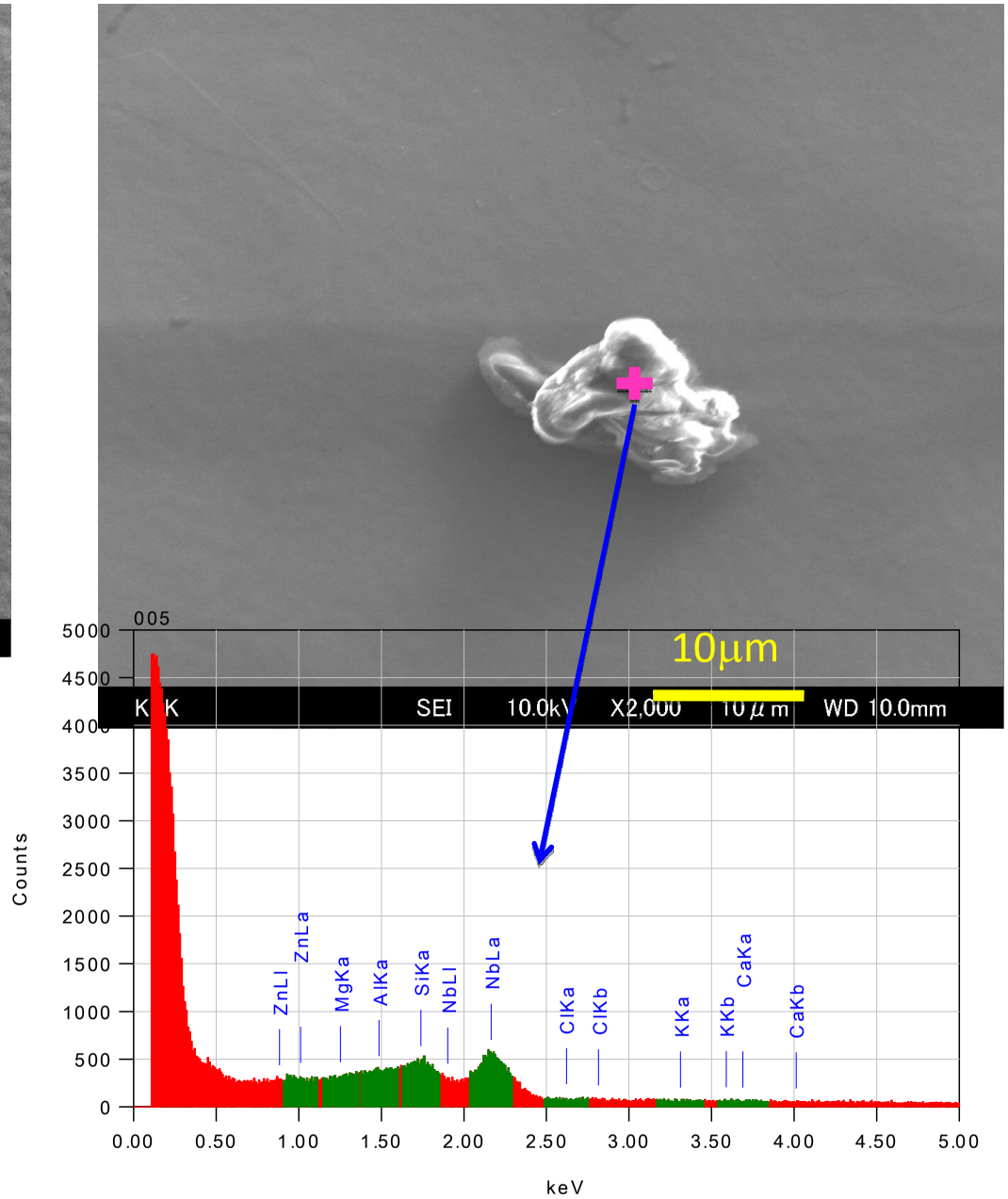




Black object is the same component as the others.
Si signal is seen other than Nb.
No S signal.

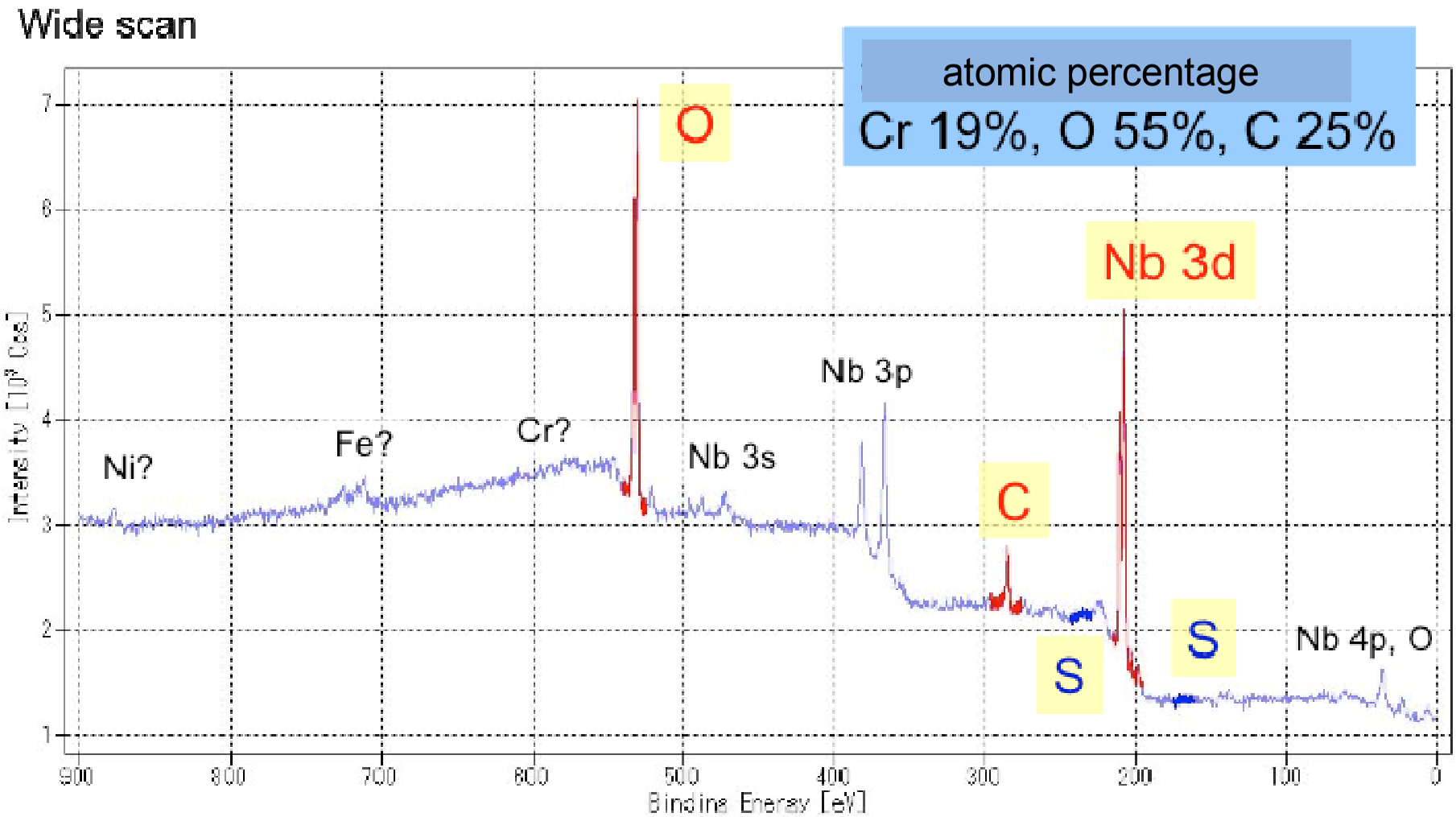
More EDX Analysis for contaminates





Si?, came from air exposure ?
at the sample transfer from XPS to SEM.

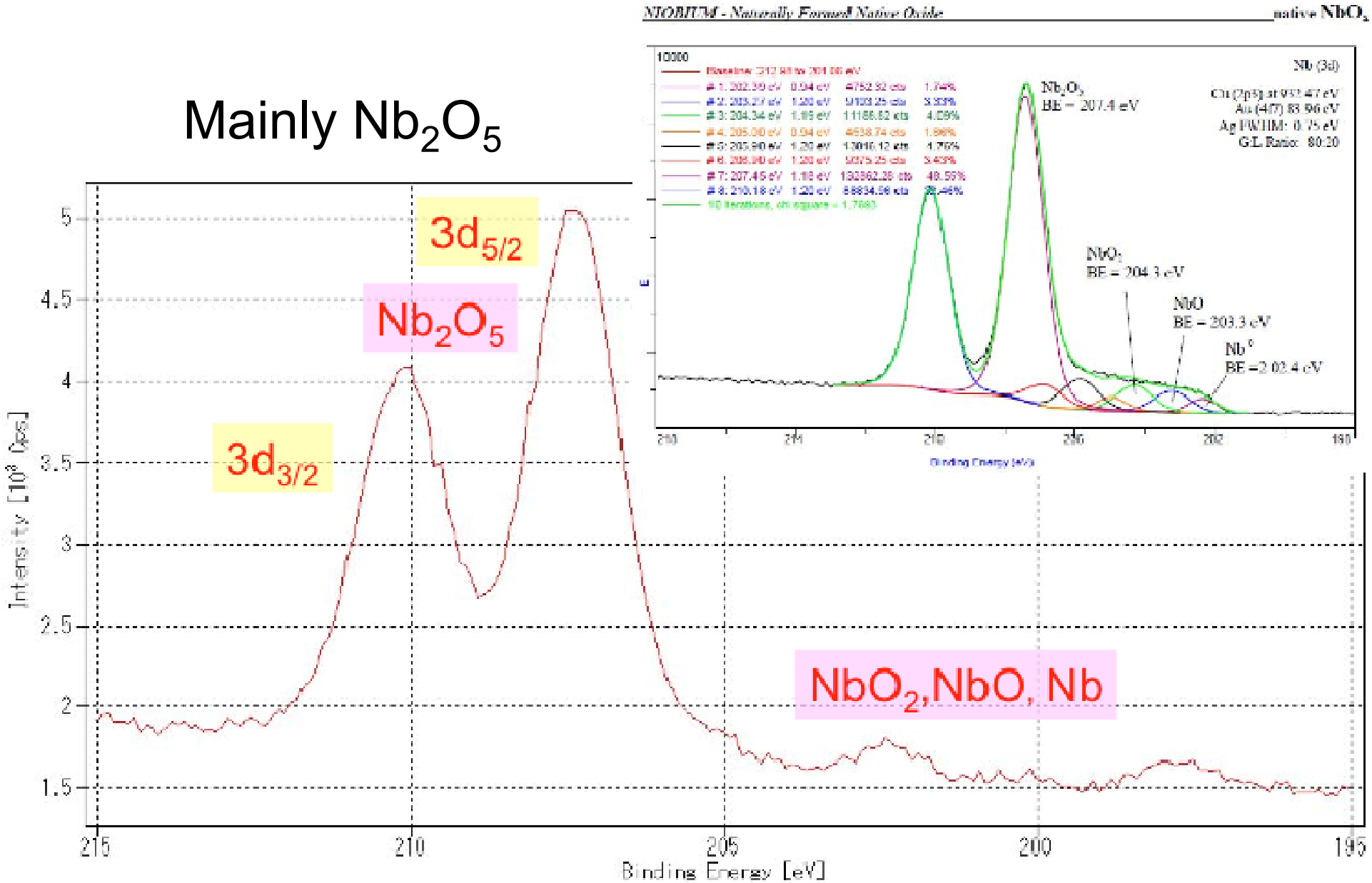
Nb-1 XPS spectrum



No observation of S,
We see SUS component contamination, maybe from the sample holder.

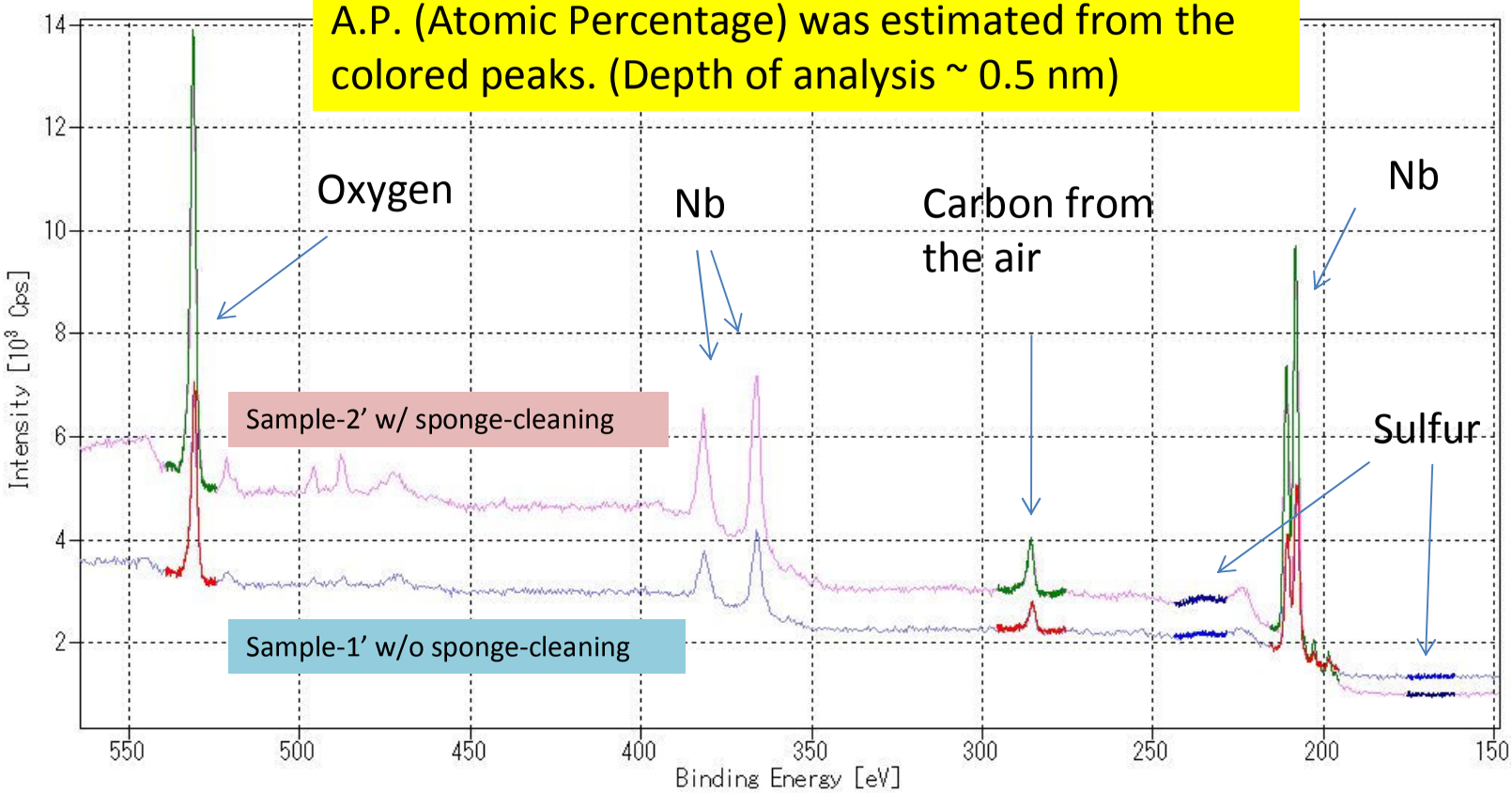
Nb-1 XPS spectrum of Nb

Mainly Nb_2O_5



XPS Analysis of KEK-type Nb-sample-1' & 2' (BCP+EP w/ & w/o sponge wipe) at KEK

A.P. (Atomic Percentage) was estimated from the colored peaks. (Depth of analysis ~ 0.5 nm)



A.P. for sample-2' w/ sponge-wipe
= Nb:20%, O:57%, C:23%

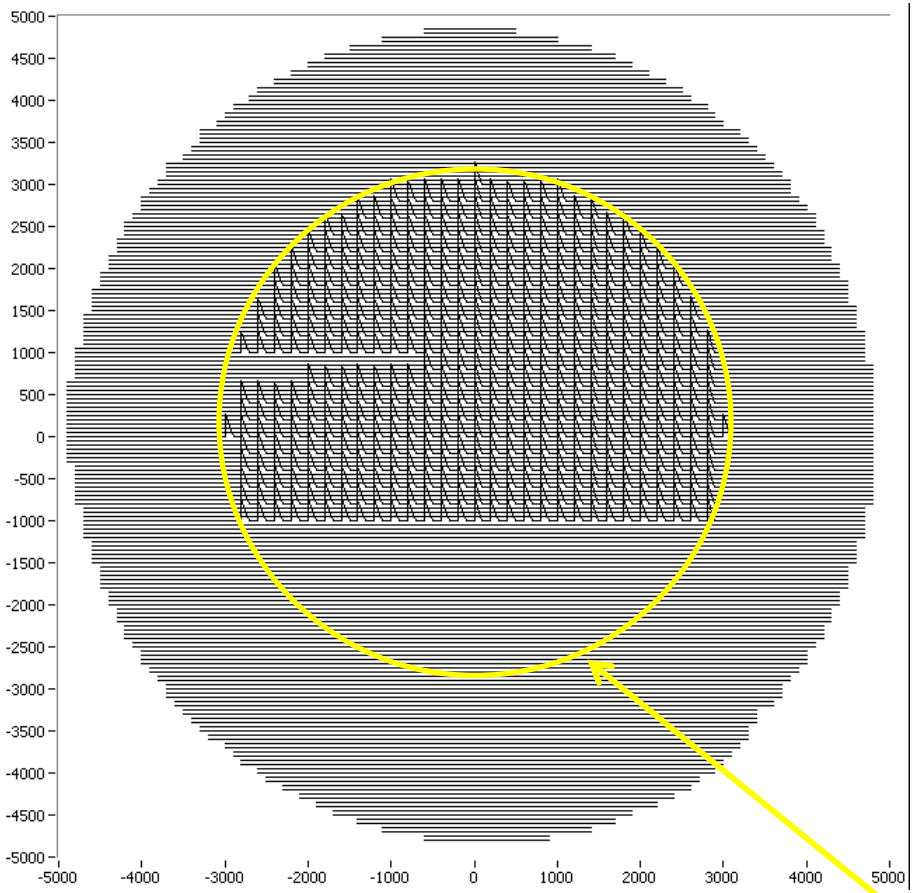
A.P. for sample-1' w/o sponge-wipe
= Nb:19%, O:55%, C:25%

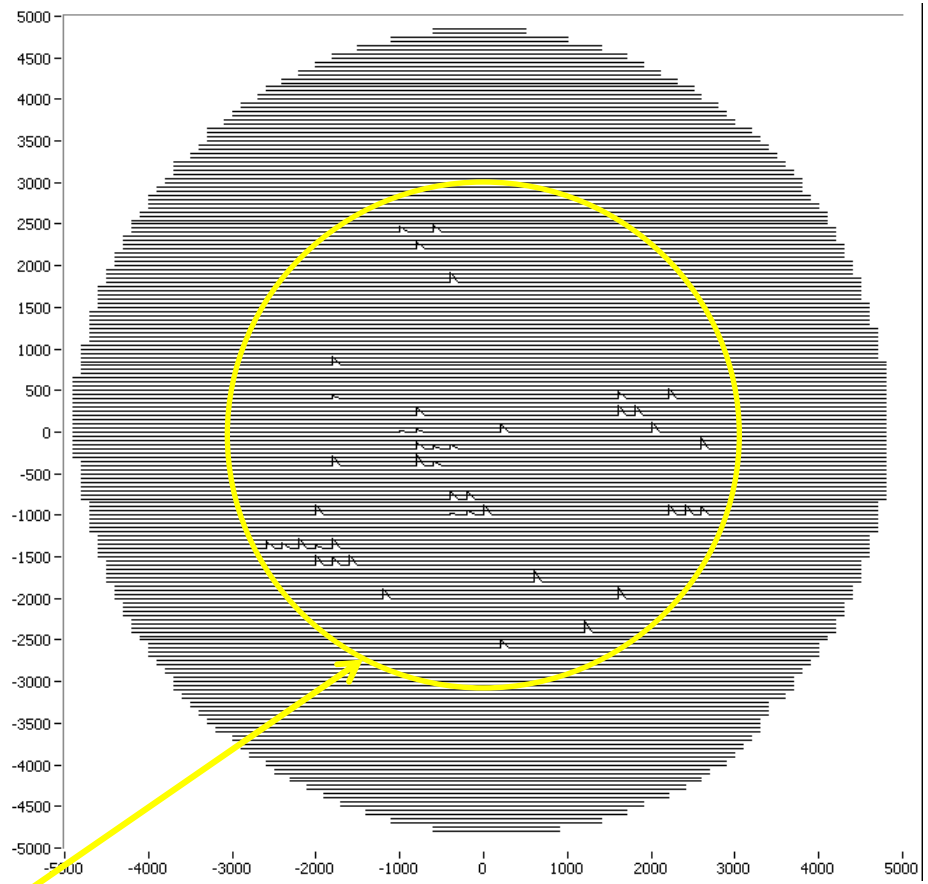
Sulfur ~ 0% for both samples w/ and w/o sponge-wipe.
No significant difference of A.P. for both samples.

Field Emission Scan by JLab (Jlab sample)

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

Sample#2: much less field emitters
Higher FE onset





EP(70 um)

Area inside circle is scanned

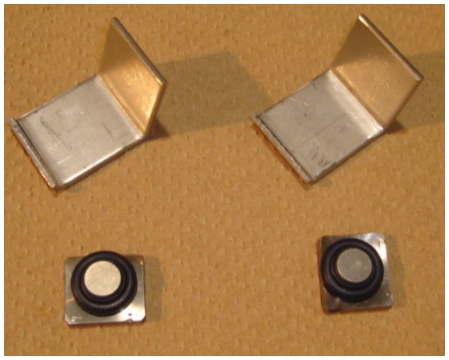
EP(70 um) + sponge wipe

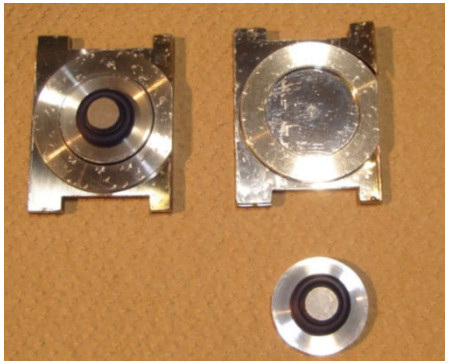
Significant difference in Field emission scan.

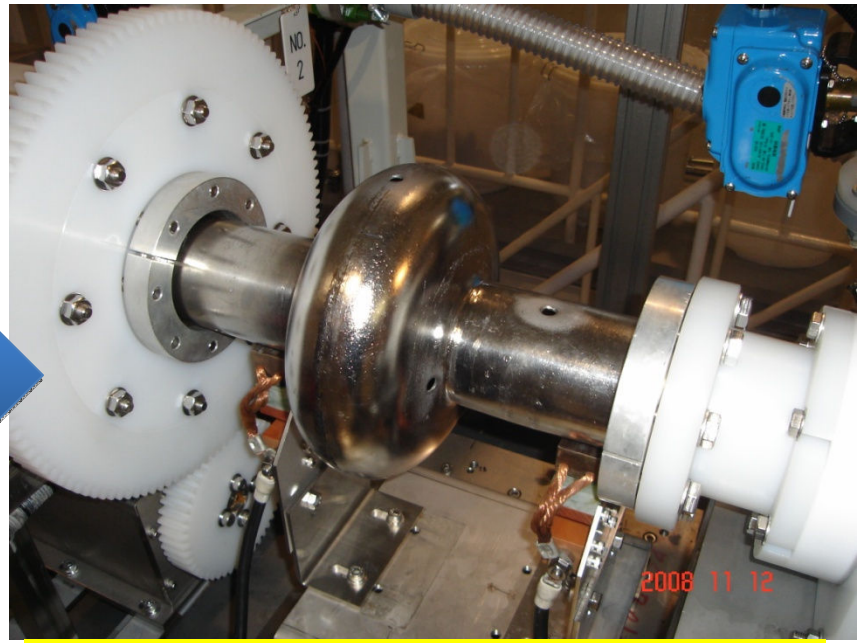
EP’d Nb sample in single-cell cavity

Special single-cell cavity for sample-study

6 Nb-samples
BCP(10um)
Weight and thickness measurements







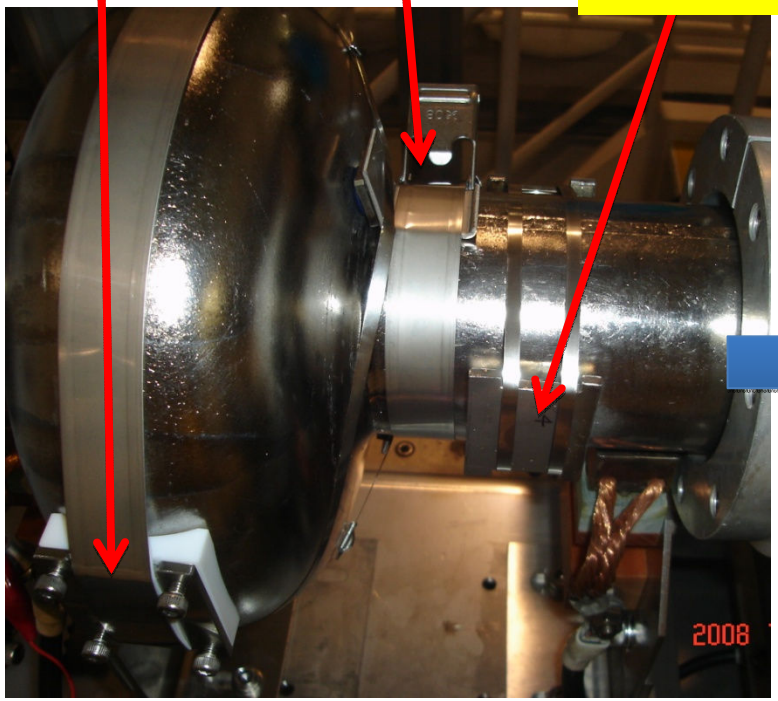
Special cavity with 6 sample-holes on the EP bed.

