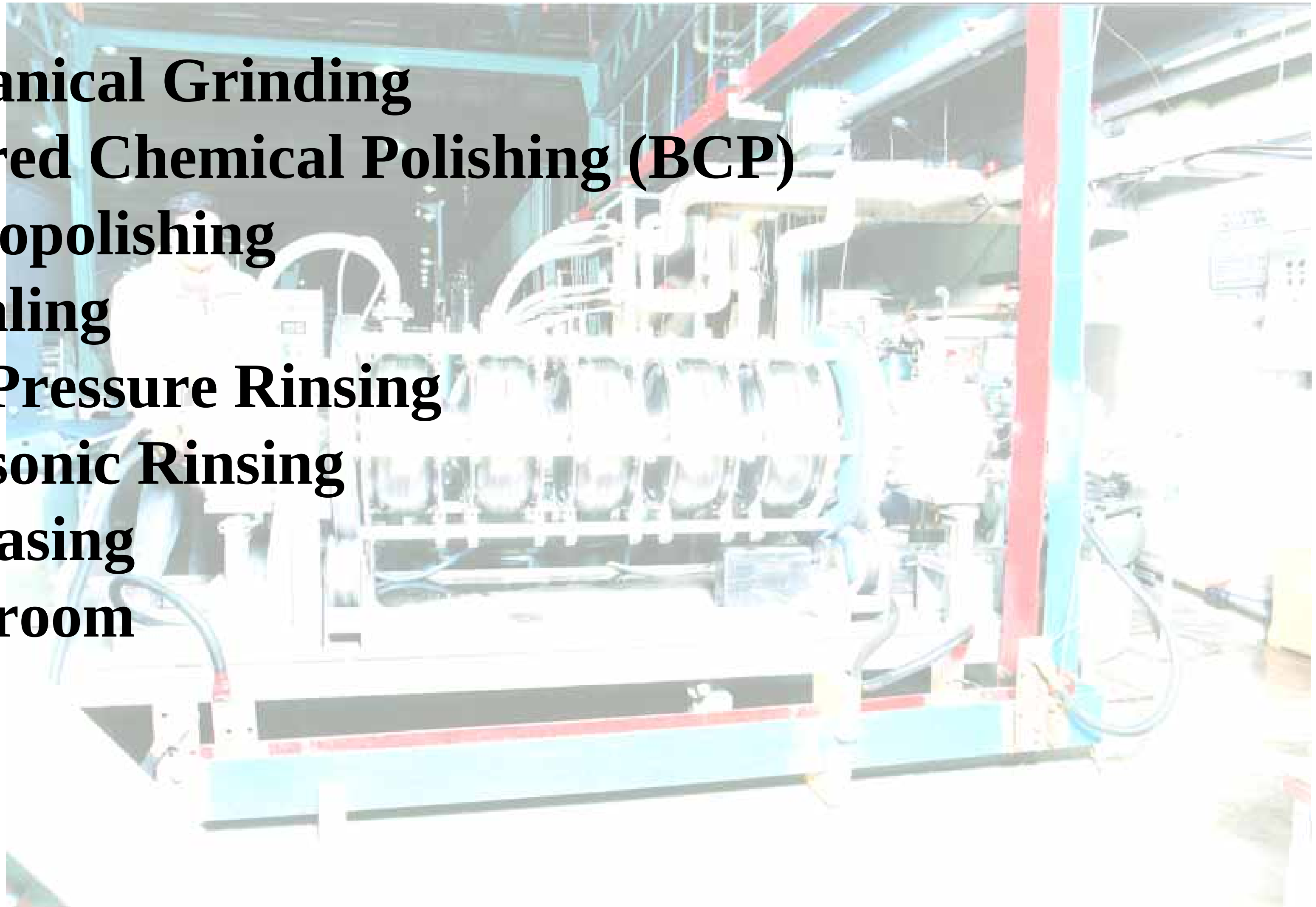
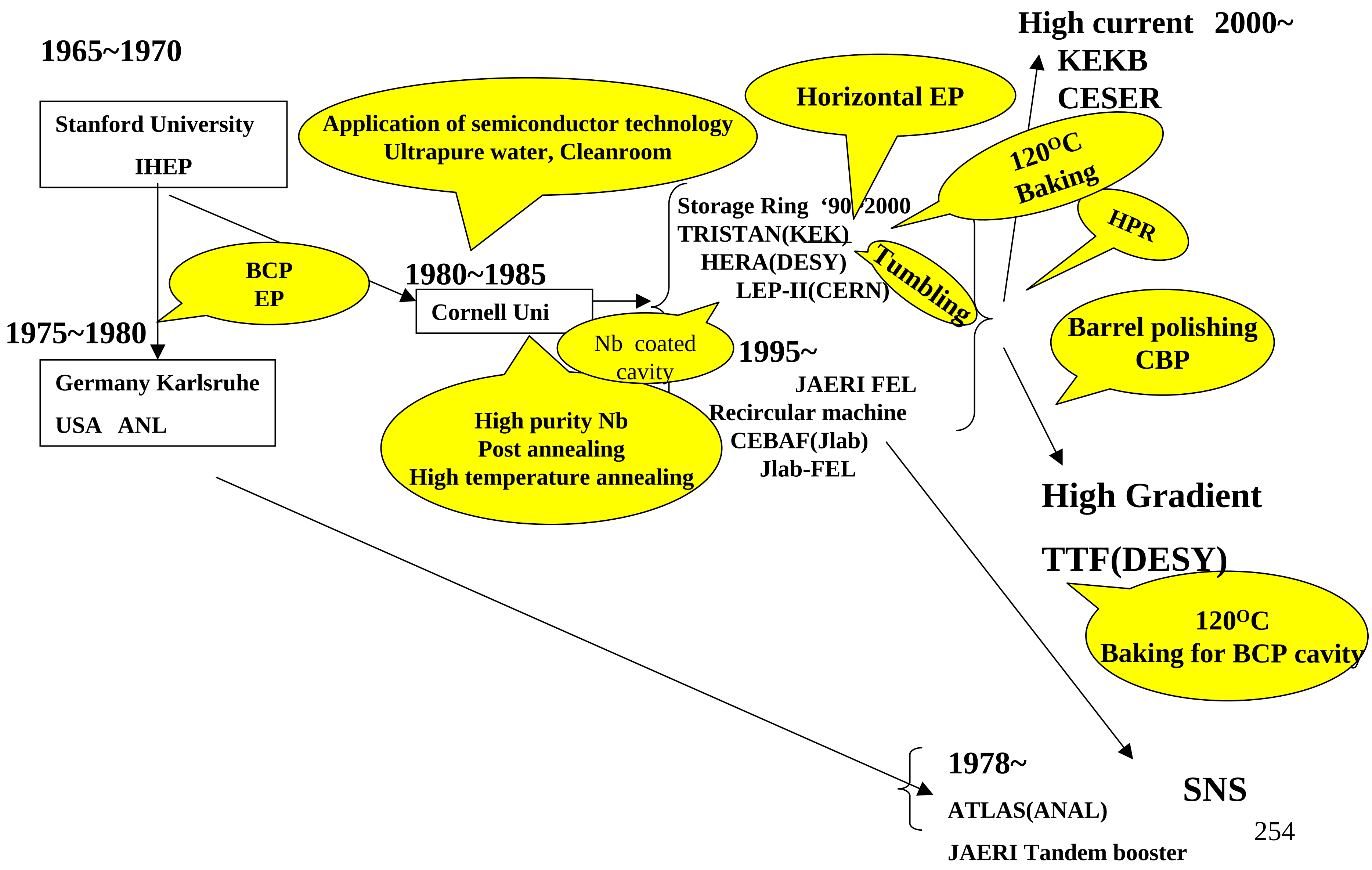
8. Surface Preparation Techniques

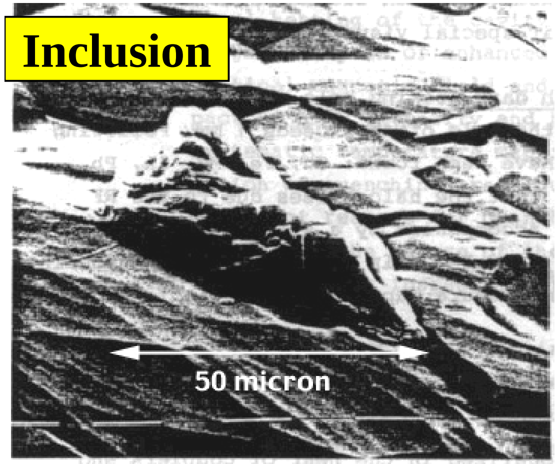
8.1 Mechanical Grinding
8.2 Buffered Chemical Polishing (BCP)
8.3 Electropolishing
8.4 Annealing
8.5 High Pressure Rinsing
8.6 Megasonic Rinsing
8.7 Degreasing
8.8 Cleanroom

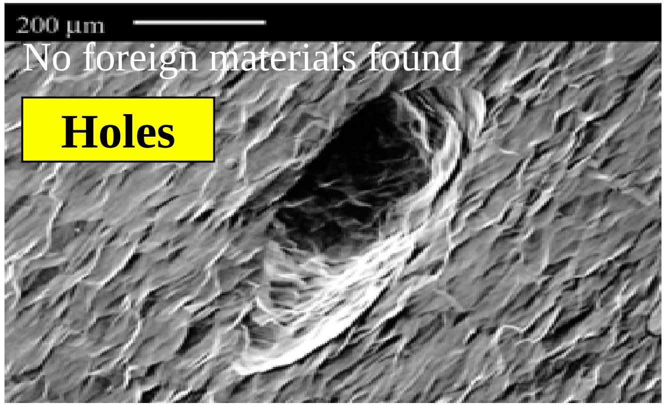


History of Preparation Technologies

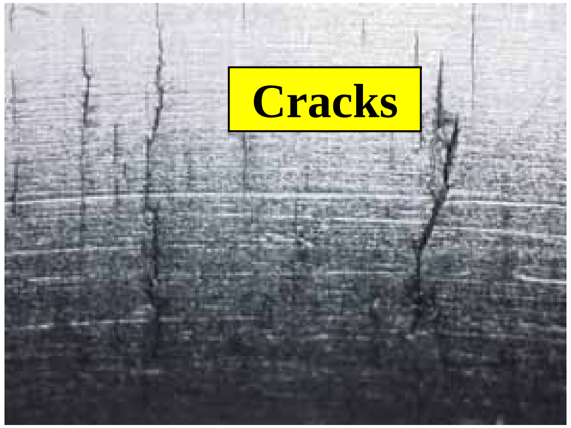


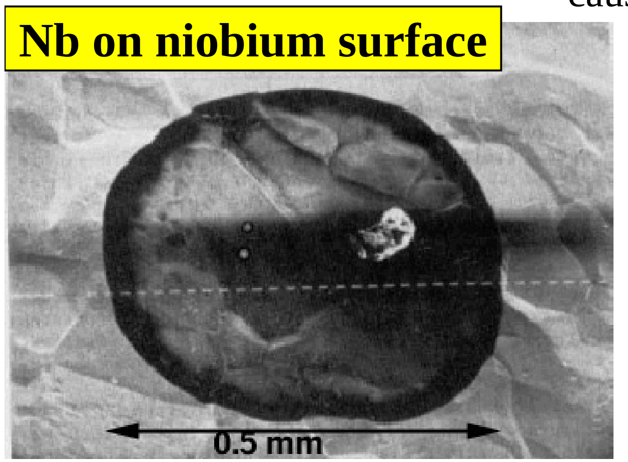
Various Surface Defects

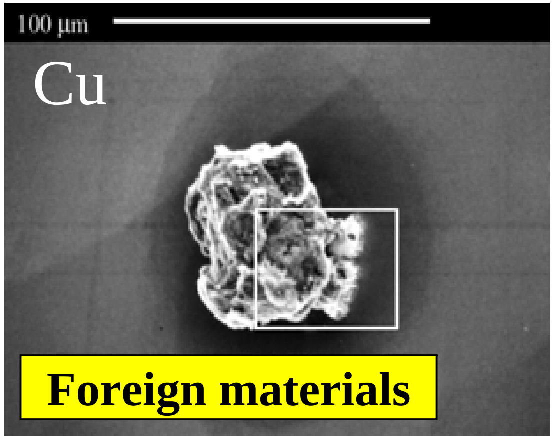


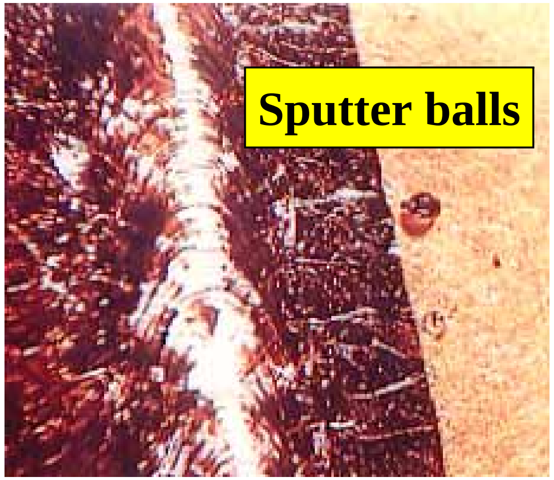


Surface defects, holes can also cause TB









Mechanical grinding is a powerful tool to remove large surface defects.

8.1 Mechanical Grinding

MG is very powerful to remove surface defects but remains Contamination on the ground surface.
It is usually used as pre-treatment before chemical preparation.

Buffing

Buffing TRISTAN 320 half cups.

- **Very powerful**
- **High reliable**
- **Well controlled the surface roughness**

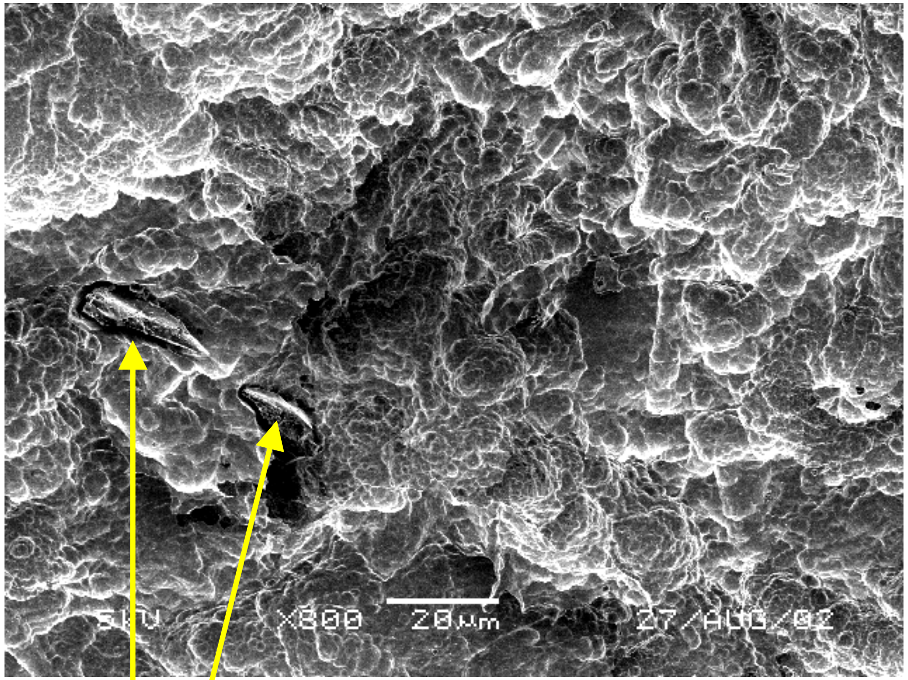
Used in the TRISTAN @ KEK

- **All half-cup were buffed.**
- **Other mechanical grinding for welding seams.**

