PHY905 Section 4
Ion Accelerator Technology
Lecture 16
- SRF Surface Preparation Technologies -

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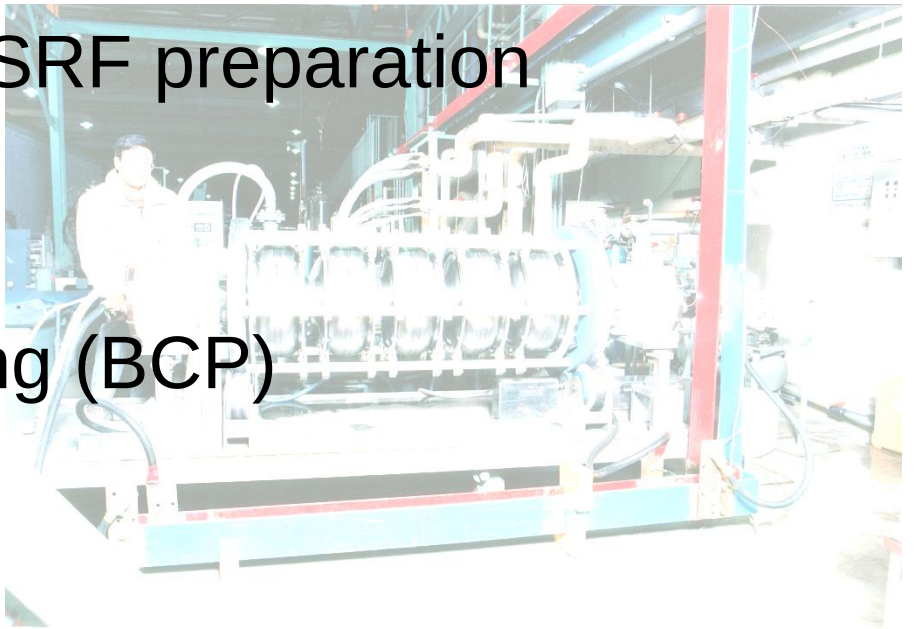
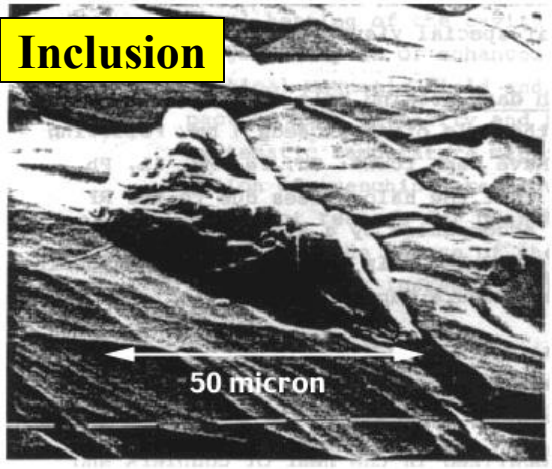
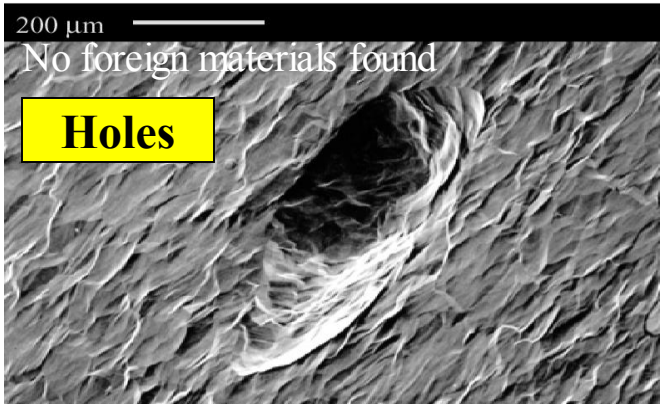
16 SRF Surface Preparation Techniques

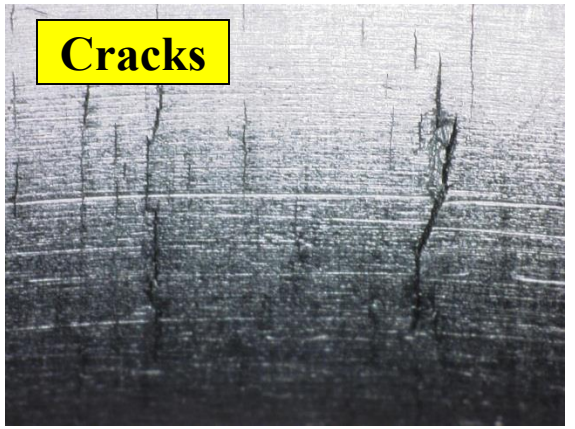


16.1 History of development of SRF preparation techniques
16.2 Mechanical grinding
16.3 Buffered Chemical Polishing (BCP)
16.4 Electropolishing（EP)
16.5 Heat treatment
16.6 Post purification
16.7 High Pressure Water Rinsing（HPR）
16.8 Megasonic rinsing
16.9 Cleaning methods post EP

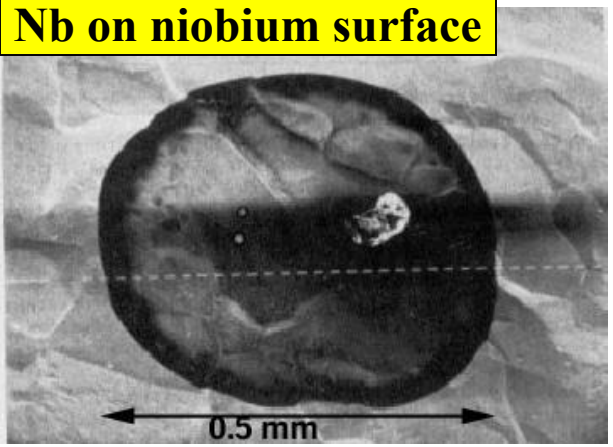
Various Surface Defects on the SRF Surface

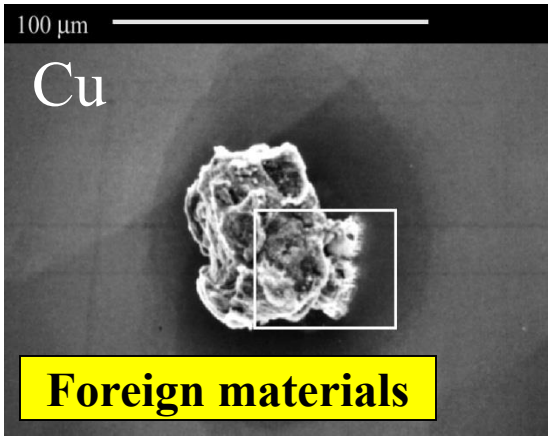


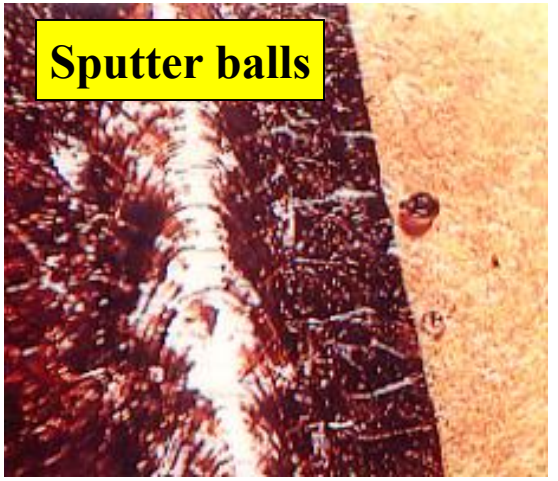




Surface defects, holes can also cause TB

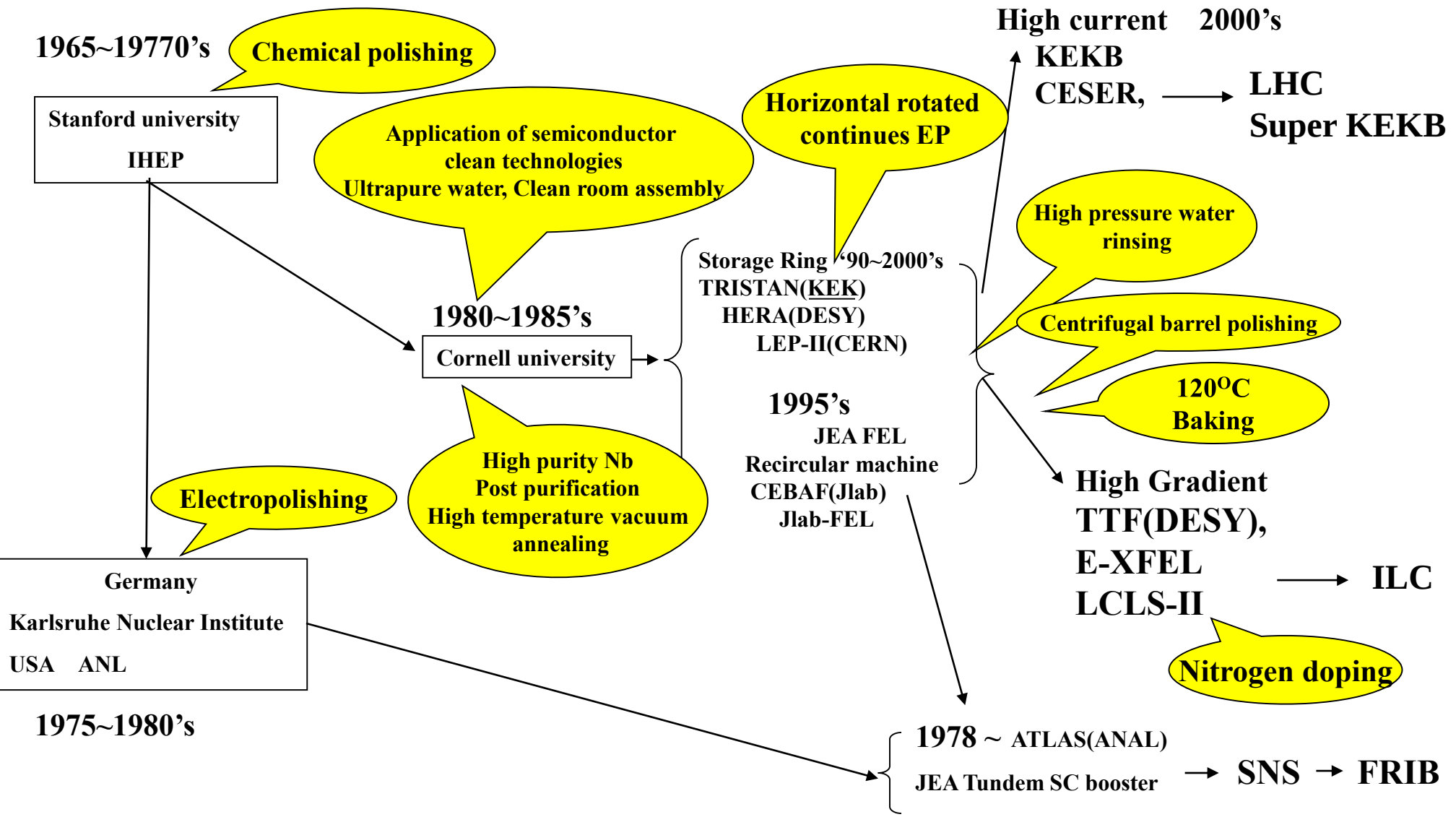






It is important to eliminate the defects on the SRF surface by mechanical grinding.

16.1 History of Development of SRF Preparation Technologies



16.2 Mechanical Grinding

Buffing all TRISTAN
320 half cells

Buffing (Emery)

Pros of buffing

- Very powerful
- High reliability for defect removal
- Surface rightness control
- Possible to remove EBS welding defects

Cons of buffing

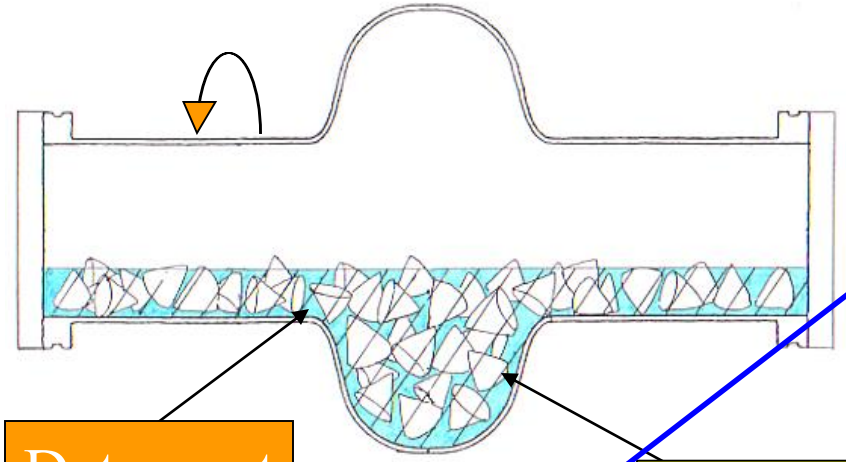
- Cost
- Buffing is difficult to apply for EBW assembled cavities

KEK TRISTAN

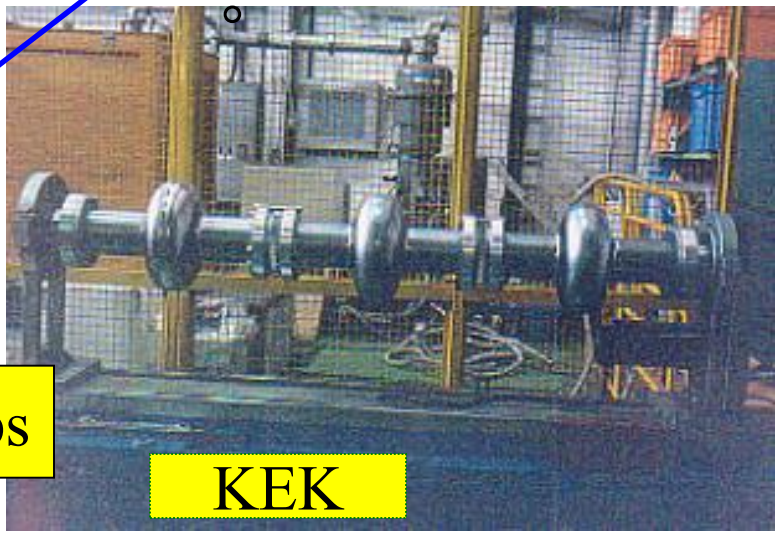
- Applied all half cells
- Mechanical grinding EBW seals

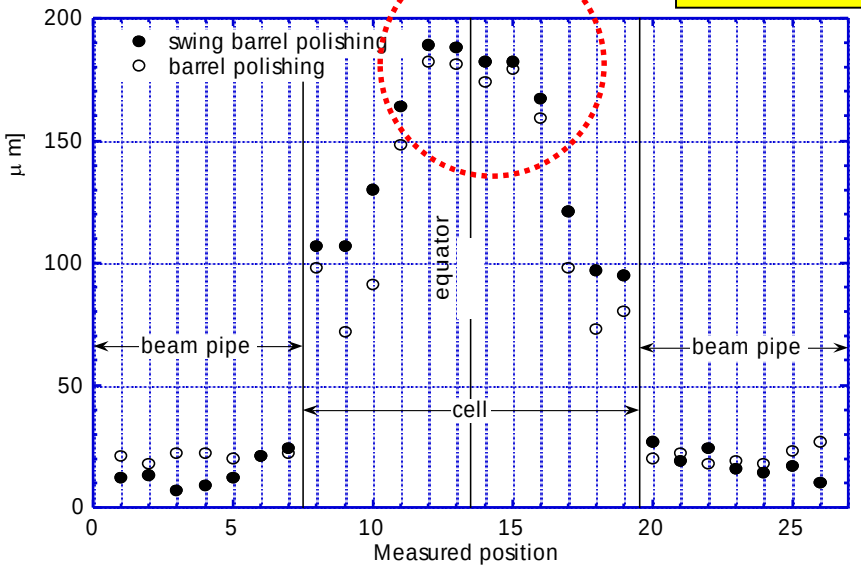
Barrel Polishing (BP)

100 rotations／min (rpm)



Maximum polishing rate at equator EBW seam.





Pros

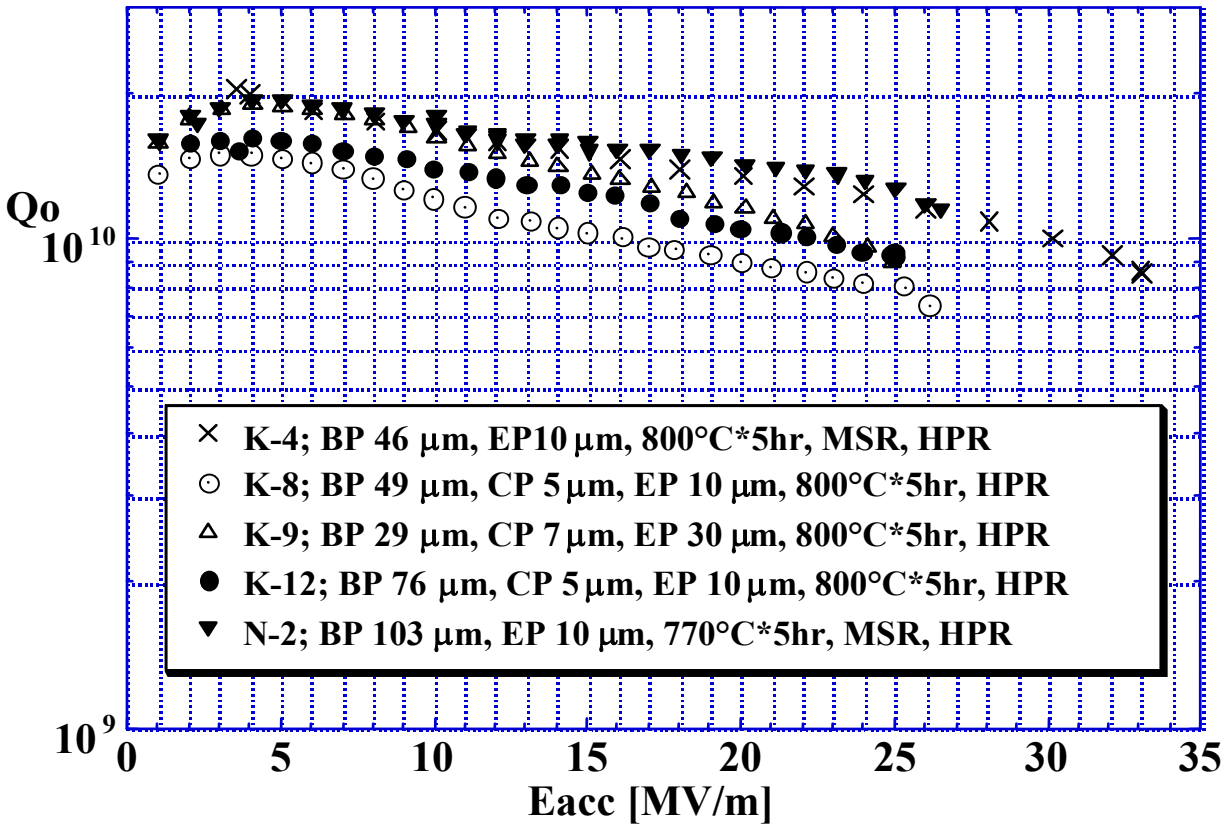
- **Simple**
- **Low cost**

Cons

- **Slow polishing speed, 3μm/day, for instance 10 days for Nb cavities**

Availability BP as the Pre-treatment of Electropolishing

ILC type 1.3GHz single cell cavities



- Reached Eacc 25MV/m with high reliability!!
- Why limited 25MV/m ?

Process:

BP(30 ~ 100 μm)

⇩ Remove surface defects by BP

EP or BCP (5 ~ 30mm) or BP + EP

⇩ Remove surface contamination by BP

Annealing at 800°C for 5hrs

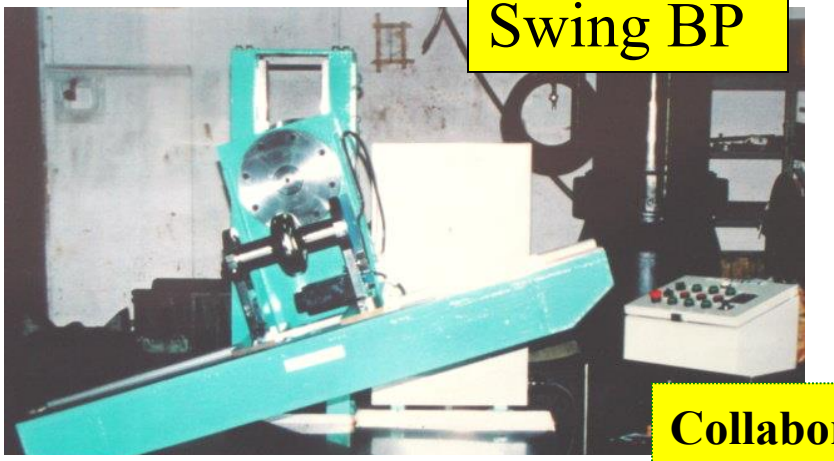
⇩ Hydrogen degassing

High pressure water rinsing

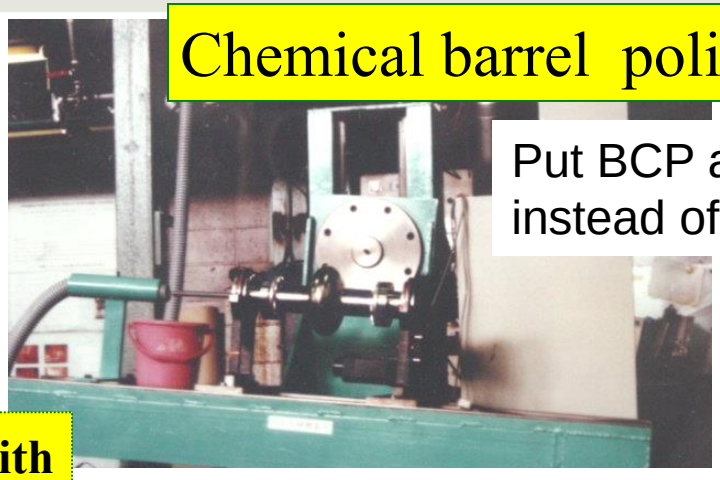
Remove particle

Further R&D of Mechanical Polishing to increase the Material Removal Rate

Swing BP

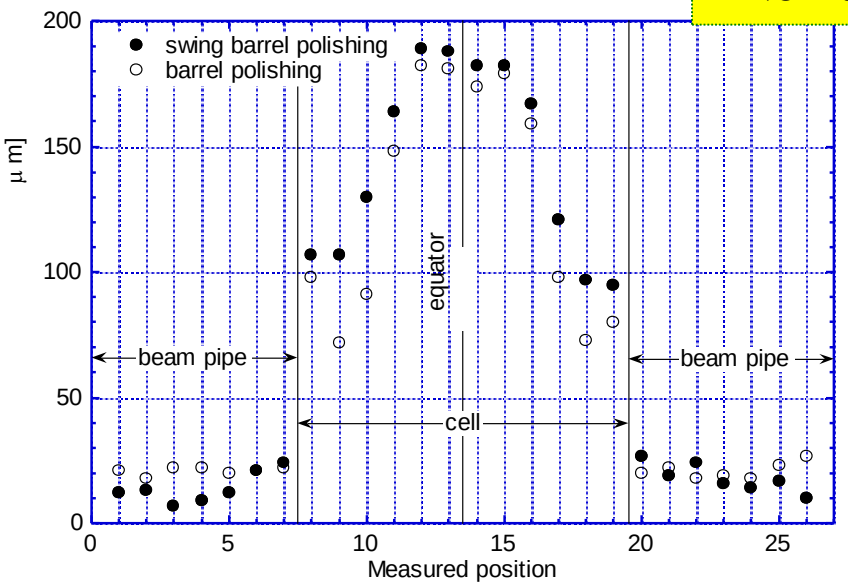


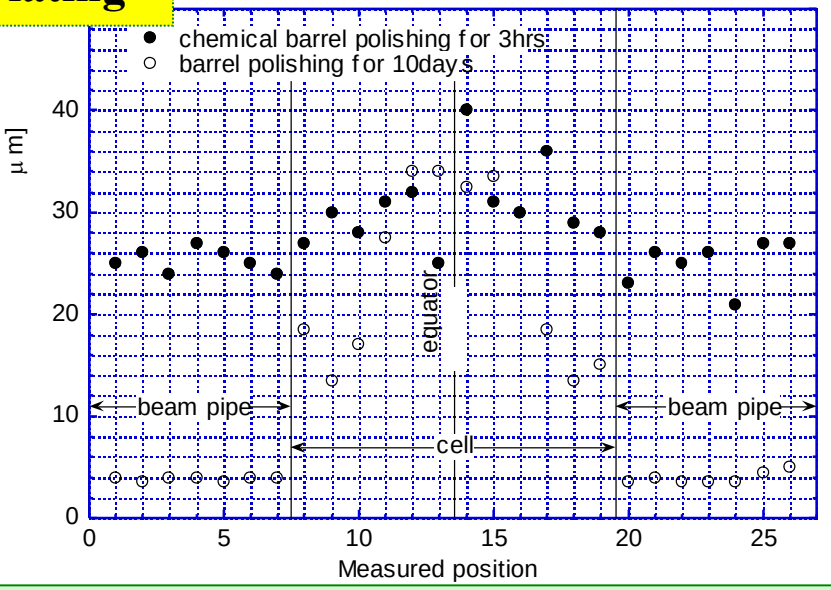
Chemical barrel polishing



Put BCP acid instead of detergent

Collaboration with Nomura Plating



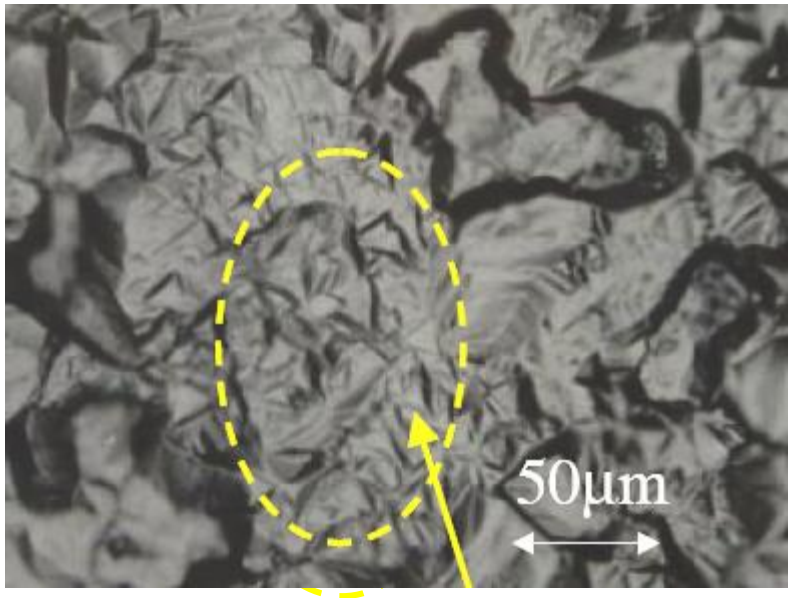


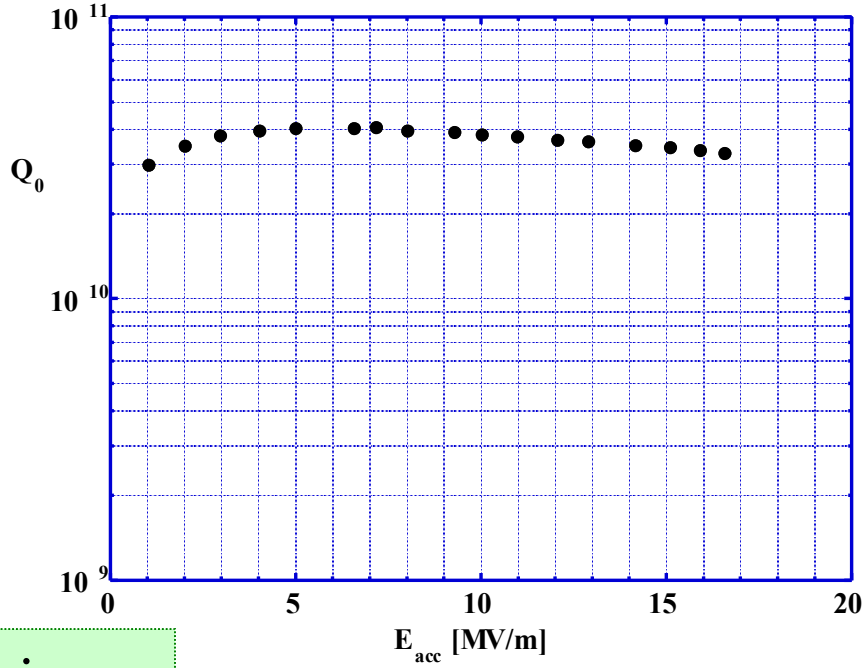
No improvement in polishing speed.

Large polishing speed but low cavity performance.

