

Surface study at KEK

- 1) sponge cleaning
- 2) special single-cell cavity with de-toutchable Nb-samples

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on behalf of Jlab-FNAL-KEK S0 study collaboration

LCWS08 at Chicago

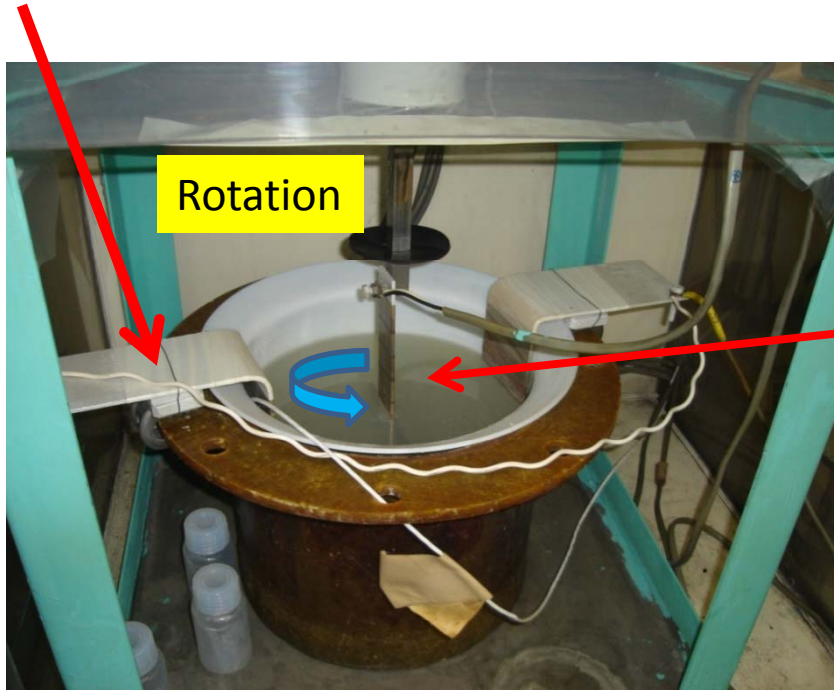
17 Nov. 2008

Sponge cleaning study with Nb samples

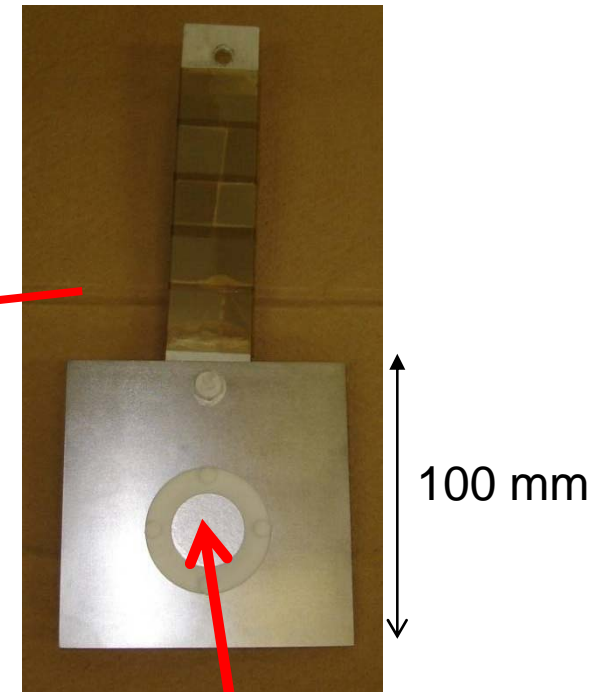
- HPR + ethanol rinse / degreaser were introduced recently because HPR-only seems not enough to eliminate FE. And quality control of HPR is still very difficult. Usage of high-pressure water (~10 Mpa), UPW quality controletc.
- Direct physical/mechanical **sponge wipe** can improve the performance? Even shorten the duration of HPR / reduce the load of HPR?
- 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 cleaning**.
- Compare sample-1 and sample-2 by **FS-SEM** at Jlab.
- Compare sample-1' and sample-2' by **XPS** at KEK.

EP(70 μm) of Nb samples at Nomura Plating

Al cathode



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



Jlab-type Nb sample

$V = 8 \sim 13$ (V), $I = 3 \sim 23$ (A) with oscillation, $T = 25 - 50$ $^{\circ}\text{C}$ w/o T control.

[Nb] in EP acid = $4.0 \sim 6.2$ g/L during the EP of two samples.

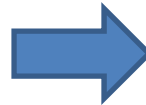
We expected sulfur contamination by aged EP acid.

U.P.W. rinse and sponge cleaning

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



Ultra Pure Water rinse (20 min)

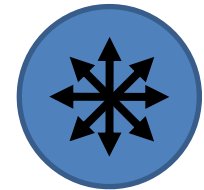


sponge cleaning with U.P.W.



+ Ultra Pure Water rinse (5 min)

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

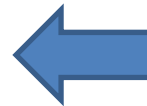


Nb-sample-2



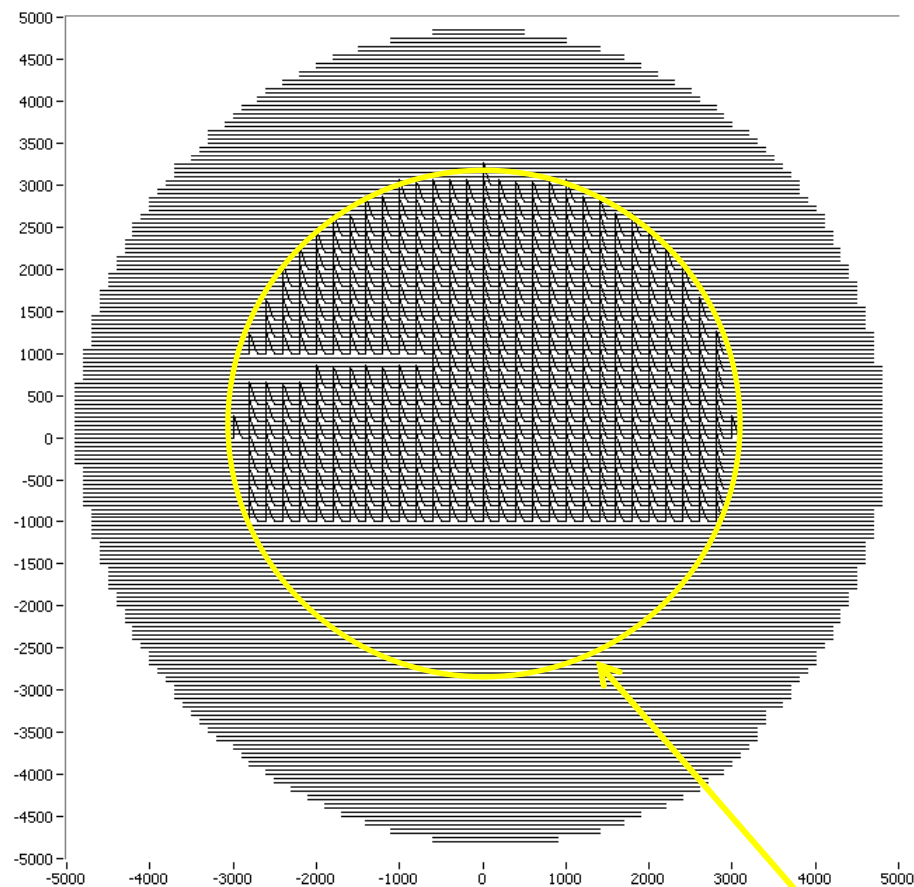
Packing in transportation container

Nb-sample-1



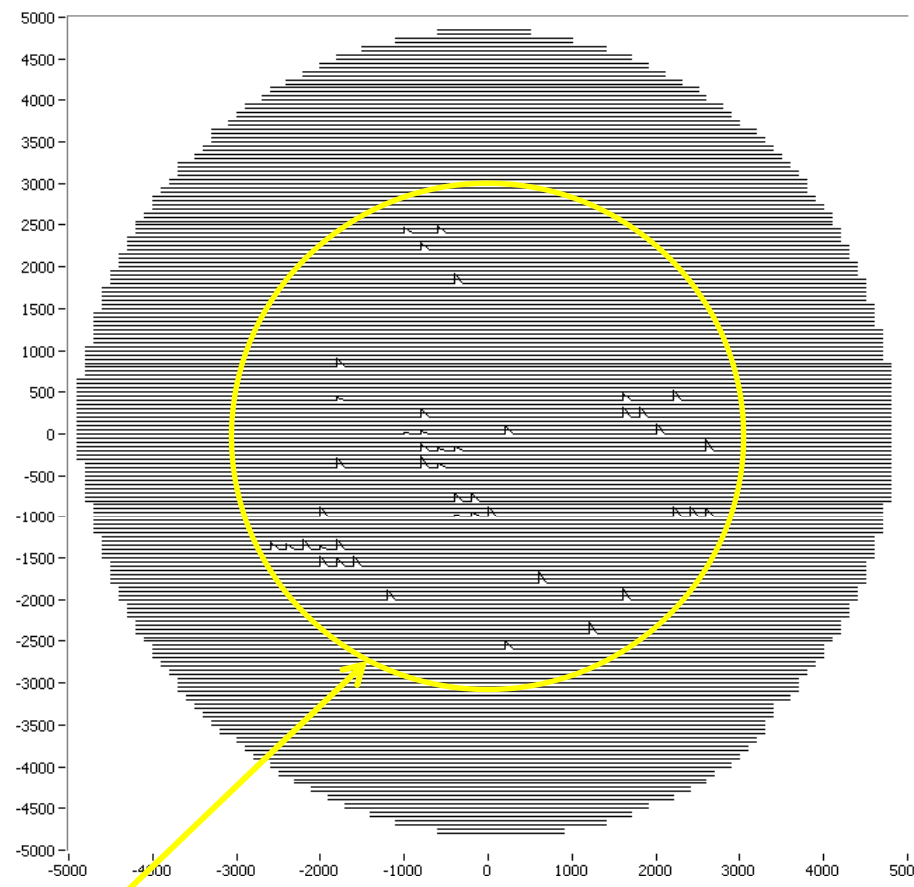
Packing under U.P.W.

