Lecture B2: Superconductive RF

Surface Preparation

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LC school 2015

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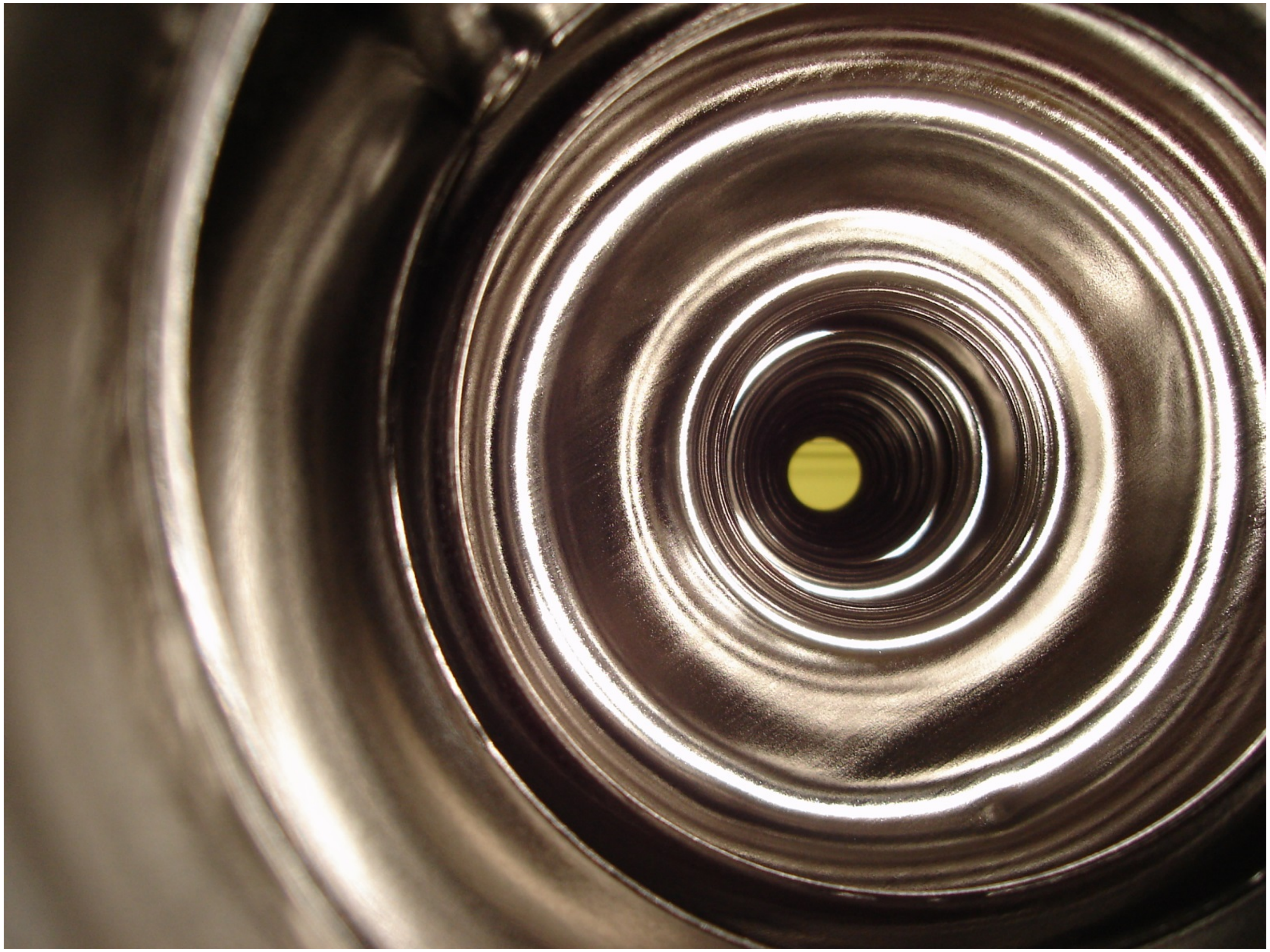
Inner Surface Preparation of SC Cavity



Overview of Inner Surface Preparation

- Thick/Rough Removal (>100 um)

  Three alternative methods:

  Buffered Chemical Polishing (BCP)

  Electro-Polishing (EP)

  Centrifugal Barrel Polishing (CBP)

- Annealing / Degassing (750 – 800 C, ~3 h)
- Final Thin Removal (10 – 30 um)

  Two alternative methods:

  Buffered Chemical Polishing (BCP)

  Electro-Polishing (EP)

- High Pressure Rinse (>7 h)
- In-situ Baking (120 – 140 C, 48 h)

Overview of Inner Surface Preparation

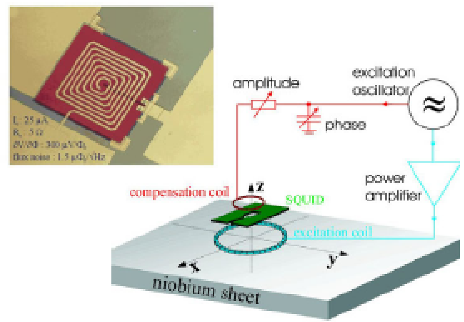
Process Name	Process	Removal Thickness (um)	Purpose
Centrifugal Barrel Polishing (CBP)	Mechanical removal with water and media (stones / plastic ….)	>100	Removal of damaged layer of Nb, or removal of defects
Buffered Chemical Polishing (BCP)	Chemical Reaction / Etching	Rough: > 100 Final: 10 - 30	Rough: Removal of damaged layer of Nb. Final : Smooth surface
Electro-Polishing (EP)	Electro-Chemical Reaction	Rough: > 100 Final: 10 - 30	Rough: Removal of damaged layer of Nb. Final : Smooth surface
Annealing / Degassing	750 ^{O}C, 3 hours Vacuum Furnace	-	Release of stress in material / Degassing of H
High Pressure Rinse (HPR)	High Pressure Rinse with Ultra Pure Water (UPW)	-	Removal of contamination / Clean surface
In-situ Baking	120 ^{O}C, 48 hours baking with Vacuum pumping inside of cavity	-	Diffusion of Oxygen

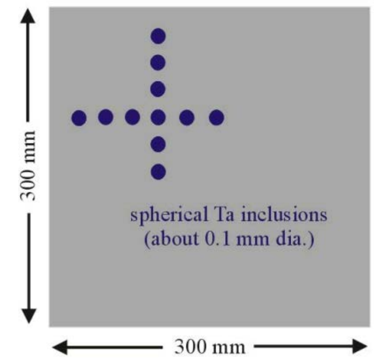
Scanning of defects with eddy current

Successfully developed at DESY to pre-screen Nb

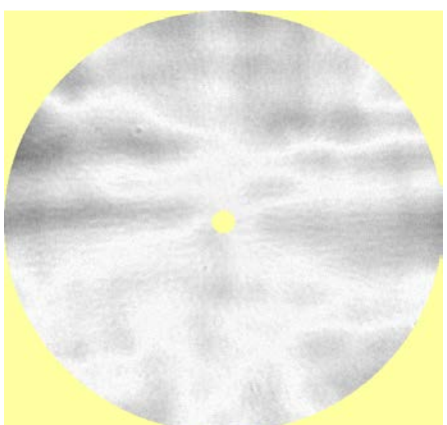
Sheets for defects: eddy current, resolution ~ 100 μm

squid, resolution < 50 μm

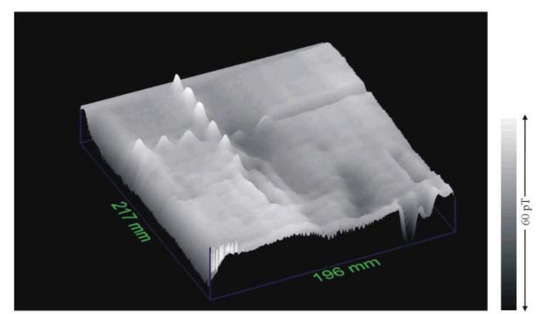




Low Tc superconducting SQUID system for eddy current testing of niobium sheets is in development



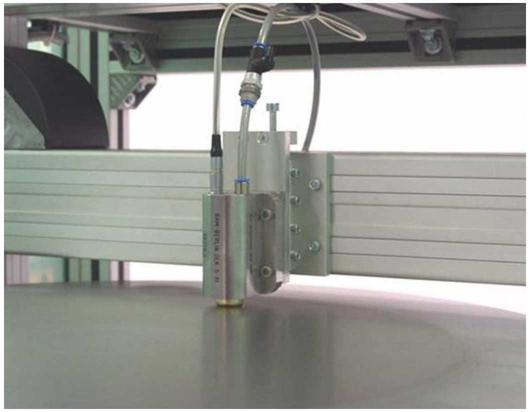
(W.Singer, X.Singer)

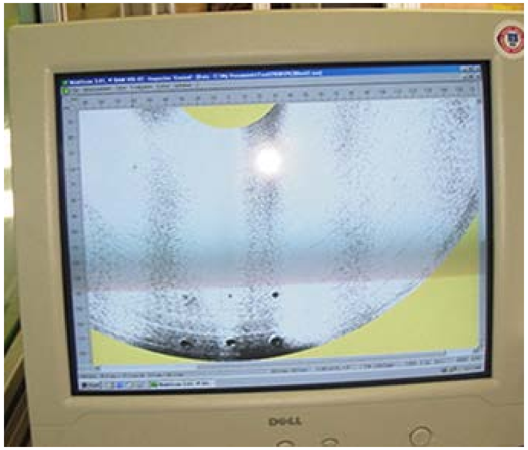


Two-dimensional distribution of eddy-current field above the niobium test sample, measured from the back side of the sample. The excitation coil had 30 turns and a diamter of 3 mm; the excitation frequency was 10 kHz. The reference phase of the lock-in amplifier was chosen such that the lift-off effect was minimized.

Scanning of defects with eddy current

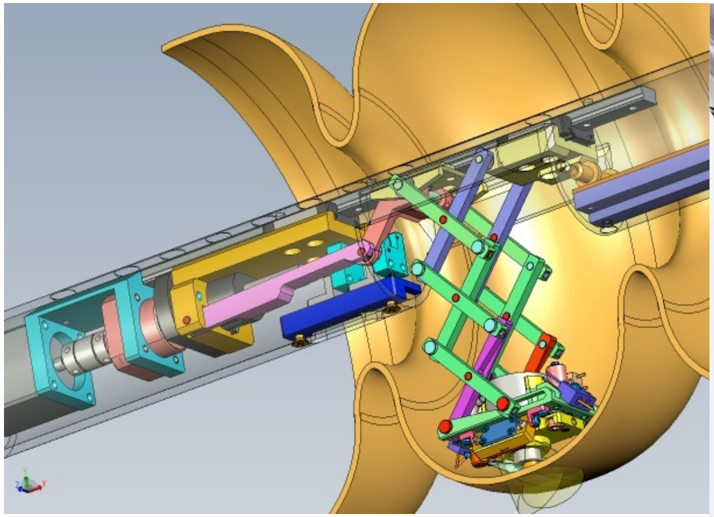
Eddy Current Scanning system for SNS high purity niobium scanning

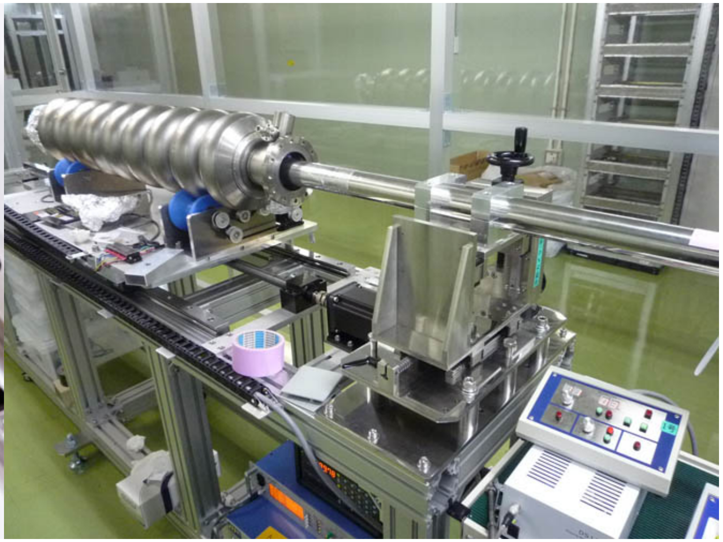




**Scanning of Nb plate/disc before fabrication.
About 1 – 2 % of Nb plates/discs have defects.**

Local grinding at KEK



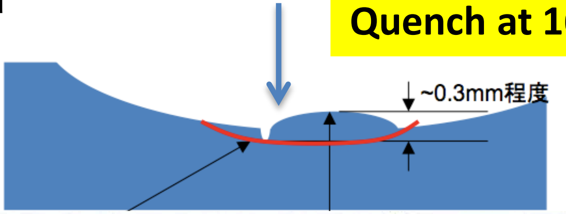


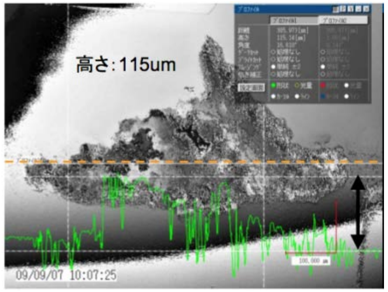
Grinding only for pit, without touching other surface

~115µm depth pit in MHI-08 cavity

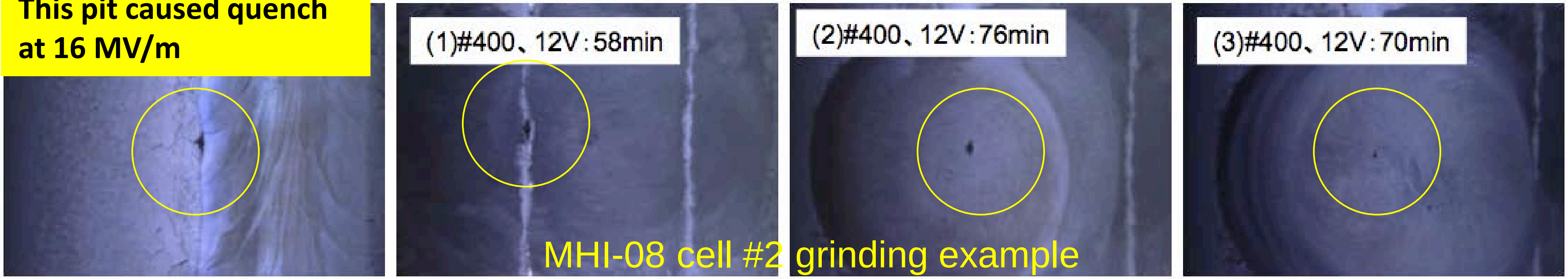
Quench at 16 MV/m

diamond powder compound with water in between were used.



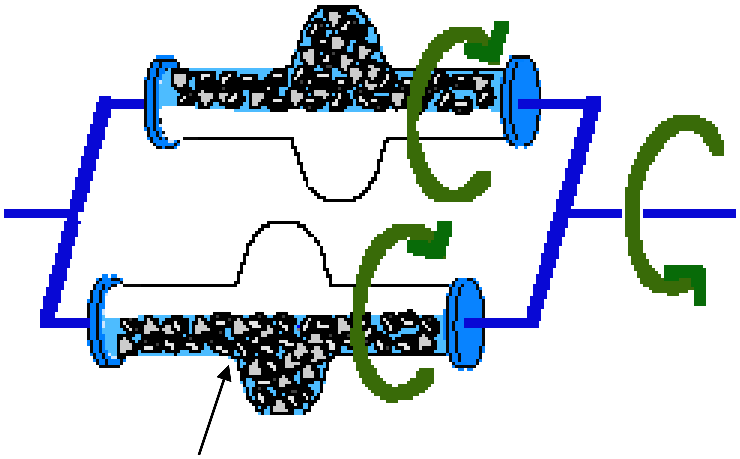


This pit caused quench at 16 MV/m



MHI-08 cell #2 grinding example

Centrifugal Barrel Polishing (CBP) Mechanical grinding



Water and media (stone, plastic, etc.)

Media : stones (rough removal)

Media : plastic (final removal)

Example (KEK recipe)

Step 1) Rough removal with stones + water : 4 hours x 3 times

Removal thickness = 25 um x 3 = 75 um

Step 2) Final removal with plastic + water : 4 hours x 3 times

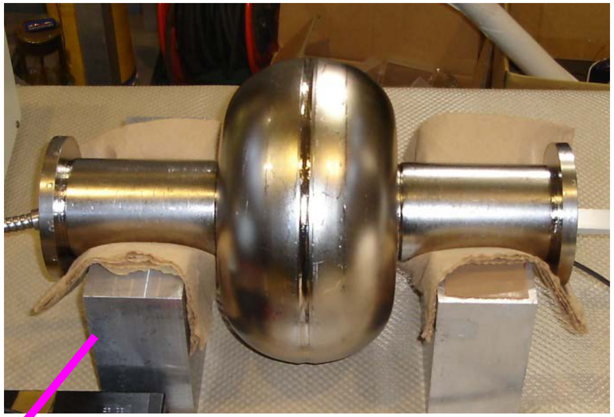
Removal thickness = 20 um x 3 = 60 um

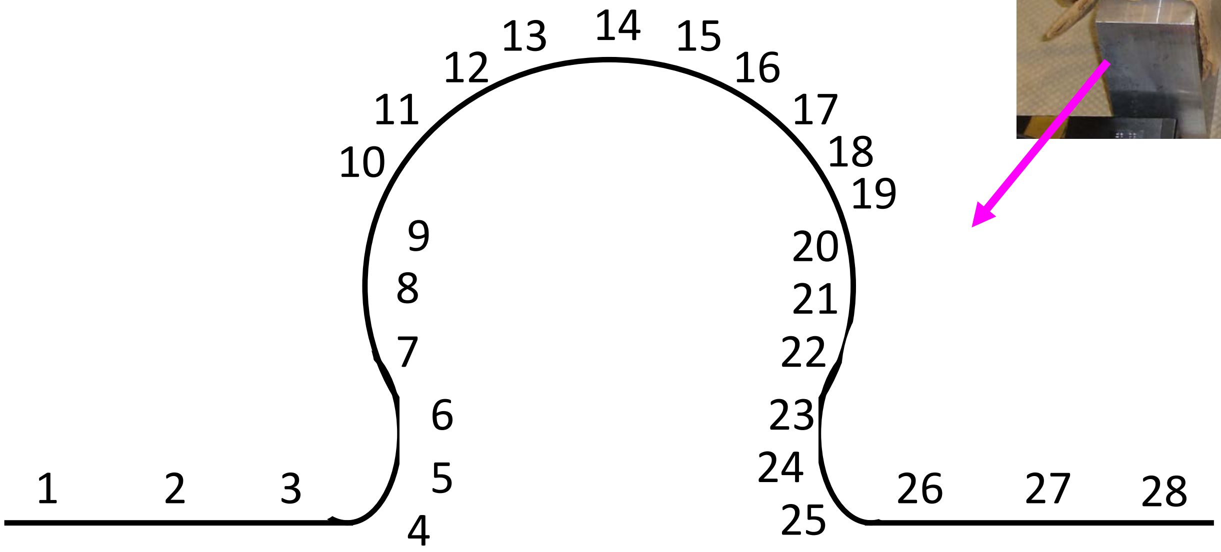
Total removal thickness = 135 um

Centrifugal Barrel Polishing (CBP)
Mechanical grinding

Measurement of Removal Thickness (CBP)