Cavity Performance by Chemical Barrel Polishing

Chemical BP＋750^{O}C Anneal + EP 50μm

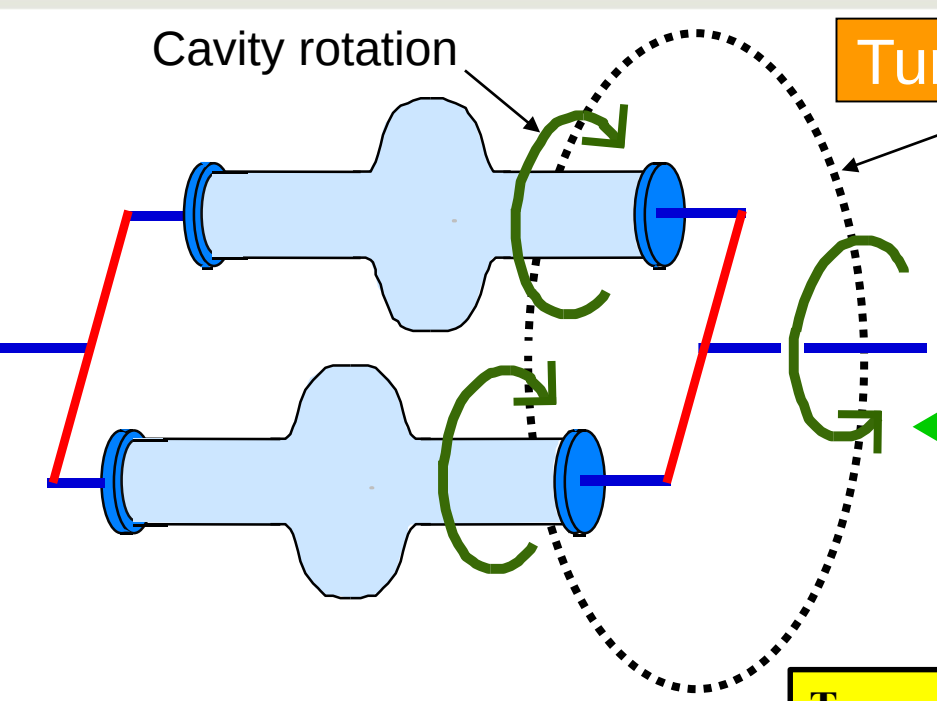




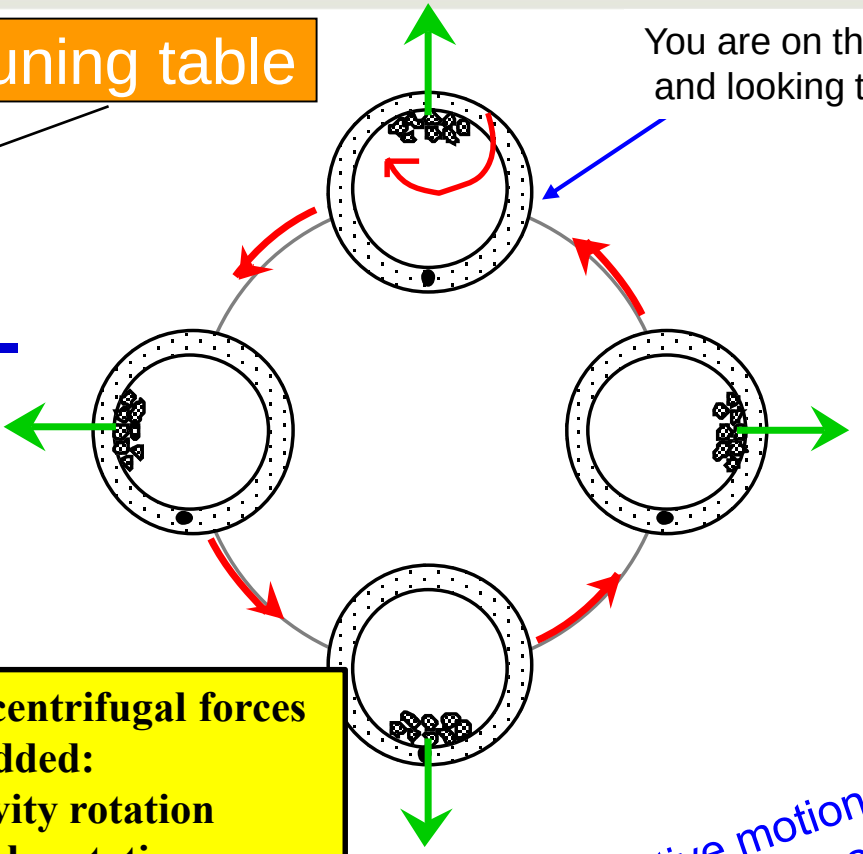
Feature: triangle shape grain boundaries 。

Gradient sharply limited below 20MV/m.

Centrifugal Barrel Polishing(CBP)
Innovation of the mechanical grinding for SRF cavities

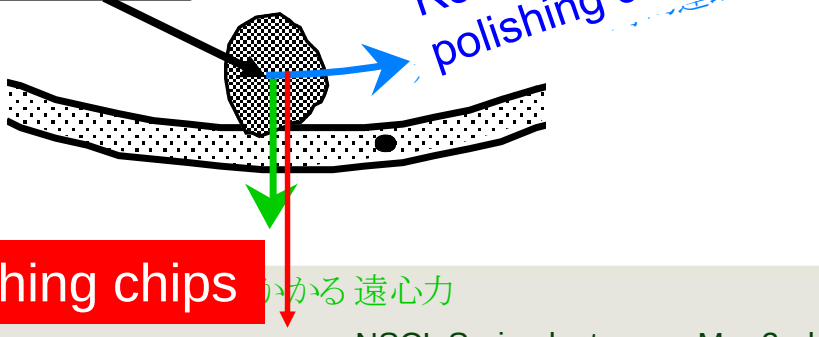


Tuning table



- **Table is tuning**
- **Cavity is oppositely rotating with the table tuning.**
- **The gear ratio between the tuning table and cavity is 1:1**

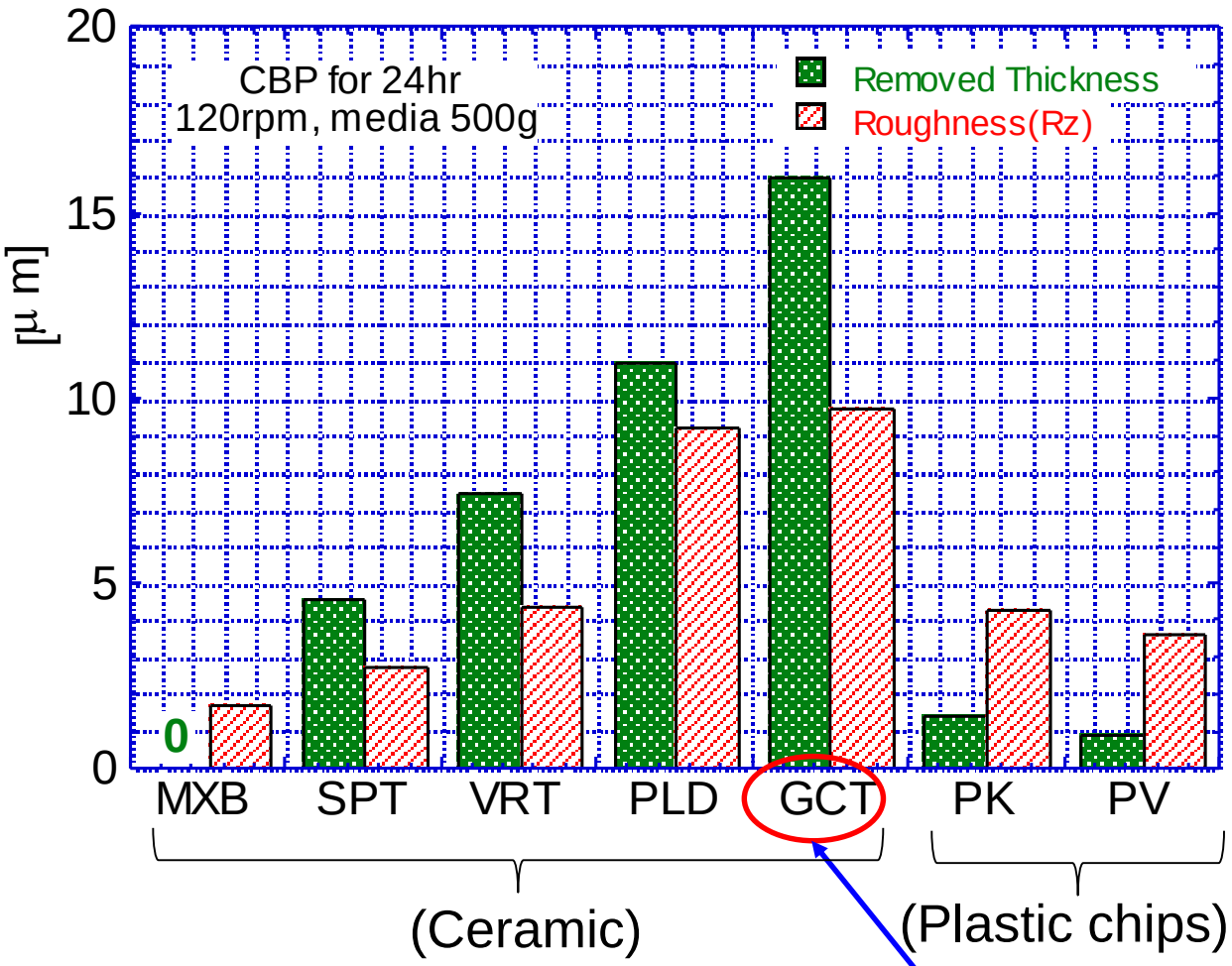
Two centrifugal forces are added:
- **Cavity rotation**
- **Table rotation**



Force loaded on polishing chips

かかる遠心力

CBP Material Removal Speed with Various Different Polishing Chips



Faster (a factor 5) than conventional BP by the sample test in the fastest case (GCT chips)

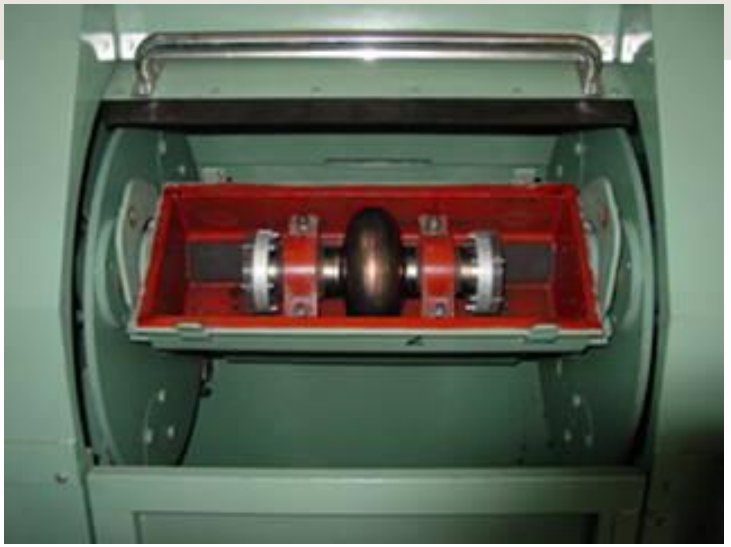
Conventional BP takes 7-10 days

⇩

CBP shortens only for 4 hrs
Big impact on polishing speed.

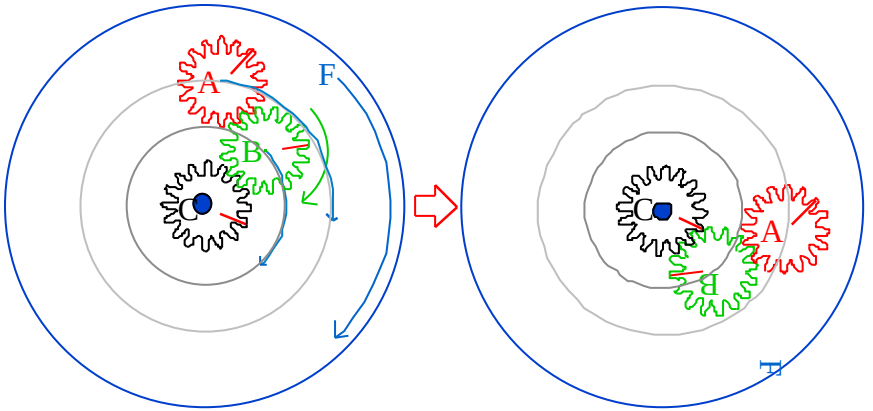
Triangle shaped SiC polishing chips

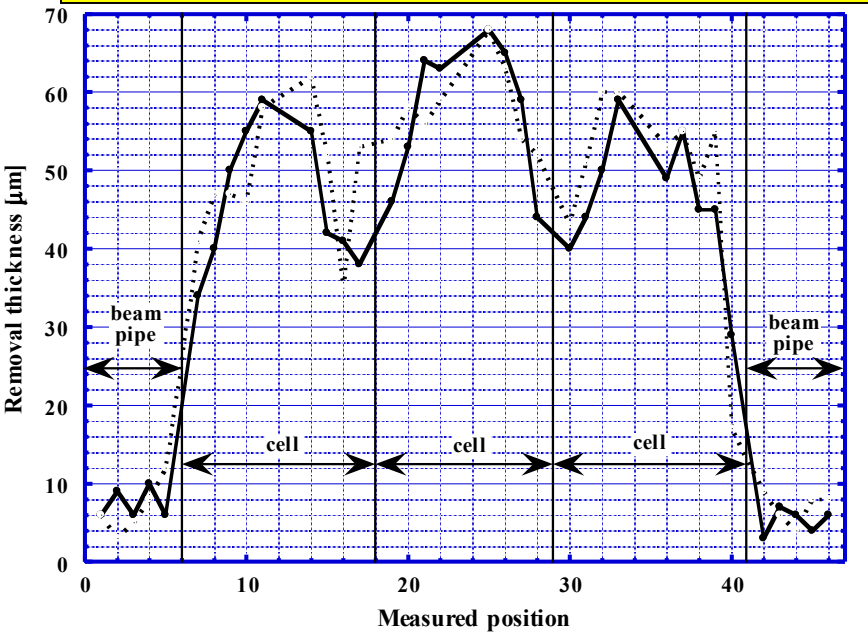
CBP System at KEK



Front view of KEK CBP system

CBP with 1300MHz 3-cell Nb cavity





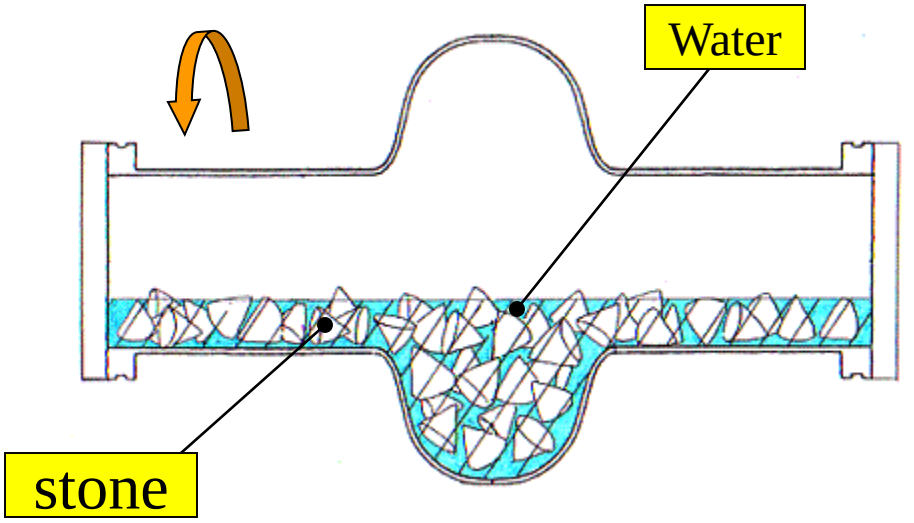
Rotation mechanism : cavity rotation/Table rotation gear ratio 1:1

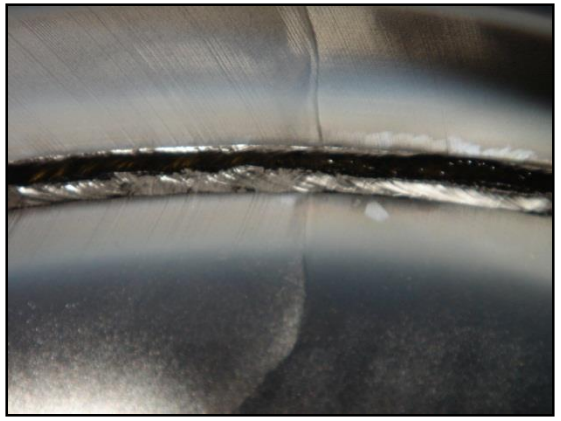
CBP'ed Surface on Equator EBW Seam

Rotated ~100 rpm on a oppositely rotating table

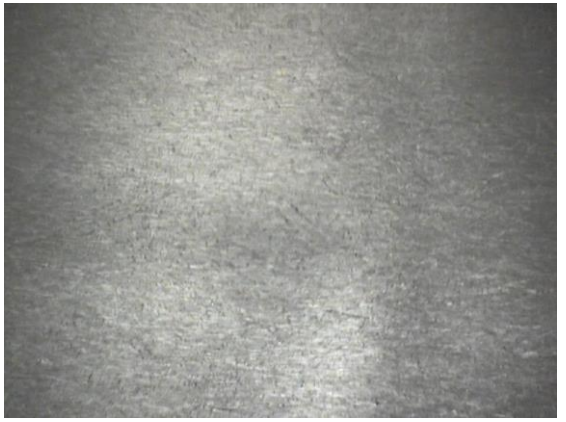
CBMM cavity

Rough stone (SiC) : 5 times
Green stone (SiC) : Once
Brown stone (plastic) : Once
White stone (plastic) : Once
Totally ~ 200 **μm removed @ equator**

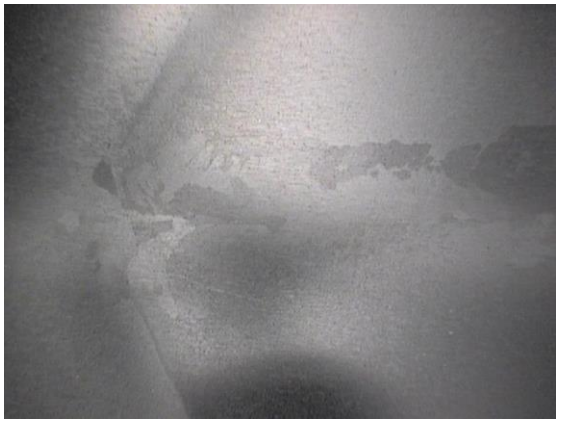




Before CBP (equator EBW seam)



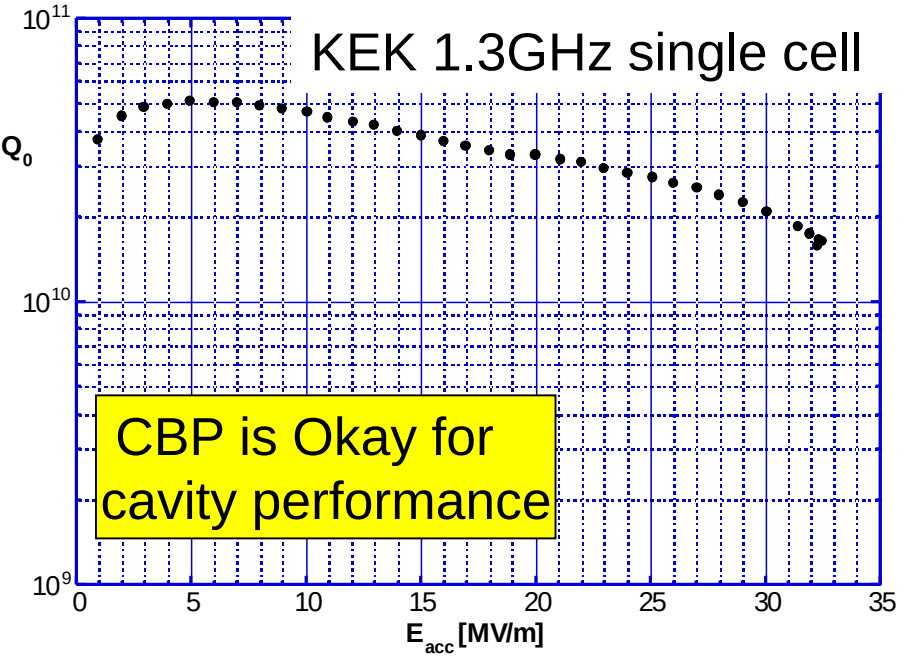
After CBP



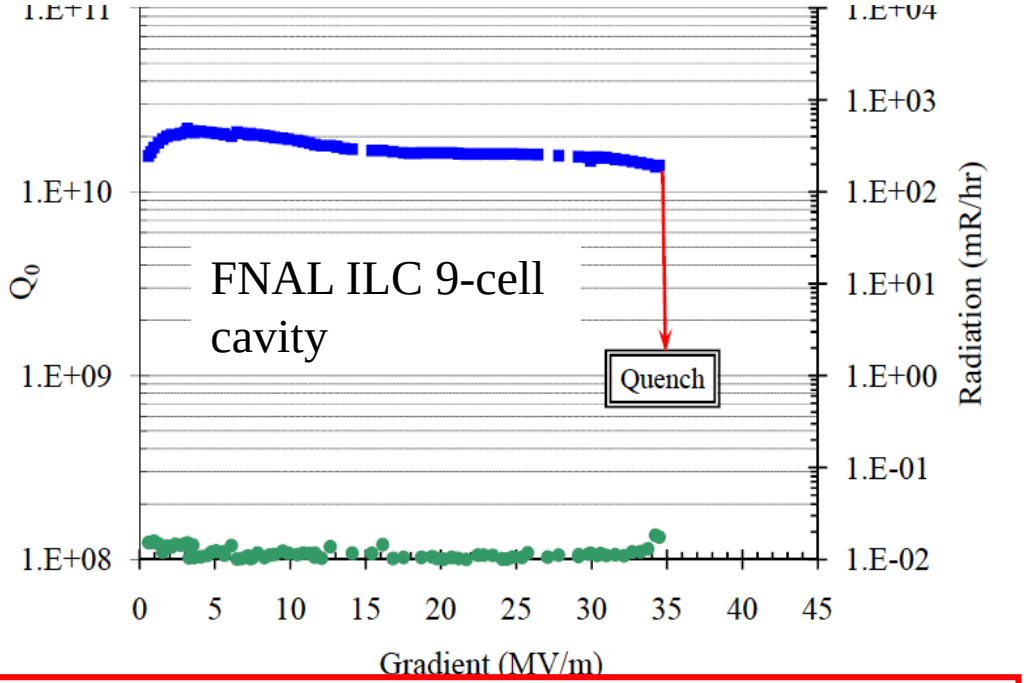
After light etching (10μm)

SRF Cavity Performance by CBP

CBP+750°C annealing + EP 50μm

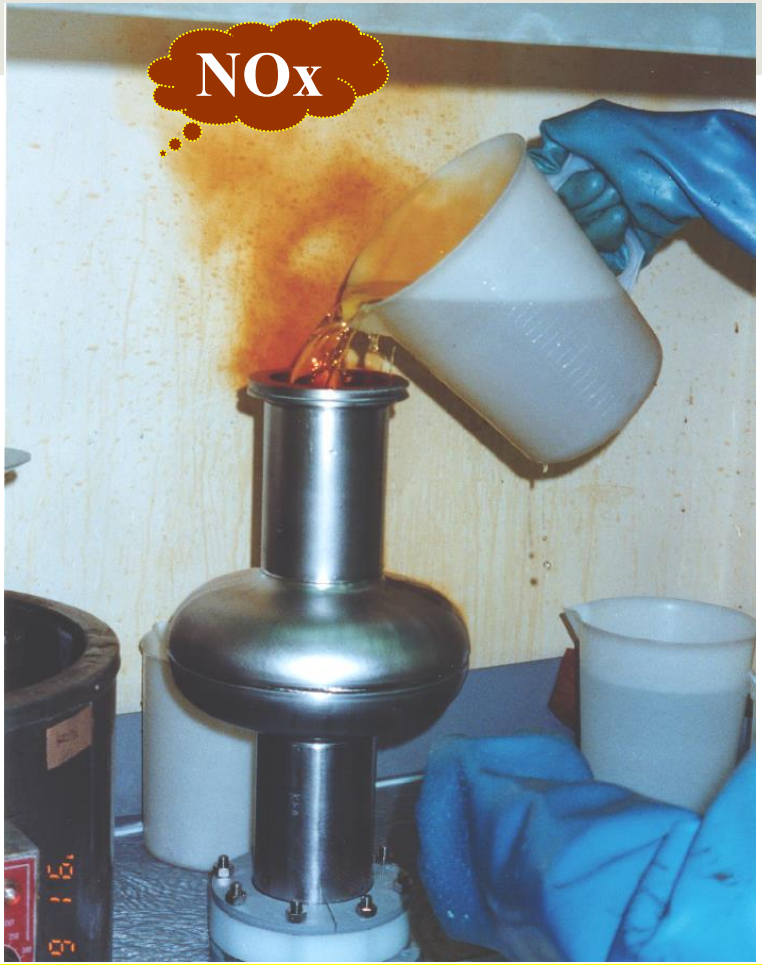


Later CBP technology has been transferred to Jlab and FNAL.



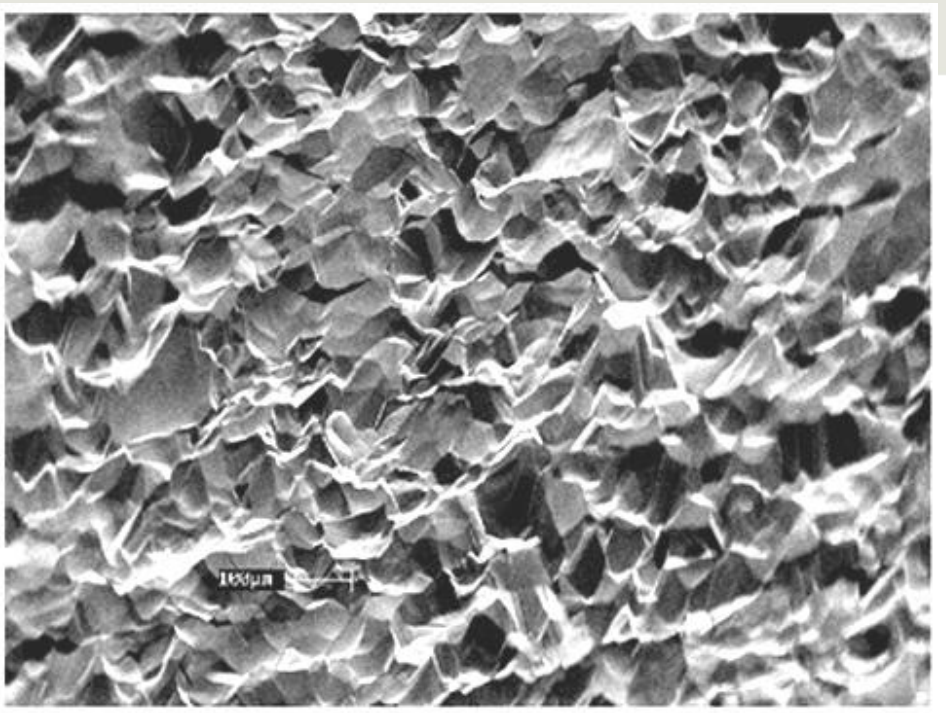
FNAL has applied CBP to the cavity (ILC 9-cell cavity) limited the gradient up to 18MV/m on December 2010 to see how the performance improved by CBP.
Process: CBP + Annealing + EP(40 μm)
The cavity gradient improved up to 35MV/m (ILC goal).