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



Nb-sample-1 w/o sponge-cleaning

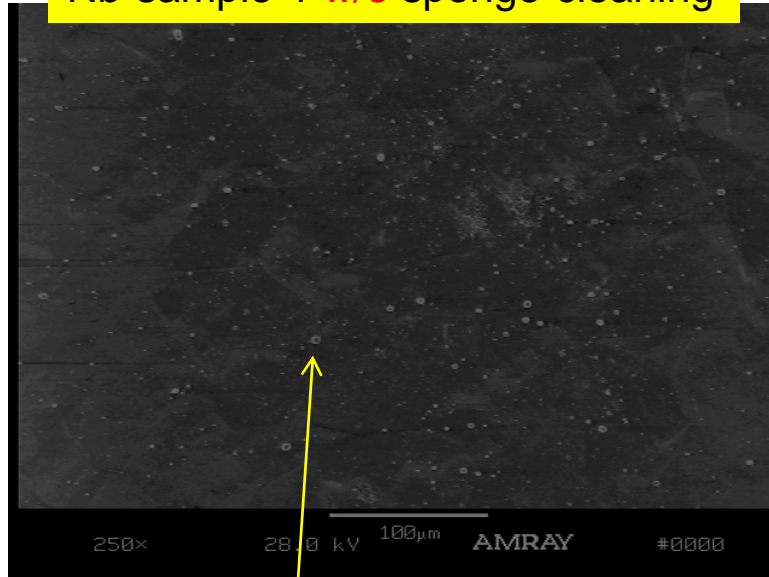
Sample#2: much less field emitters
Higher FE onset



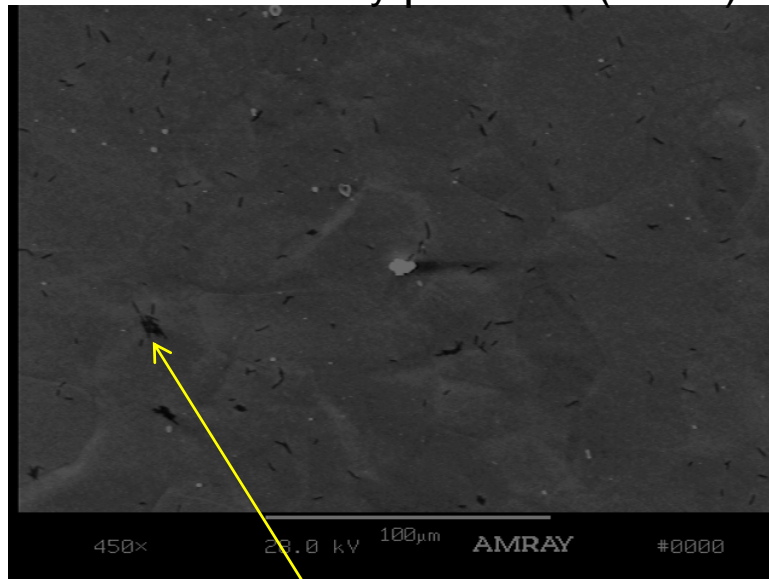
Nb-sample-2 w/ sponge-cleaning

Area inside circle is scanned by FS-SEM.

Nb-sample-1 w/o sponge-cleaning

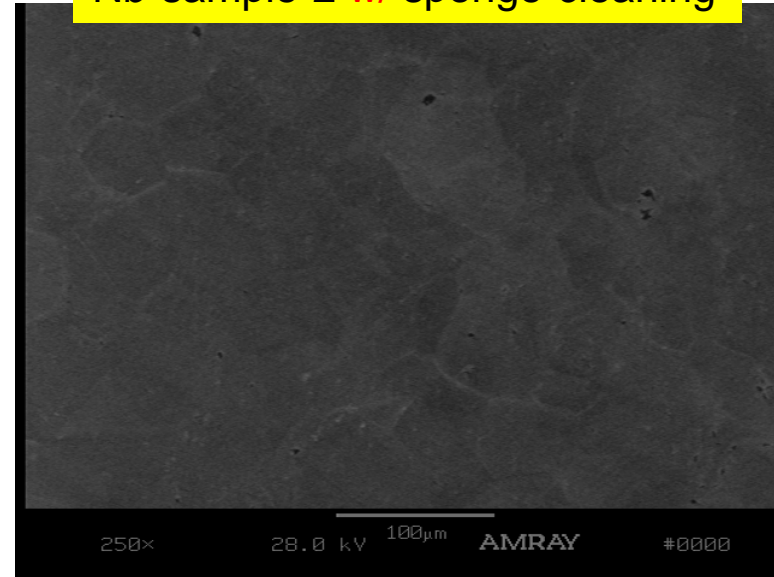


Lots of Nb_xO_y particles (white)

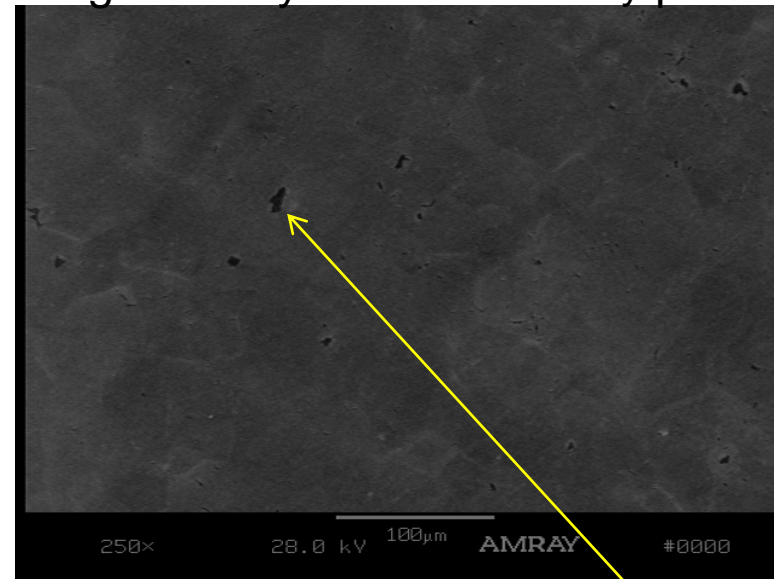


Suspected bacteria (black worm)