Problem with buffing

1) High cost
2) Impossible to completed structure

Contamination by mechanical grinding

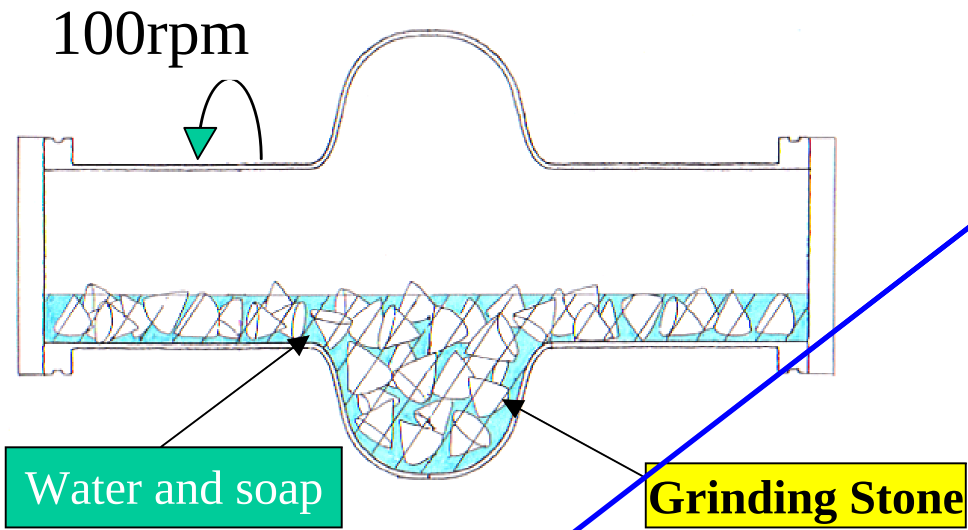


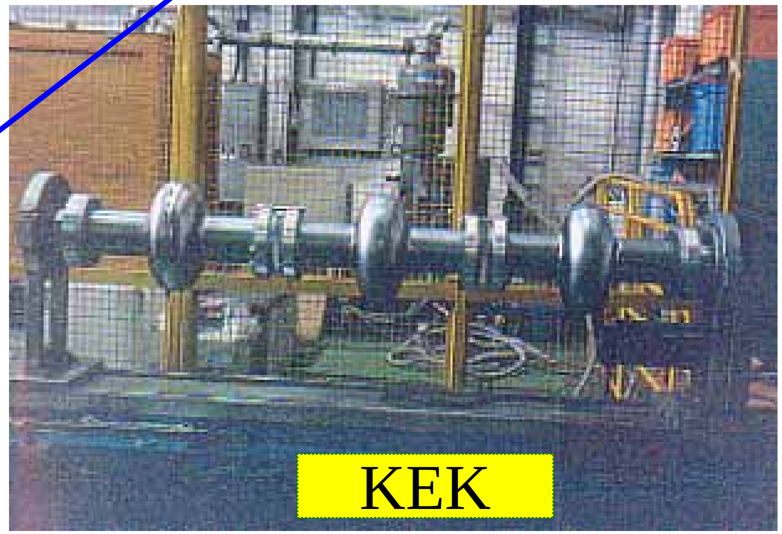
Remained grains of grinding material
(Barrel polishing)

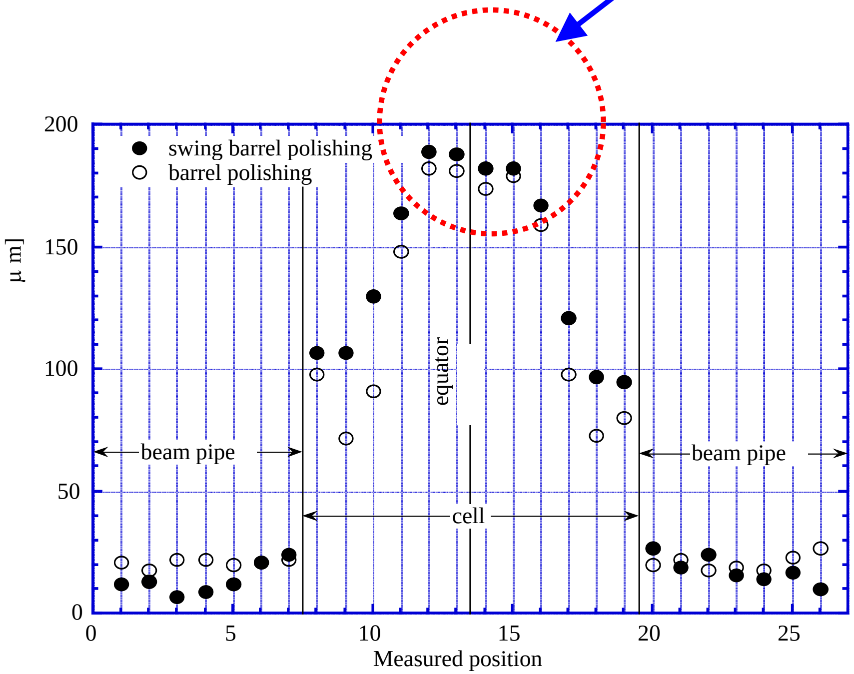
Need to make a chemical preparation in order to remove these contamination.

Tumbling or Barrel Polishing(BP)

Easy for EBW seam at equator







- Simple
- Possible to a competed structure
- Low cost

Problem in BP

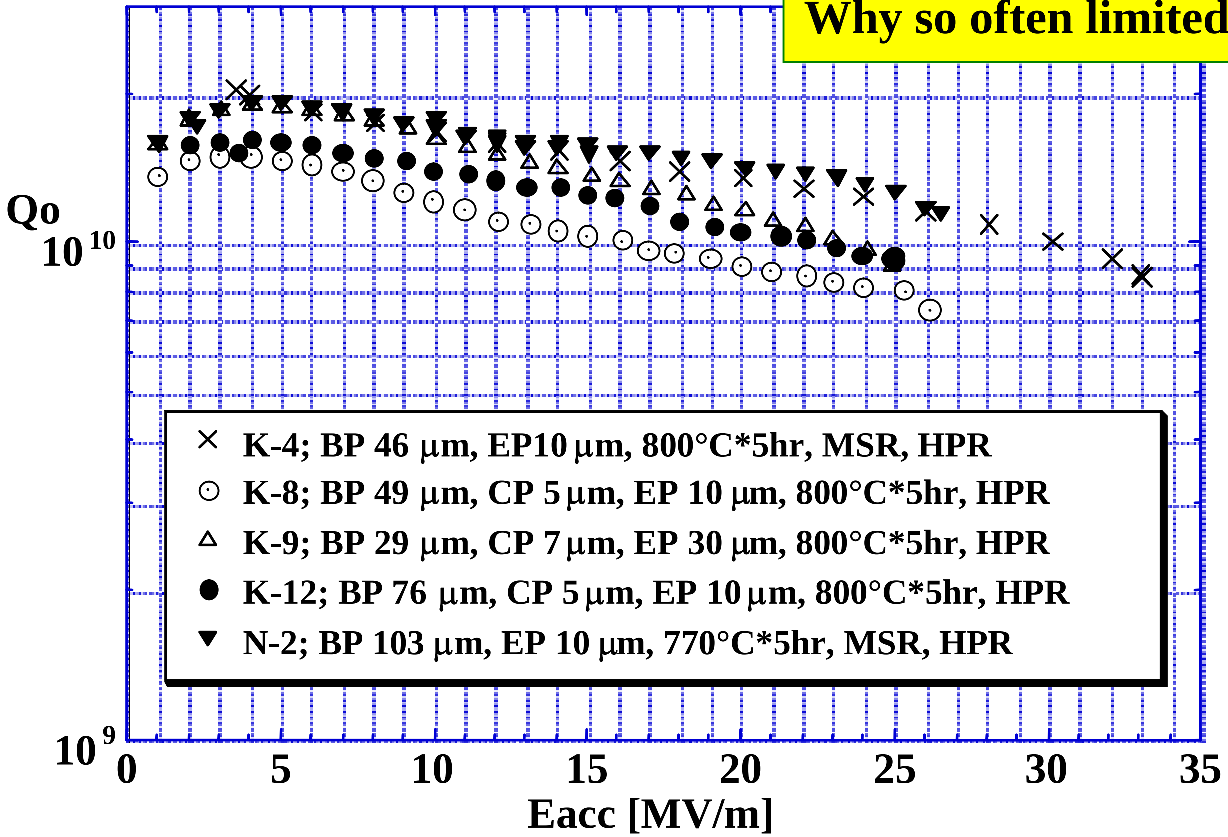
Slow material removal speed 3μm/day

Takes "one week" to remove 30μm.

Dopes hydrogen in the Nb material

Confirmation of the BP effectiveness as pre-treatment prior to EP

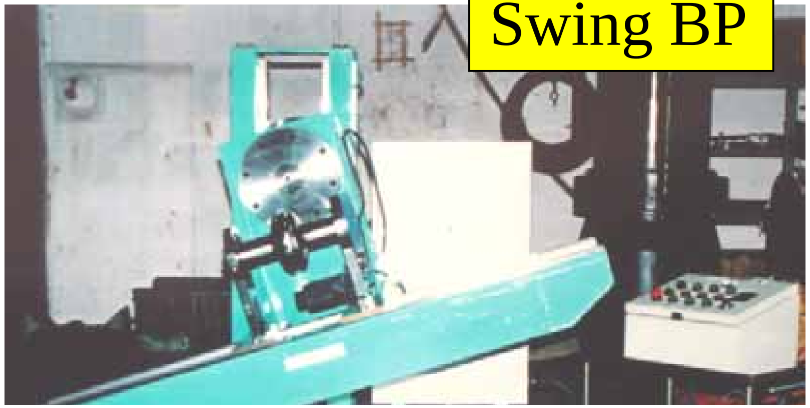
Cavity was annealed after BP.
Why so often limited at 25MV/m?



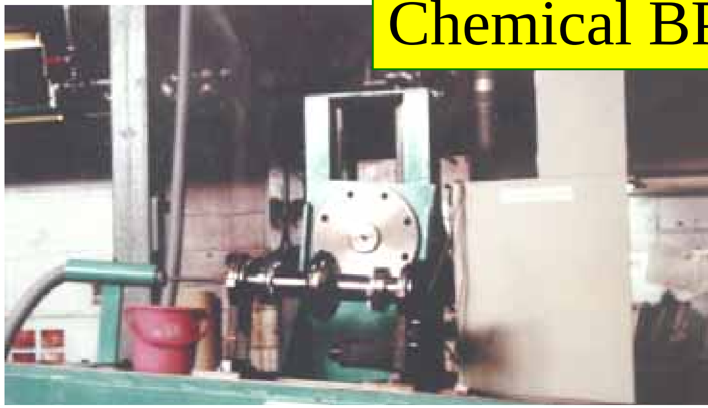
Confirmed 25MV/m by combination BP+Annealing + EP,
25MV/m was enough high gradient in those days (1995).

Some trials to improve the material removal speed of BP

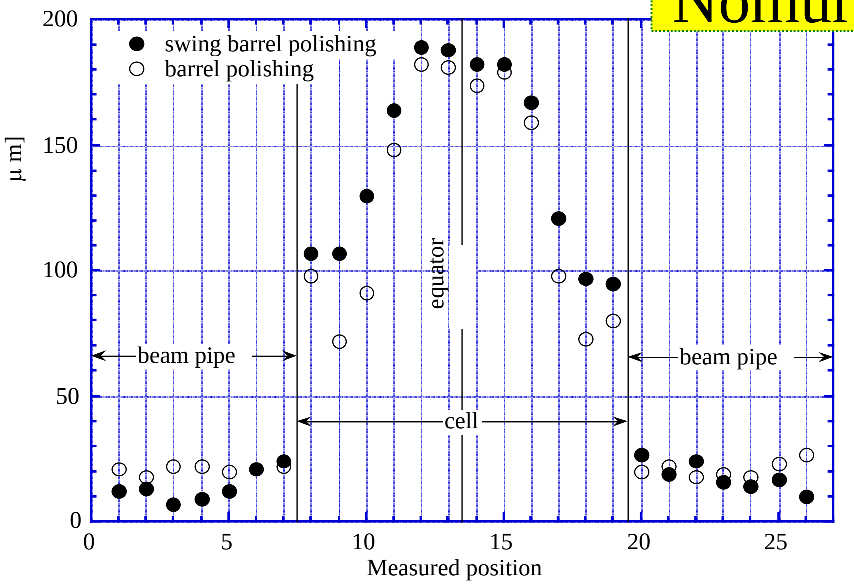
Swing BP

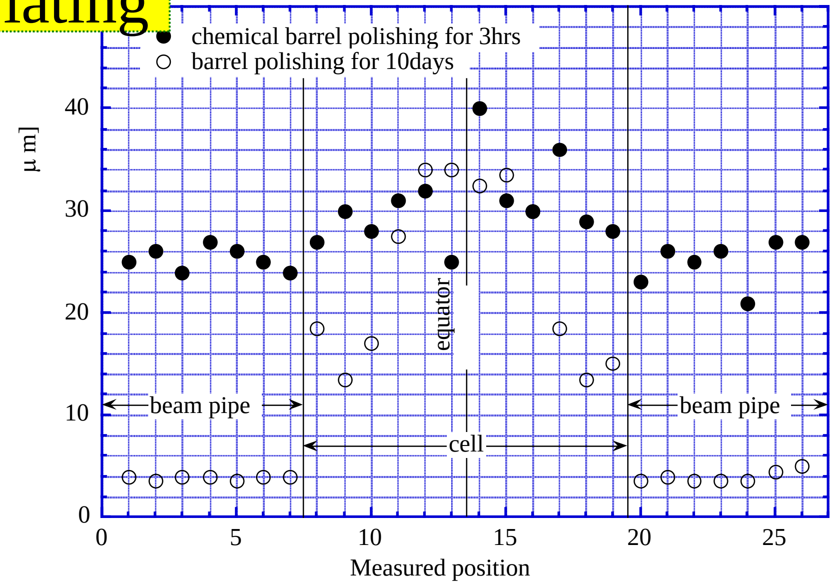


Chemical BP



Nomura Plating

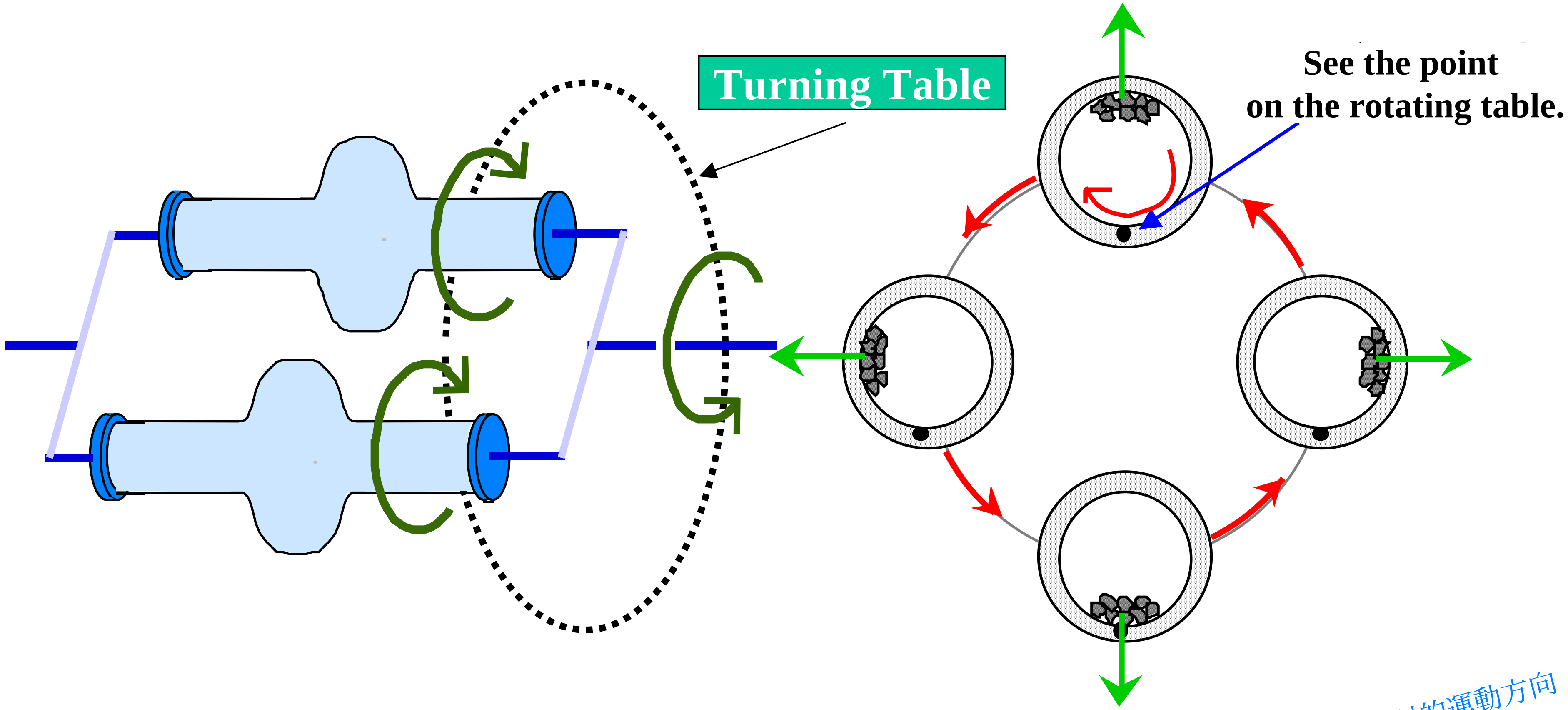




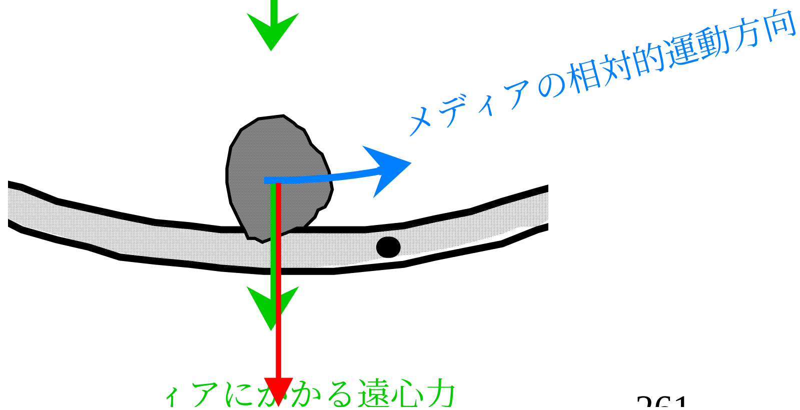
No improvement on the removal speed

Large removal speed
but could not good cavity performance

Innovation Centrifugal BP (CBP)