16.3 Buffered Chemical Polishing (BCP)



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

No contributes to chemical reaction, Buffers reaction (reduce the reaction speed)



BCP'ed surface (Nb)

Pros of BCP

- **Simple and high polishing speed**

Cons

Surface roughness is not as low as as EP

Chemical reaction:

$$6Nb+10HNO_3 \rightarrow 3Nb_2O_5+10NO \quad +5H_2O$$
$$Nb_2O_5 +10HF \rightarrow 2NbF_5 + 5H_2O$$

$$6Nb+10HNO_3+30HF \longrightarrow 6NbF_5+10NO \quad +20H_2O$$

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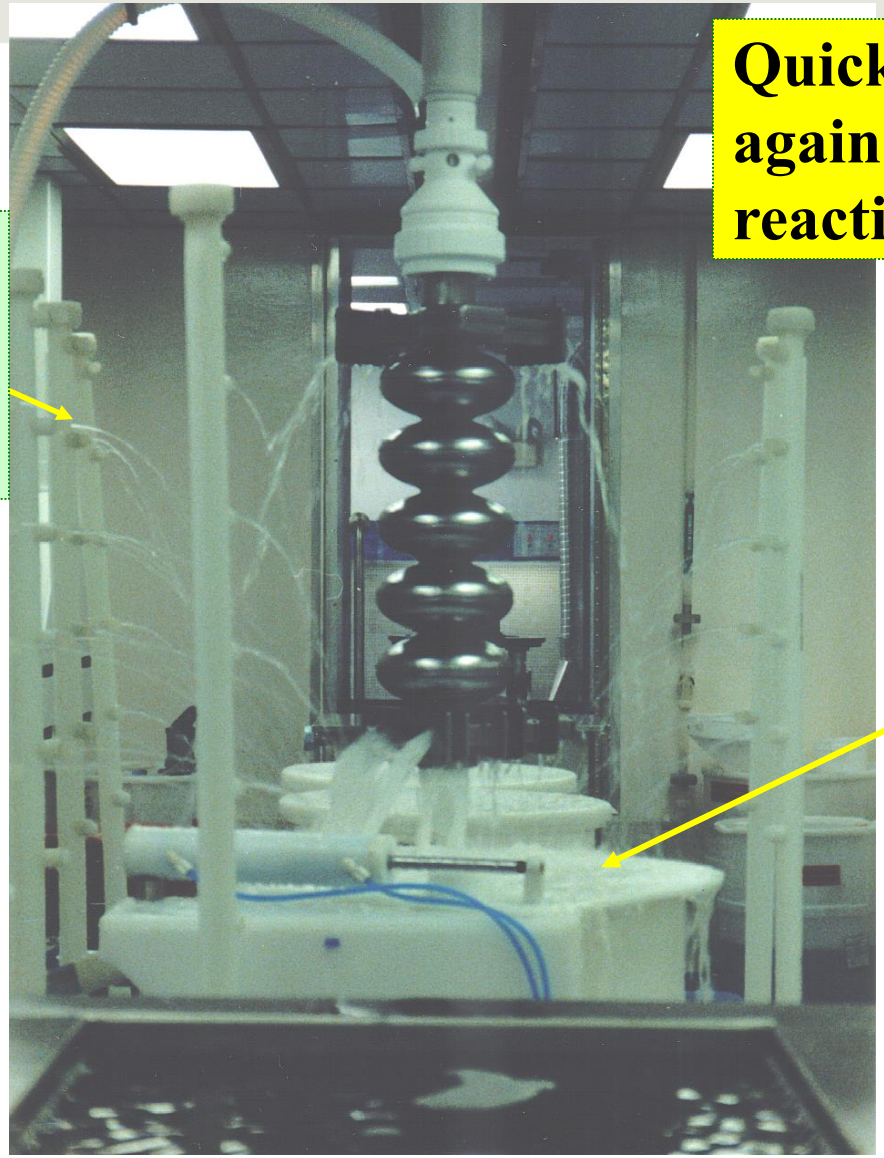
CEBAF BCP & Rinsing

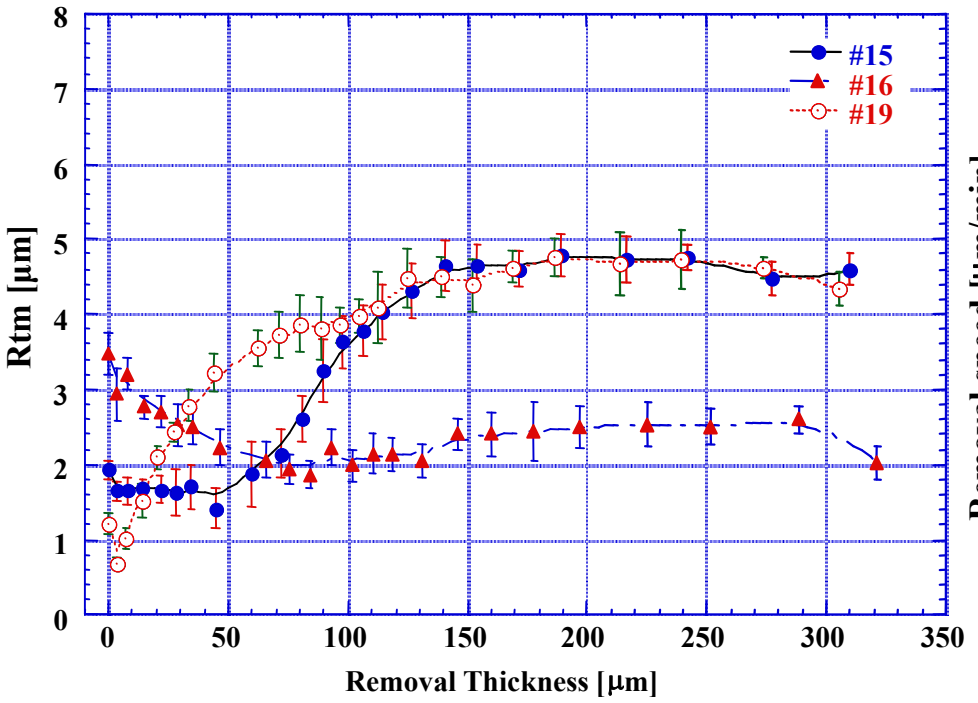
Shower for Rinsing the outer cavity surface

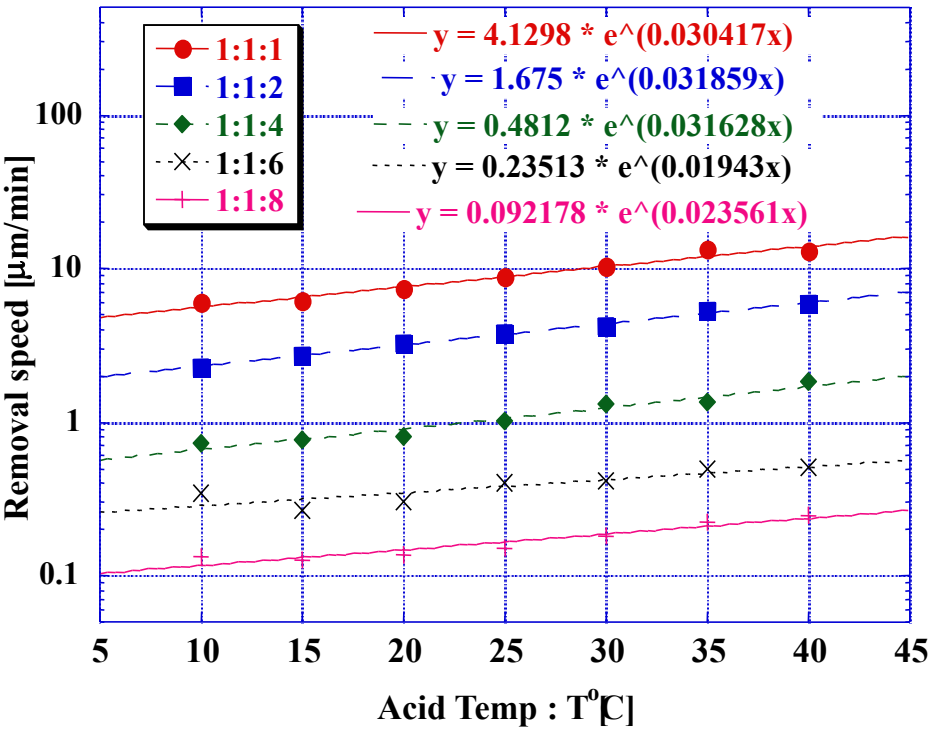


Quick rinsing against the runaway reaction

CP acid tank
Make BCP by immersing cavity in the BCP acid.
Inner and outer both surfaces are polished

Feature of BCP



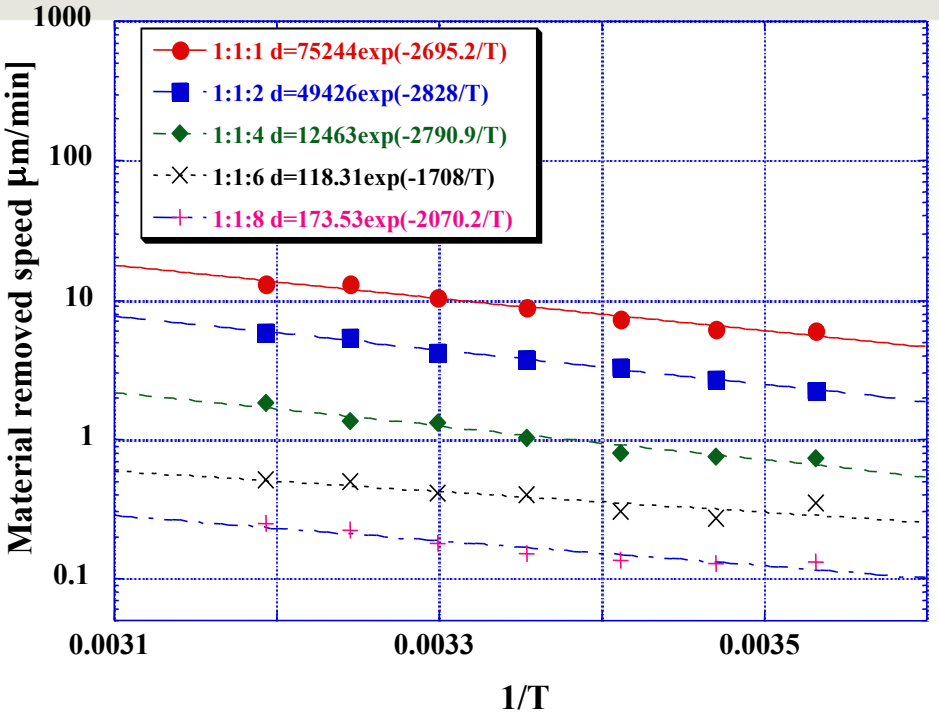


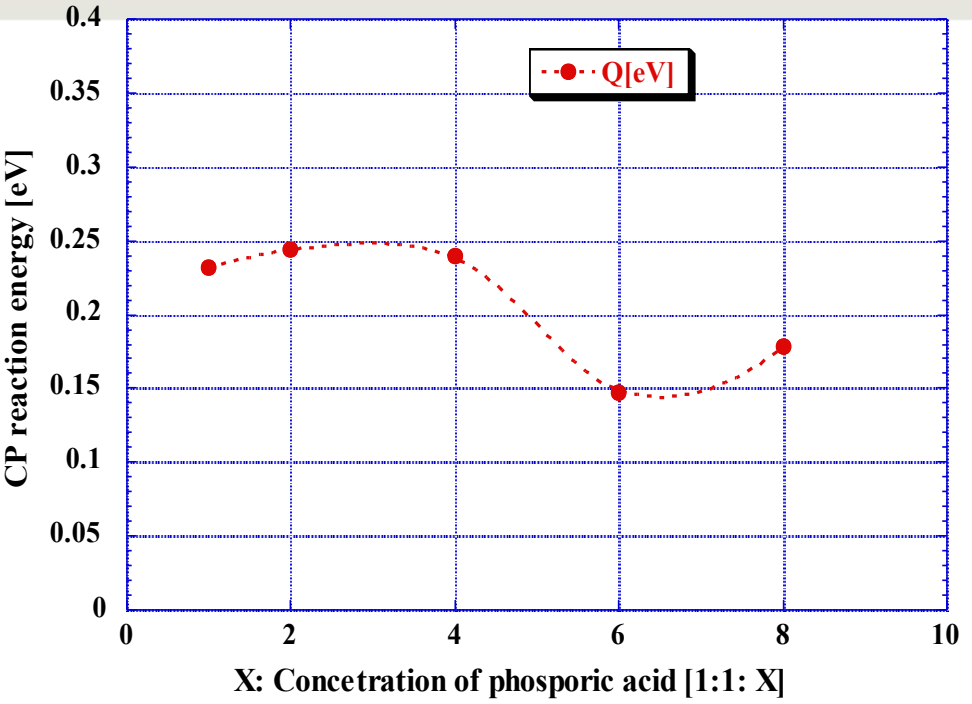
Typical surface roughness = 2 ~ 5 μm after 100μm CP,
Material removal speed ~ 4 μm/min at the room temperature with BCP acid 1:1:2 at 25°C

CP is faster in material removing than EP (0.4μm/min).

Finished surface roughness depends on grains of the Nb surface.

Reaction Energy of BCP (1:1:1)

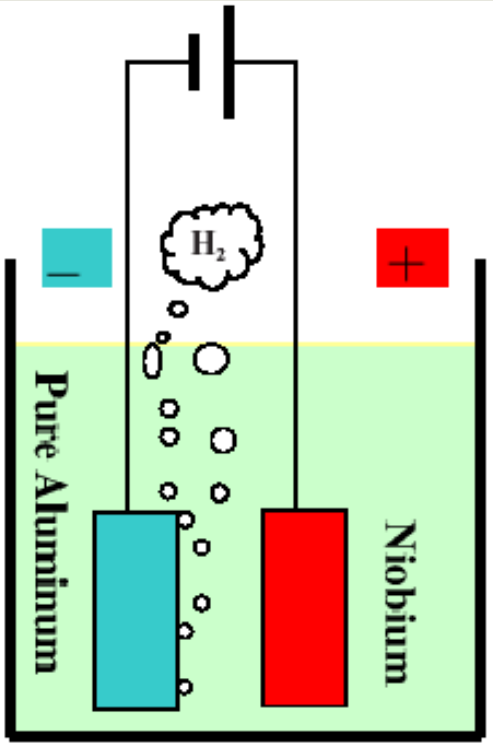




$$d(T) = d_o \cdot \exp(-\frac{Q_{CP}}{k_B T})$$: E reaction energy of CP $Q_{CP} \sim 0.232$eV @ 1:1:1

Q_{CP} is an important information for hydrogen Q-disease.

16.4 Electropolishing (EP)

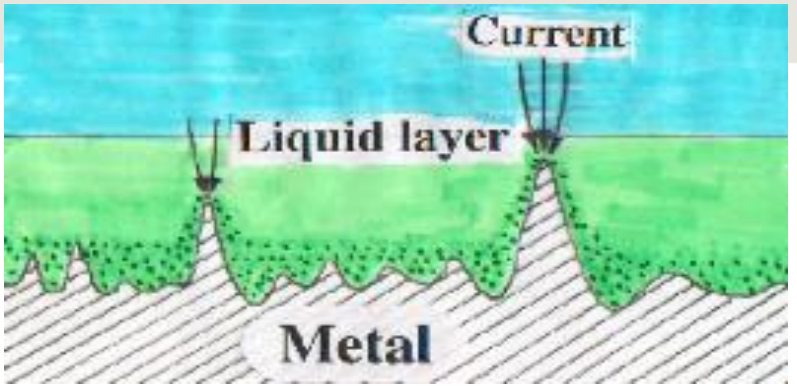


Acid:
H_2SO_4 (>93%): HF(46%)=10:1 V/V

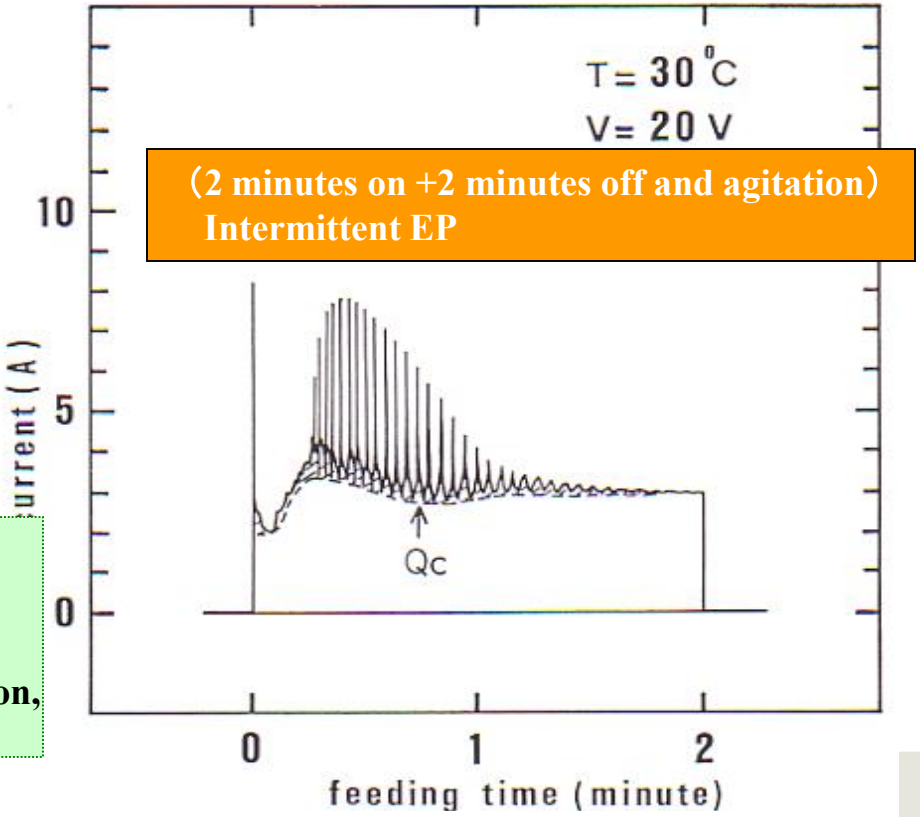
Chemical reaction:

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

Sulfuric acid has no contribution to the chemical reaction, Only to increase viscosity in the EP acid.

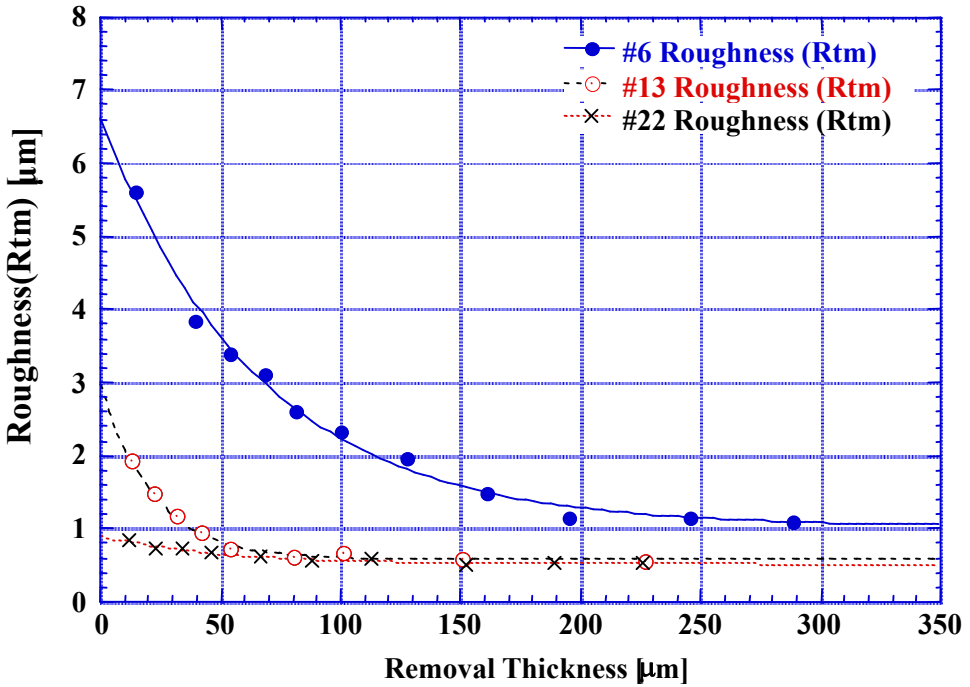


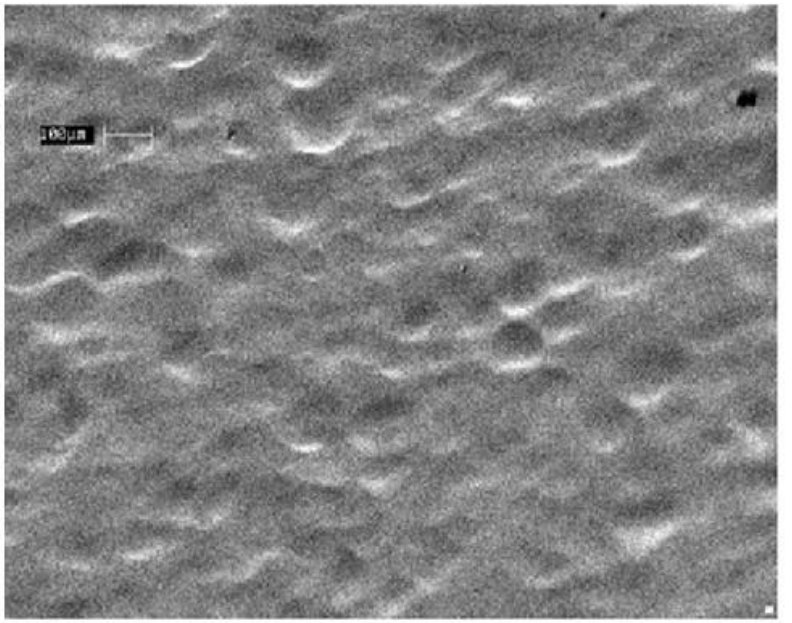
Principle of leveling of EP



Example of EP current

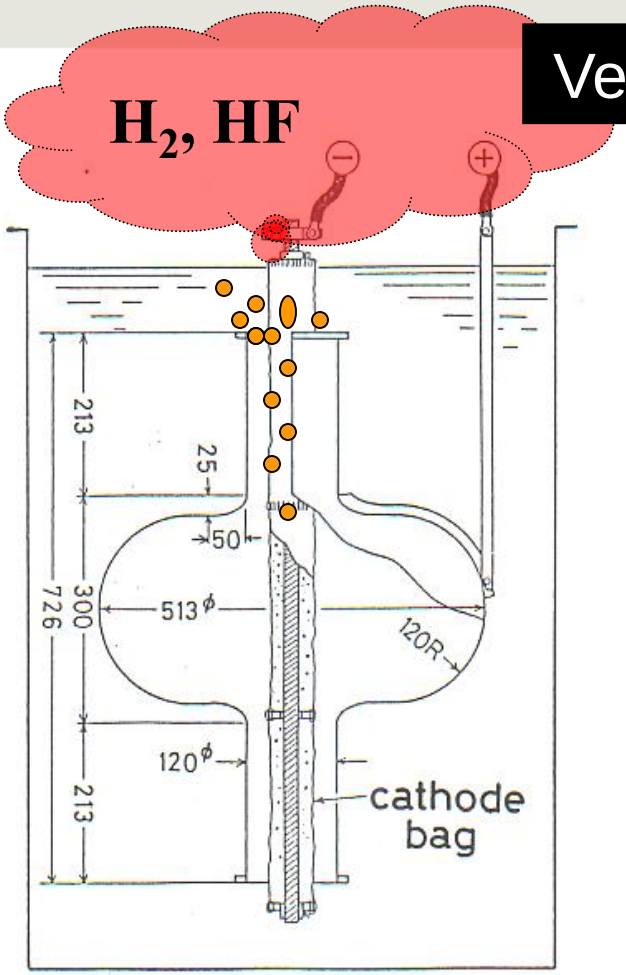
EP Finished Surface Roughness





1) EP finishing surface depends on the initial surface roughness
2) Finishing surface become smoother exponentially with increased removed thickness
3) EP has no preferred grain etching.
4) Finished surface is very smooth
5) Control of surface finishing is easy

Early KEK EP Method



Very hazardous working environment

Light hydrogen Q-disease

Medium hydrogen Q-disease

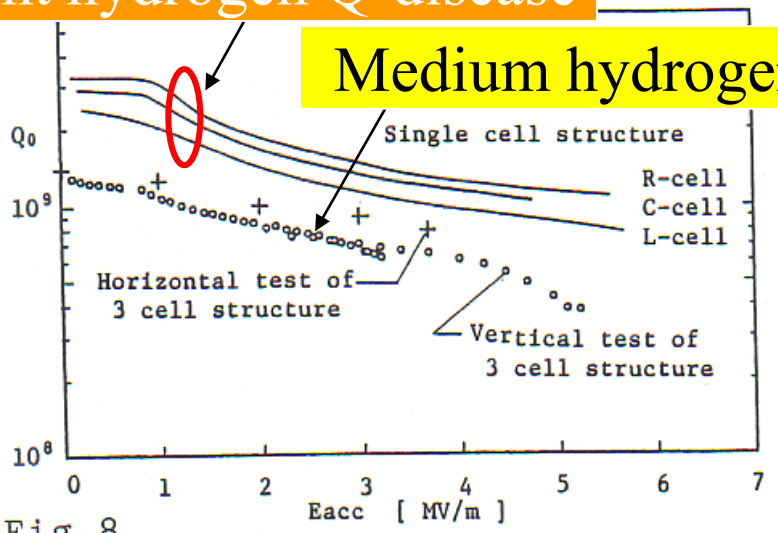


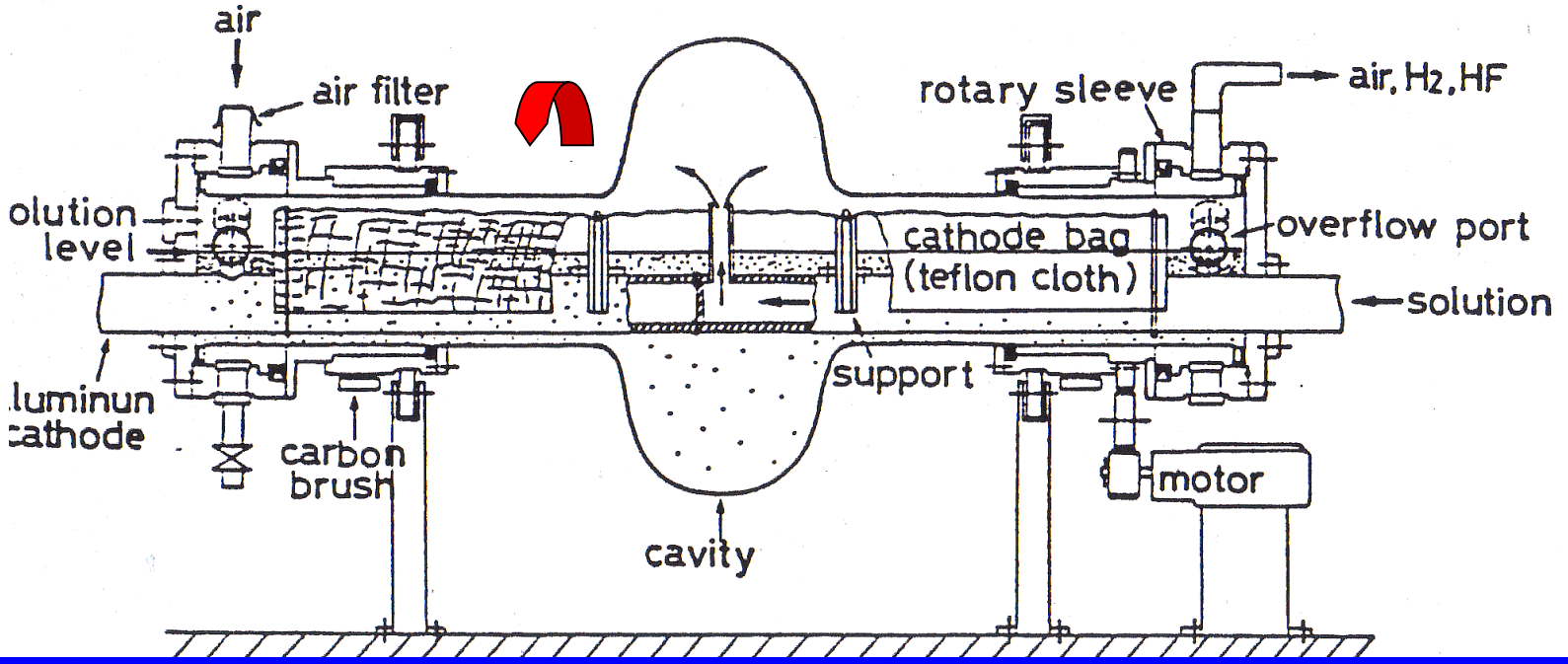
Fig.8
Qo-Eacc curves of a three-cell cavity electropolished using the vertical EP method. R,C,L-cell are single cell cavities before completing the three cell cavity.