Nb-sample-2 w/ sponge-cleaning

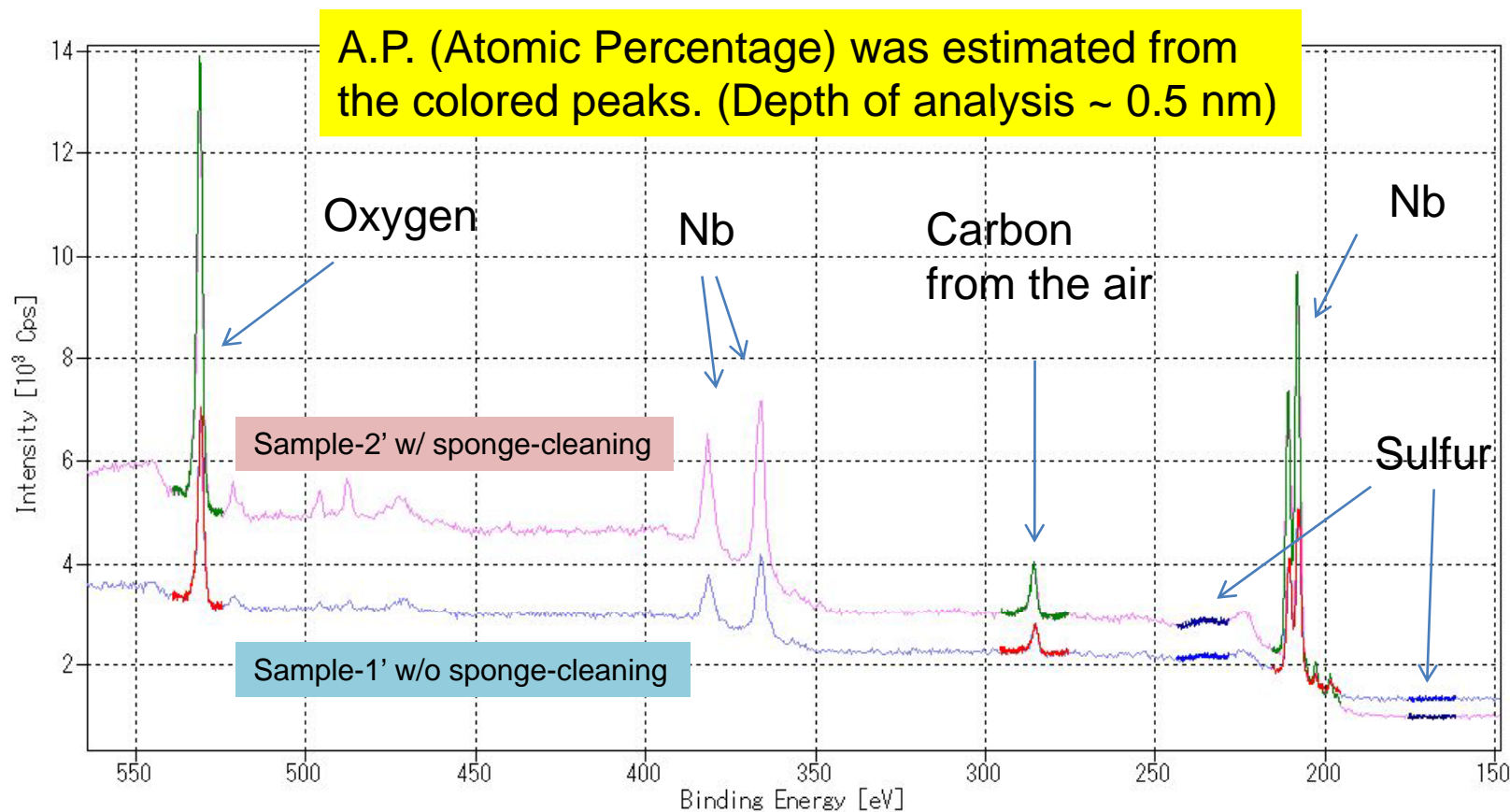


Significantly reduced Nb_xO_y particles



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

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

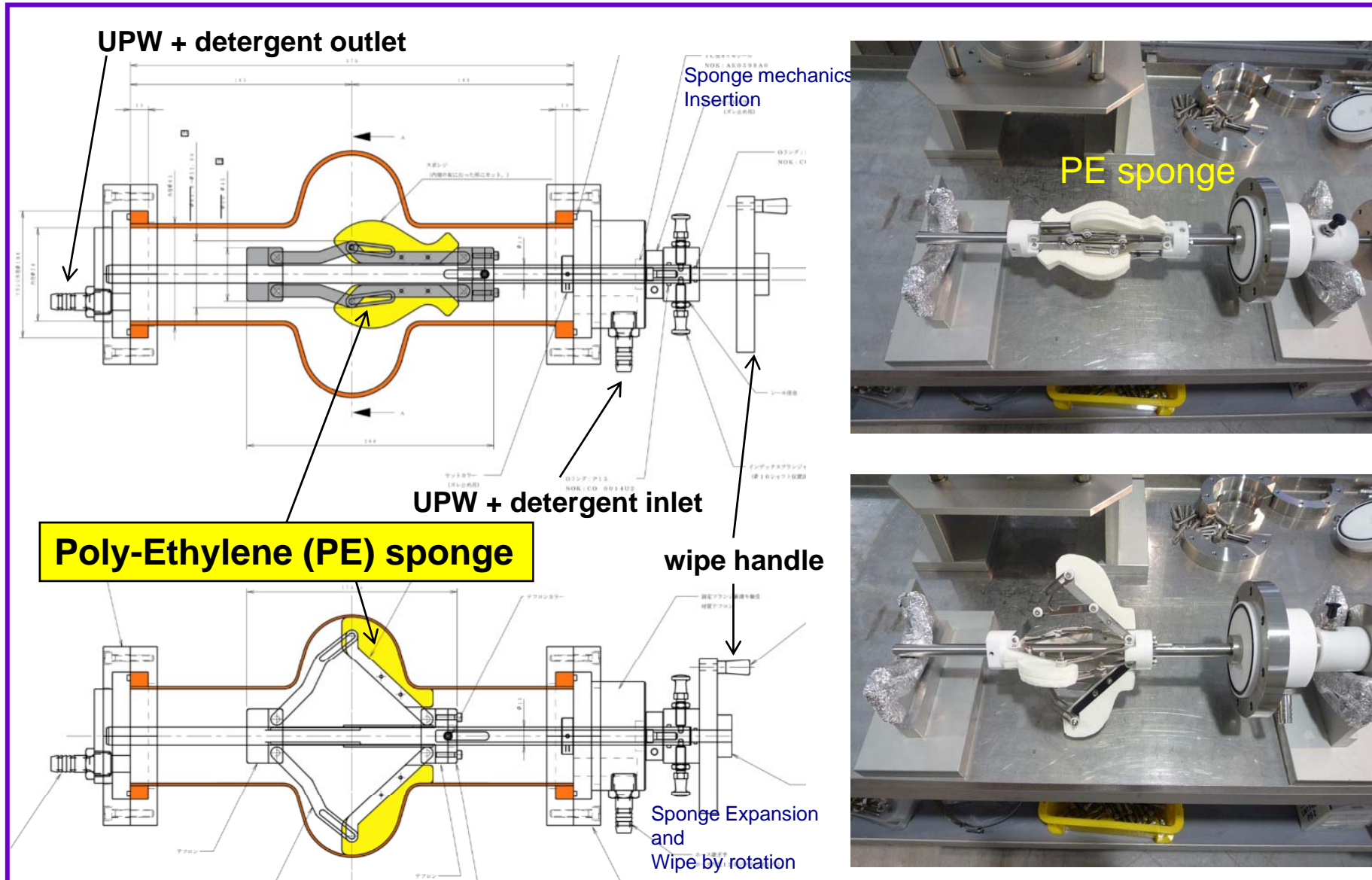


A.P. for sample-2' w/ sponge-cleaning
= Nb:20%, O:57%, C:23%

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

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

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

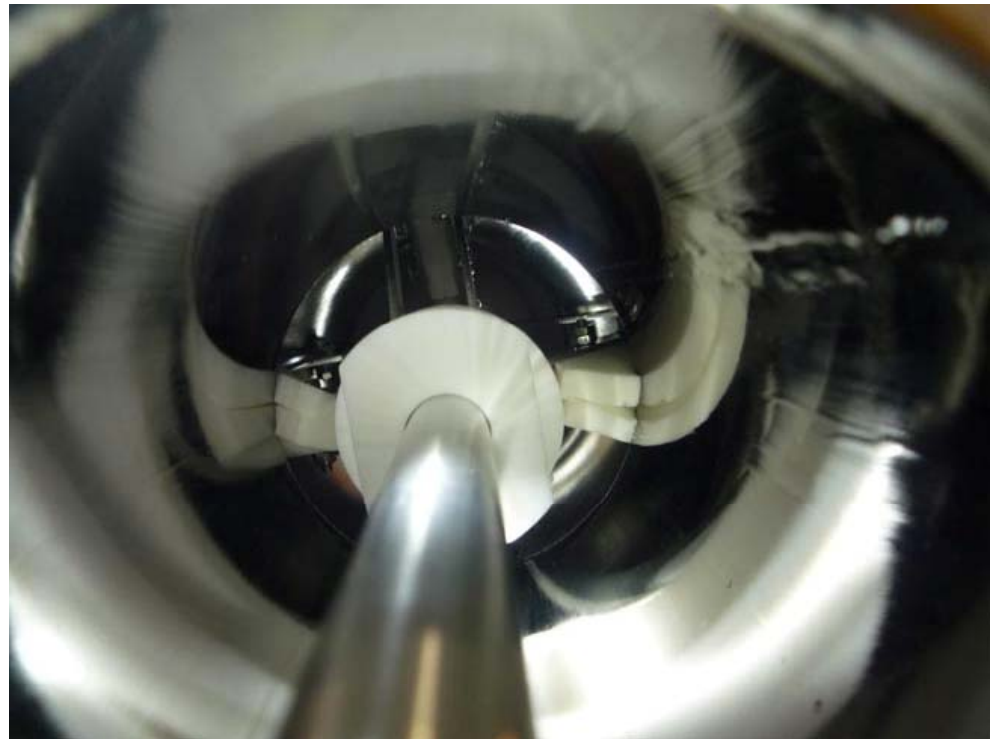


The single-cell cavity was EP'ed and UPW-rinsed before the sponge-cleaning test.

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.

Analysis methods

- Filtering drained UPW (10L) through the filter-paper of 0.20 μ m
→ Condensation of filtered UPW (10L) to 70 ml.
→ Ion Chromatography (IC) analysis for filtered /condensed UPW (70 ml).
- Residuals on the filter-paper → Optical Microscope → Component-analysis by X-ray fluorescence (XRF) analyzer



