EP parameter comparison between JLab and KEK

28 Mar. 2010

ILCW2010 at Beijing, GDE/Main Linac session

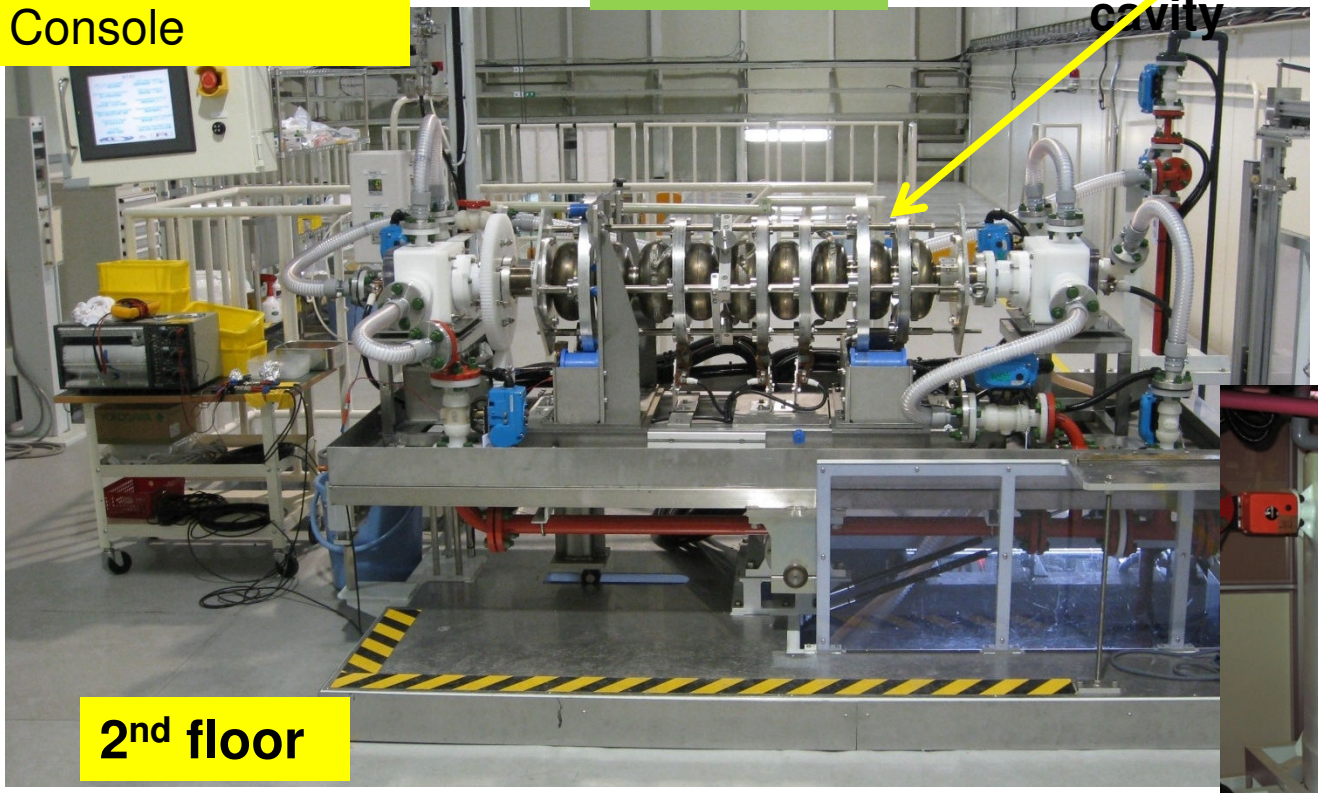
T. Saeki

EP facility at STF/KEK

Automatic Operation Console

EP bed

9-cell cavity



2nd floor

1st floor

EP solution reservoir tank

New EP facility at KEK was constructed in 2008, instead of old Nomura EP facility, which was closed in the summer of 2009.

Stain problem at KEK (Summer 2009)

After the EP acid is replaced with brand-new one (1000-L tank), we had heavy field emission in vertical test of EP-processed cavities.