Issue in the early EP method

- Low Q due to hydrogen disease
- Hazardous working environment

Development of Horizontally Rotated Continuous EP

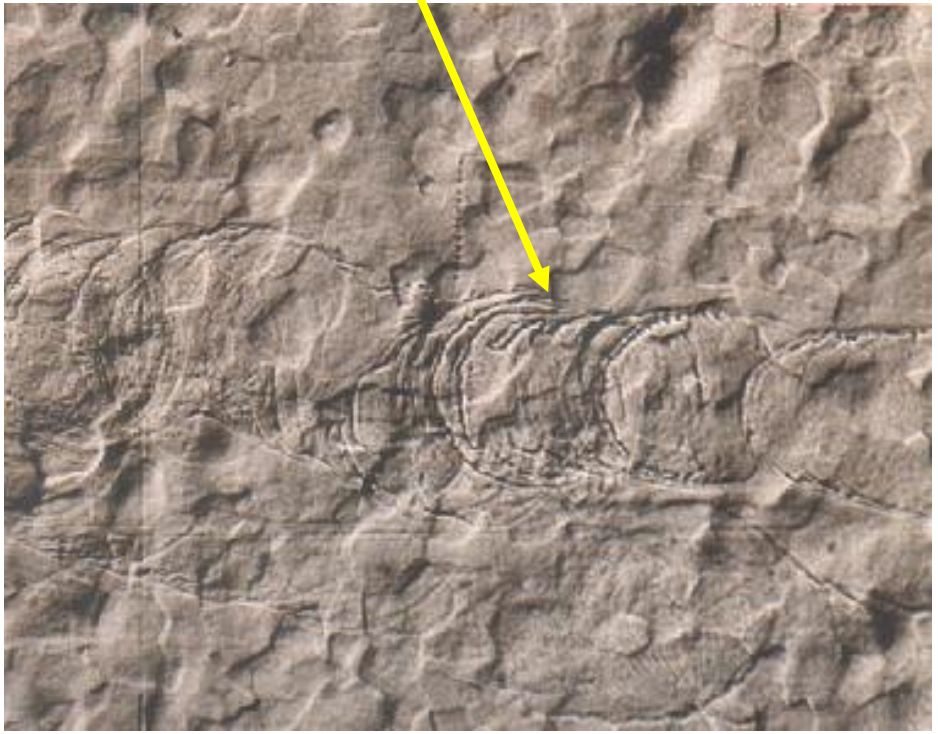
Continuously slowly rotated

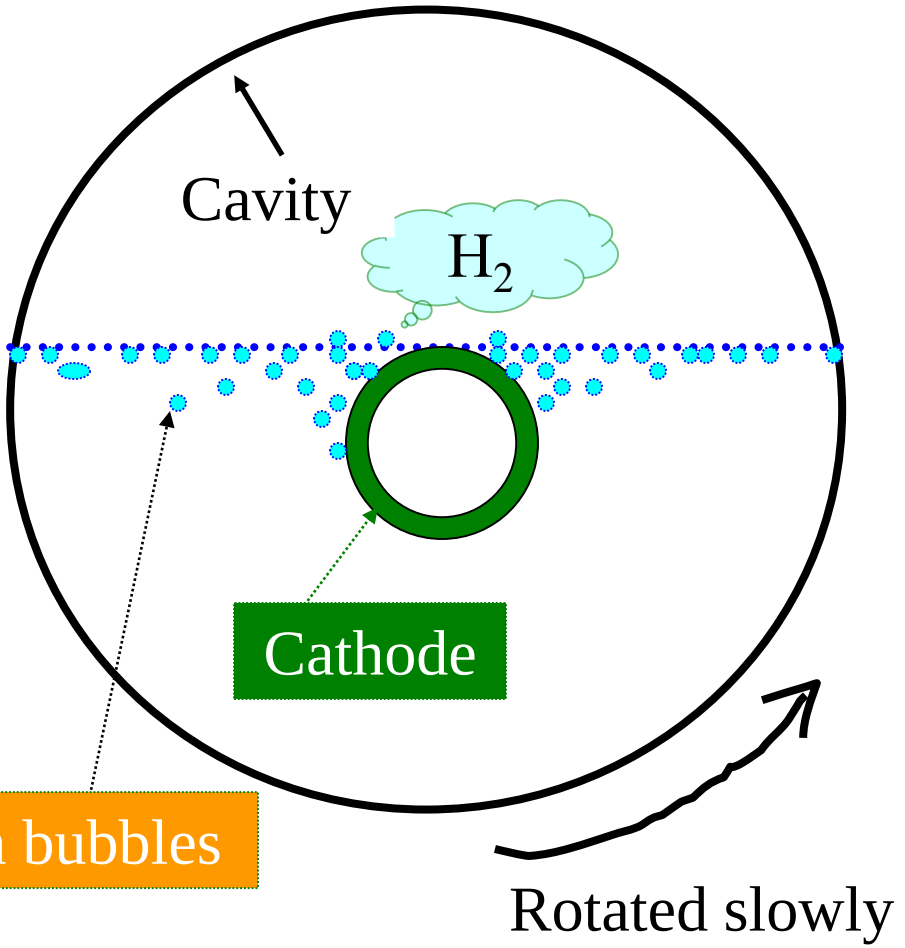


- EP acid is filled just above half level and recirculated through the overflow ports. EP acid is recirculated in the closed system
- Cavity is continuously slowly rotated during EP as to give an agitation on EP surface
- EP voltage is applied continuously (Continuous EP)
- Hydrogen gas is exhausted through beam pipe

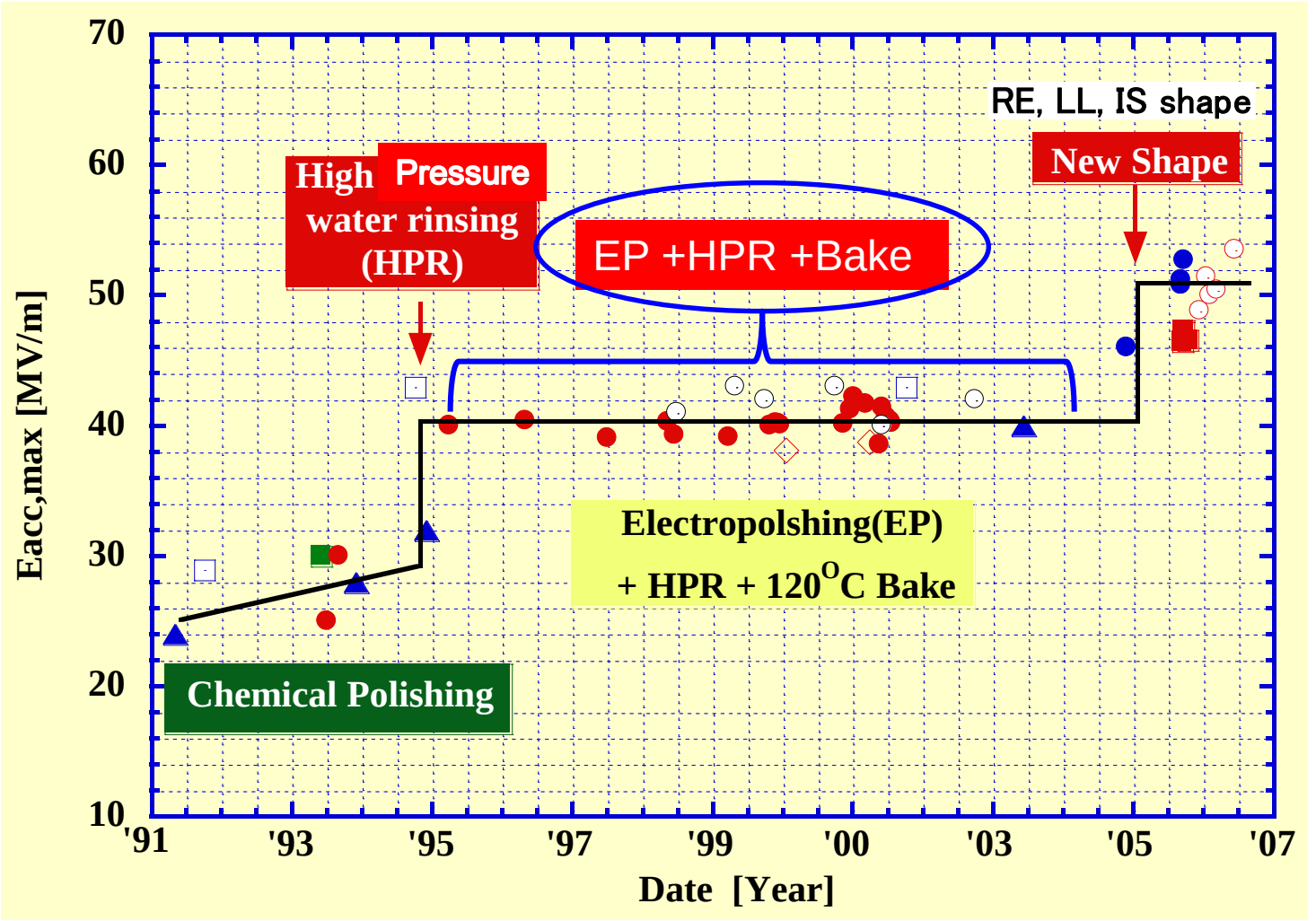
Cathode Bag Necessary

Hydrogen bubble trace on Nb surface





Ten Years Later, EP Made the Breakthrough for 40MV/m

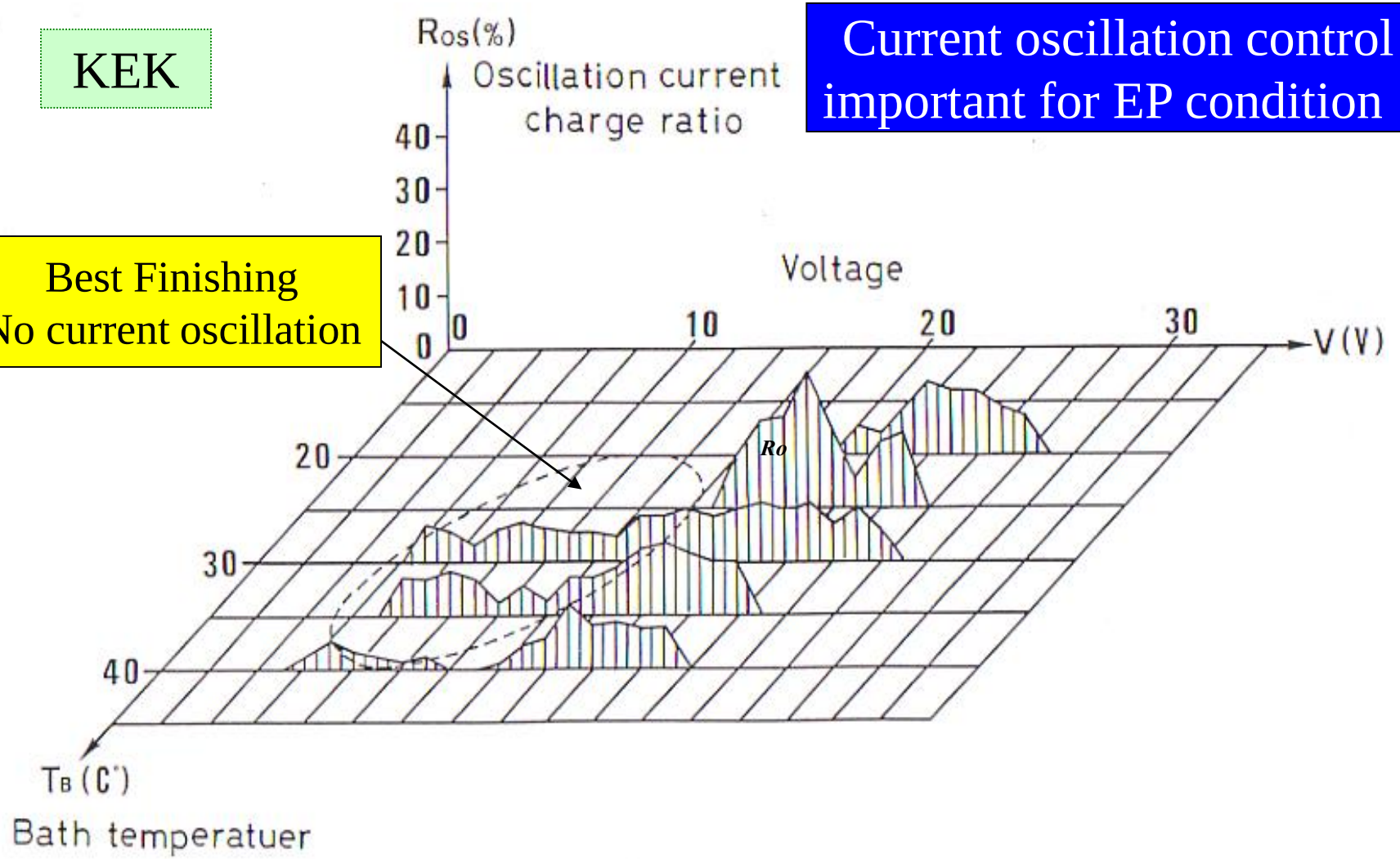


Reconsideration of the Current Oscillation

KEK

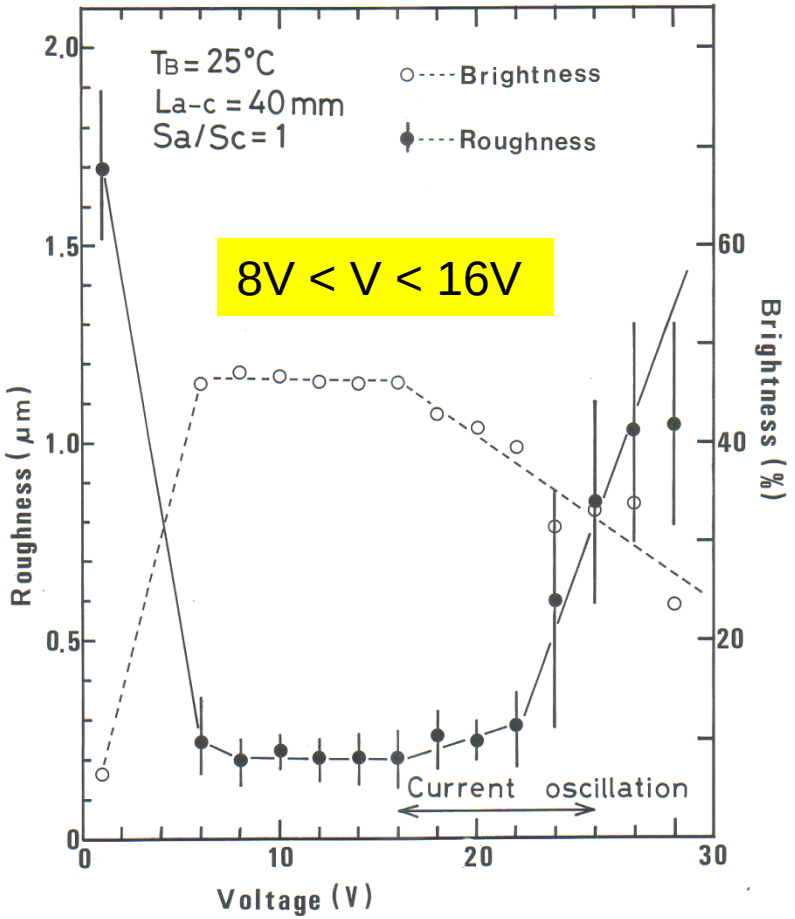
Current oscillation control is not important for EP condition

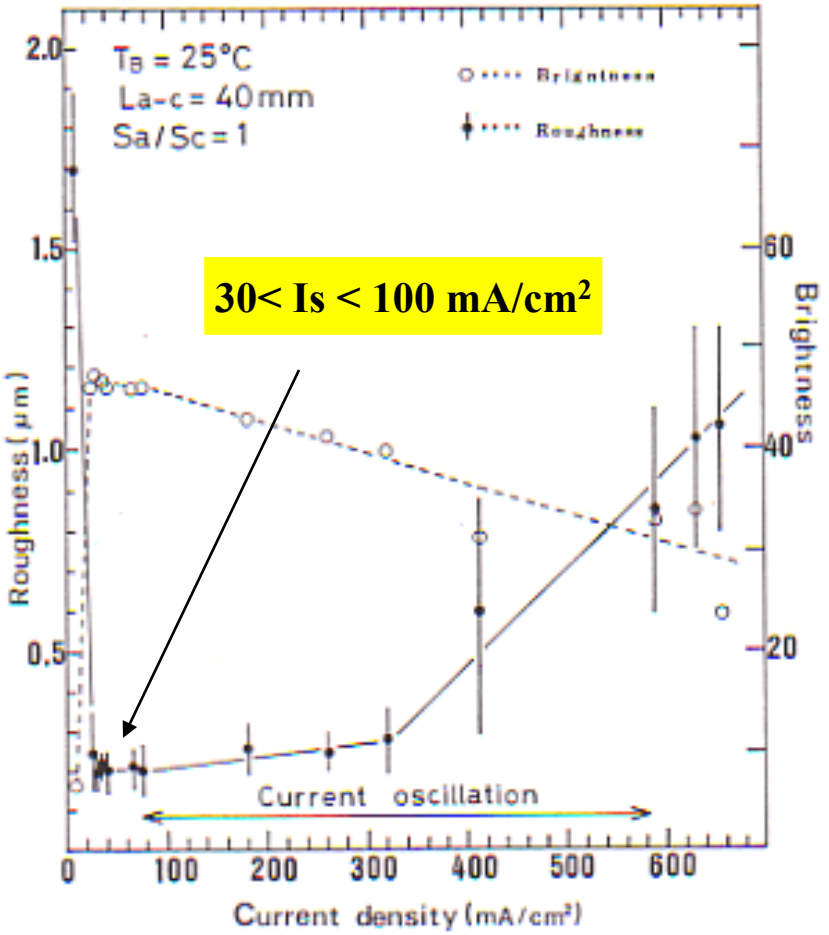
Best Finishing
No current oscillation



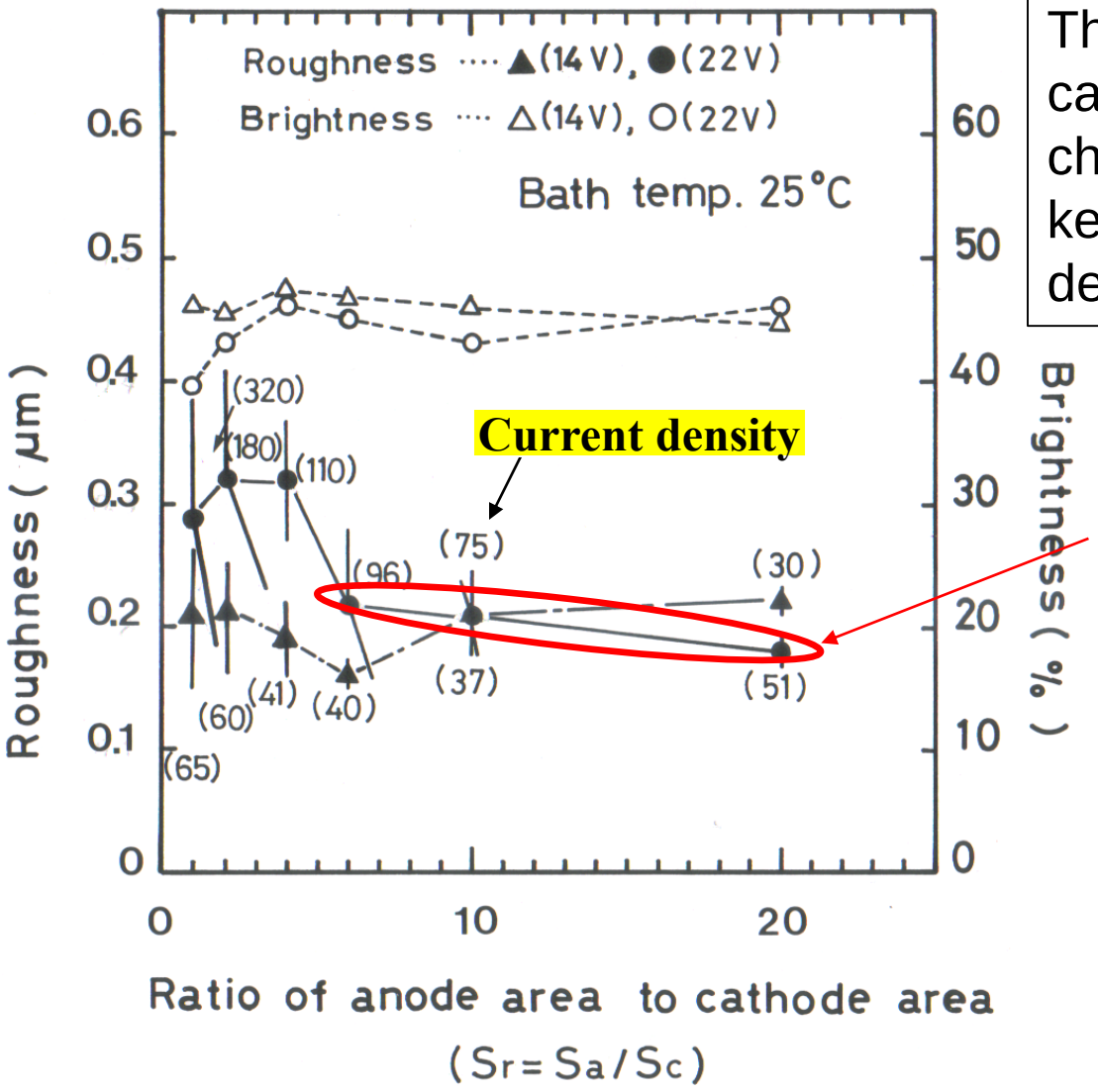
What is the Critical EP Parameter ?

Voltage or Current density ? This is a coupled problem.





Separation of I and V Effects



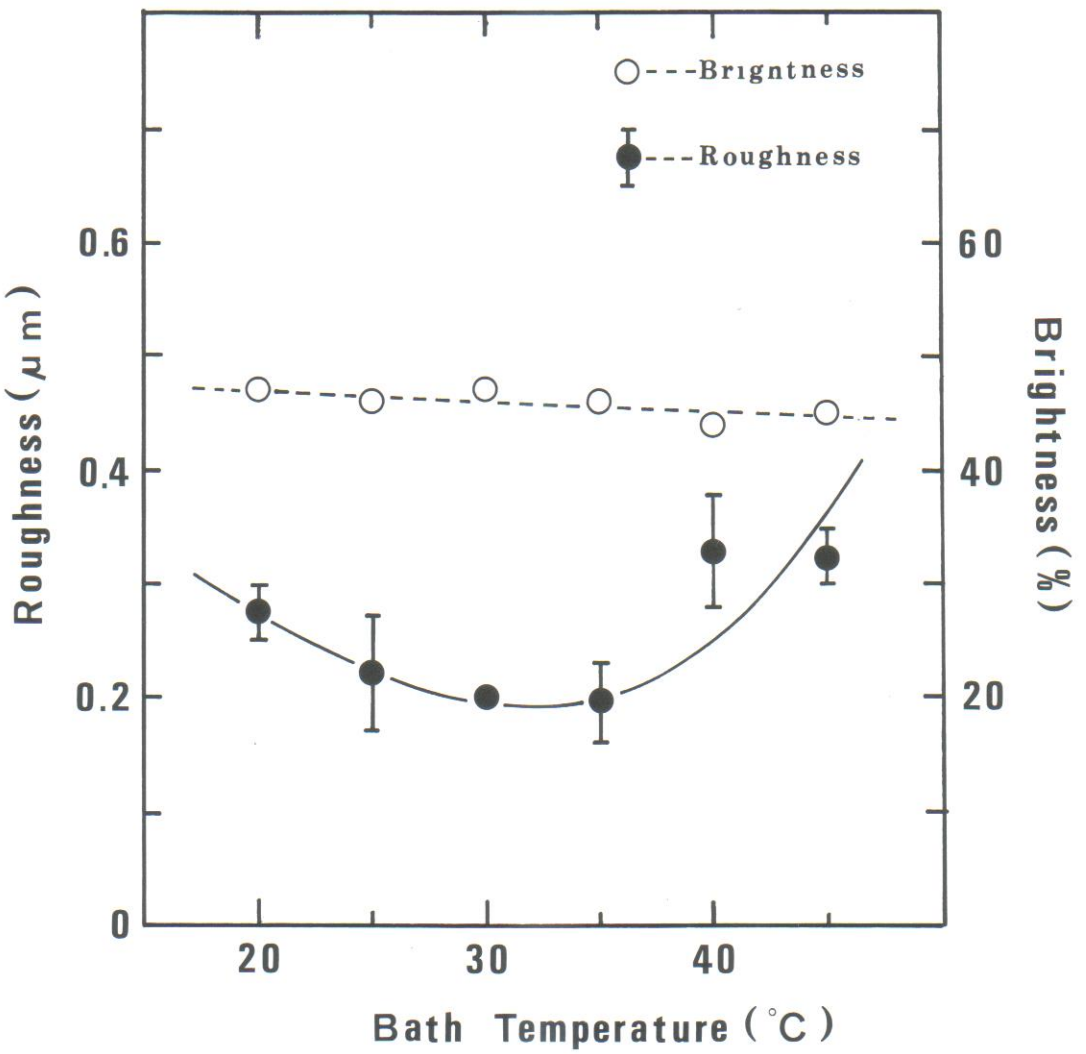
The surface ratio between anode and cathode (Sr) impacts on the I-V characters of EP, for instance even kept the same voltage the EP current density reduces if Sr made smaller.

Even 22V, smooth surface can be obtained. if I_s in $30 < I_s < 100 mA/cm^2$.

Current density is the crucial condition.

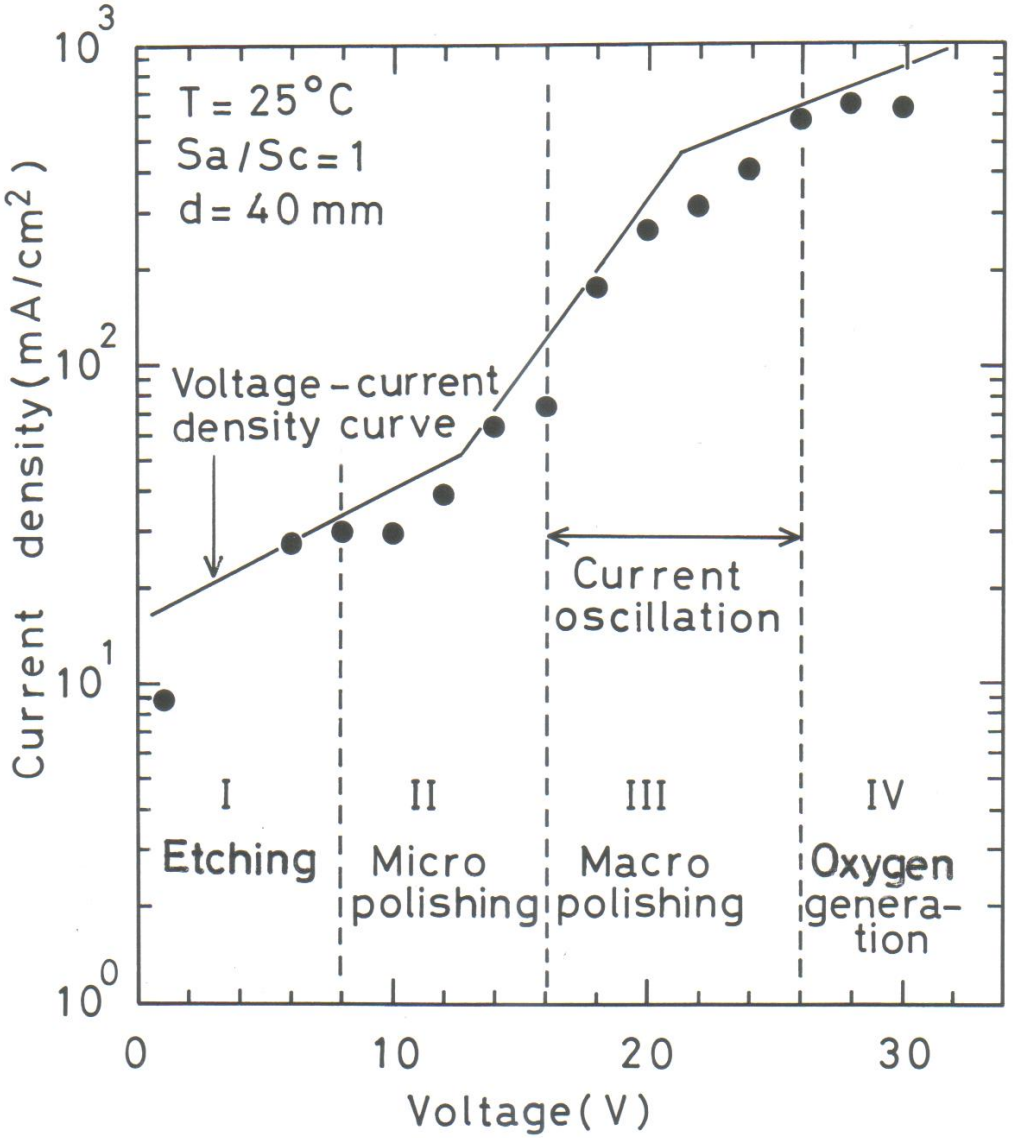
$30 < Is < 100\ mA/cm^2$

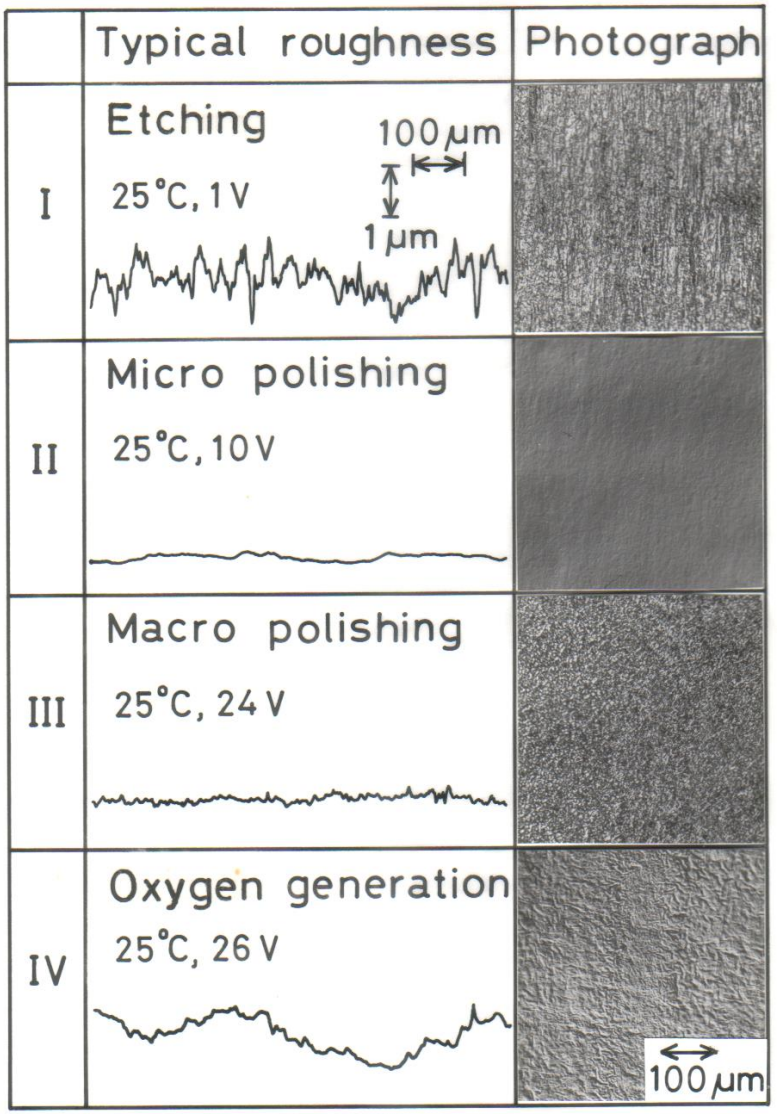
Temperature Effect on EP Finishing



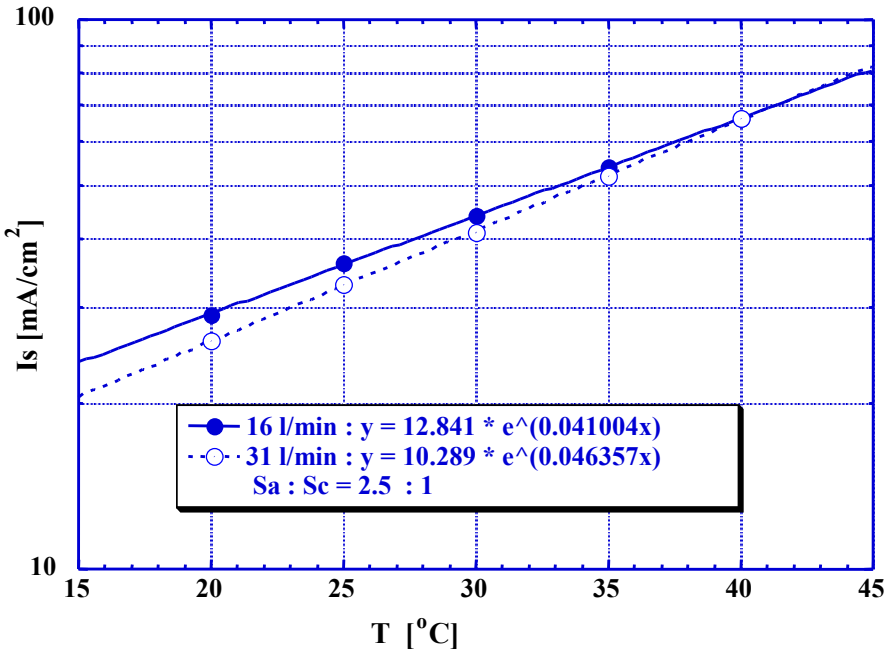
EP acid temperature
$25 < T < 35\ ^{O}C$
produces good finishing.