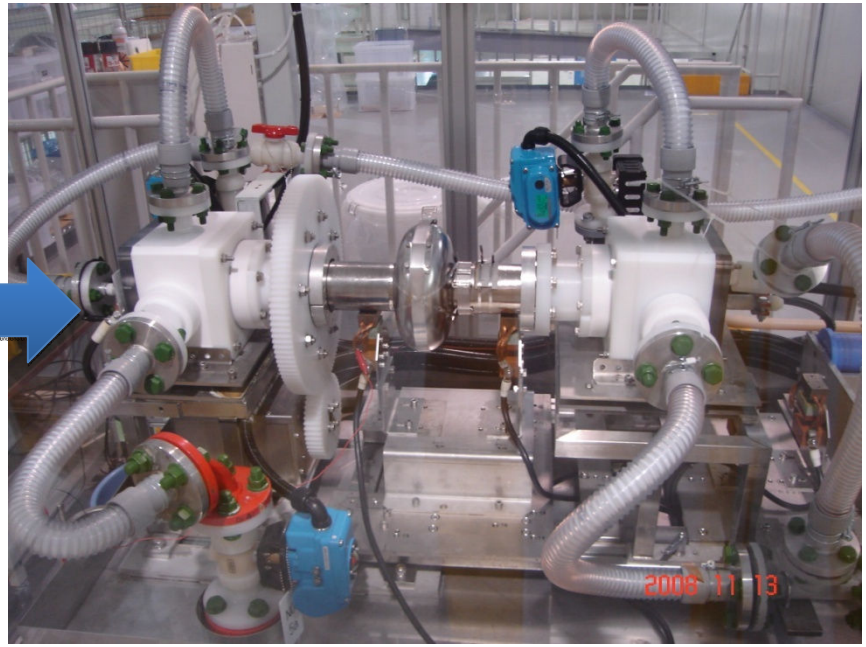
Special single-cell cavity for sample-study

2 samples at equator

2 samples at iris

2 samples on a beam-pipe



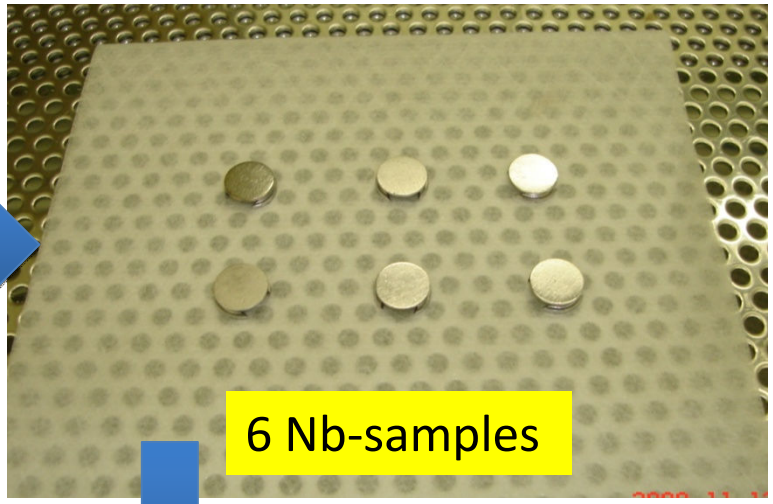


6 samples were dressed on the special cavity

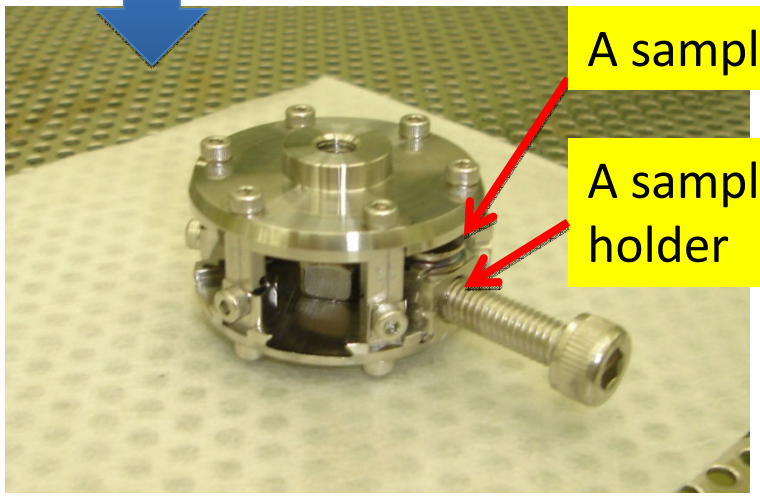
EP 20 um + UPW rinse (No ultrasonic-rinse)

Special single-cell cavity for sample-study

Disassembly in class-10 Clean-Room



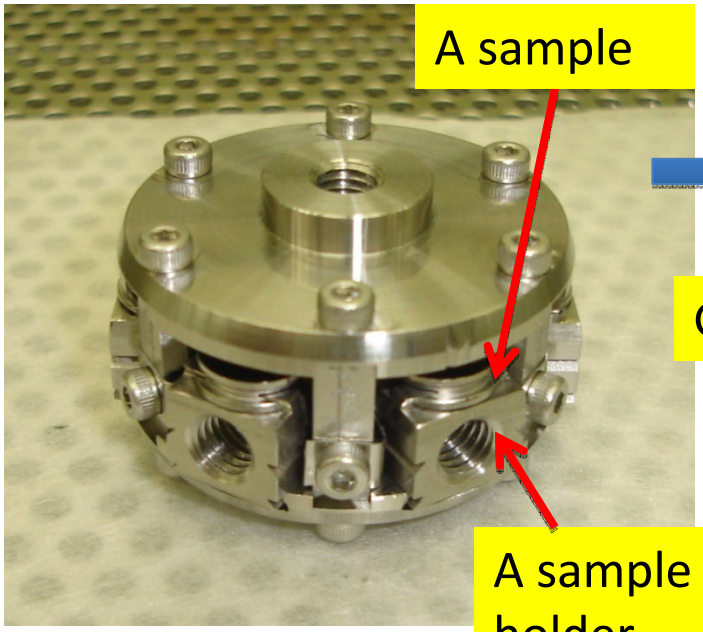
6 Nb-samples

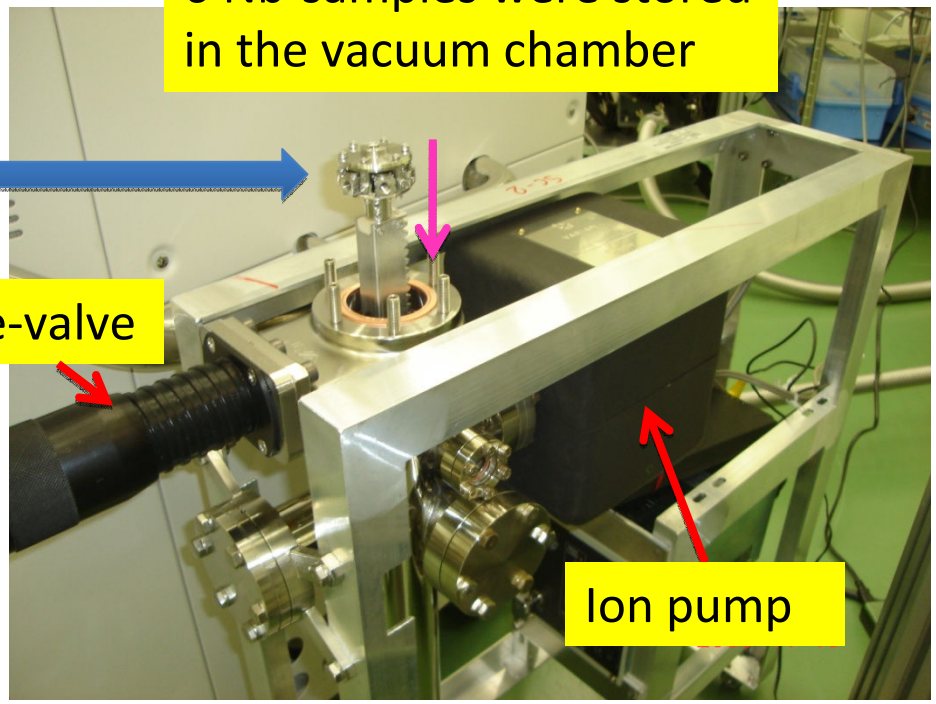


A sample

A sample holder

Special single-cell cavity for sample-study



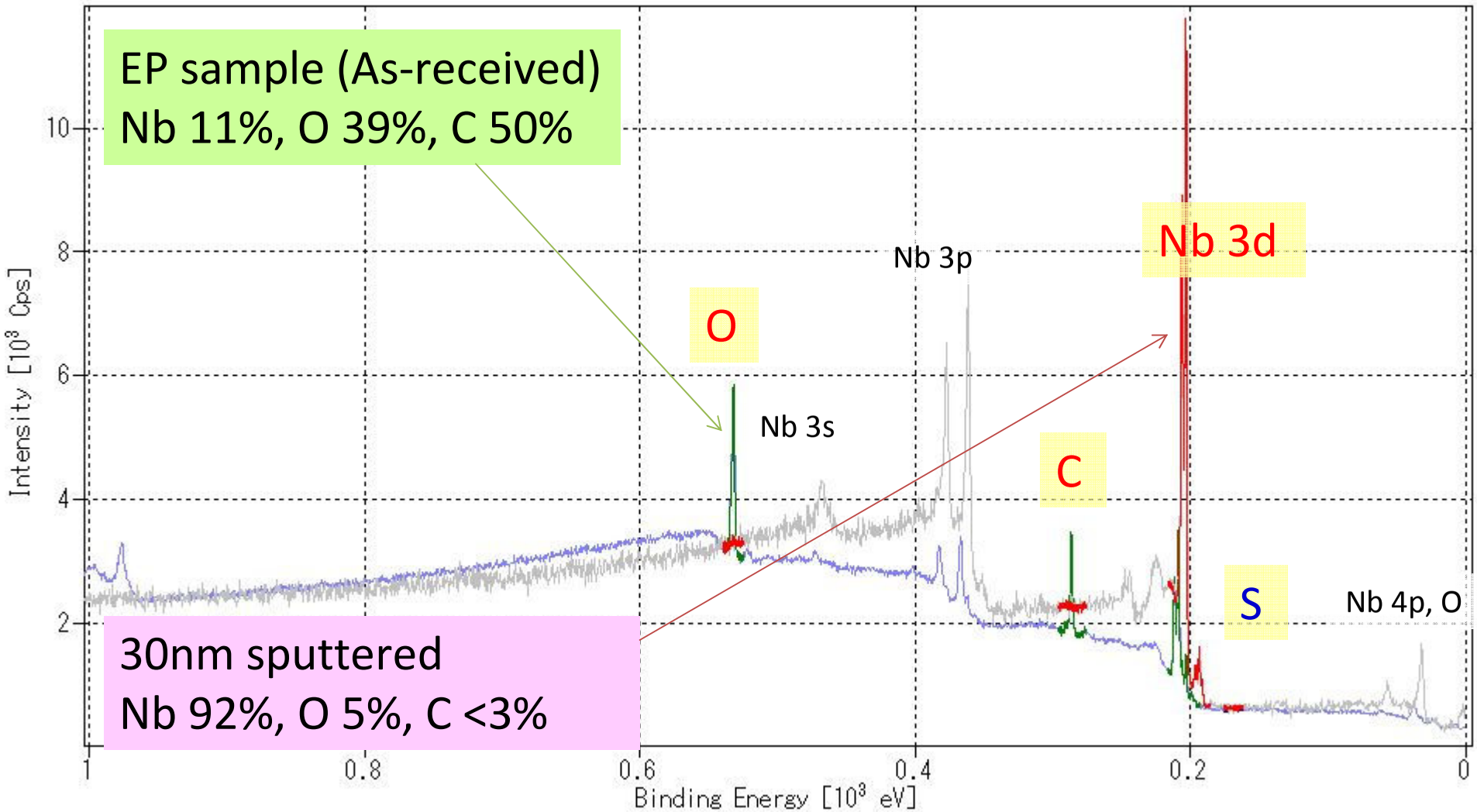


6 Nb-samples + sample-holders

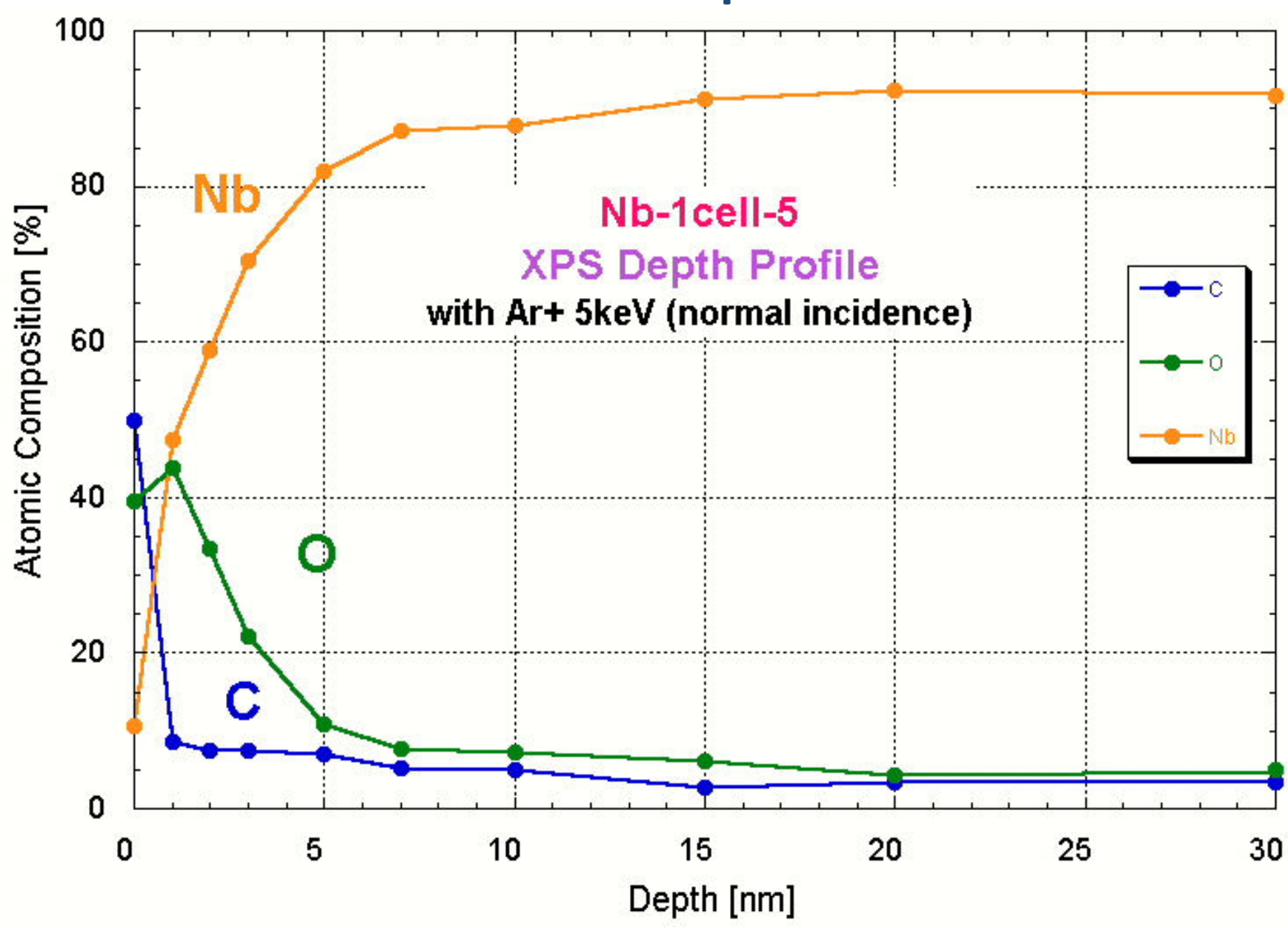
Surface analysis of EP'd Nb (Single-cell cavity EP sample)

Nb-1cell-5 XPS spectrum

Wide scan



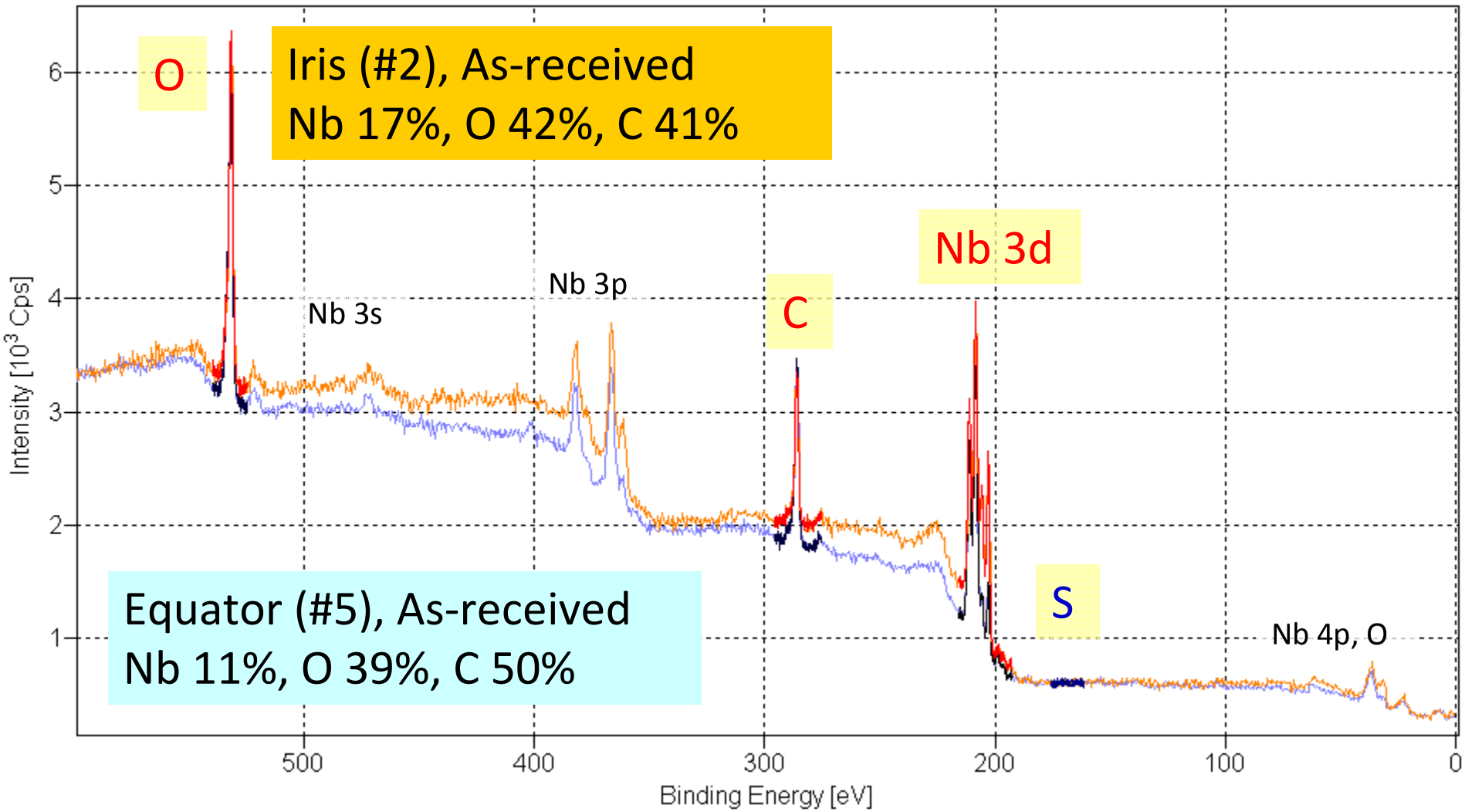
Nb-1cell-5 Depth Profile

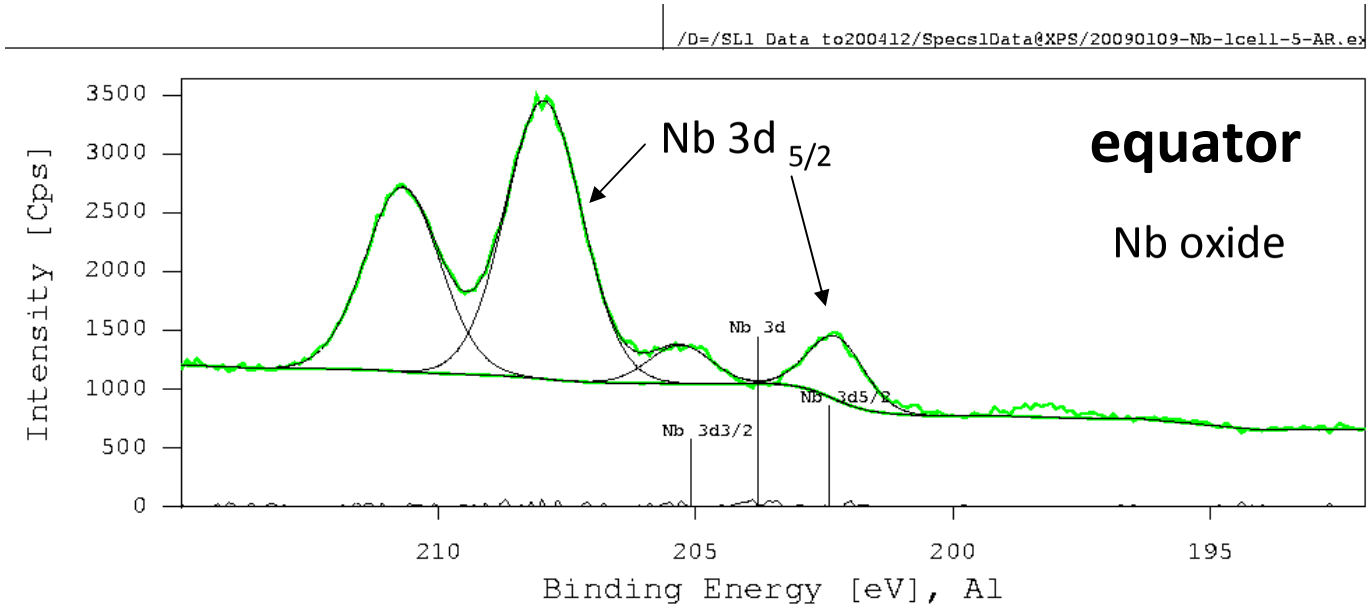


Nb-1cell equator & iris sample

XPS spectrum

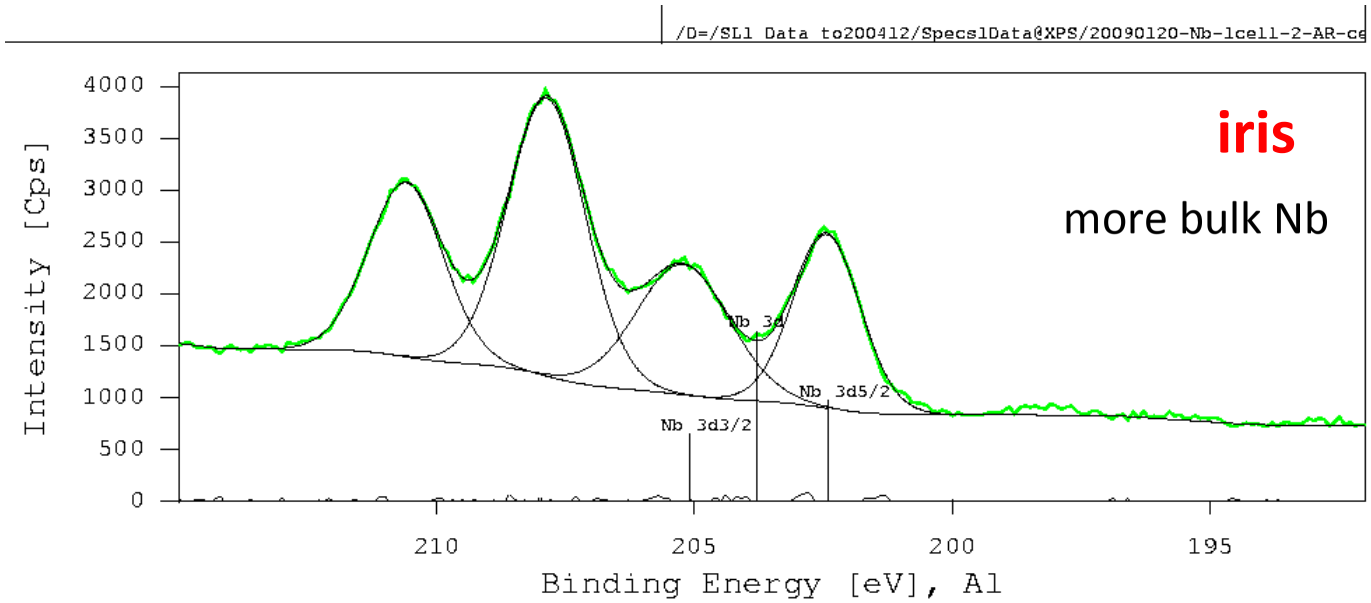
Wide scan





Ratio of Nb-oxide and bulk Nb

~ 6 : 1

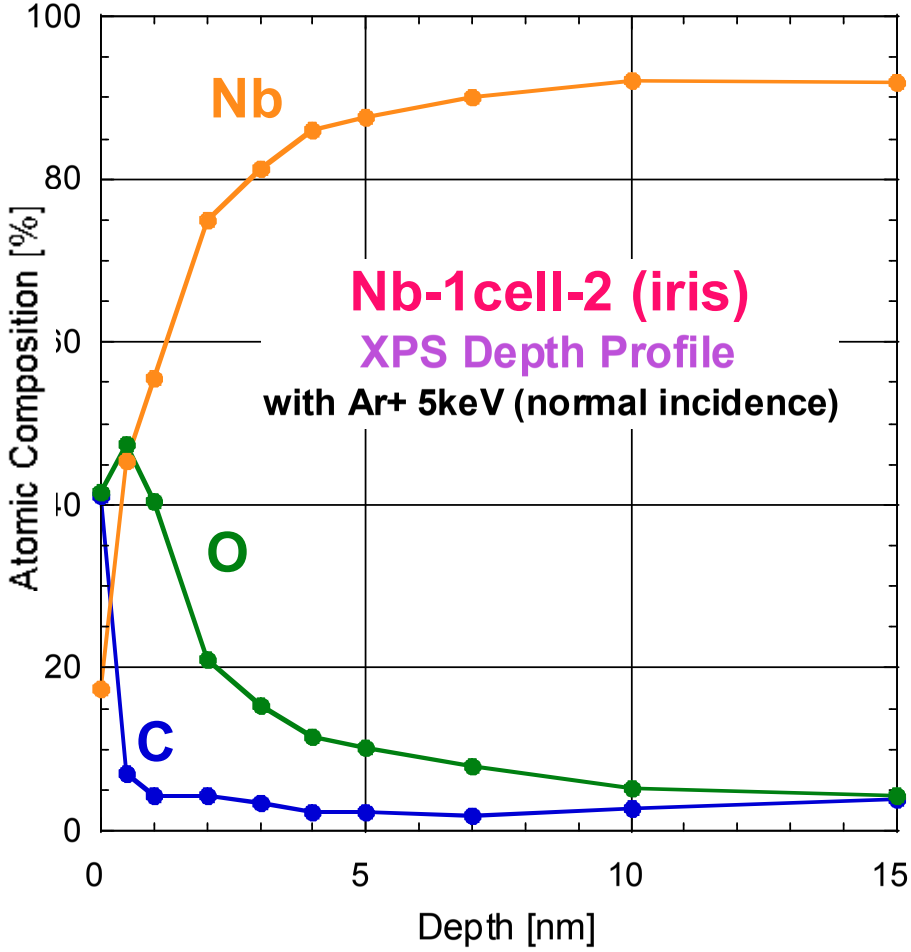


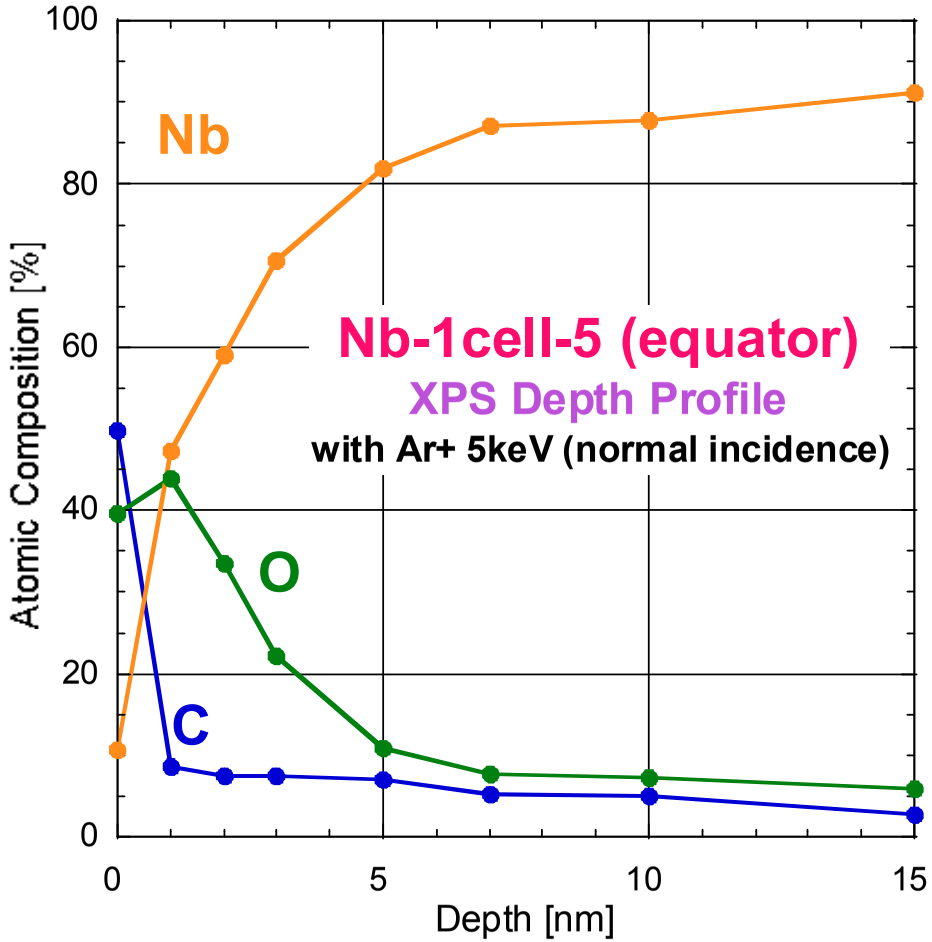
~ 1.8 : 1

There will be different EP effect between equator and iris.

XPS Depth Profile of EP'd sample on the single cell

BCP(10μm) + EP(20μm) + UPW rinse (No ultrasonic-rinse)



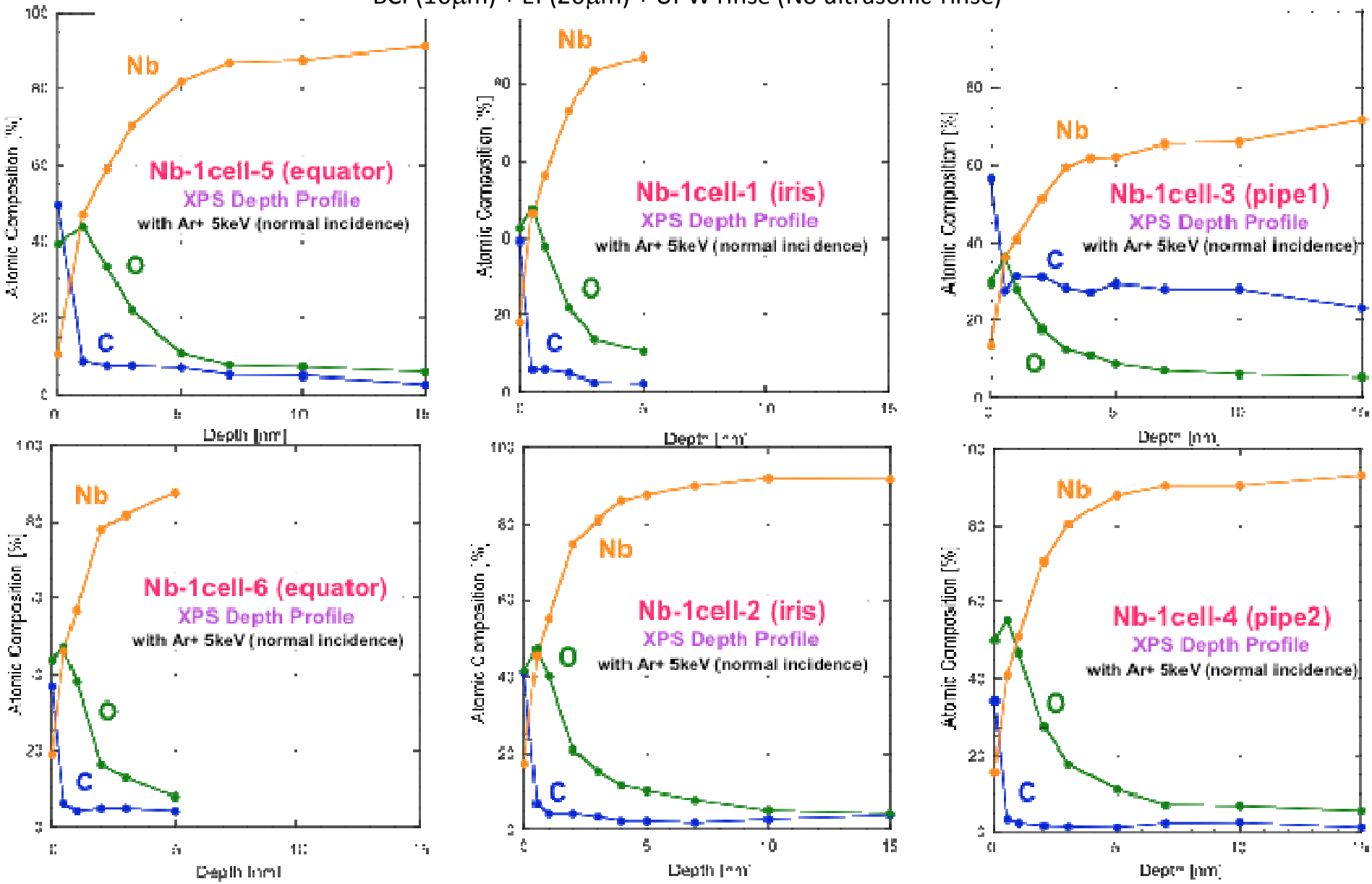


thickness of oxide layer iris : equator = 2 : 3

There will be a possibility of
the difference of the ratio Nb-oxide and bulk Nb, came from oxide layer thickness.