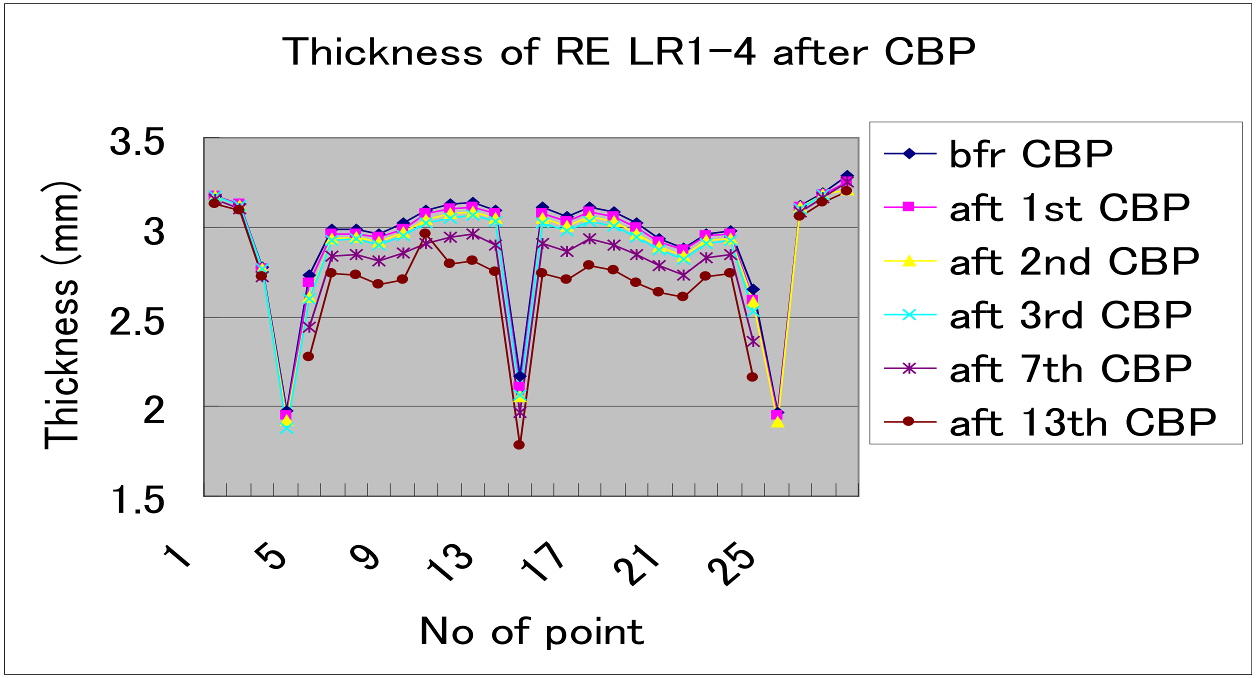
Measurement of thickness by ultrasonic thickness gauge



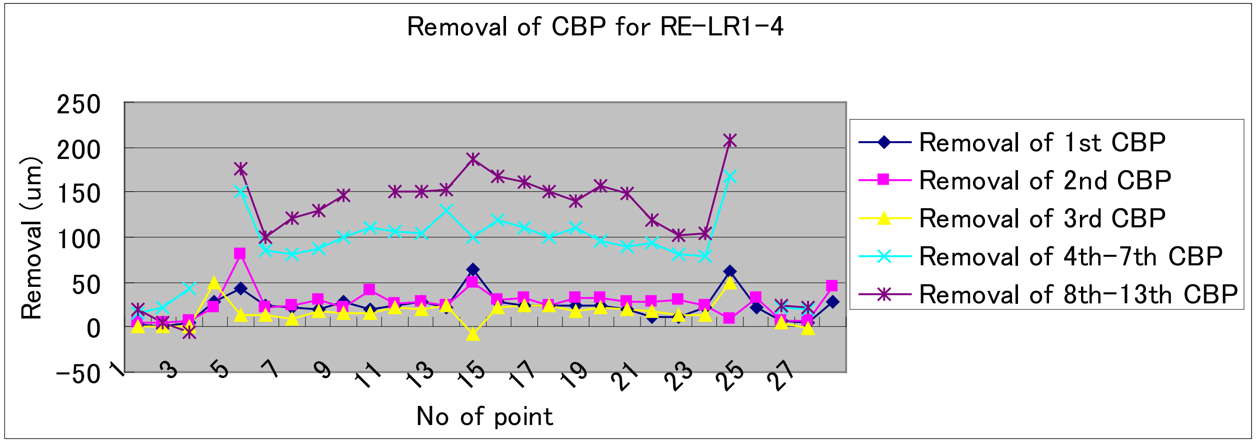


Positions of measurement

Measurement of Removal Thickness (CBP)



Thickness measurements by ultrasonic thickness gauge



Removed weight

1st CBP: 21.0 g

2nd CBP: 21.0 g

3rd CBP: 16.5 g

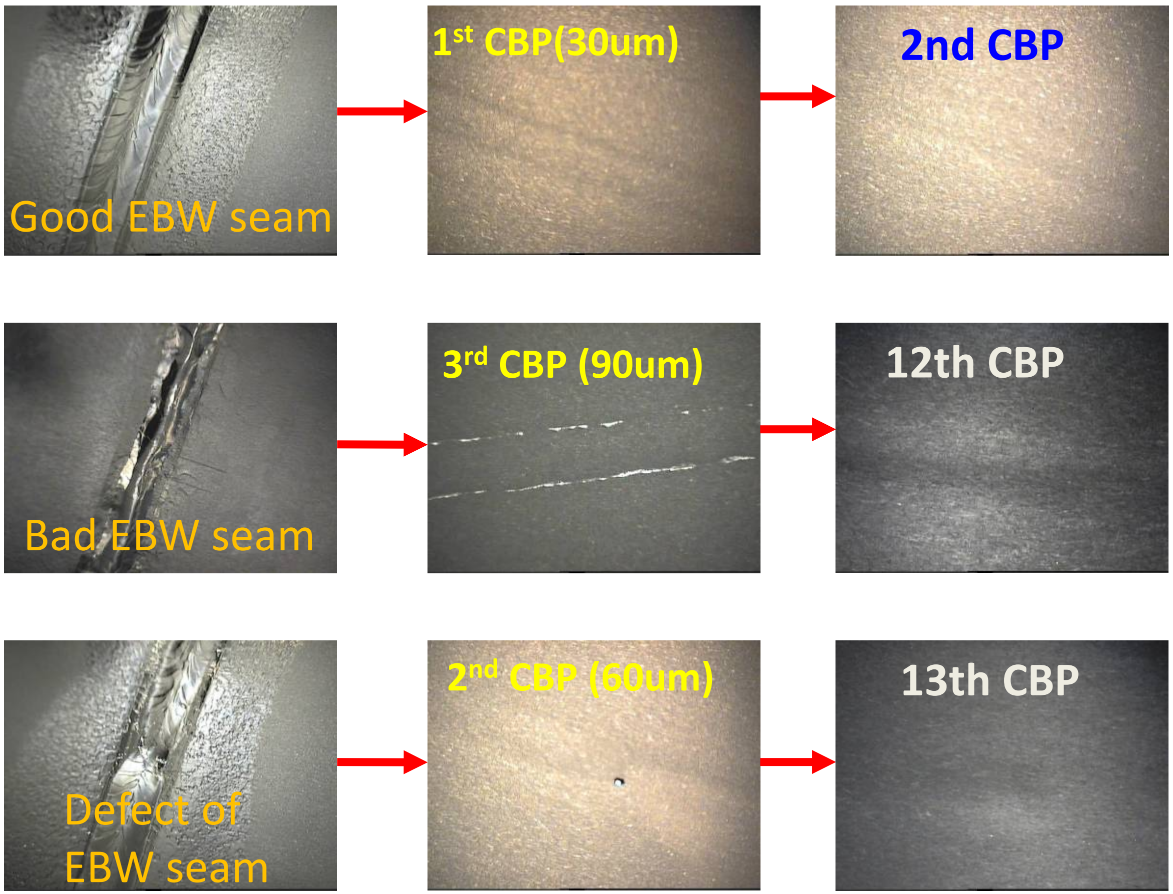
4th-7th CBP: 78.0 g

8th-13th CBP: 94.3 g

Total of removed weight = 230.8 g

Centrifugal Barrel Polishing (CBP) Mechanical grinding

EBW seam before CBP

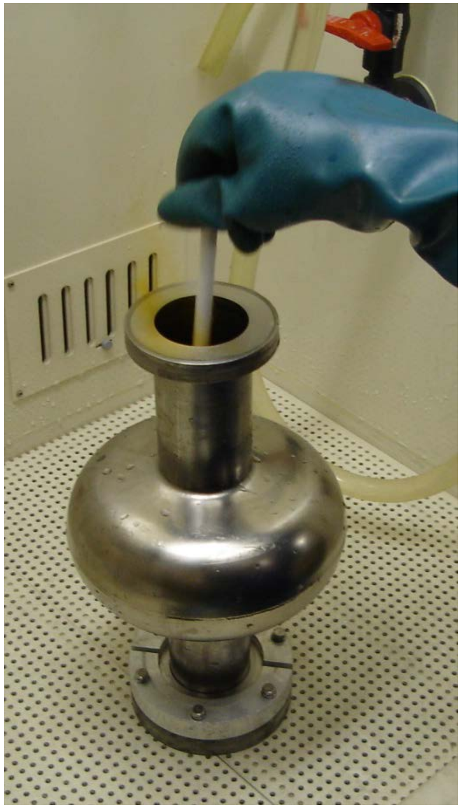


Removal thickness = 60 um.

Removal thickness > 200 um.

Removal thickness > 200 um.

Buffered Chemical Polishing (BCP)



Components of BCP acid (KEK recipe)
HF (HF 46%) : HNO_3 (HNO_3 60%) : H_3PO_4 (H_3PO_4 80%) = 1 : 1 : 1 (Volume Ratio)
H_3PO_4 can be increased if you like slow etching (1:1:2, 1:1:3, etc…)

Various BCP systems in the world

BCP:

Mixture of $HF/HNO_3/H_3PO_4$ in ratios 1:1:1 or 1:1:2 @ 10-15C

$2Nb + 5NO_3 \rightarrow Nb_2O_5 + 5NO_2$

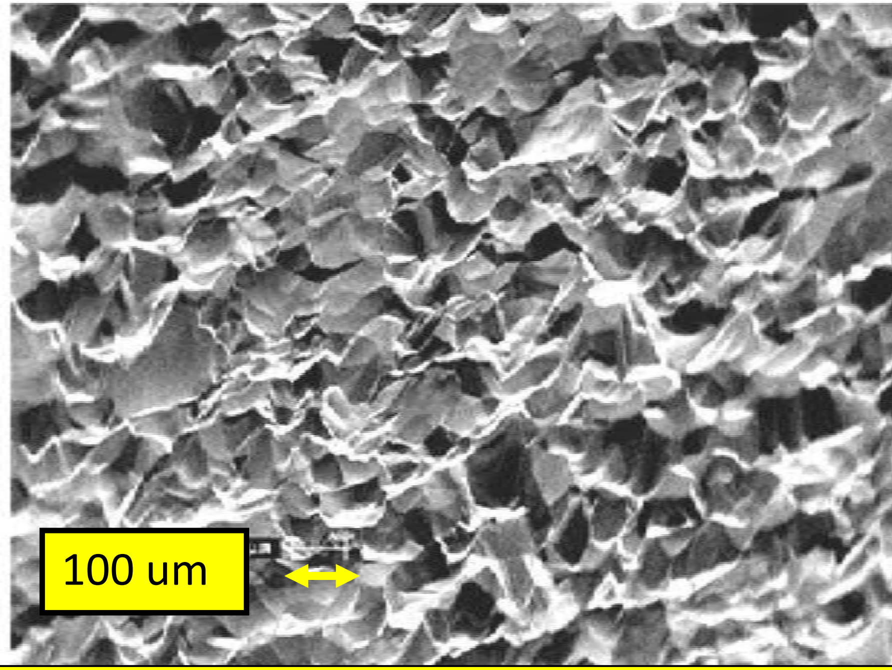
$Nb_2O_5 + 6HF \rightarrow \underbrace{H_2NbOF_5}_{\text{soluble}} + \underbrace{NbO_2F_* 0.5H_2O}_{\text{non soluble}} + 1.5H_2O$

$NbO_2F_* 0.5H_2O + 4HF \rightarrow \underbrace{H_2NbF_5}_{\text{soluble}} + 1.5H_2O$

Exothermic reaction
Removal rate ~ 2 μm/min @ 10C

Buffered Chemical Polishing (BCP)

Nb Surface after BCP



Simpler than EP, but the surface is rougher than EP.
The roughness is coming from the difference of etching among the grain. Steps are made along the grain-boundary.

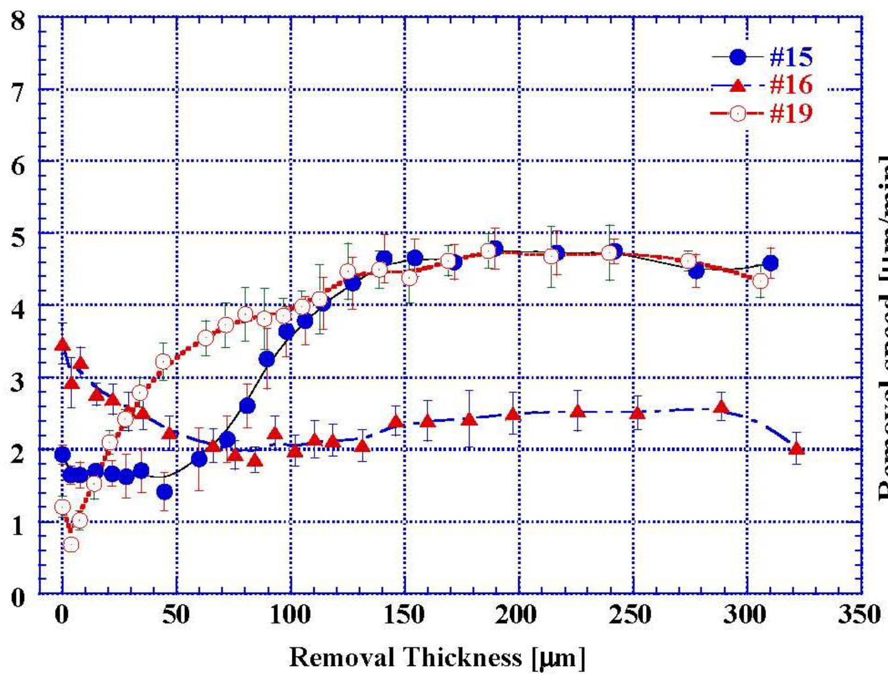
Chemical reaction:

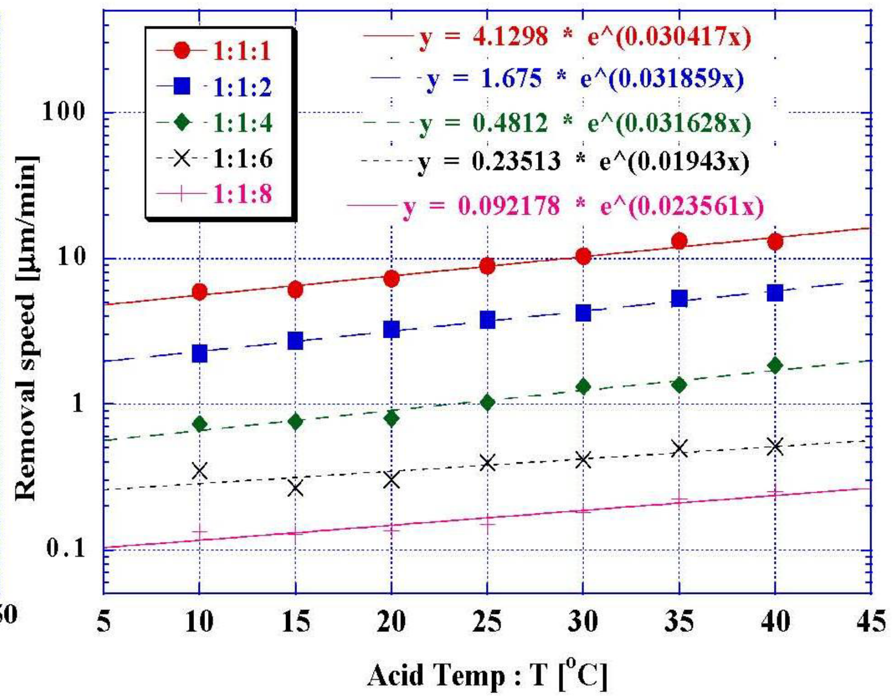
$6Nb + 10HNO_3 \rightarrow 3Nb_2O_5 + 10NO\uparrow + 5H_2O$

$Nb_2O_5 + 10HF \rightarrow 2NbF_5 + 5H_2O$

$6Nb + 10HNO_3 + 30HF \rightarrow 6NbF_5 + 10NO\uparrow + 20H_2O$

Buffered Chemical Polishing (BCP)





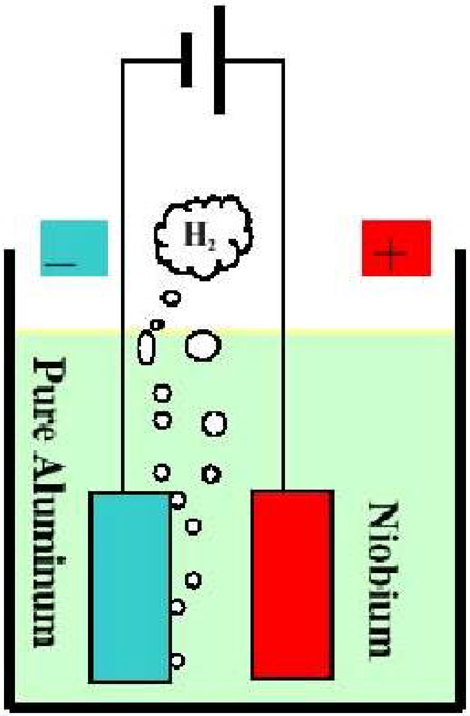
Typical surface roughness = 2 ~ 5 μm after 100μm CP,

Material removal speed ~ 10μm/min at the room temperature with CP acid 1:1:1

CP is faster in material removing than EP.

The roughness is related to the steps along the grain-boundary.
And so the toughness changes if the grain-size of Nb material changes.

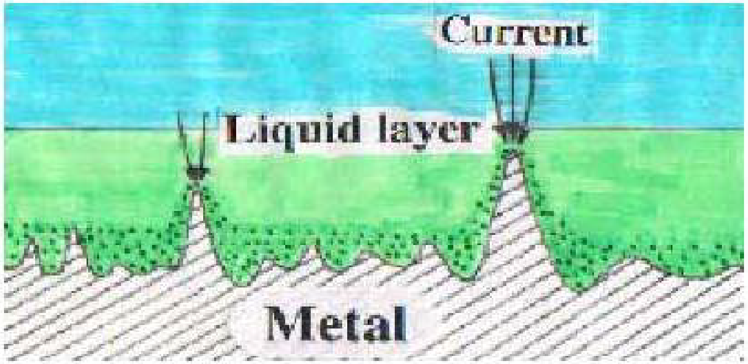
Electro-Polishing (EP)