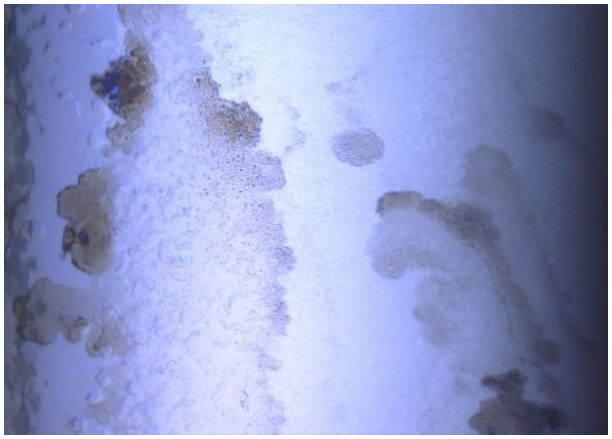
Brown spot and traces;

MHI-06 : spot or traces on BP-#1、#1、#2、#3、#4、#8、#9、#9-BP (red indicate bad)

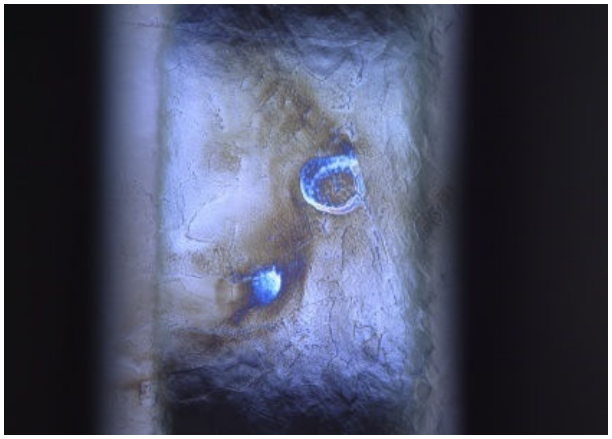
MHI-07 : BP-#1、#1、#2、#3、#4、#5、#6、#7、#8、#9、#9-BP

Kyoto-camera picture examples using new LED illumination

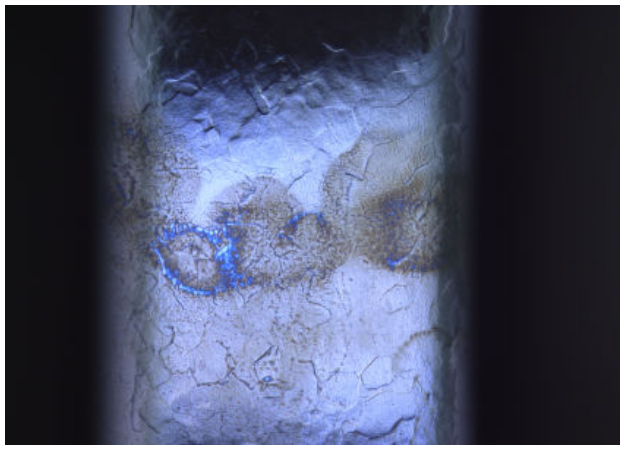
BL#6 1-cell equator, t = 306 deg.
Downstream : Outside weld area