XPS Depth Profile of EP'd sample on single cell)

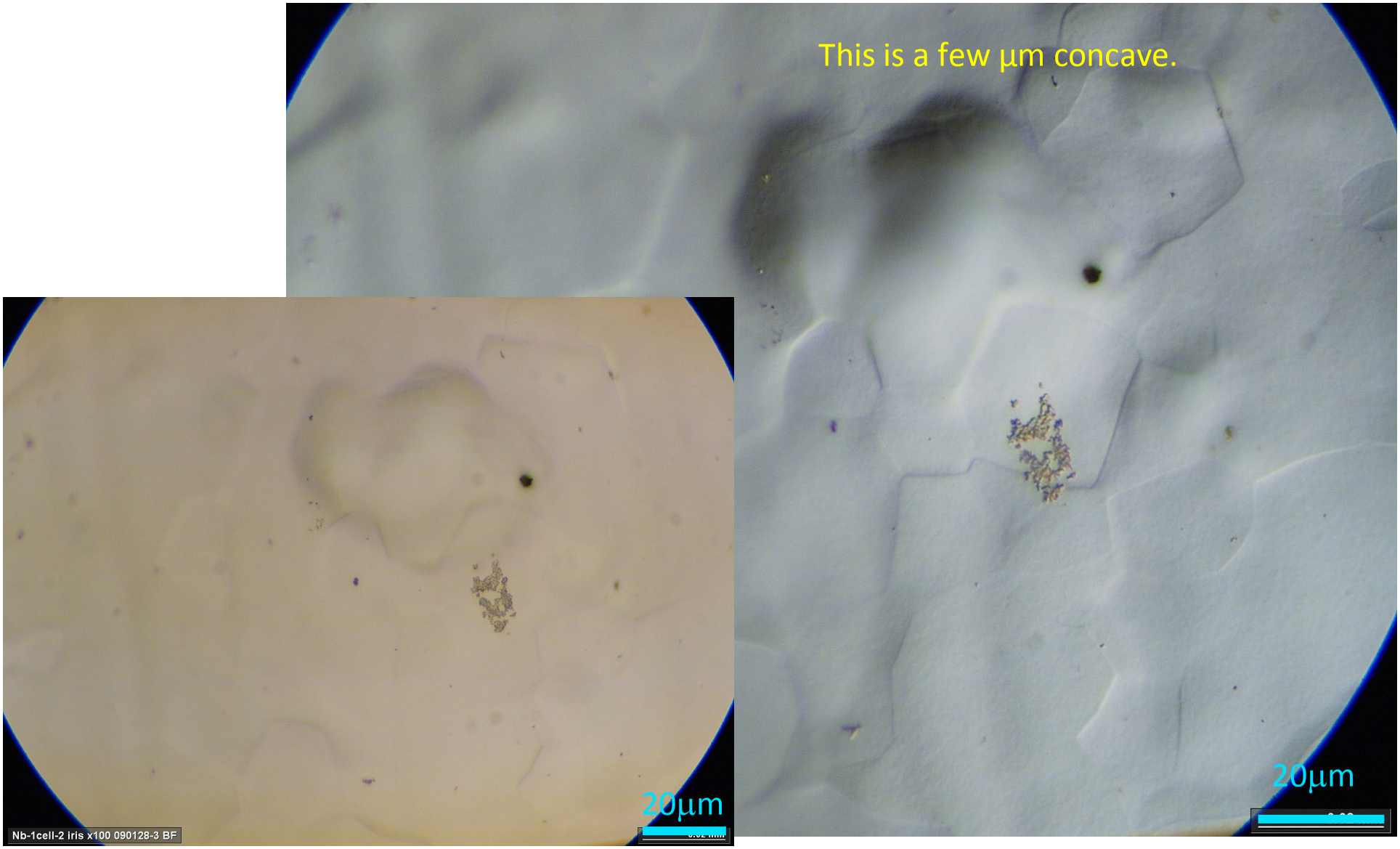
BCP(10μm) + EP(20μm) + UPW rinse (No ultrasonic-rinse)



#3 sample has a lot of carbon, the reason is not yet known.

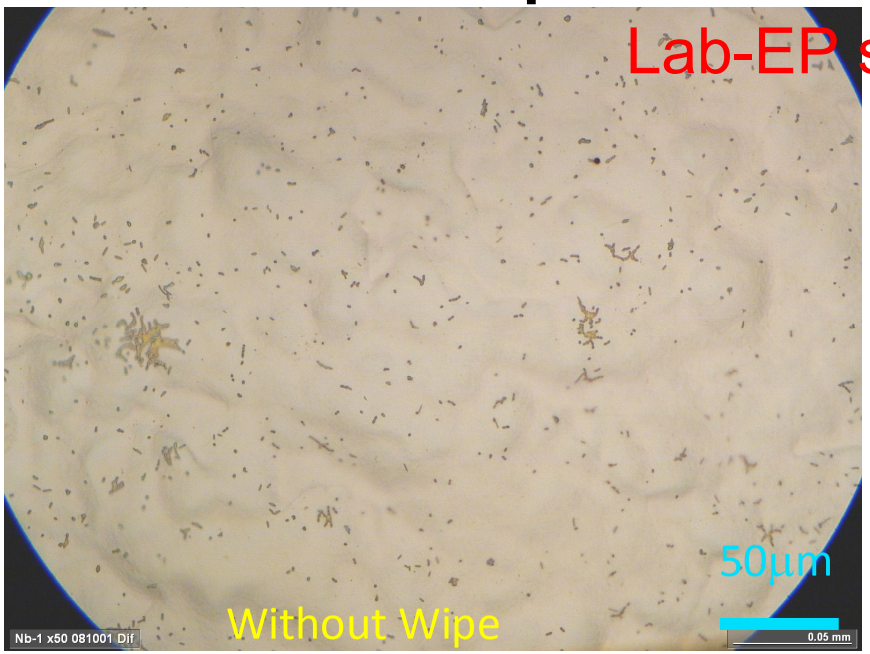
Oxide layer are 2-3nm in all the samples.

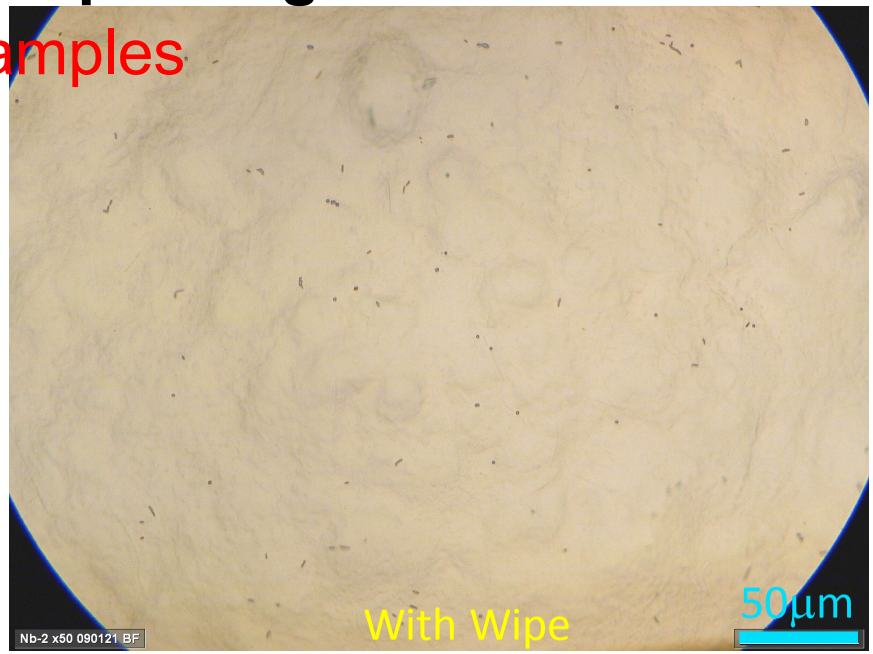
Optical Microscope Images sample#2(Iris)

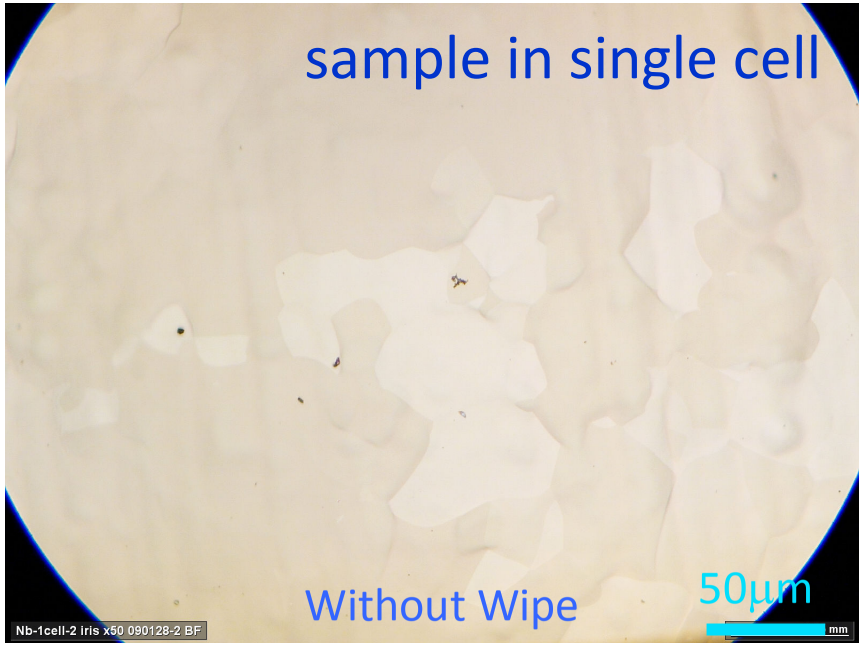


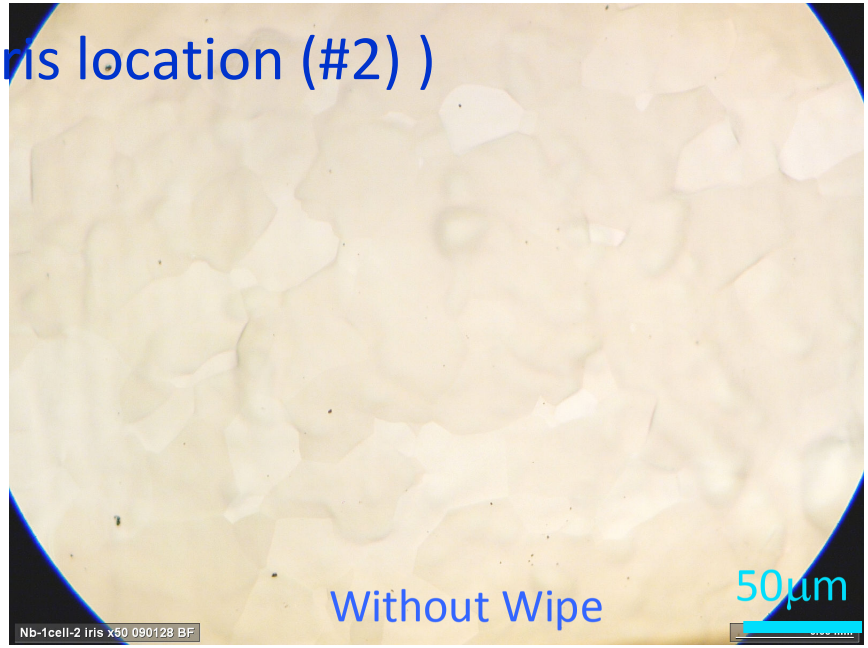
SEM and EDX analysis is underway, now.

Optical Microscope Images





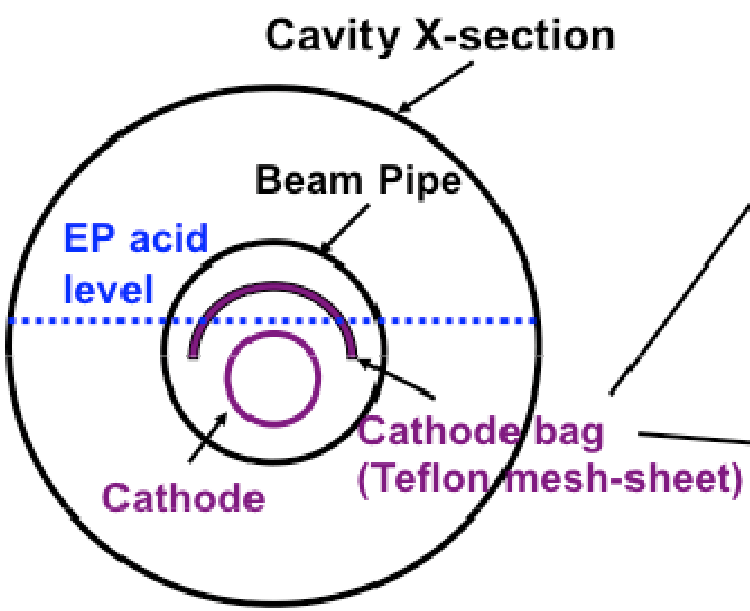


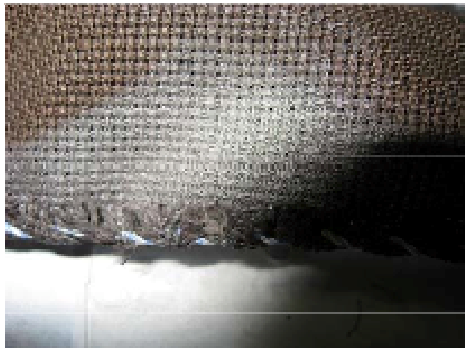


The difference is maybe by water storage (Lab-EP) and by vacuum storage (single cell sample)

Sulfur removal
(captured sulfur on EP cathode bag)

Powder on cathode bag at STF/KEK



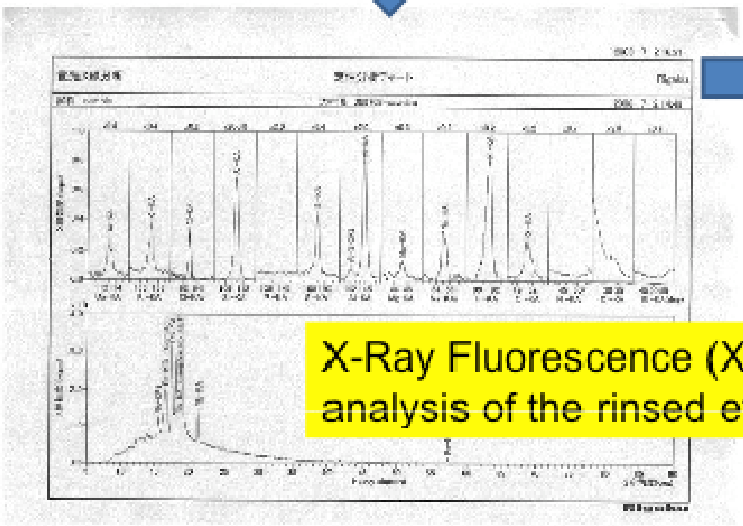


30 June – 1 July 2008

Location:
Iris Cell#4-cell#5, Iris cell#5-cell#6

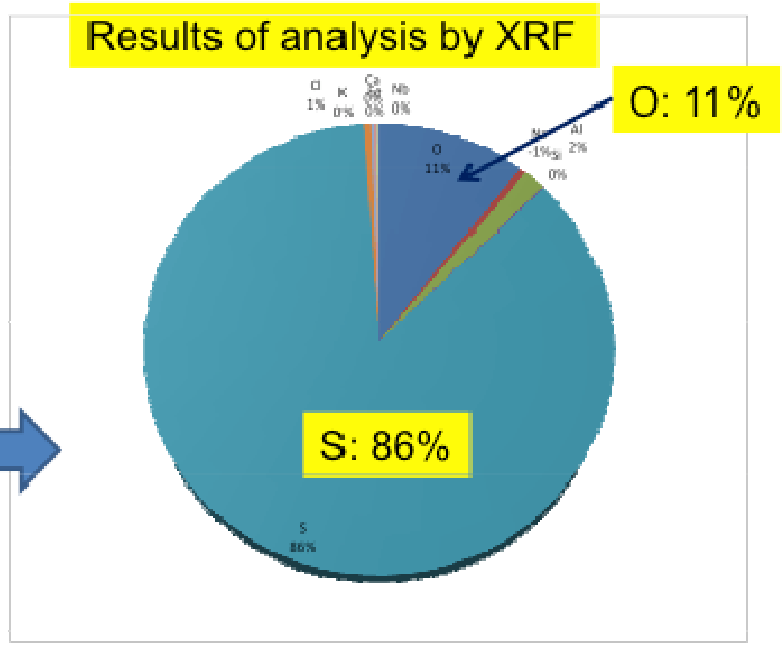
Analysis of powder on the cathode bag

Ethanol rinse of cathode bag

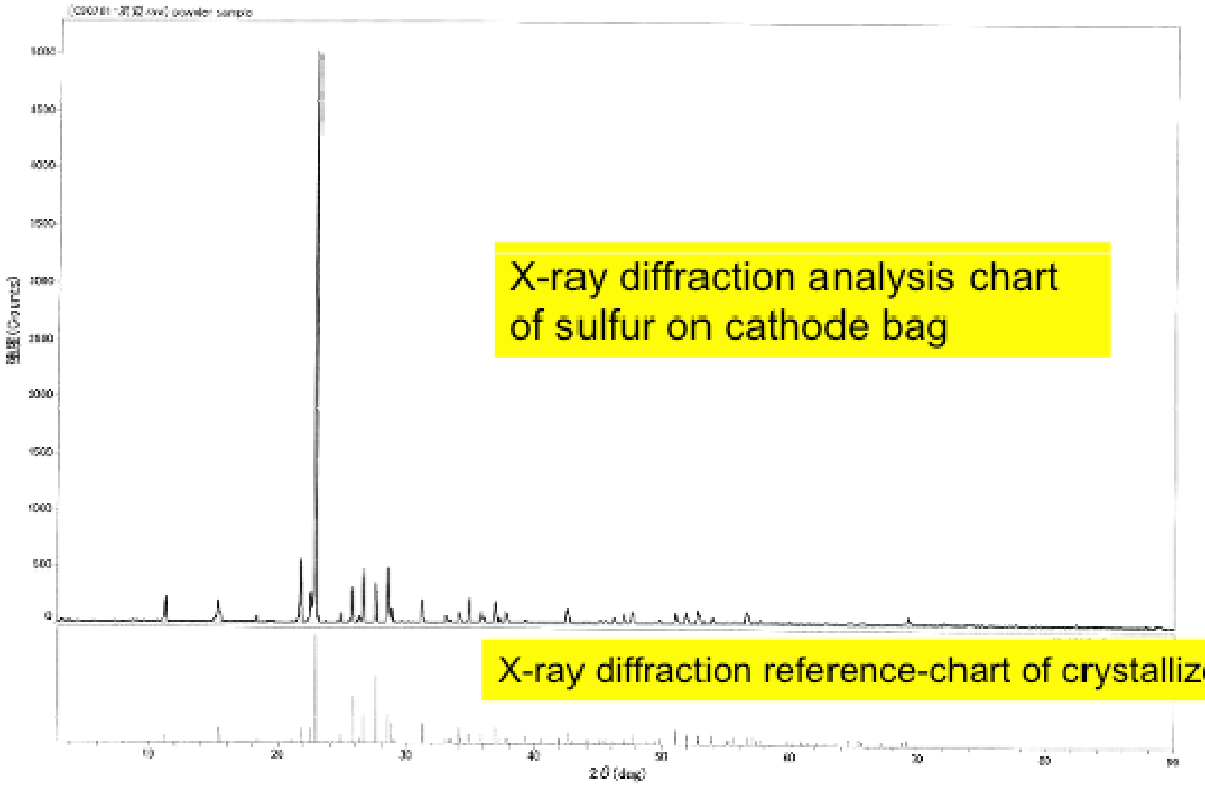


X-Ray Fluorescence (XRF) analysis of the rinsed ethanol

Results of analysis by XRF



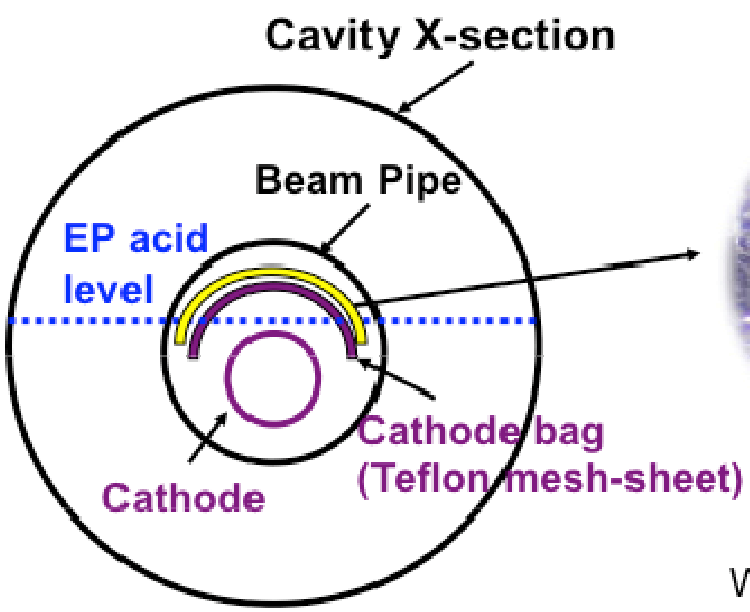
X-ray diffraction analysis

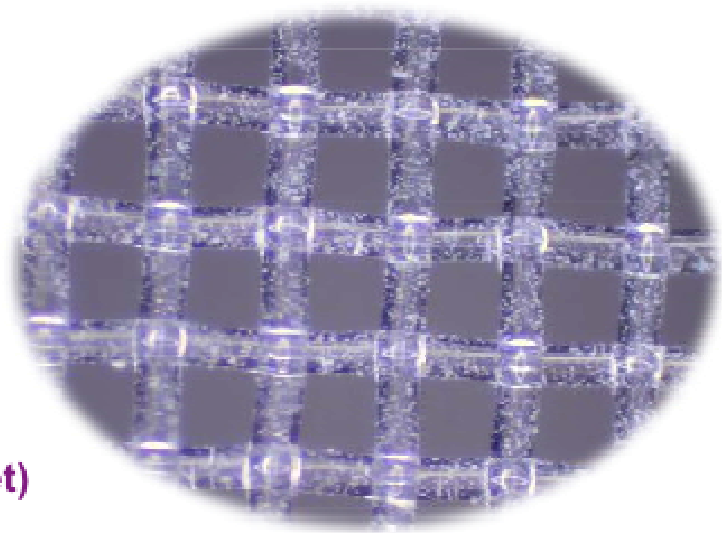


High Energy Accelerator Research Organization

The results shows that sulfur is in a crystallized form

Collection of sulfur powder on cathode bag

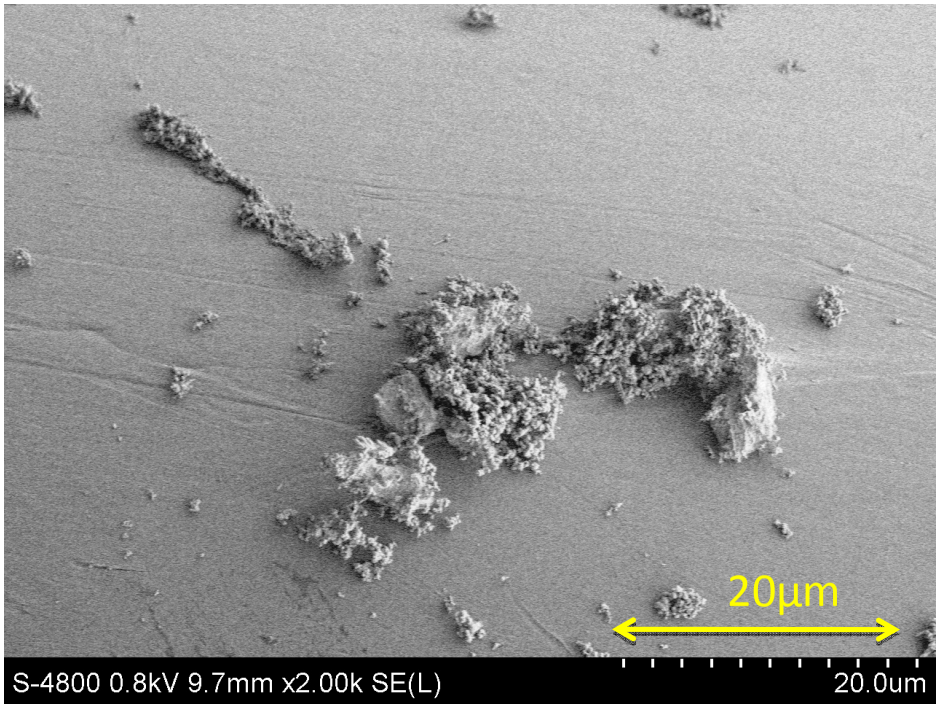


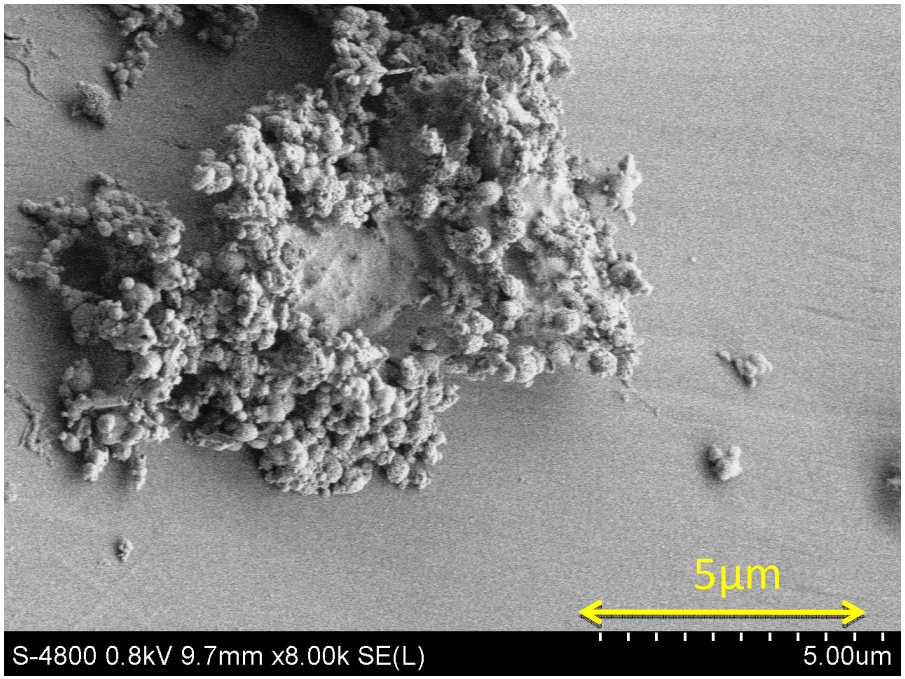


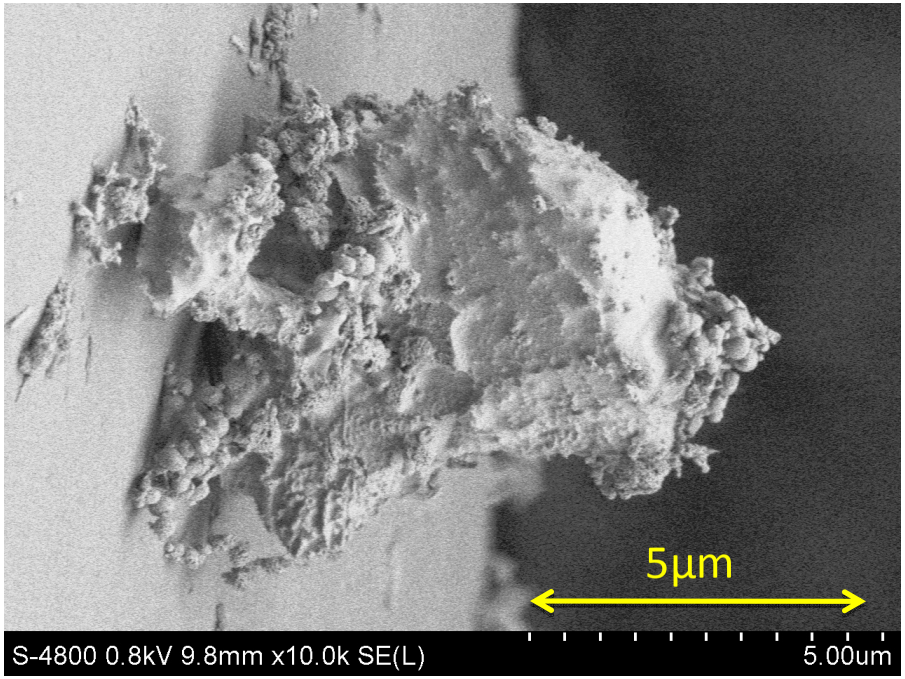
Location:
Iris Cell#4-cell#5, Iris cell#5-cell#6

We added detachable piece of Teflon mesh-sheet over the cathode bag. The sheet collected sulfur powder in the EP acid during the EP processes of cavities at STF/KEK.