- Rotate cavity on a "rotating table"
- Rotation directions are opposite each other



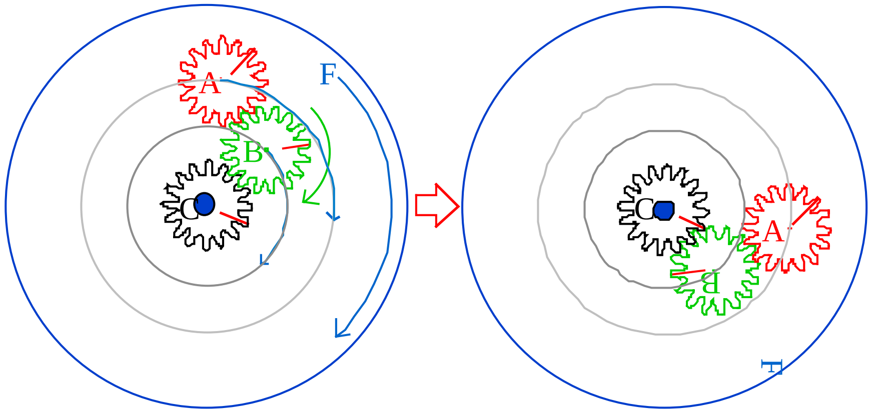
Two centrifugal forces are added on the grinding stones

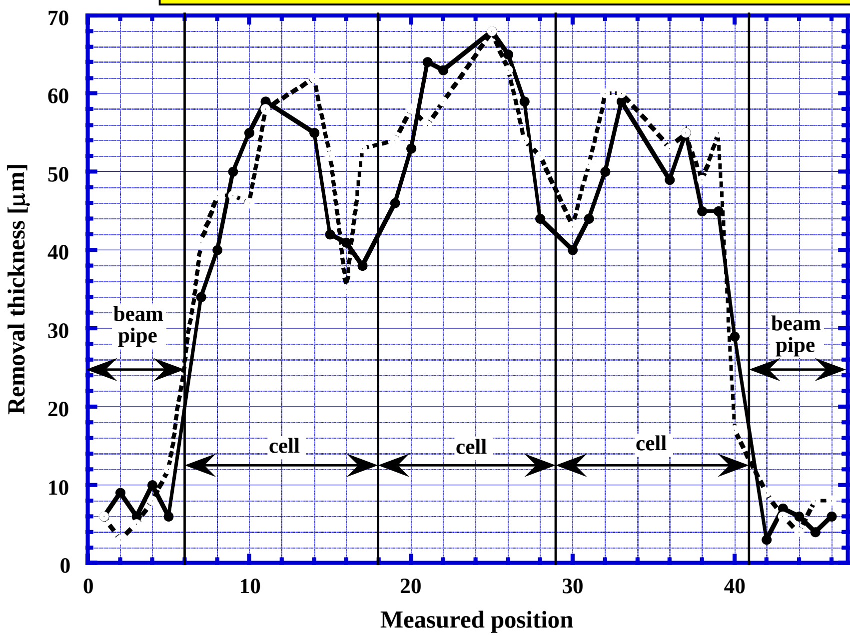
Developed CBP Machine

Front surface

KEK

Three cell CBP (1300MHz)





Rotation mechanism:

Cavity rotating/table rotation

CBP Finishing Surface

Large Grain cavity case

Rough stone (rough) : 5 times (4 hour each)

Green stone (medium) : Once

Brown stone (medium): Once

White stone (for final fine finish) : Once

Totally ~ 200 **μm removed @ equator**

Very fast removal speed!

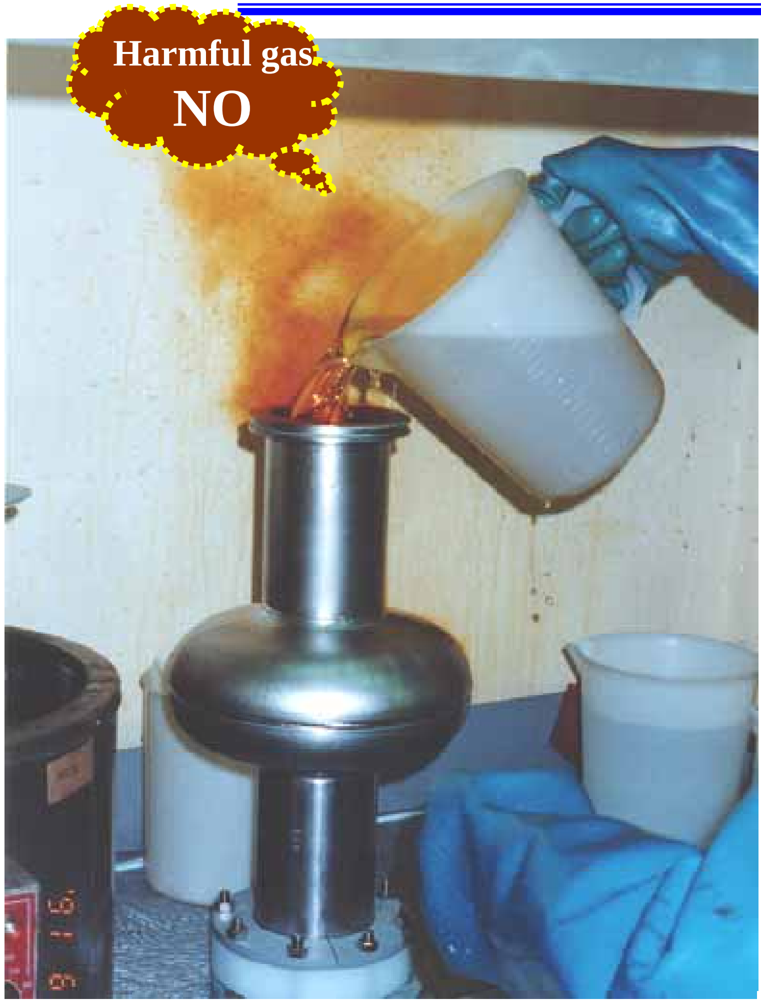
Material removal speed: “one week”(BP) → 4hr (CBP)

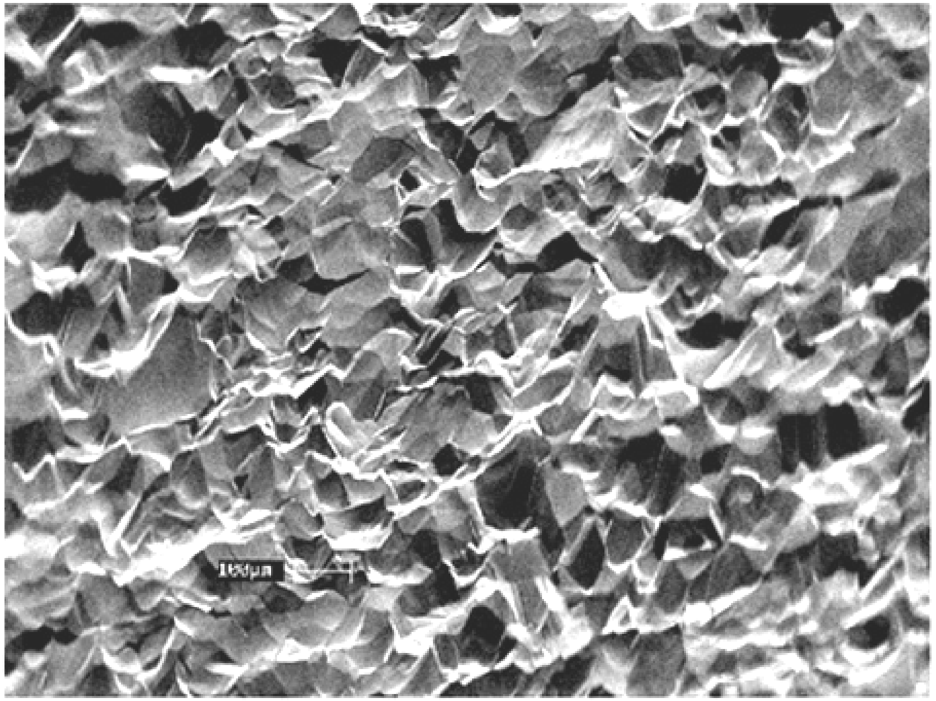
Before CBP (equator EBW seam)

After CBP

After light CP(10μm)

8.2 Buffered Chemical Polishing (BCP)





$HF(46\%):HNO_3(60\%):H_3PO_4$
1:1:1 (V/V)

No reaction with Nb, Mild the reaction, Increase viscosity of the acid.

- **Simple and A large material removal speed (10μm/min @ R.T.)**

Problem of BCP: Surface is not so smooth.

Chemical reaction:

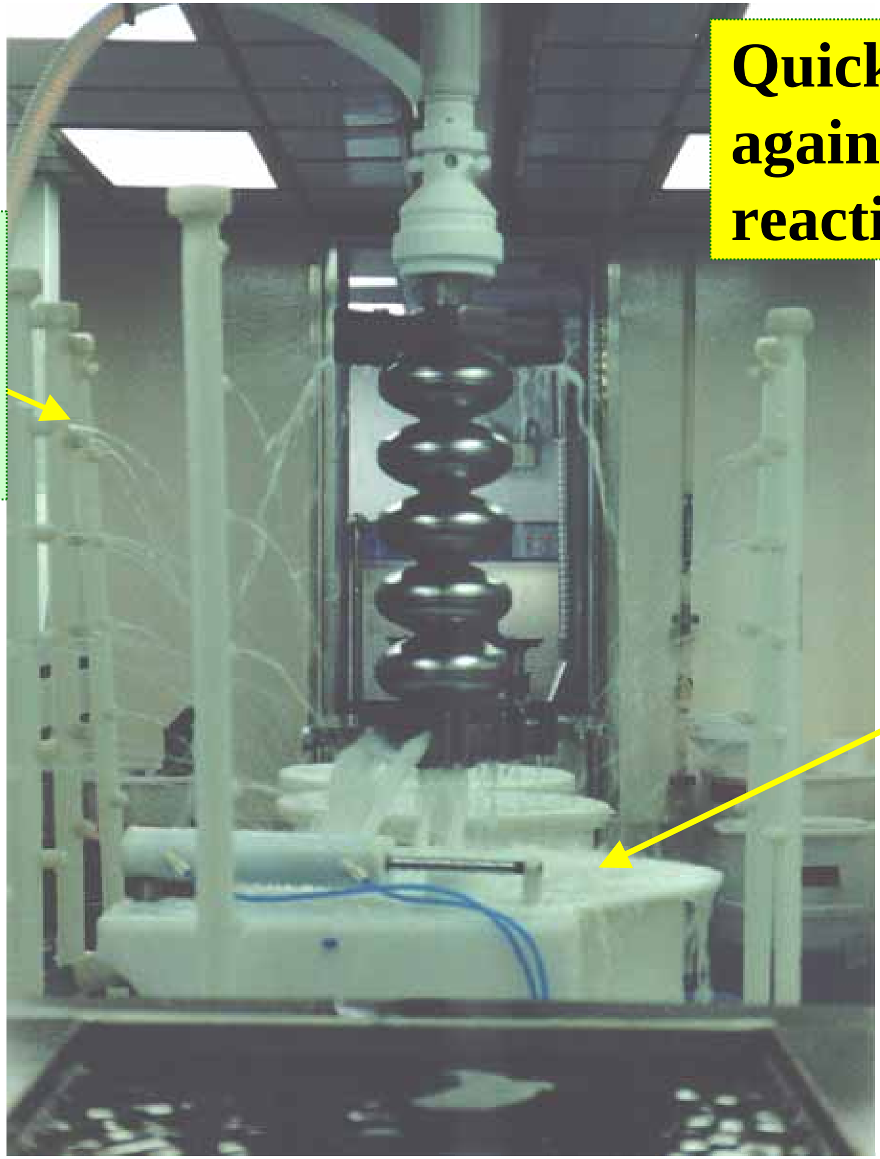
$$6Nb+10HNO_3 \rightarrow 3Nb_2O_5+10NO\uparrow+5H_2O$$

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

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

CEBAF CP & Rinsing

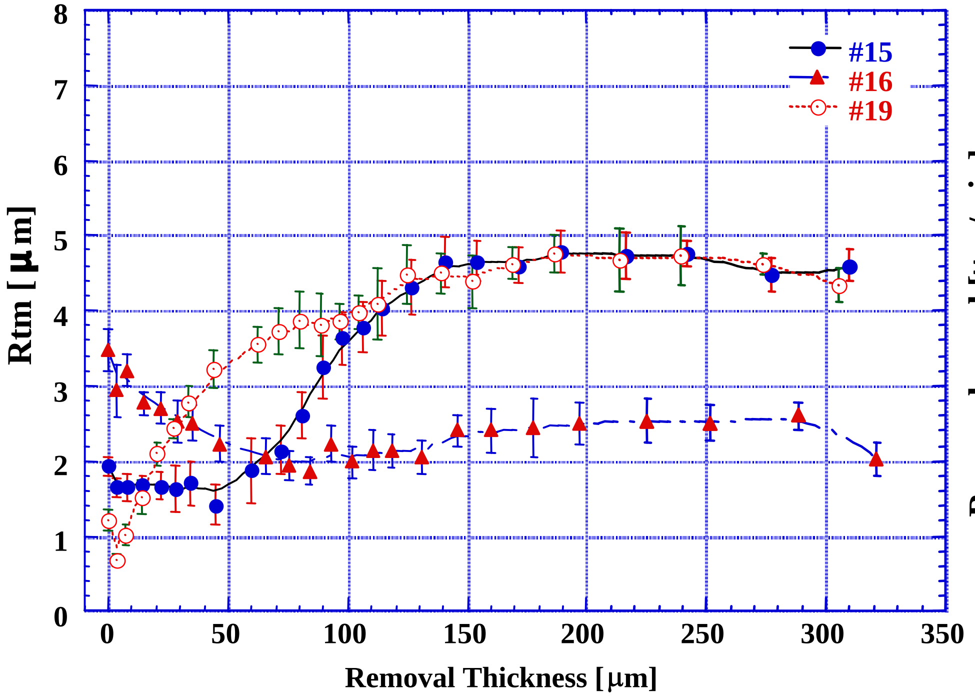
Shower for Rinsing the outer cavity surface

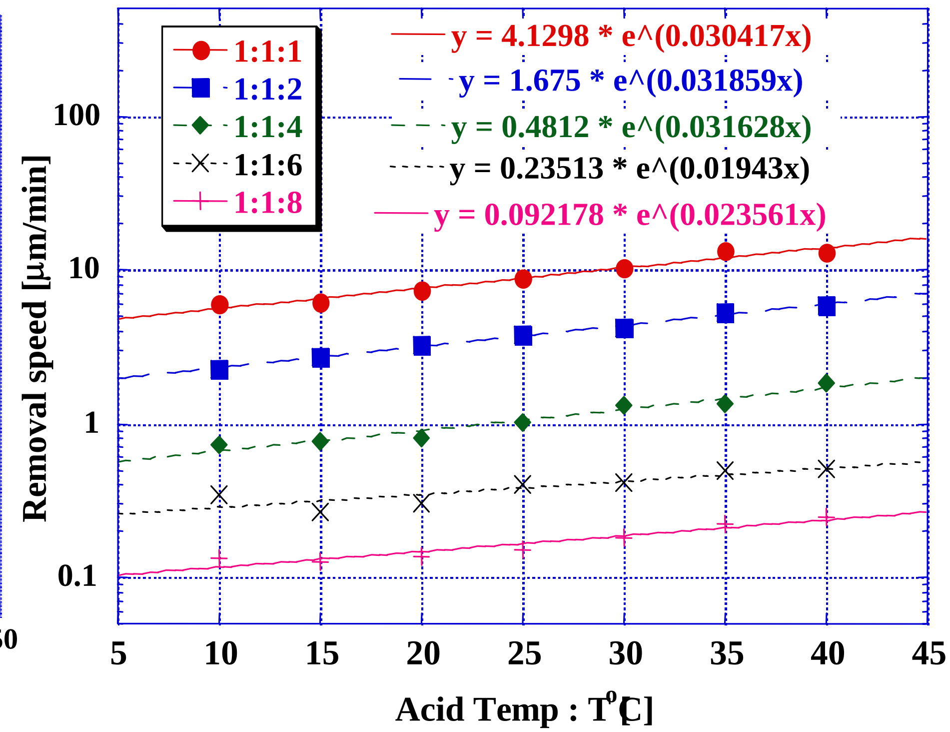


Quick rinsing against the runaway reaction

CP acid tank Cavity is immersed in the BCP acid.