Optical Microscope

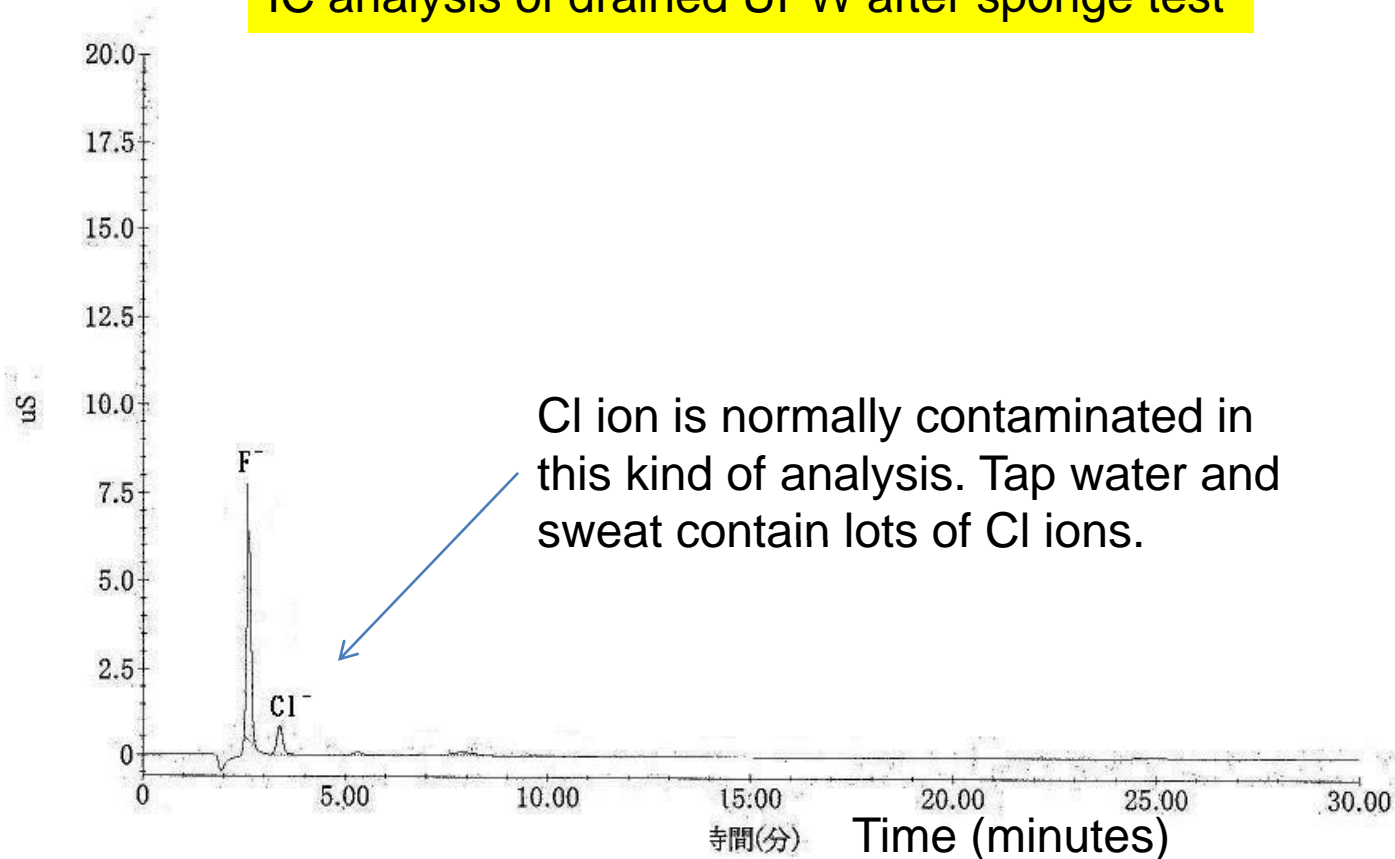


XRF

Result of IC analysis

- 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



Residuals on the filter-paper

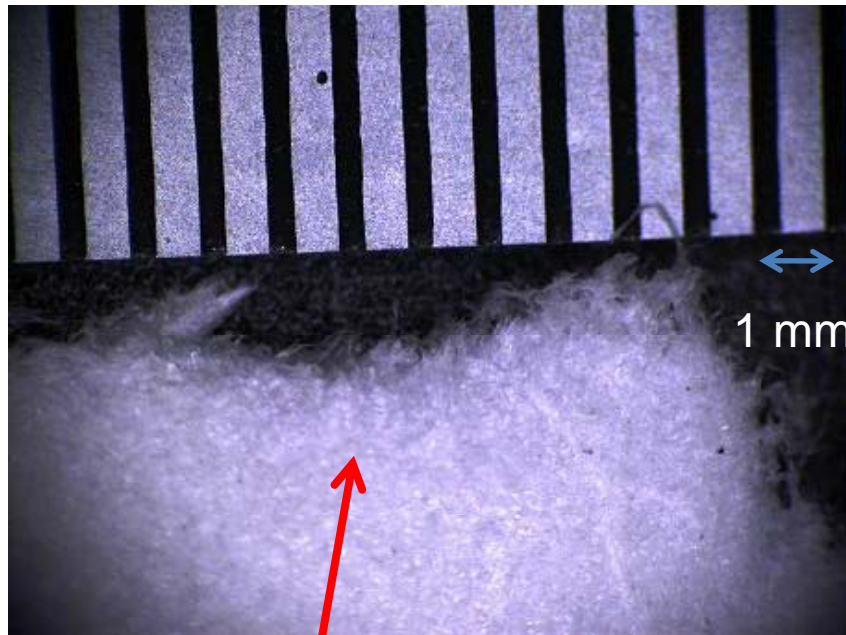


Residuals look like fibers/particles



1 mm

Poly-Ethylene (PE) Sponge



PE sponge fibers



Blue particles in PE sponge fibers.
We are not sure what these particles are, but vendors normally mix non-flammable materials in chemical products. Anyway, we need to ask the vendor what they are.

Results of XRF

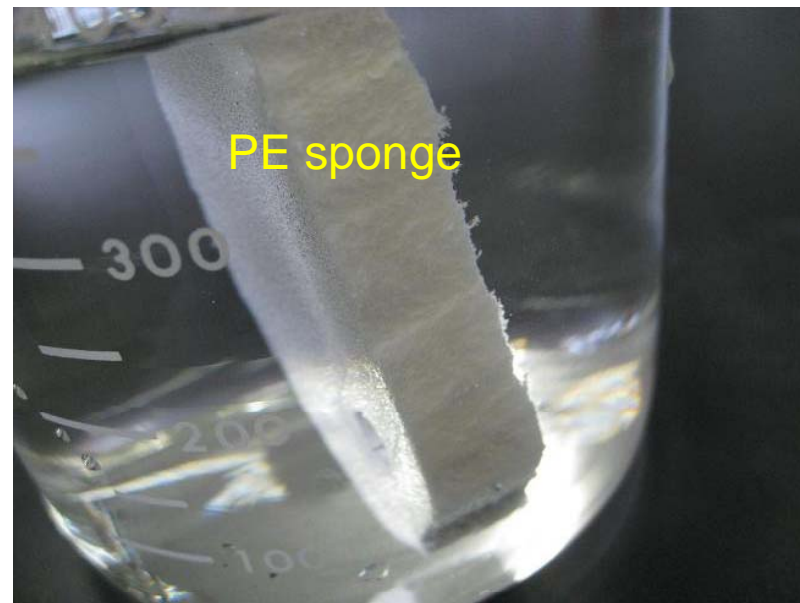
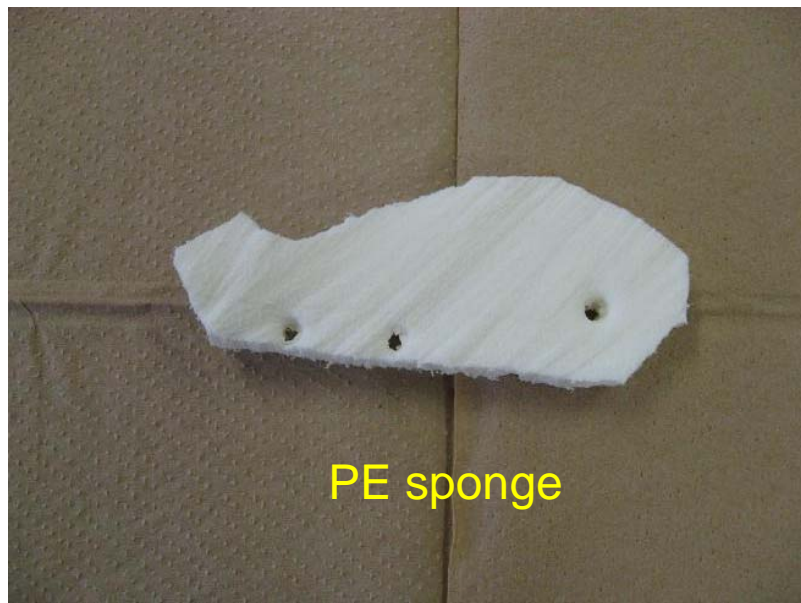
	Sponge test UPW (residuals on filter-paper)			Beaker sponge test UPW (residuals on filter-paper)			PE sponge (used in the sponge test)		
K α	Peak Position (d e g)	Peak intensity (k c p s)	Sample /Blank (Ratio)	Peak position (d e g)	Peak intensity (k c p s)	Sample /Blank (Ratio)	Peak position (d e g)	Peak intensity (k c p s)	Sample /Blank (Ratio)
Mo-K α	20.295	1.983	0.95				20.328	1.536	0.74
Ga-K α	38.923	1.394	1.07	38.918	2.235	1.25	38.933	1.646	1.27
Zn-K α	41.799	1.339	0.77	41.783	2.554	1.96	41.802	10.894	6.26
Cu-K α	45.015	2.926	1.14	45.024	4.037	1.01	45.028	2.322	0.90
Ni-K α	48.673	6.474	1.02	48.671	7.717	1.11	48.648	7.794	1.23
Fe-K α	57.522	74.235	1.09	57.501	81.77	0.88	57.499	69.124	1.02
Mn-K α	62.956	3.576	1.16	62.941	3.92	0.93	62.972	2.67	0.87
Cr-K α	69.348	18.595	1.09	69.352	22.653	1.15	69.325	23.02	1.34
Ca-K α	113.185	2.242	1.35	113.17	2.665	0.99	113.209	1.154	0.69
K-K α	136.656	0.499	-	136.713	0.283	1.40	136.758	0.779	-
Cl-K α	92.887	0.824	3.04	92.878	0.348	1.15	92.852	0.399	1.47
S-K α	110.846	4.602	4.29	110.892	1.787	1.44	110.854	1.406	1.31
P-K α	141.12	5.409	21.38	141.145	0.639	1.15	141.163	0.247	0.98
Si-K α	109.102	4.817	10.56	109.103	2.944	4.59	109.12	1.792	3.93
Al-K α	144.81	0.772	2.21	144.779	0.51	2.49	145.079	3.03	8.66
Mg-K α	45.346	0.083	4.61	45.293	0.047	1.81	45.302	0.127	7.06
Na-K α	55.349	0.032	-	55.336	0.038	1.90	55.307	0.053	-



Phosphorus (P) contamination might come from the pipes and flanges, other BCP'ed cavities?

PE sponge rinse test in a beaker

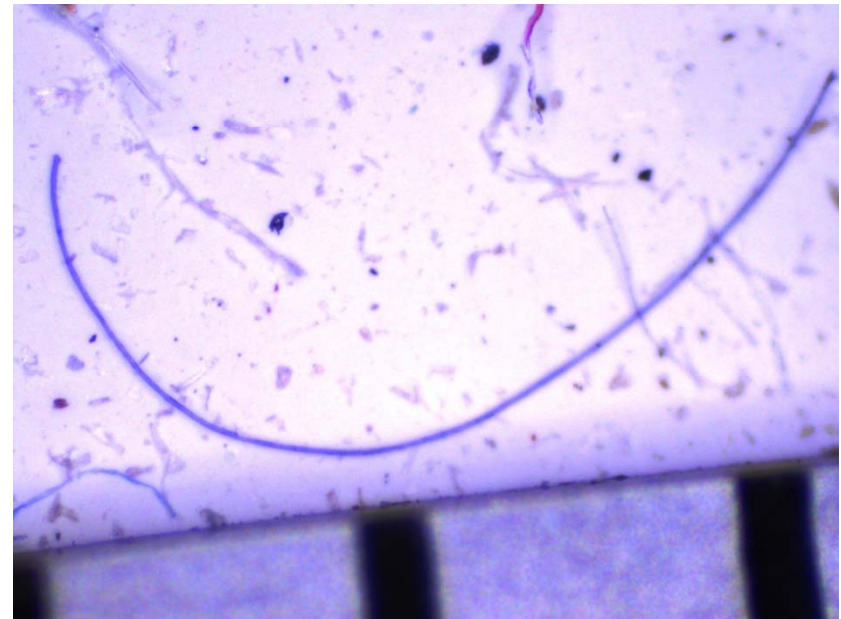
- Pour 400 ml UPW into a 500ml-beaker.
- Wipe inside the beaker by PE sponge for 10 minutes.
- Filter the UPW in the beaker by filter-paper.
- Analyze residuals on the filter-paper.