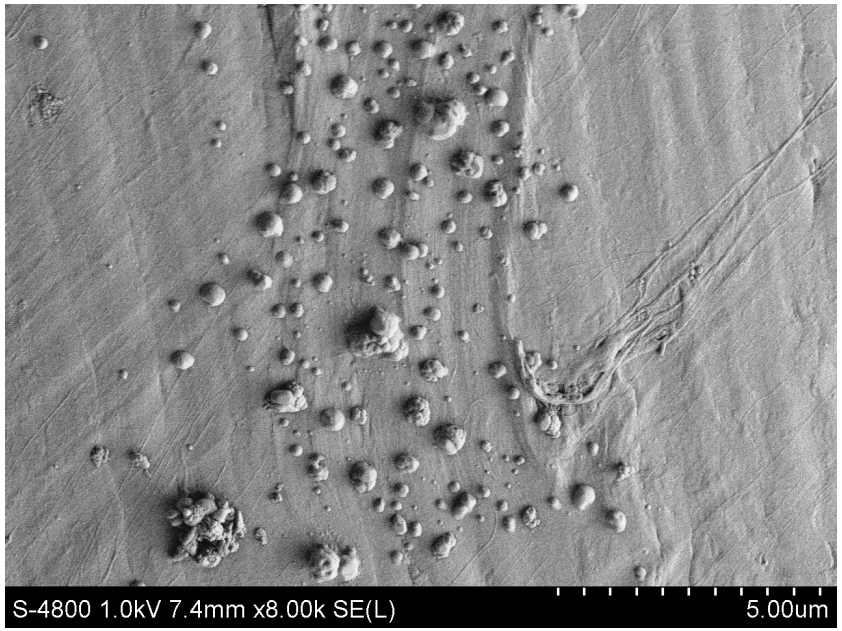
SEM images of Sulfur on the Teflon mesh (1)

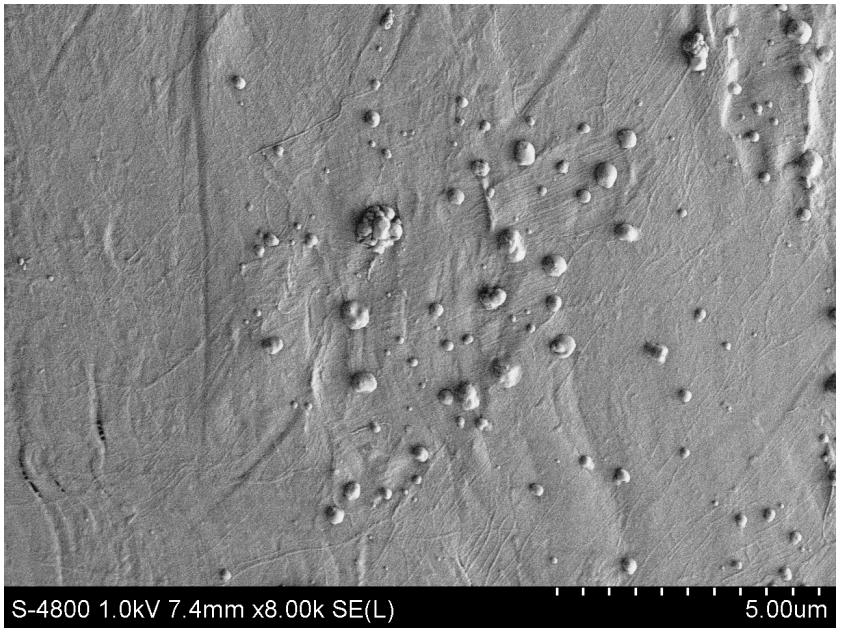


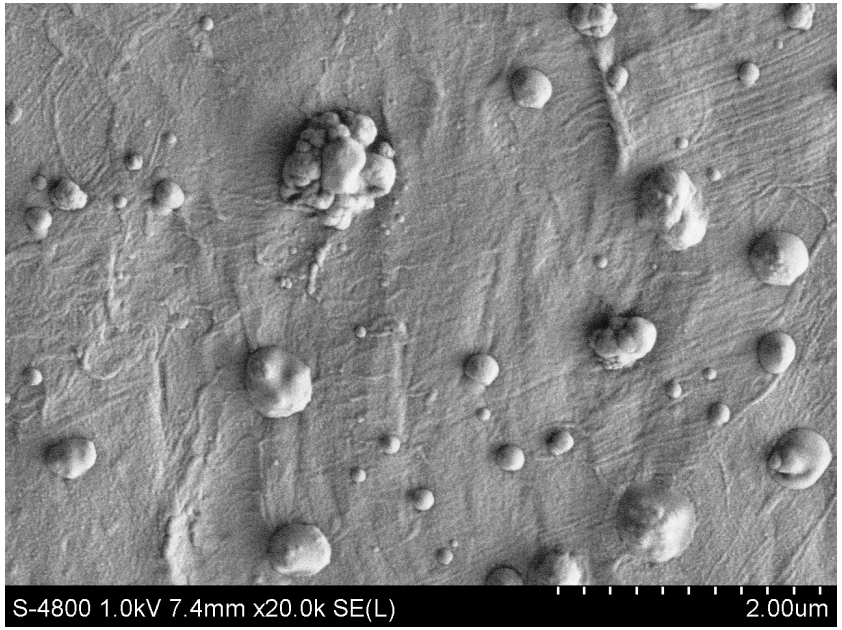


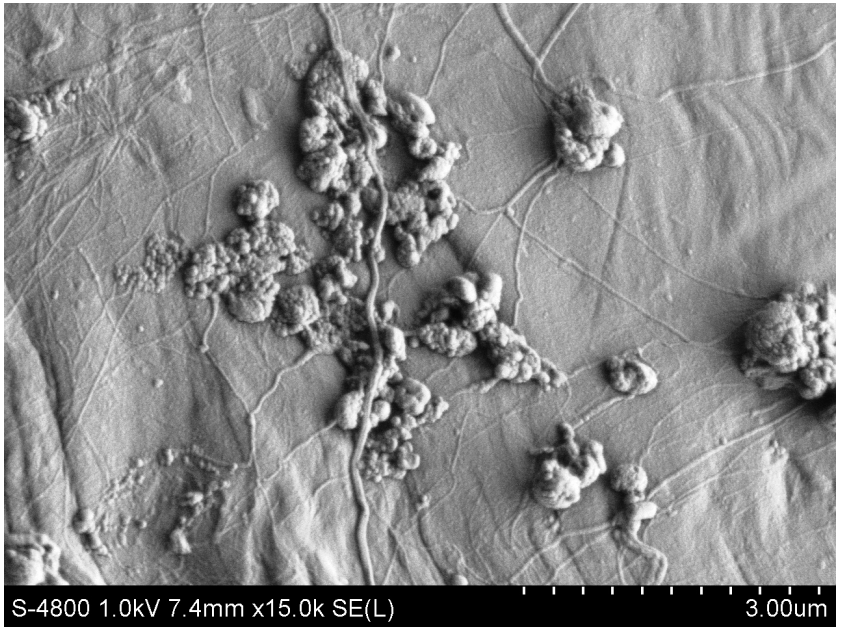


SEM images of Sulfur on the Teflon mesh (2)

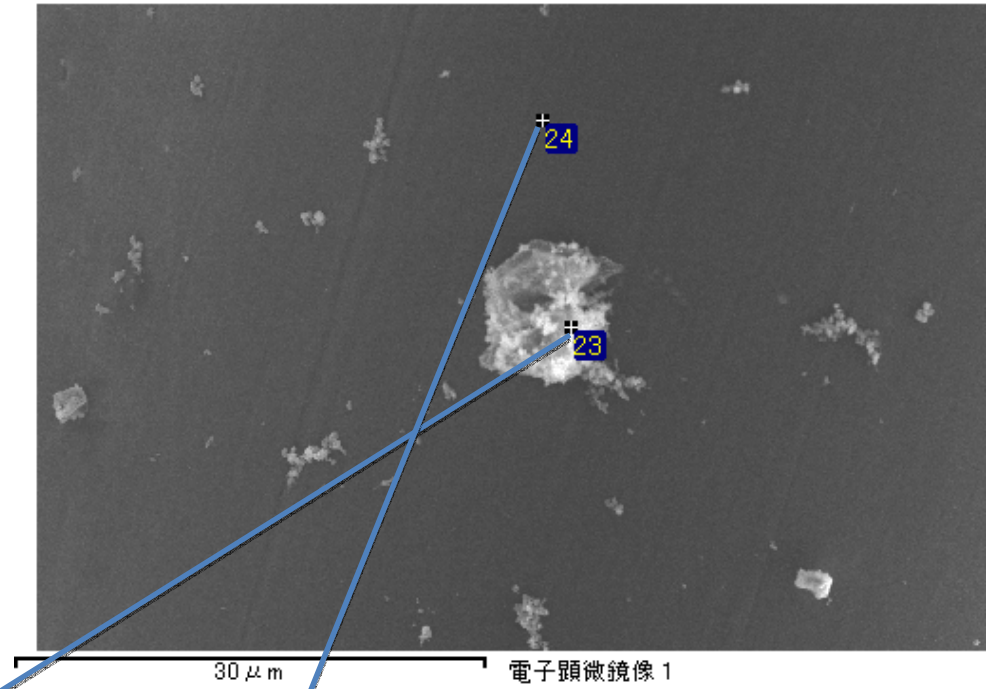




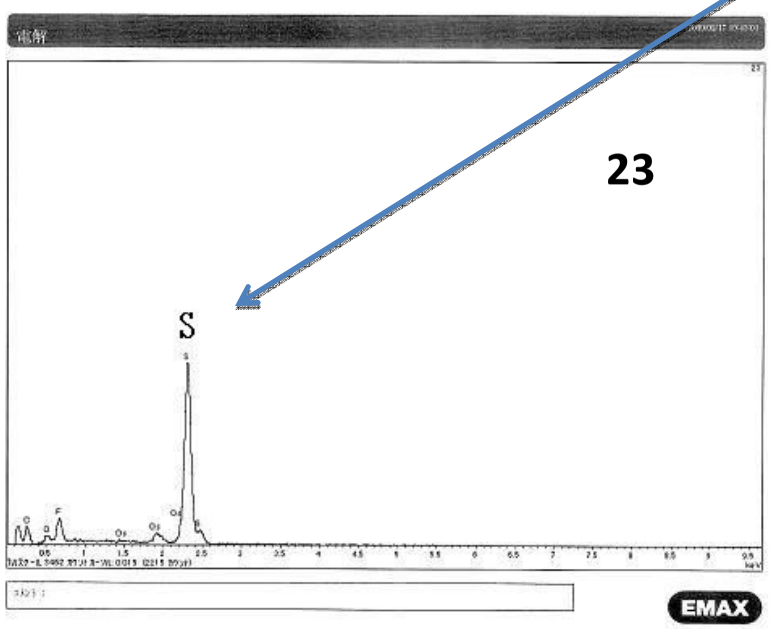


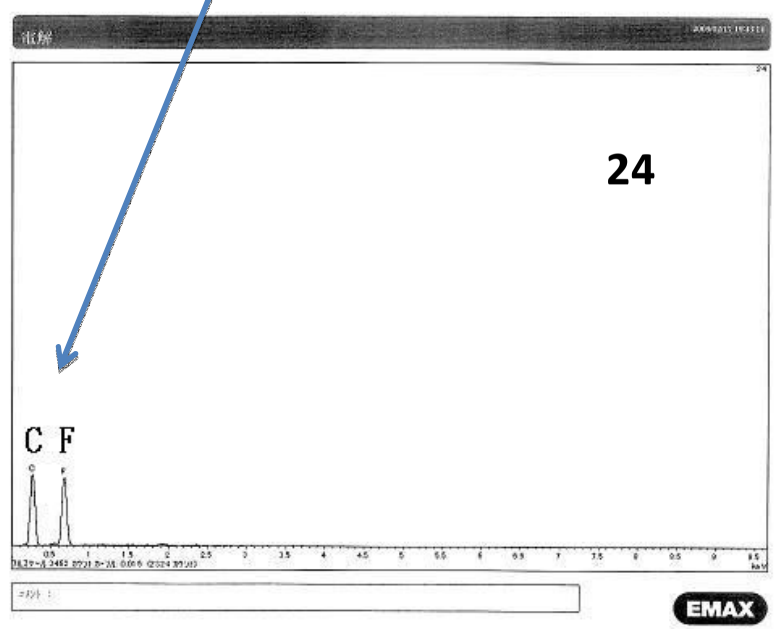


EDX analysis on the SEM images of Sulfur



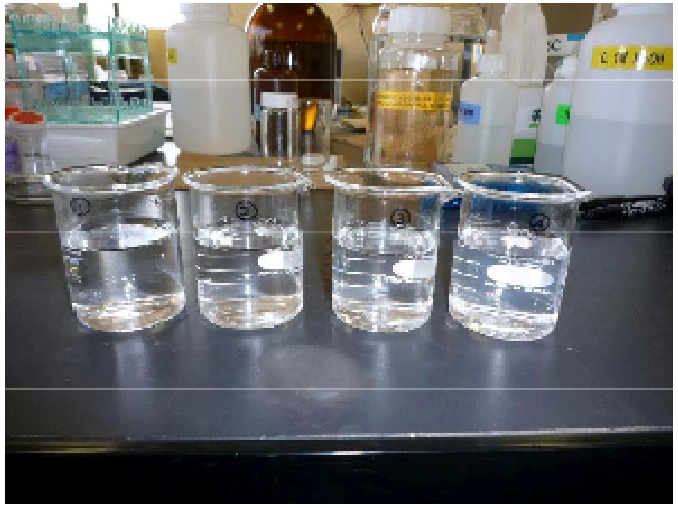
It's sulfur.



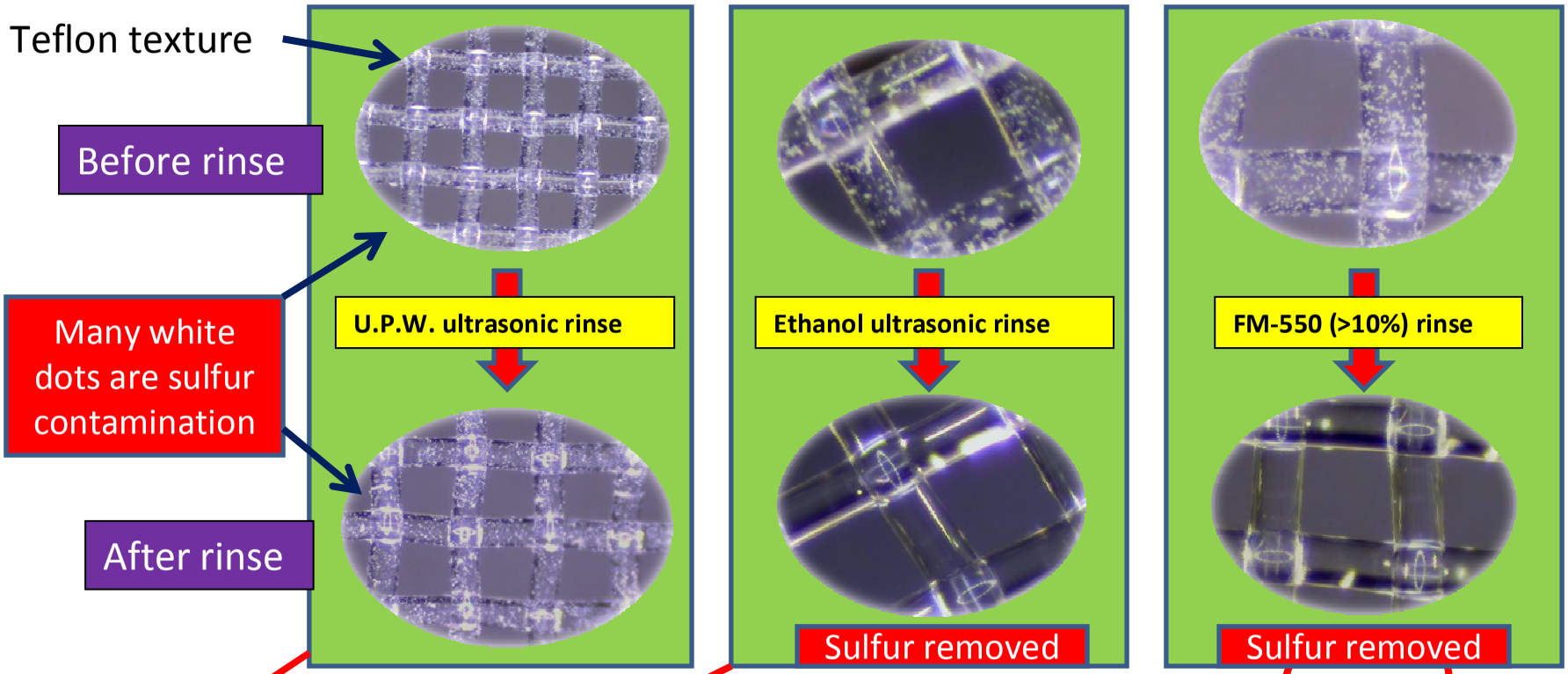


Rinsing effect of Degreaser（FM-550)

- Degreaser（LION SUNWASH FM-550）
- concentration = 0%, 2%, 5%, 10%
- 50ml, 50℃, 1hour, Ultrasonic rinse



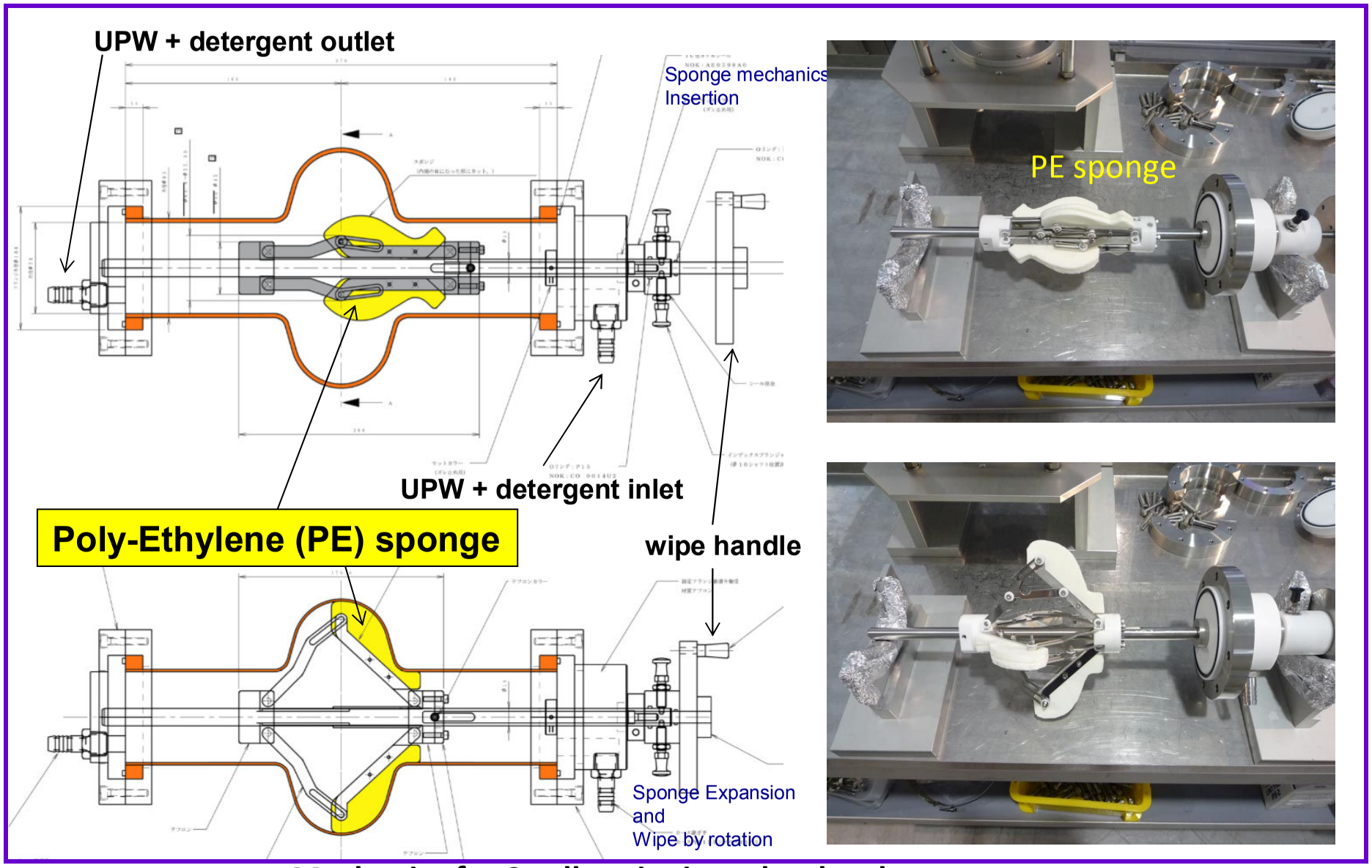
Sulfur Removal Test



	U.P.W. ultrasonic rinse	Ethanol rinse (vibration)	Ethanol ultrasonic rinse	Detergent FM-550 2 %	Detergent FM-550 5 %	Detergent FM-550 10 %	Detergent FM-550 20 %
Cleaning Result	✖	□	○	□	□	○	○

Development of sponge wipe mechanism for cavities

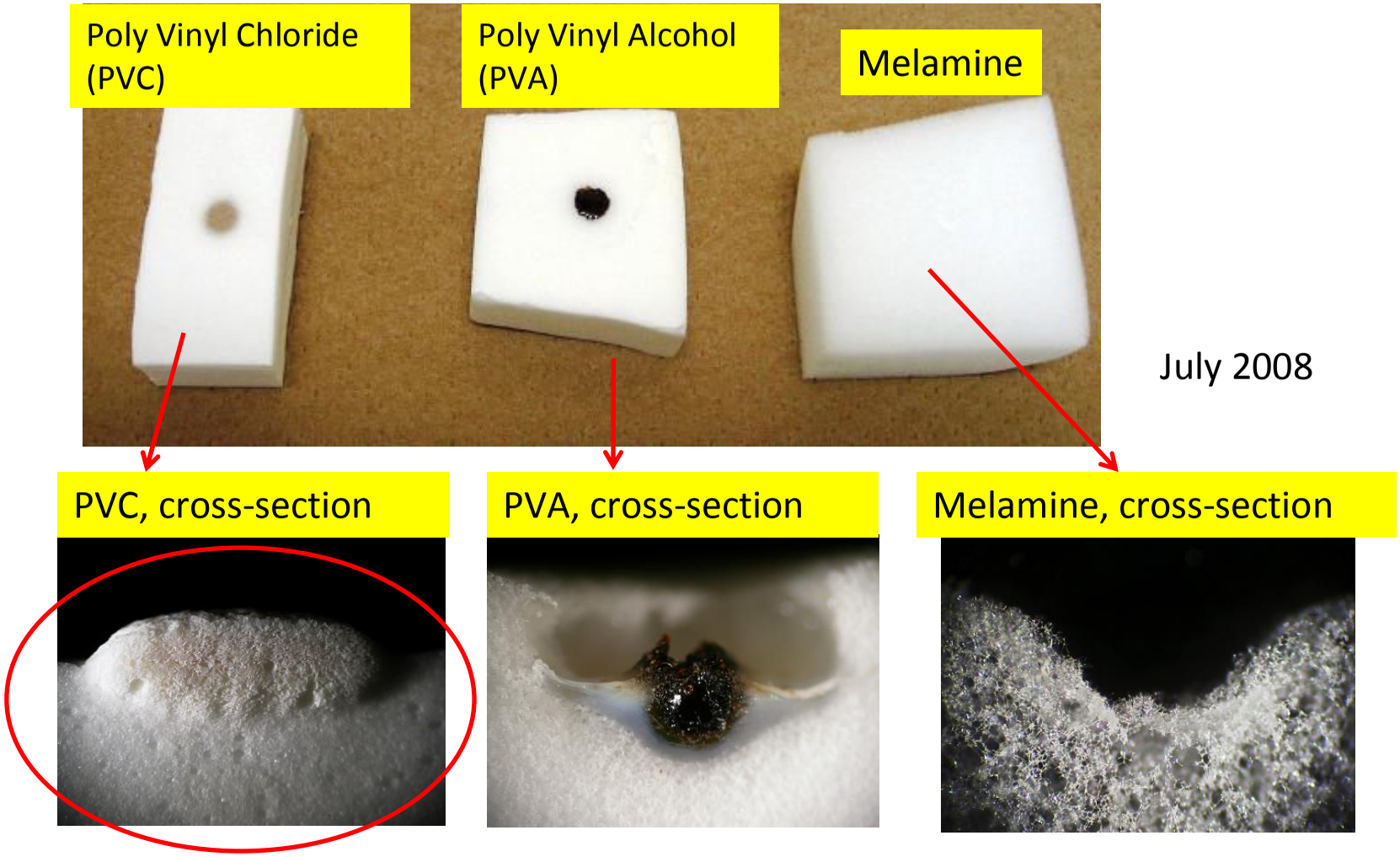
First wiping/fitting test of proto-type sponge-cleaner for single-cell cavity



Mechanics for 9 cell cavity is under development.

Melting test of sponges for EP solution

20 micro litter of EP solution (no dilution) was dropped on each sponge.



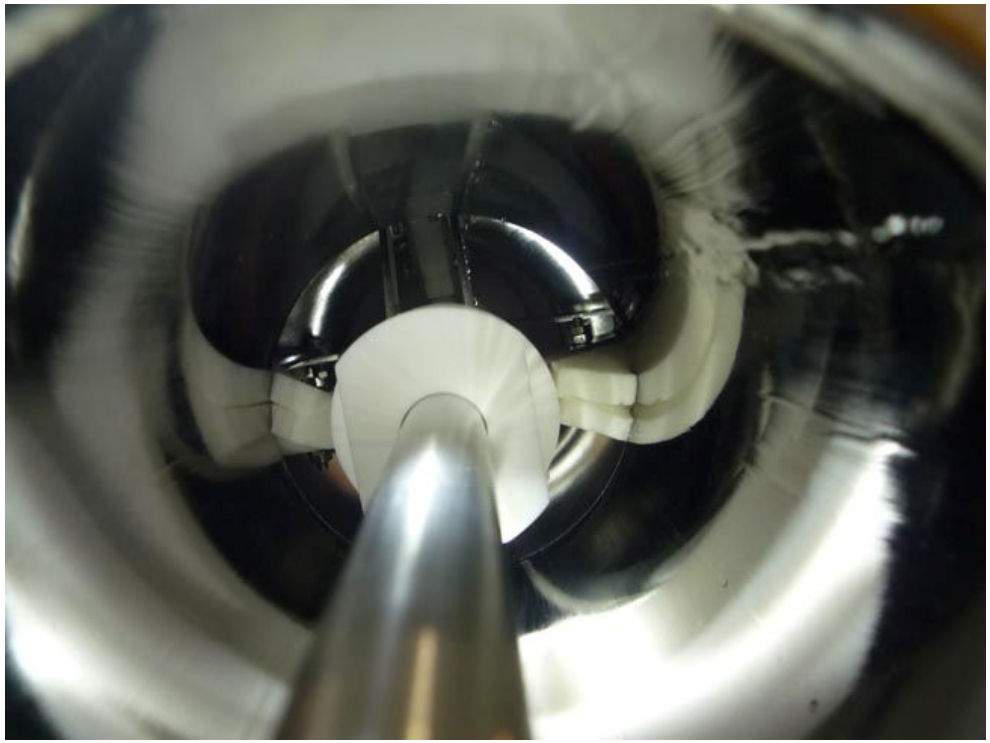
July 2008

We used PVC sponge for the first Nb-sample test (EP + Sponge cleaning).

The single-cell cavity was EP'ed and UPW-rinsed before the sponge-cleaning test.

Insertion of sponges

Expansion and rotation of sponges inside cavity



Feeding UPW during the rotation of sponges
for 1 – 2 minutes.

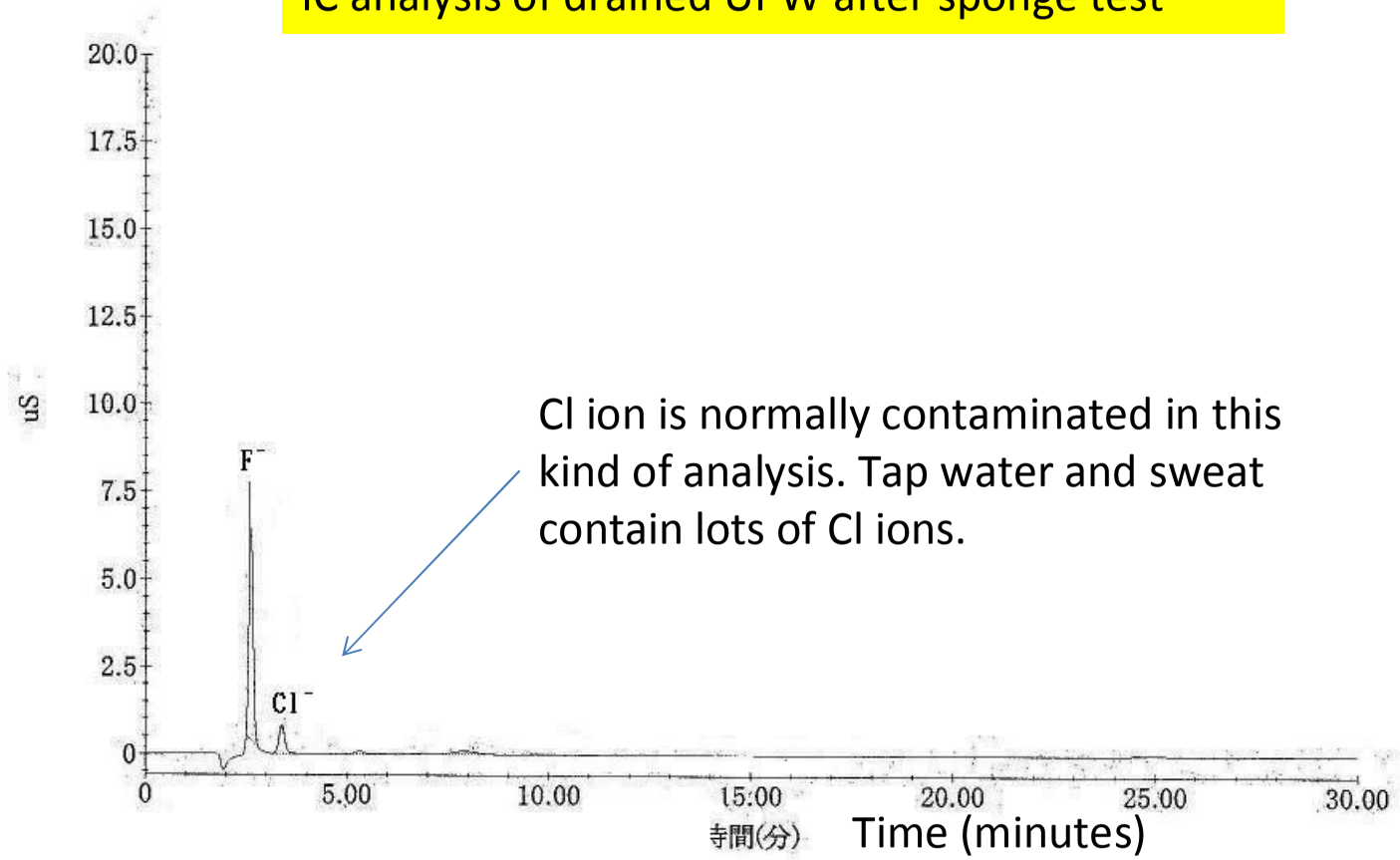
Draining UPW inside the cavity

Drained UPW was kept in a container to analyze the components/contamination.