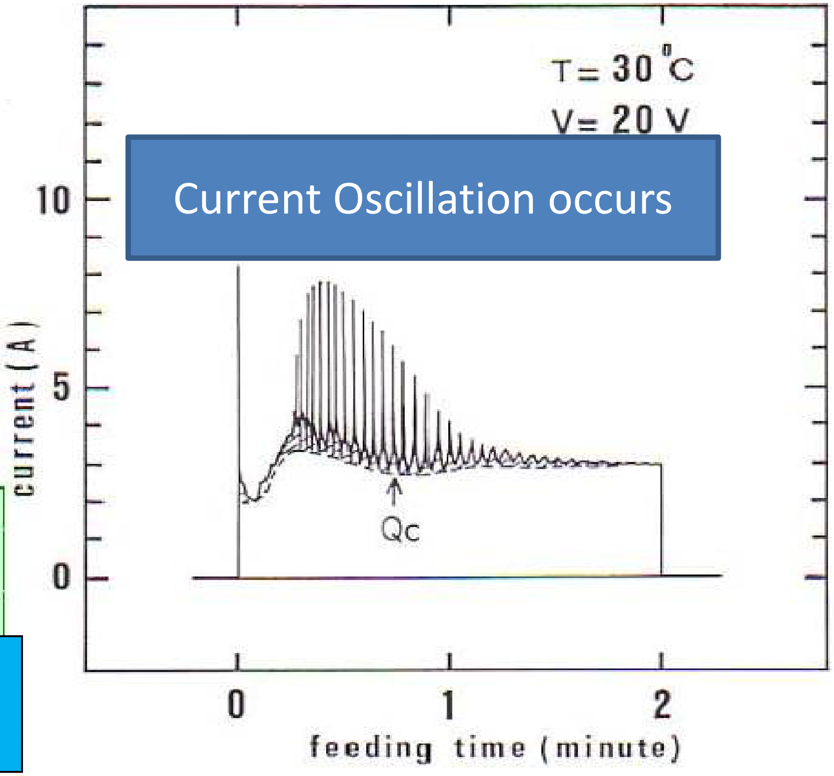


Acid:
H_2SO_4 (>93%): HF(46%)=10:1 V/V

Chemical reaction:
$2Nb + 10HF + 2H_2O \rightarrow 2H_2NbOF_5 + 5H_2\uparrow$

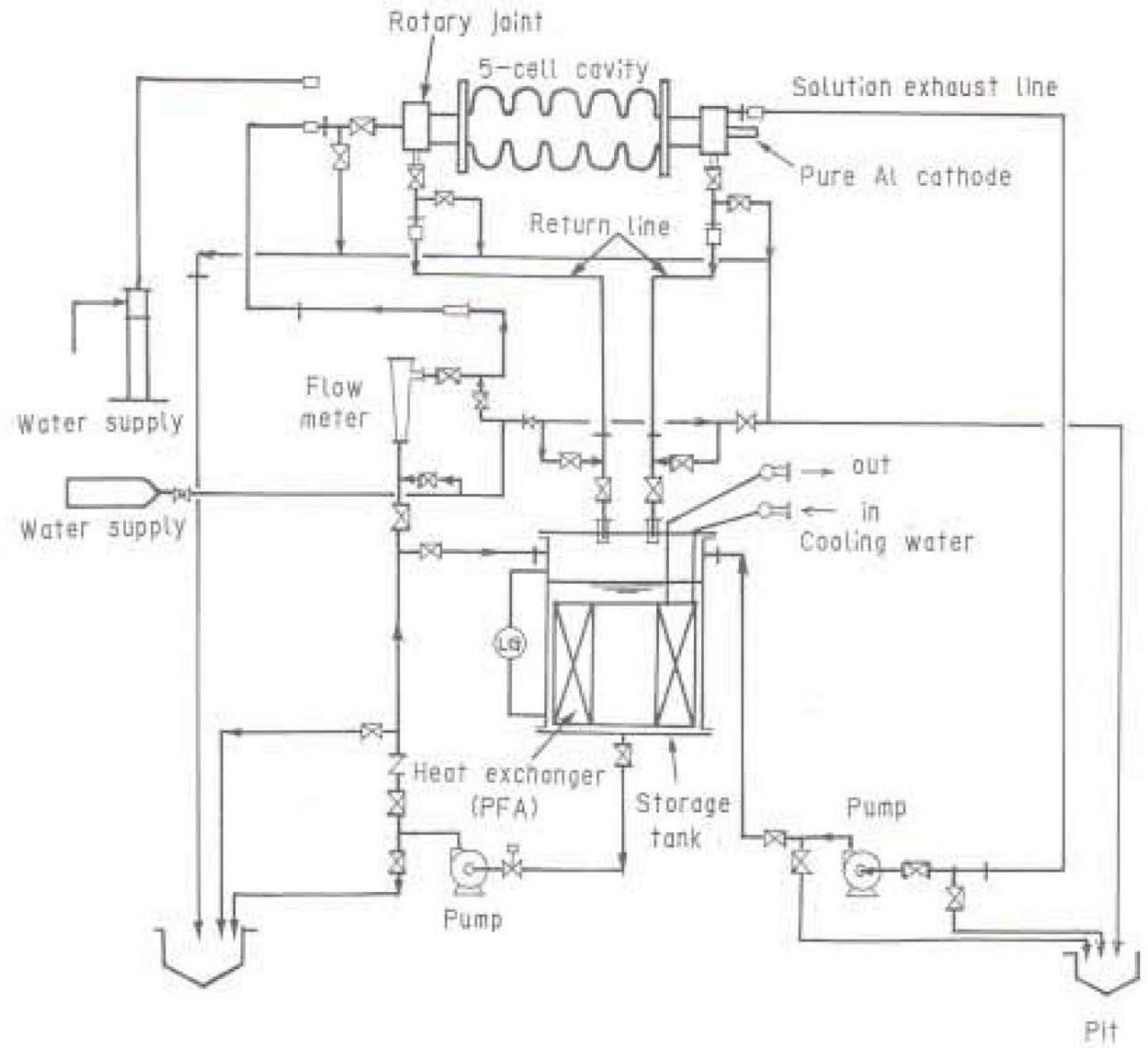
Sulfuric acid is not included in the reaction.
It gives the viscosity to the electrolyte.



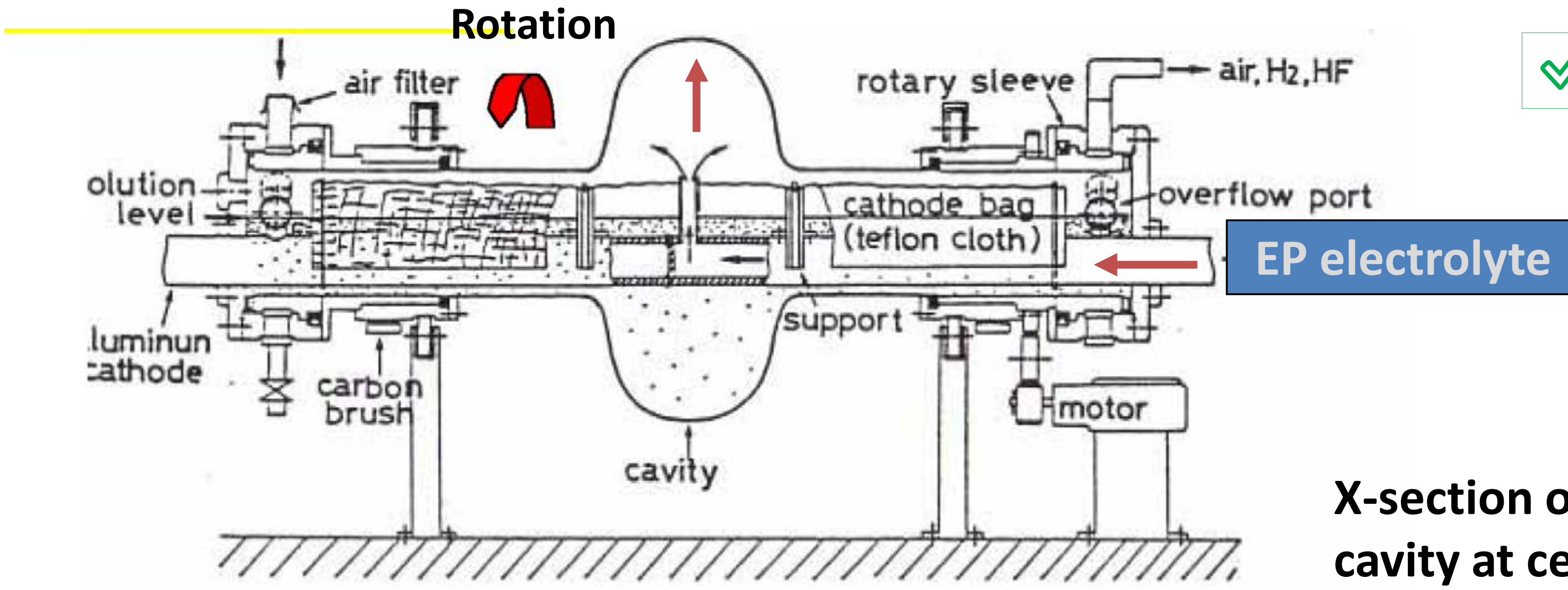


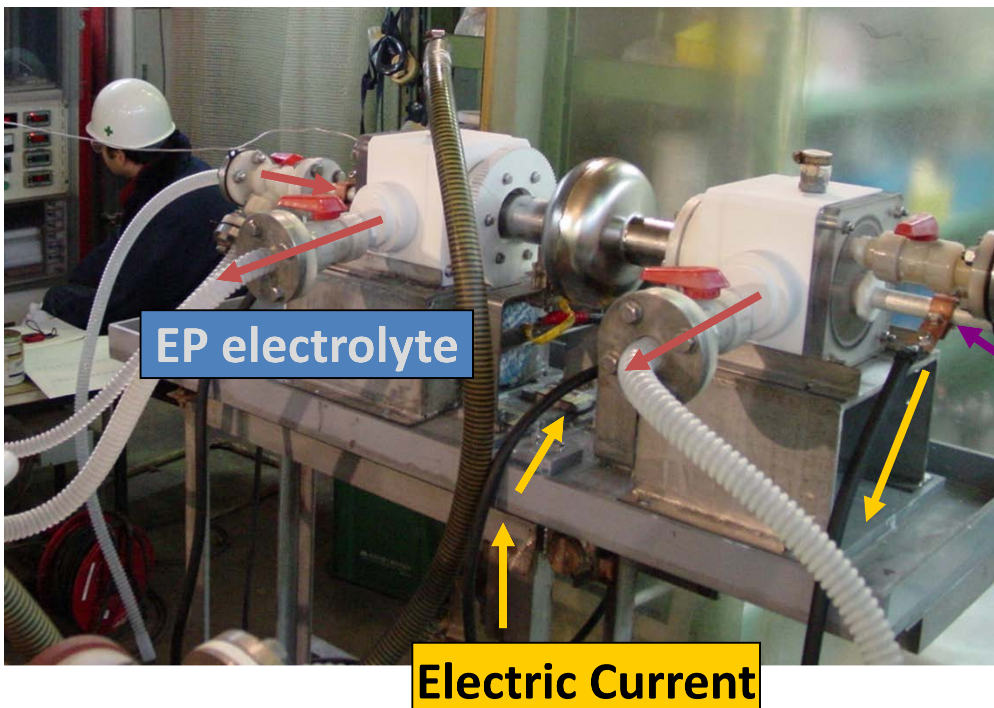
Current Oscillation occurs

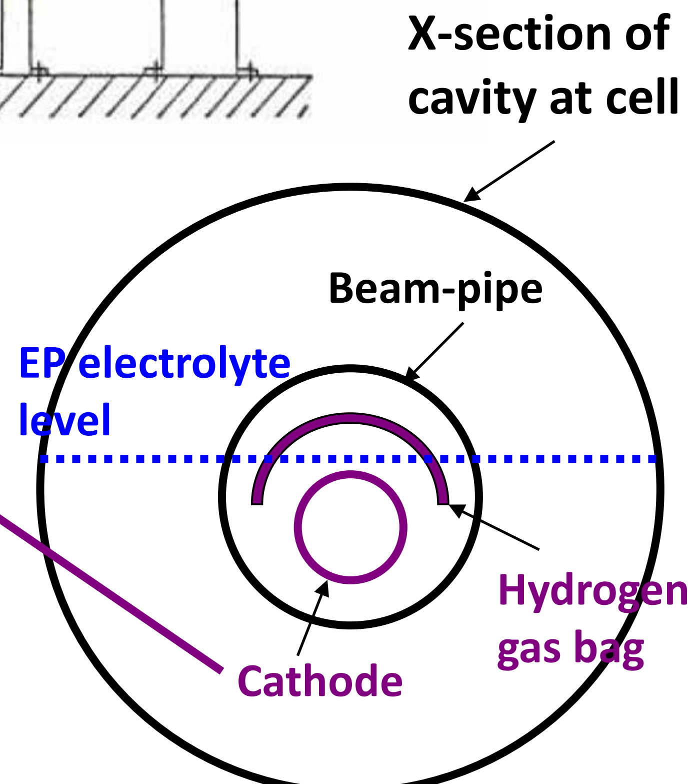
EP System Flow



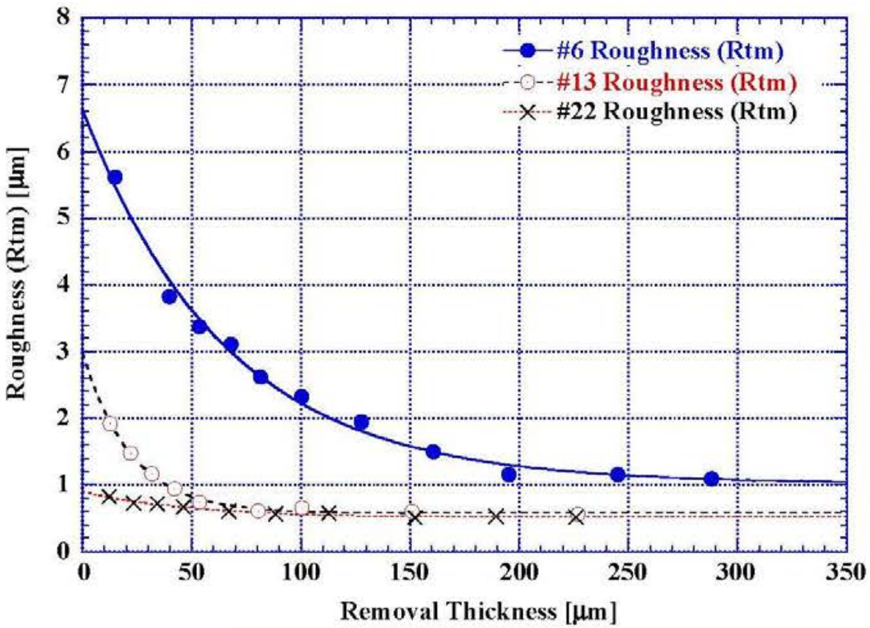
Horizontal EP system for single-cell cavity

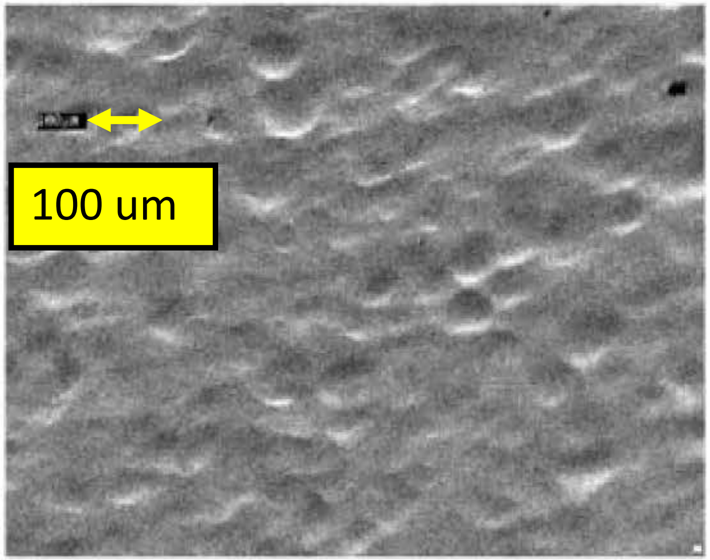

Rotation
air filter
rotary sleeve
air, H2, HF
olution level
cathode bag (teflon cloth)
overflow port
EP electrolyte
luminun cathode
carbon brush
support
motor
cavity


EP electrolyte
Electric Current


X-section of cavity at cell
Beam-pipe
EP electrolyte level
Hydrogen gas bag
Cathode

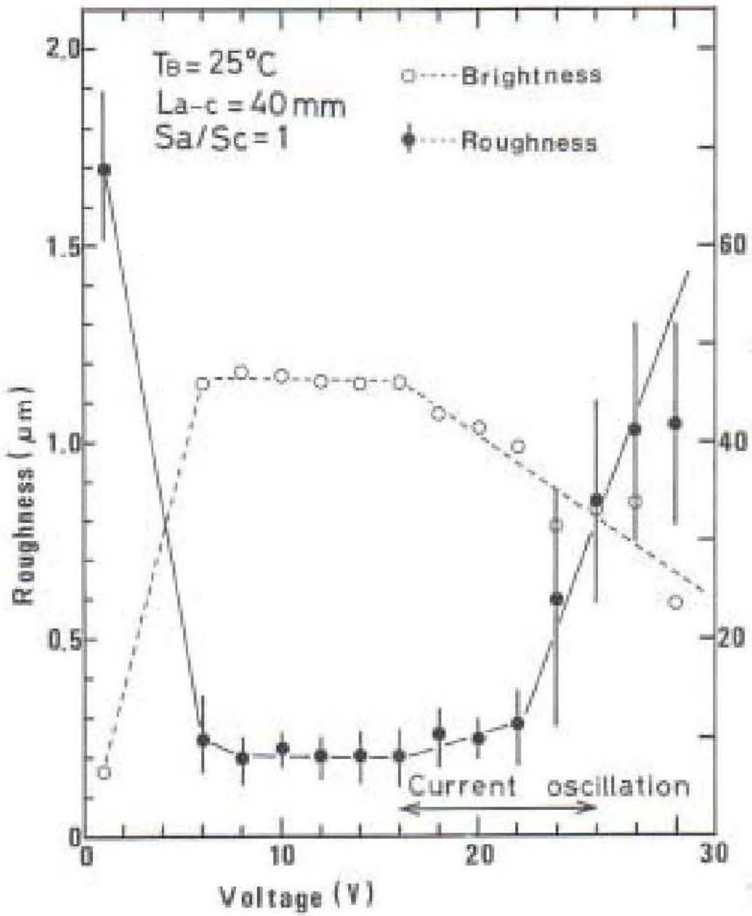
EP Finishing

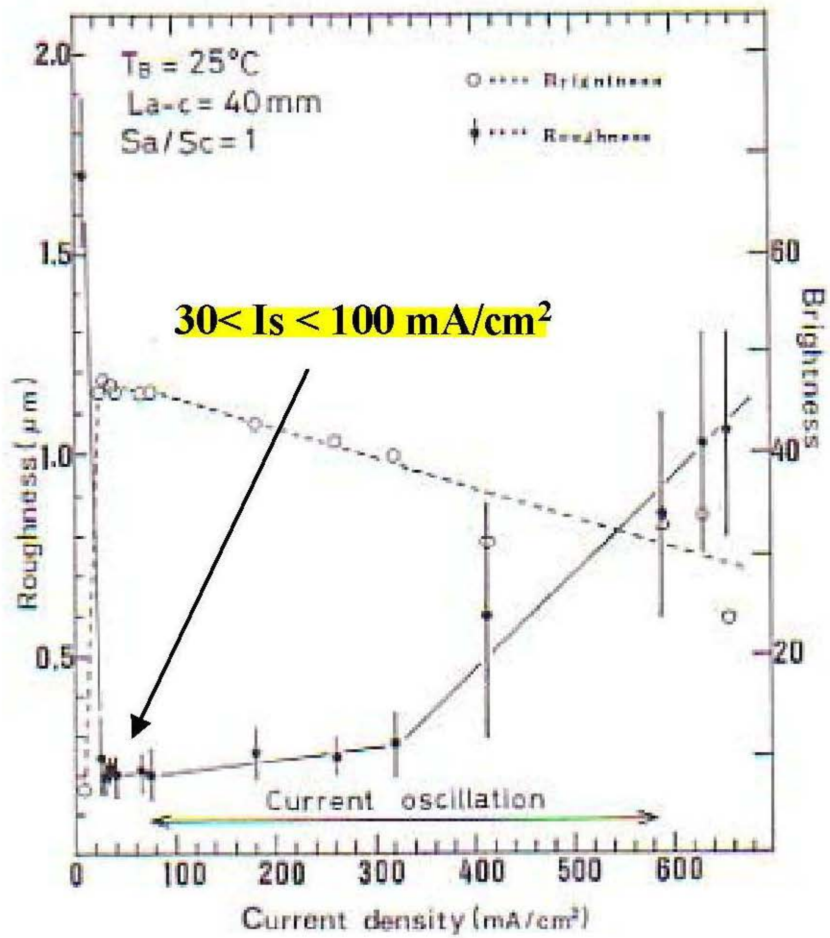




1) The final roughness depends on the initial roughness.
2) The roughness goes down as the exponential function to the removal.
3) Steps are not created along the grain-boundary. This causes smooth surface.
4) If voltage is switched off, the process stops. The control of process is easier than BCP.

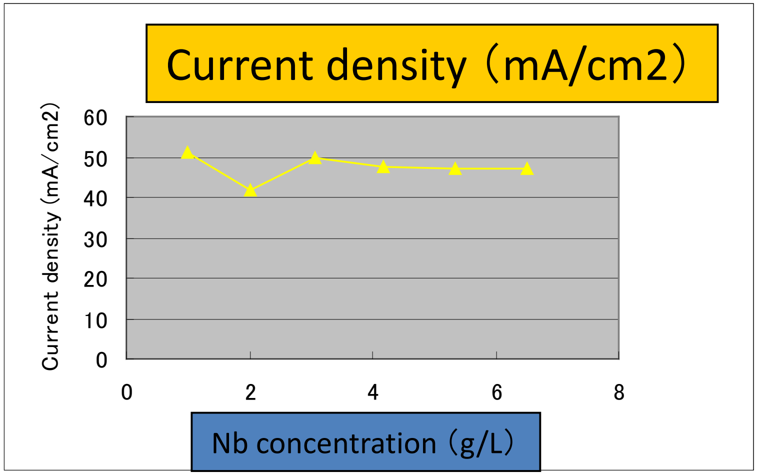
What is the curtail EP parameter?