Residuals on filter-paper from beaker UPW

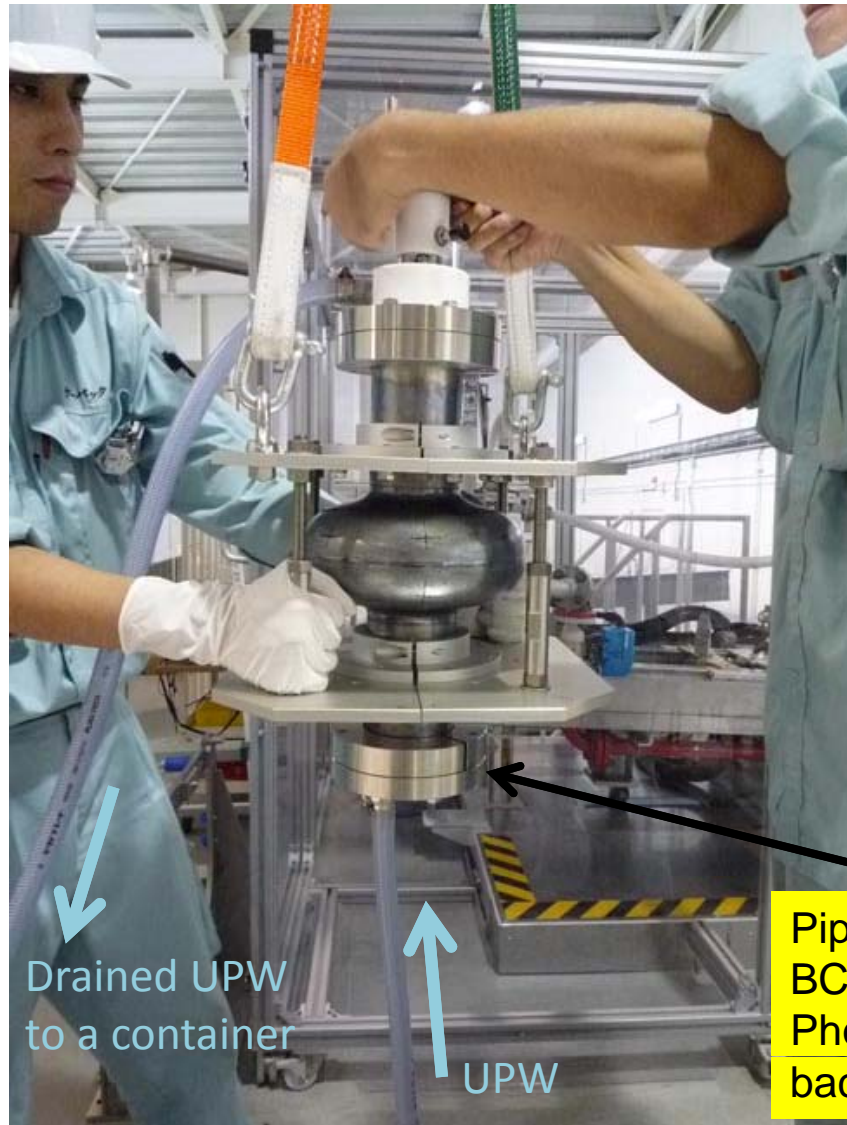


Residuals look like fibers/particles



←→
1 mm

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



Draining UPW inside the cavity



Pipes and flanges are commonly used for other BCP'ed cavities. This caused the contamination of Phosphorus (P) in the drained water. We will come back this later.

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

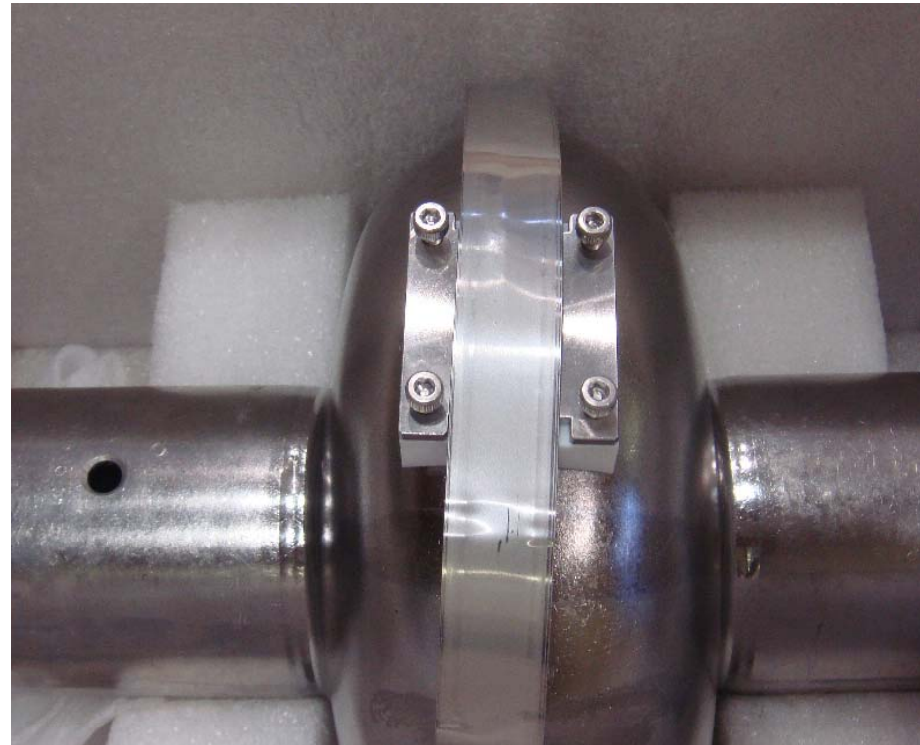
Summary of sponge cleaning study

- Two samples w/ and w/o sponge cleaning after EP-process were analyzed by FS-SEM and XPS.
- lots of field emitters (Nb_xO_y) were found on the sample w/o sponge cleaning.
- Few field emitters were found on the sample w/ sponge cleaning.
- Sulfur was not found by XPS on the samples.
- First wiping/fitting test of sponge-cleaner was done with a single-cell cavity after EP process.
- UPW was fed during sponge wipe, and drained water was analyzed by IC and XRF.
- Sponge fibers/particles and S, P, Cl, F were found in the drained water.
- Next step: Can fibers/particles be removed by HPR? Selection of new sponges? Collaboration with sponge vendor for special (contamination-free) sponge?
- More detailed information can be found at <http://ilc.kek.jp/JFK-S0/>

Special single-cell cavity for sample-study

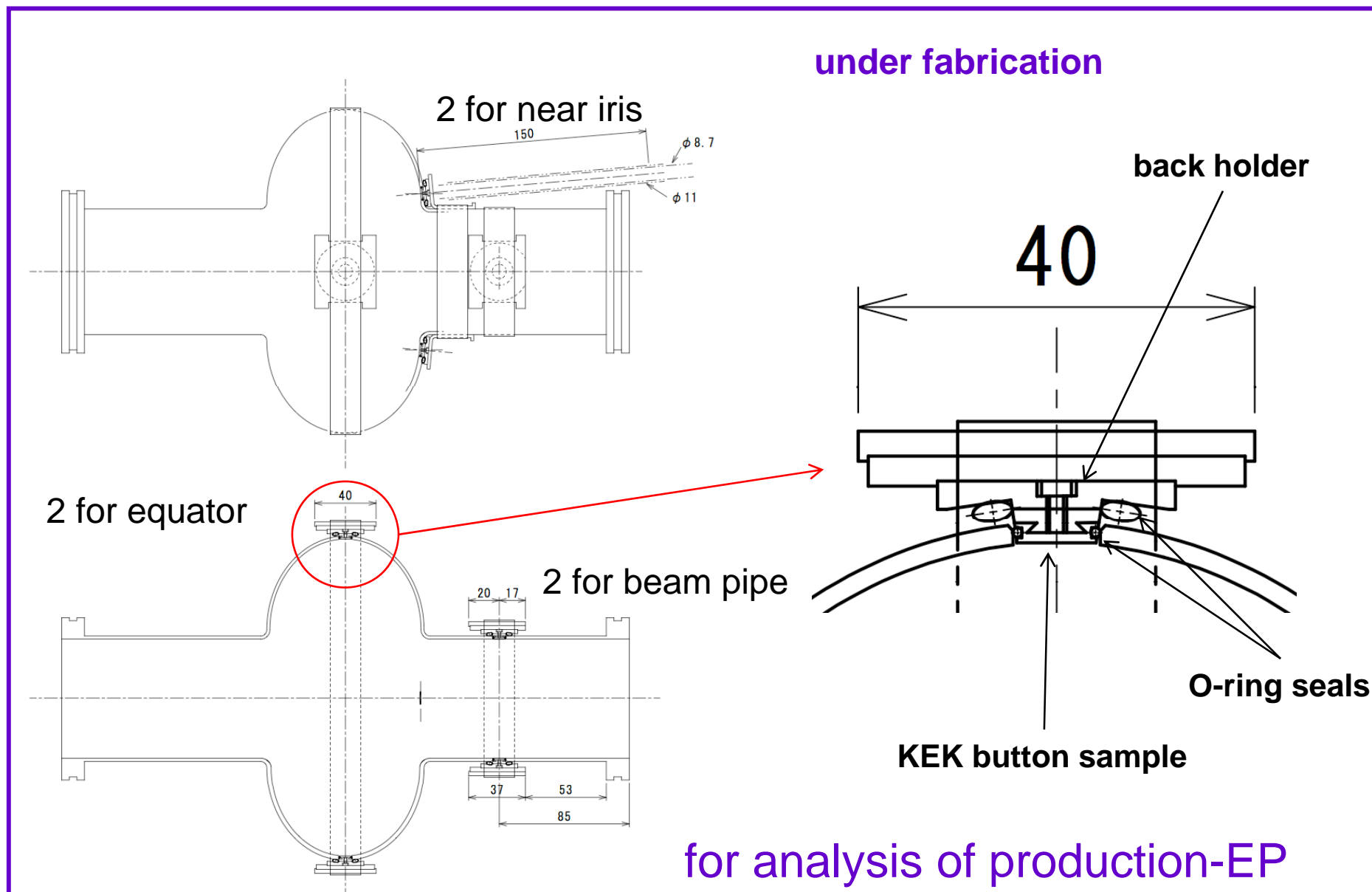


Machining of 6 holes done



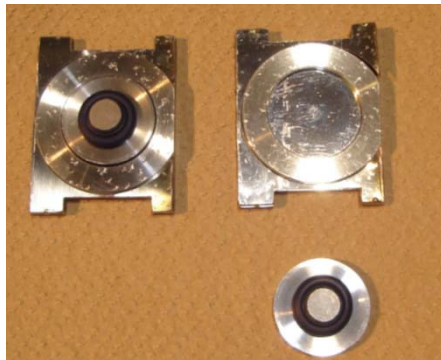
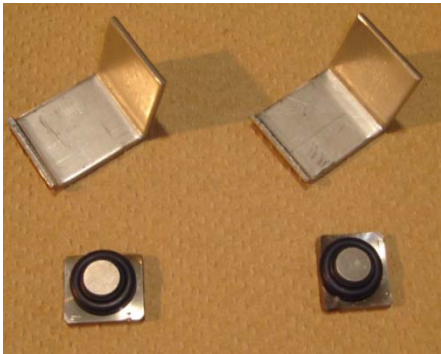
Nb samples and all parts were already delivered. (17 Oct. 2008).