Ion Chromatography for drained UPW

- **4.9 mg/L of F^- was detected from filtered/condensed UPW (70ml).**
- **Sulfur ion (S, SO_4, etc) was not detected. The components were different from that of EP acid.**

IC analysis of drained UPW after sponge test



Cl ion is normally contaminated in this kind of analysis. Tap water and sweat contain lots of Cl ions.

Residuals on the filter-paper

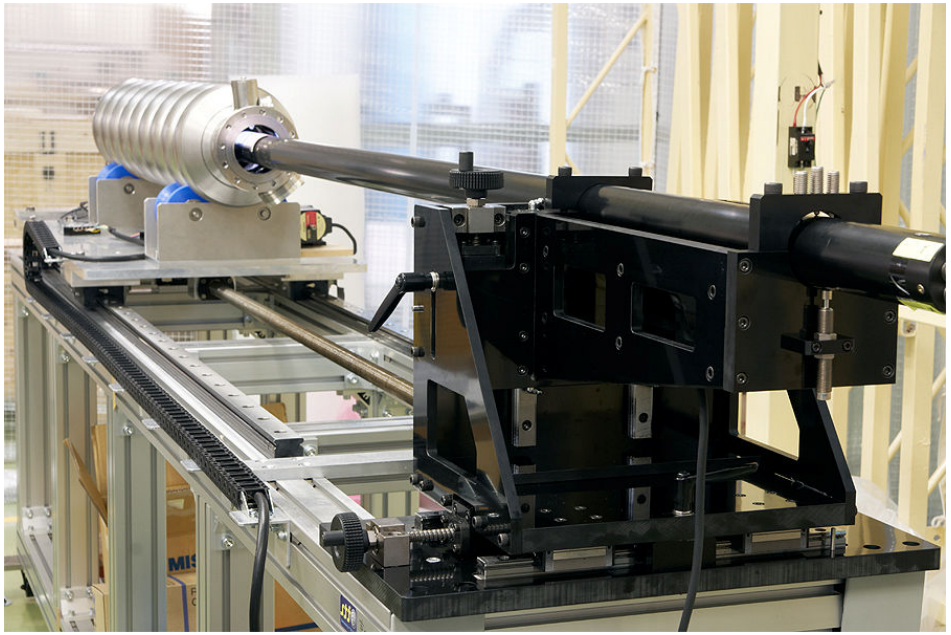
↔
1 mm

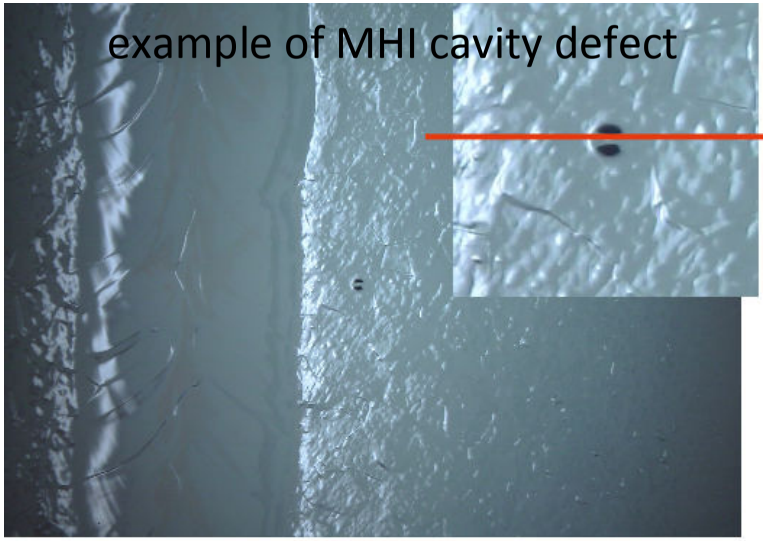
Residuals look like fibers/particles

Problems of sponge wipe? → HPR will help to clean up.

Upgrade of Inspection Camera

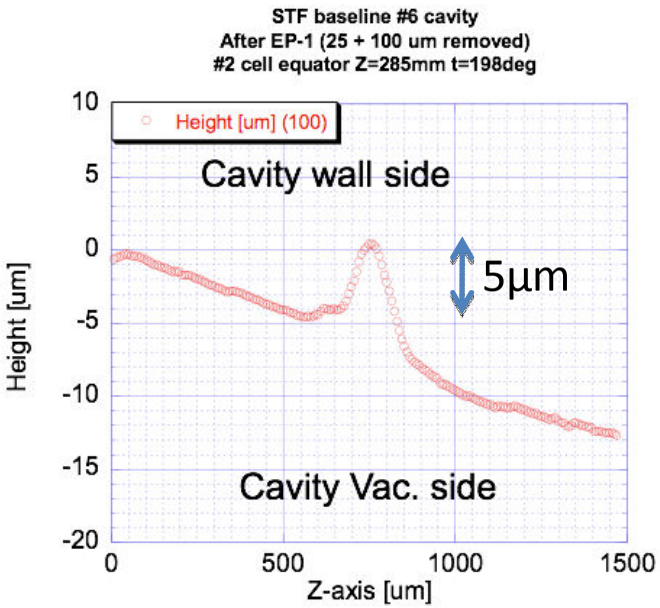
Inspection Camera



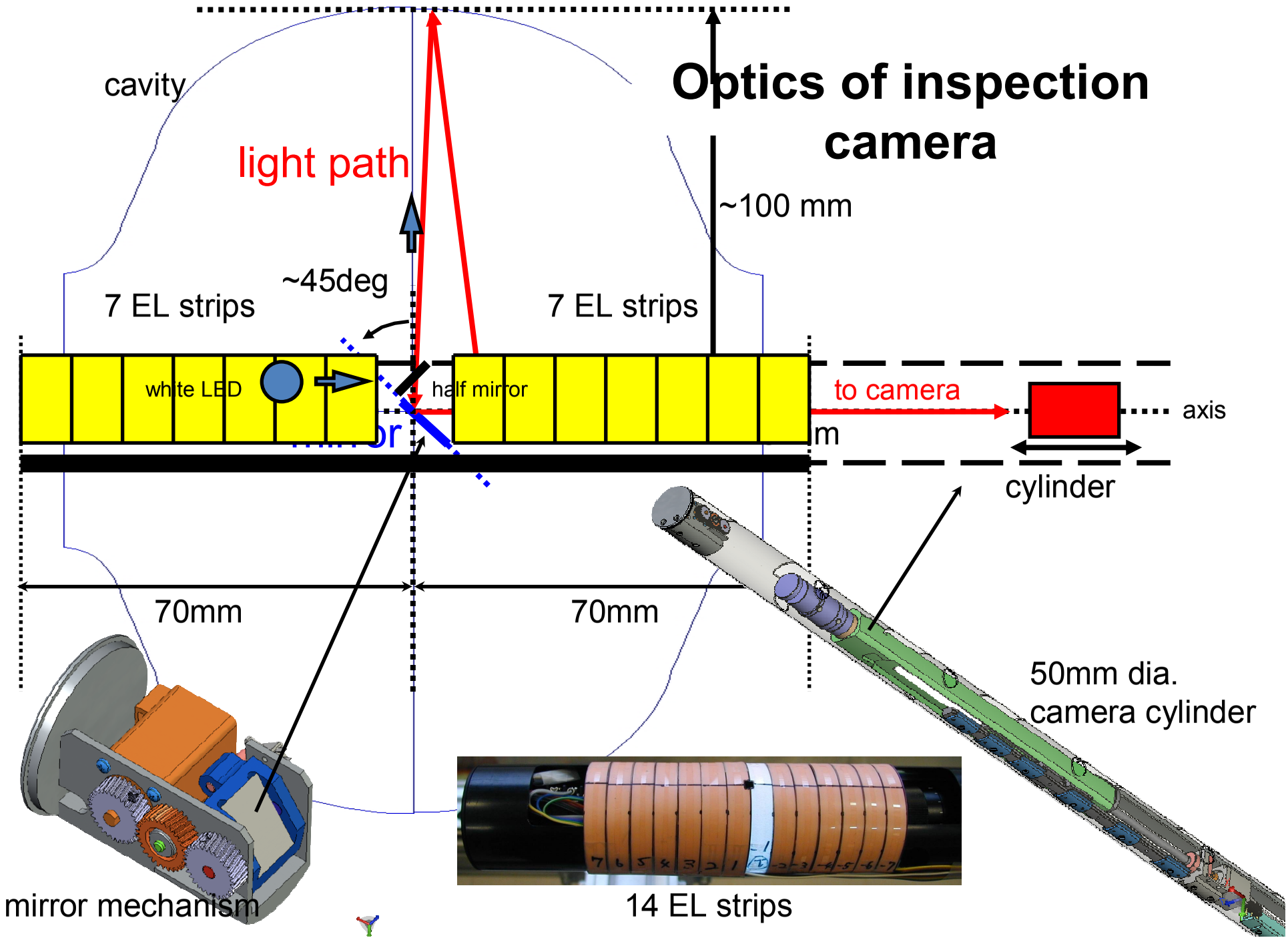


(1) 7µm/pix resolution, 13mm x 9mm view,
several 10µm object observable.

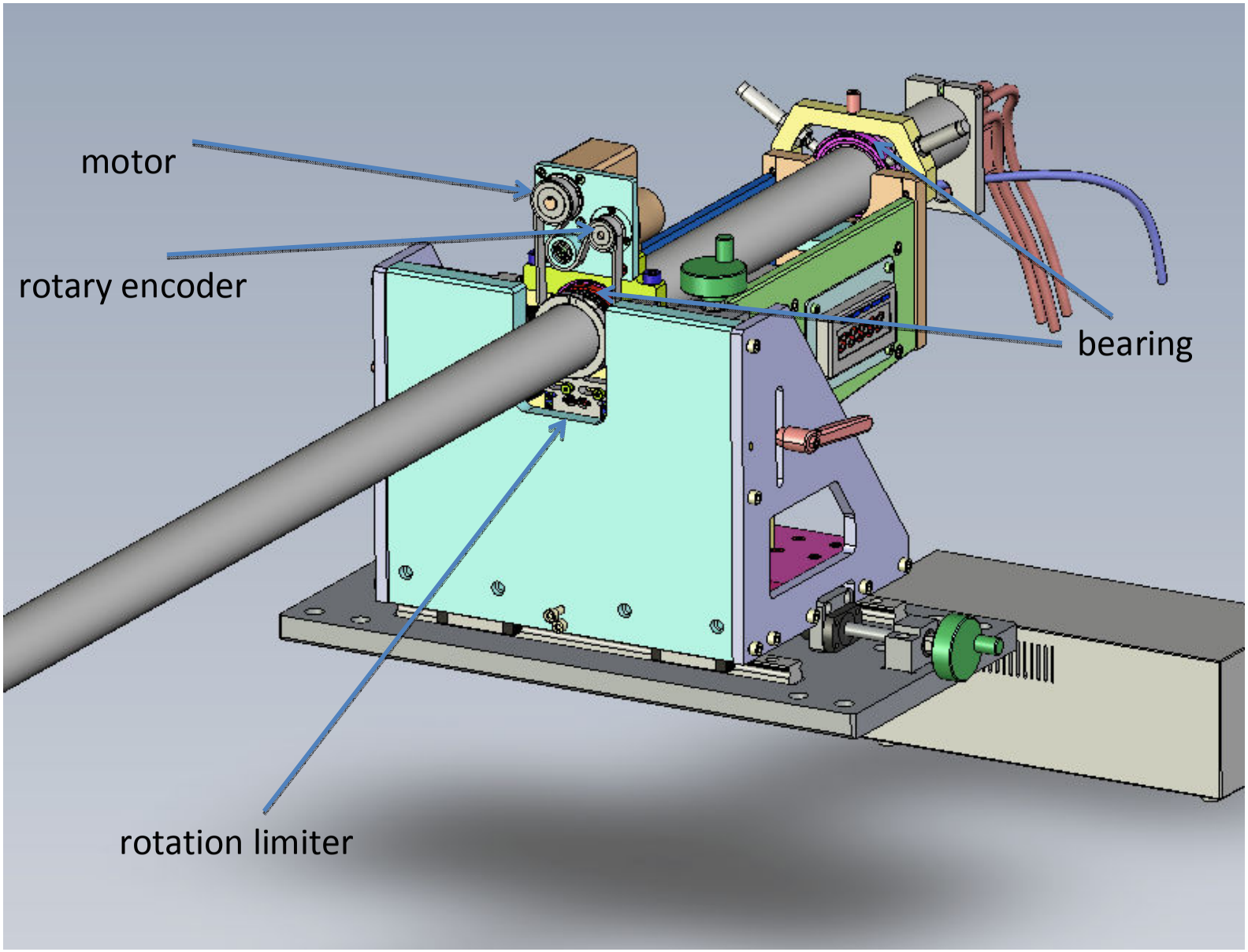
(2)direct LED illumination,
14 EL strip illumination for
wall gradient measurement capability



Optics of inspection camera



Introduction of camera-cylinder rotation mechanism
for cavity inspection with EP-VT support jigs,
and for jacketed cavity.



Upgraded camera under fabrication

(1)CCD camera upgrade

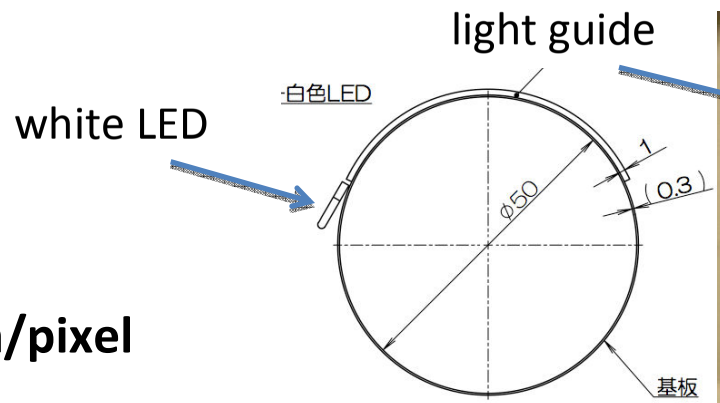
from 5μm CCD pixel
to 2.2μm CCD pixel camera.

(2) Lens upgrade

more magnification
with more larger aperture.

(3) illumination upgrade

EL panel has limited life with more high voltage
(for more brightness).
-> LED + light guide with scattered surface (twice more light).



~7μm/pixel → targeting ~3 μm/pixel

Image capture automation

(1)control of cavity position

being done by VB application.

(2) Image capture and automated file-save

already done by VB application.

(speed is enough fast, but must wait for vibration damping)

(automated focus is the next concern)

(3) defect pattern matching

the software already fabricated in 2007

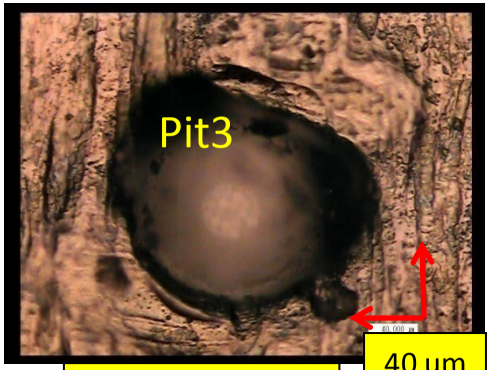
was tested using recent high quality pictures.

-> no good results, so far.

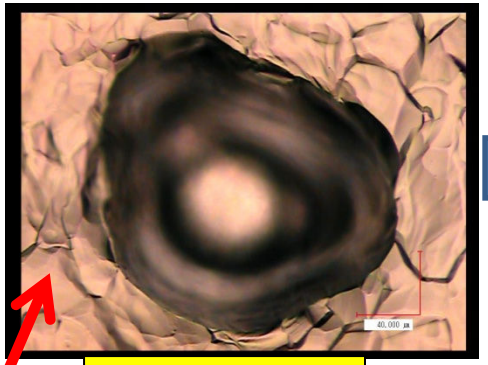
(match to every bright traces, not suspected defects only)

Development of Local grinding mechanism for cavities

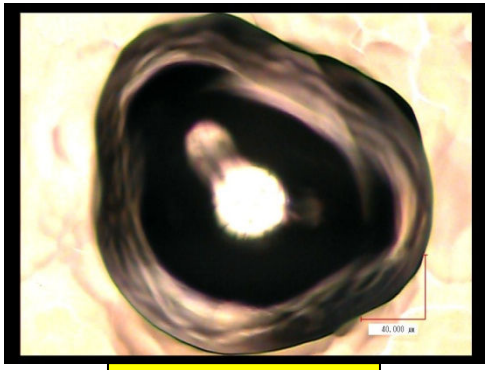
EP test of intentional pit sample (Jlab made)



As received

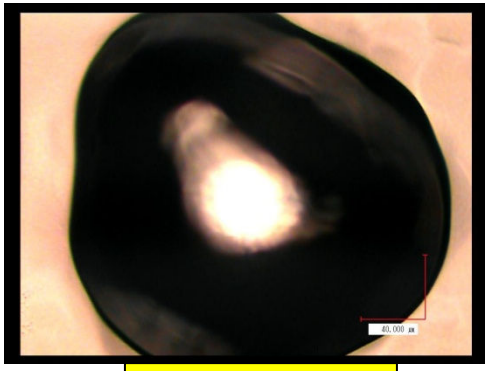


BCP (30um)

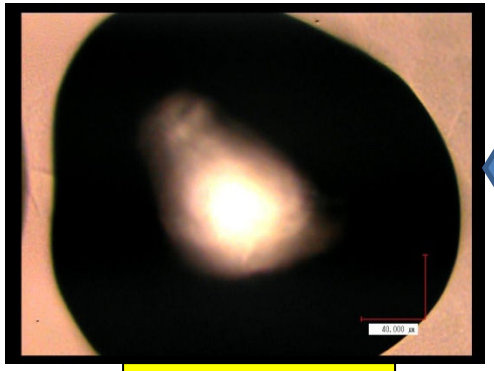


EP (30um)

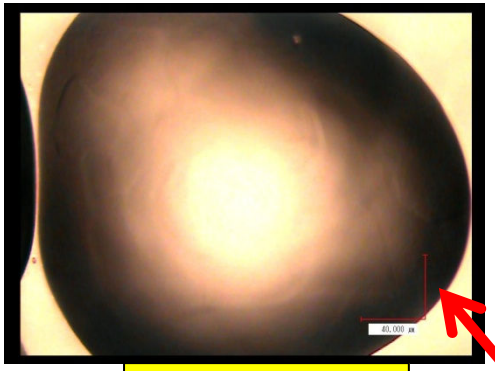
Large steps at grain-boundaries after BCP.



EP (60um)



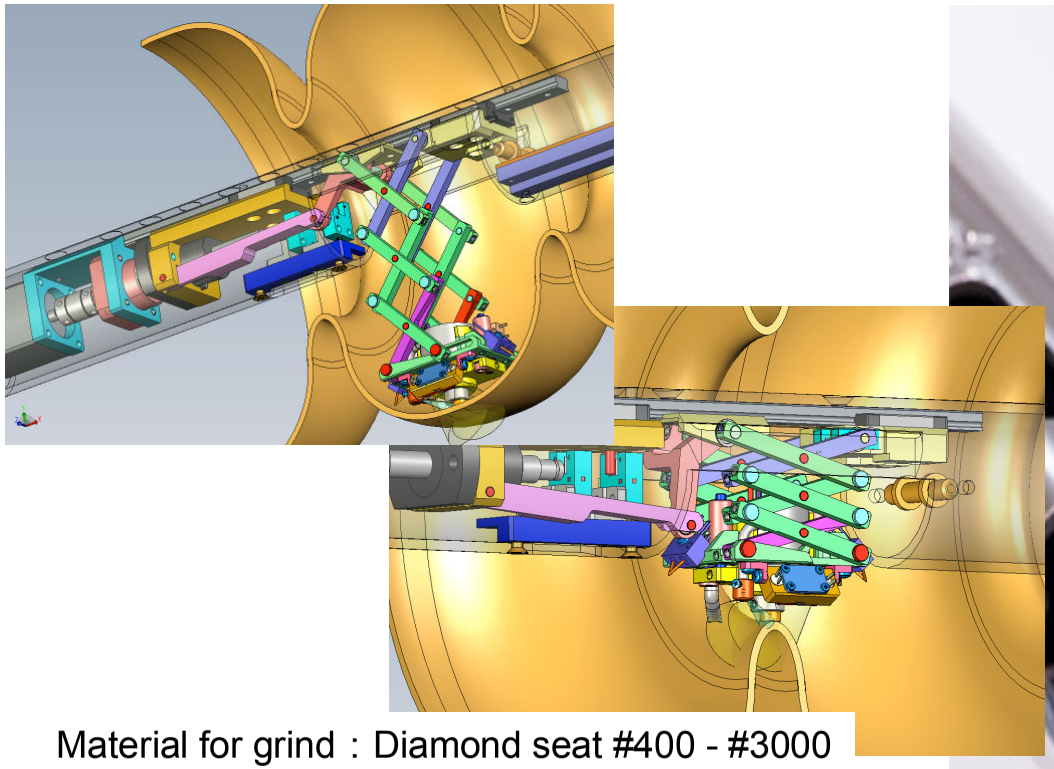
EP (90um)

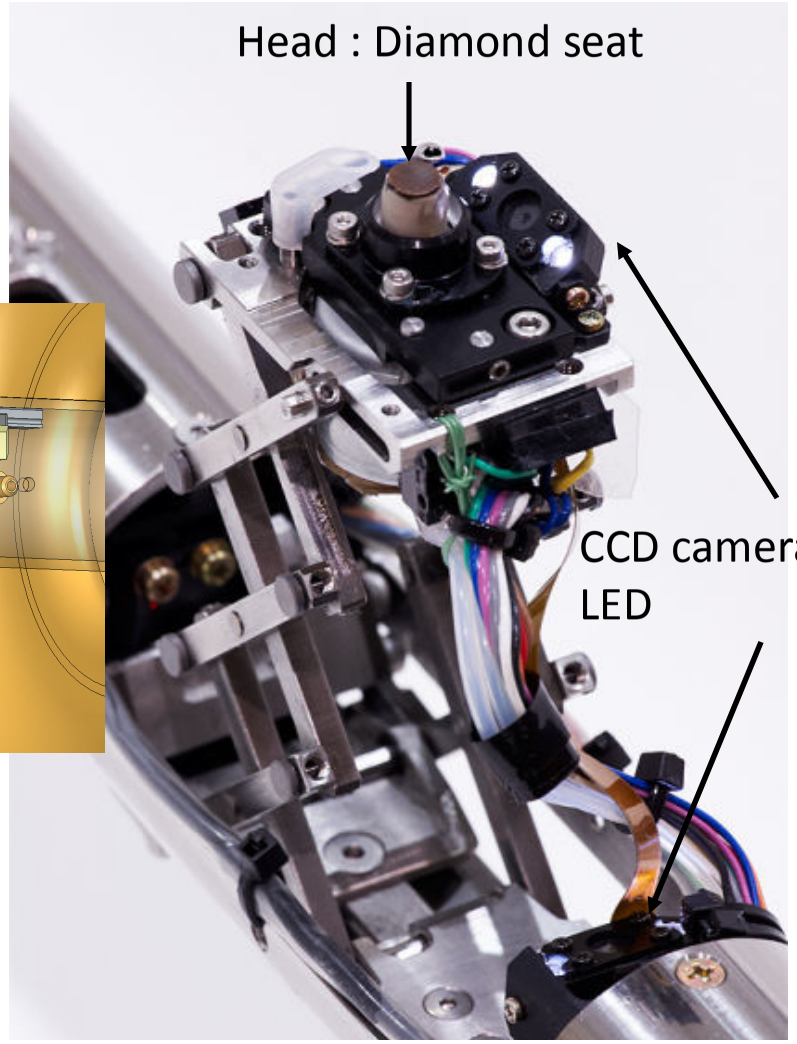


EP (120um)

Still sharp edge even after BCP(30um)+EP(120um).
EP seems difficult to remove pit sharp edge.

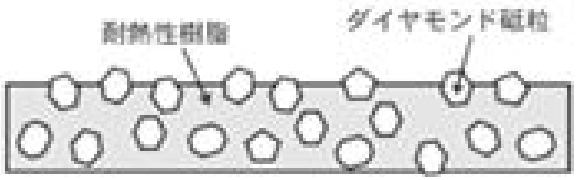
Local Grinder development





Material for grind : Diamond seat #400 - #3000

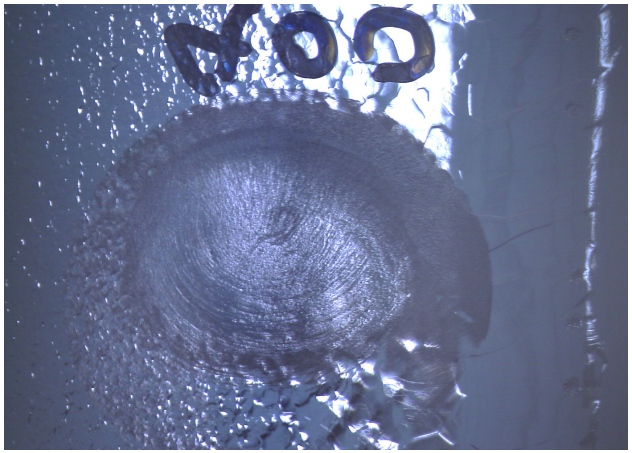
(particle size = 40 ~ 3 um), (POLYMOND)



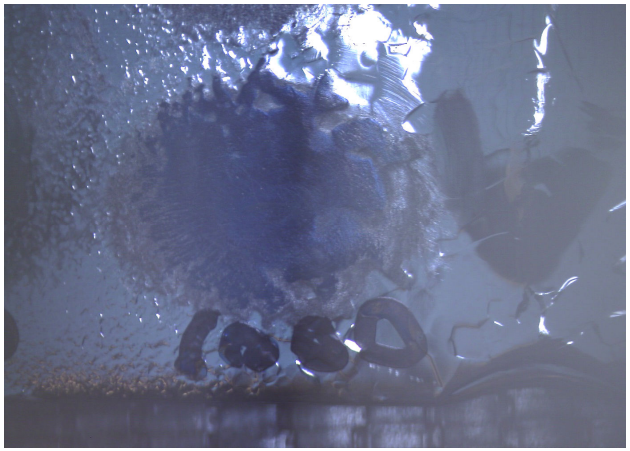
Grinding machine was delivered, and tested.