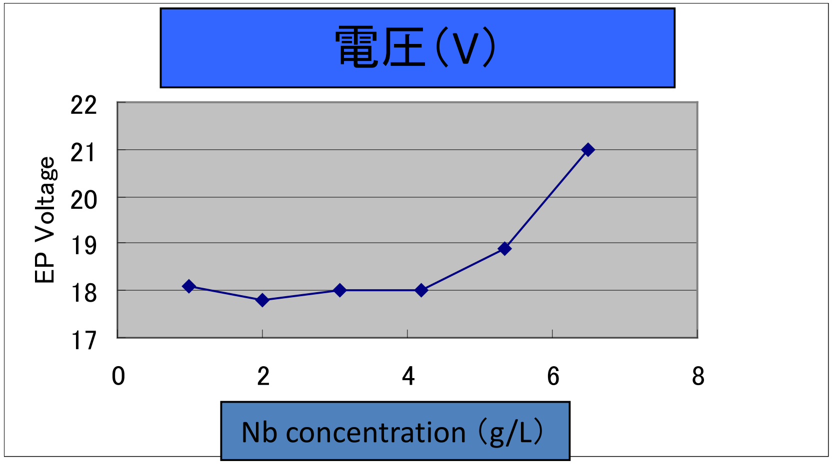




Voltage or Current density ? This is a coupled problem.

Nb concentration in EP electrolyte





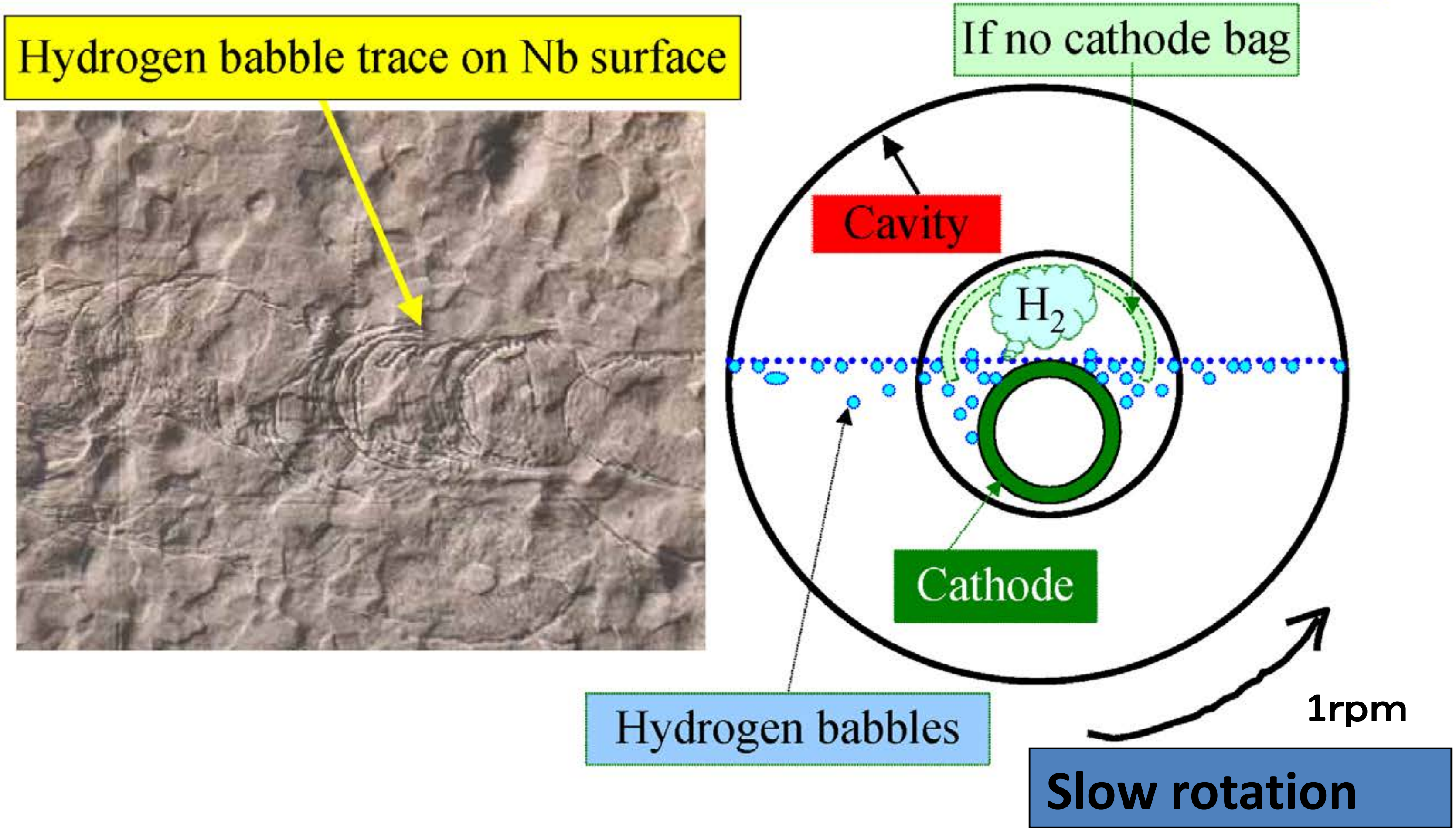
Repeating EP processes under the constant current density(= 50mA/cm^2).

If Nb concentration increased beyond 9 g/L, the surface finish is going to be rough. And sulfur contamination increase also.

＝＞If this difficulty is overcome, the cost of mass-production can be lowered.

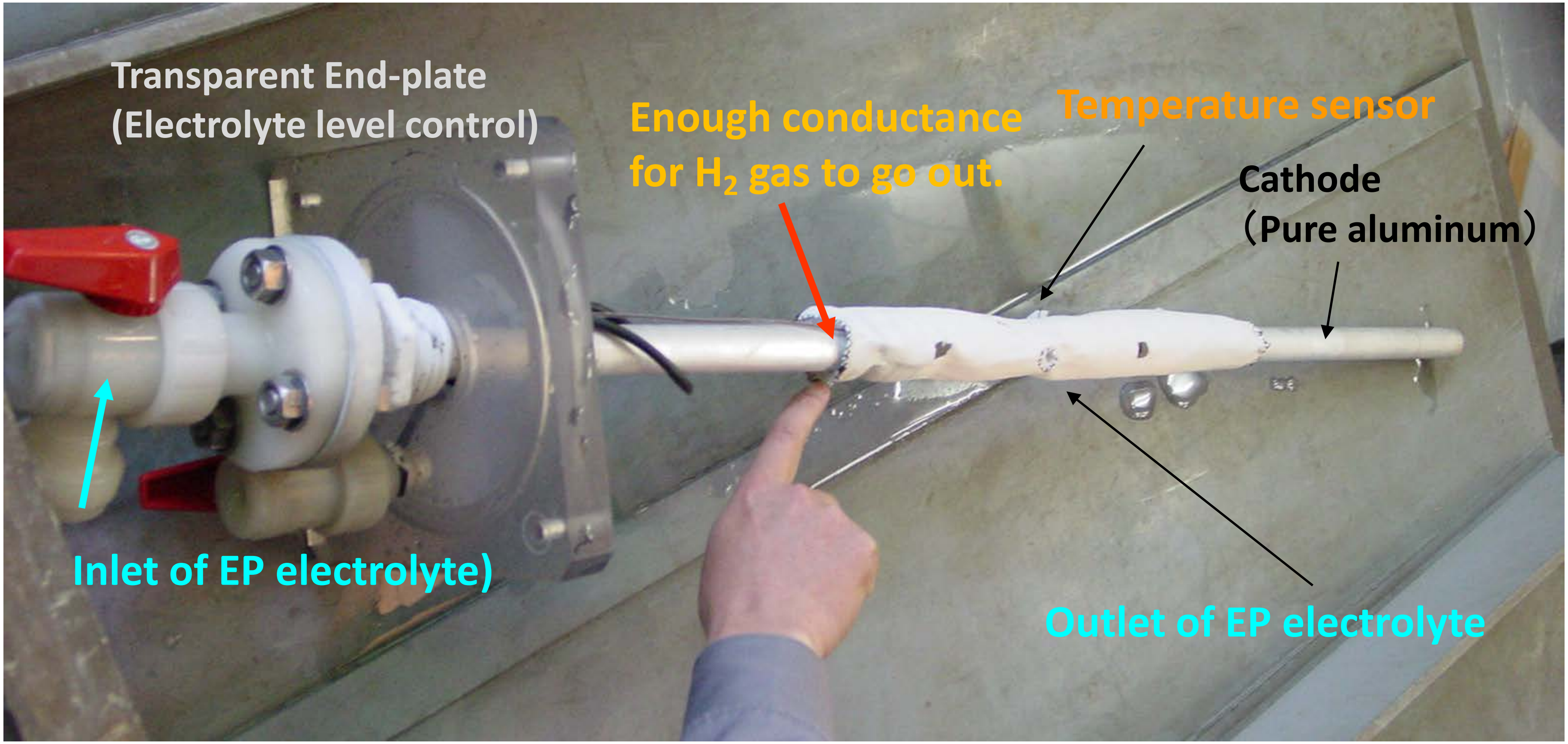
In this experiment, the voltage increased with the increase of Nb concentration.

Cathode Bag

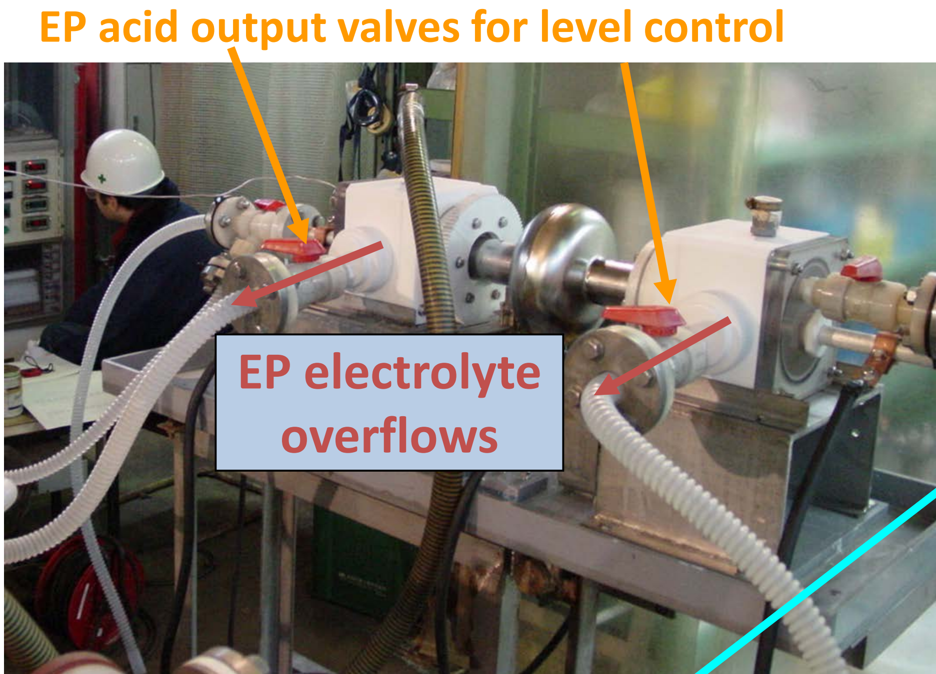


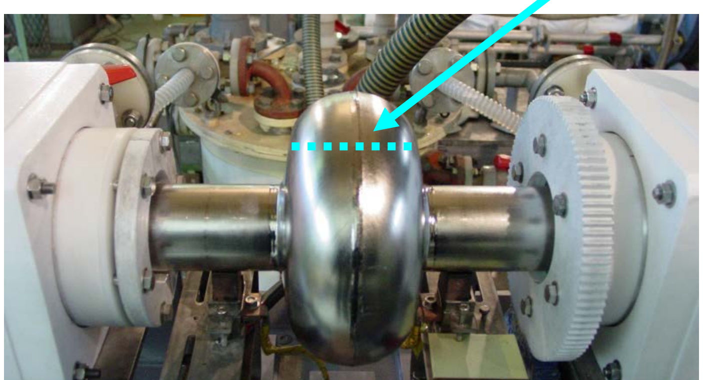
Hydrogen-gas bag should be set on the cathode appropriately. Otherwise, hydrogen-gas goes into the inner wall of cavity and causes Q-disease (low Q from low gradient).

Cathode and H2 Gas Bag

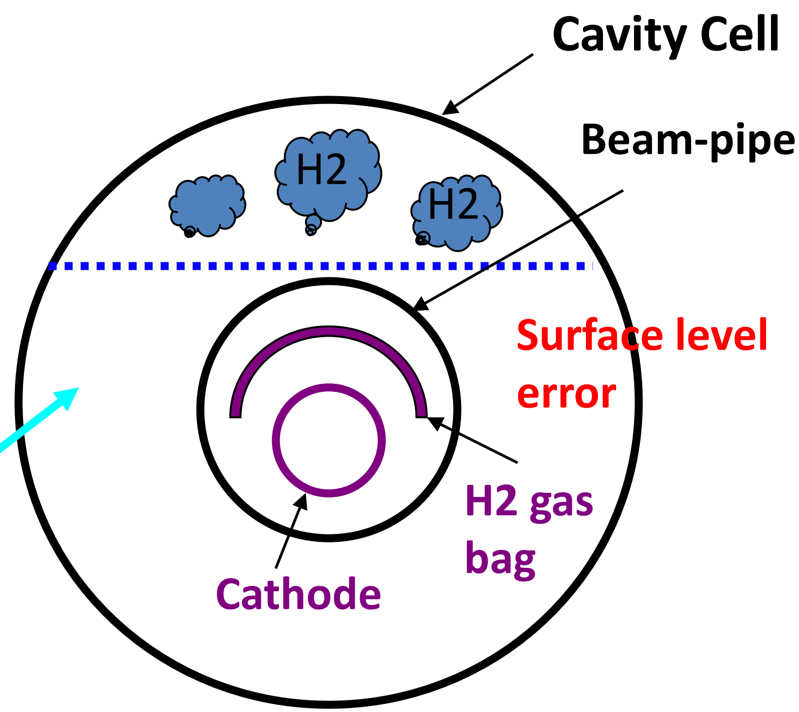


Level control of EP electrolyte





Surface level error



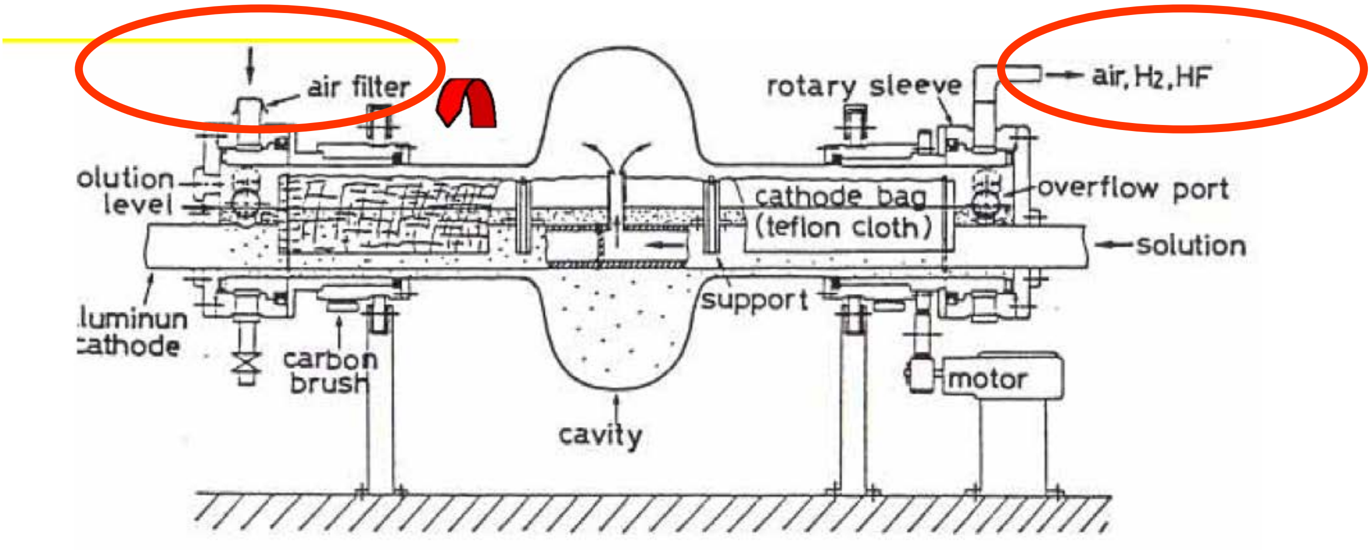
Failure of surface level of EP acid causes accumulation of H_2 gas in the cell.

↓

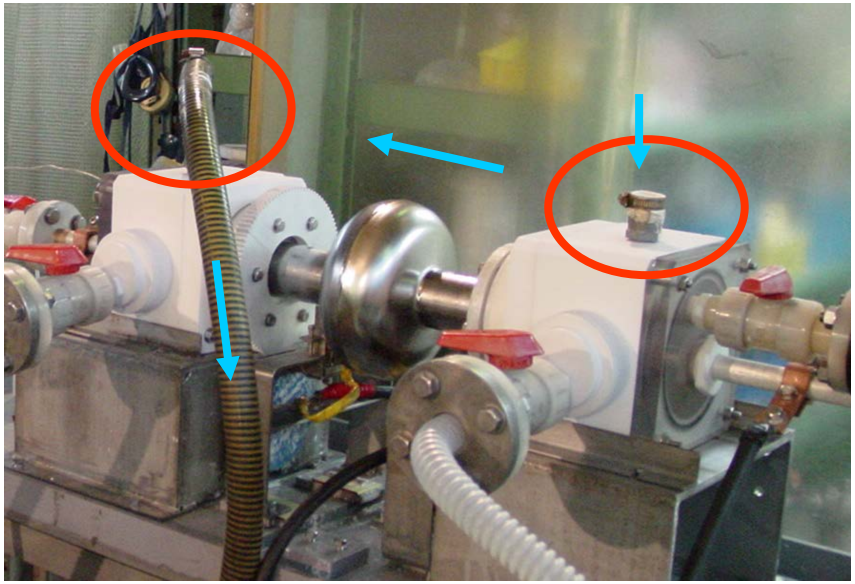
Q-disease

(Low Q from low gradient)