BL#6 #9-BP, t = 241 deg. -1

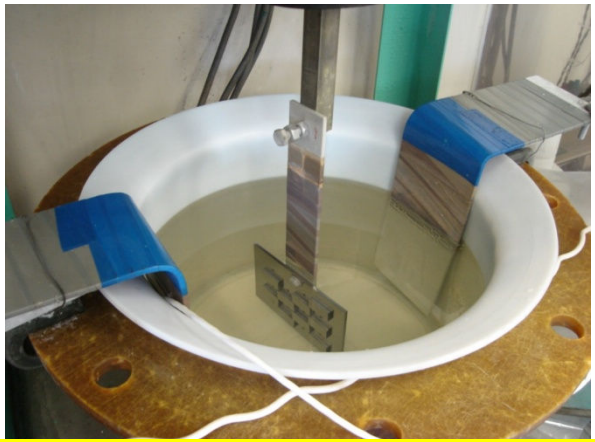


BL#6 #9-BP, t = 241 deg. -2

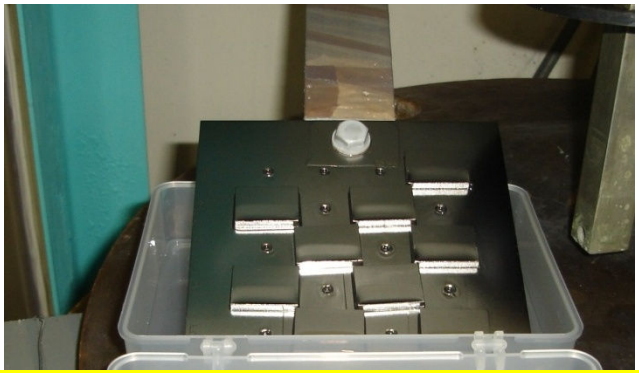


Labo-EP of samples at Nomura

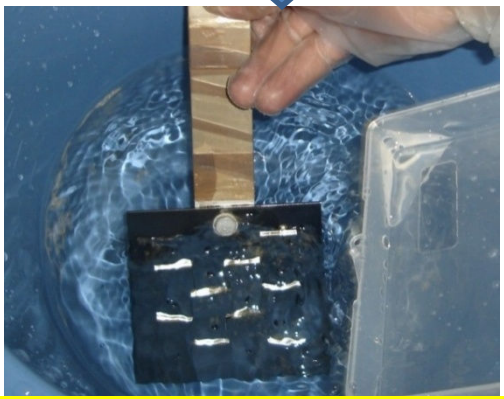
11 Aug. 2009



Labo-EP (20 um) with new EP acid ([Nb] = 0 ~ 0.4 g/L) at Nomura



Exposing the samples to the air for 70 min. w/o Pure-Water (P.W.) rinse. No stain appeared.



Light P.W. rinse for a few 10's seconds.

Stains appeared within a minute after exposing the samples to the air.

Summary of sample tests

[Nb] of EP acid (at the end of EP)	Duration of exposure to the air. Stains?	Duration of light P.W. rinse	Duration of exposure to the air. Stains?
0.4 g/L	70 min. No stain	A few 10's sec.	Stains appeared within a min.
4.8 g/L	30 min. No stain	A few 10's sec.	6 min. No stains
8.6 g/L	30 min. No stain	A few 10's sec.	4 min. No stains

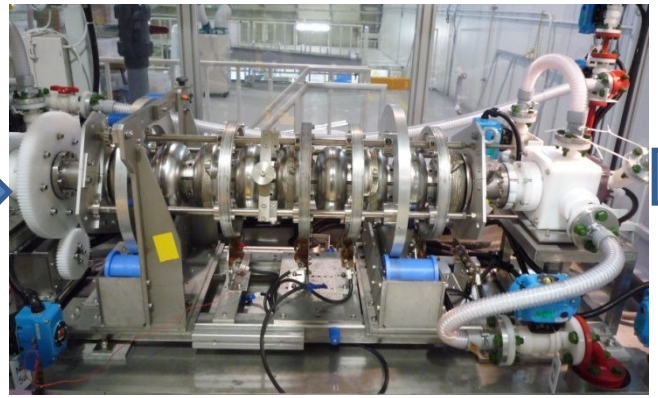
Considerations

- KEK EP-facility has a big EP-tank of 1000L. When we replace the EP acid with new one, we will keep using new EP acid with some 9-cell cavities for a while.
- Should we change the amount of EP-acid in the tank? Or should we develop a new U.P.W. rinse method, like introducing N2 during first U.P.W. rinse duration?

Modification of first U.P.W. rinse process

First U.P.W. rinse duration after EP-acid-draining at STF/KEK was extended.

Drain EP acid for 15 min.



Horizontal for 5 min.