Grind Test Results for Nb plate

Just after grinding (~6mm diameter region)



15min grind by polymond#400
(40-60μm diamond)

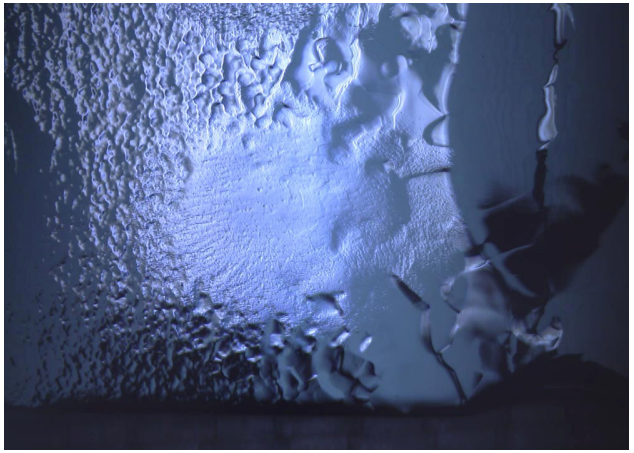


15min grind by polymond#1000
(10-20μm diamond)

30min grind by polymond#3000
(3-8μm diamond)

After 50μm EP

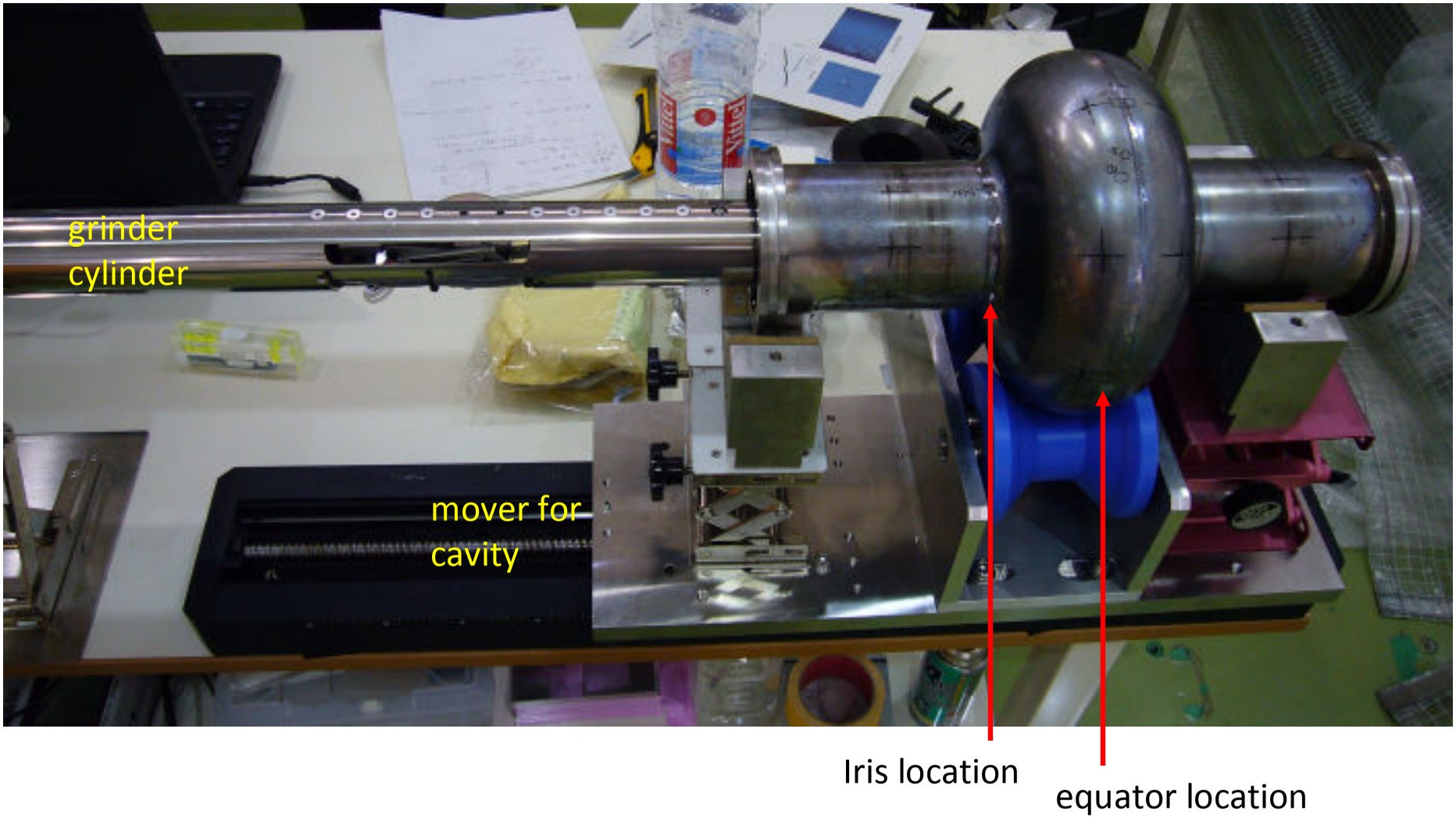
NG



Good

excellent

Single cell cavity grind test

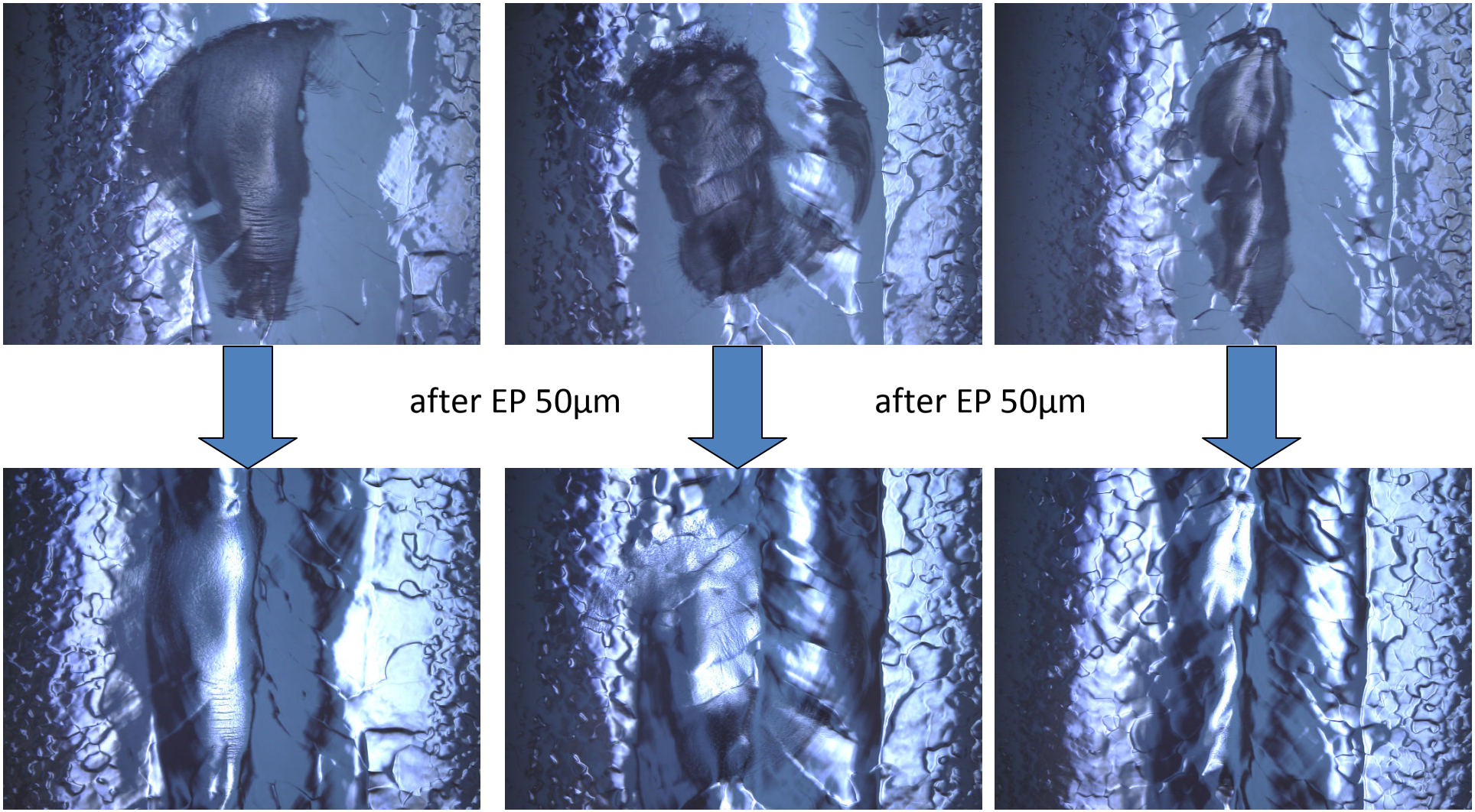


Grinding examples at equator

equator(1) new#1000, 15min

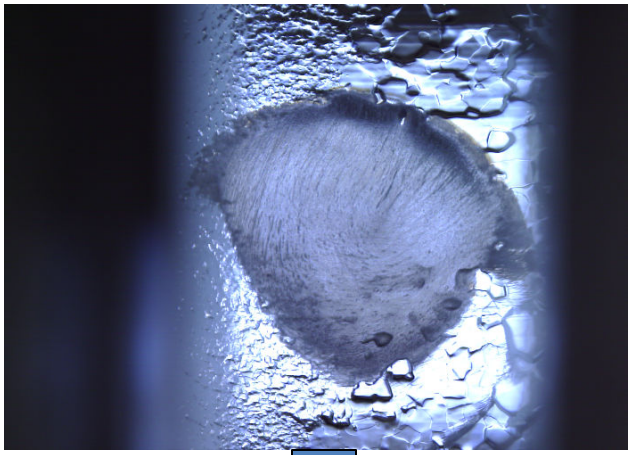
equator(2) used #1000, 15min

equator(3) used #1000, 15min

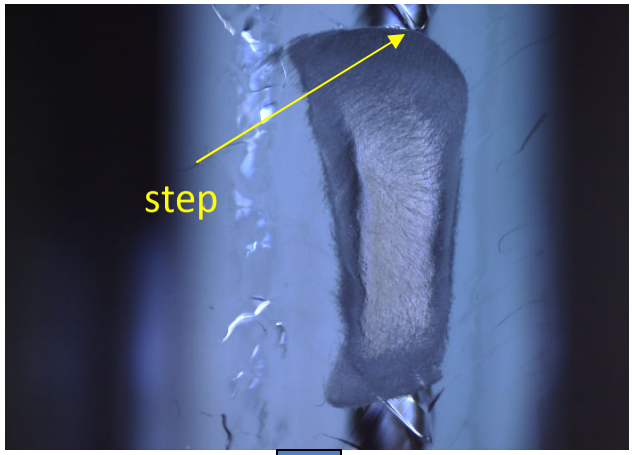


Grinding examples at iris

Iris(1) new#1000, 15min



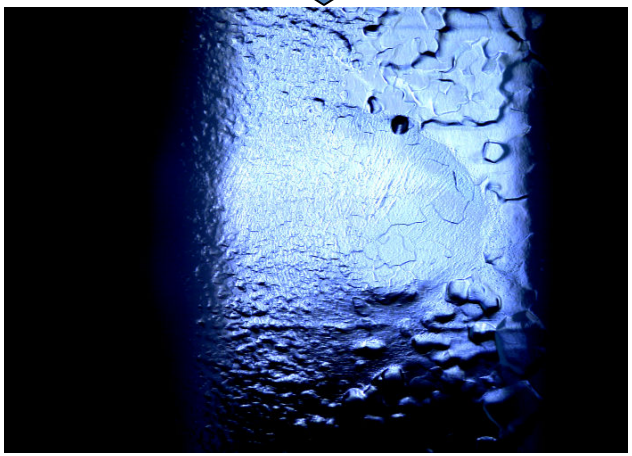
Iris(2) new #1000, 15min

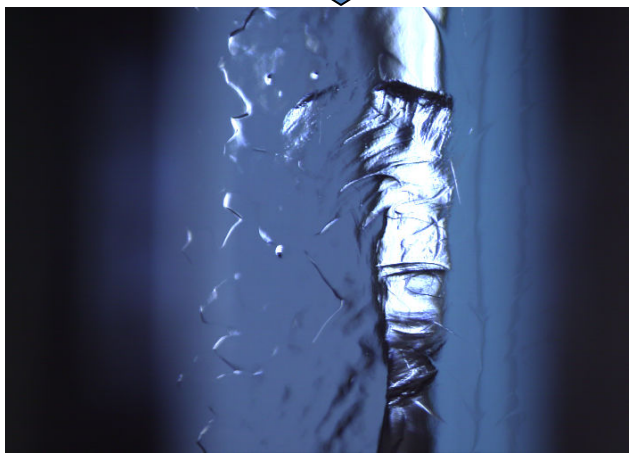


Iris(3) used #1000, 15min

after EP 50µm

after EP 50µm





The grinder touches on small area of iris, need to pay attention for smooth start of grind. Otherwise it make scratches.

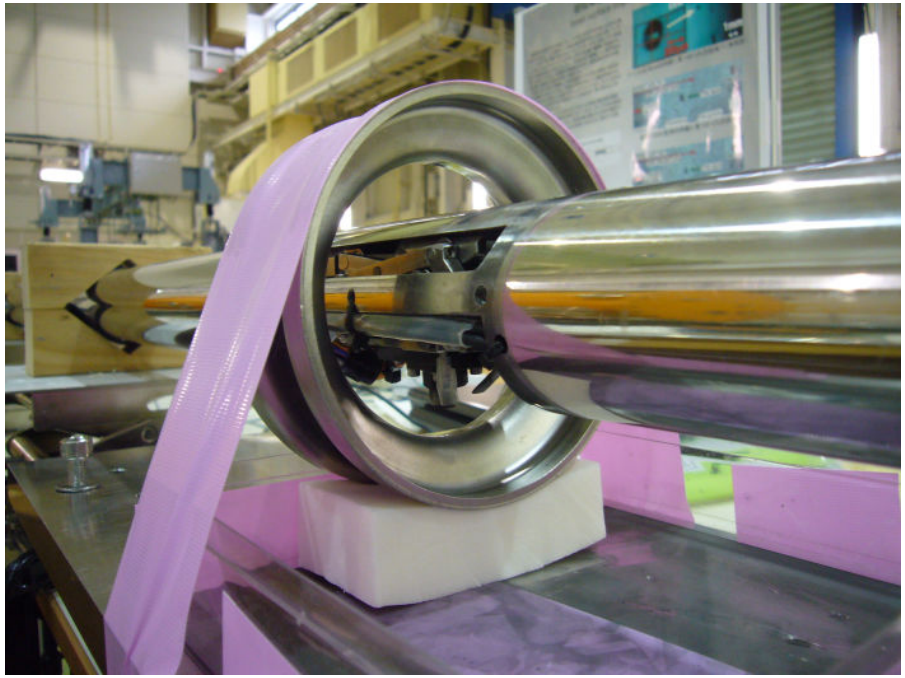
Grinding test using Cornell Iris weld sample

Before to grind AC71 iris, the grinding test was done using Cornell Iris weld sample, which was given to Akira Yamamoto last year. Thanks to Cornell people.

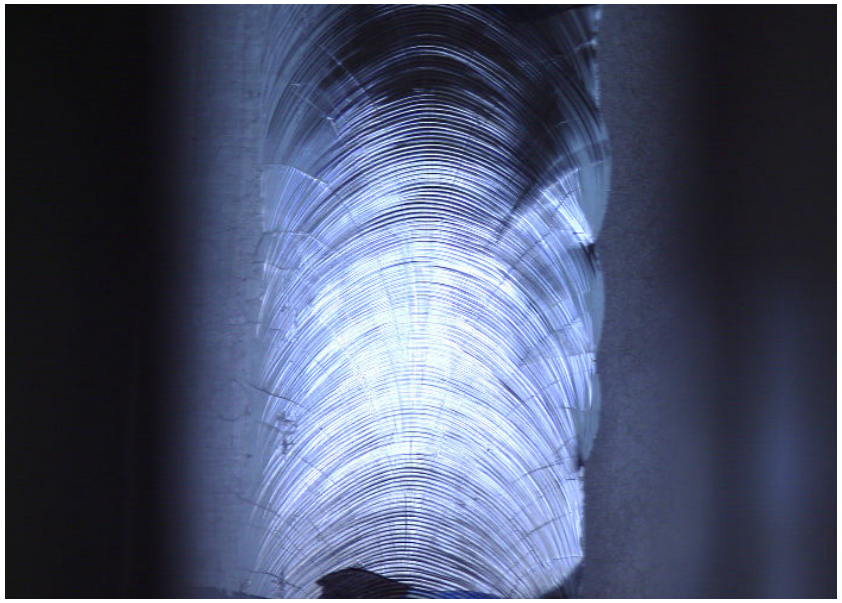
The iris is 78mm diameter with flat EBW seam, no surface treatment.

(1) Check of the grinding head fitting to iris EBW seam.
(2) evaluate surface finish for #1000 diamond sheet or #3000 diamond sheet.
(3) estimate grinding time, grinding rotation speed.
(4) use of water injection pipe.
(5) use of pre-installed CCD camera (grinder camera).

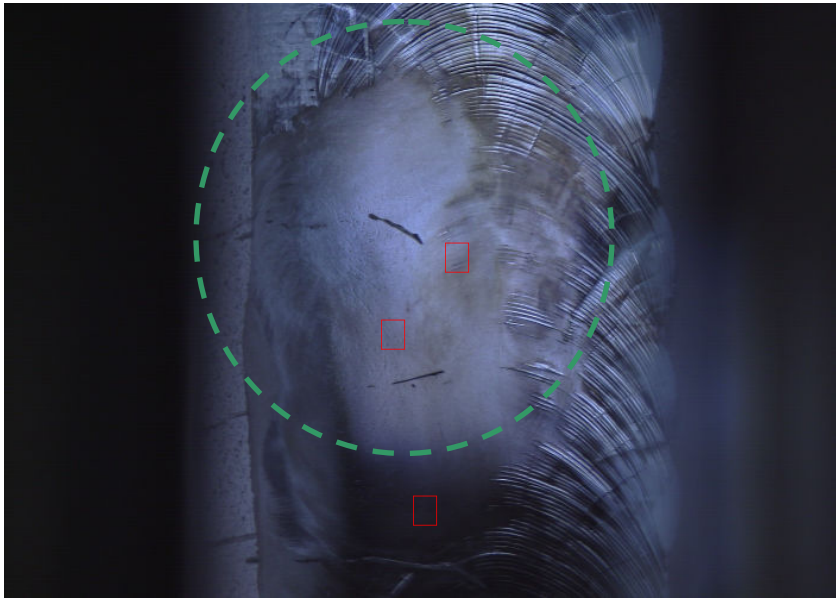
The water should be injected just before to start rotation.
Otherwise,
rotation head is easy to bite into Nb surface, and no rotation anymore.



Before scratching (view by Kyoto-camera)

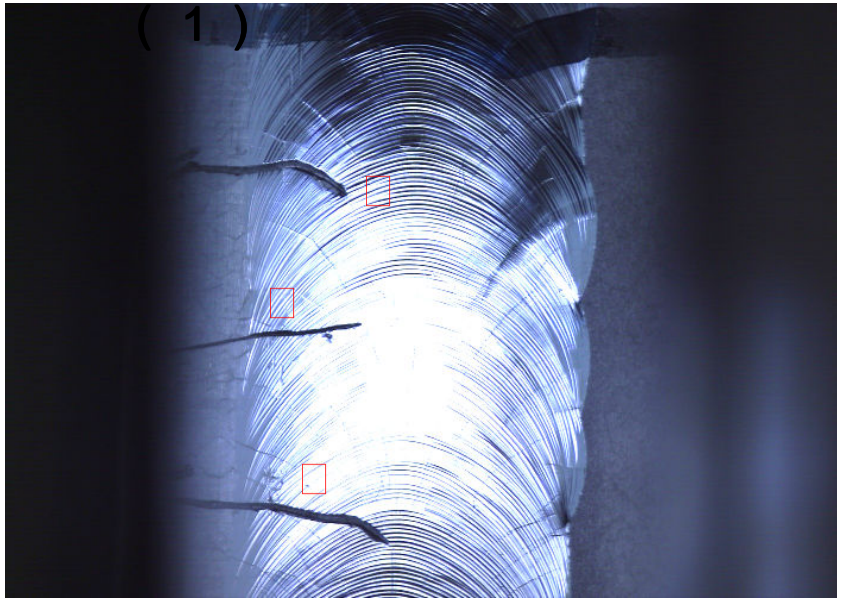


using #3000 diamond sheet
with 110 + 20 + 30 min

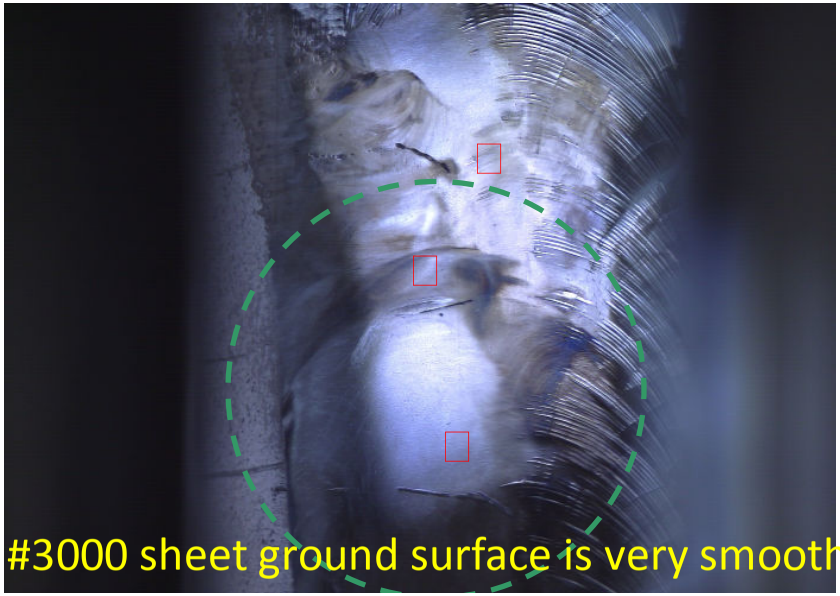


After intentional scratch

(1)



using #3000 diamond sheet
with 110 + 20 + 30 + 80 min



Surface roughness measurement

Instrument : Mitutoyo SJ-301

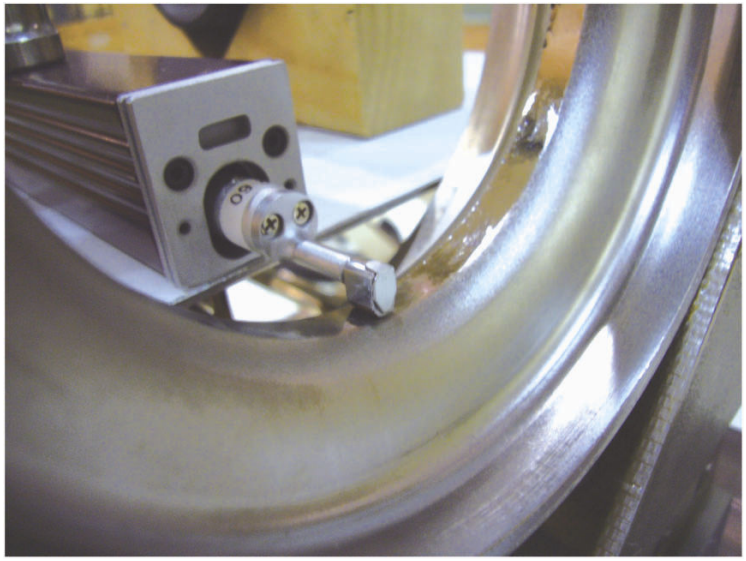
sweep scan along beam direction for

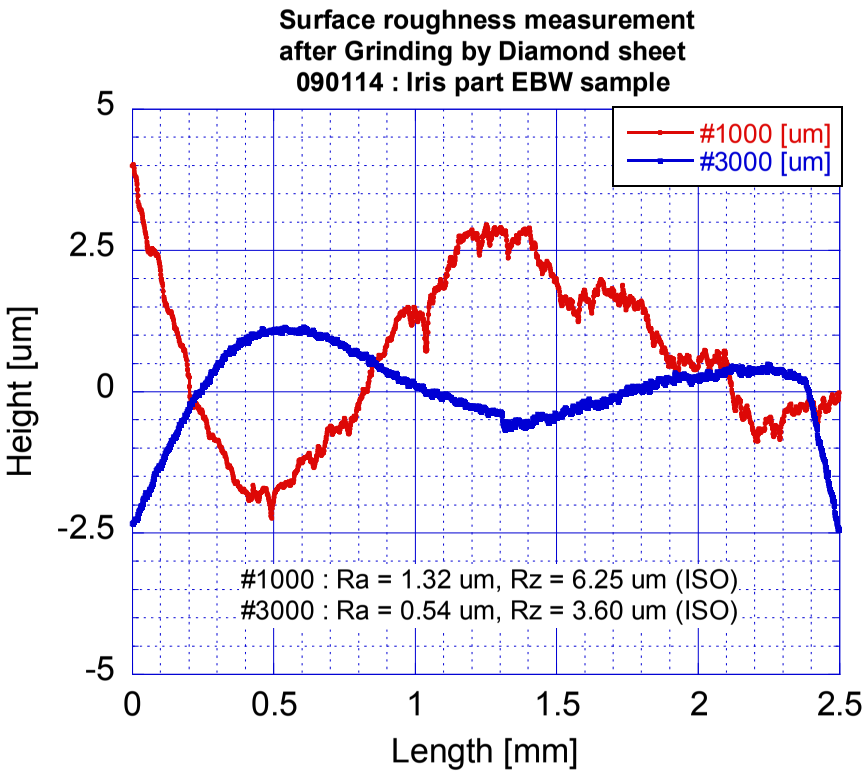
(1)ground surface by #3000 sheet

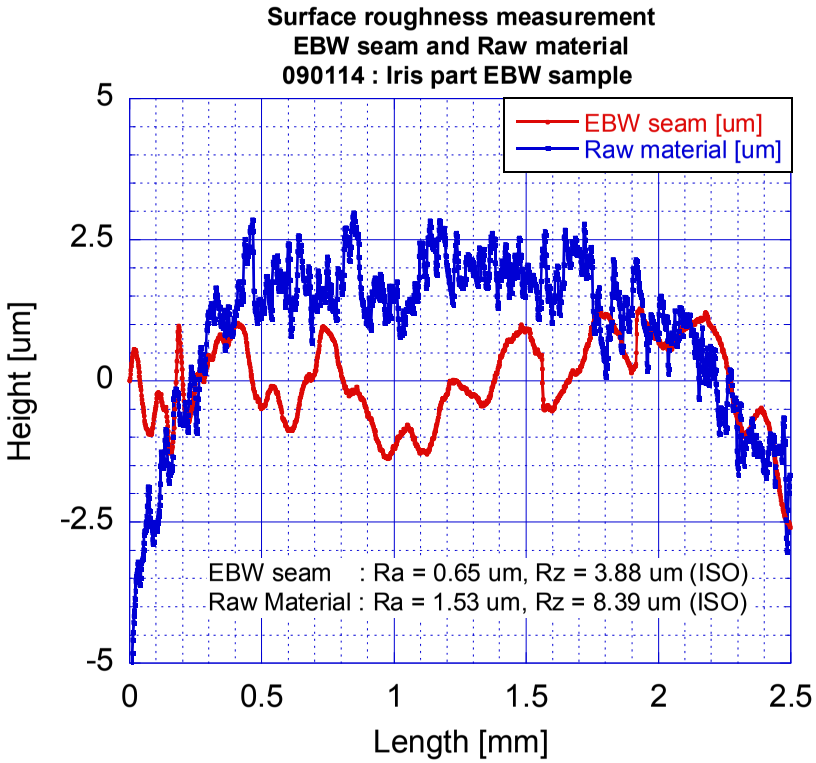
(2)ground surface by #1000 sheet

(3)EBW surface

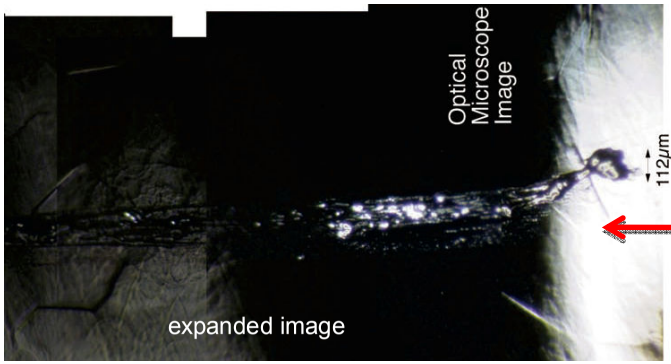
(4)raw Nb surface

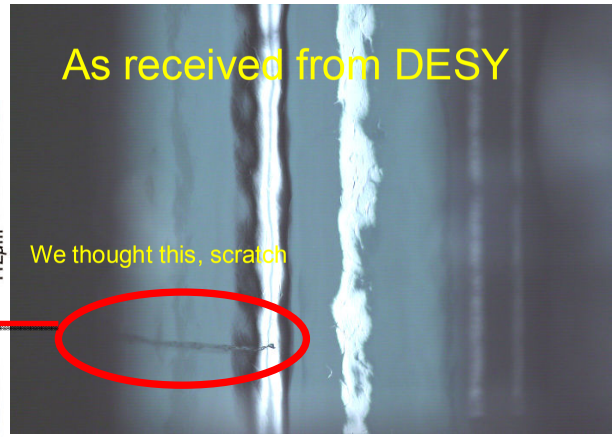




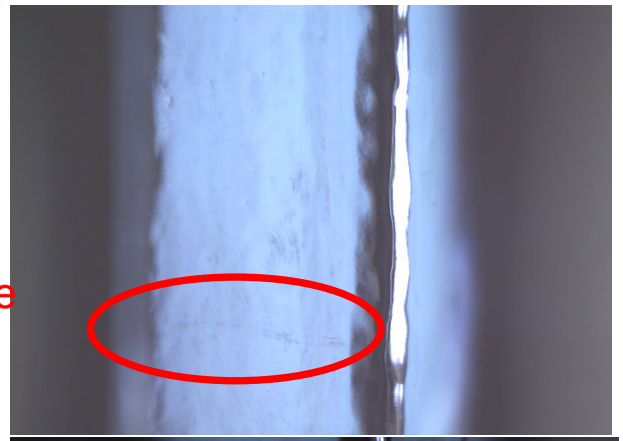


Local grinding for AC71 #1-#2 Iris

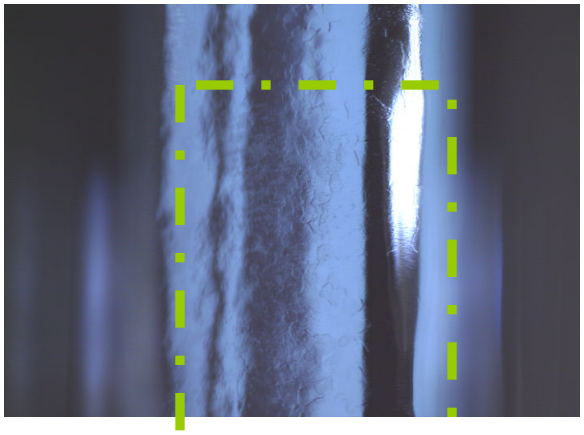


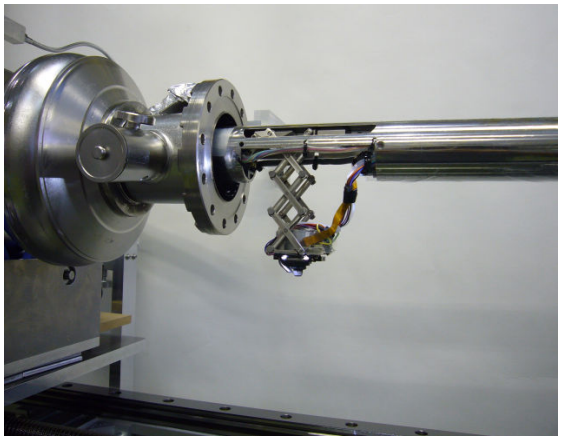


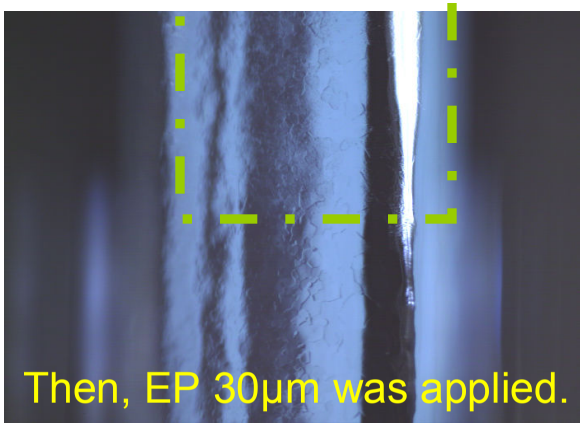
AC71 was local sponge wipe with ethanol,
then, mechanical grinding,
then, sponge wipe and HPR,
then, EP 30μm, ultrasonic UPW rinse, HPR
drying in clean room, inspection,
Then, sent back to DESY.



before grinding, we applied sponge wipe with ethanol. Then it went away. Still there is some trace.