Exhaust/Suction of Hydrogen Gas

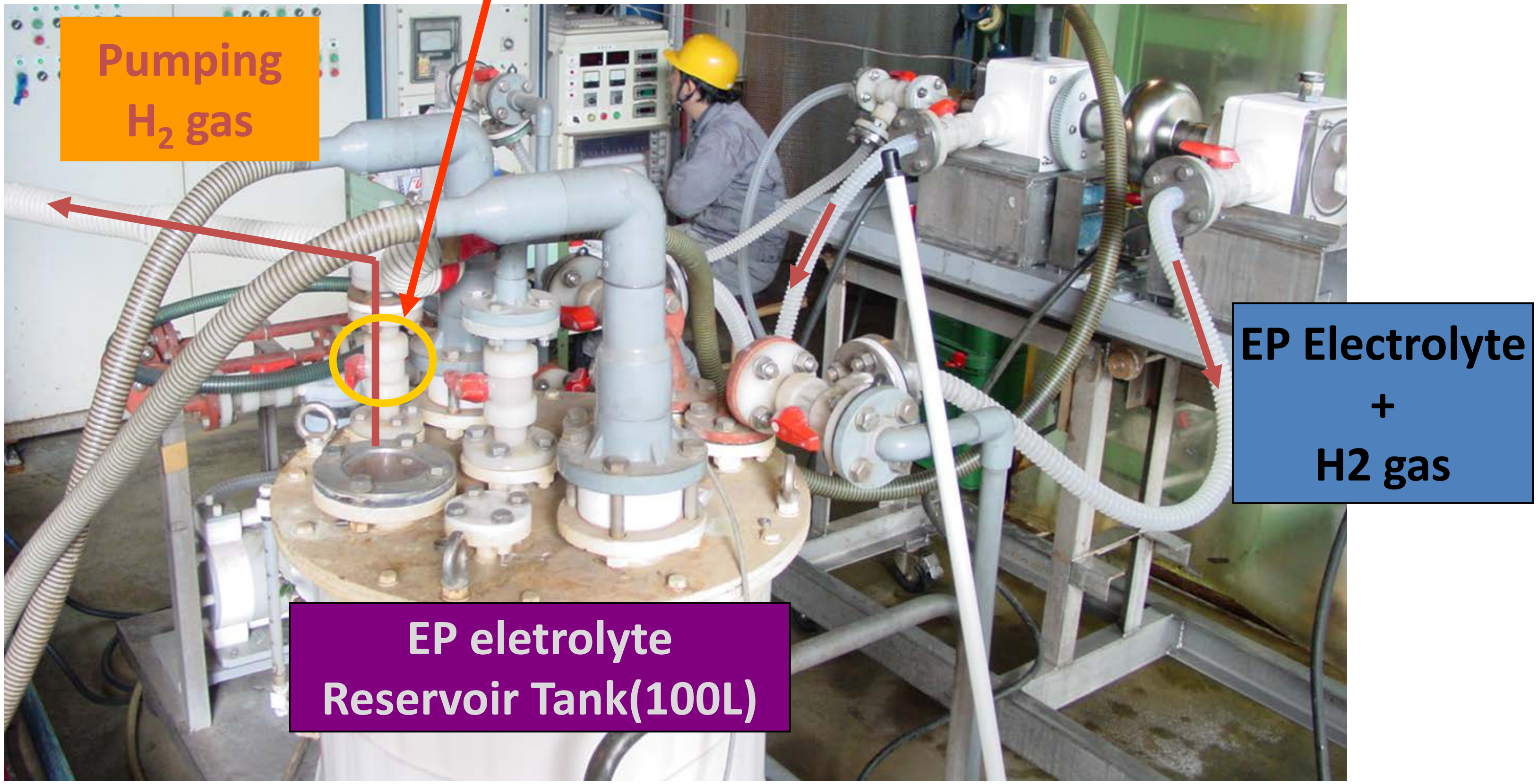


Exhaust/Suction of H_2 gas

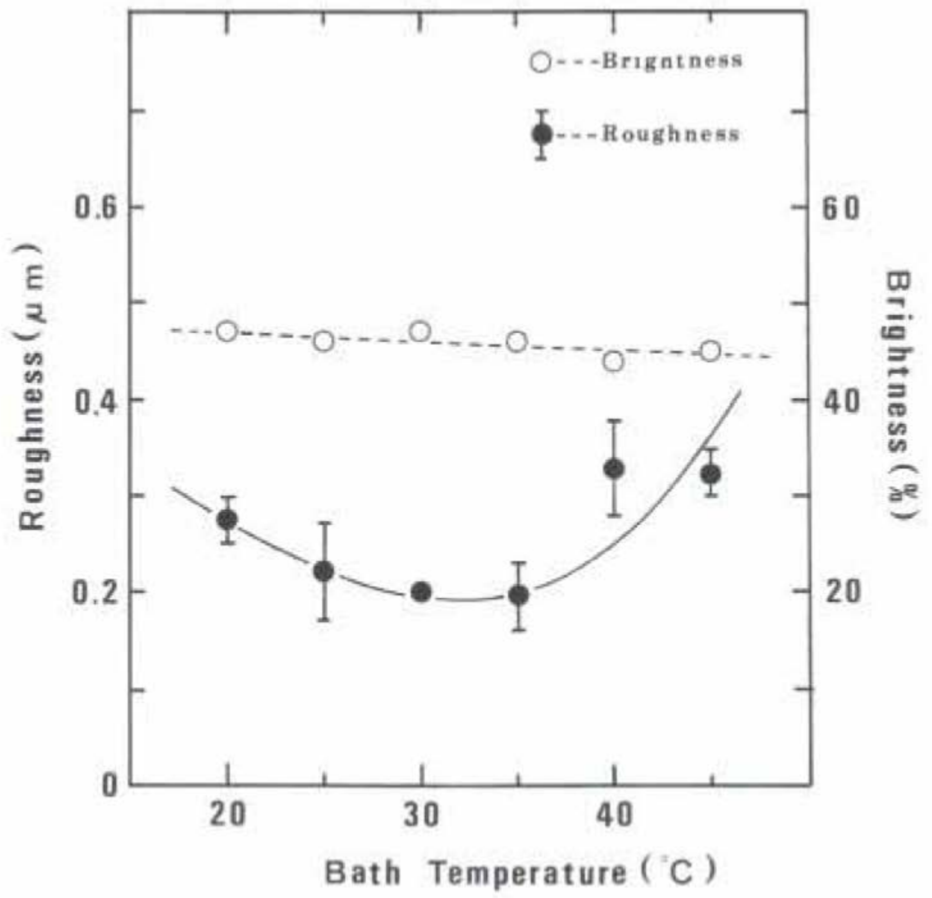


Evaporation of HF from EP electrolyte

This valve is open during the EP process in order to exhaust hydrogen gas.
But after EP process finished, this valve should be closed.
If you keep this valve open, the HF evaporates and you lose HF concentration in EP electrolyte.



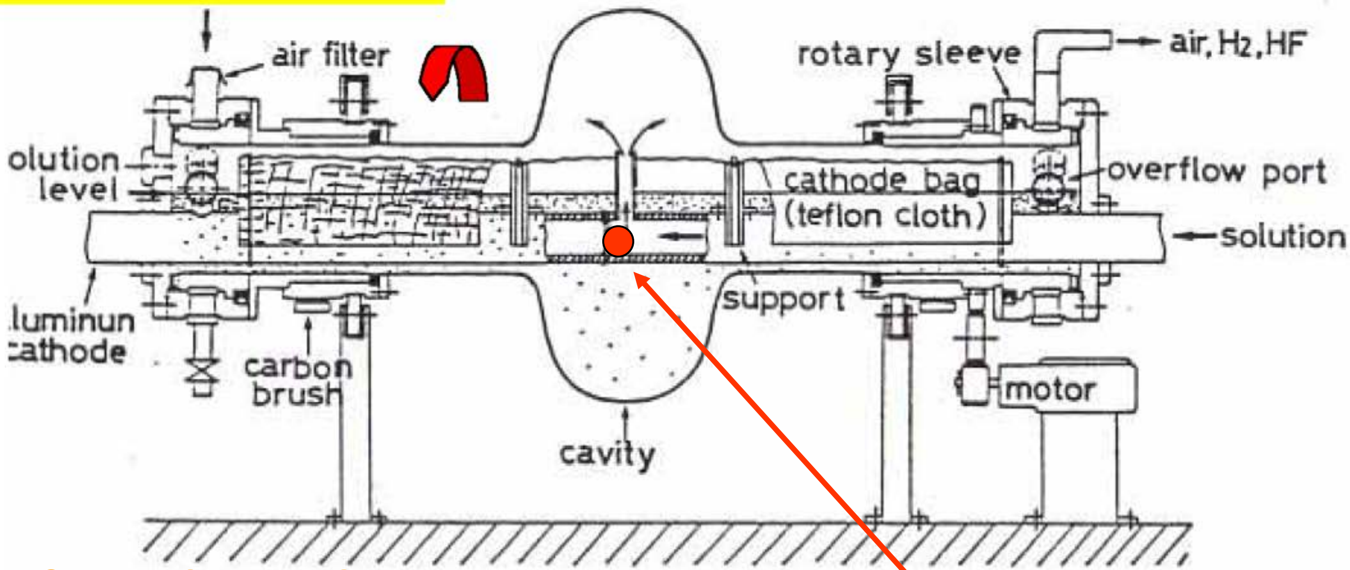
Temperature Effect on EP Finishing



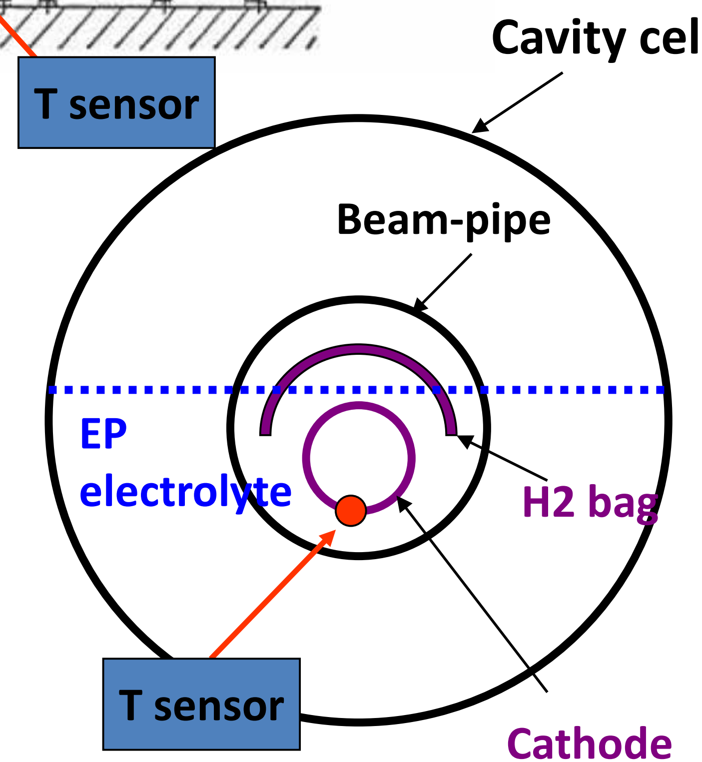
25 < T < 35 ^{O}C

T > 35 deg. C

=> Higher risk for Q-disease



Control of EP electrolyte temperature T inside cavity is important.



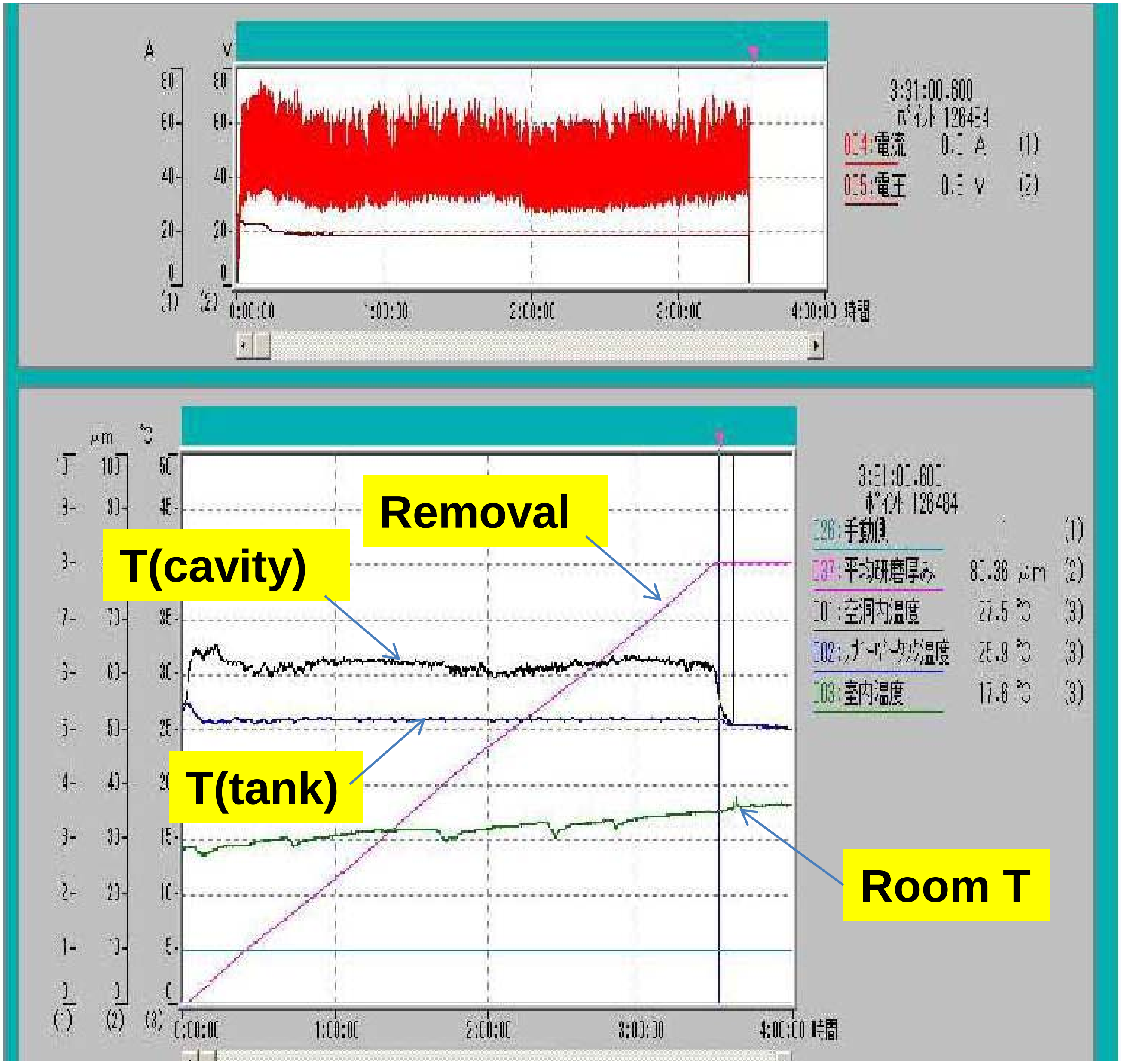
Related parameters to T (inside cavity):

V, I increase, then T(cav.) increases.

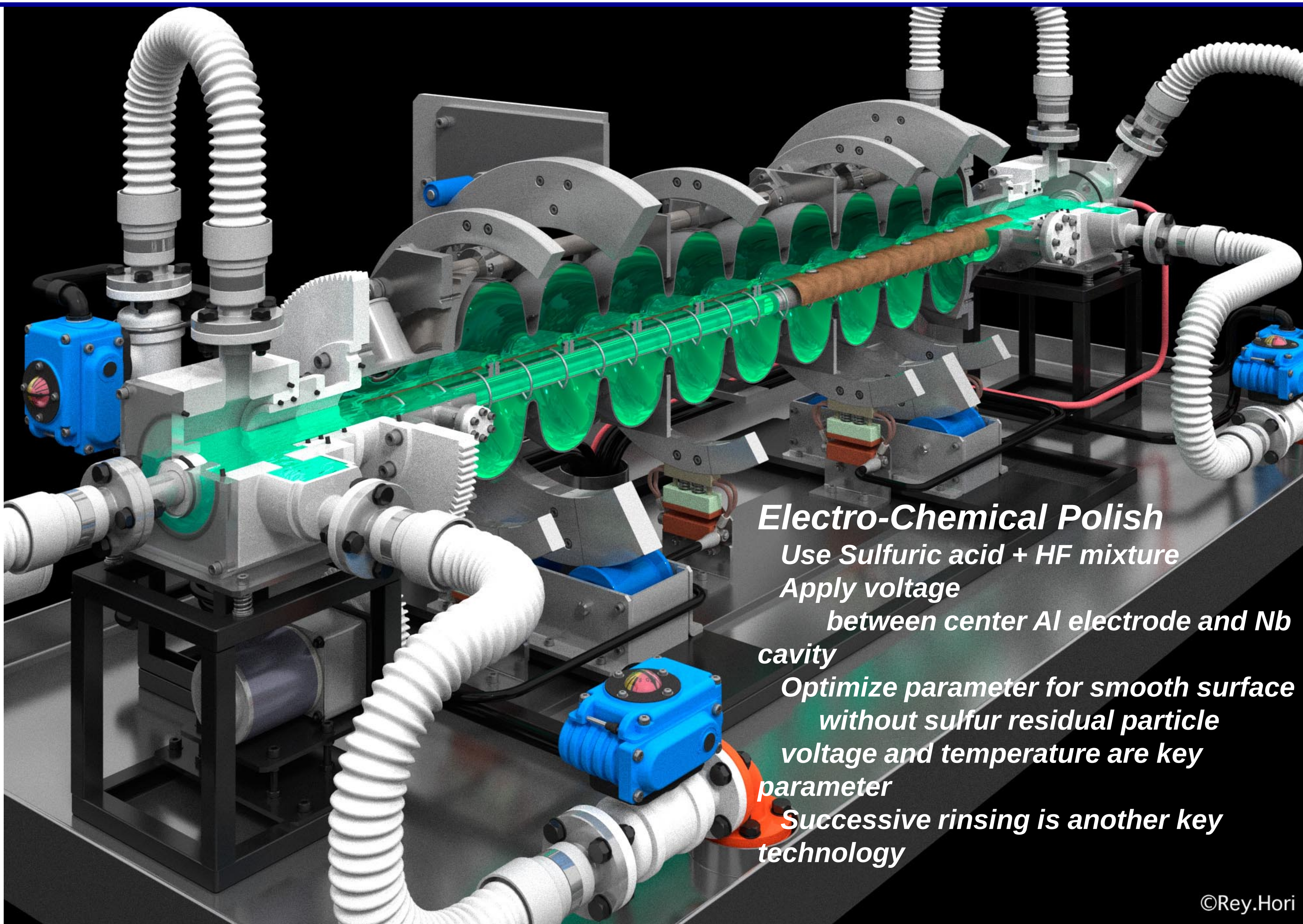
Flow rate of EP electrolyte increases, then T(cav.) decreases.

T of EP electrolyte in EP tank increases, then T(cav.) increases.

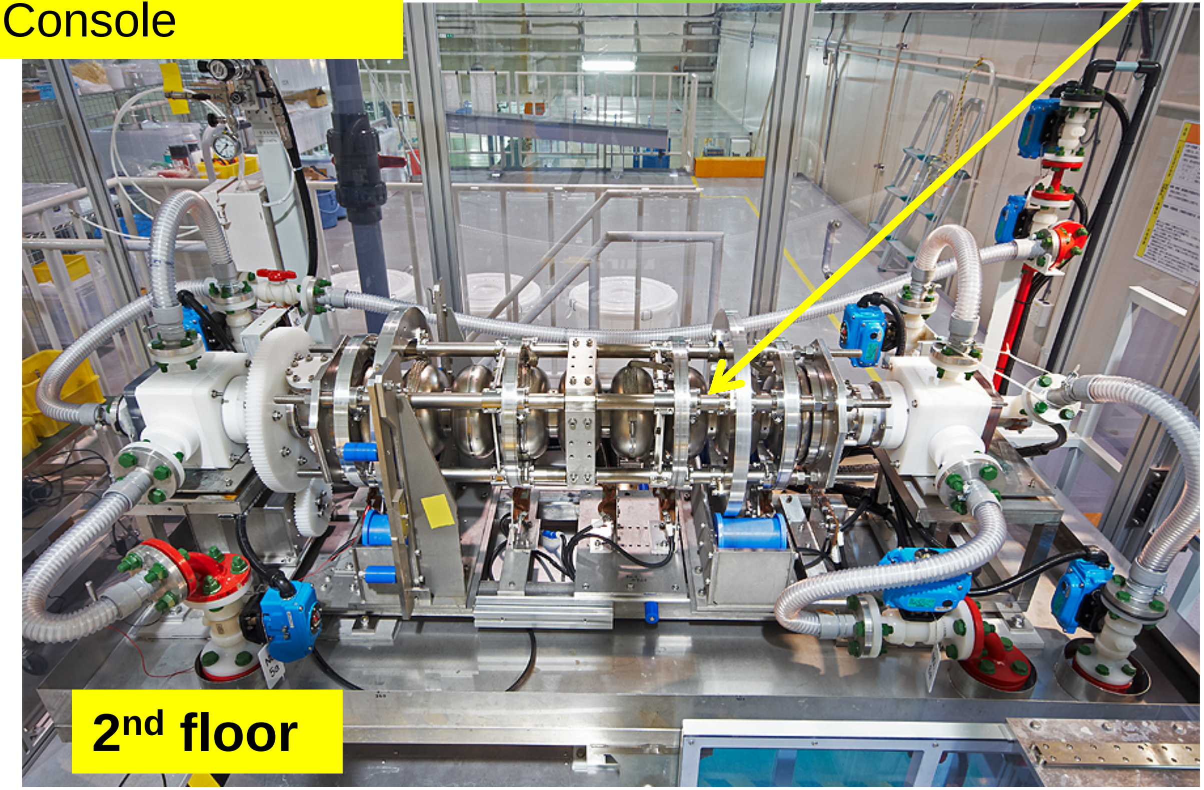
Parameters (EP)



Electro-Chemical polishing inside 9-cell cavity



Electro-polishing facility at STF/KEK

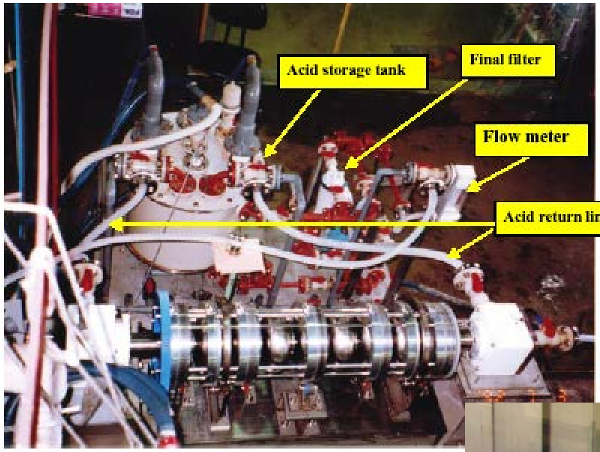


EP facility at KEK

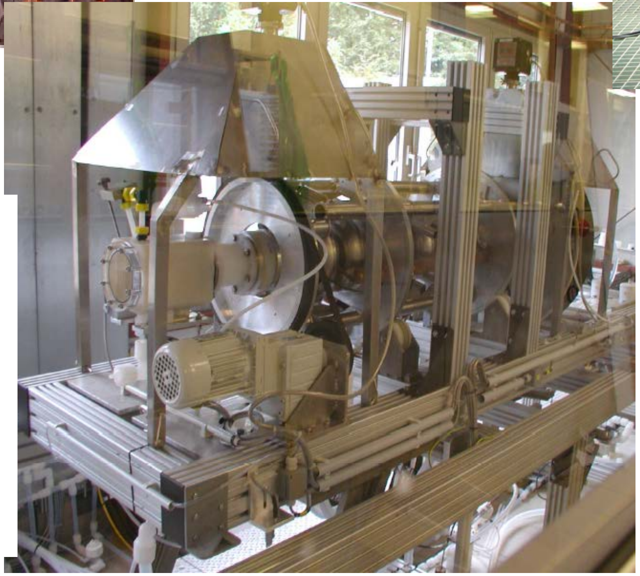
EP acid: HF + H_2SO_4, Aluminum anode,
surface removal speed: 20µm/hour, V ~18V, I ~270A, T ~30degC (for 9-cell),
cavity rotation: 1 rpm.