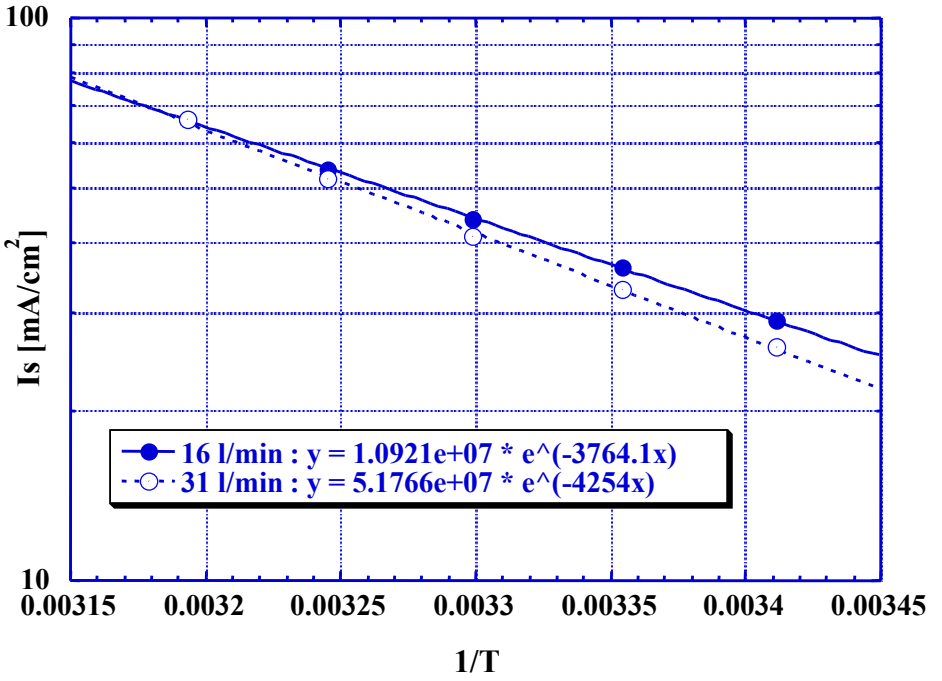
Electrolishing Characteristics with Nb





EP Reaction Energy





Material removal speed in EP

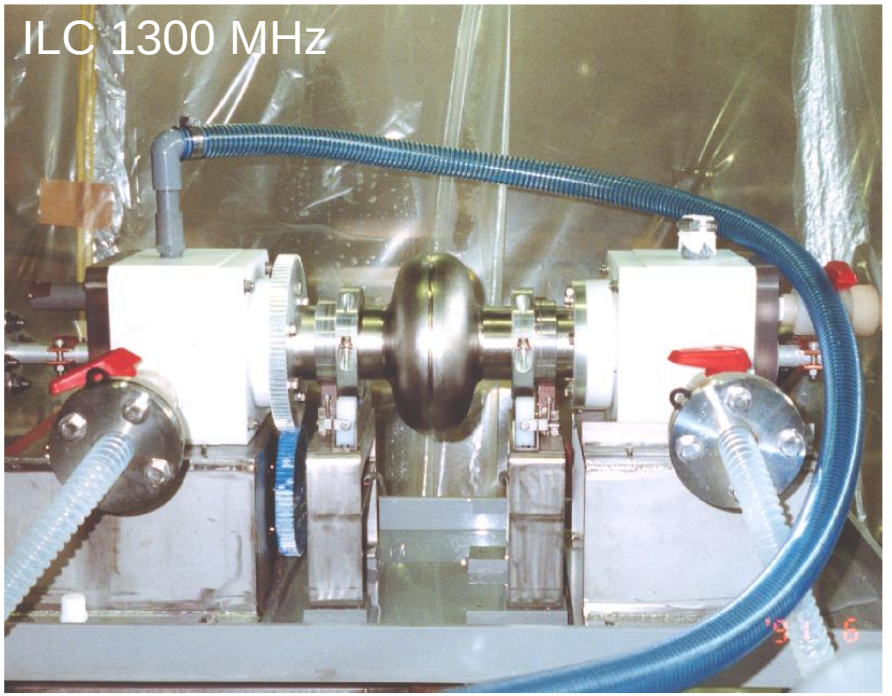
$$I_s(T) = I_0 \exp(-\frac{Q_{EP}}{k_B T})$$

EP reaction energy : Q_{EP} = 0.325 ~ 0.367 eV

A little larger compared to BCP (0.232eV)

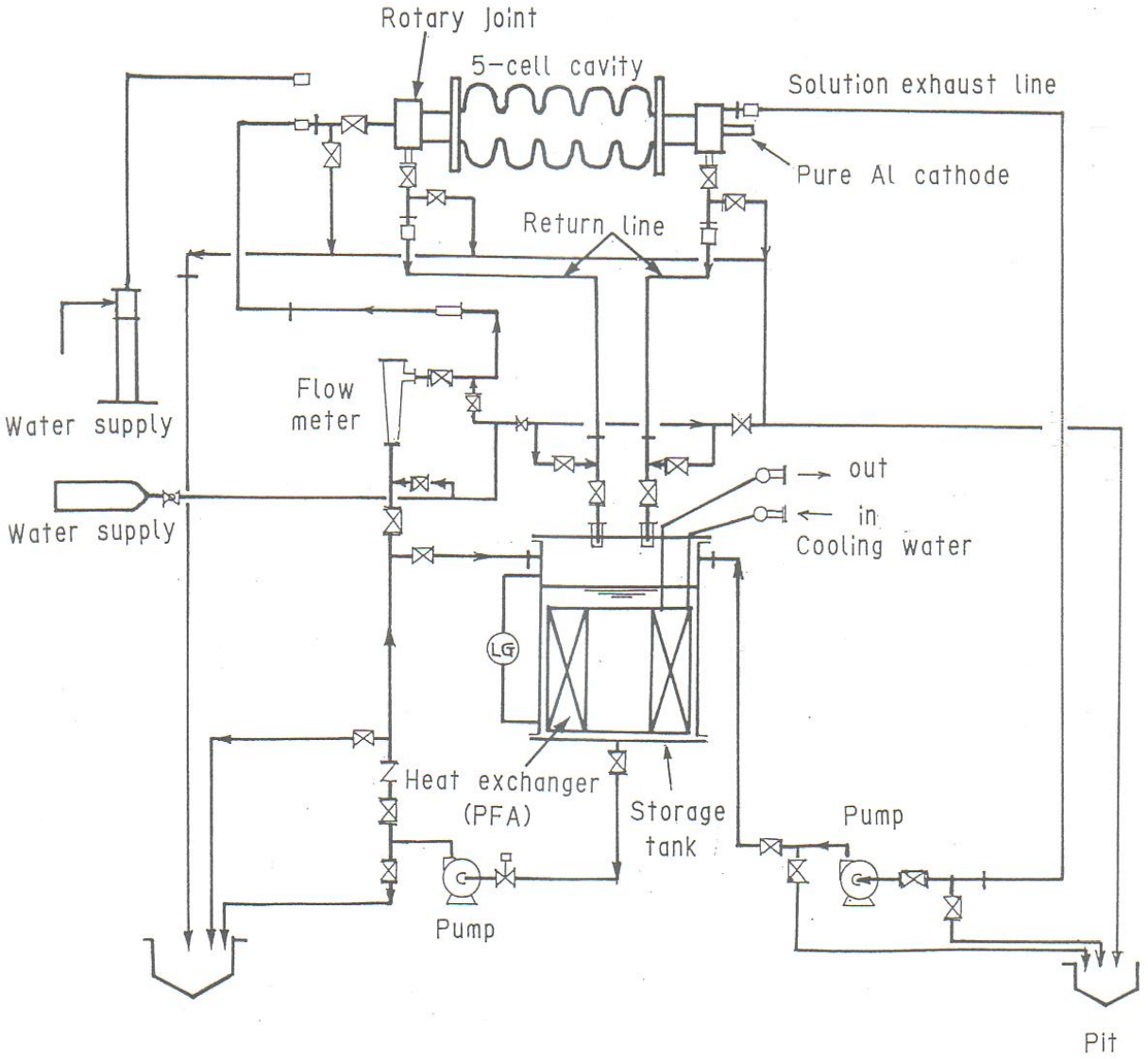
Horizontally Rotated Continuous EP (HEP) Developed in KEK (1985)

- HEP system was developed in KEK for TRISTAN 508MHz 5-cell cavity preparation in 1985 under the collaboration with Nomura Plating Co. Ltd.
- This method produced excellent cavity performance.
- Later, this method was transferred DESY, Jlab, FNAL in 2000's



Collaboration with Nomura Plating Co. Ltd.

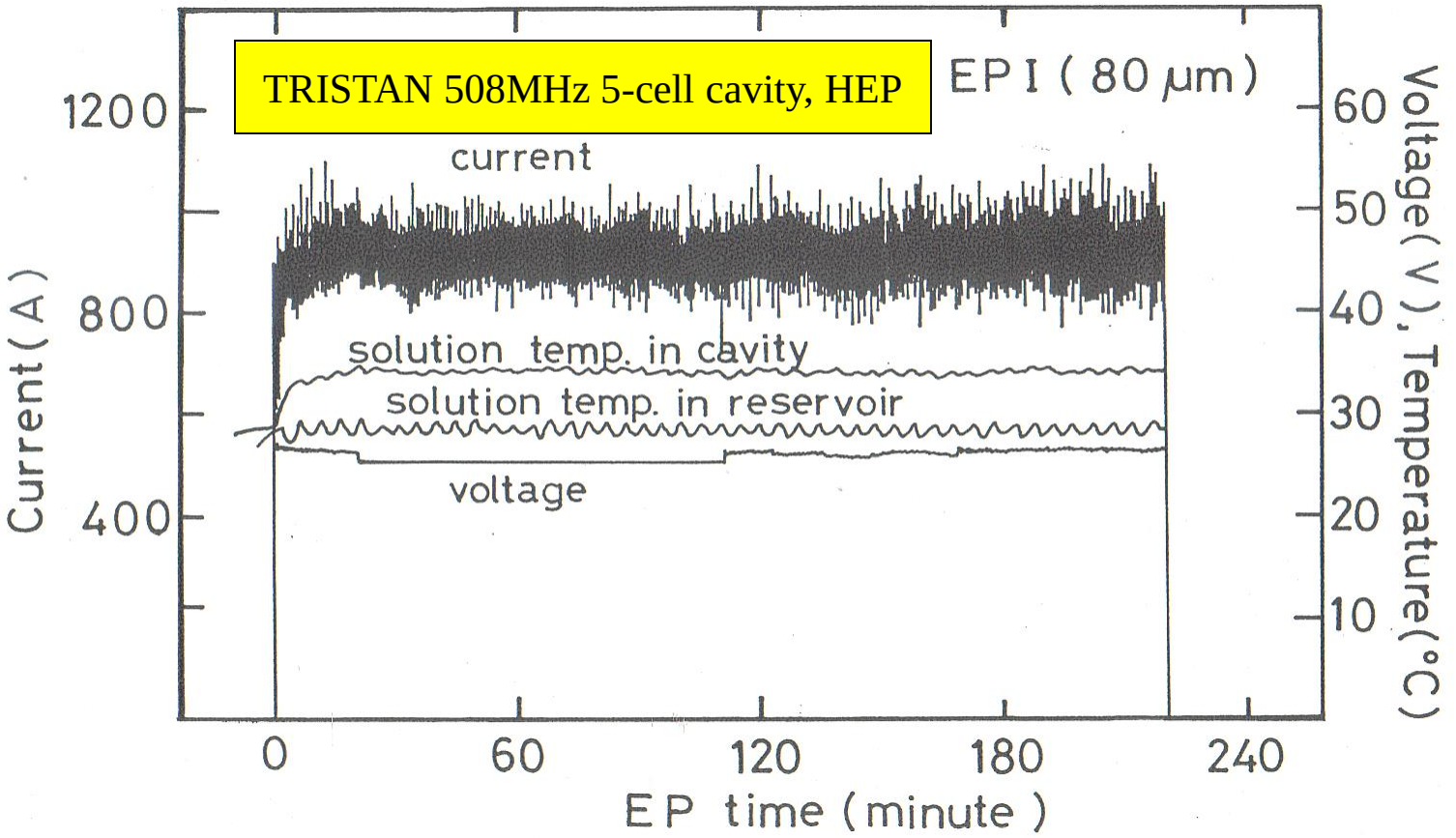
EP System Flow for Production



EP Control Data Example

Very easy control and very safe

- Continuous operation
- Closed EP acid circulation
- Hazardous gases during EP (HF, H2, Sox all exhausted through the pipe and neutralized)



Extraction of Cathode after HEP

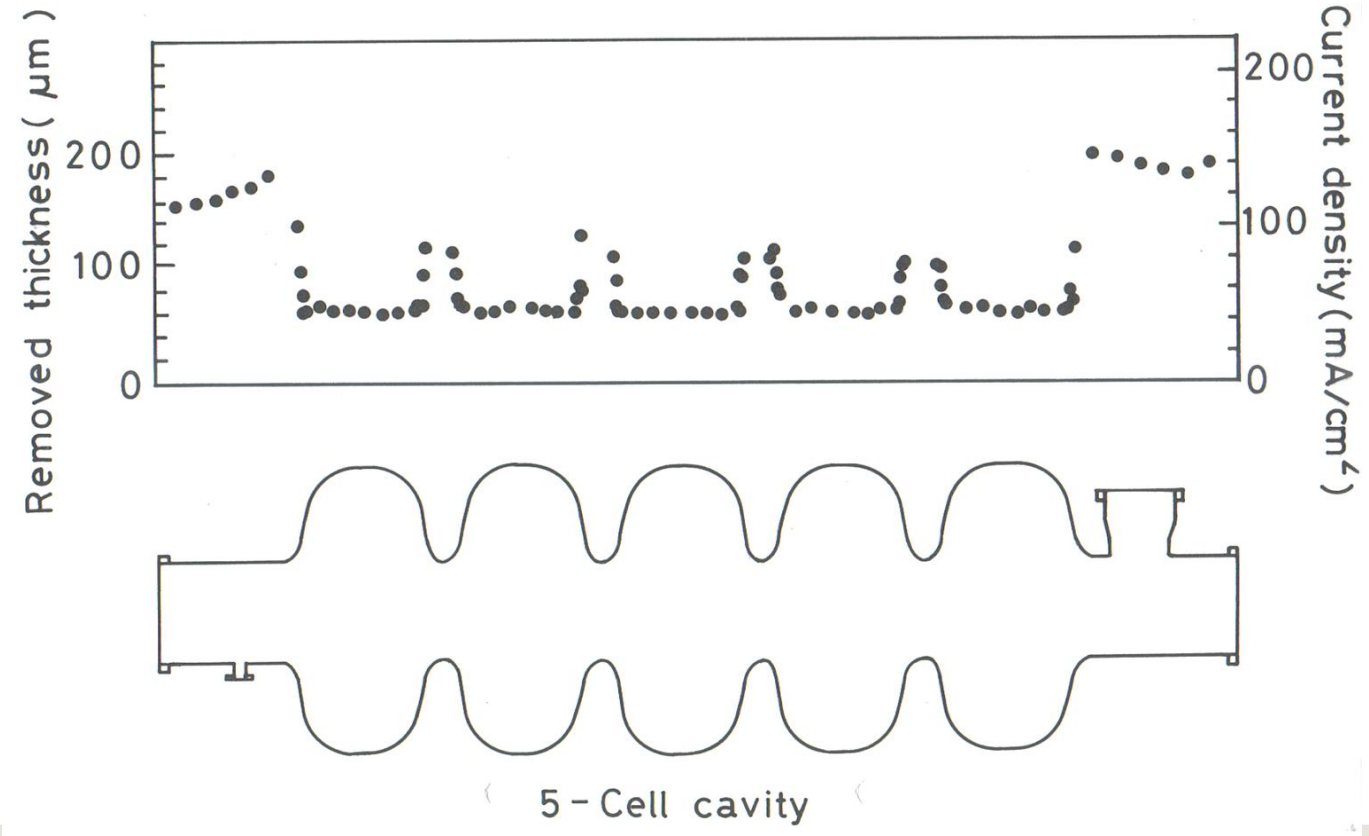
After HEP:

- Cavity is turned vertical position
- Extracted EP acid from the cavity
- First DI water rising putting water into the cavity
- Extraction of cathode from the cavity
- Move to medium pressure water rinsing
- Hot water rinsing with ultrapure water
- Disassembled HEP fixture

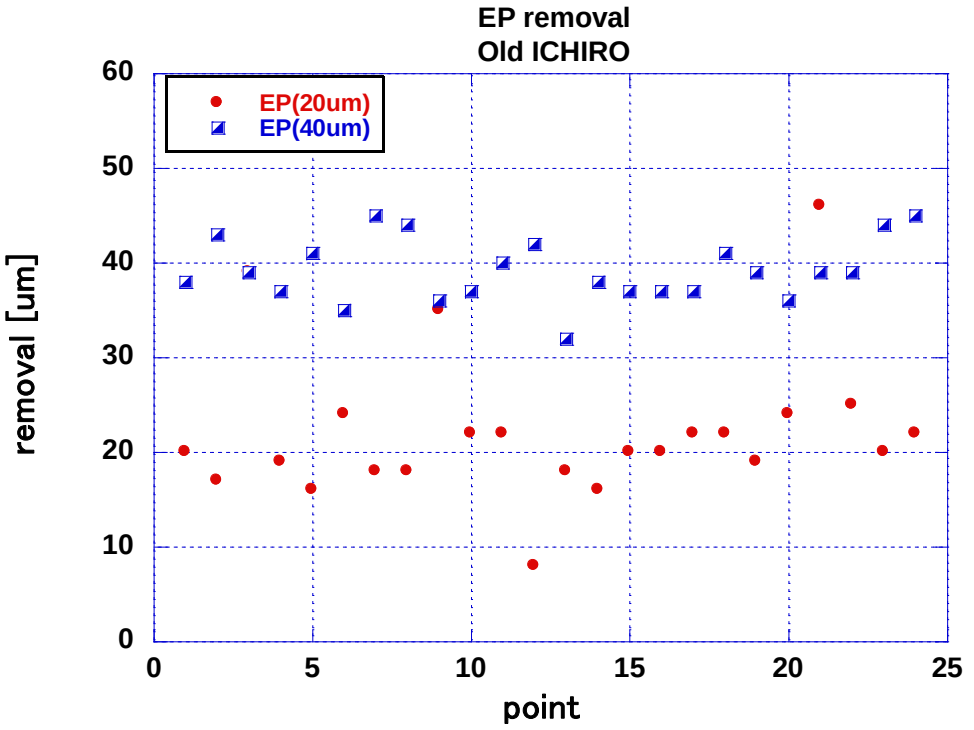
Removal Distribution with TRISTAN 508MHz 5-Cell Cavity HEP

Goal: 80 μm remove in average

- Uniformly removed in each cell as expected.
- Irises area is removed doubly than equator section.
- Beam area is removed three times larger than equator section.



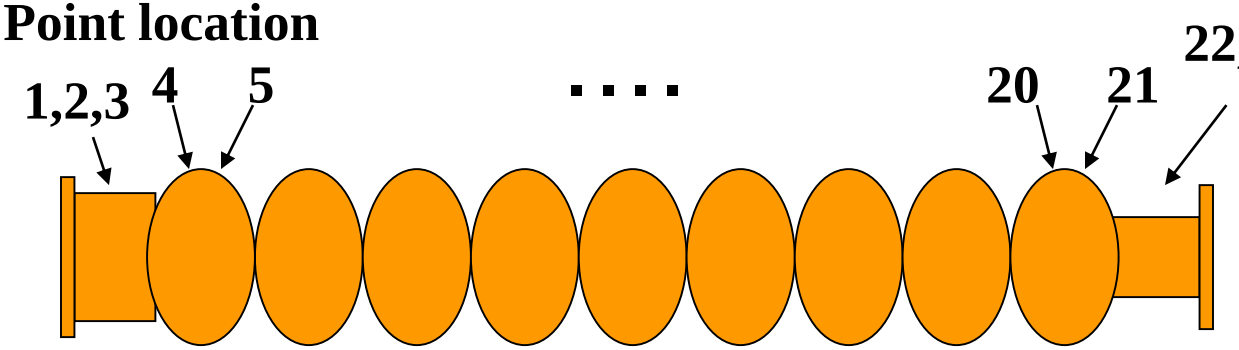
EP Material Removal and Frequency Shift ILC Ichiro cavity



- Material removal is more uniform for ILC cavity because of shorter distance between cathode and anode
- The EP removal set is close to the measured removal averaged.
- Frequency shift is ~ -10kHz/mm for ILC cavity

Cavity	df [kHz/μm]	N
Old I9 w/ large BP	10±2	2
Single	10±5	44
Old I9 #0, #1	8±6	6

Point location



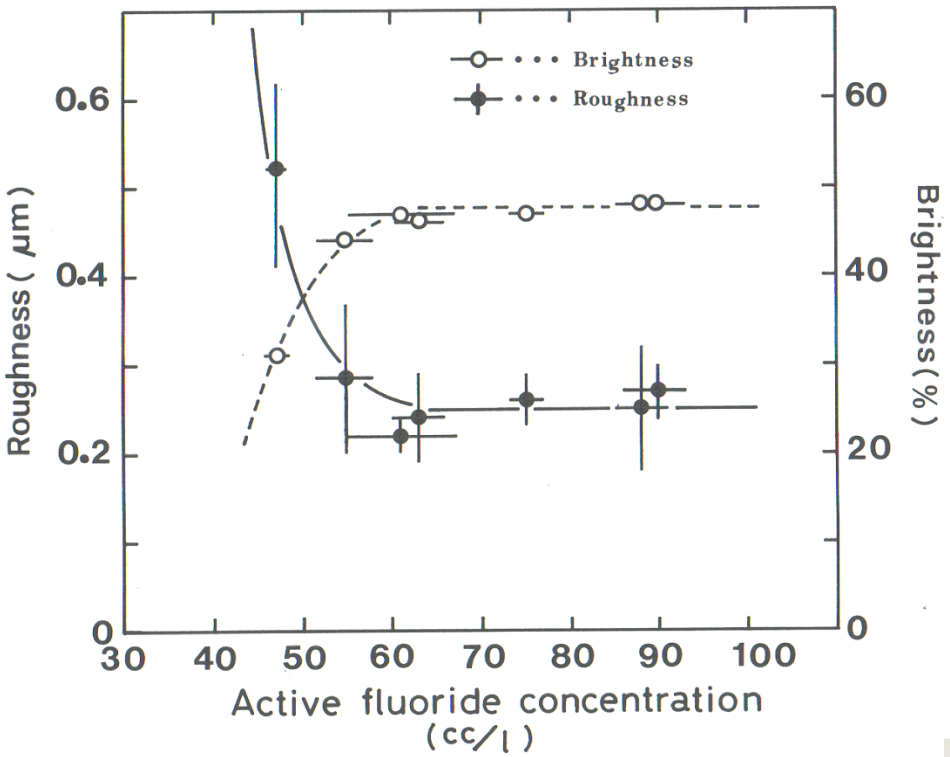
EP set value	Removal meas.
40 μm	39±3 μm
20 μm	22±5 μm

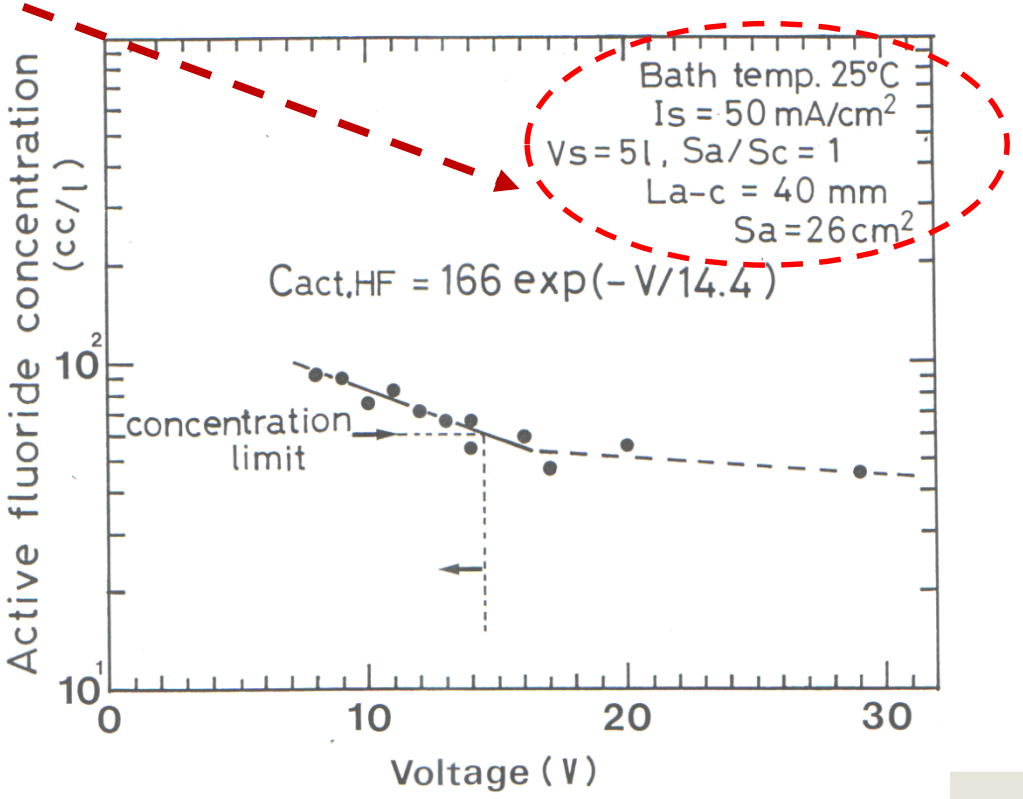
EP Aid Quality Control (QC)

HF concentration becomes too low < 55 cc/L (converted to 40% concentered HF acid) , EP finishing is rough.

The HF concentration is easily measured by EP voltage.

- Sample 5 L from EP acid reservoir tank.
- Take EP under the same EP electrode configuration.
- If EP voltage is higher than 14.4 V to have EP current 50 mA/cm^2, it concluds the acid aged.

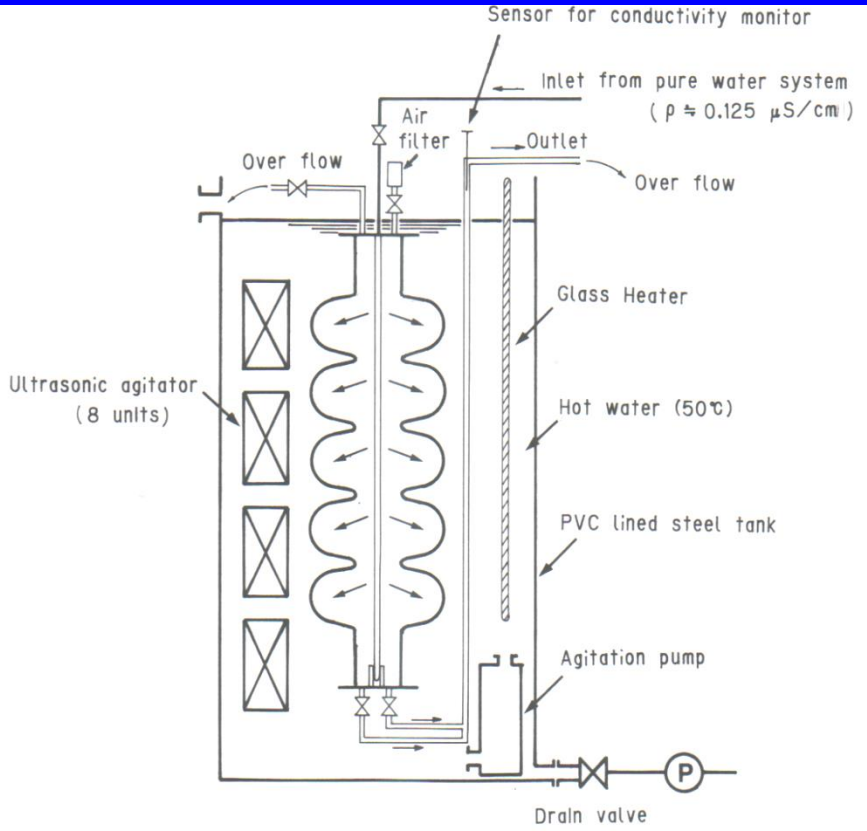


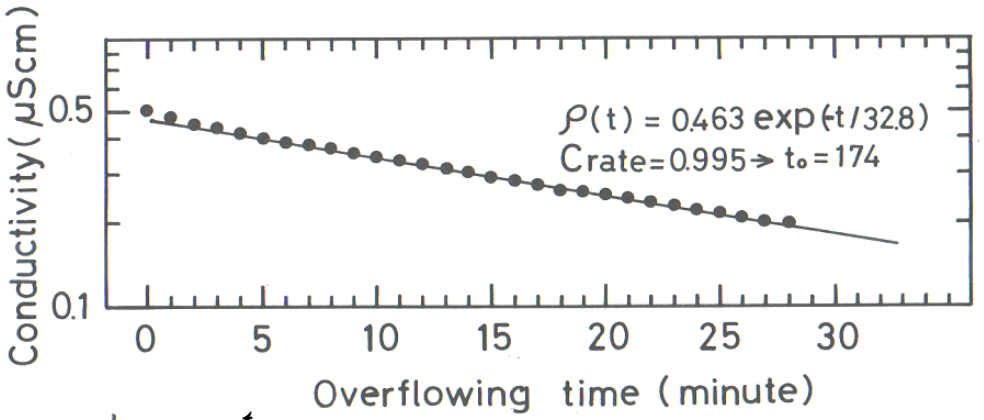


Rinsing QC

Rinsing QA is done by measuring the electric conductivity of waste water from the cavity.