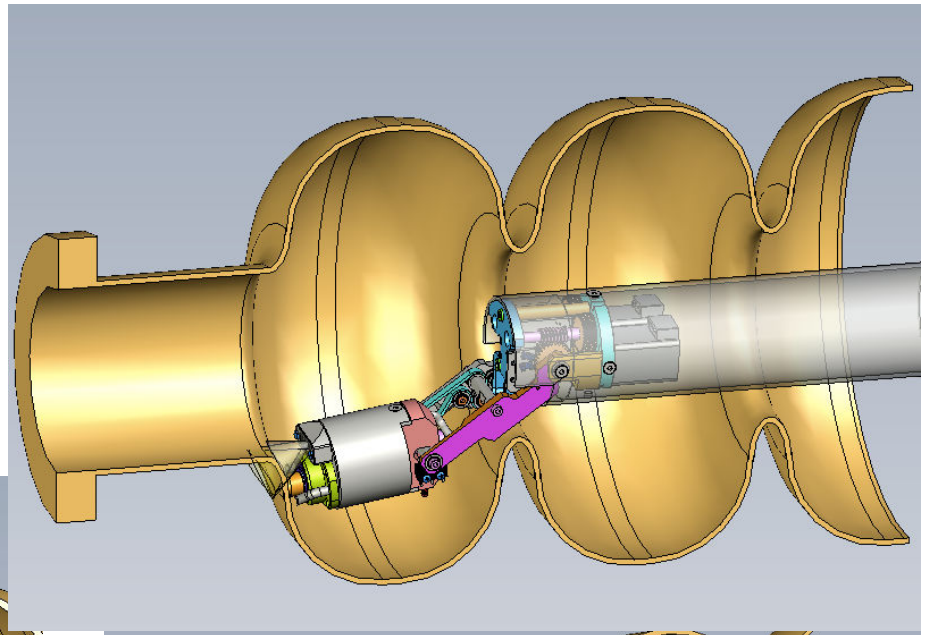


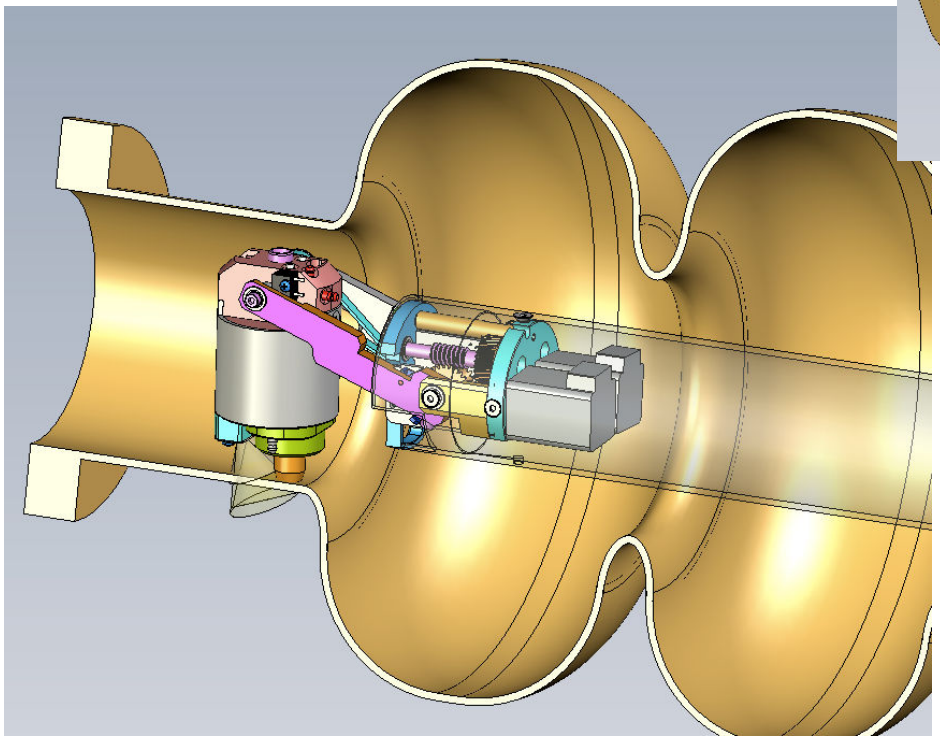


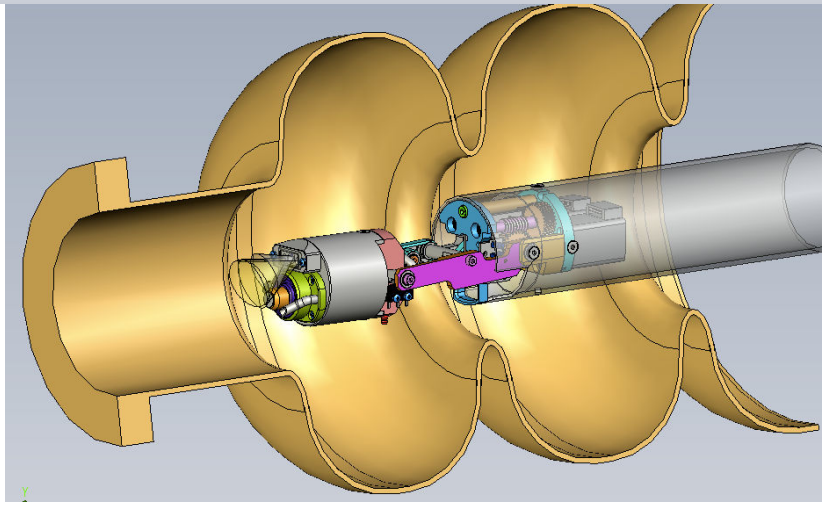
We could get smooth surface.

Next version of local grinder

for flexibility of angle of grinder surface,
easy to adjust grinder head to cavity surface







Now under fabrication.

Summary of High Gradient basic R&D in STF

1. Electro-chemical Polishing Facility in STF

Commissioned in last summer, 9 cell cavity process is in routine.

1. Surface Analysis of EP'd Nb
2. Sulfur removal
3. Sponge wipe

First round of analysis is under-going.
Caught Sulfur is utilized for rinse method comparison.
Sponge wipe is proposed to remove contaminations.
Its mechanism is under development.

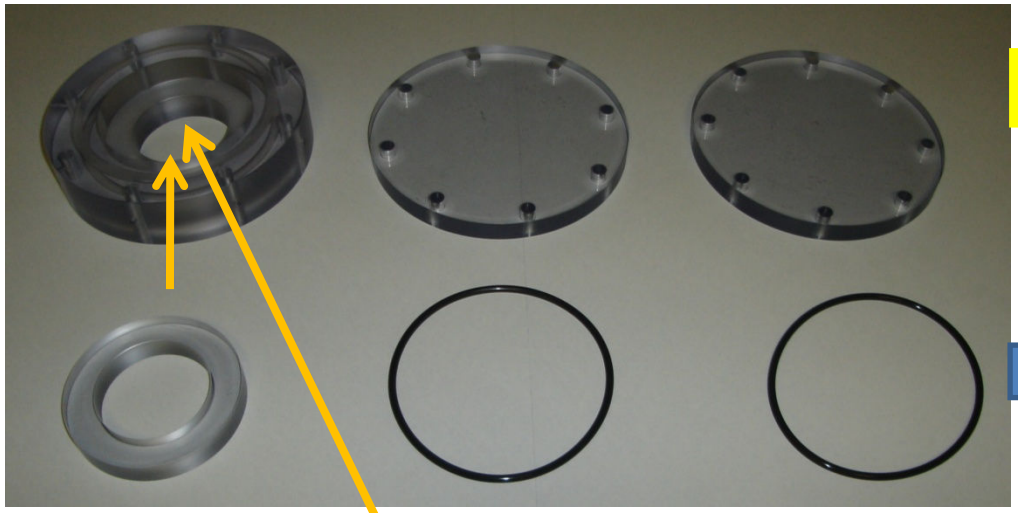
1. Inspection Camera
2. Local grinding

Inspection camera was demonstrated its capability of defect catch.
For more performance, the camera is under upgrading.
Local grinding is proposed to remove defects in the cavity.

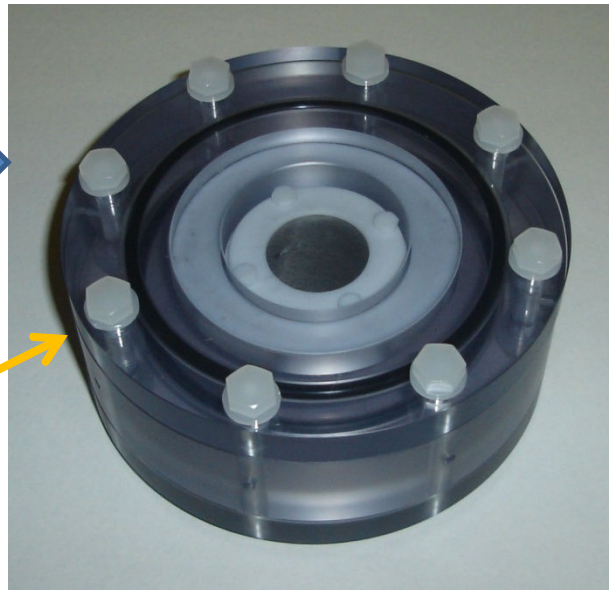
end

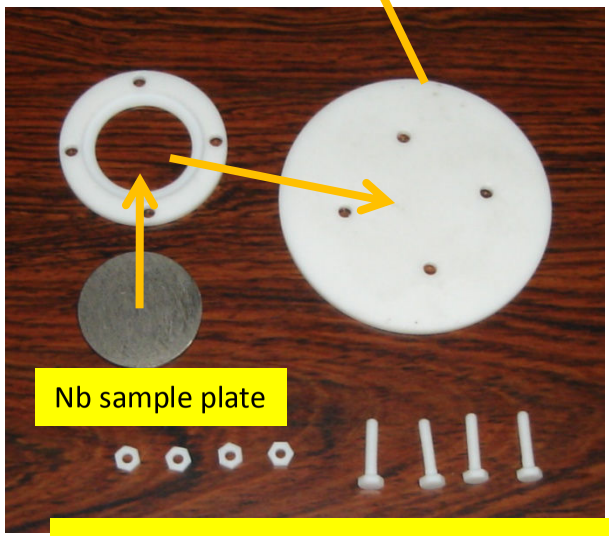
Backup slides

UPW filled container for transportation



transparent PVC container





Nb sample and Teflon parts

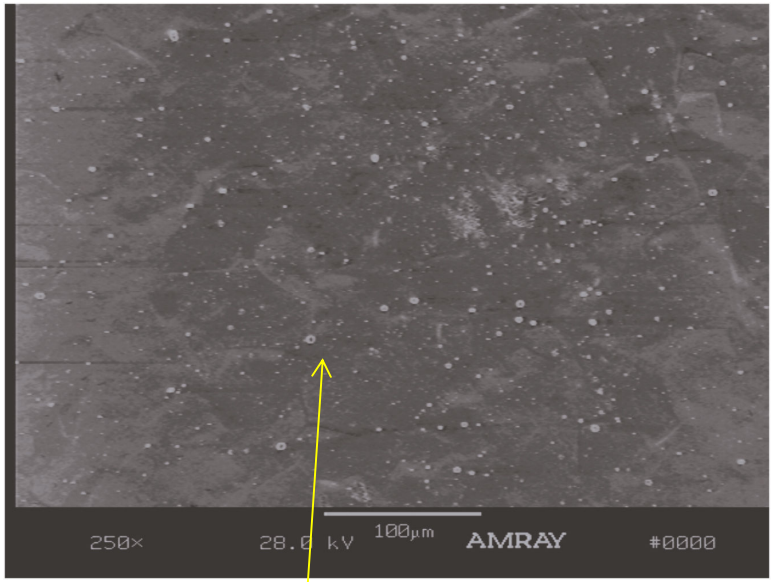
PVD bolts

Use of UPW container is intended for long distance transport like JPN-US

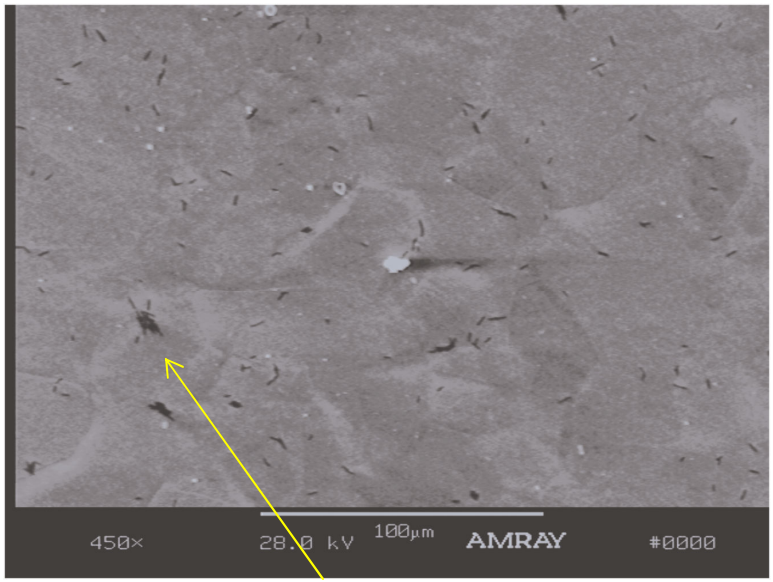
JLab sample

SEM@JLab

Sample#1(just EP'd)

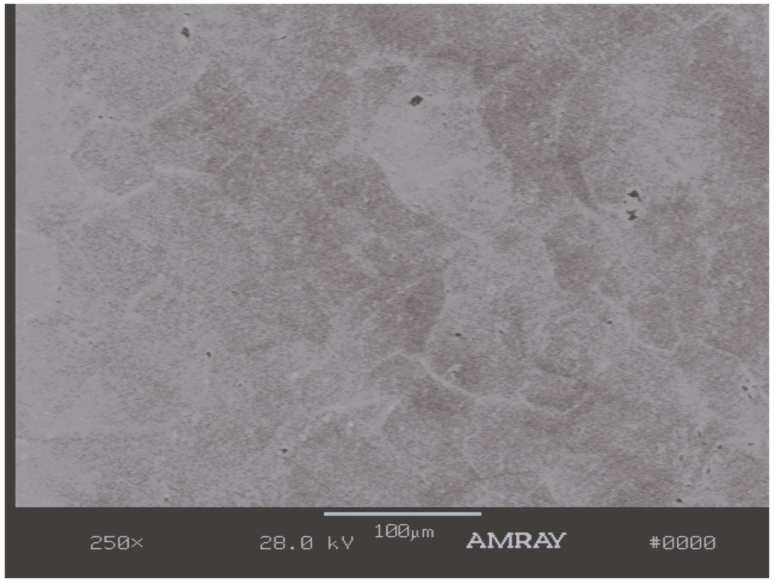


Lots of Nb-O particles (white)

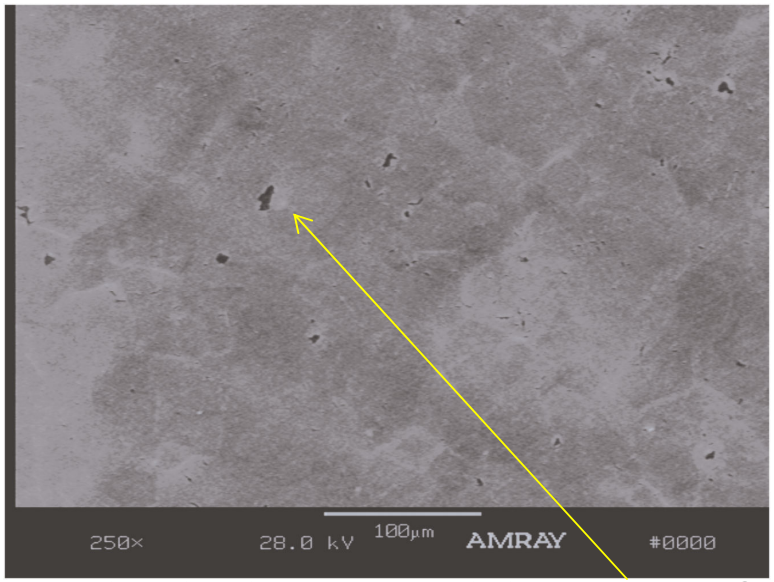


Suspected bacteria (black worm)

Sample#2 (EP + sponge wipe)

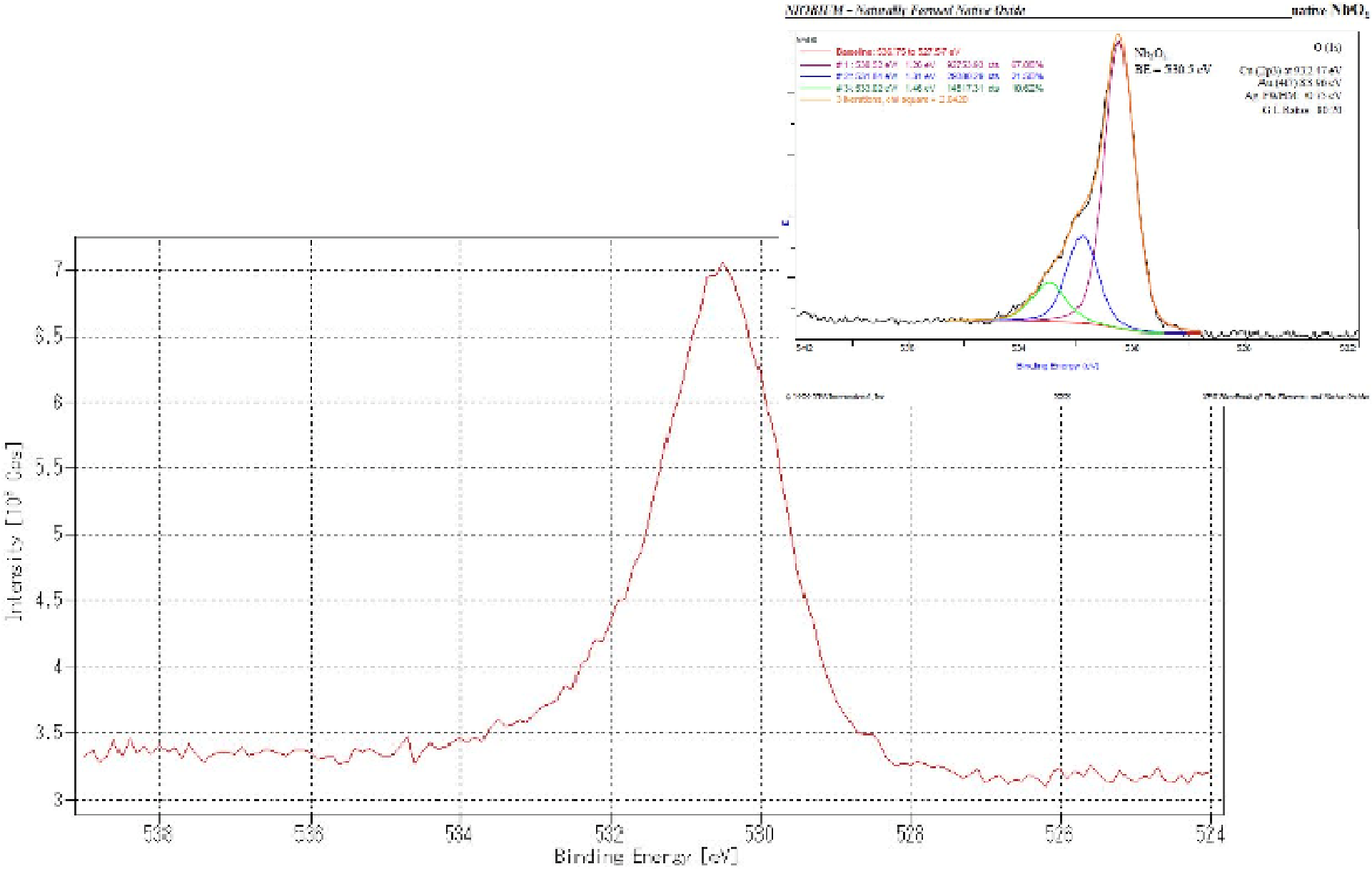


Significantly reduced Nb-O particles

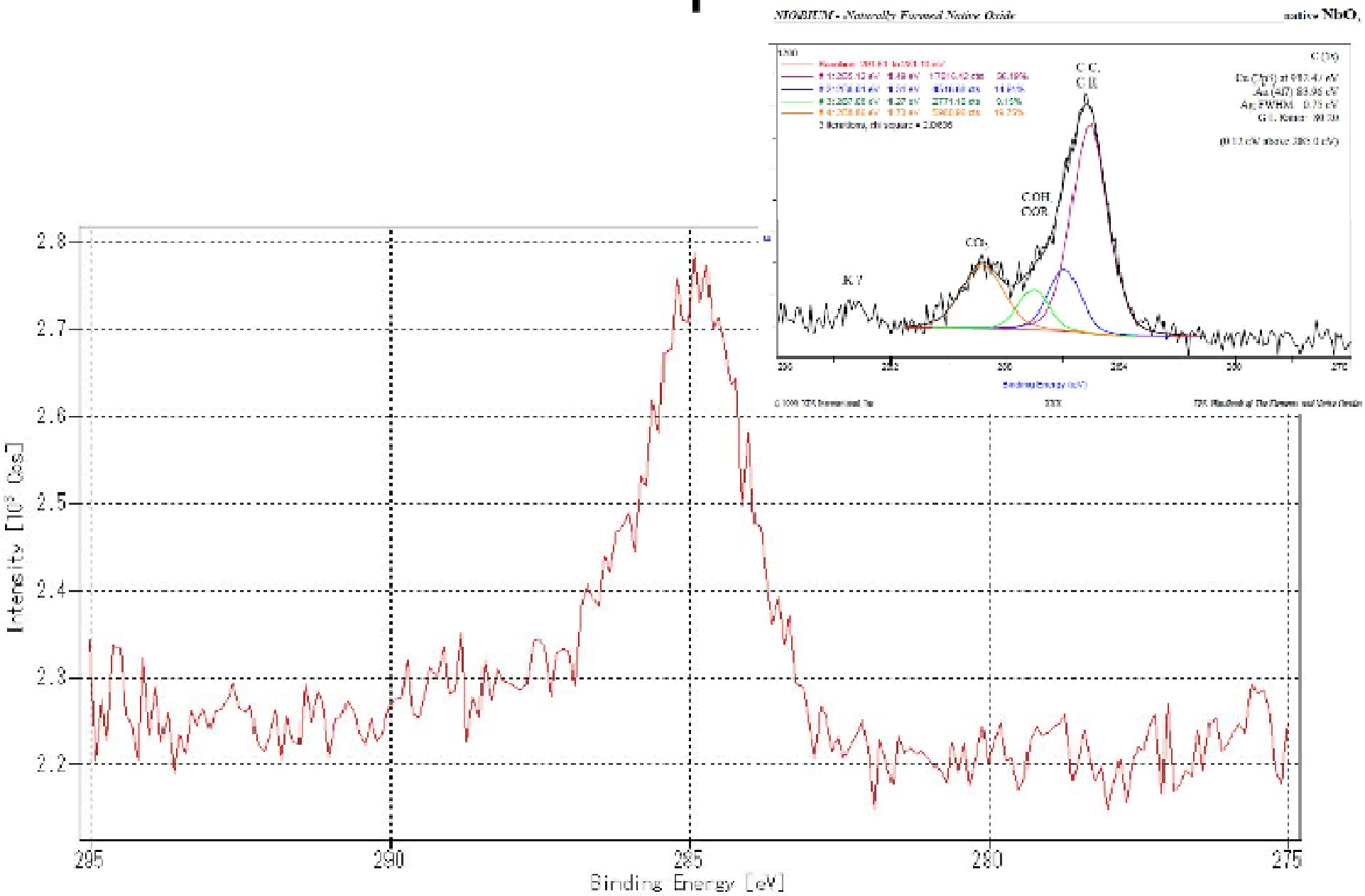


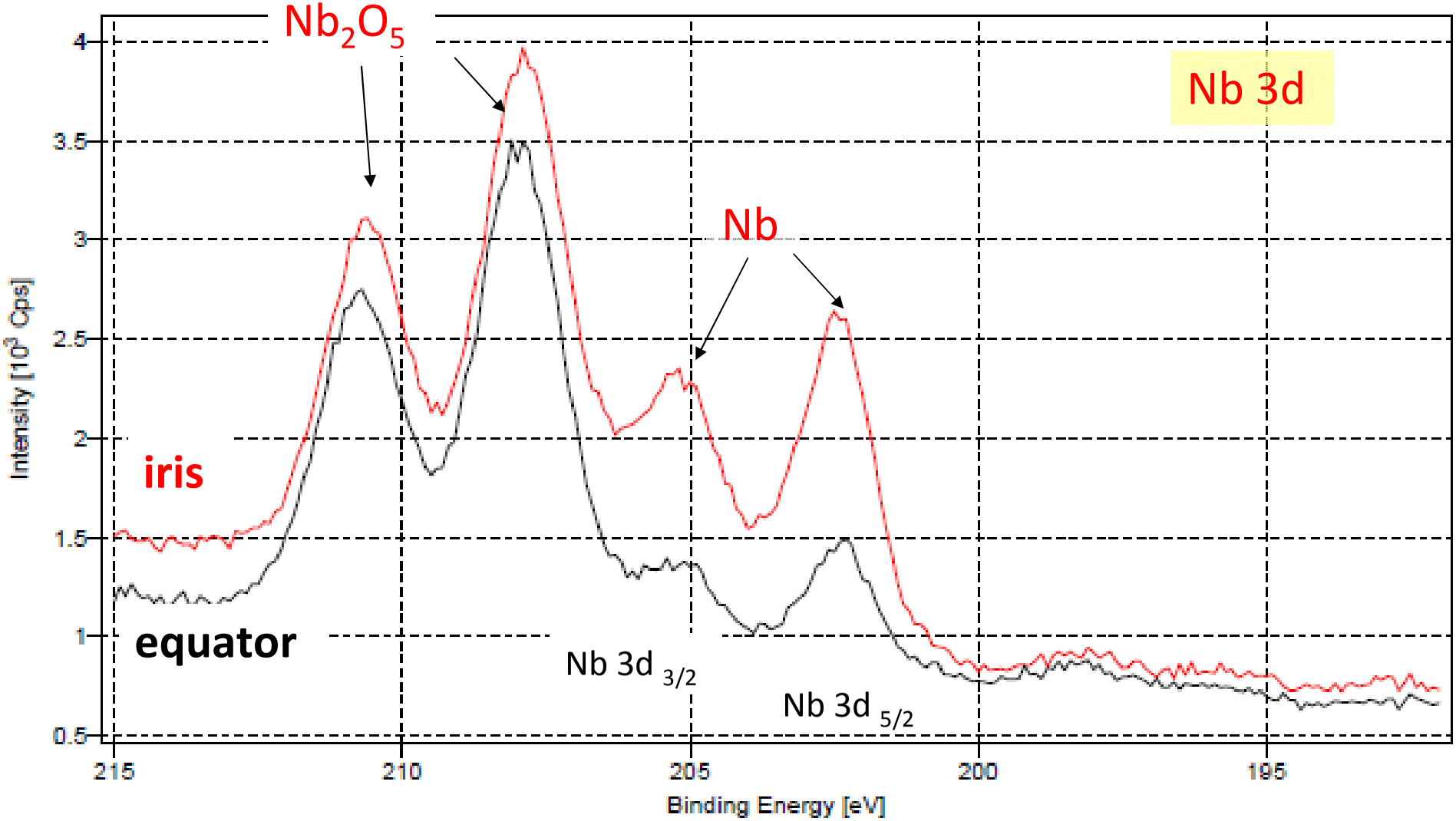
Somehow less bacteria growth, but new type of Contamination (black particulate)