Characteristics of BCP



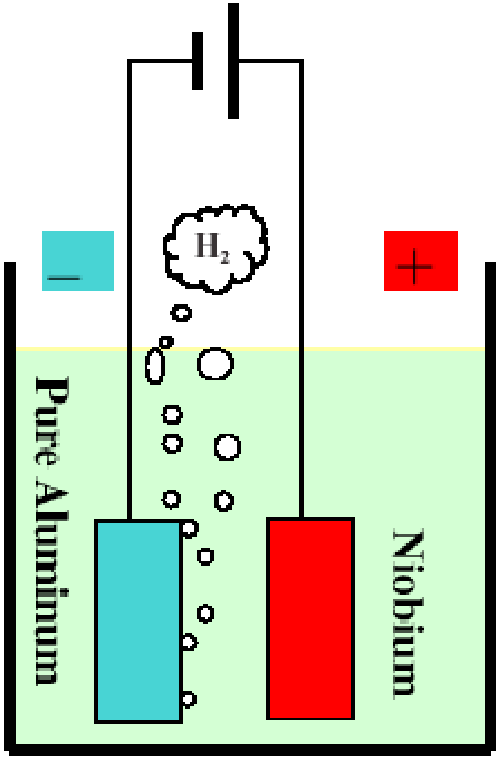


Typical surface roughness = 2 ~ 5 μm after 100μm CP,
Material removal speed ~ 10μm/min at the room temperature with CP acid 1:1:1

CP is faster in material removal speed than EP.

The finished surface roughness strongly depends on the grain size of the Nb material.

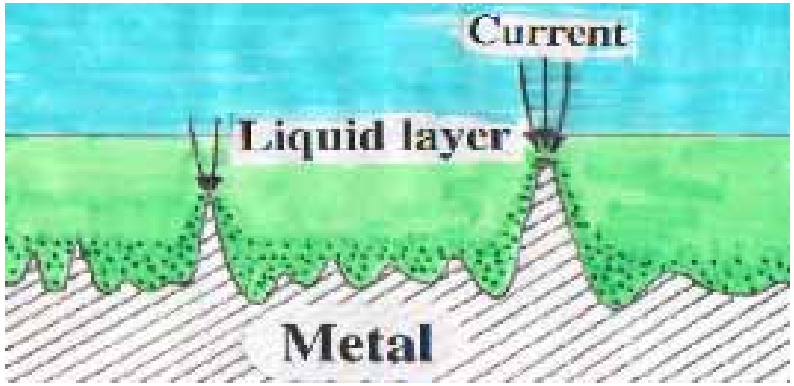
8.3 Electropolishing

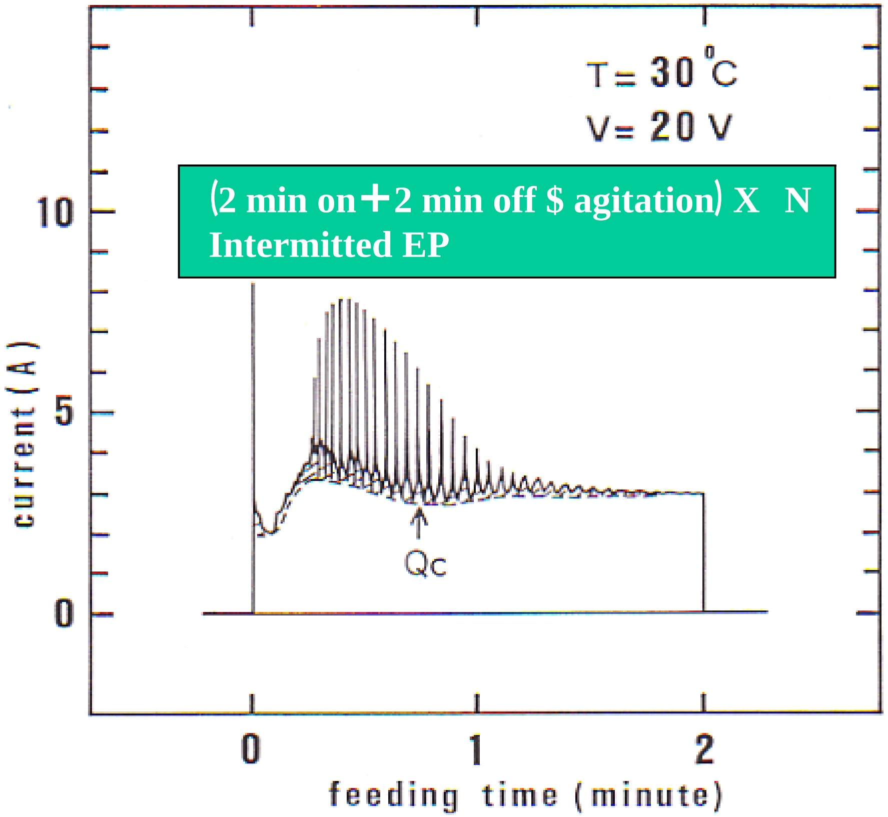


Acid:
H_2SO_4 (>93%): HF(46%)=10:1 V/V

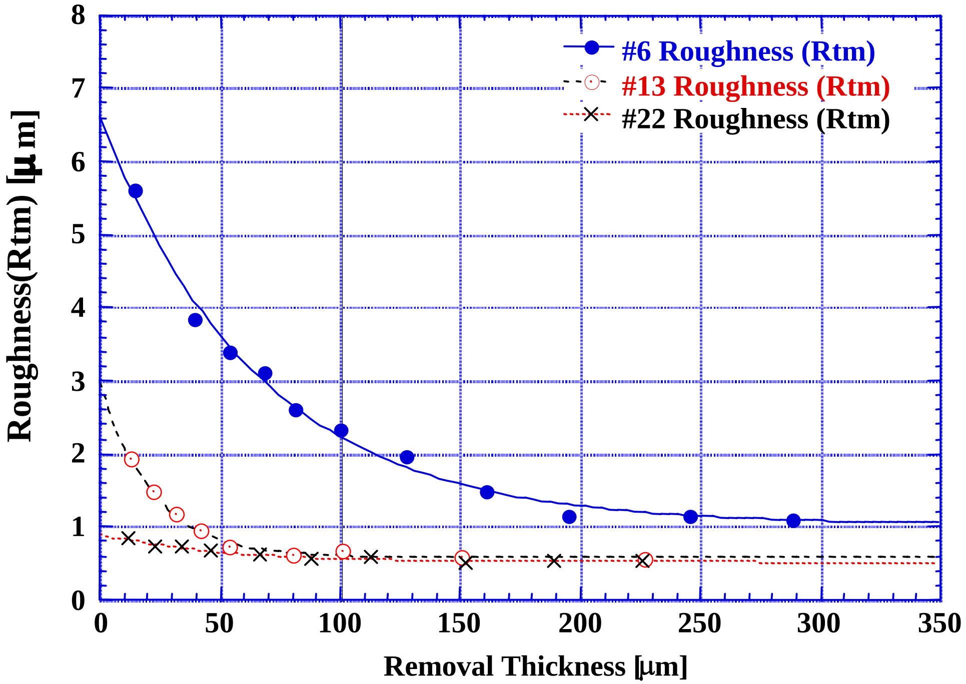
Chemical reaction:
$2Nb + 10HF + 2H_2O \rightarrow 2H_2NbOF_5 + 5H_2\uparrow$

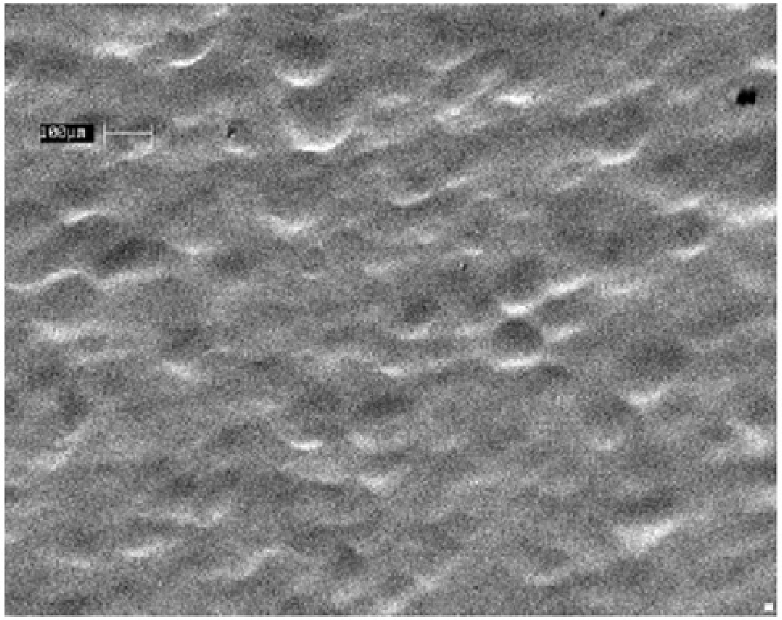
H_2SO_4 does not react with Nb, which make viscosity in the acid.





EP Finished Surface

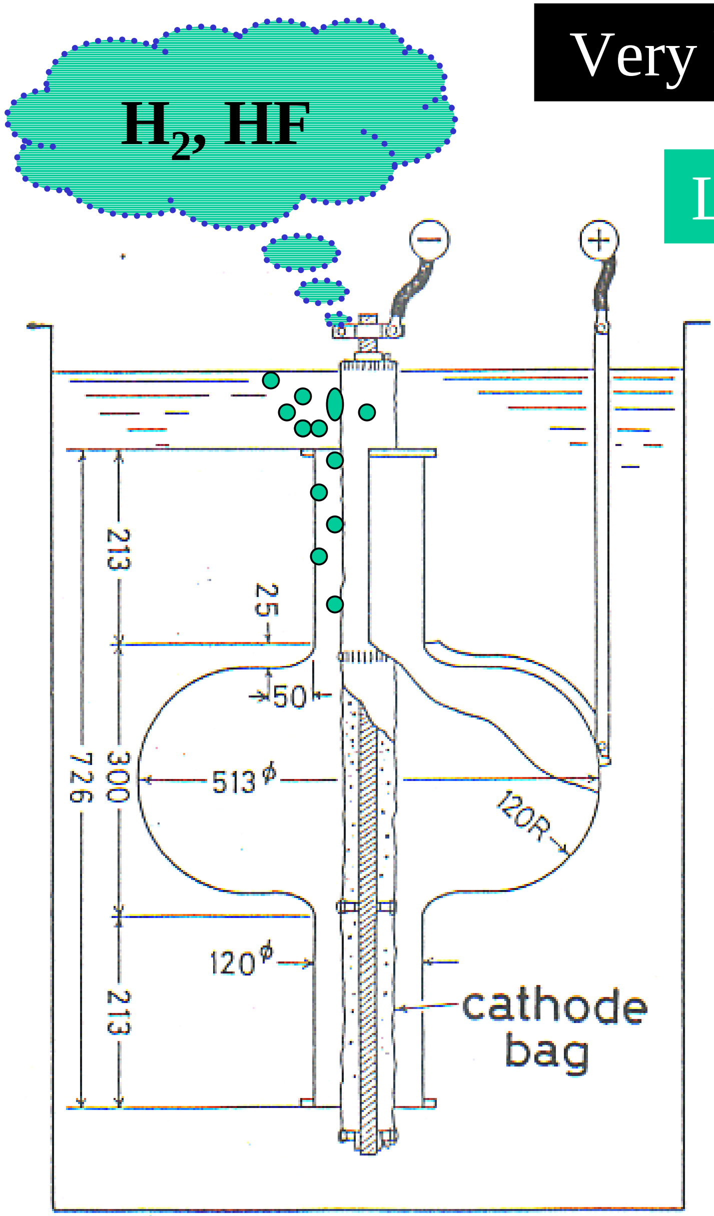




1) The finishing surface roughness depends on that of the initial surface.
2) The finishing roughness becomes smooth exotically with the material removal.
3) Grain boundary is not sharp edge as that of BCP case.
4) Easy control of surface roughness.

KEK Early EP (Vertical EP)

Very hazardous working environment



Light hydrogen Q-disease

Hydrogen Q-disease

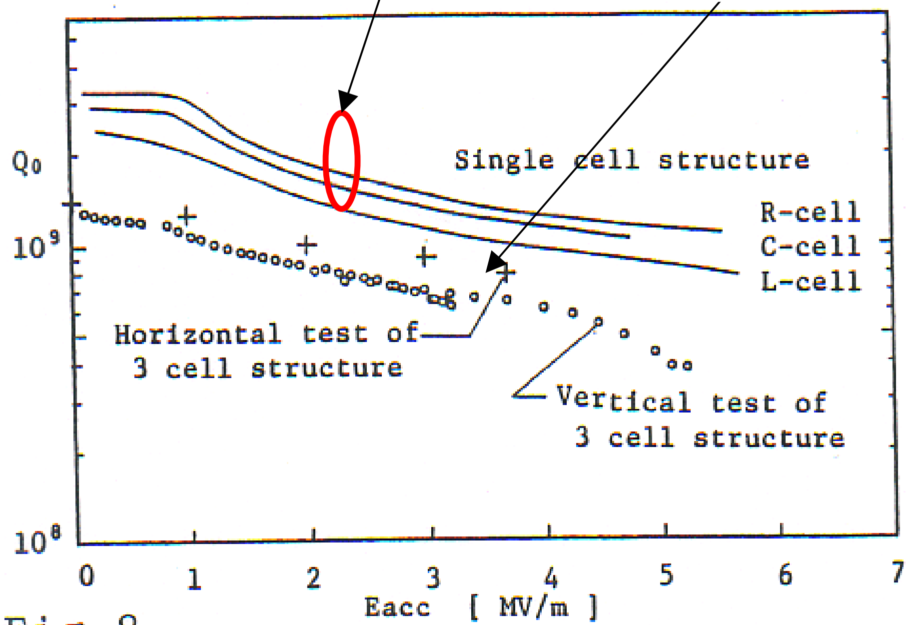
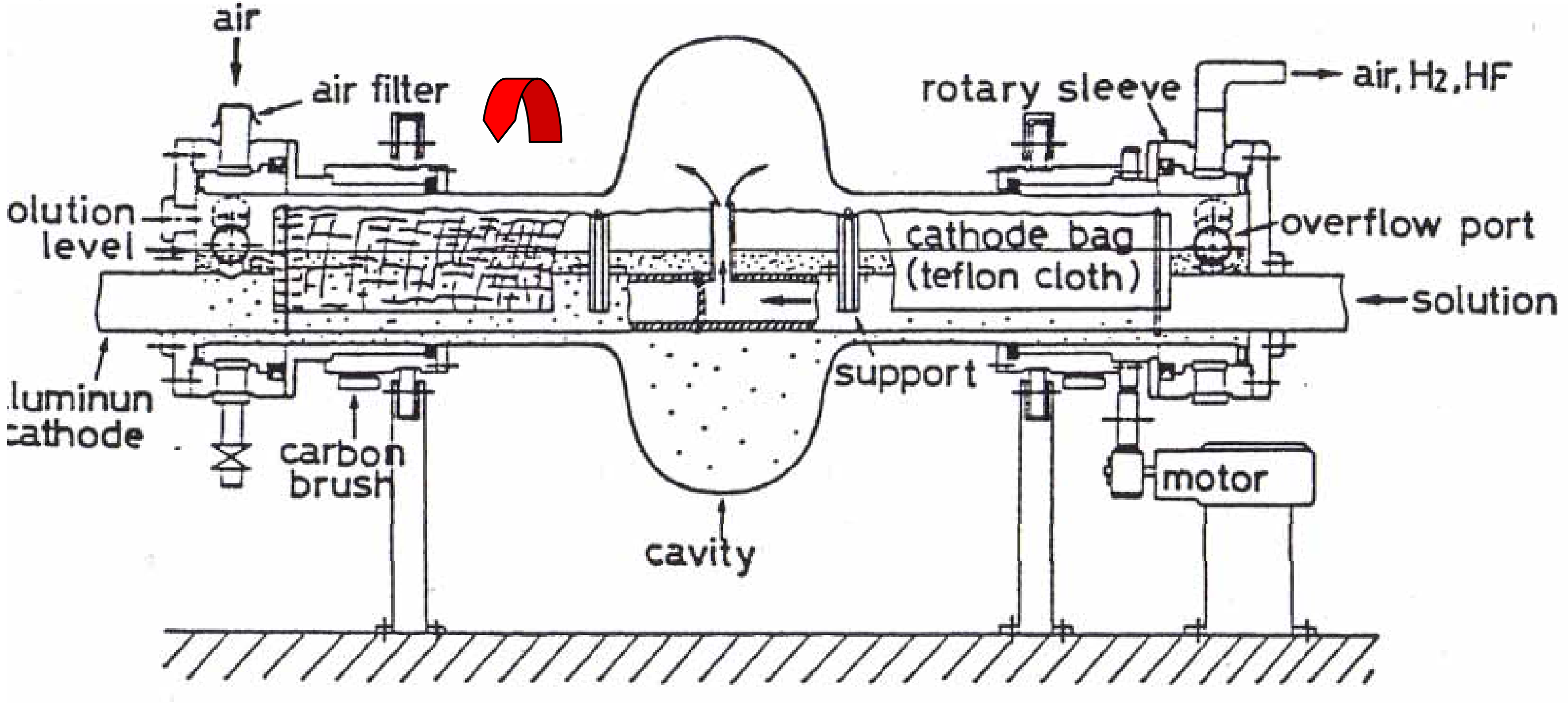


Fig.8
Qo-Eacc curves of a three-cell cavity electropolished using the vertical EP method. R,C,L-cell are single cell cavities before completing the three cell cavity.