Various EP systems in the world

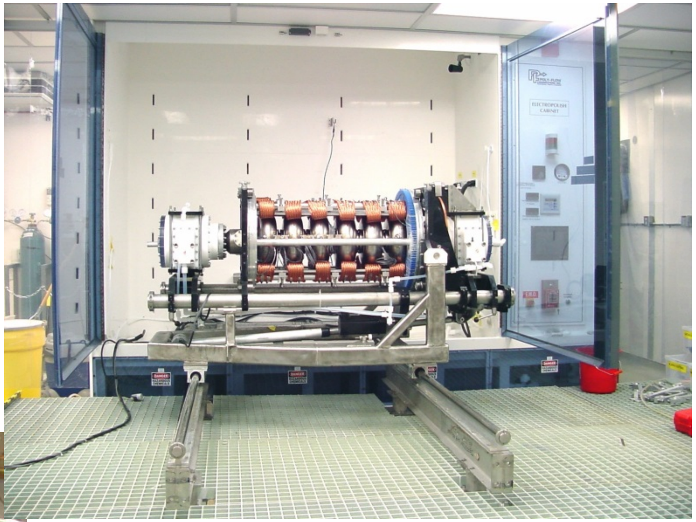
KEK/Nomura Plating



DESY

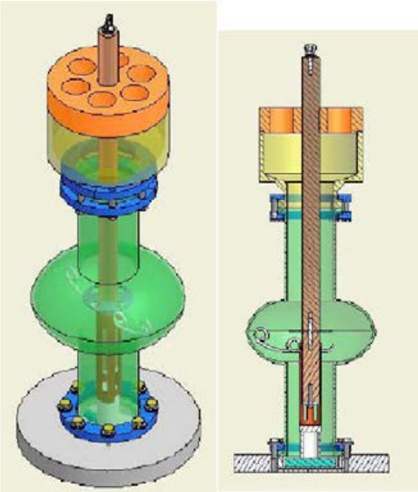


JLab



INFN

Cornell

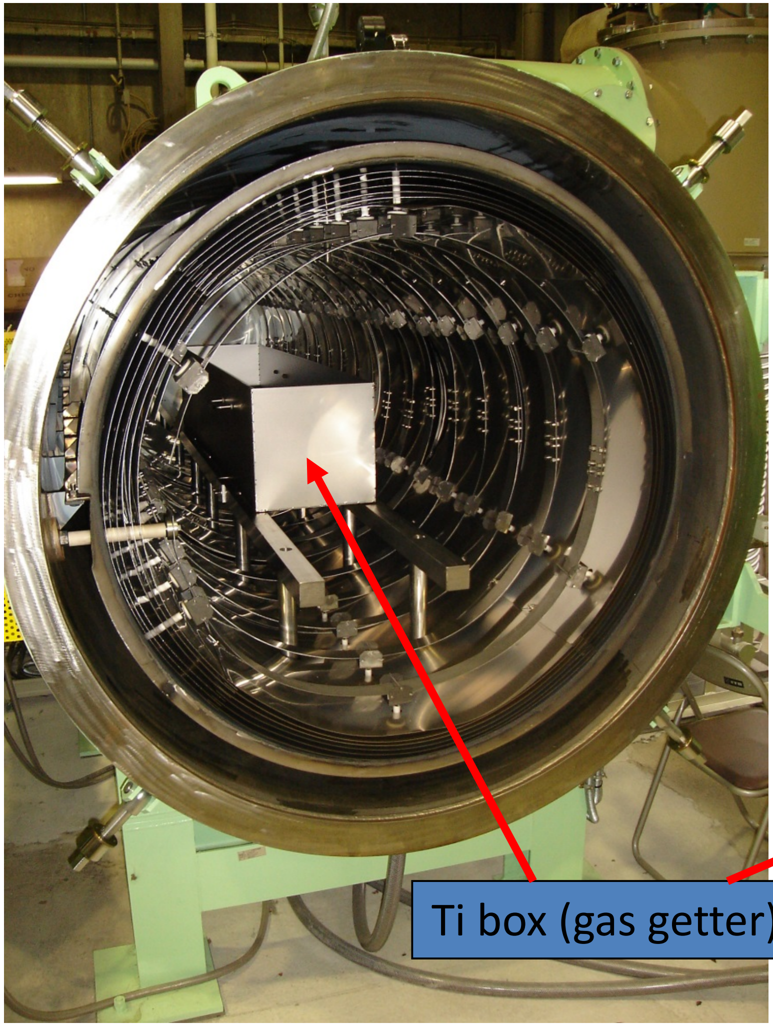


Annealing / Degassing

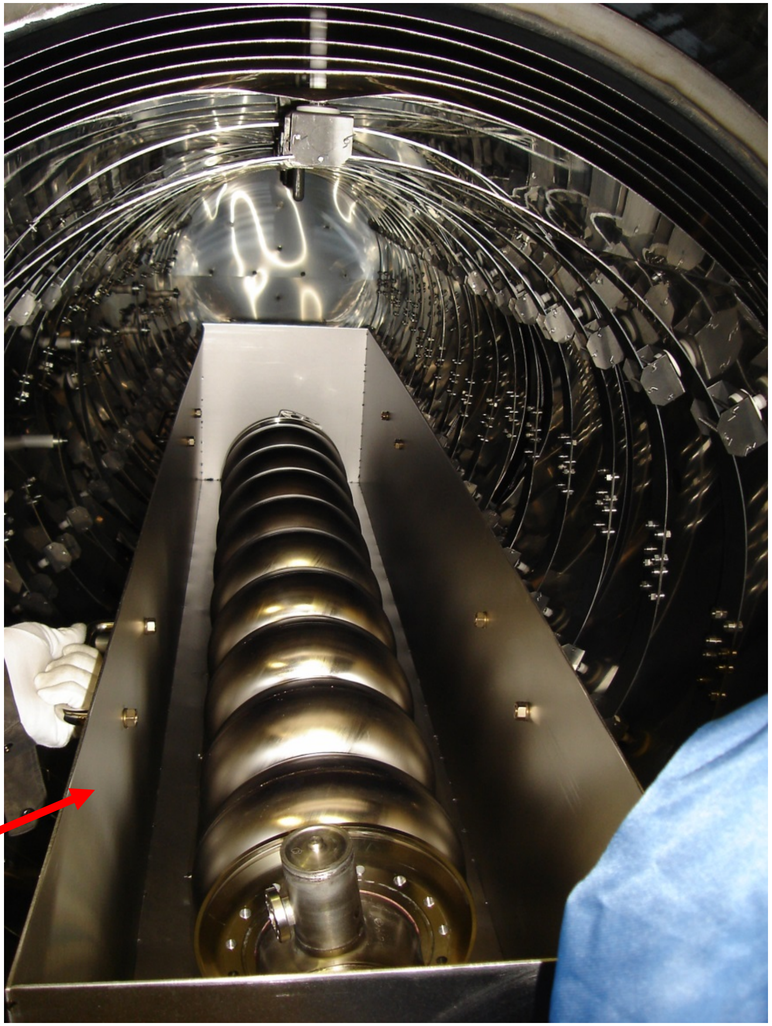
Annealing / degassing furnace at KEK：Two 9-cell cavity can be processed at once. Designed to consider about the supper-structure (Super-structure is consisting of two connected 9-cell cavities with one input-coupler).

Annealing / Degassing

Vacuum Furnce

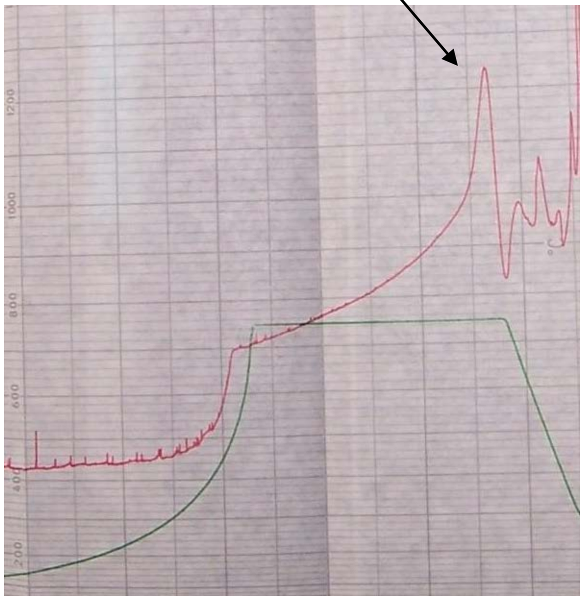


Cavity is set in a Ti box (gas getter)



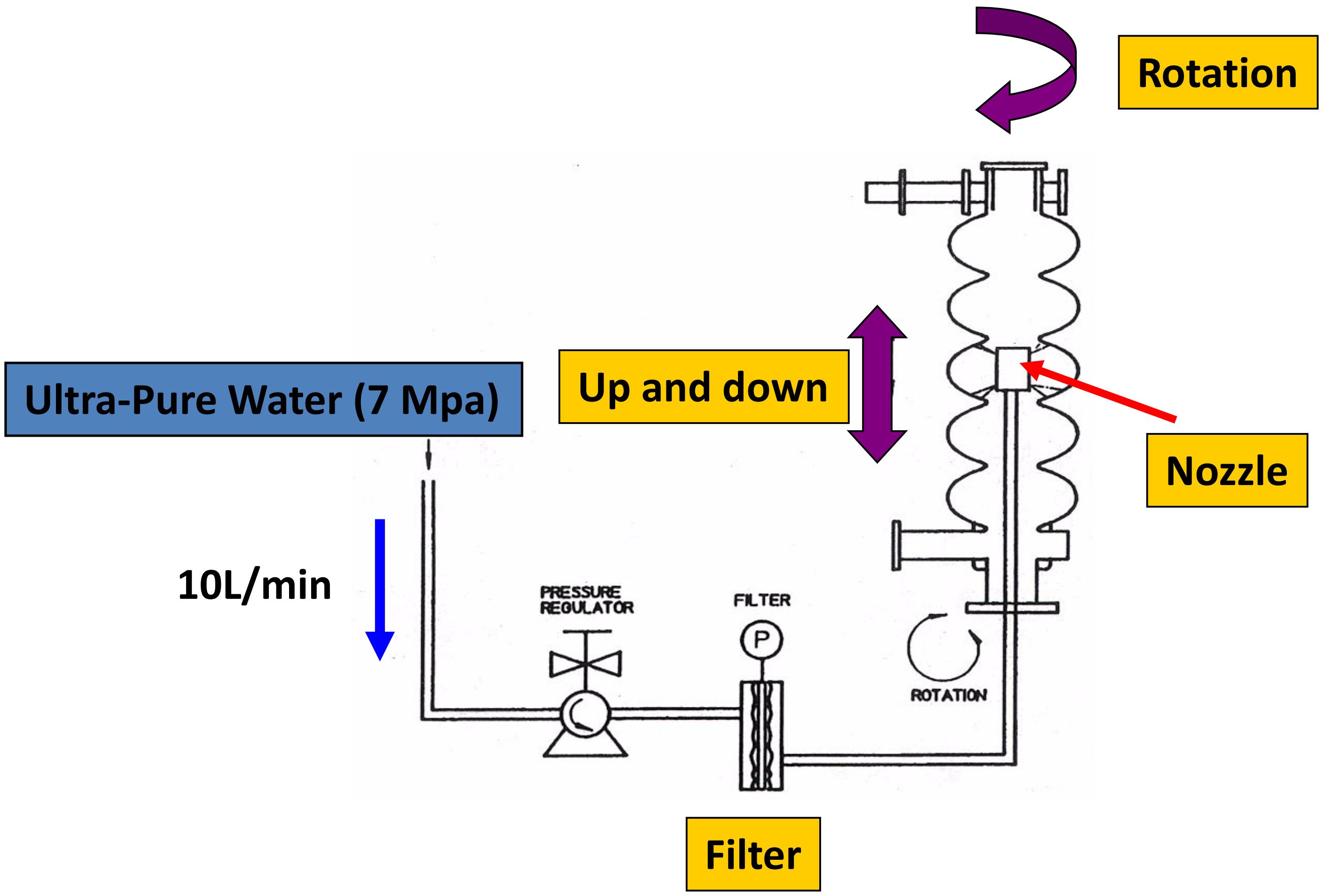
Ti box (gas getter)

Annealing / Degassing



KEK recipe : 750 ^{0}C, 3 hours.
Hydrogen gas can be degassed.
Hydrogen in the Nb material causes Q-disease that degrades the Q value.

High Pressure Rinse (HPR)



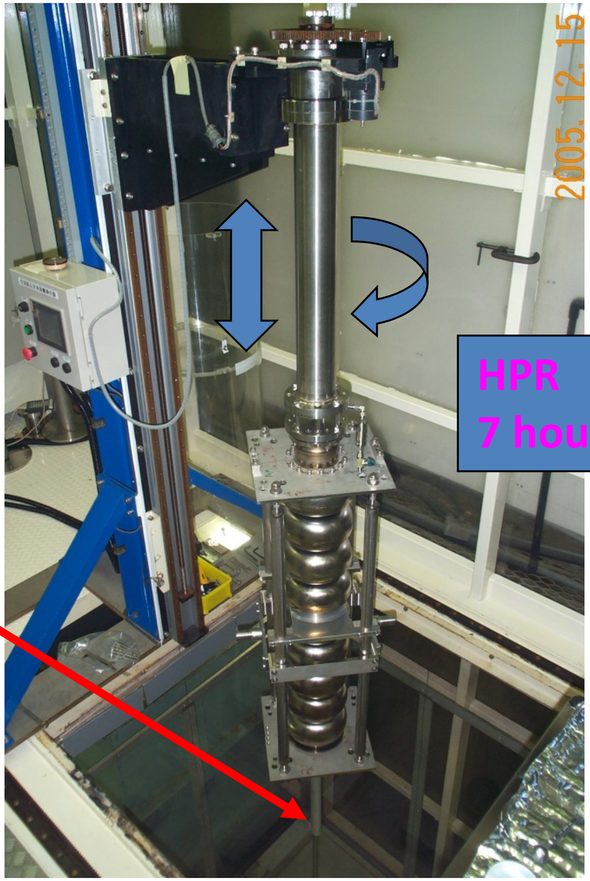
High Pressure Rinse (HPR)

Cavity

Pressure = 7 MPa

Flow rate = 10 L/min.

Nozzle



HPR
7 hours

Ultra Pure Water
Specific resistance = 18 M Ohm cm
TOC = 10 – 20 ppb
Bacteria = 0 – 3 count / mL

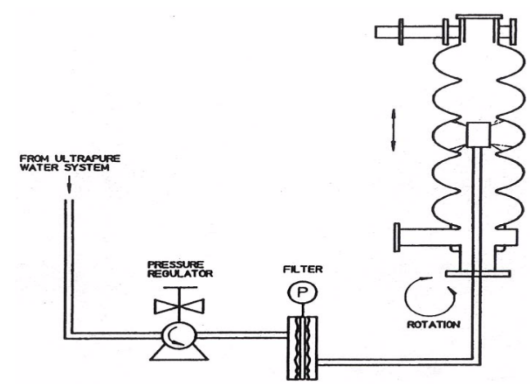
HPR is a strong tool to clean up the inside of cavity.

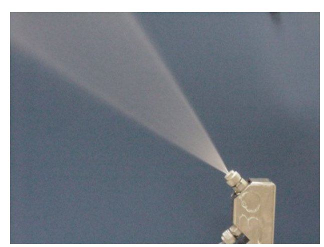
Various High Pressure Rinse (HPR) machines

DESY-System

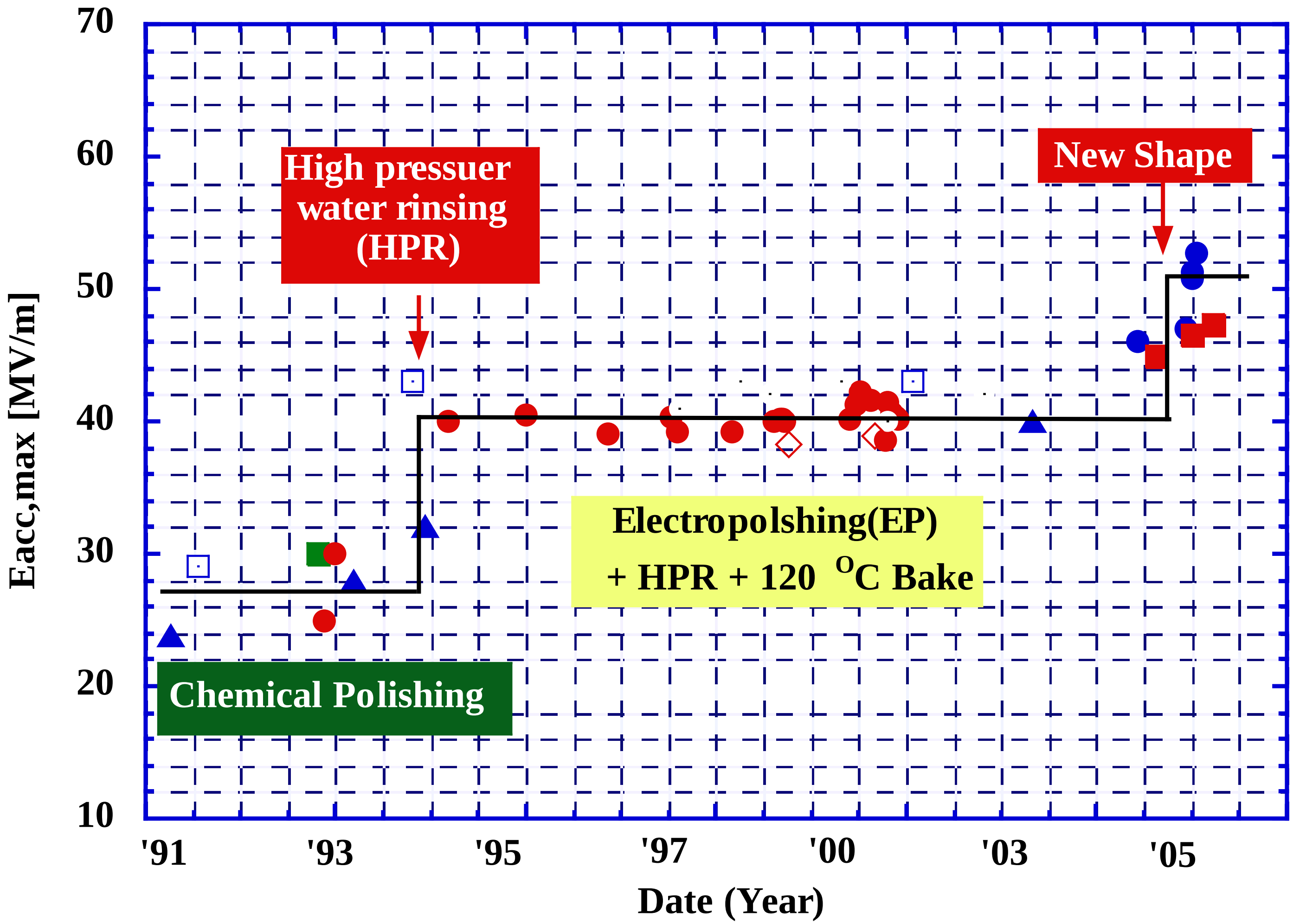
Jlab HPR Cabinet

KEK-System





Breakthrough by HPR



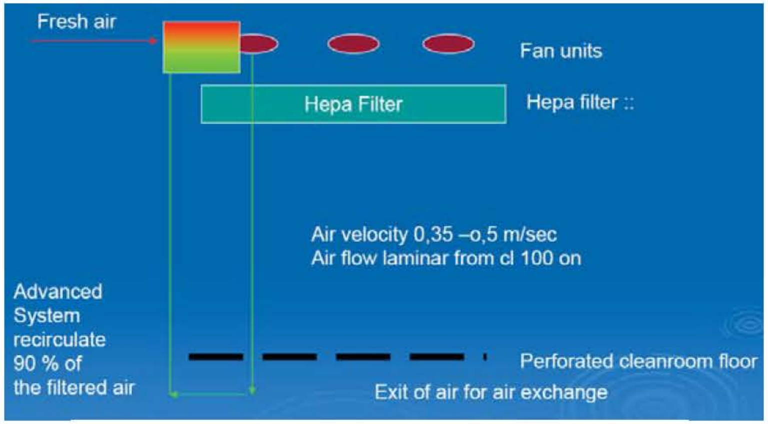
Assembly in Clean Room

HEPA filter (class 100)