- Electric conductivity decreases exponentially with increase rinsing time
- The decay time τ provides the necessary rinsing time.
  For instance, set rinsing rate 99.9% against chemical residues, the necessary rinsing time (t_0) is 174 mi $t_0 = -\tau \times log_e(1 - 0.995), \tau = 32.8\,min$





$$C(t) = C_0 \cdot e^{-\frac{t}{\tau}}$$

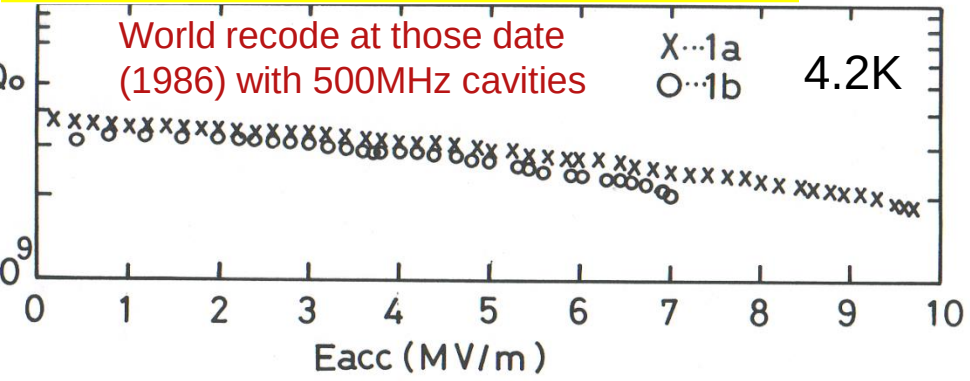
$$\text{Rinsing rate} = \frac{\int_0^{t_o} C_0 e^{-\frac{t}{\tau}} dt}{\int_0^{\infty} C_0 e^{-\frac{t}{\tau}} dt} = 1 - e^{-\frac{t_o}{\tau}}$$

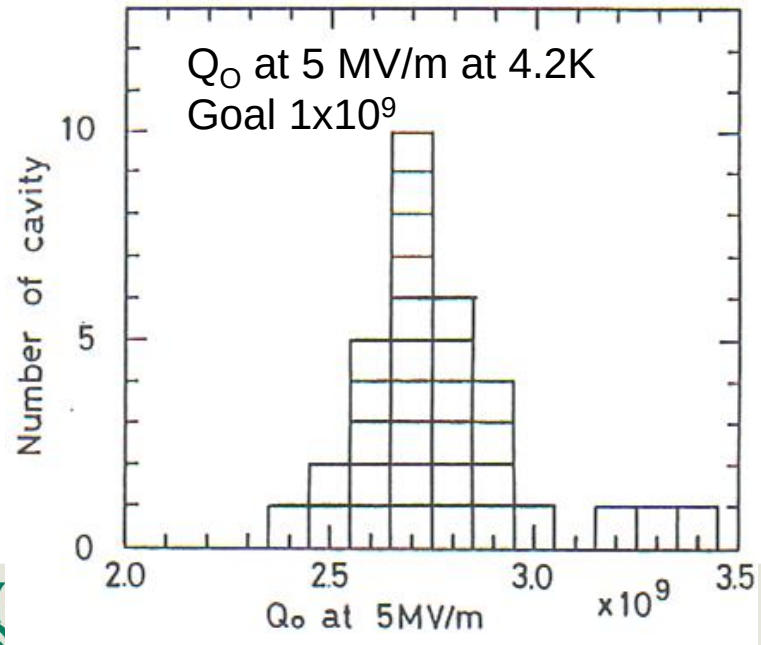
TRISTAN SRF Cavity Performance

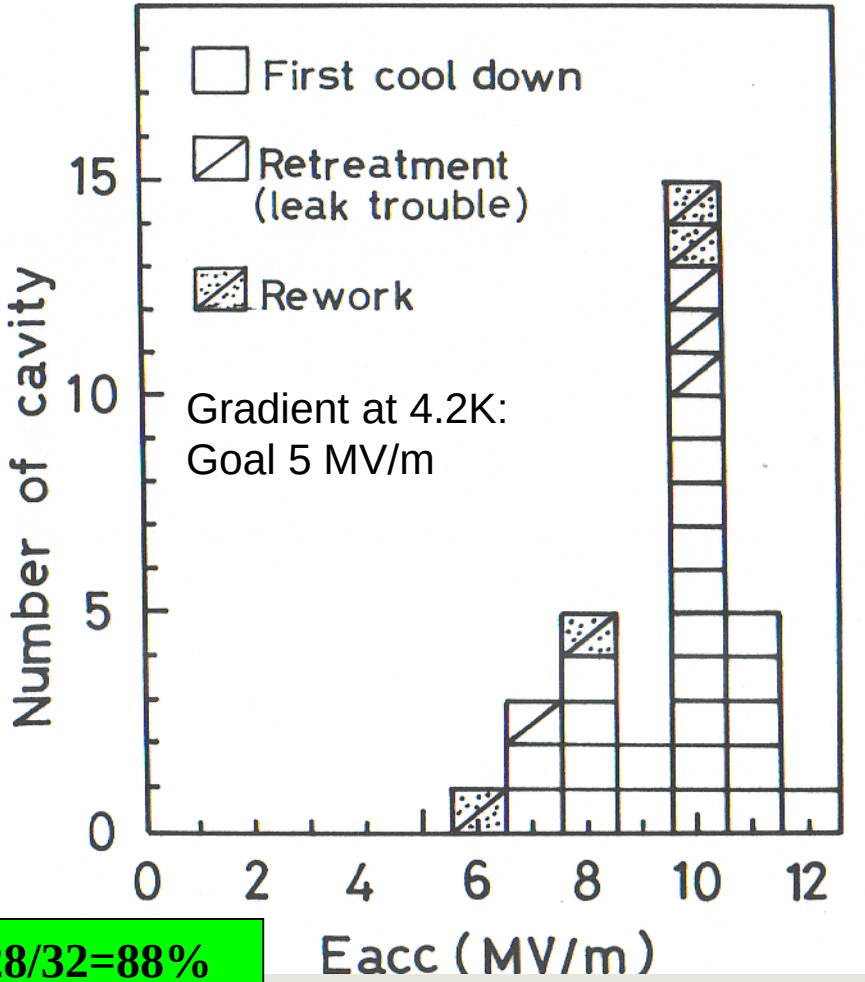
TRISTAN SRF Goal
Q_O > 1x10^9 at 5MV/m @ 4.2K
Eacc = 5MV/m

Cavity performance statistics:
32 TRISTAN cavities.
Highest performance in the world at that time

TRISTAN production cavities #1 and #2





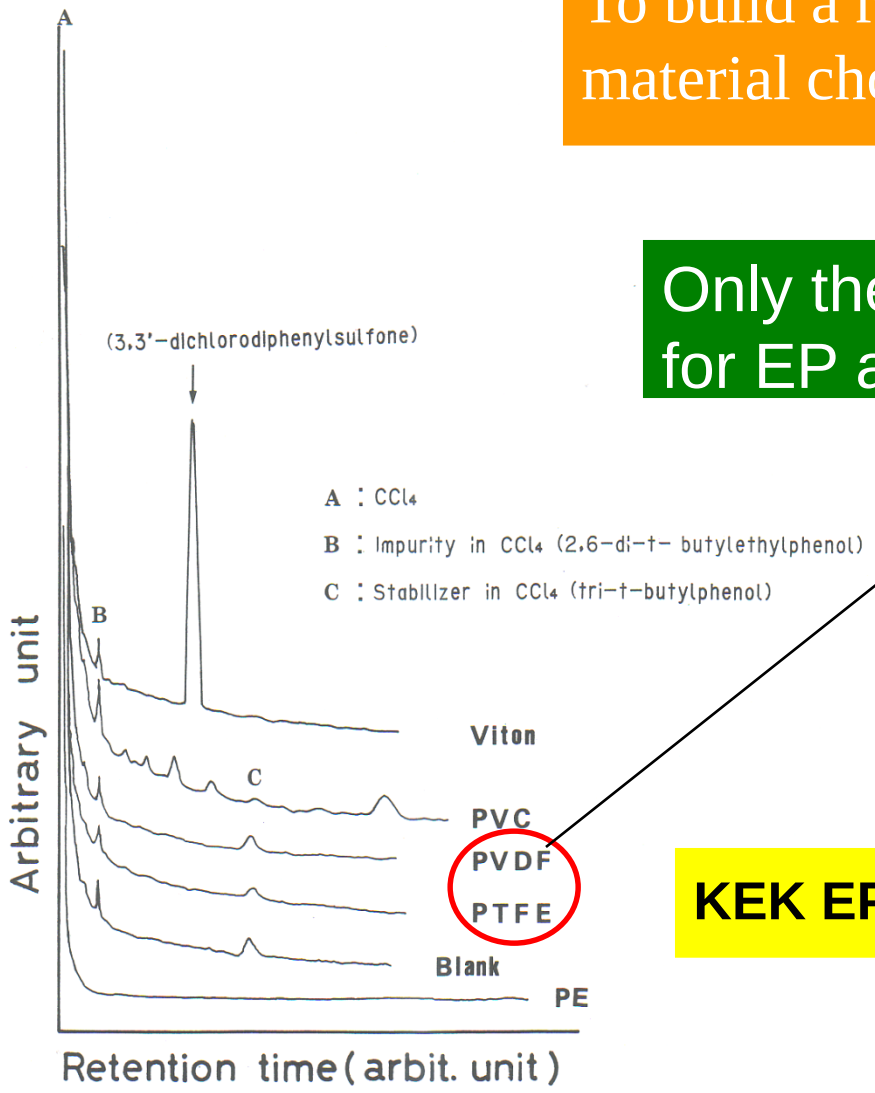


Yield =28/32=88%

Material Choice for the Reliable EP System

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

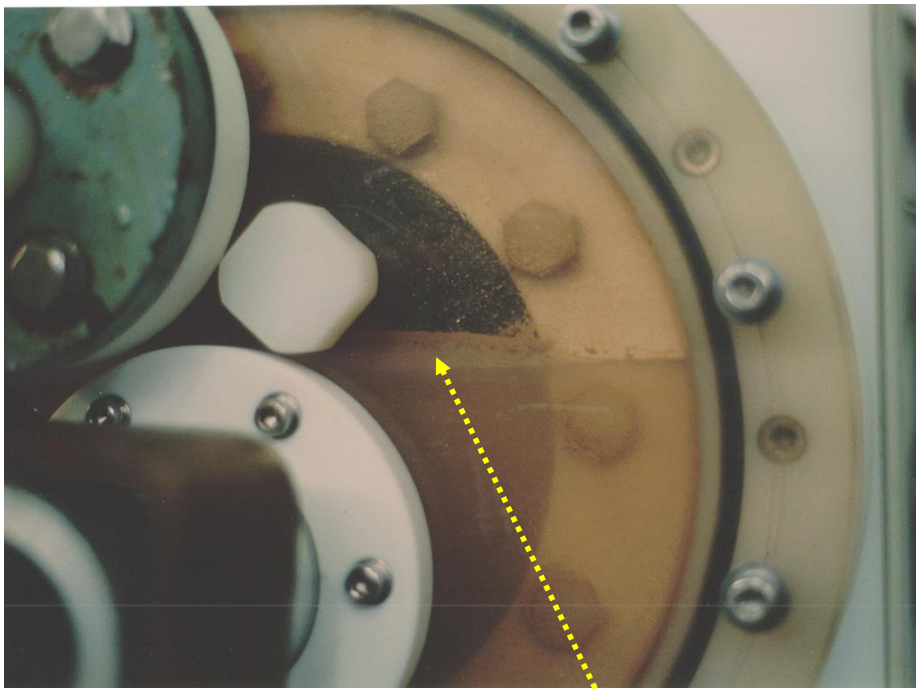
Only the Teflon is the reliable material for EP acid line.

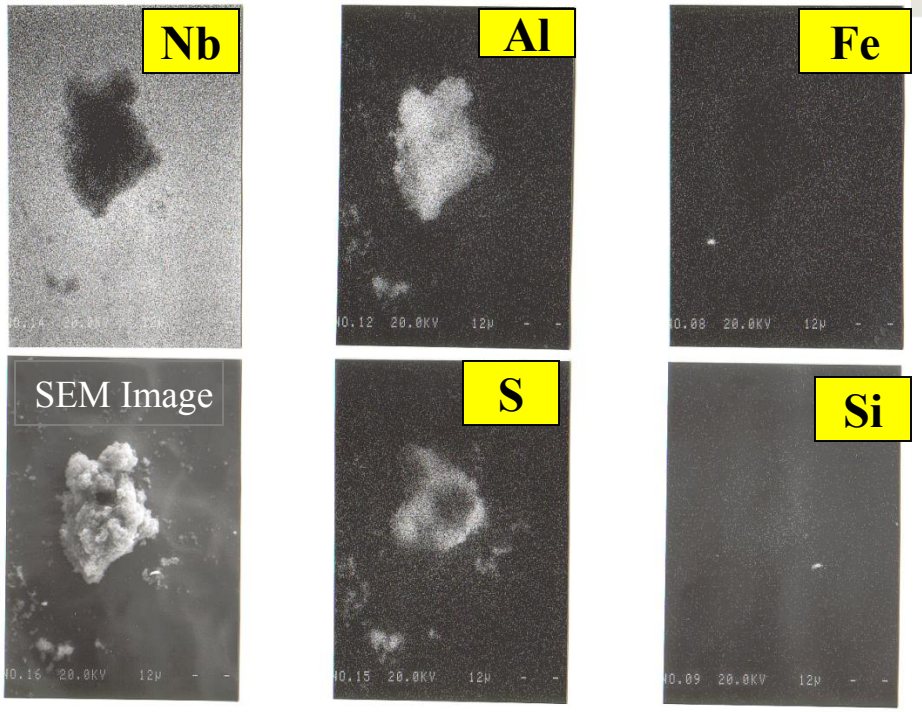


KEK EP system is still working after 20 years.

Dissolved chemicals into EP acid from Plastic Materials

Contamination Problem from Buffing Residues



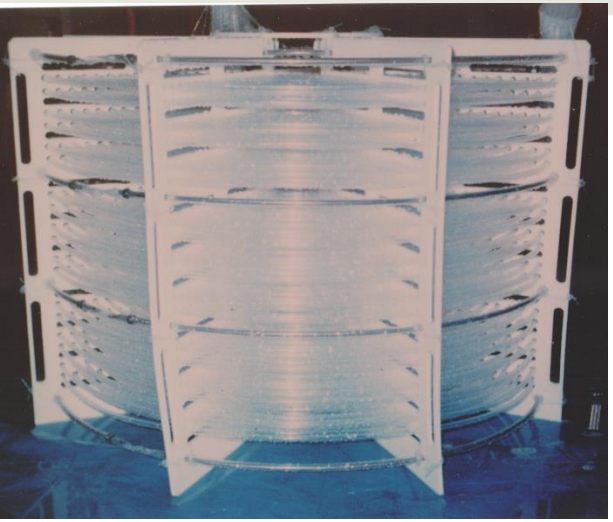


Precipitation of contaminants in the rotary sleeves

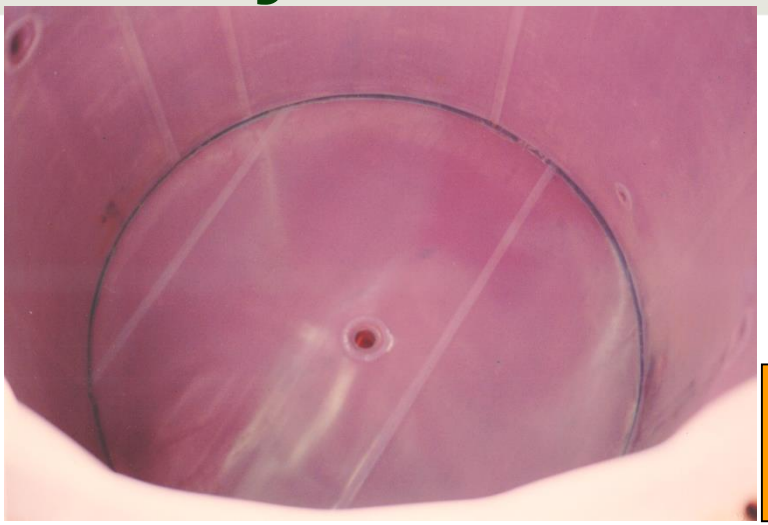
Al, Si, Fe come from the buffing residues
Sis produced by reductive reaction of H_2SO_4 acid.

TRISTAN met the field emission problem during production.
EP system was cleaned once in the production and EP procedure was modified to mitigate the contamination issue.

Sulfur Contamination in EP System



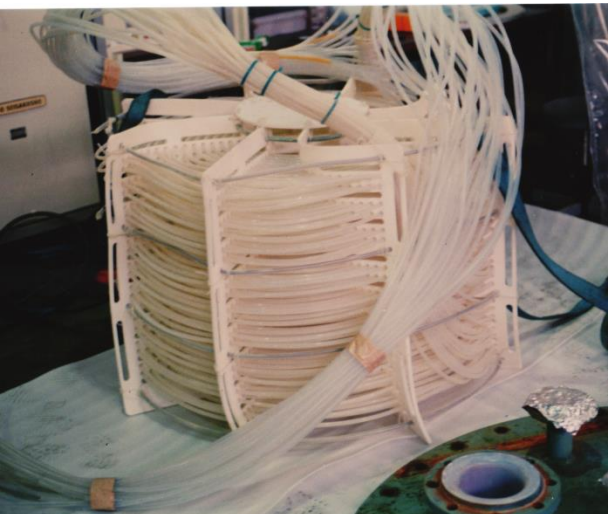
Teflon heat exchanger tube in the EP acid tank (brand new)



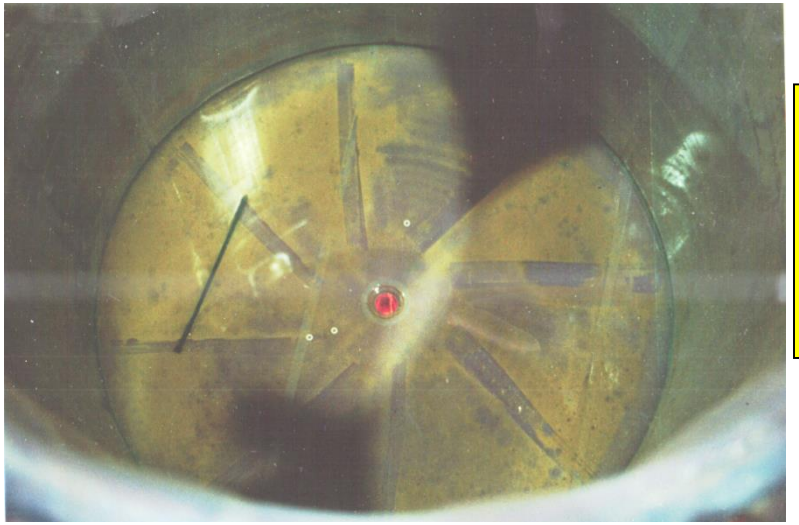
Teflon lined EP acid tank (brand new)

Generation of Sulfur by the reduction reaction of sulfuric acid

Sulfur precipitates around buffing residues



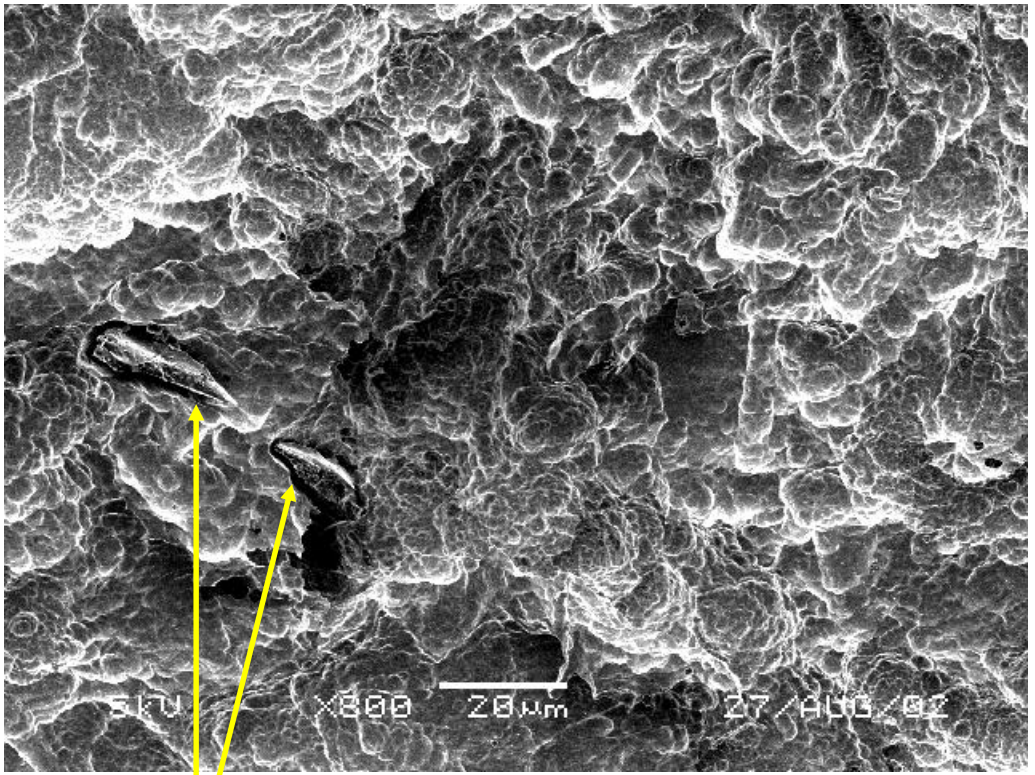
Contaminated Teflon heat exchanger tube



Contaminated EP acid tank

EP system
Was disassembled and clean up during the TRISTAN production.

Contamination Cure for EP System：Pre-EP



Buffing residues on the cavity surface
（Abrasives by Buffing）

Buffing
Embedding of abrasives

↓

Pre-EP
2~3μm removed by
off-line EP
using closed EP acid
in the cavity (no circulation)
Remove the contaminants

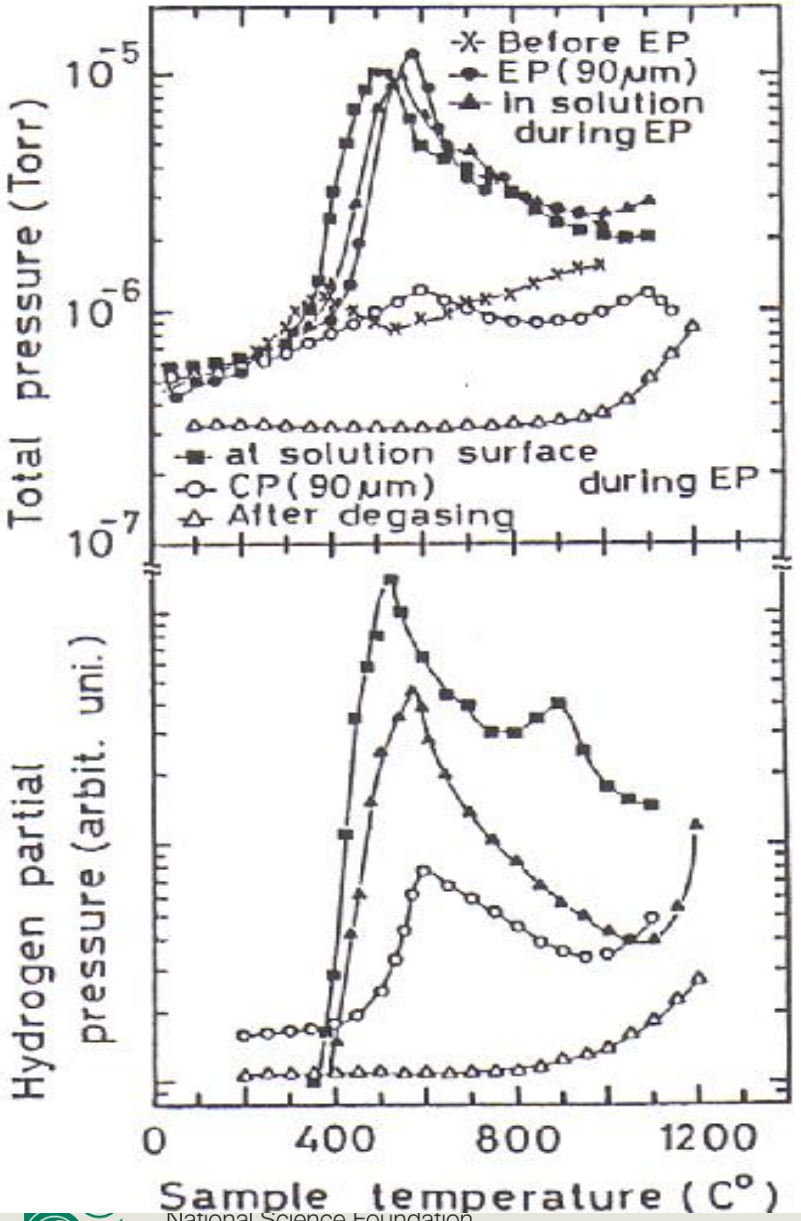
↓

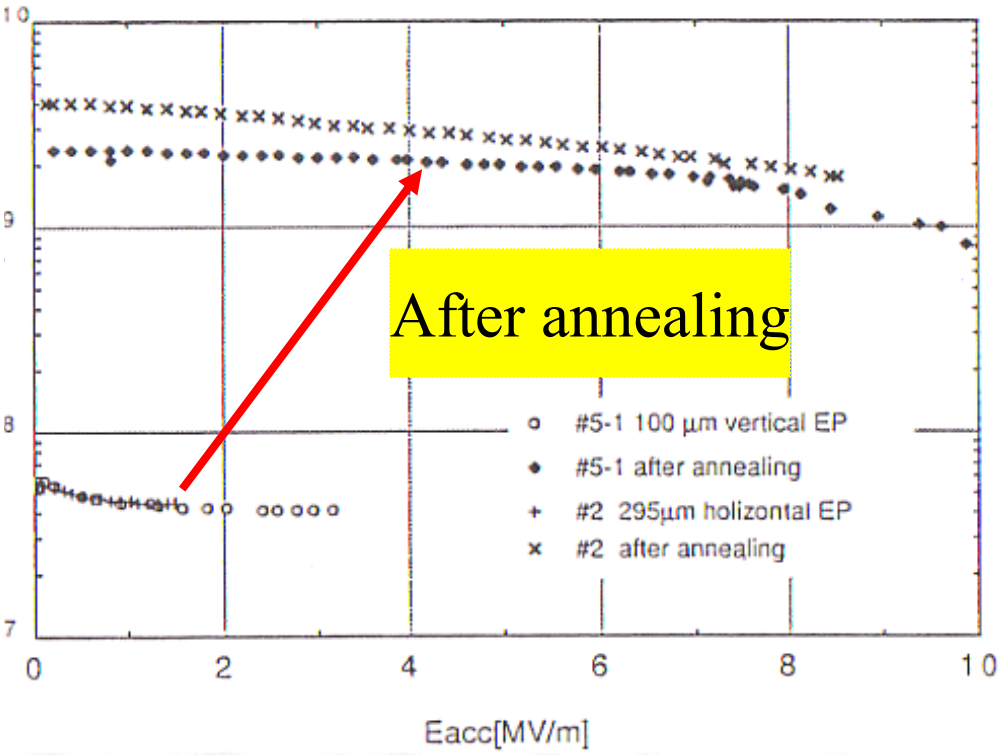
Dump the EP acid

↓

Heavy EP

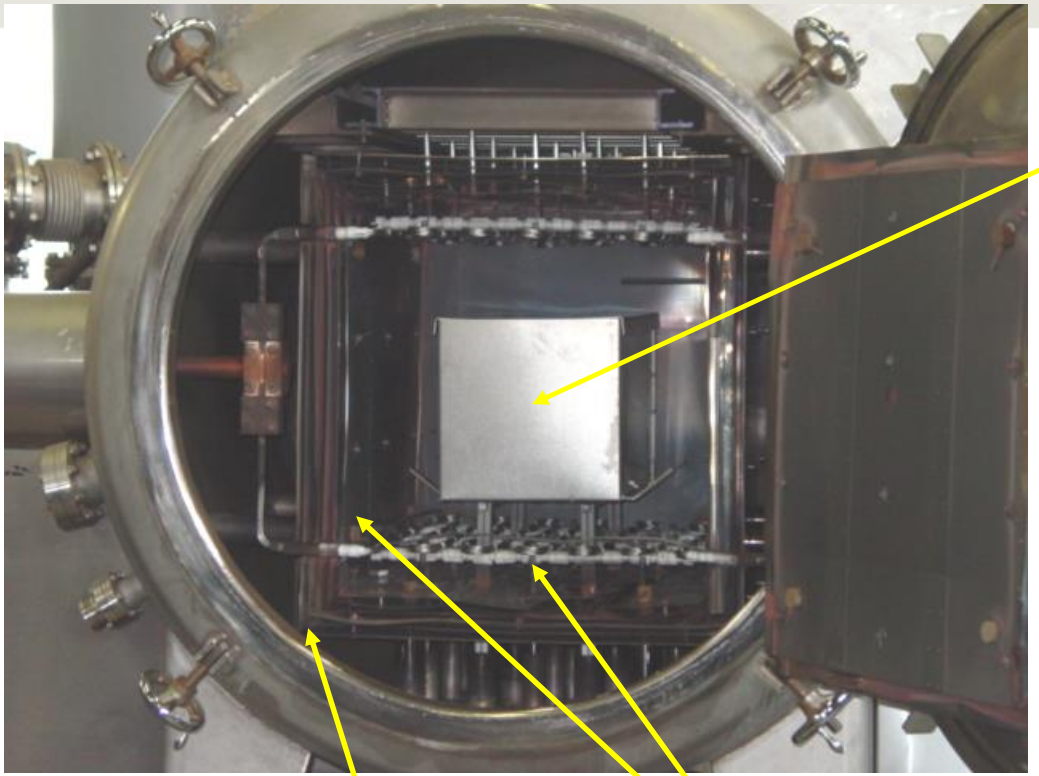
16.5 Heat Treatment (Hydrogen Degassing)





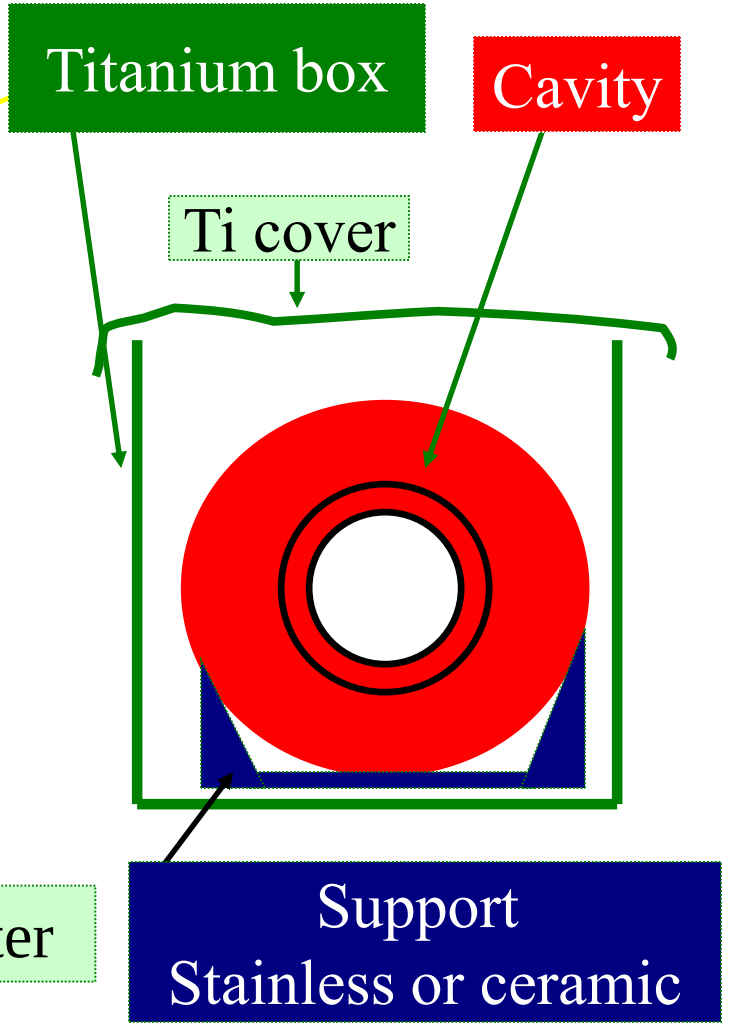
Vacuum annealing at 700~800^{O}C is very effective for hydrogen degassing. Nb material is not softened so much .