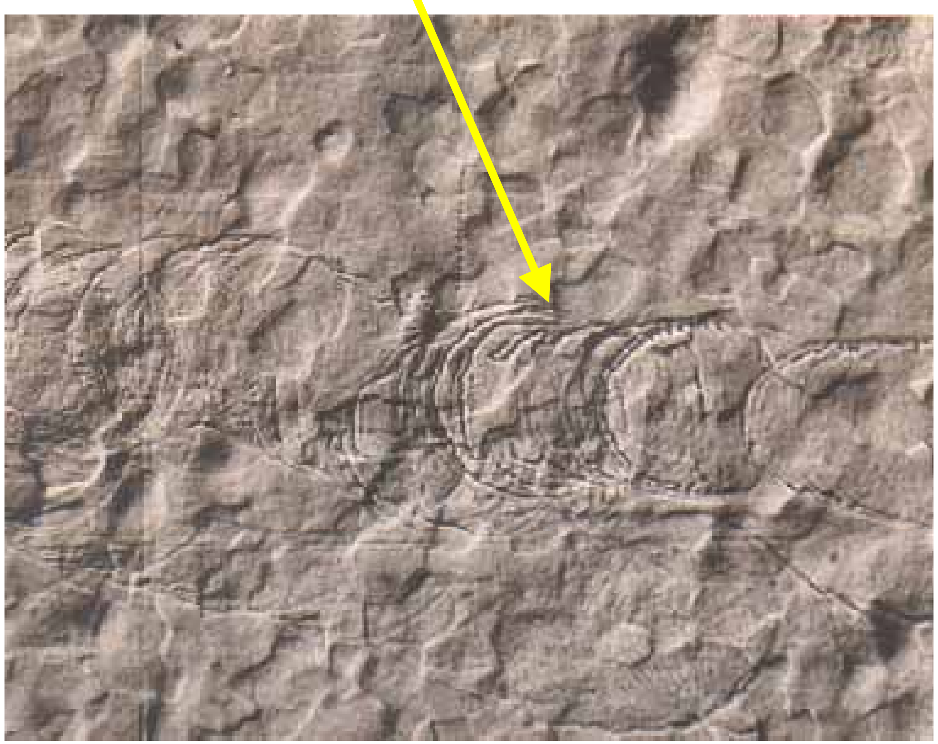
Innovation of the Horizontal EP

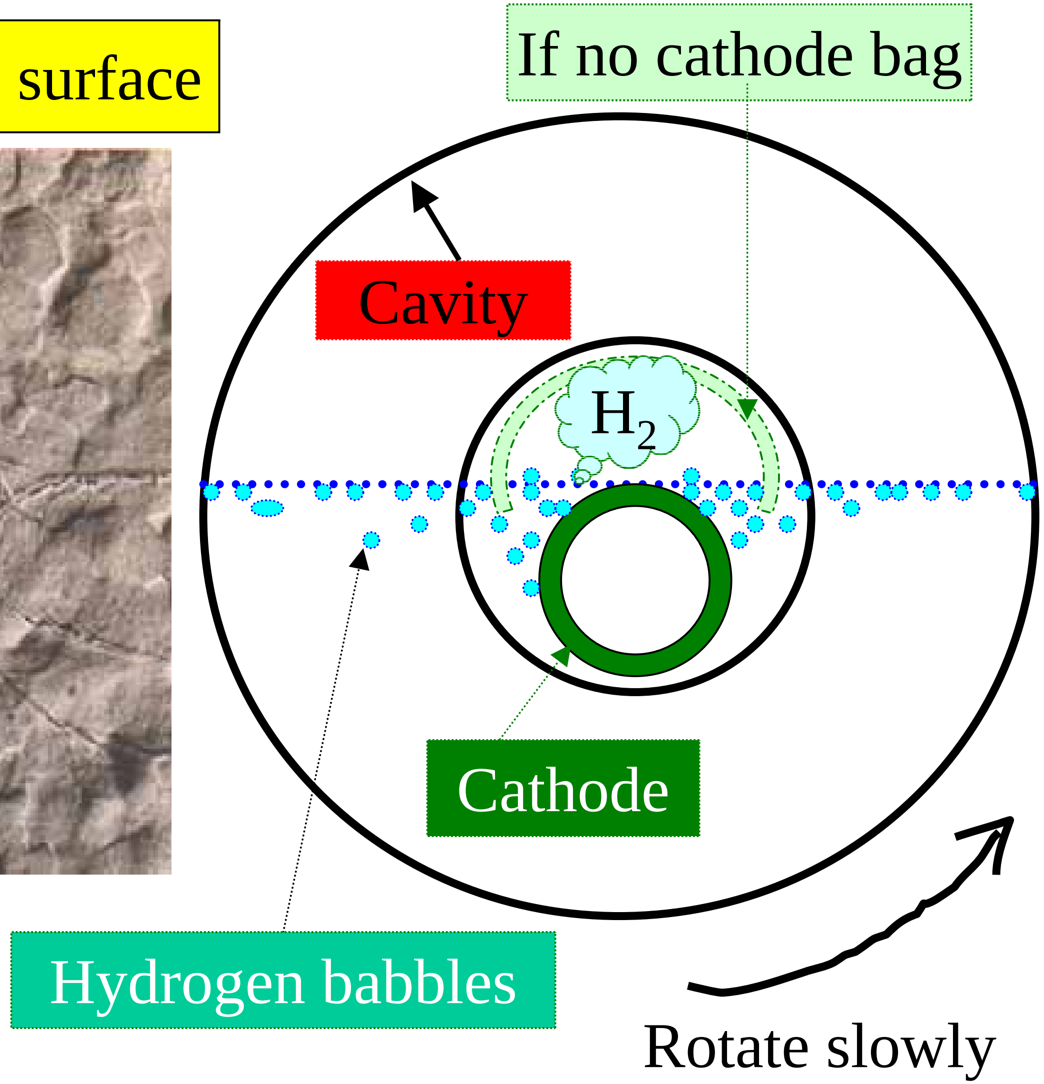


1) Close the EP acid in the EP system to improve the working environment.
2) Easy H_2 gas evacuation even for multi-cell cavity.
3) Uniform material removal in each cell for multi-cell cavity.
4) Simple control.

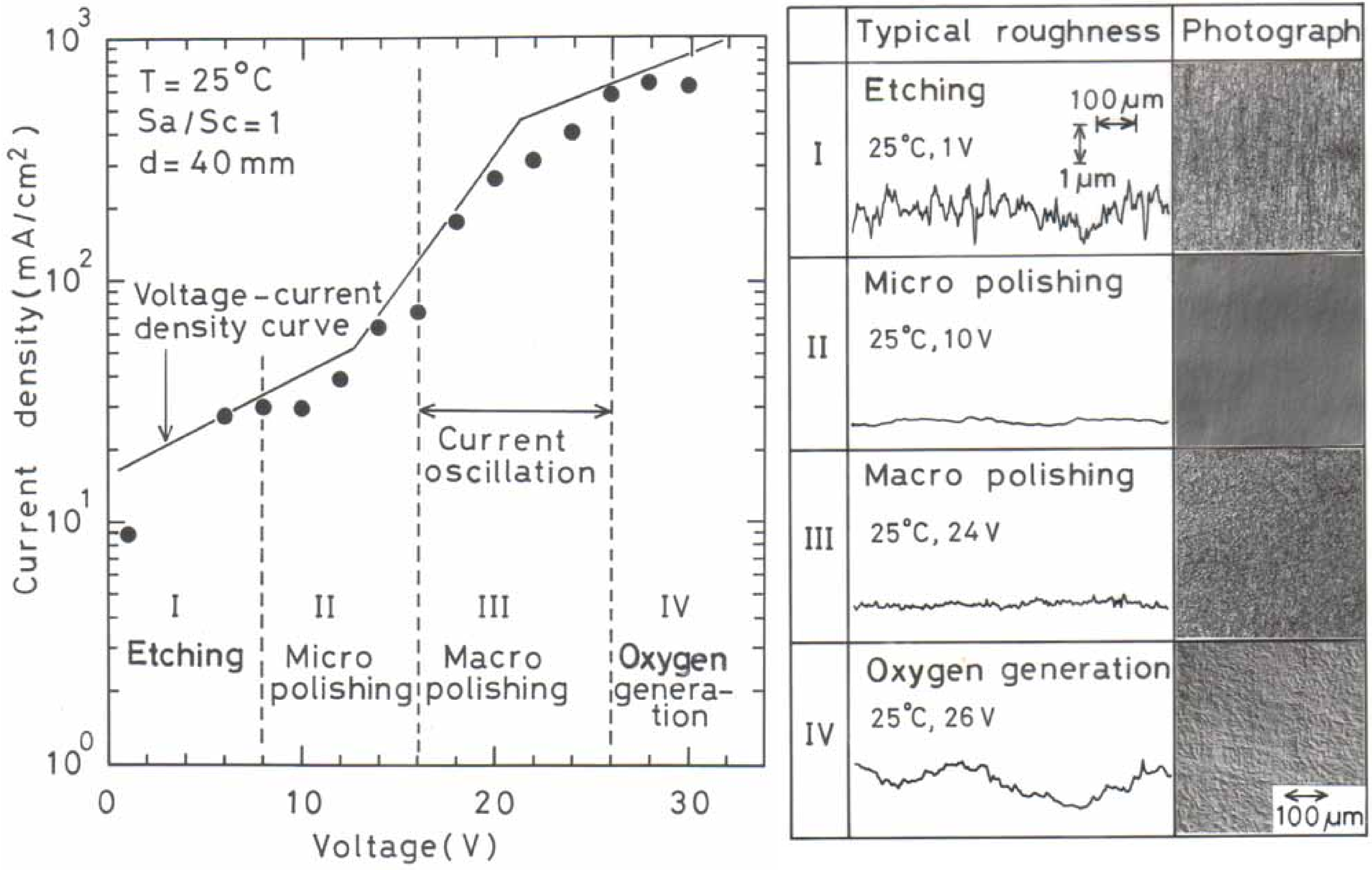
Cathode Bag

Hydrogen babble trace on Nb surface





Electrolishing Characteristics with Nb

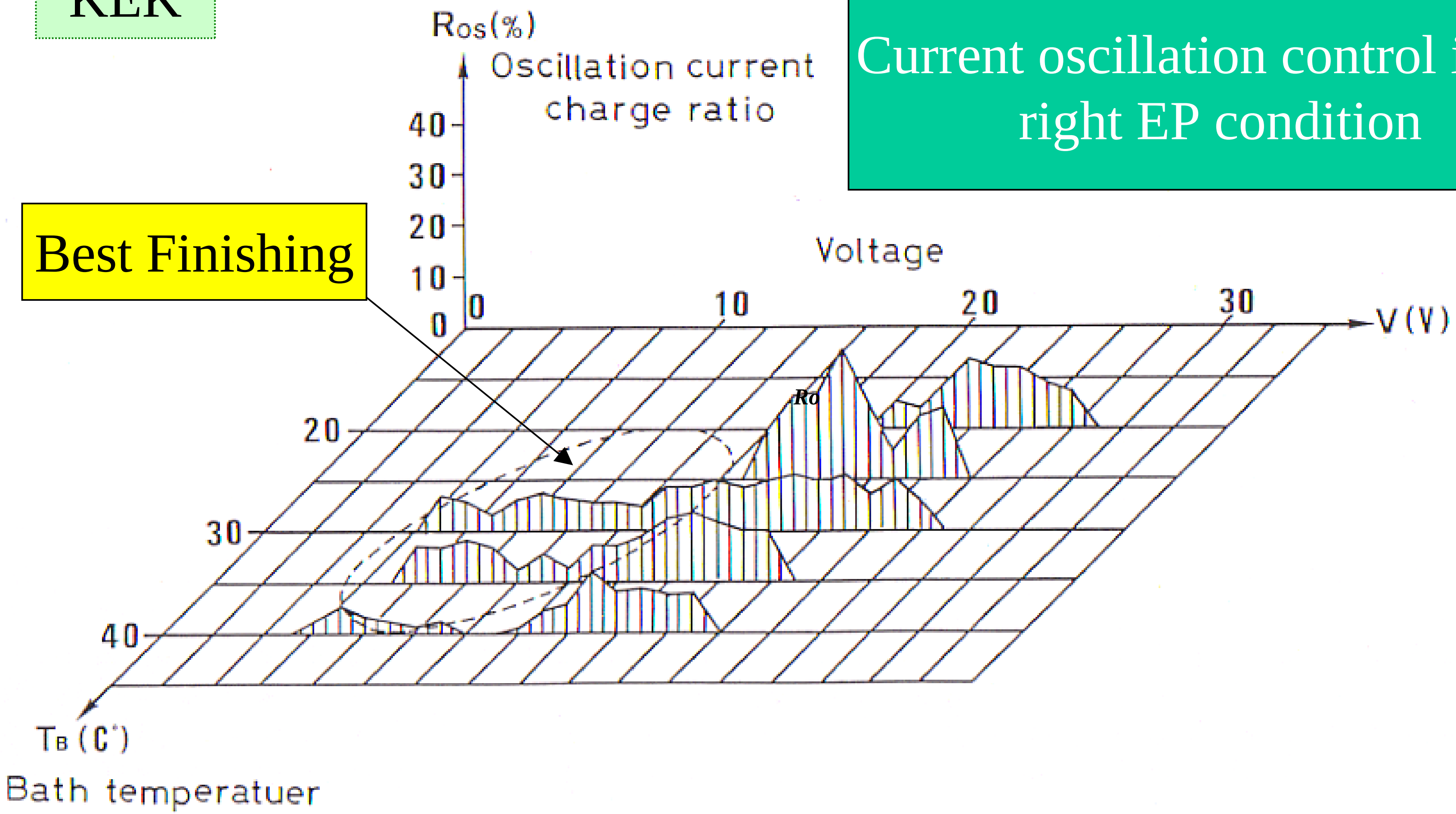


Reconsideration of the Current Oscillation

KEK

Current oscillation control is not right EP condition

Best Finishing



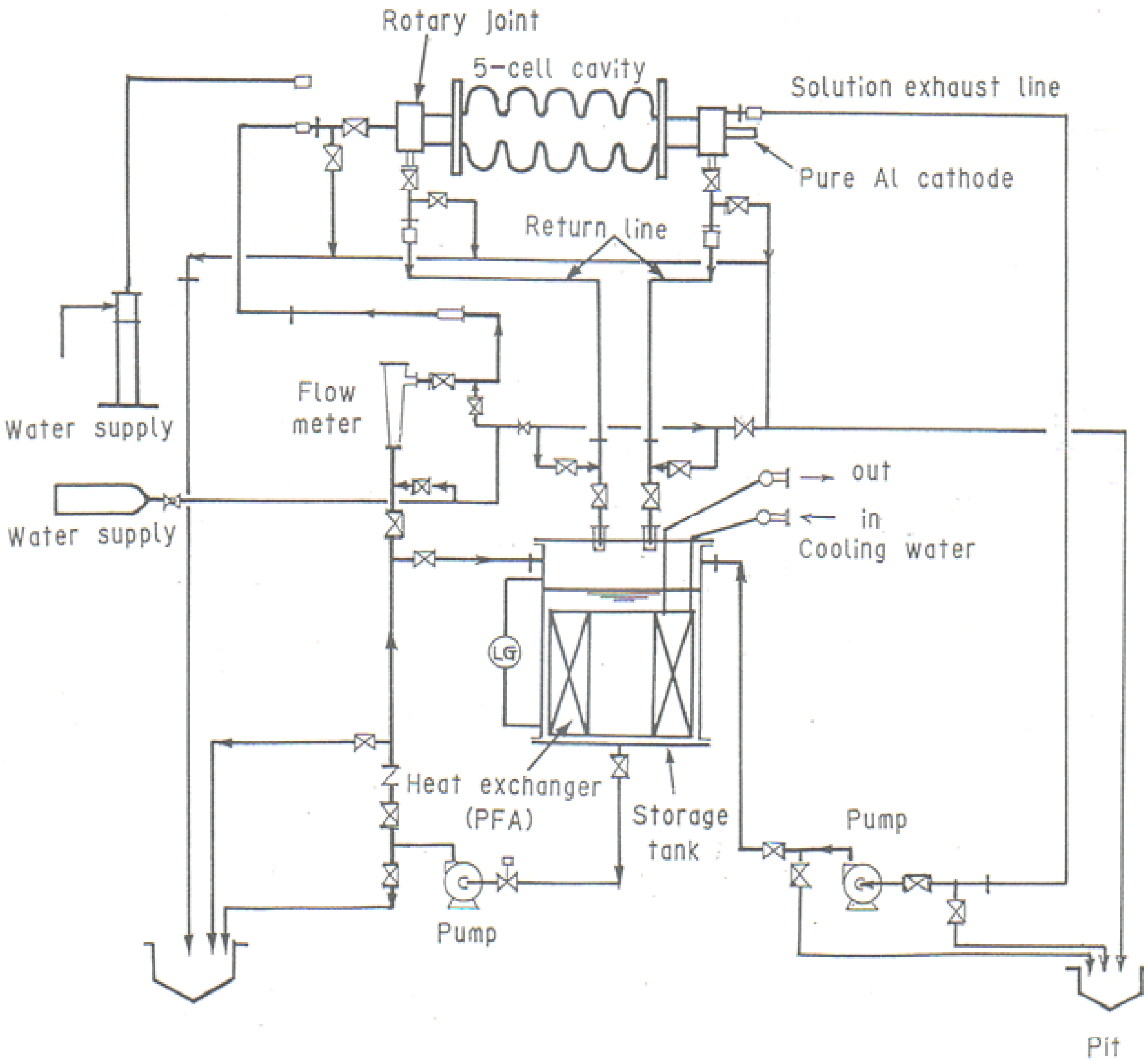
Successfully developed Horizontal EP system