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

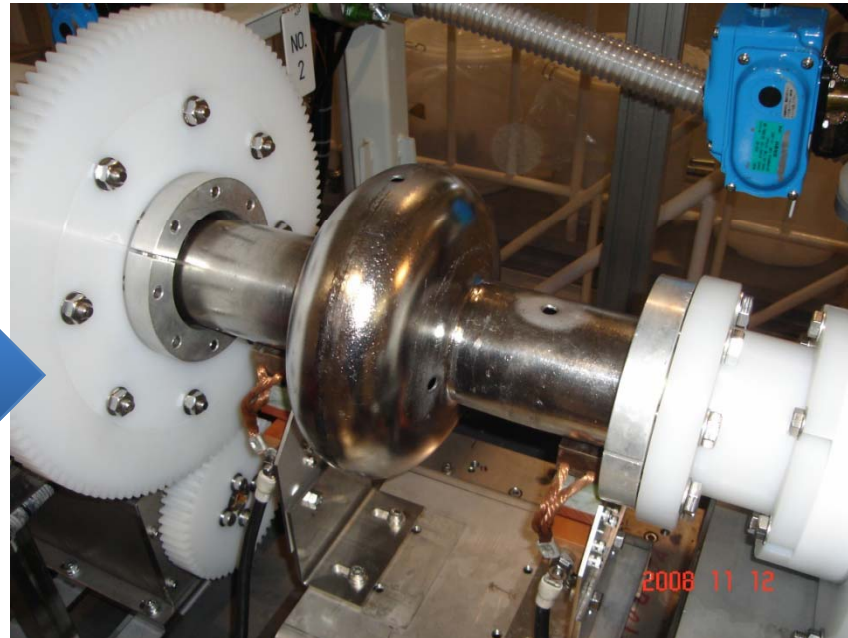


Special single-cell cavity for sample-study

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



First EP at STF/KEK
Recipe:
BCP(10um)+EP(20um) + UPW rinse



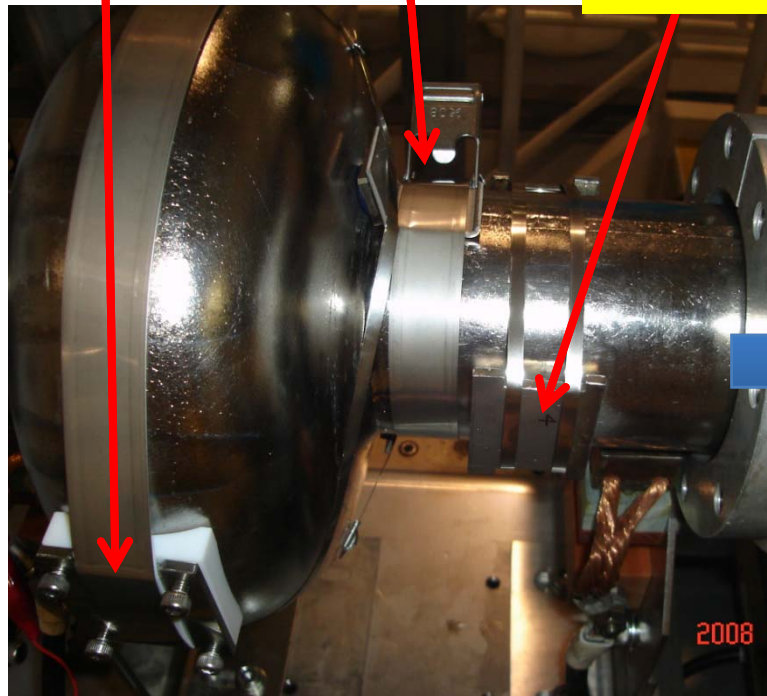
Special cavity with 6 sample-holes
on the EP bed.

Special single-cell cavity for sample-study

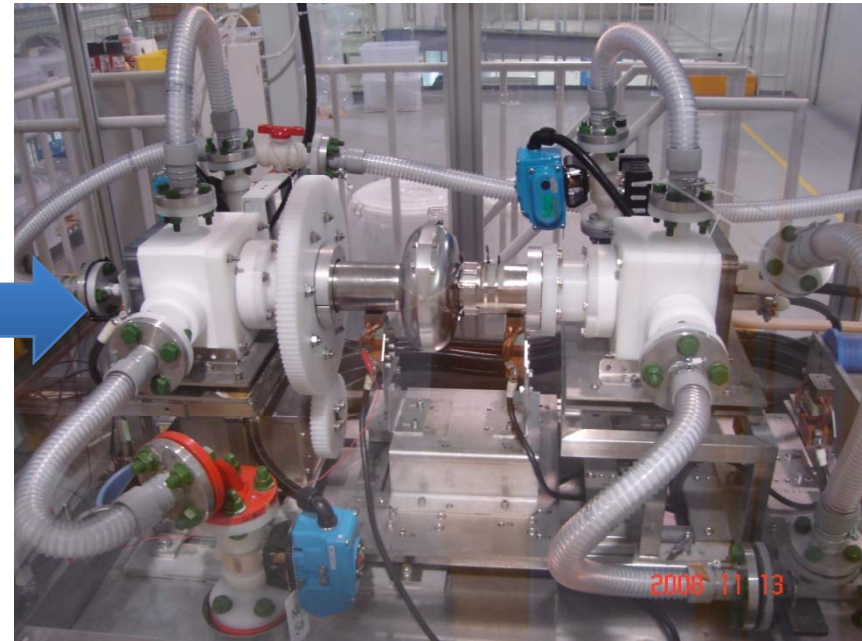
2 samples
at equator

2 samples
at iris

2 samples on
a beam-pipe



6 samples were dressed on
the special cavity

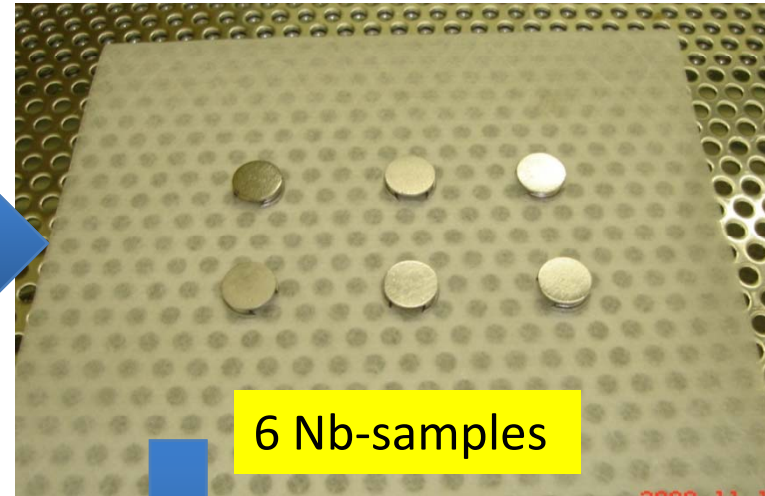


EP 20 um + UPW rinse
(No ultrasonic-rinse)

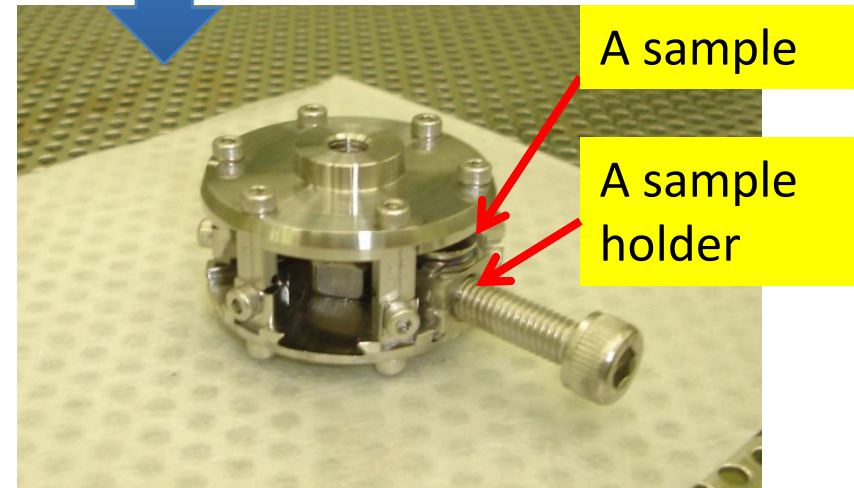
Special single-cell cavity for sample-study