R&D Annealing Furnace in KEK Machining Center



Vacuum furnace at KEK machining center

1300^{O}C max, 1 x 10^{-6} Torr

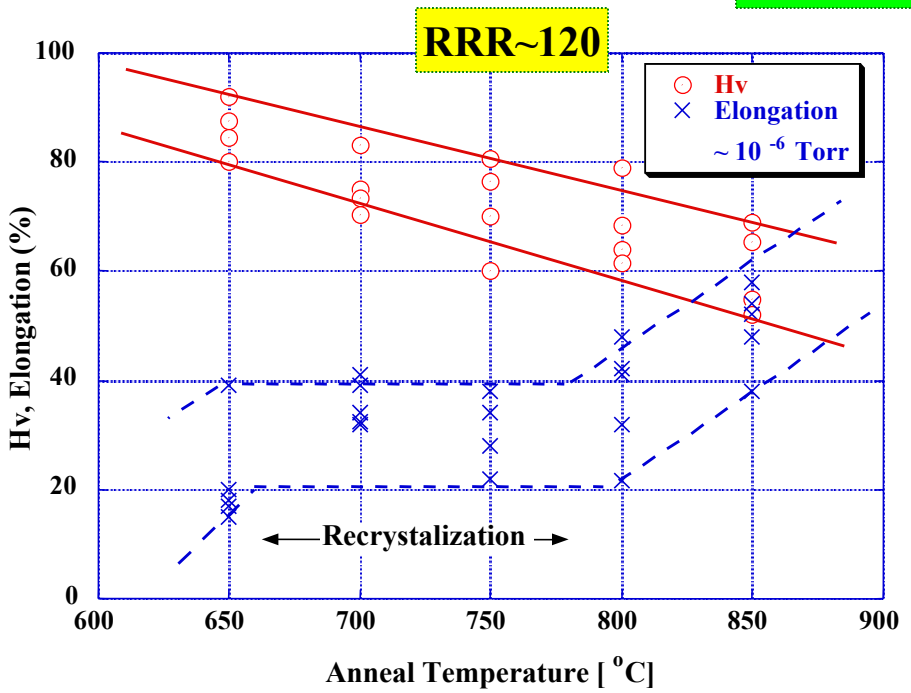


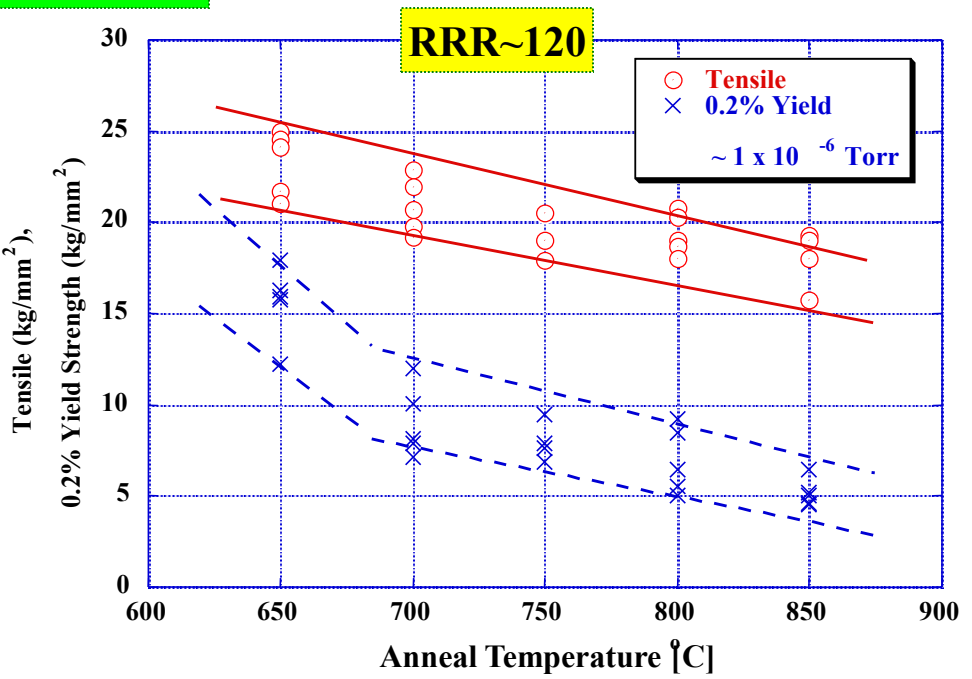
Large Vacuum Furnace for ILC Cavities (KEK Machining Center)

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

Annealing Temperature and Niobium Mechanical Properties

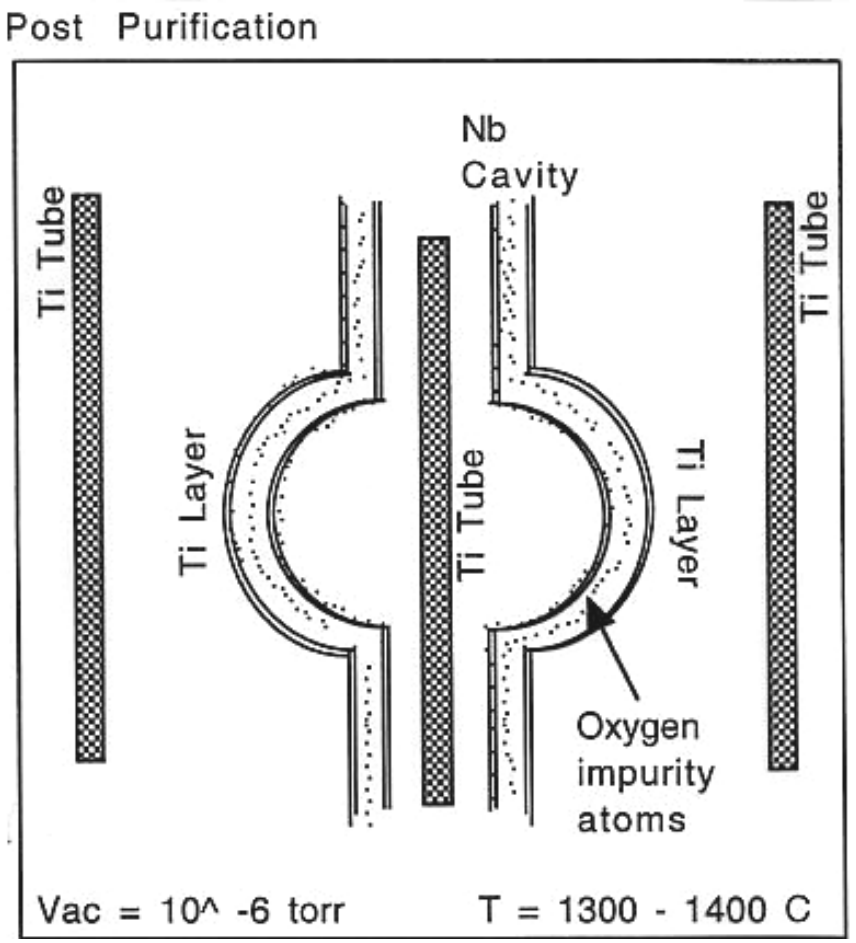
TRISTAN





Re-crystallization Temperature : 680 ~ 780°C
Vacuum : ~10^{-6} Torr

16. 6 Post Purification (Titanium Treatment)



- **Cavity is annealed surround Titanium sheets around it in the vacuum at the temperature higher than 850^{O}C.**
- **Titanium is evaporated and attached on cavity surface, which acts getter action and pick up Oxygen from the niobium bulk.**

Diffusion Coefficients

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

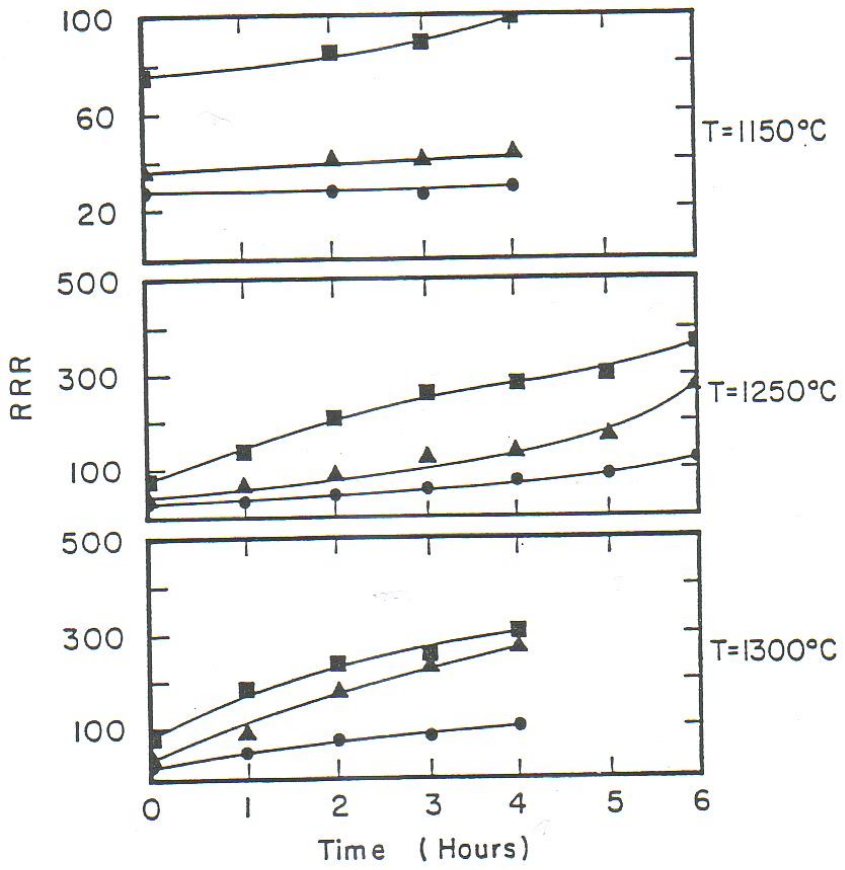
$$C : 0.043\exp(-\frac{1.670\cdot 10^4}{T})\ \ cm^2/sec$$

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

T (°C)	O mm/hr	N mm/hr	C mm/hr
900	0.4	0.005	0.0
1000	0.6	0.008	0.0
1100	0.9	0.13	0.1
1200	1.2	0.19	0.1
1400	2.0	0.38	0.3

DESY initially planned to make post Purification for TTF cavities at 1300^{O}C.

RRR Improvement with the Post Purification



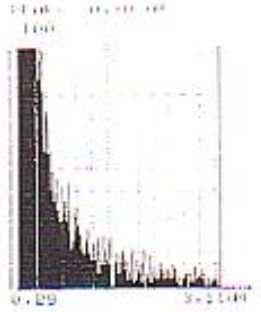
Dependence of the RRR-value on reaction temperature and reaction time for niobium samples of different purity exposed to titanium vapor (● 1/8" thick, RRR=27; ▲1/8" thick, RRR=37; ■ 1/16" thick, RRR=77)

Improved by a factor 3 by 6hr post purification process
but material softening is a concern.

P. Kneisel, Cornell/Jlab

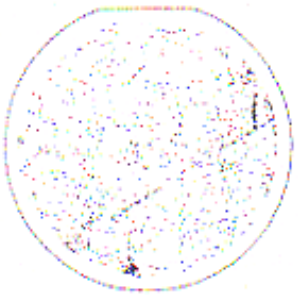
16.7 High Pressure Water Rinsing (HPR)

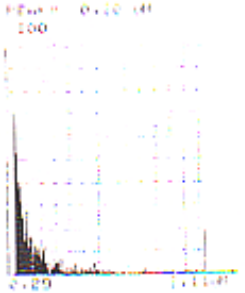
TRISTAN rinsing method



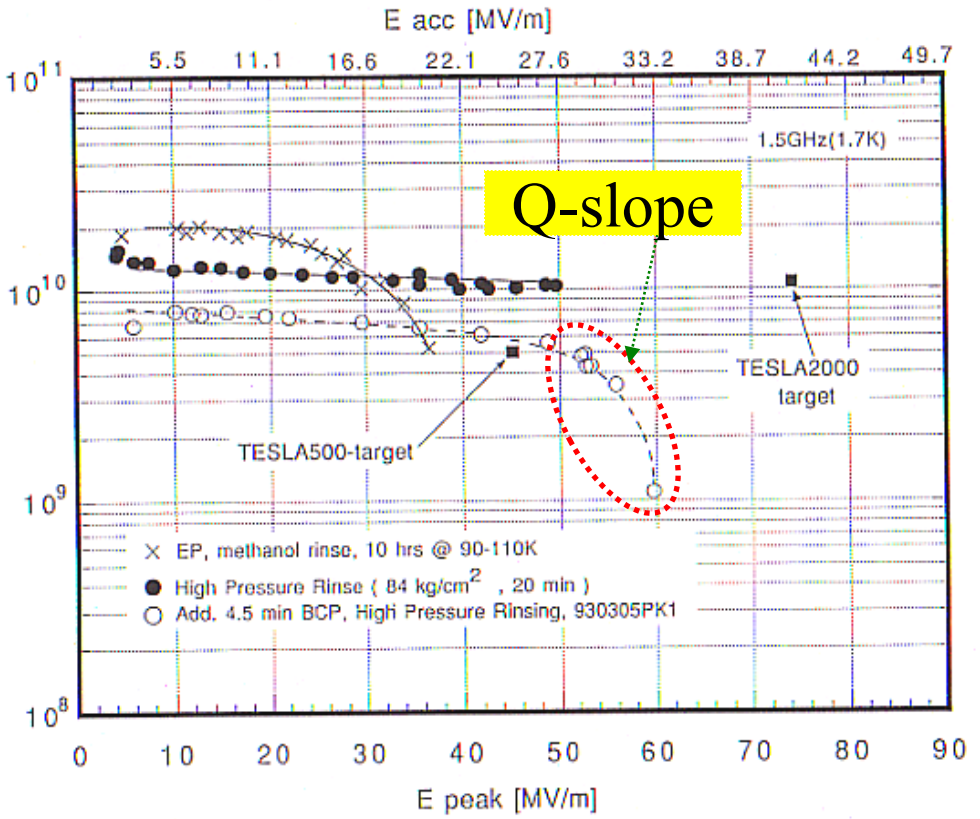
Particle size	Count
0.30-1.20 μm	5825
1.20-2.01 μm	405
2.01-3.00 μm	2720
> 3.00 μm	1069
Total	10019

HPR rinsing



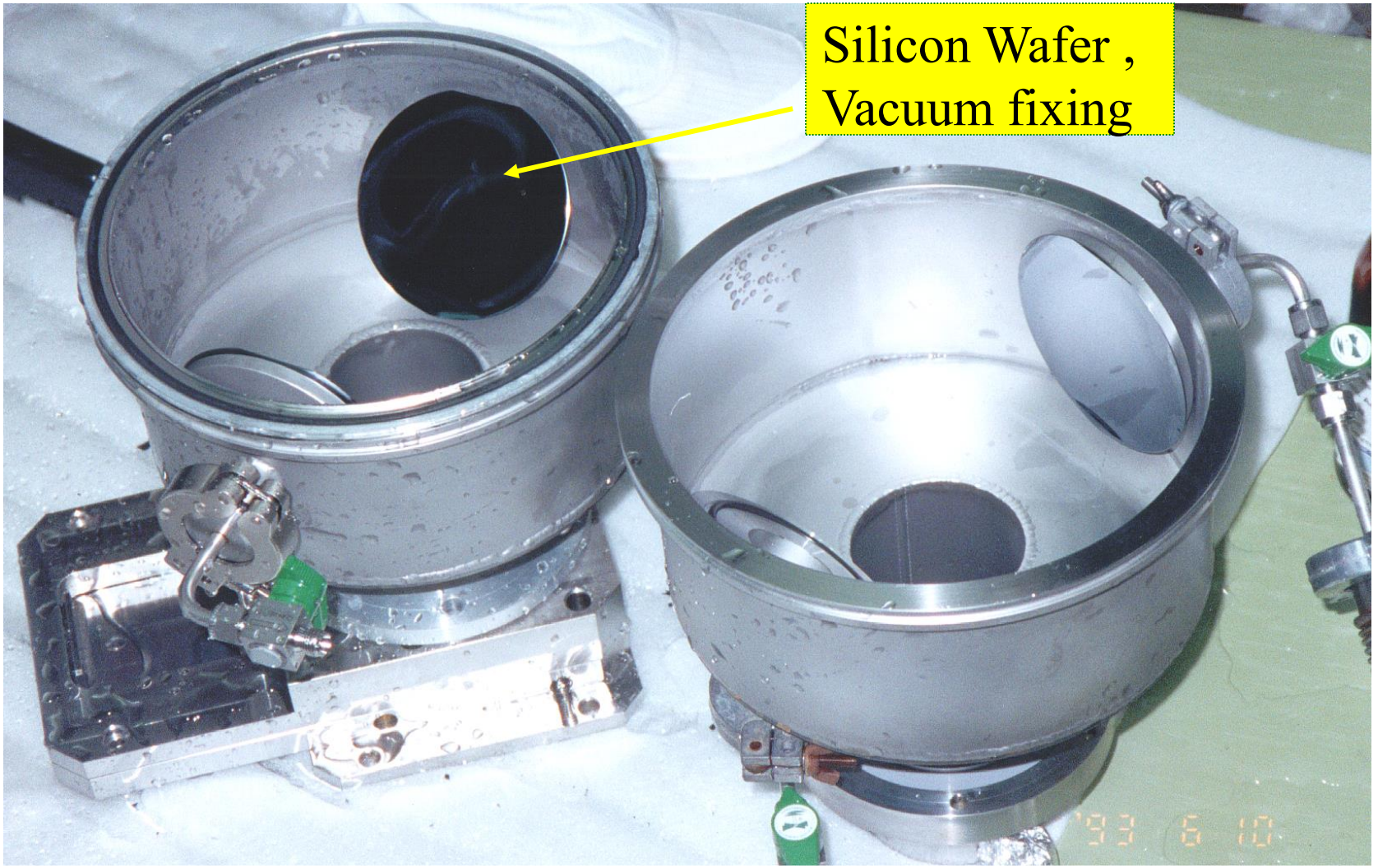


Particle size	Count
0.30-1.20 μm	646
1.20-2.01 μm	52
2.01-3.00 μm	282
> 3.00 μm	37
Total	1017

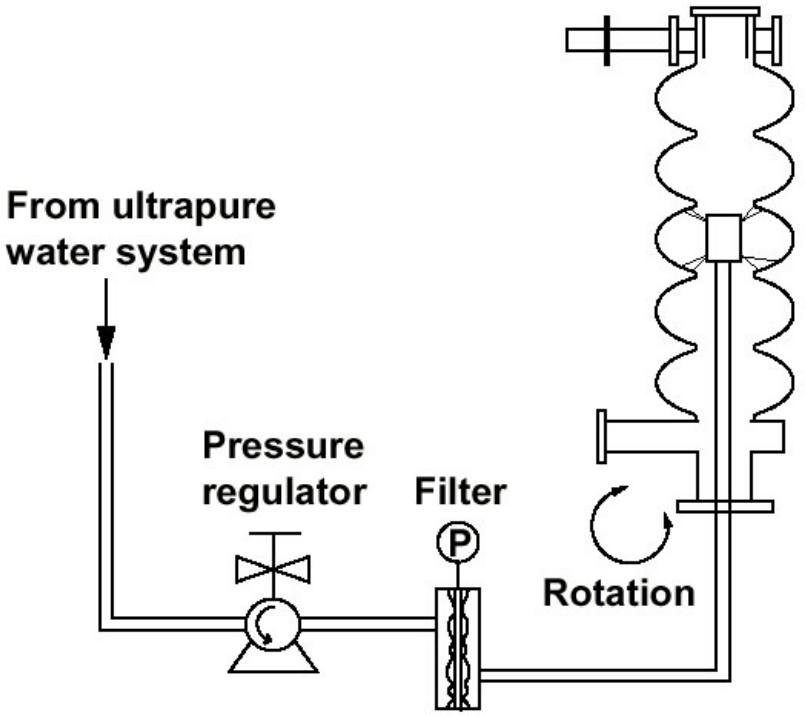


HPR is a very powerful tool to remove the particle contamination on niobium cavities.