TRISTAN SRF cavity

KEK/Nomura Plating

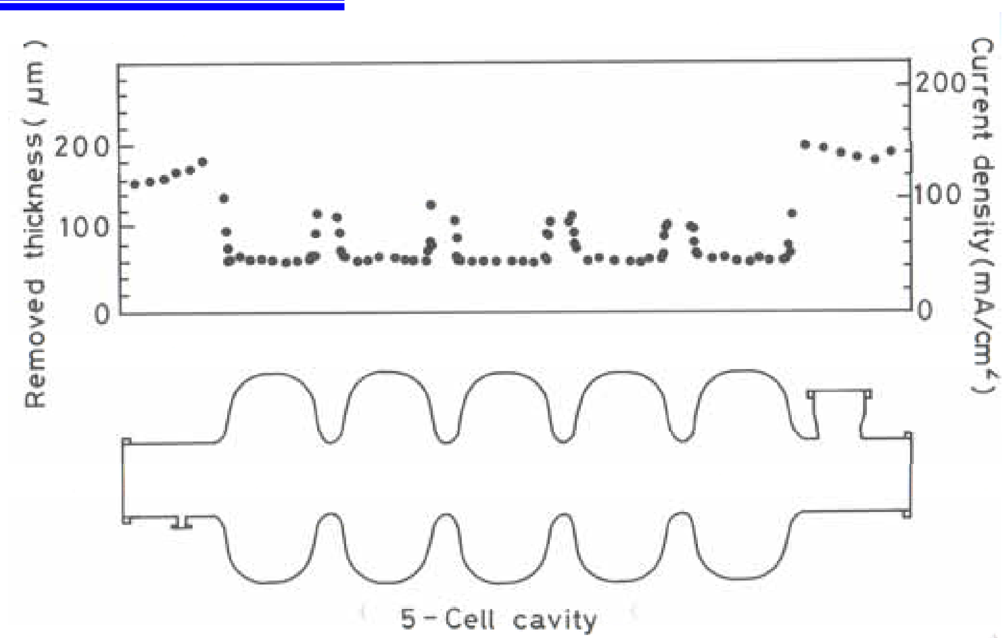
1300MHz single cell cavity

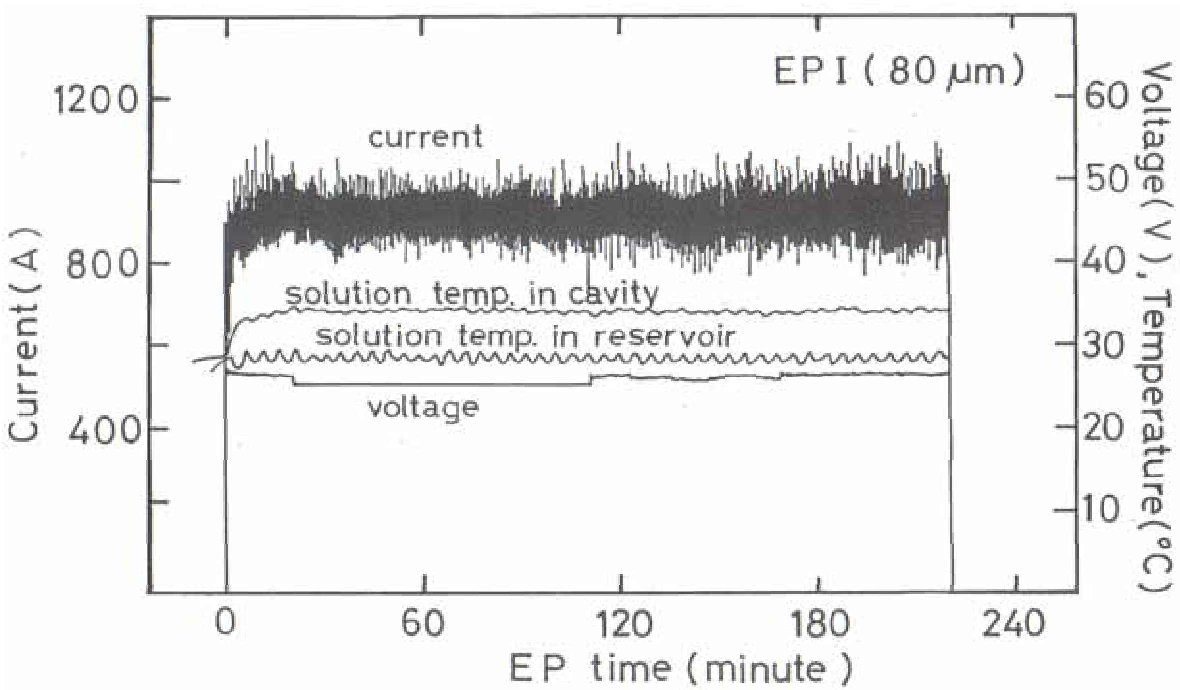
EP System Flow



EP Control

TRISTAN 508MHz 5-cell cavity, Continuously EP



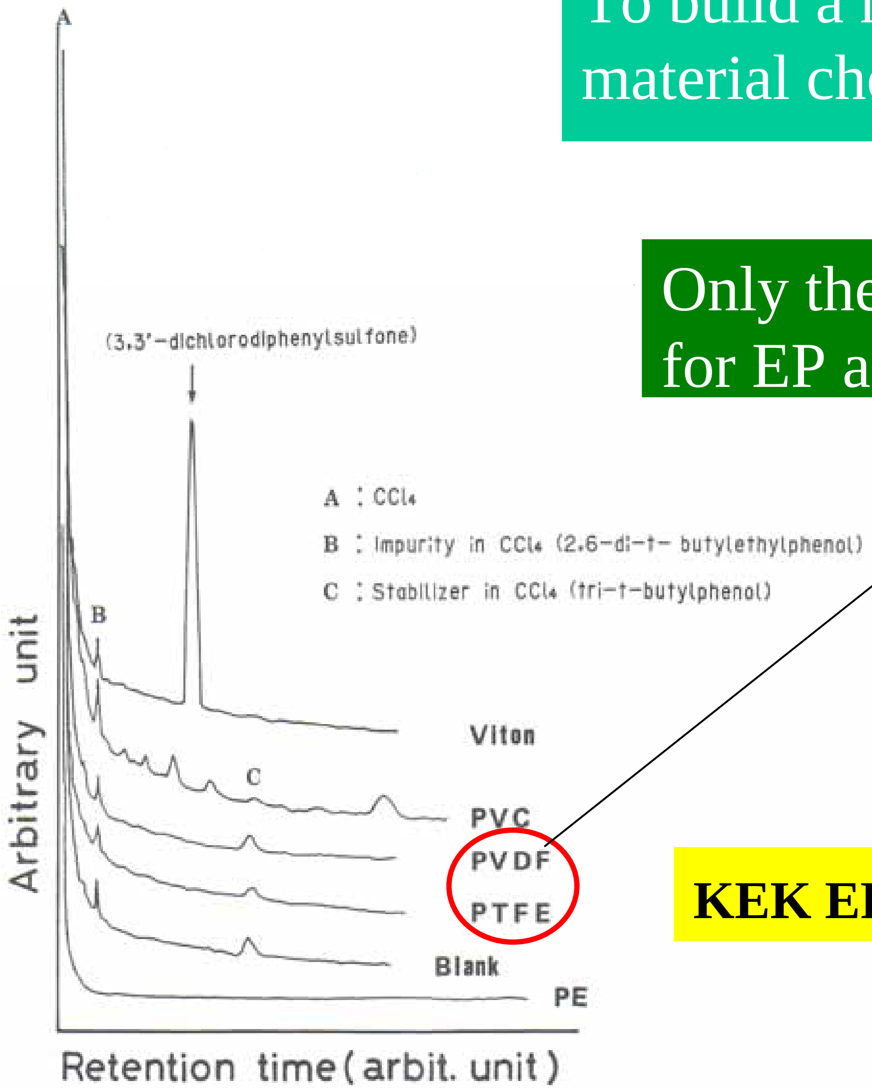


Uniform removal in each cell

Cathode extraction after EP：TRISTAN

Material Choice for the Reliable EP System

To build a reliable EP,
material chose in the EP acid line is curtail.

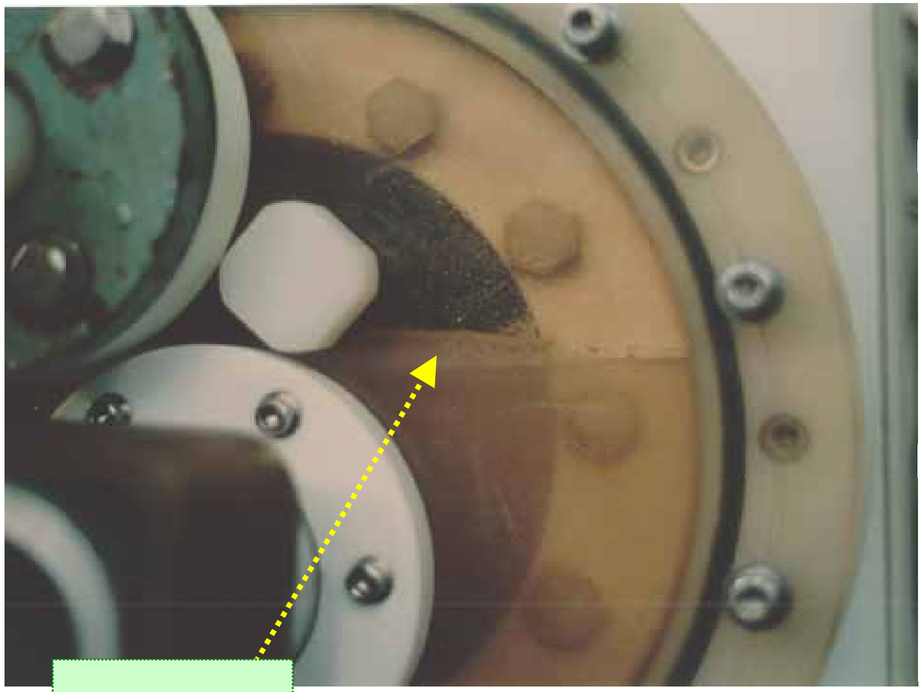


Only the Teflon is the reliable material for EP acid line.

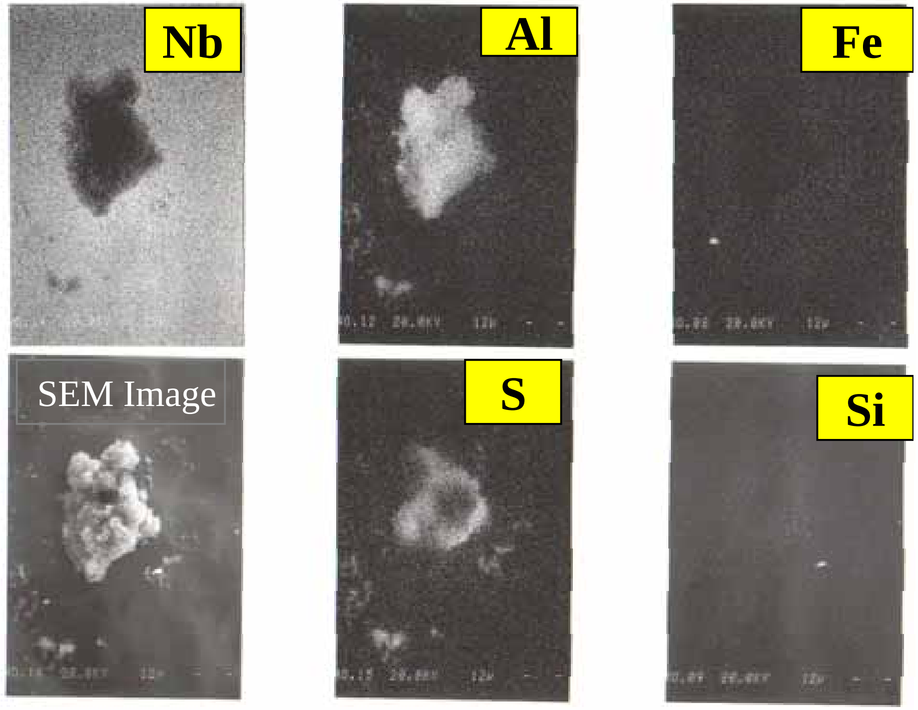
KEK EP system is still working for 20 years.

Dissolved chemicals into EP acid from Plastic Materials

Contamination Problem from Buffing



Sulfur



Al, Si, Fe are originated from buffing (TRISTAN)
S is due to decomposition of H_2SO_4 during EP process.

In the early stage of the TRISTAN mass production, these contamination brought heavy field emission on cavity performance.
The EP system was overhauled once. See next slide.

Sulfur Contamination in EP System

Teflon heat exchanger tube (Brand-new)

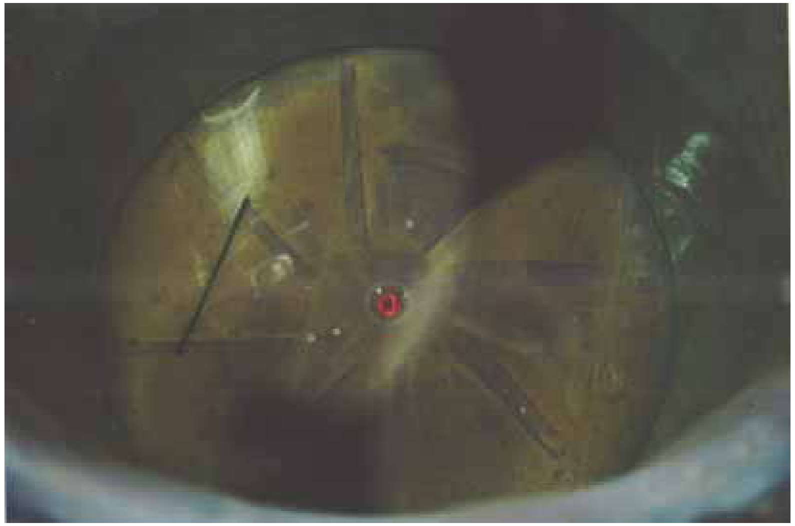
Teflon lining EP acid tank (brand-new)