Disassembly in class-10
Clean-Room



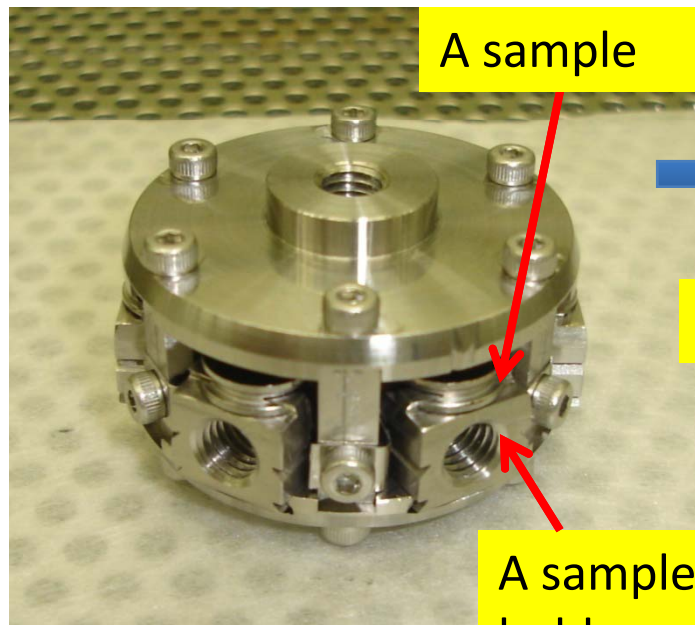
6 Nb-samples



A sample

A sample
holder

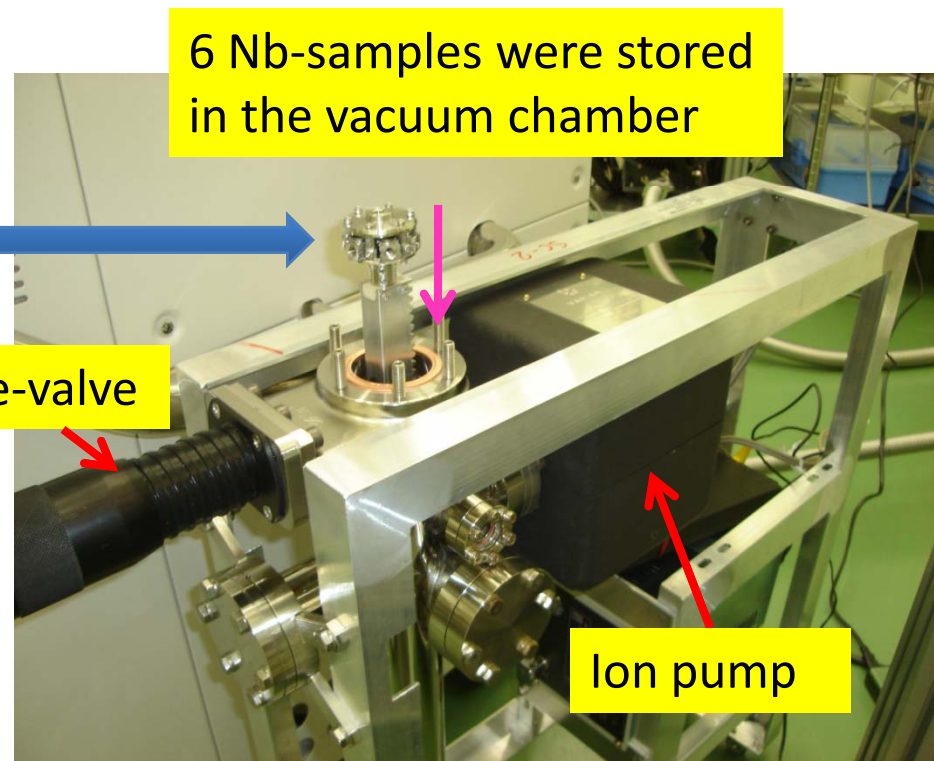
Special single-cell cavity for sample-study



A sample

A sample holder

6 Nb-samples + sample-holders

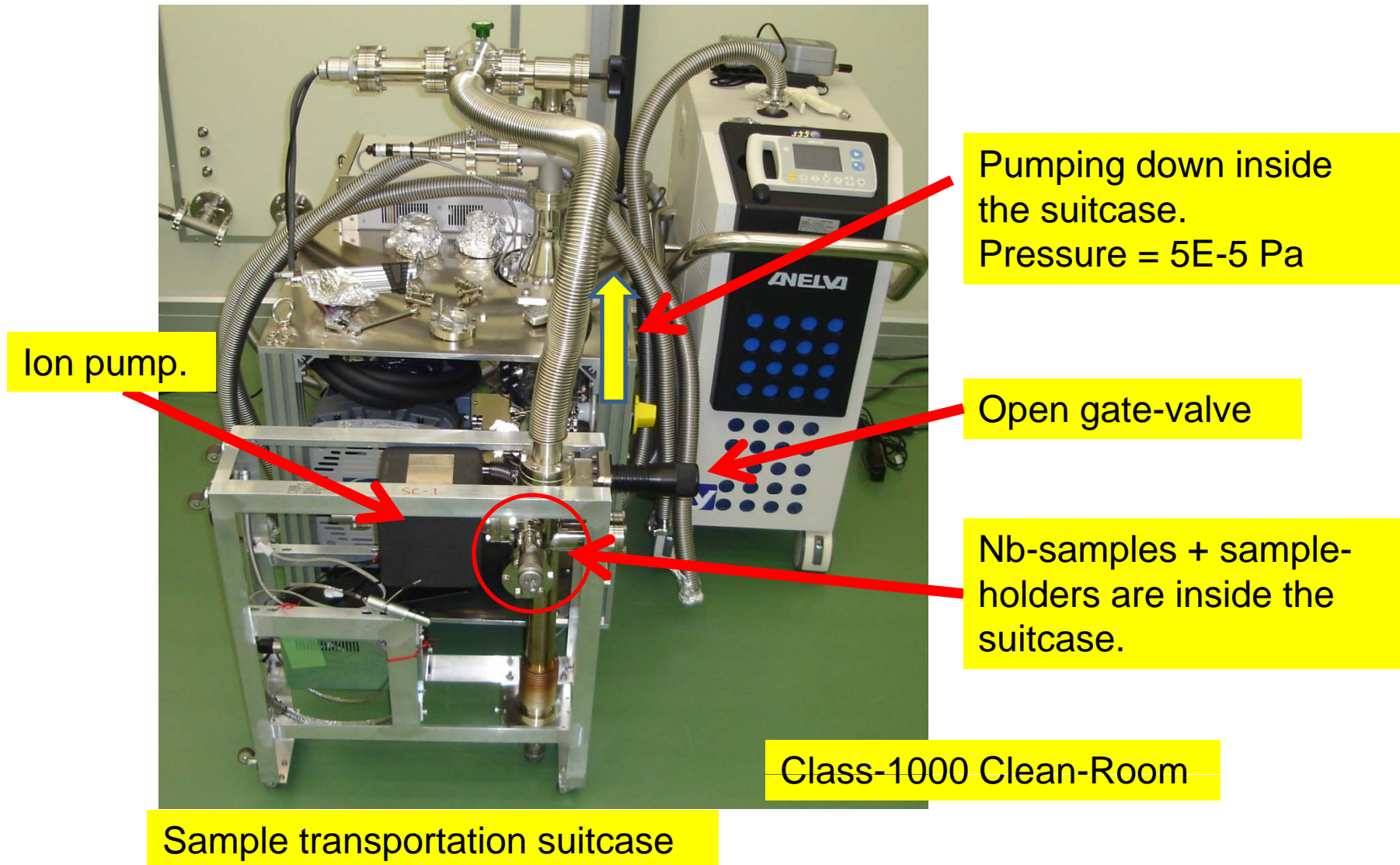


6 Nb-samples were stored in the vacuum chamber

Gate-valve

Ion pump

Sample transportation suitcase



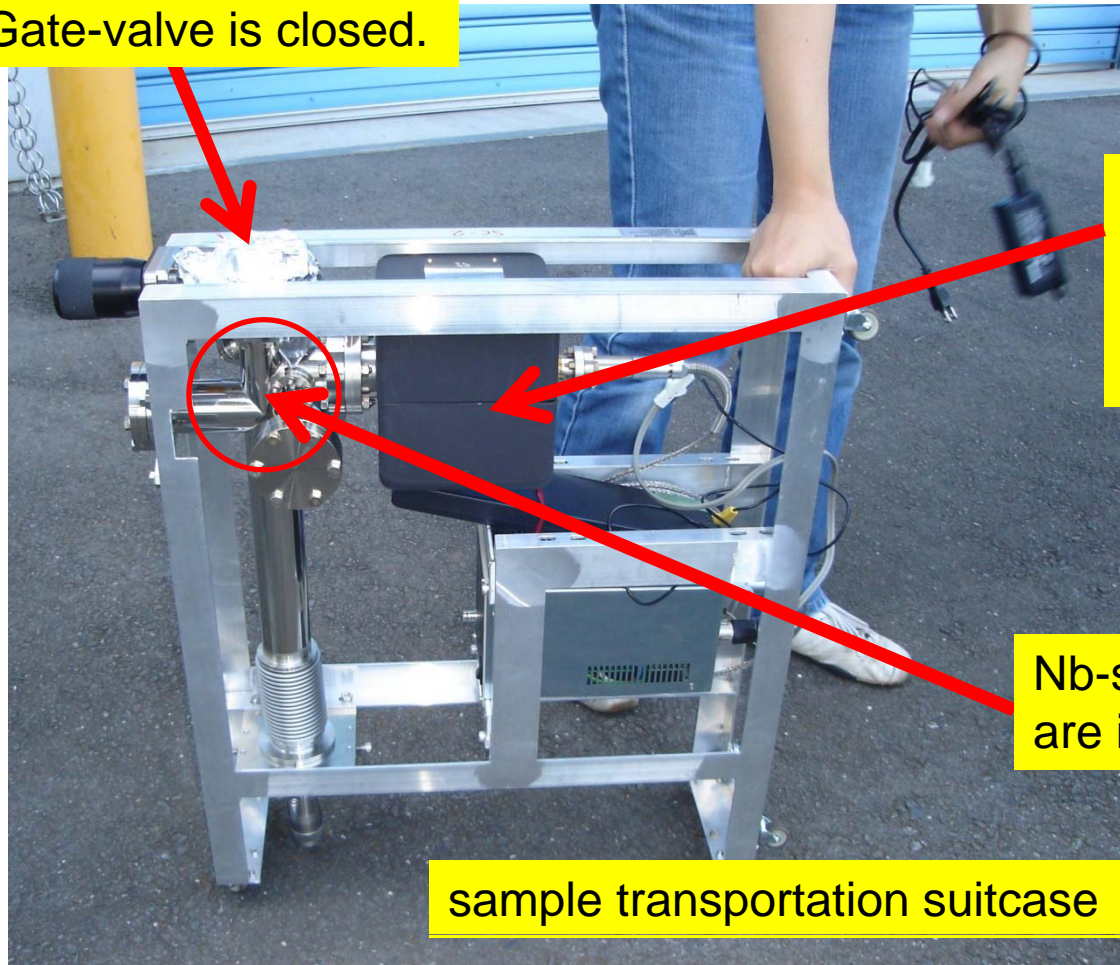
Transportation of samples to XPS analysis room

Gate-valve is closed.