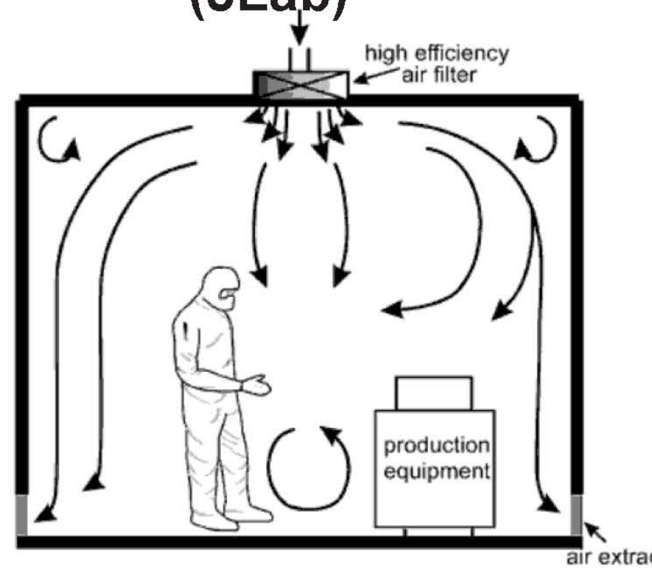
ULPA filter (class 10)

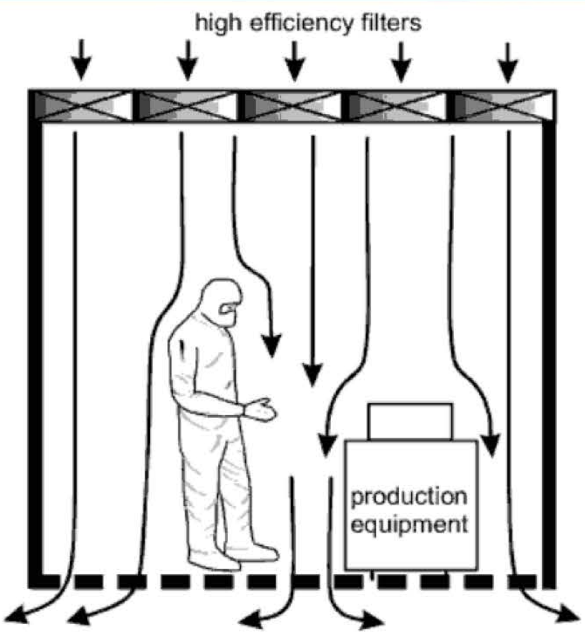
Clean-room

Type of Cleanrooms



Non-Unidirectional airflow type (JLab)





Unidirectional airflow type (DESY)

Cleanroom Classification

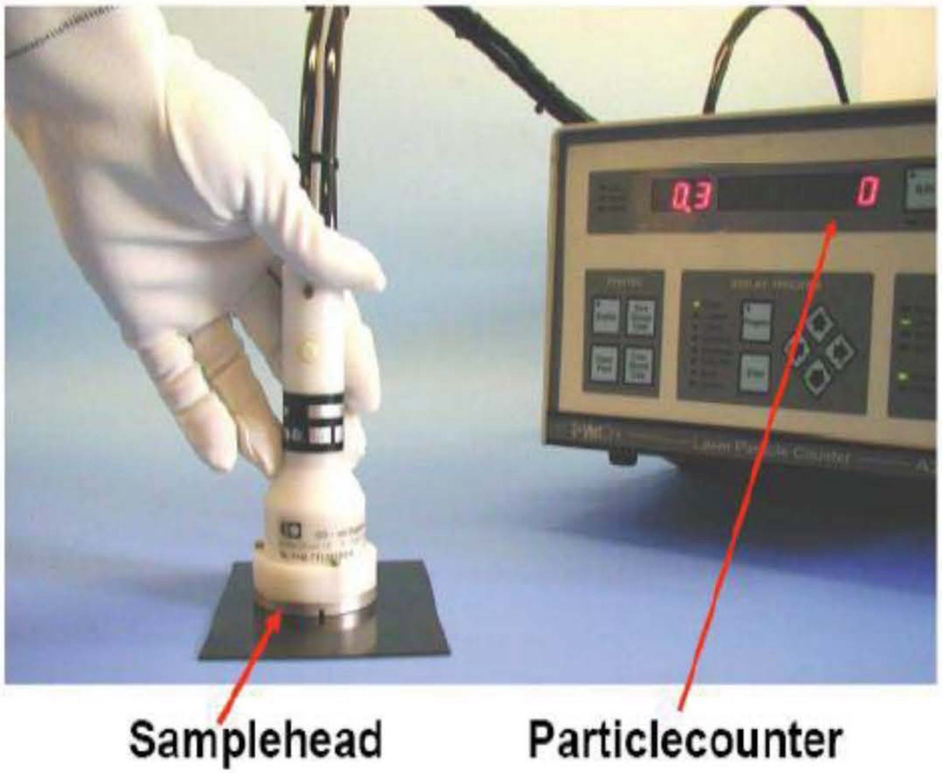
ISO Classification number	Maximum concentration limits (particles/m³ of air) for particles equal to and larger than the considered sizes shown below					
	>=0.1µm	>=0.2µm	>=0.3µm	>=0.5µm	>=1µm	>=5.0µm
ISO Class 1	10	2				
ISO Class 2	100	24	10	4		
ISO Class 3	1 000	237	102	35	8	
ISO Class 4	10 000	2 370	1 020	352	83	
ISO Class 5	100 000	23 700	10 200	3 520	832	29
ISO Class 6	1 000 000	237 000	102 000	35 200	8 320	293
ISO Class 7				352 000	83 200	2 930
ISO Class 8				3 520 000	832 000	29 300
ISO Class 9				35 200 000	8 320 000	293 000

ISO 14644-1 Classes	Class 3	Class 4	Class 5	Class 6	Class 7	Class 8
FS 209 Classes	Class1	Class 10	Class 100	Class 1000	Class 10, 000	Class 100, 000

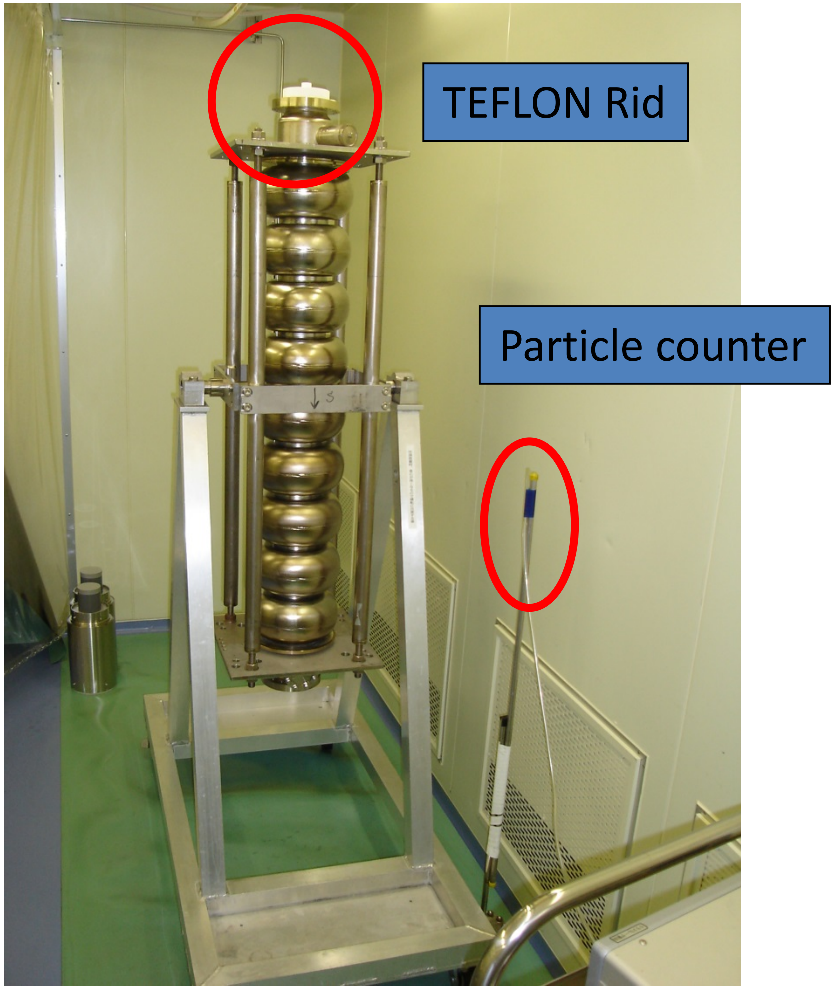
Cavity assembly (→ Class 10)

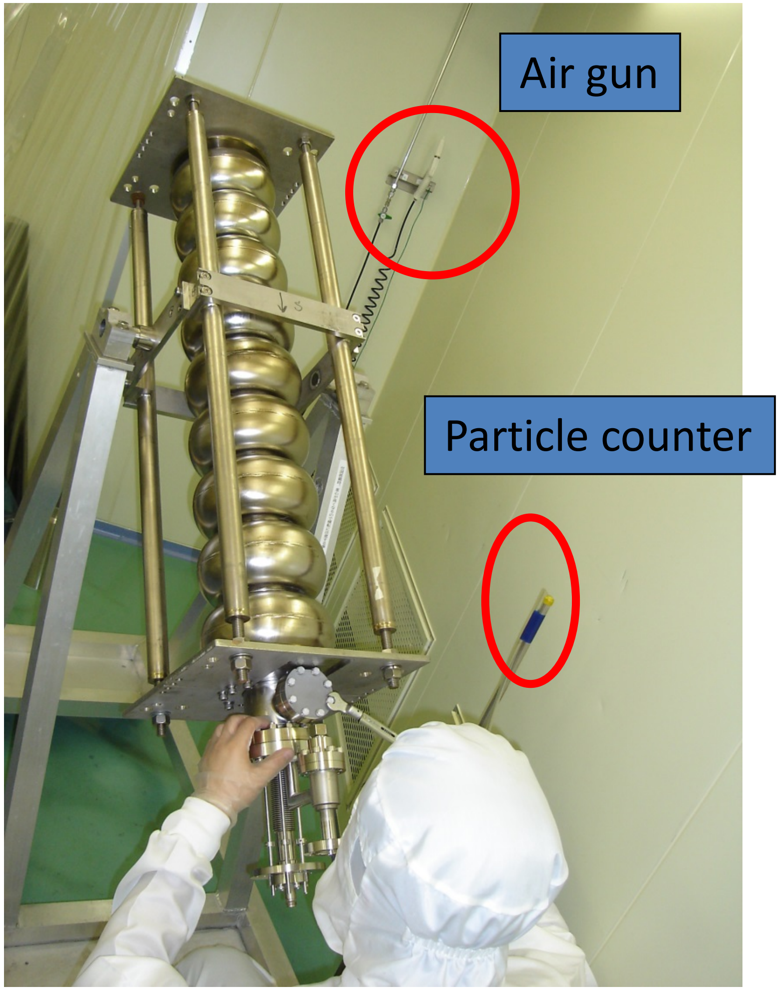
Cleanroom for SRF (→ Class 100)