Reduced H_2SO_4

↓

S precipitated on the contaminants

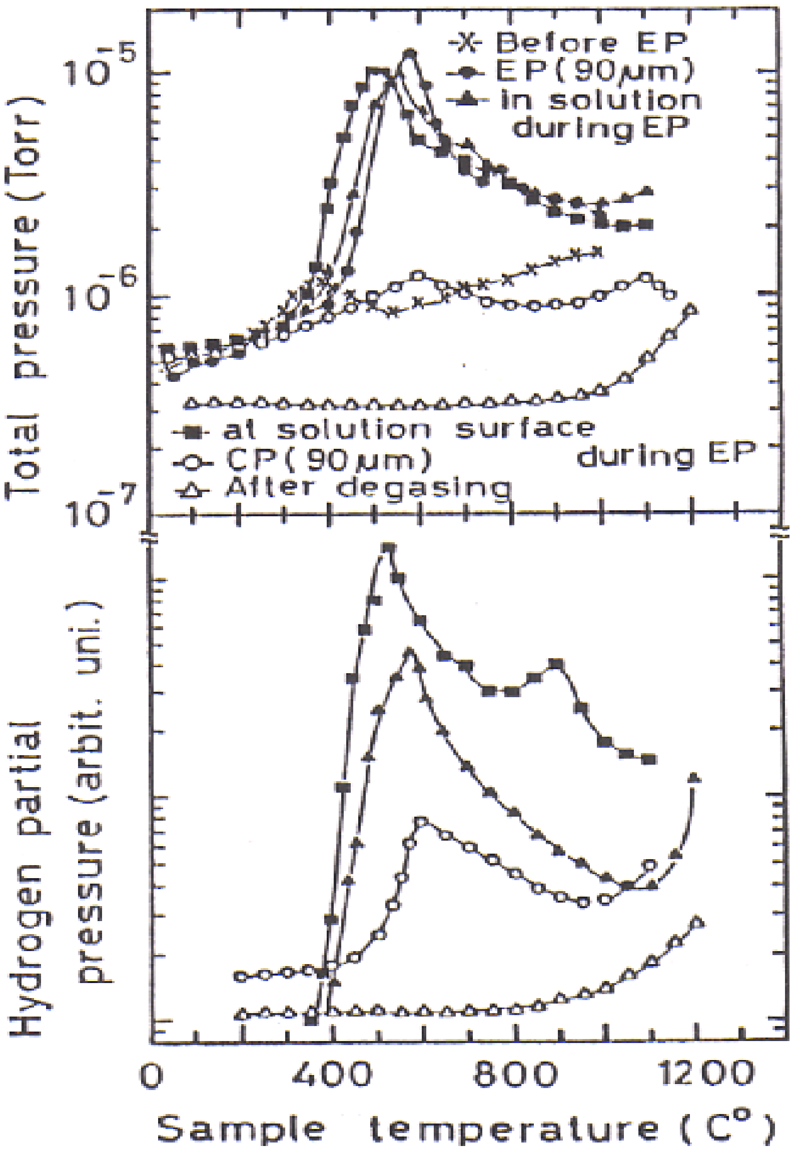
The contaminated heat exchanger

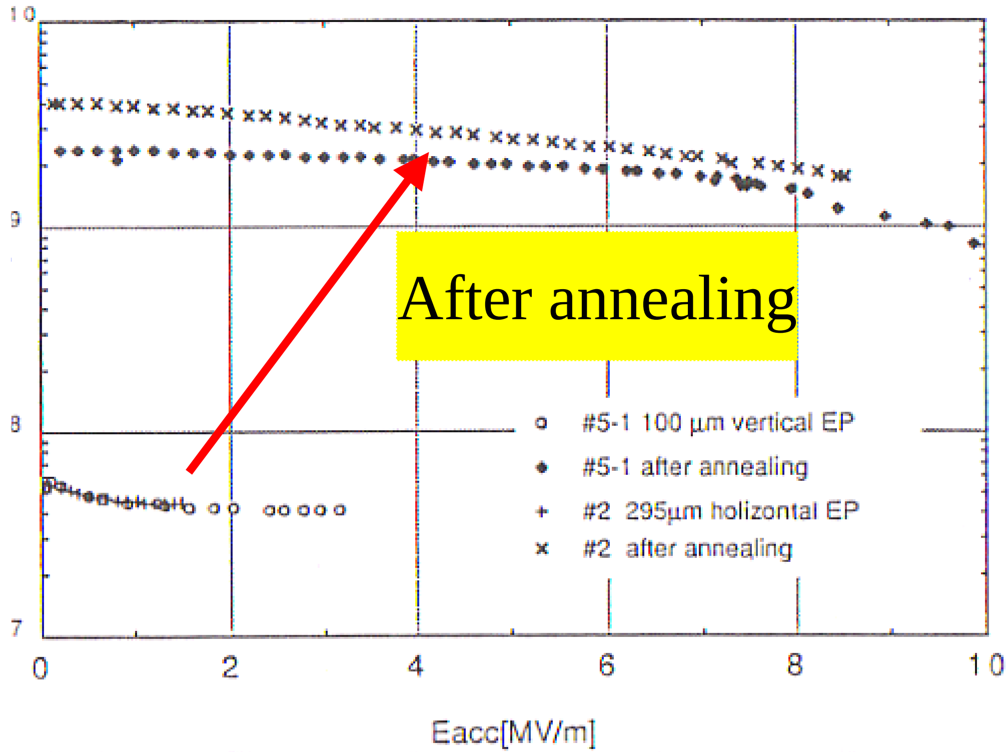


The contaminated EP acid tank

EP system was cleaned up.

8.4 Annealing

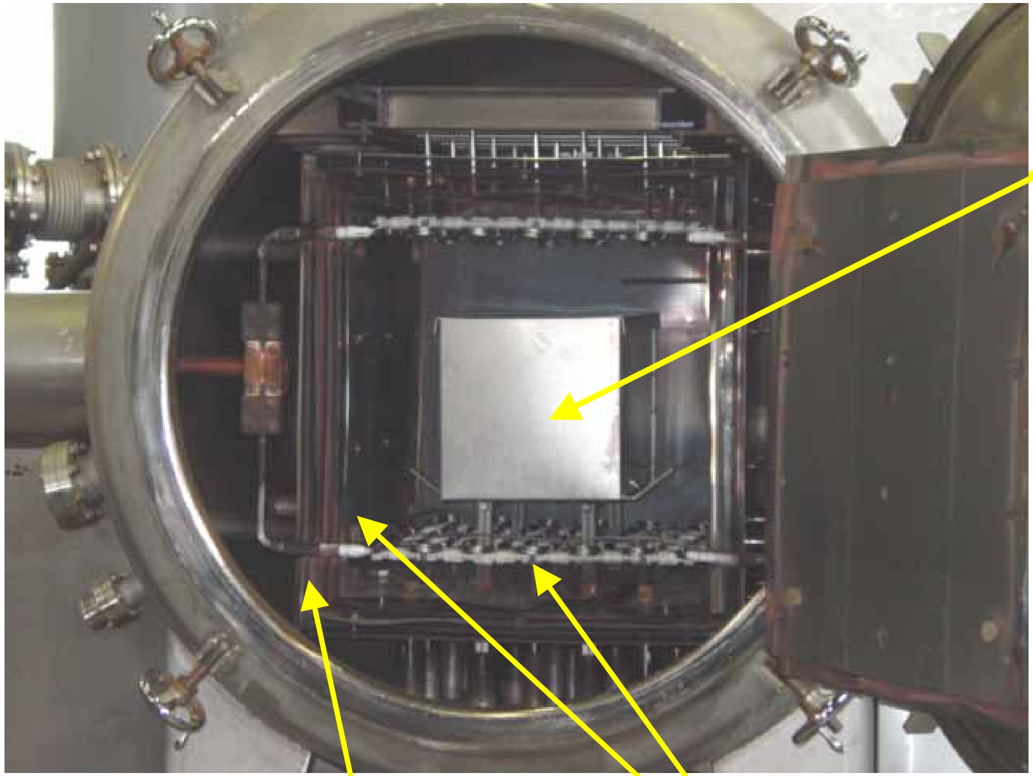




Hydrogen doped in niobium material is easily degassed at 700~800^{O}C.

This temperature dose not soft the niobium material.

Annealing Furnace in KEK Machining Center

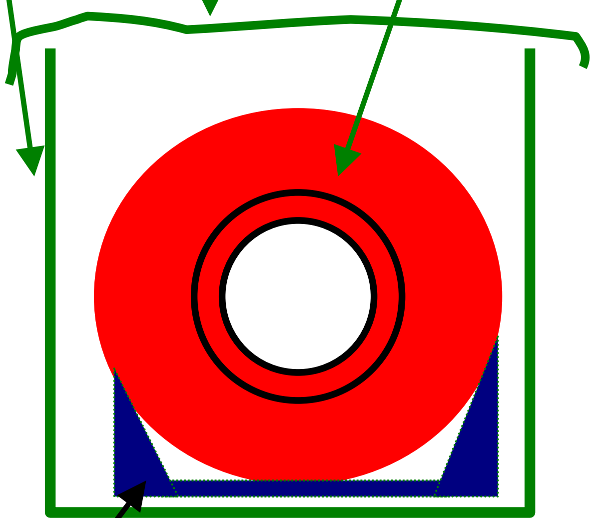


KEK Machining Center

Titanium box

Cavity

Ti cover



Molybdenum Heater

$1300^{O}C$ max, $1X10^{-6}$ Torr

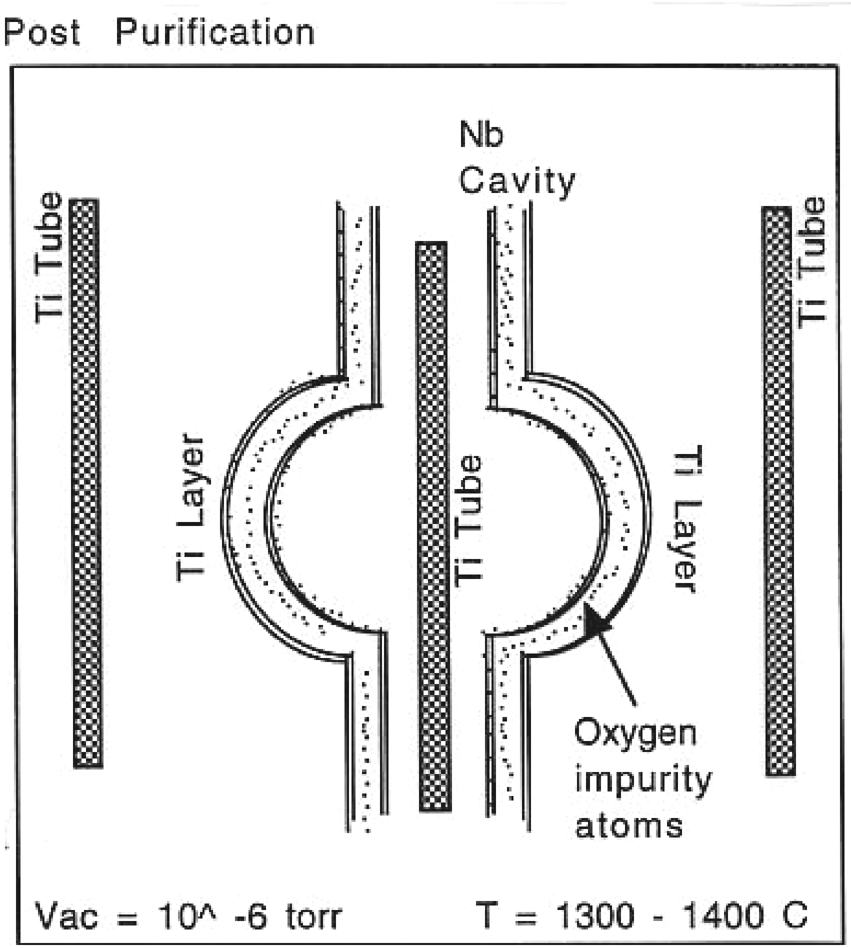
Radiation shield

Support
Stainless or ceramic

Large Vacuum furnace for ILC cavity (KEK Machining Center)

Specification：800^{O}C, ~E-6 Torr,
Working zone 500$^{\phi}$x 3000L

Post Purification (Titanization)



1400^{O}C annealing with Ti @ DESY TTF cavity

Using Titanium getter effect, Oxygen in Nb material can be reduced.

RRR cab be increased by this process.

Problem: Softening of the material

Diffusion Coefficients

$$O: 0.20\exp\left(-\frac{1.354\cdot 10^{4}}{T}\right)\ cm^{2}/sec$$

$$C: 0.043\exp\left(-\frac{1.670\cdot 10^{4}}{T}\right)\ cm^{2}/sec$$

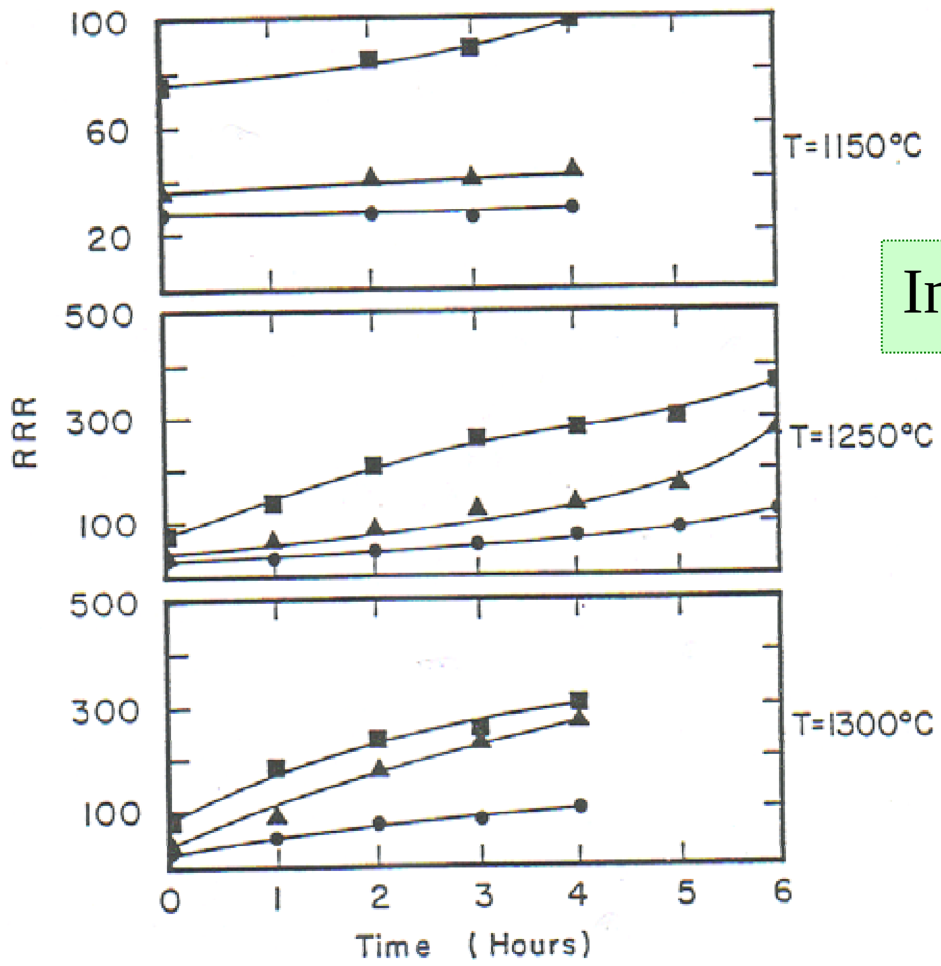
$$N: 0.0085\exp\left(-\frac{1.758\cdot 10^{4}}{T}\right)\ cm^{2}/sec$$

T - (^{o}C)	O - mm / hr	N - mm / hr	C - mm / hr
900	0.4	0.005	0.005
1000	0.6	0.008	0.008
1100	0.9	0.13	0.13
1200	1.2	0.19	0.19
1400	2.0	0.38	0.38

RRR Improvement by Post Purification

P.Kneisel

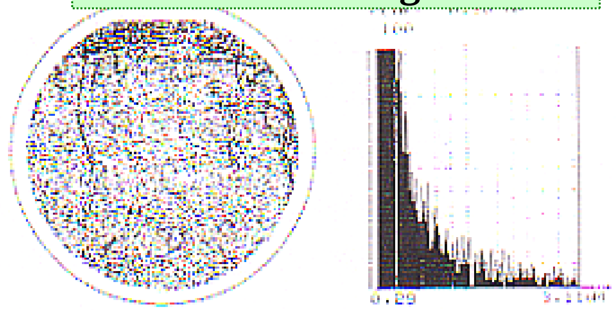
Improved by a factor 3



Dependence of the RRR-value on reaction temperature and reaction time for niobium samples of different purity exposed to titanium vapor (● 1/8" thick, RRR=27; ▲1/8" thick, RRR=37; ■ 1/16" thick, RRR=77)

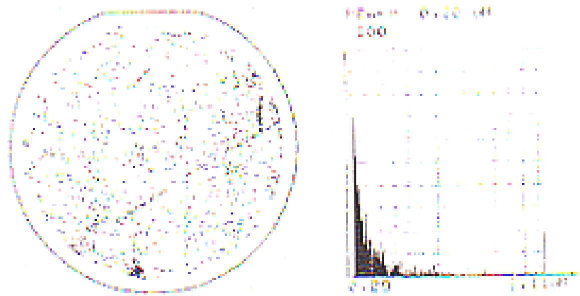
8.5 High Pressure Water Rinsing

TRISTAN rinsing method

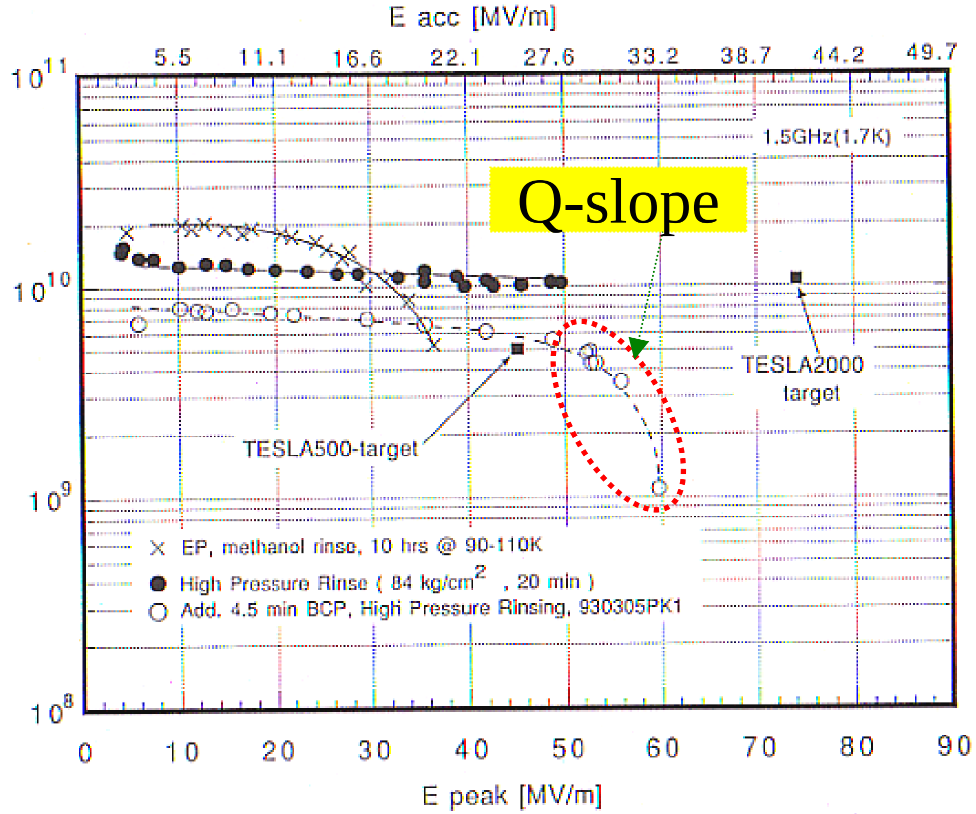


Particle size	Count
0.30-1.20 µm	5825
1.20-2.01 µm	405
2.01-3.00 µm	2720
> 3.00 µm	1069
Total	10019