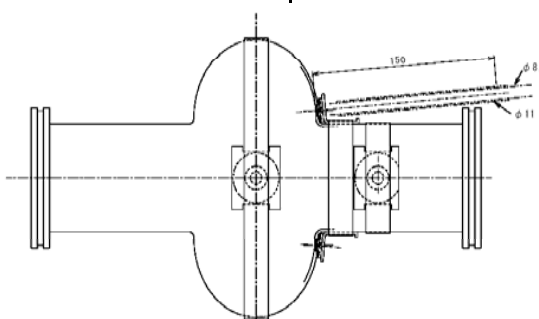
Pumping down inside the suitcase with ion pump powered by battery.
Pressure = $5\text{E-}5$ Pa

Nb-samples + sample-holders are inside the suitcase.

sample transportation suitcase



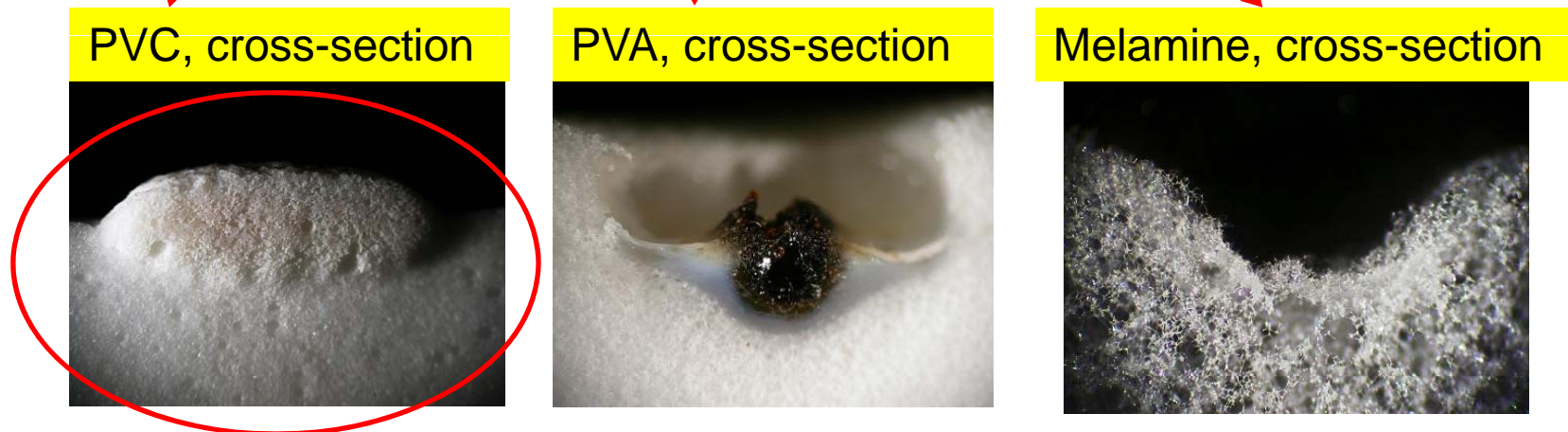
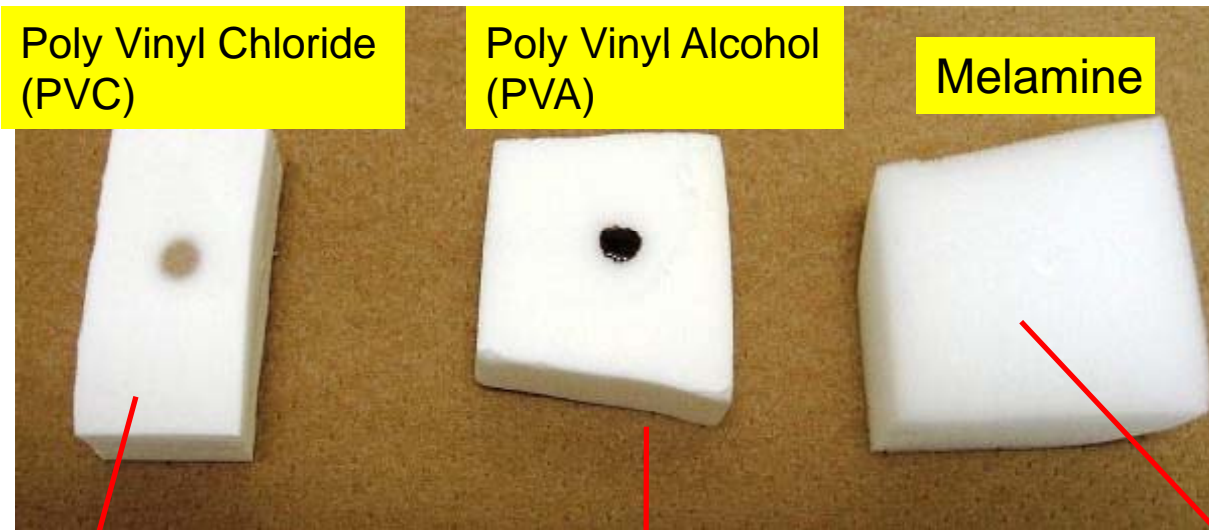
Plan for KEK-type samples in special cavity

November 3	November 10	November 17	November 24	December 1	December 7
<p>KEK-samples</p>  <p>Fitting test on the EP bed at STF/KEK</p> <p>First EP at STF/KEK Recipe: BCP(10um)+EP(20um) + UPW rinse</p>					
				First XPS / SEM analysis at KEK	

Backup slides

Melting test of sponges for EP solution

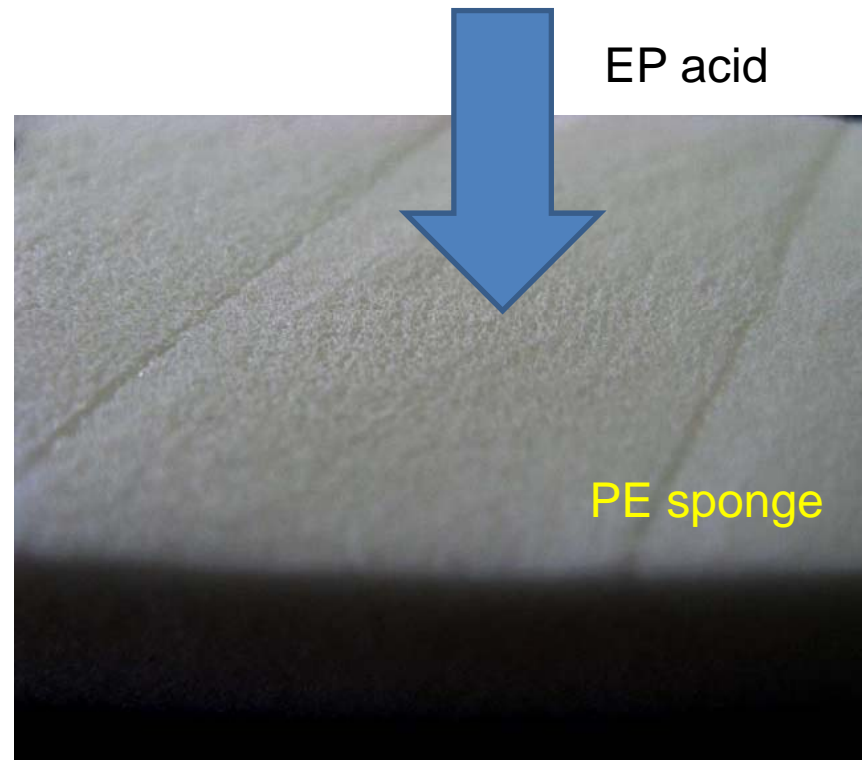
20 micro litter of EP solution (no dilution) was dropped on each sponge.



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

Poly-Ethylene (PE) sponge test with EP acid

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

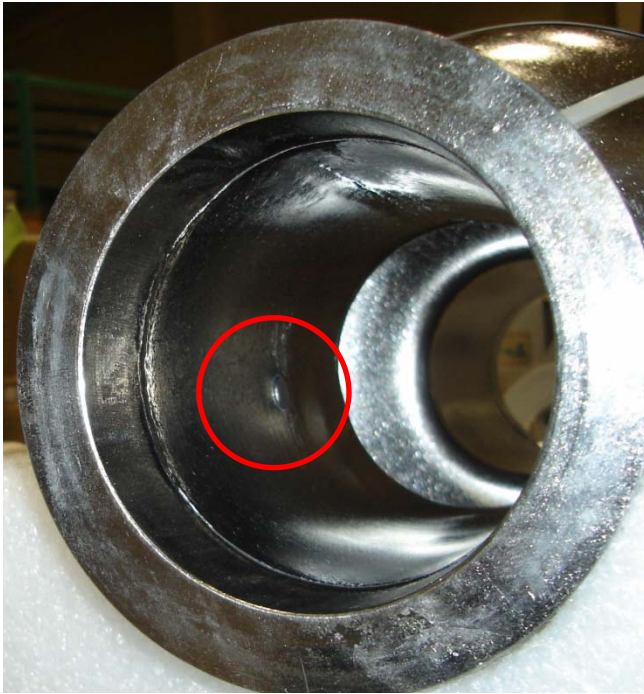


October 2008

No corrosion after dropping EP acid on PE sponge.

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

Special single-cell cavity for sample-study



Nb-sample at beam-pipe



Nb-sample at iris