Drain EP acid for 10 min.
Then UPW rinse starts.

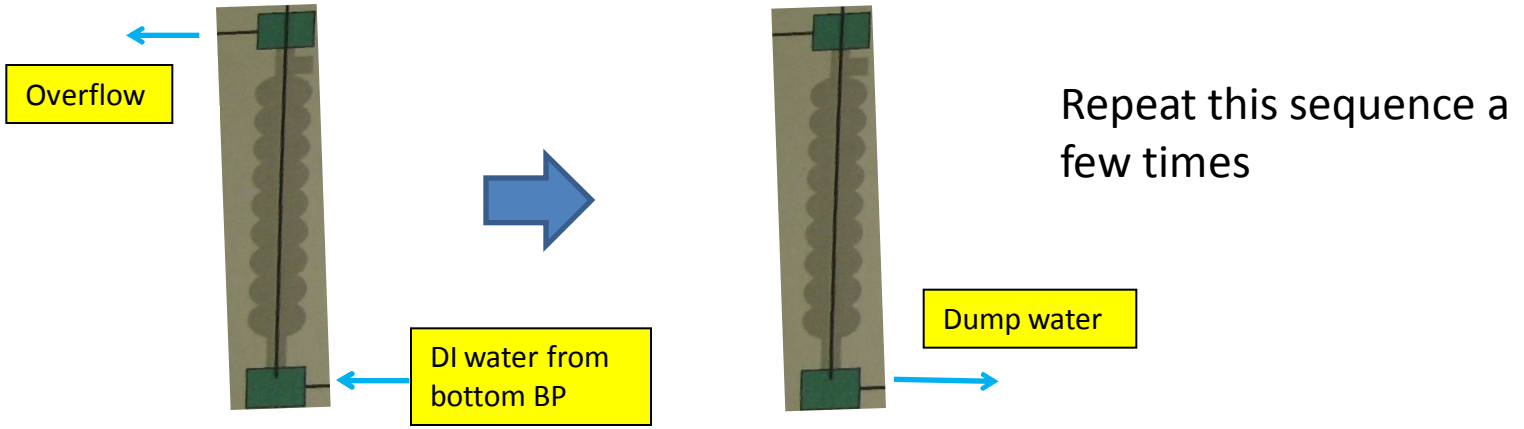
The first U.P.W. rinse was extended with overflow for a longer time.
Original sequence: [pouring U.P.W. for 7 min. + draining for 5 min.] x 5
Modified sequence: [pouring U.P.W. for 60 min. + draining for 10 min.] + [pouring U.P.W. for 20 min. + draining for 7 min.] x 3.