HPR rinsing

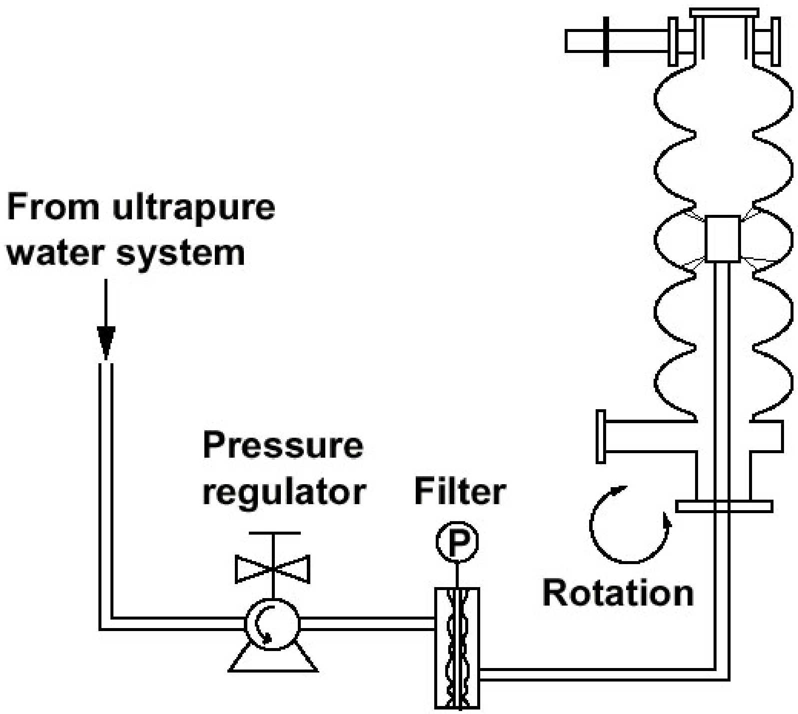


Particle size	Count
0.30-1.20 µm	646
1.20-2.01 µm	52
2.01-3.00 µm	282
> 3.00 µm	37
Total	1017



HPR is a very powerful tool to remove the particle contamination on niobium cavities.

HPR System

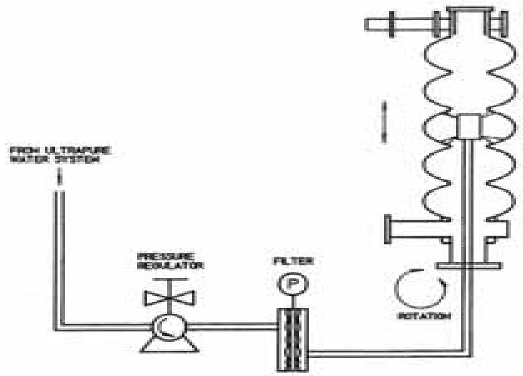


Nomura Plating

HPR Systems at other labs

DESY-System

Jlab HPR Cabinet

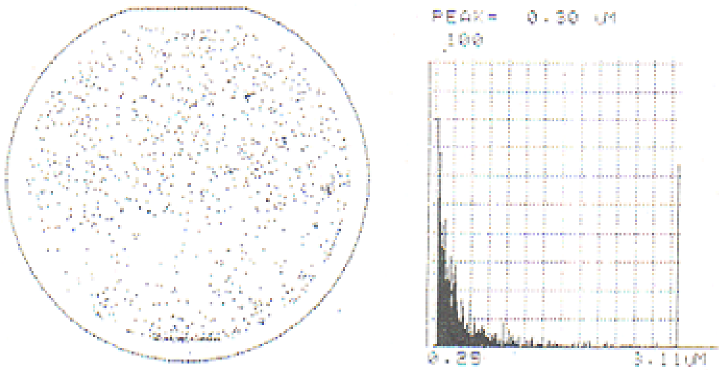


8.6 Megasonic Rinsing

An attractive rinsing method if compact oscillator can be product.

Megasonic Rinsing Effect

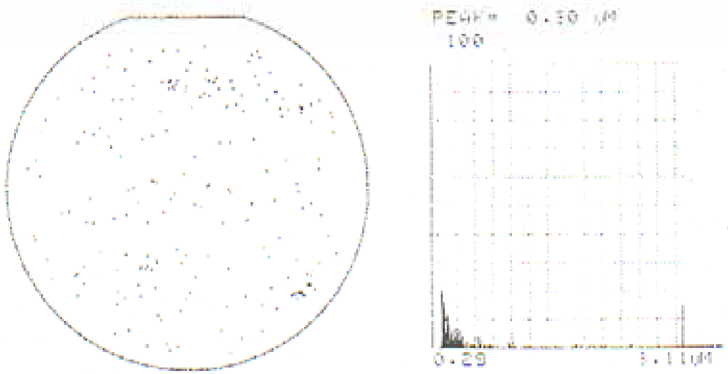
HPR rinsing



Particle size	Count
0.30-1.20 µm	673
1.20-2.01 µm	50
2.01-3.00 µm	305
> 3.00 µm	90
Total	1118

Fig. 11 Residual particles on a wafer surface after HPR; rinsing condition 19 in Table

Mega-sonic rinsing



Particle size	Count
0.30-1.20 µm	158
1.20-2.01 µm	5
2.01-3.00 µm	43
> 3.00 µm	18
Total	224

Fig. 12 Residual particles on a wafer surface after megasonic rinsing; rinsing condition 16 in Table 1.

Amount of particle Contamination is reduced to 1/5 of HPR

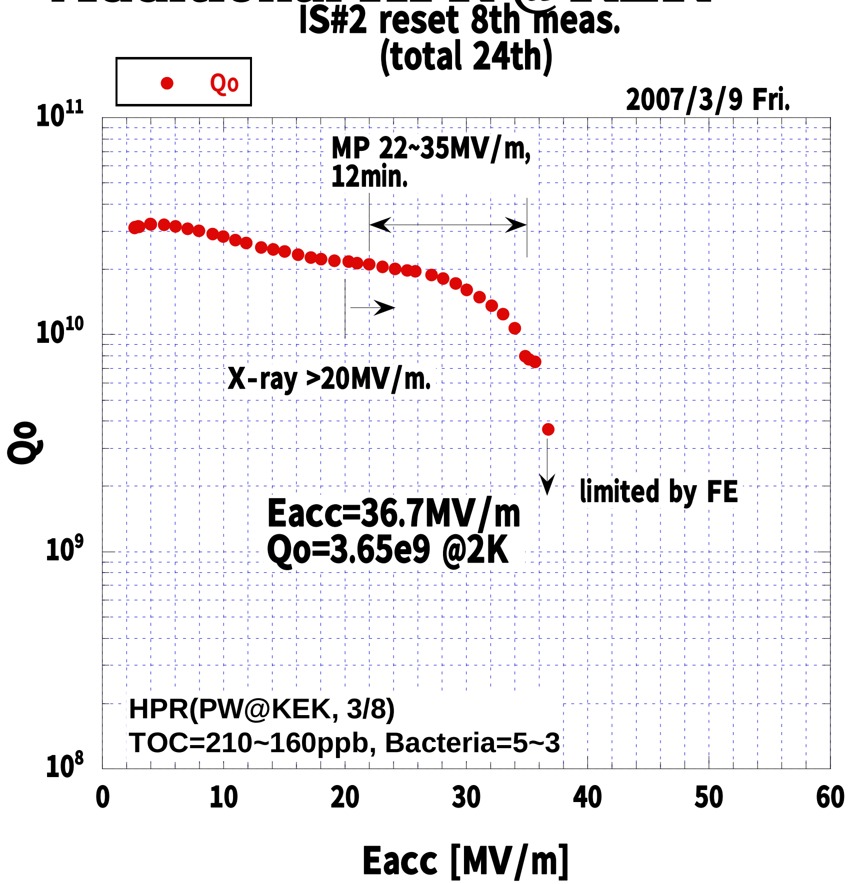
Megasonic rinsing can be an alternative of HPR ?

KEK will start investigation of Megasonic.

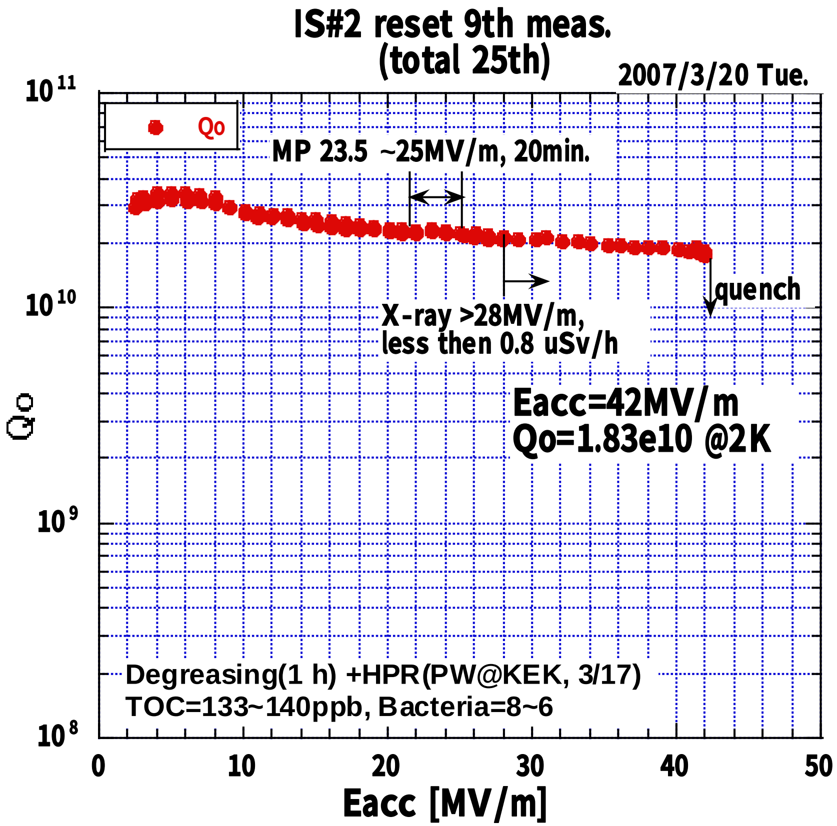
8.7 Degreasing after EP

Developed @ JLAB, J.Mammosser

Additional HPR @KEK



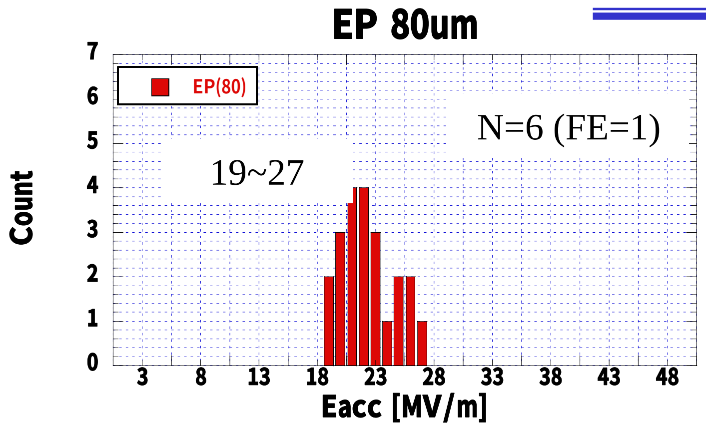
Additional Degreasing + HPR@KEK

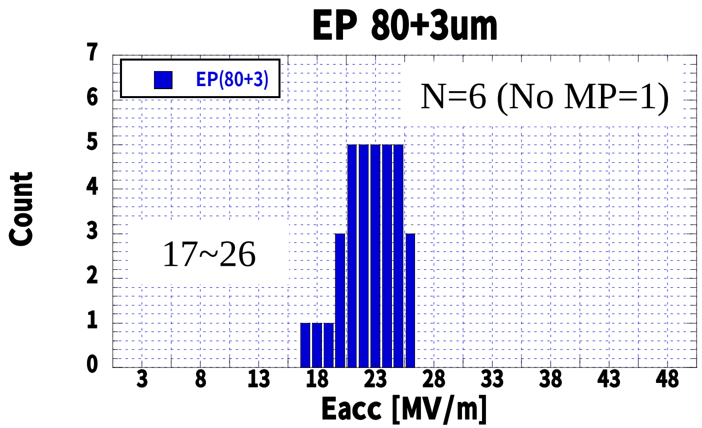


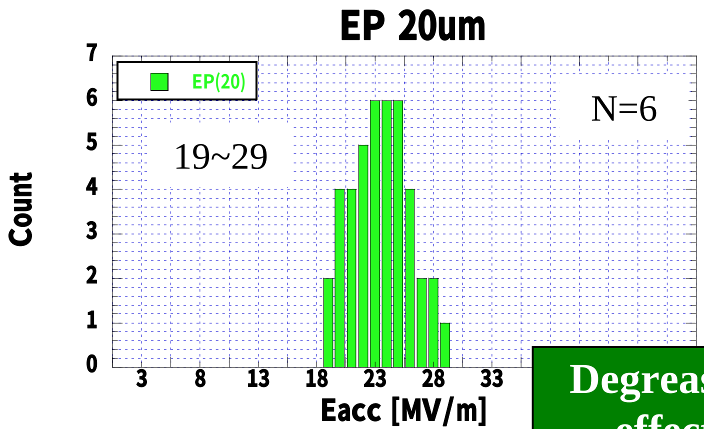
(Use Japanese degreaser)

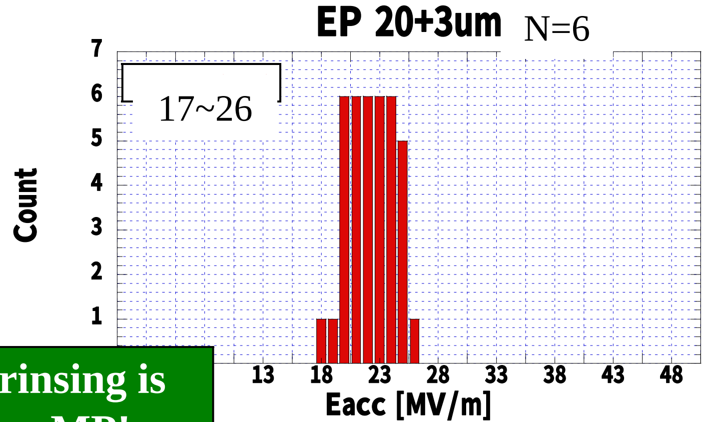
Degreasing is very much effective to eliminate contamination !

Multipacting

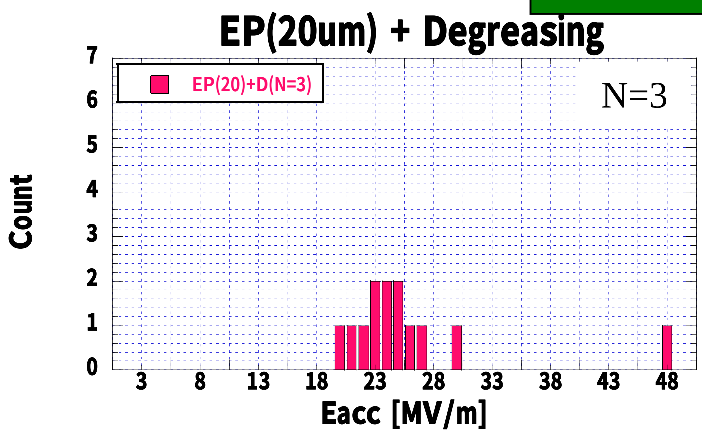


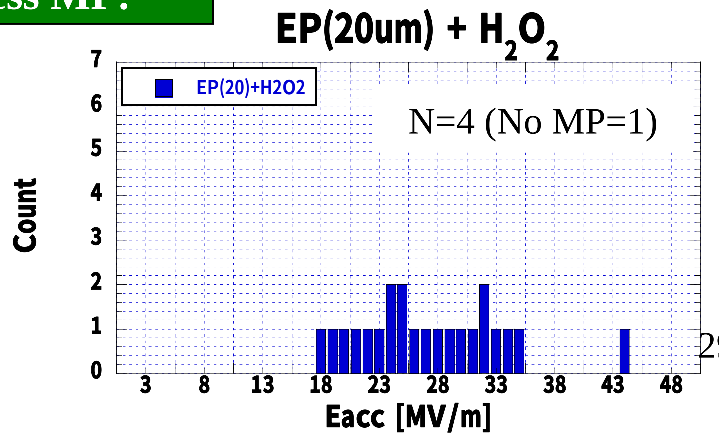






Degreasing or H_2O_2 rinsing is effective to suppress MP!





8.8 Cleanroom Assembly