HPR Test Using Silicon Wafers

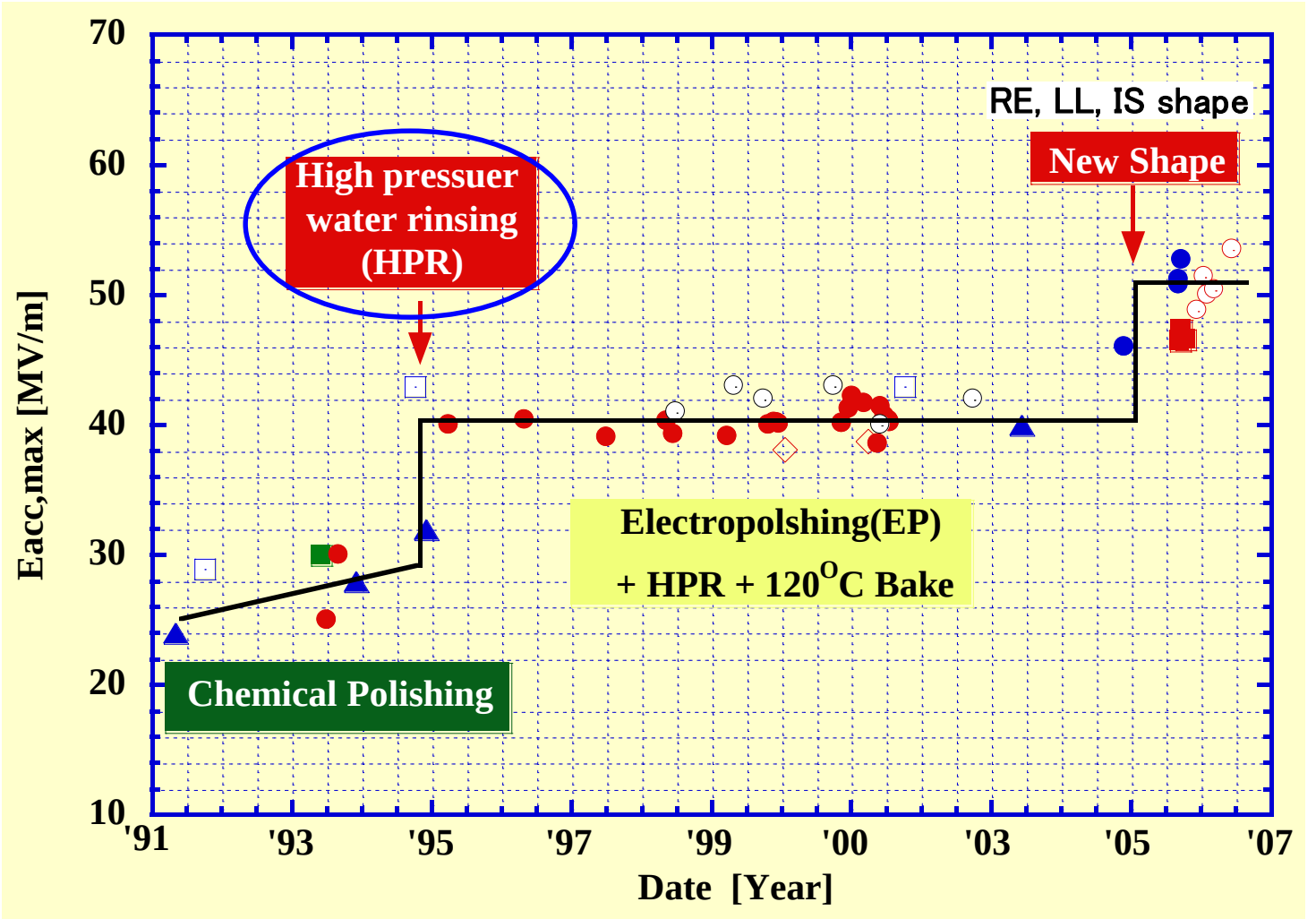


HPR System

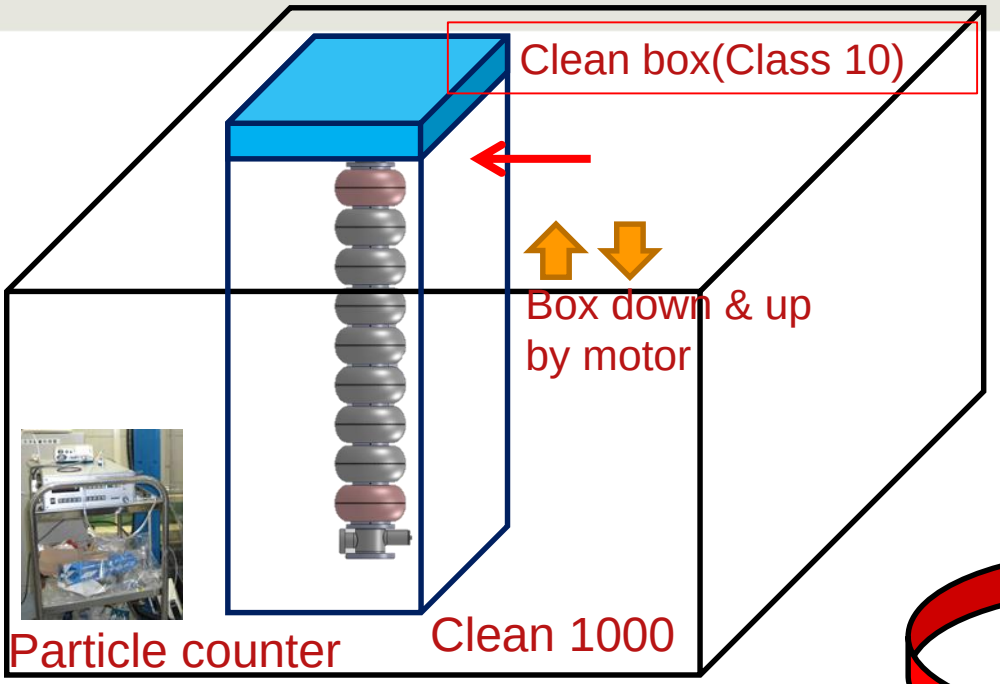


Nomura Plating Co. Ltd.
（株）野村鍍金鹿沼工場

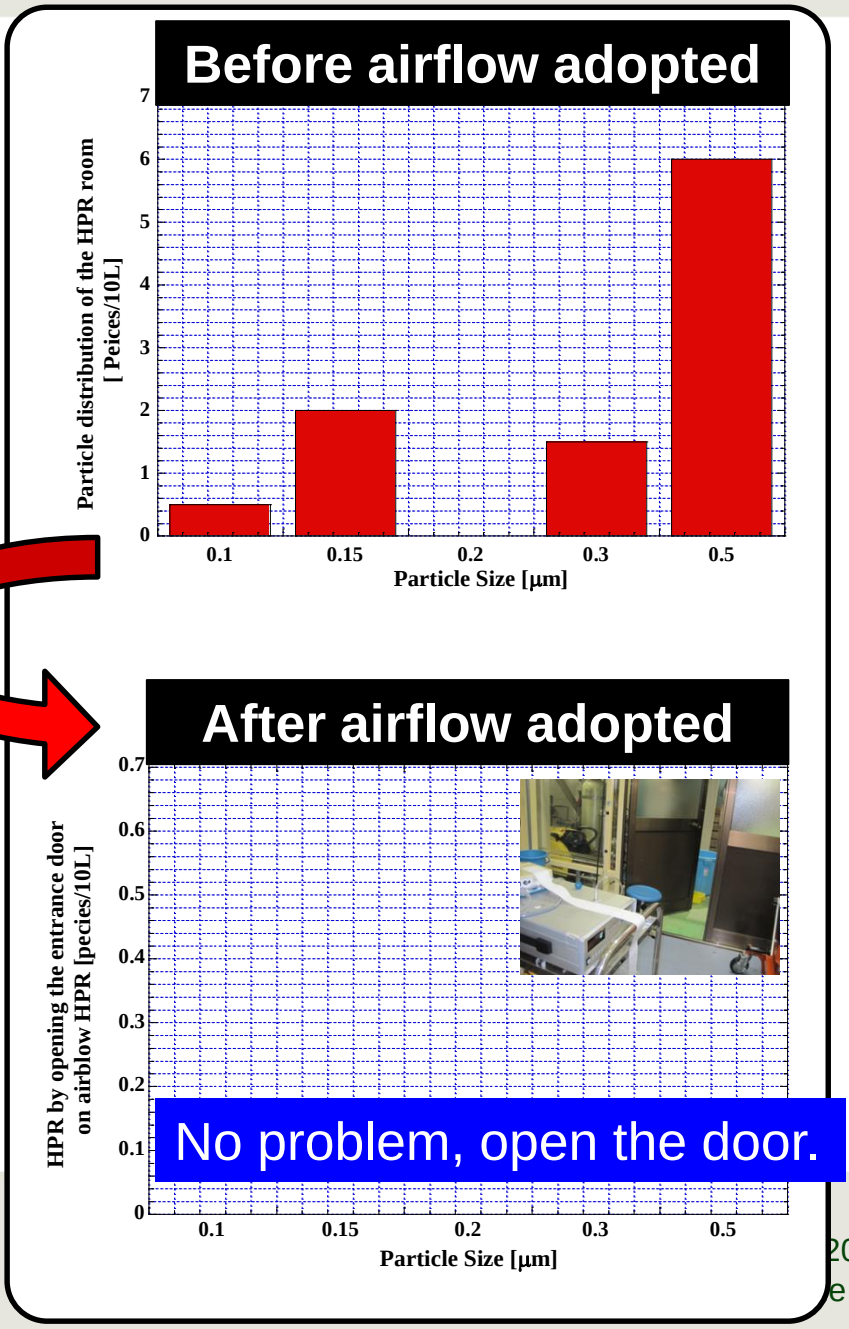
Breakthrough for 40MV/m



Before and After Particle Free Airflow Adopted



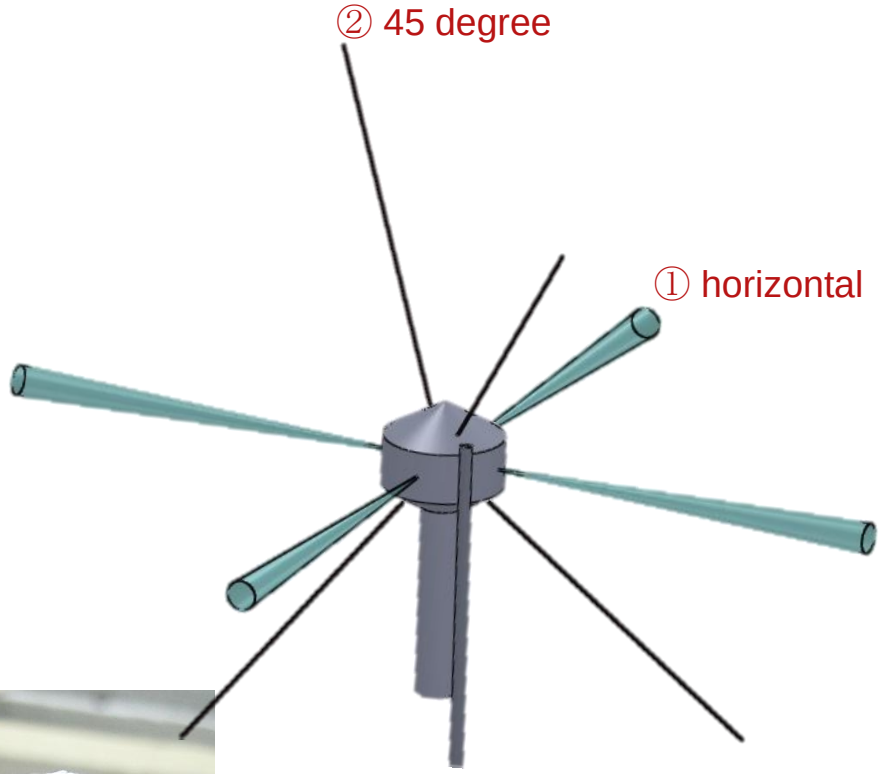
Our HPR room

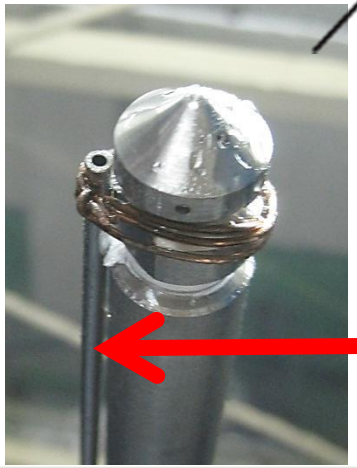


- Investigated the particle environment in our HPR room. Lots of particles were observed in the cavity.
- Adopted a clean airflow method.
- Then, we got the of particle free result inside the cavity.

Even opened the entrance door, no particles comes into the cavity during HPR.

HPR Nozzle and Airflow





Clean air flow line

Parameter of HPR Nozzle	
Flow velocity	10 L/min
Hole radius of Nozzle	0.5 mm
Spread angle of Horizontal ①	2 degree
Spread angle of 45deg ②	0 degree

Parameter of Airflow line	
Inside diameter	2 mm
Outer diameter	3 mm
Flow velocity	0.4 MPa

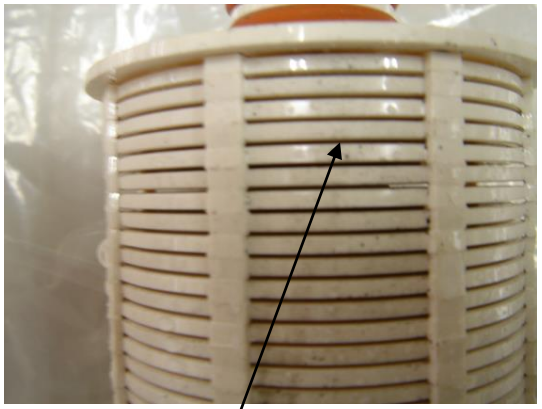
HPR Contamination Trouble

Be careful, HPR pump is sometimes a source of contamination

Replaced to new HPR pump @ Nomura
(plunger type)

HPR final filter was contaminated due to
new plunger, poor rinsing for ethylene-glycol or other reason

Contaminated filter



Oil ?

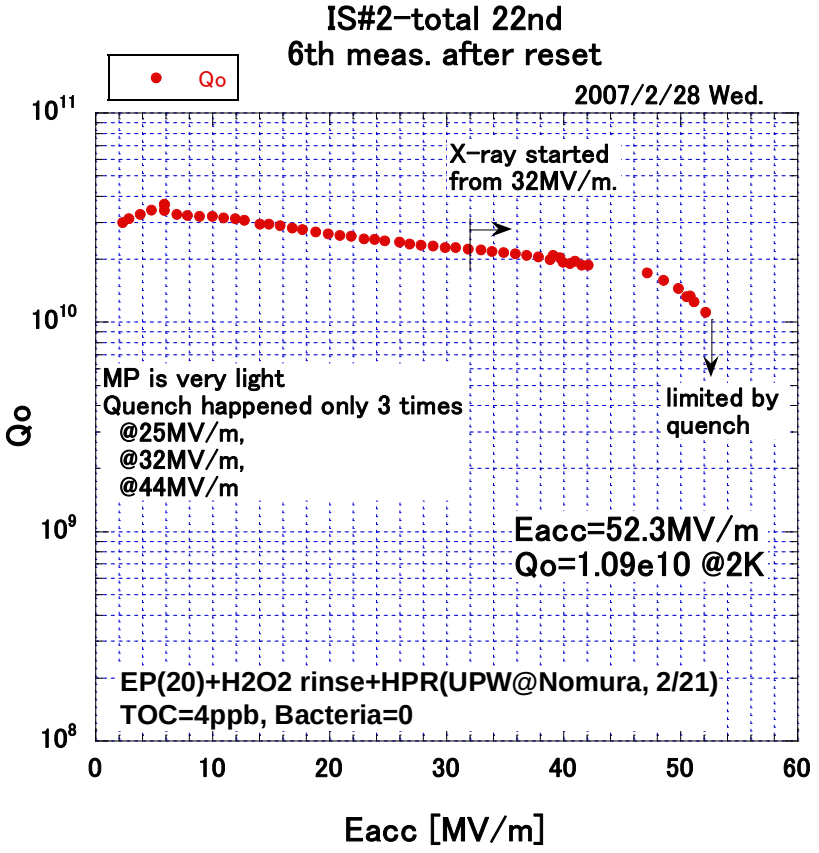
New filter

The maker shipped the pump filling Ethylene-glycol.

HPR pump @ KEK : KÄRCHER HDS8/14C

Contamination Incident by HRP Pump

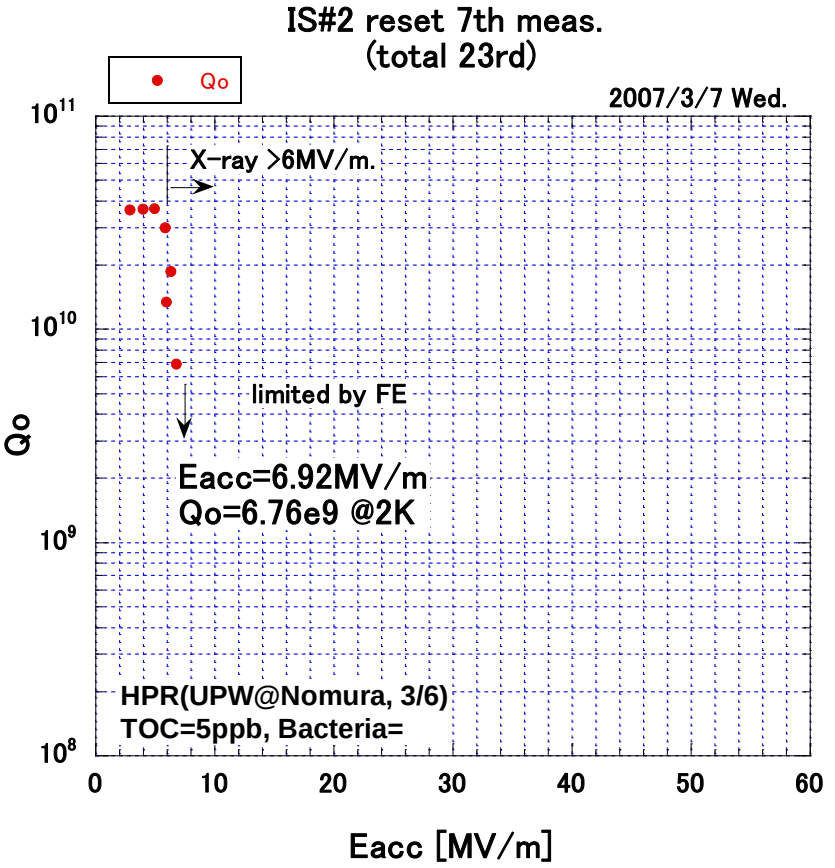
EP(20)+H_2O_2+HPR @Nomura



Before the HPR contamination

Additional HPR @Nomura

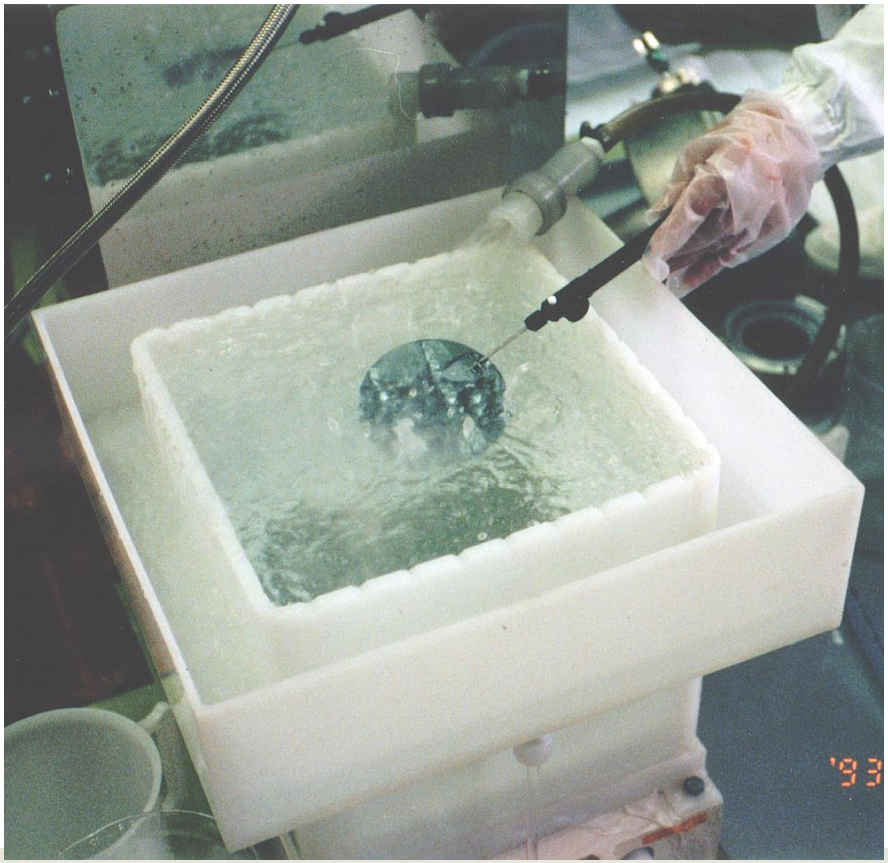
Contaminated by HPR pump !!



After the HPR contamination

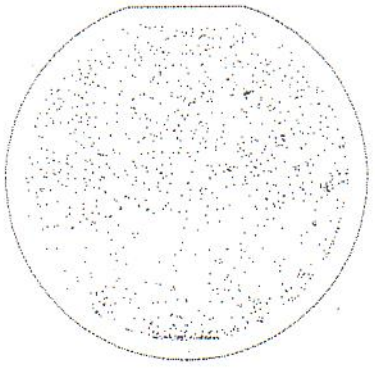
16.8 Megasonic Rinsing

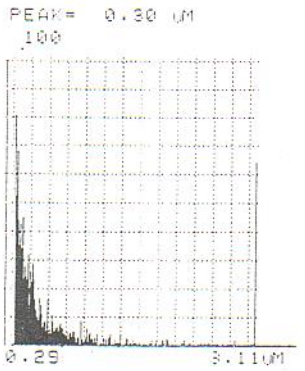
- Megasonic is the similar to ultrasonic (20-100kHz) but the frequency is 950 KHz.
- Megasonic is more powerful to remove particle contamination.
- Megasonic is an attractive rinsing method if the oscillator is well developed for SRF cavities.



Megasonic Rinsing Effect

HPR rinsing

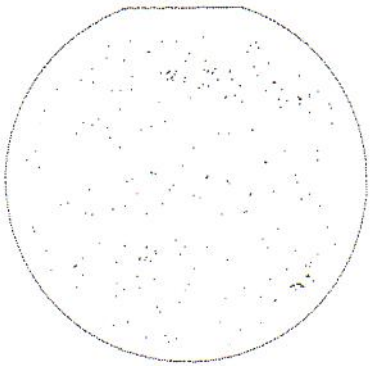


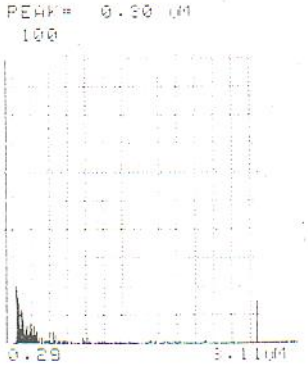


Particle size	Count
0.30-1.20 µm	673
1.20-2.01 µm	50
2.01-3.00 µm	305
> 3.00 µm	90
Total	1118

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

Megasonic 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.

The amount of particles is 1/5less than HPR in case of Megasonic rising

Developed Megasonic Rinsing Oscillator for SRF Cavities

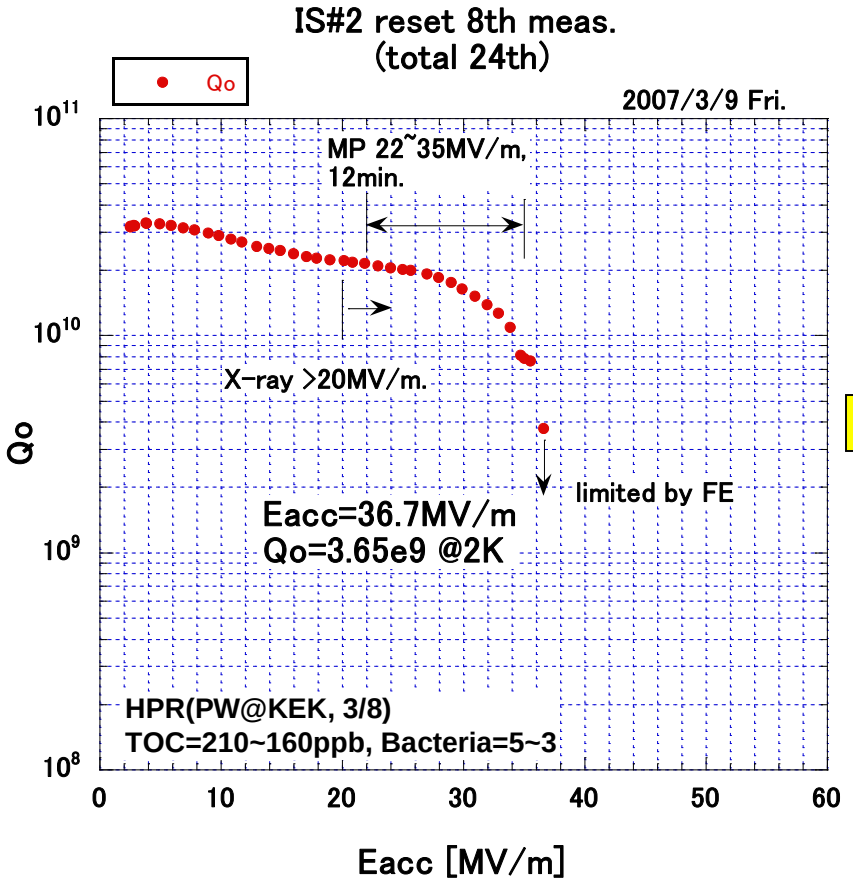
The oscillator is mounted in the cavity.

10.9 Cleaning Methods Post EP

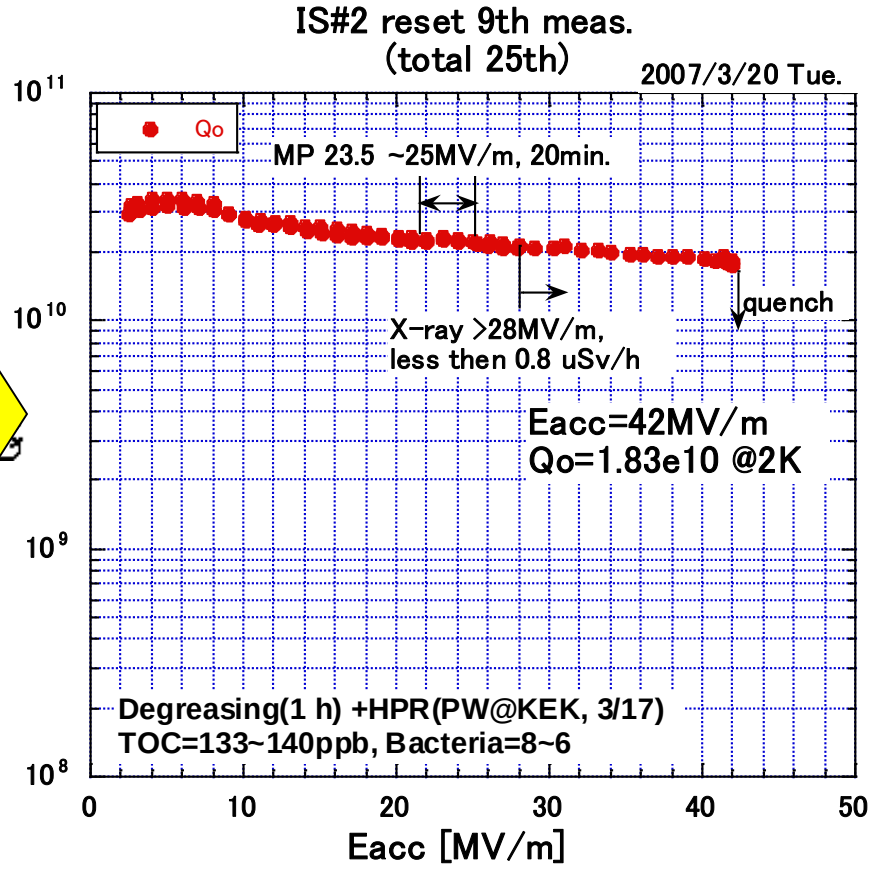
- EP produces surfer contamination which is caused by decomposition of H_2SO_4 during EP. Surfer is good field emitter.
- Up to 1986, Anodizing or Alcohol rinsing took place to remove the surfer contamination (cleaning post EP) .
  - Anodizing
    - Make oxide (Nb_2O_5) file 2000A applying voltage up to 100V in the ammonium solution
    - Remove the oxide film immersing HF acid
    - Very hazardous work and costly
  - Alcohol rinsing
    - Put Ethanol alcohol in the cavity and rinse
    - Need explosive-protection system
- KEK applied H_2O_2 (10%) rinsing instead these method in 1986 (TRISTAN SRF)
  - Cheaper and safe
- In the ILC R&D at KEK, high cavity performance was obtained with single cell cavity even without the cleaning post EP.
- When HREP technology was transferred to DESY, they met the field emission problem with their 9-cell cavities but the performance still scattered a lot.
- The post EP cleaning was concerned as the biggest issue early 2000's

Degreasing Post EP, Developed at Jlab

Additional HPR @KEK

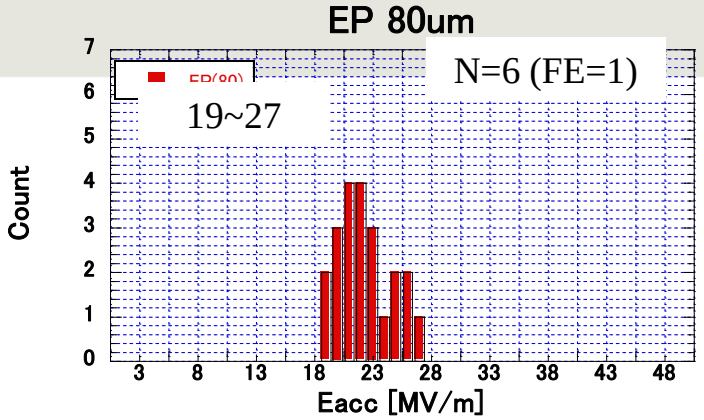


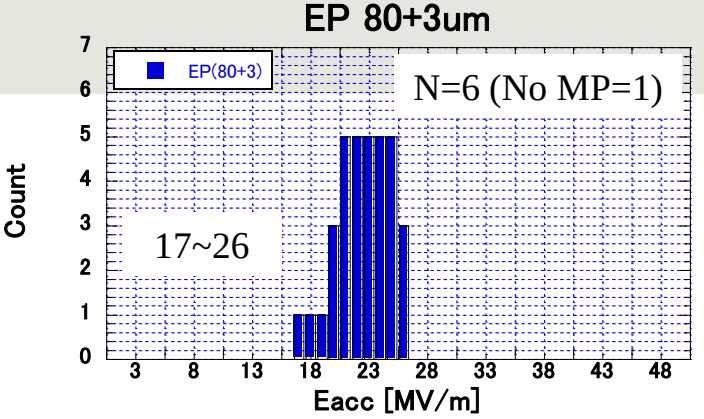
Additional Degreasing + HPR@KEK
(Use Japanese degreaser)

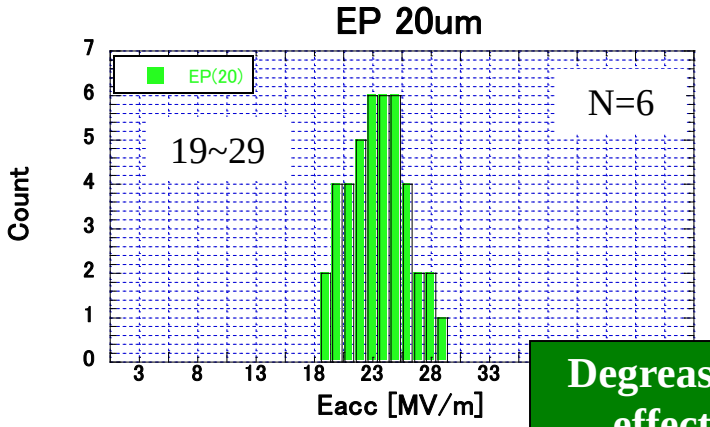


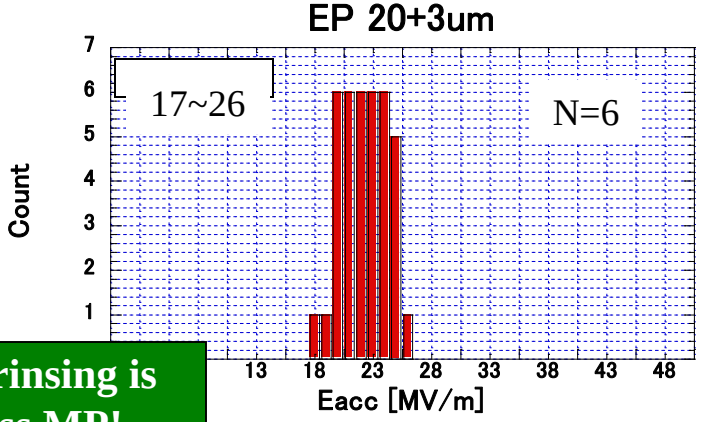
Degreasing is very effective to eliminate Sulfur contamination !

Various Cleaning Post EP and Multipcating

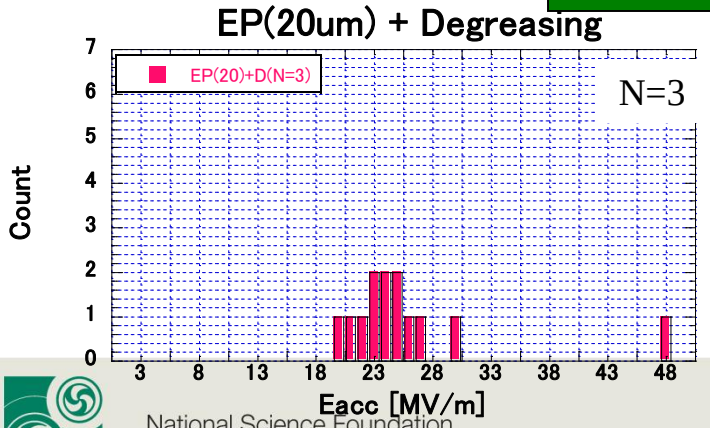


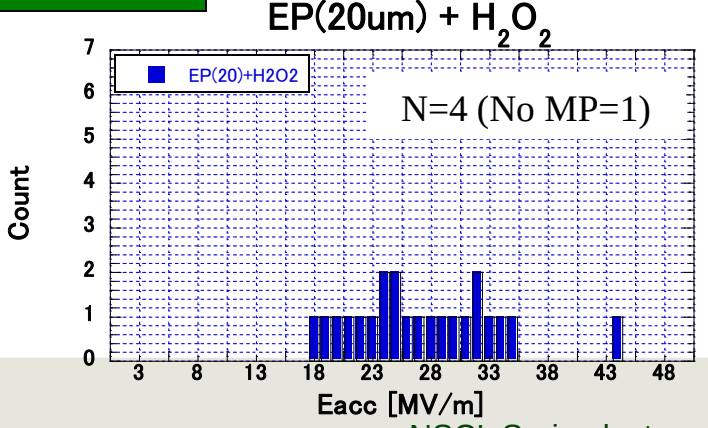






Degreasing or H_2O_2 rinsing is effective to suppress MP!





Homework

- **16.1 Homework**

In this lecture various SRF surface preparation technologies were introduced. However, these technologies will become not to be used by improving of the cavity fabrication technology, for instance post purification is not used anymore because of high RRR niobium sheet commercial production.

Anyway, the SRF preparation technologies are very complicated. They need to be replaced with more simple and cost-effective way.

Describe your opinion which kind of preparation technology can be replaced by new technology. What is your idea?