Particle Counters

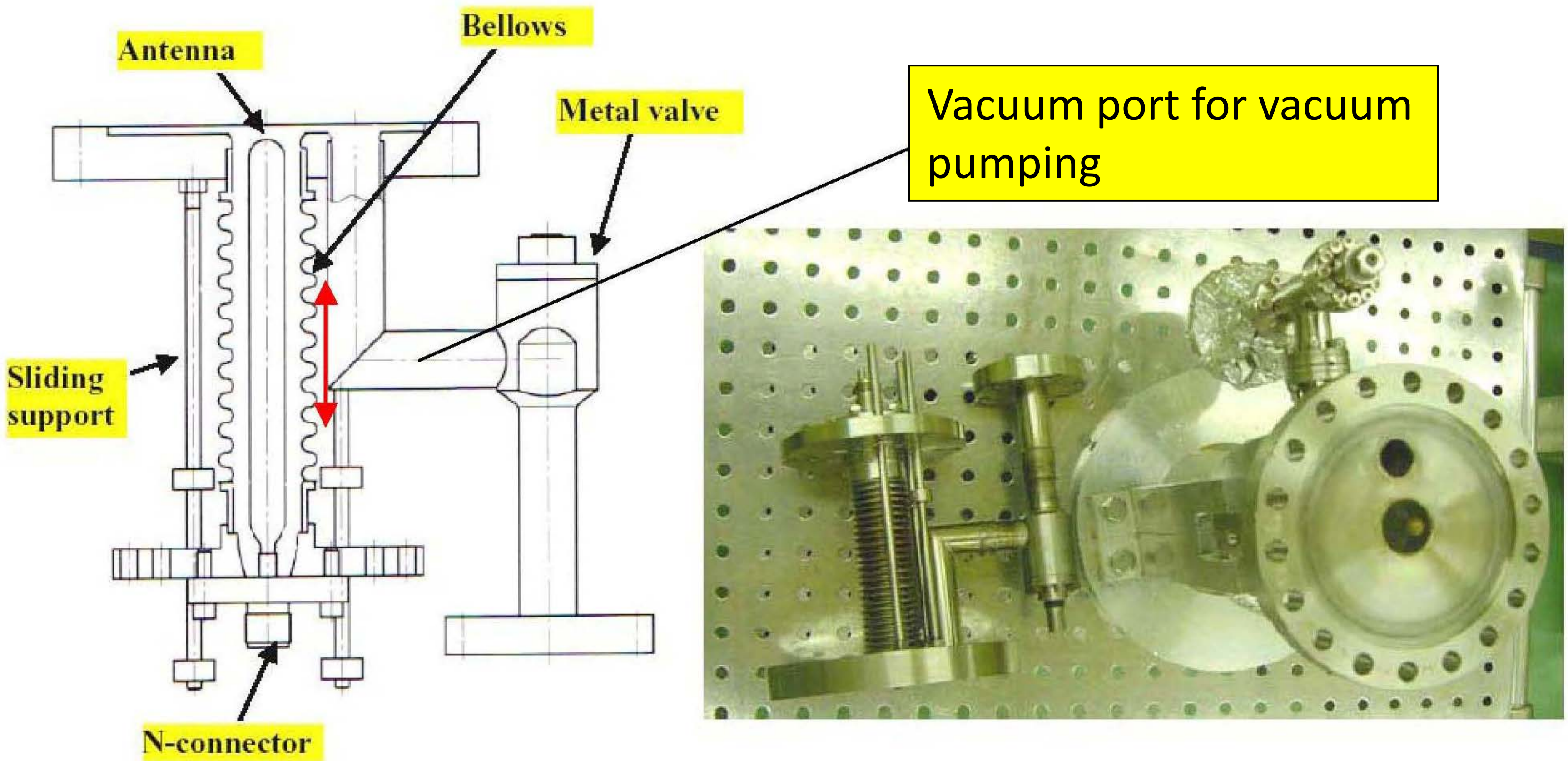


Assembly in Clean Room



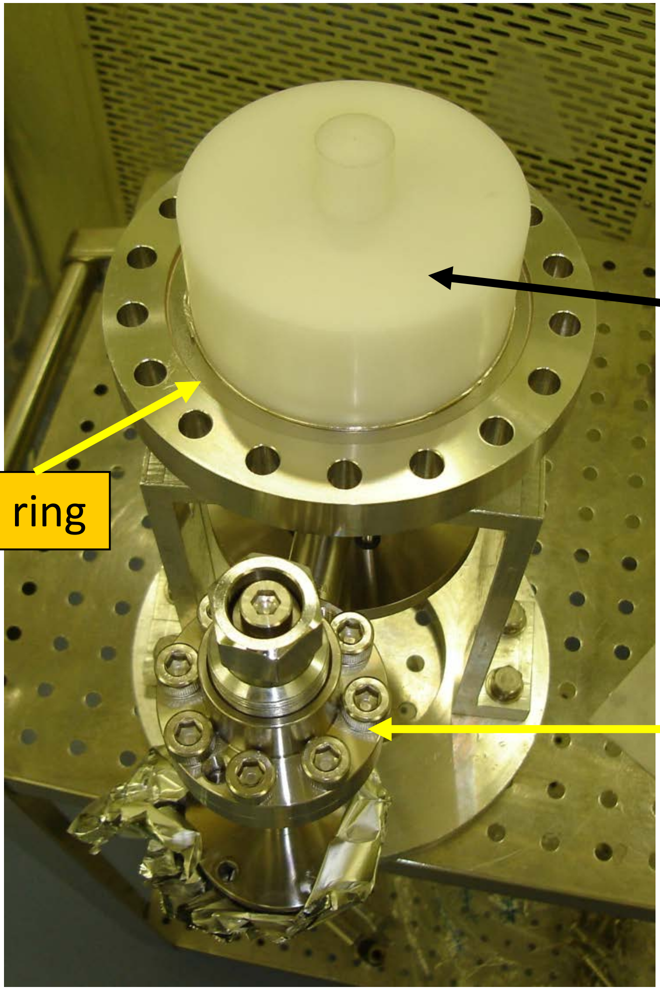


Input-coupler for RF vertical test

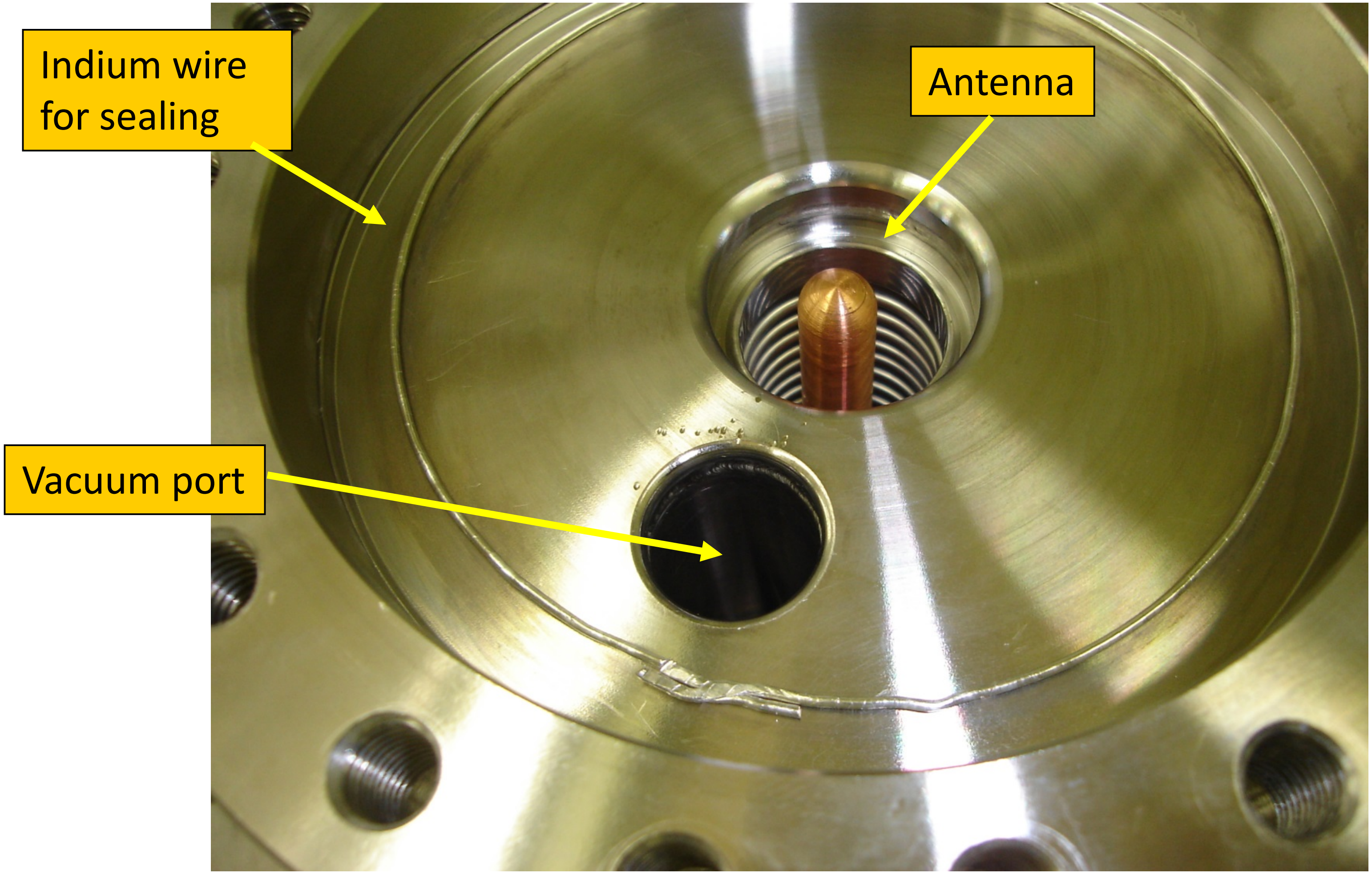


Variable input coupler for the vertical test in KEK

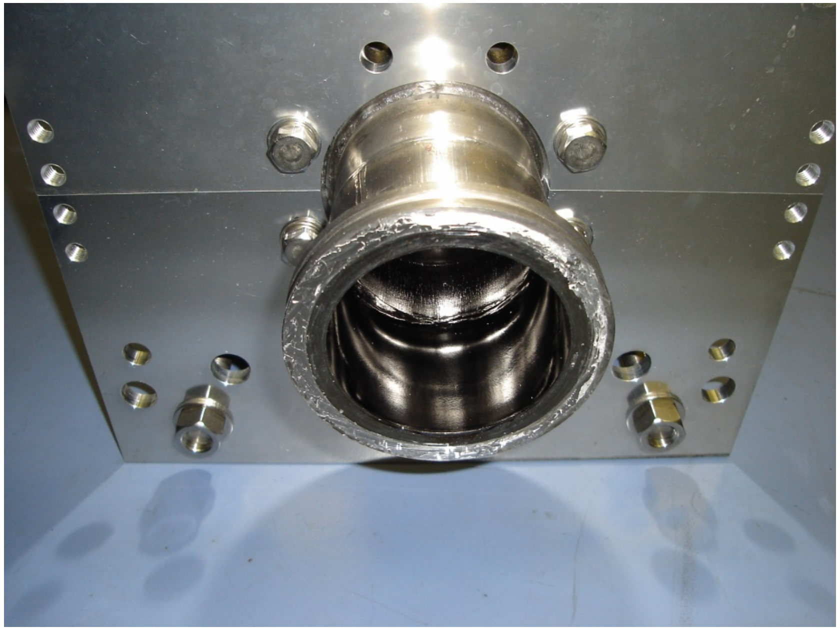
Indium Sealing

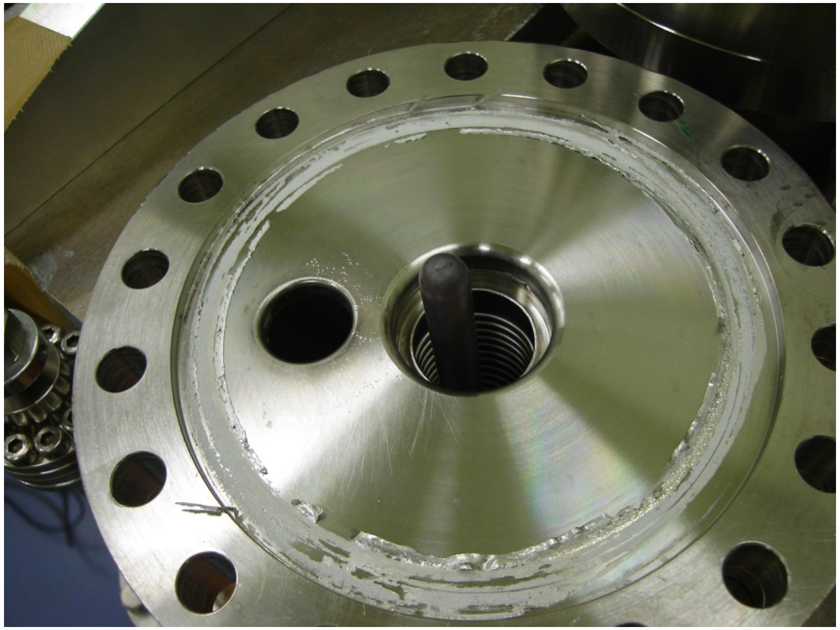


Indium Sealing



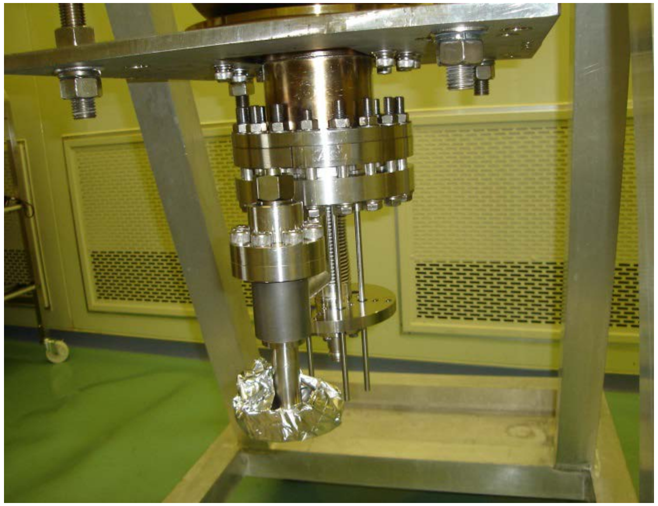
Indium Sealing



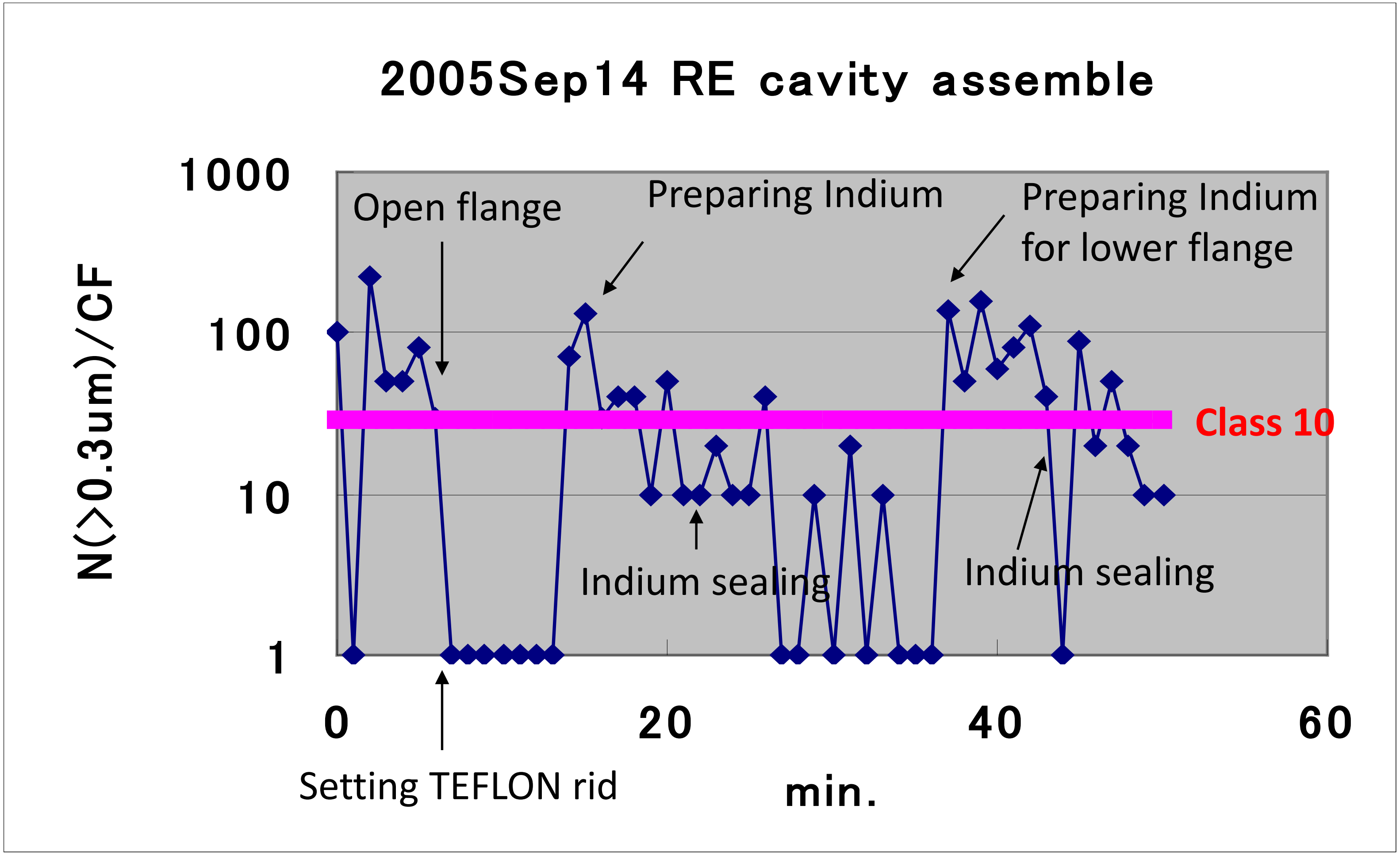


Indium is soft material. So the operation is complicated.
Even when disassembly of cavity, the removal of indium is not easy work.
And because the melting point of indium is low : T = 156 C, you should pay attention to the temperature control of In-situ baking (T=120 – 140).

Assembly in Clean Room

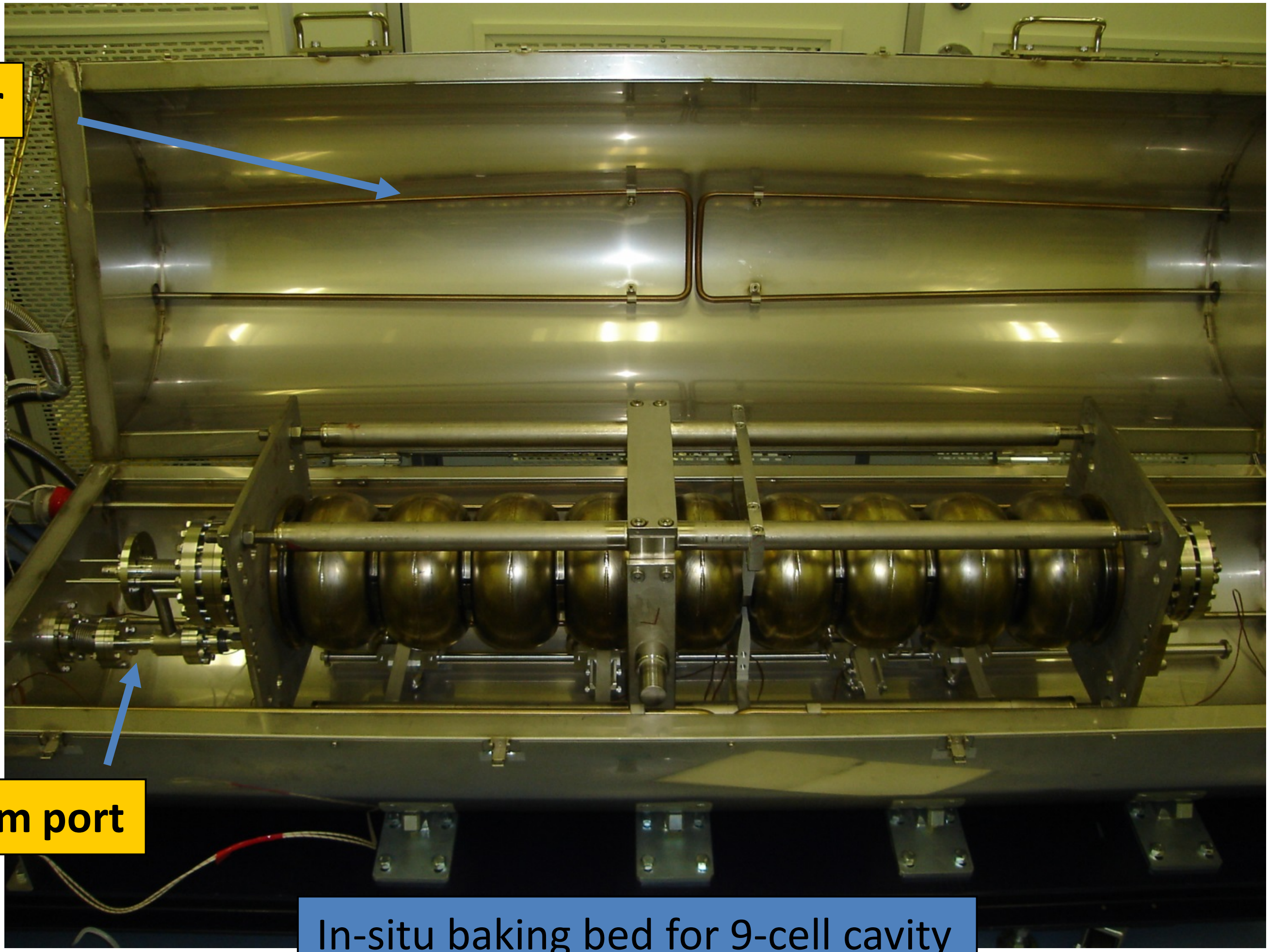


Assembly in Clean Room



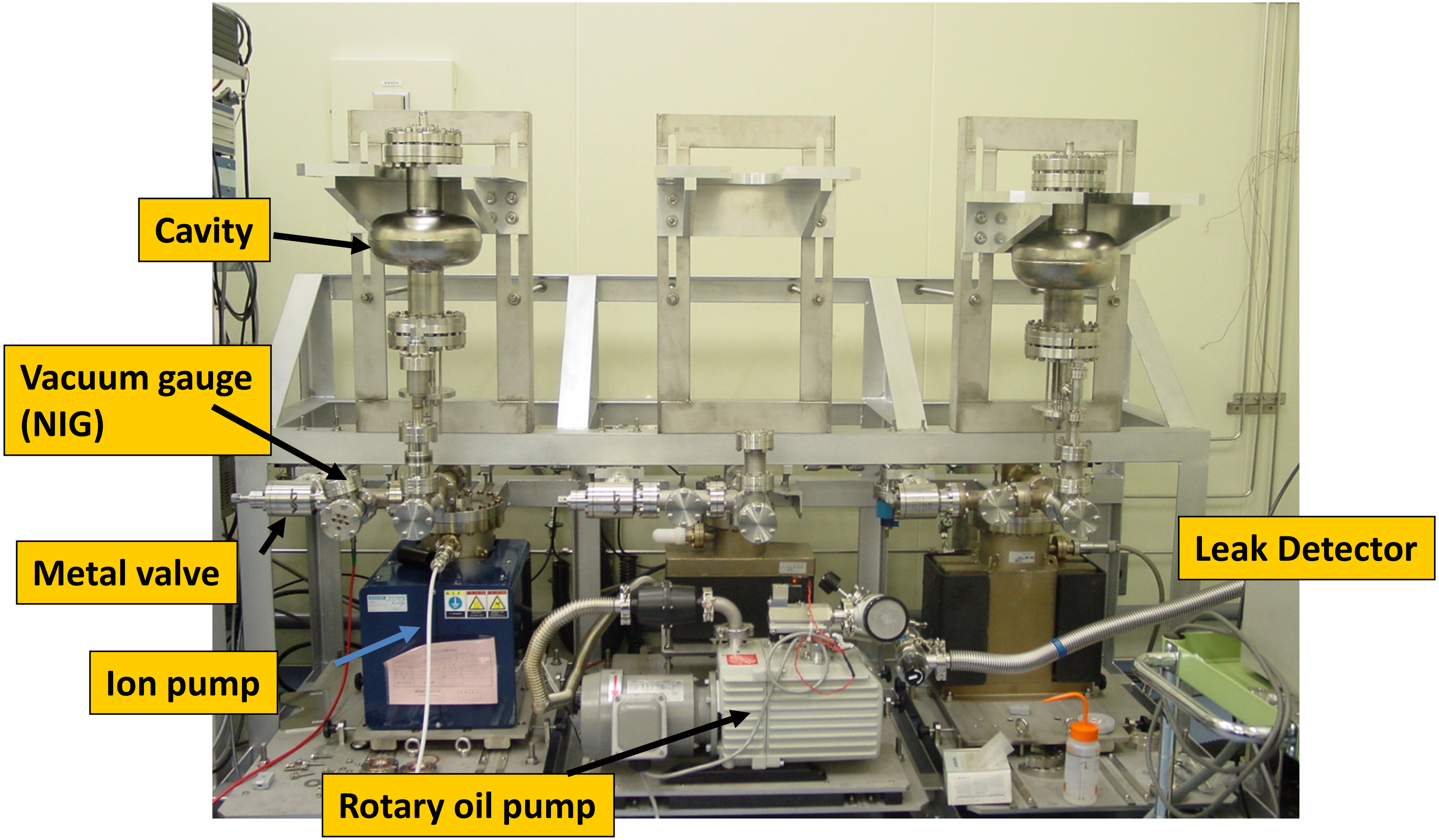
28 particles (>0.3um) / CF = class 10

In-situ Baking System for 9-cell Cavity



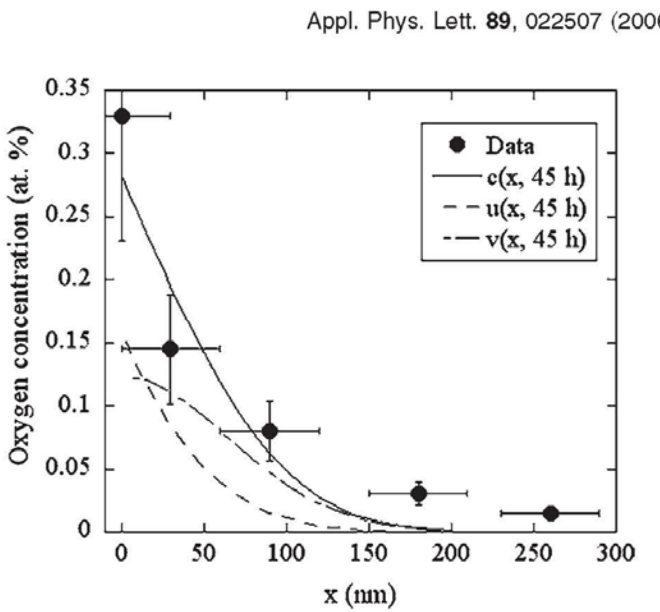
In-situ baking bed for 9-cell cavity

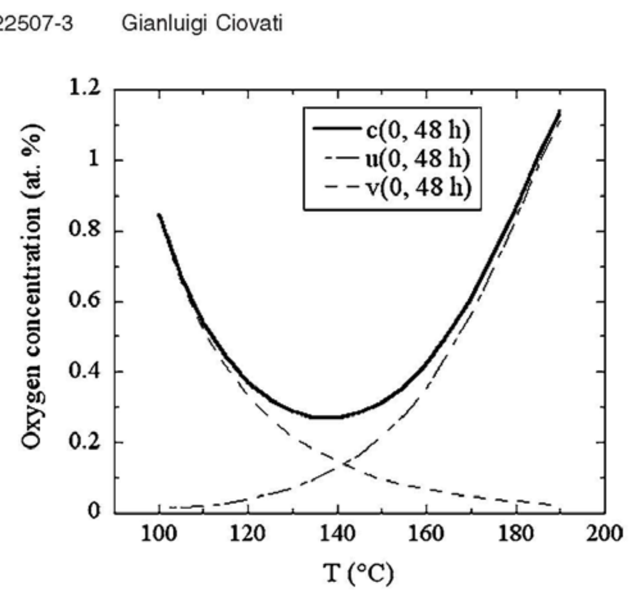
Vacuum System for In-situ Baking



Diffusion of Oxygen by In-situ Baking

The SC current inside the cavity has the depth of ~40 nm (London Penetration Depth)





X(nm): Depth from the surface

Oxygen diffuses into bulk area.

c = total O concentration (~ u + v)
u : O concentration from Nb_2O_5 decomposition
(Nb_2O_5 => NbO_2 =>NbO)
v : Initial O concentration after diffusion by baking

A hypothesis is that oxygen causes Q-drop only at high gradient.