Nb-1 XPS spectrum of O

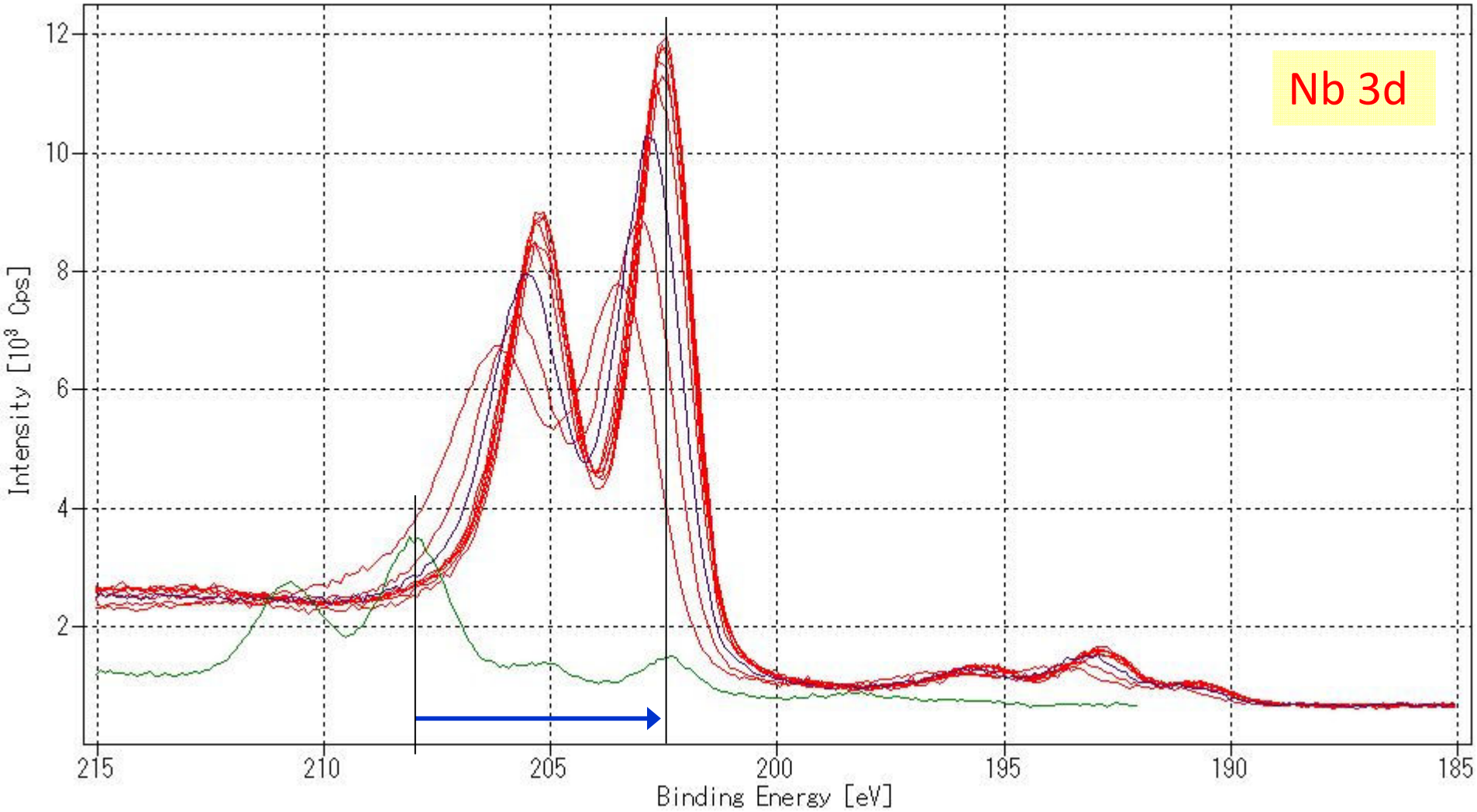


Nb-1 XPS spectrum of C





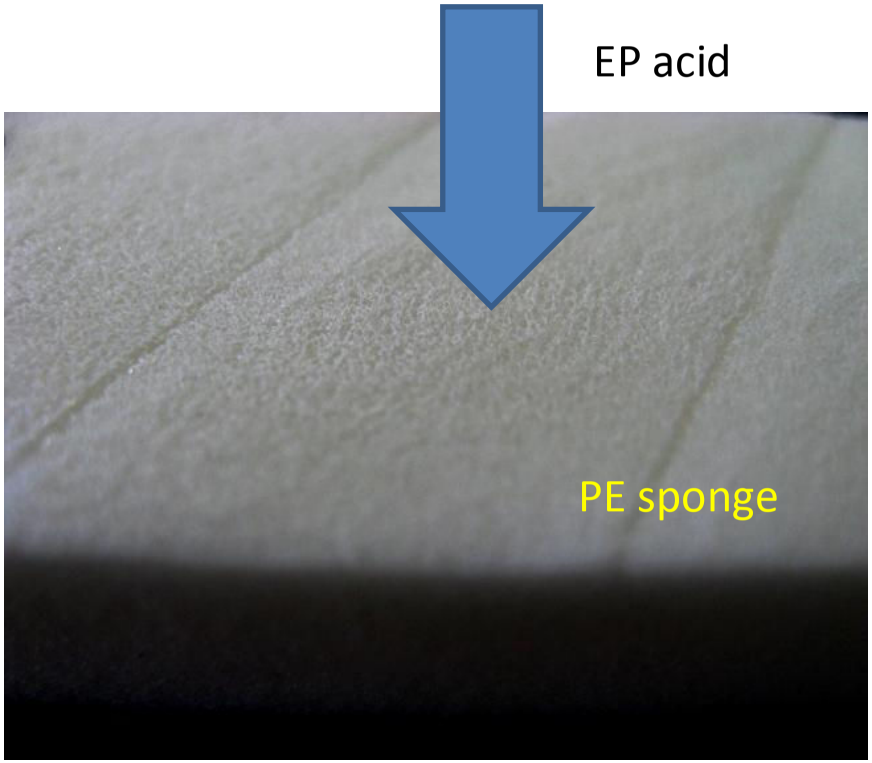
Iris と equatorで、酸化ニオブと金属ニオブの比率が違う



酸化二オブから金属二オブへ

Poly-Ethylene (PE) sponge test with EP acid

- **Almost no corrosion of PE sponge by the EP acid.**

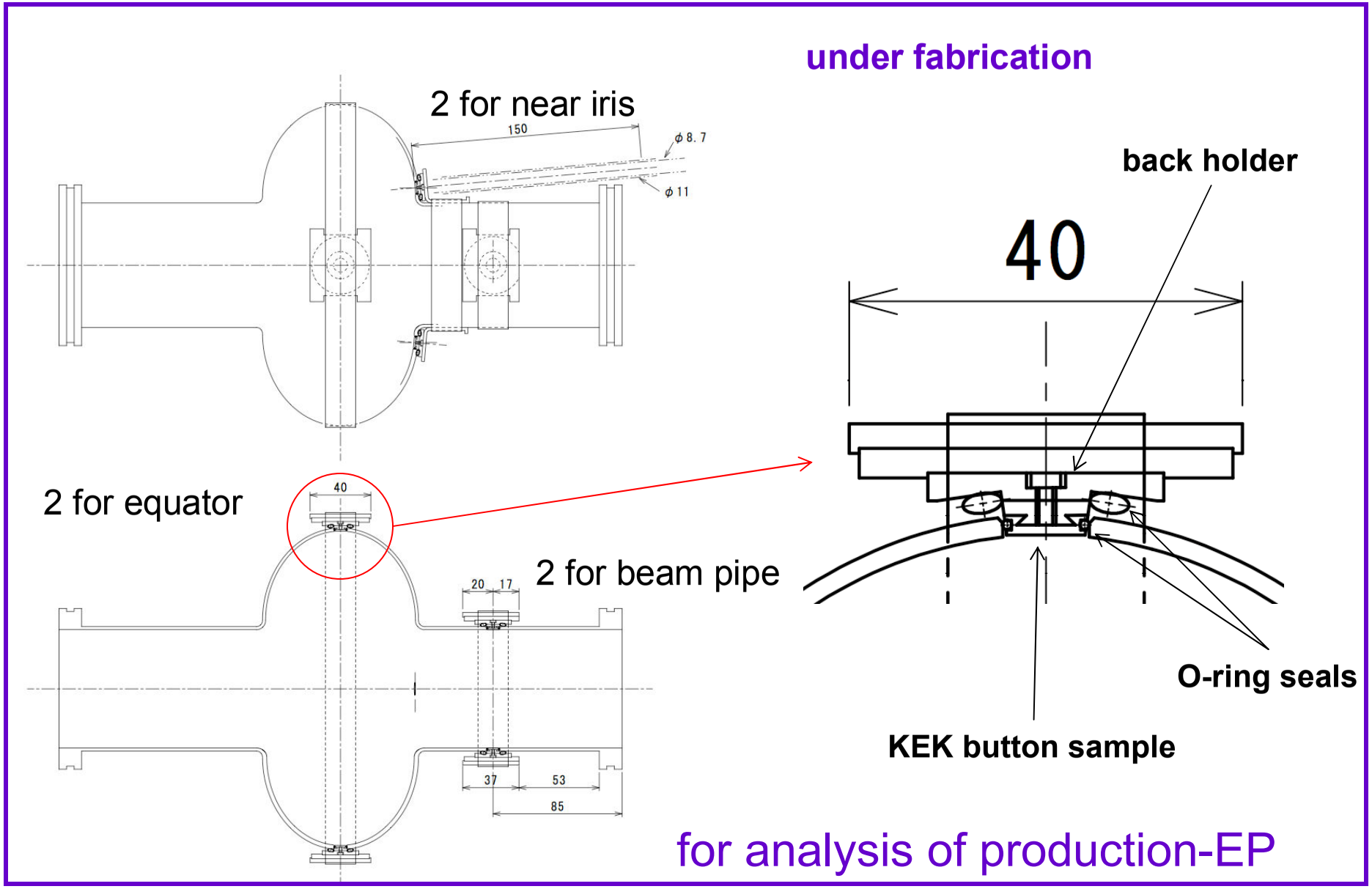


October 2008

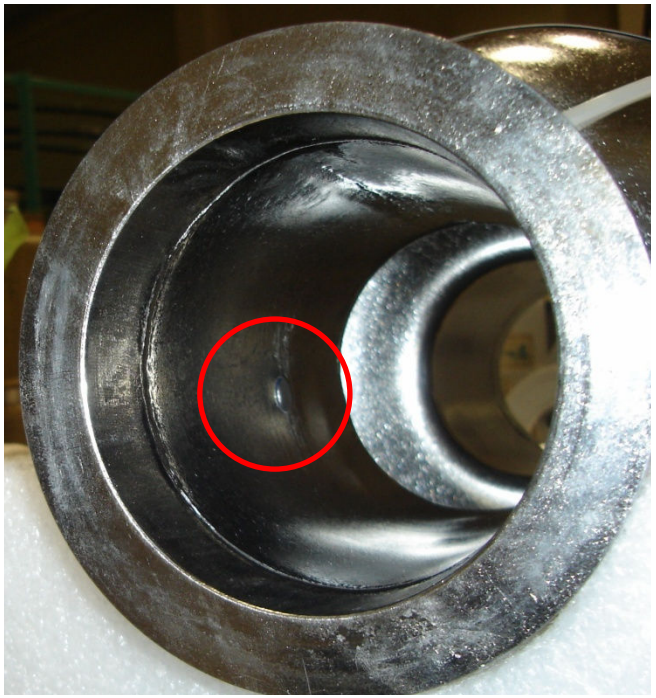
No corrosion after dropping EP acid on PE sponge.

Let's made a sponge-cleaning tool for single-cell cavity with PE sponge.

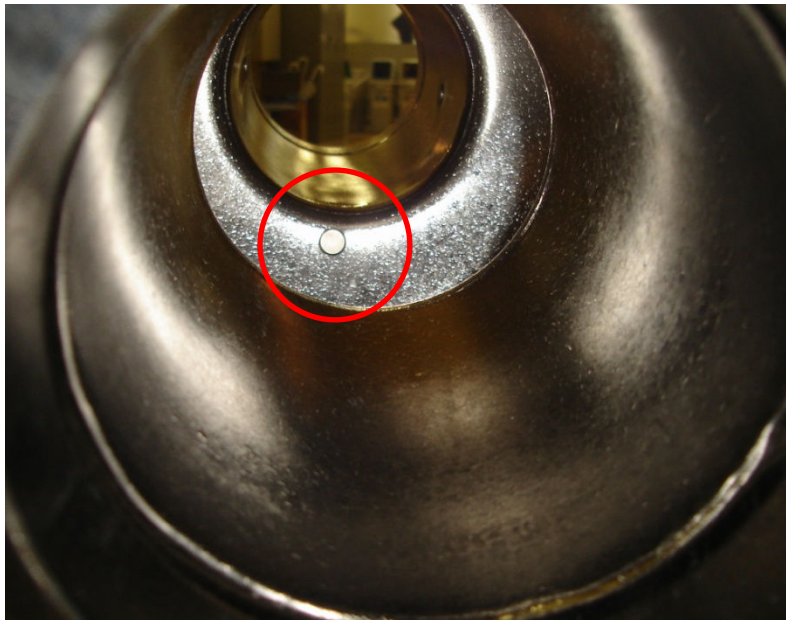
Analysis of EP'd surface by using single-cell cavity with de-toutchable button Nb-samples



Special single-cell cavity for sample-study



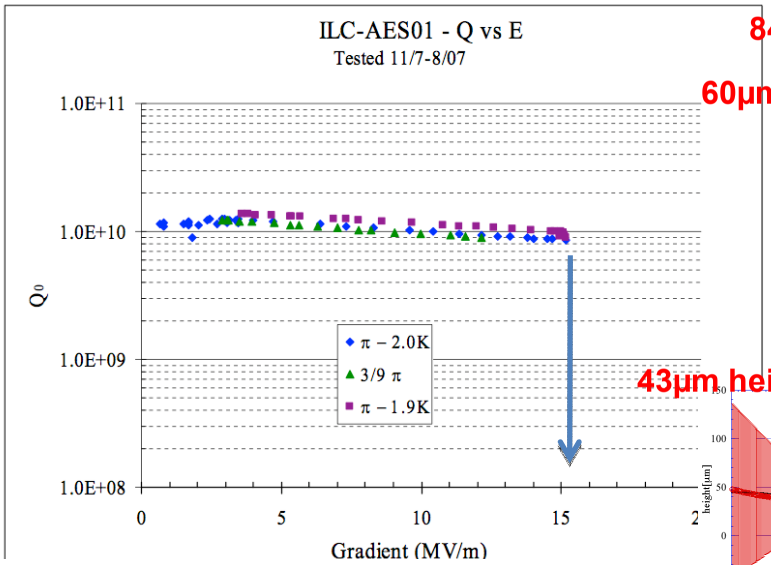
Nb-sample at beam-pipe



Nb-sample at iris

Inspection of AES01 (FNAL)

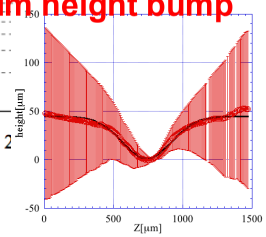
3rd Test Results



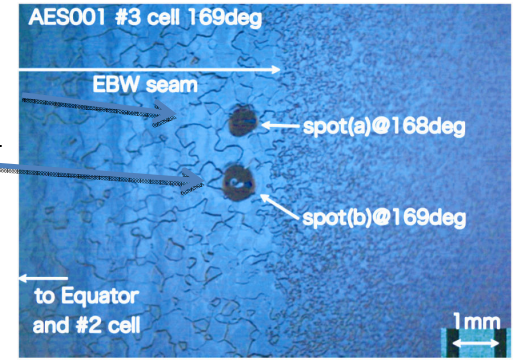
84μm height bump

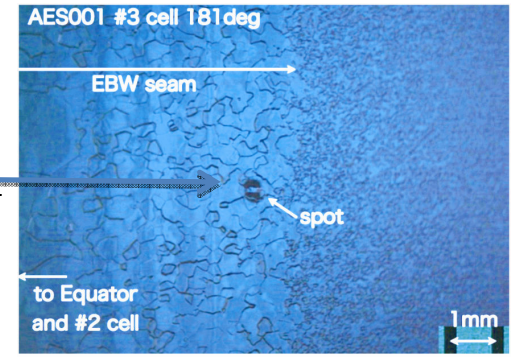
60μm height bump

43μm height bump

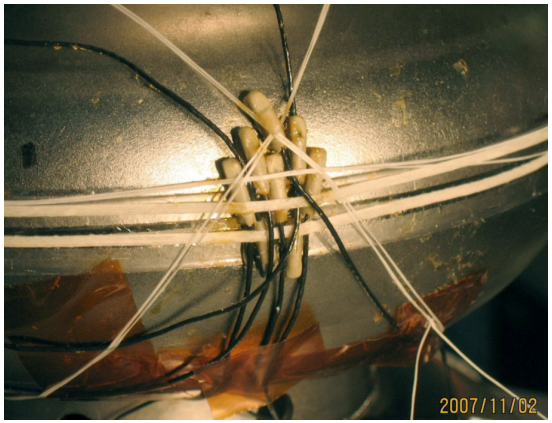


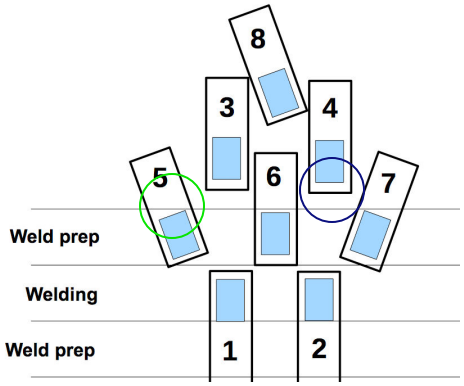
~21mm

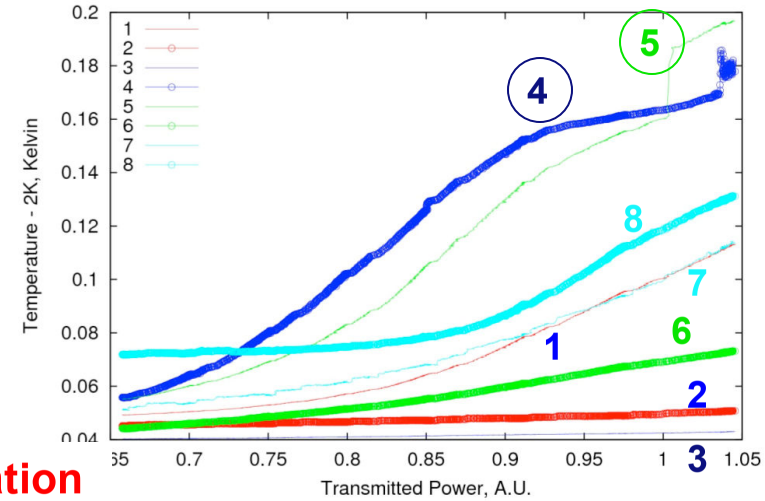




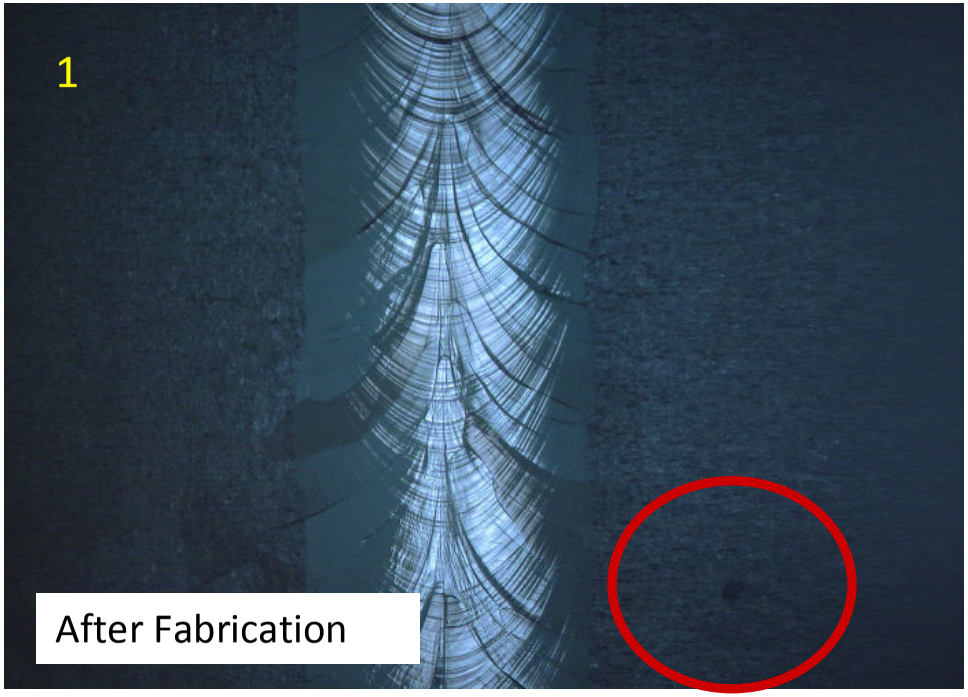
AES01 has hard quench at 15MV/m, its location was identified by Cernox at FNAL.





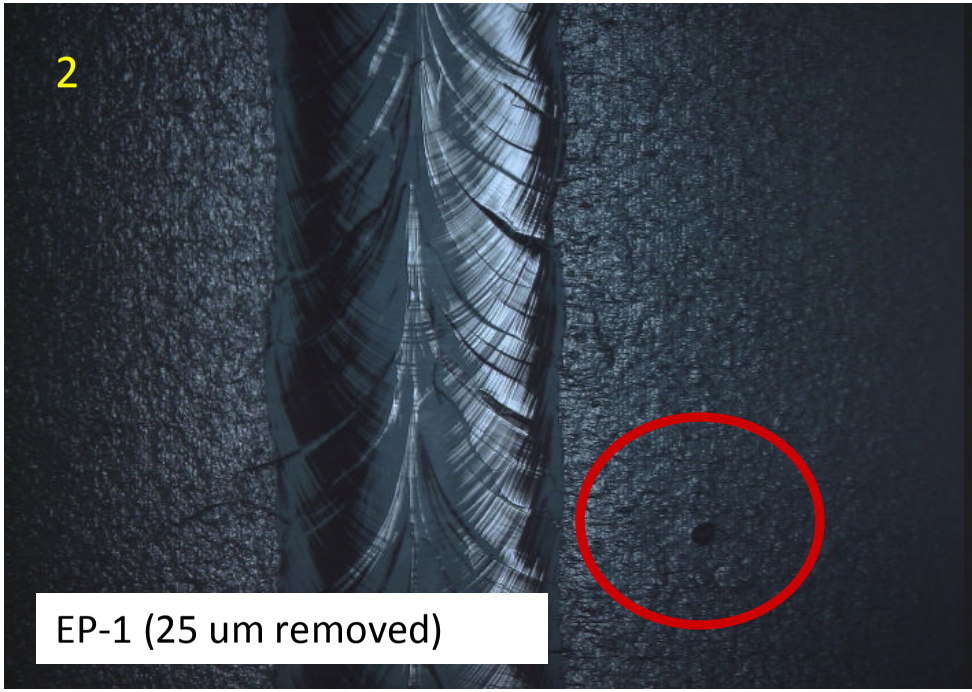


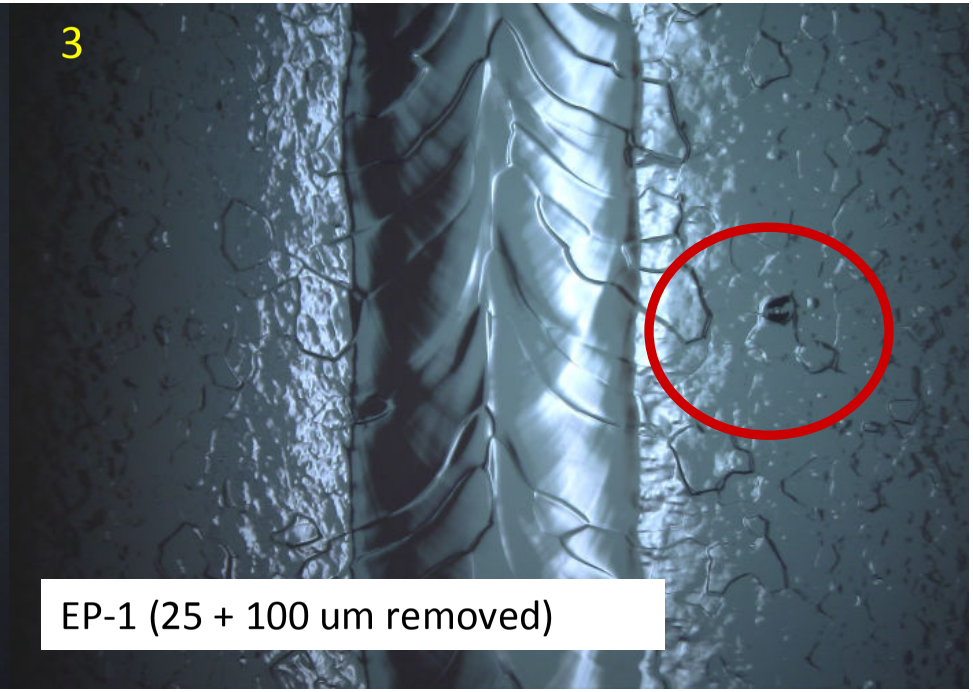
Kyoto-camera found 3 spots in their exact location

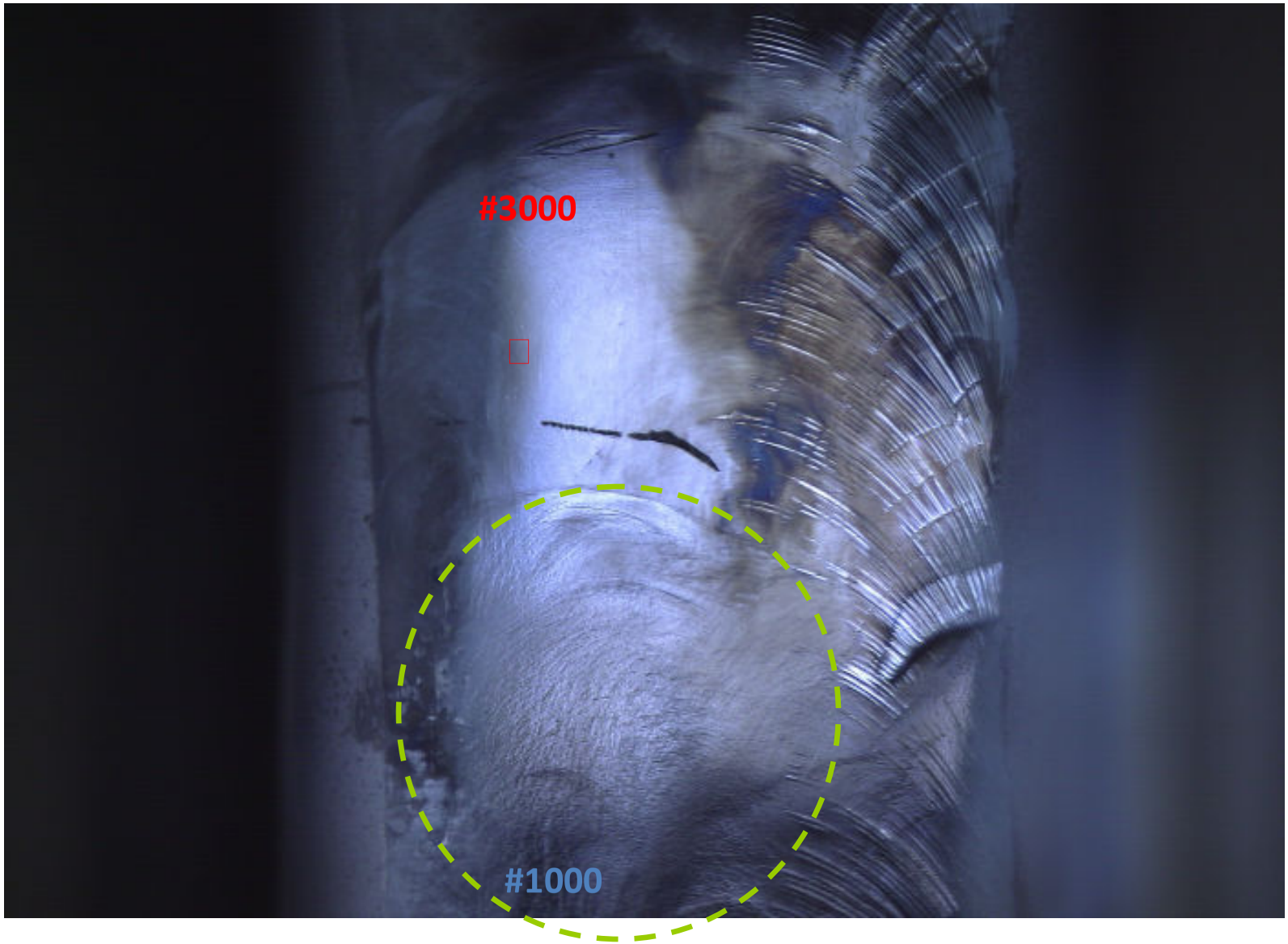


Comparison of each EP treatment
#3 cell equator, Z=402mm, t=10 deg

Pit exist from the beginning, and not disappear

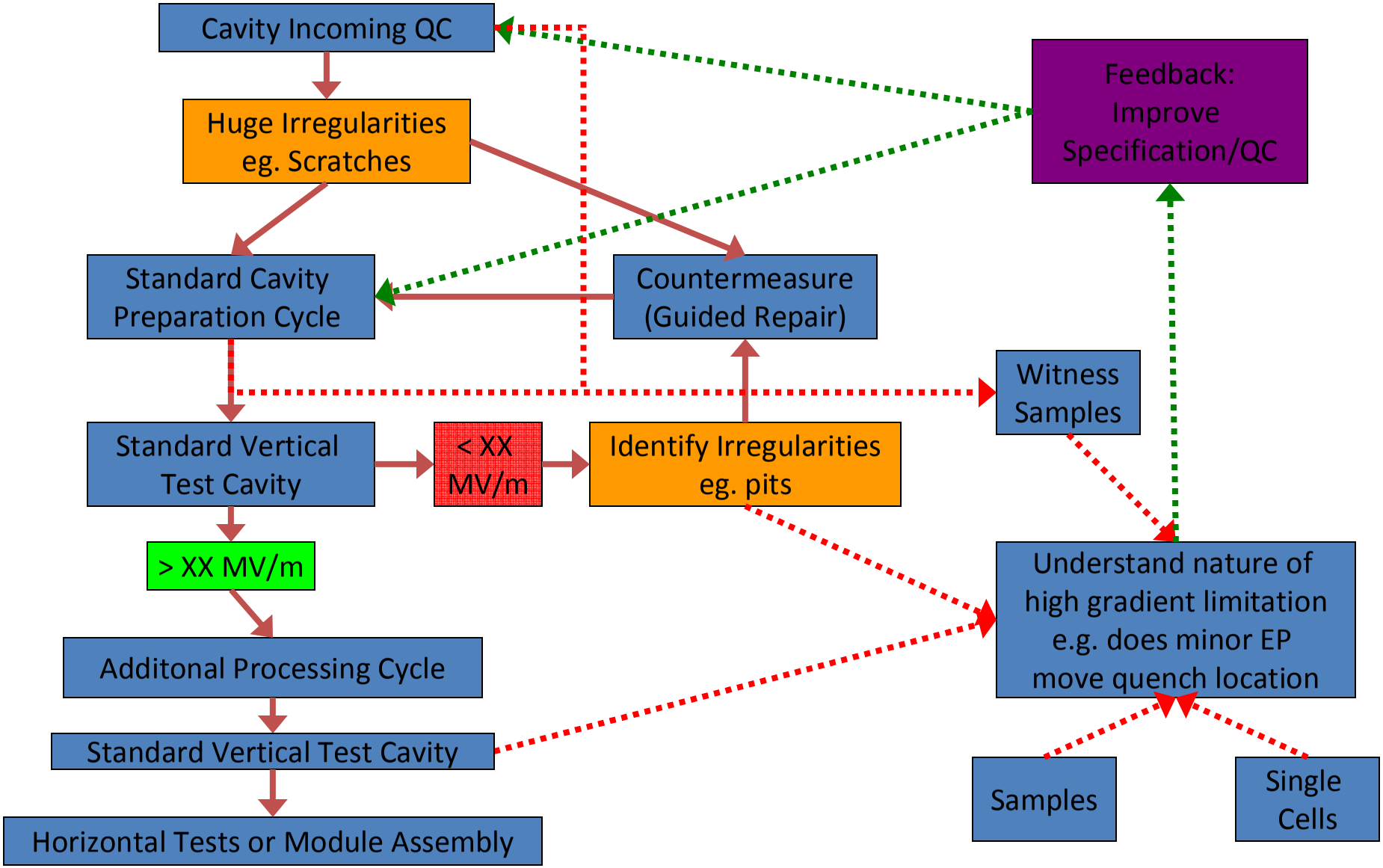






Additional grind by #1000 diamond sheet for 2-5min in 4 locations

ILC S0 Feedback Loop



4.4.2008 Lutz Lilje
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