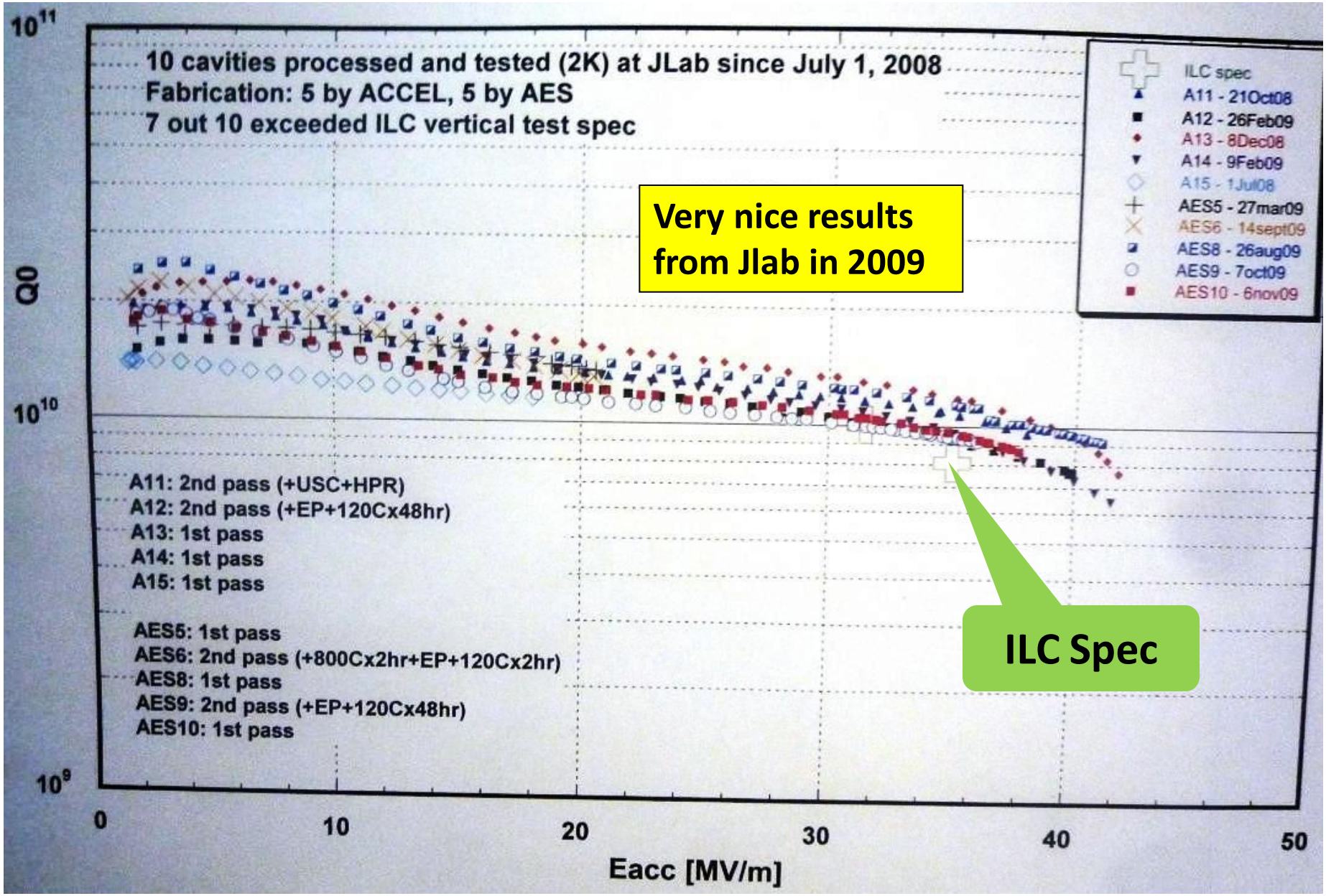
MHI#9 (9-cell cavity) was processed with this modified sequence and reached Eacc = 27 MV/m at Q0 = 9x10^9 (quench), even with some field emission.

Comparison of EP process among laboratories

DESY and JLab have no stain problem.

- DESY: The final EP is done with the EP acid of [Nb] ~ 10g/L.
- JLab: New EP acid is used for the final (20 um) EP 2 times, bulk (>100um) EP, and then dumped. So new EP acid is used for the final EP is similar to KEK. But the rinse process is different from KEK.




10 cavities processed and tested (2K) at JLab since July 1, 2008
Fabrication: 5 by ACCEL, 5 by AES
7 out 10 exceeded ILC vertical test spec
Very nice results from Jlab in 2009
ILC spec
A11 - 21Oct08
A12 - 26Feb09
A13 - 8Dec08
A14 - 9Feb09
A15 - 1Jul08
AES5 - 27mar09
AES6 - 14sept09
AES8 - 26aug09
AES9 - 7oct09
AES10 - 6nov09
A11: 2nd pass (+USC+HPR)
A12: 2nd pass (+EP+120Cx48hr)
A13: 1st pass
A14: 1st pass
A15: 1st pass
AES5: 1st pass
AES6: 2nd pass (+800Cx2hr+EP+120Cx2hr)
AES8: 1st pass
AES9: 2nd pass (+EP+120Cx48hr)
AES10: 1st pass
ILC Spec
Q0
10^{11}
10^{10}
10^{9}
0
10
20
30
40
50
Eacc [MV/m]

KEK researchers visited JLab

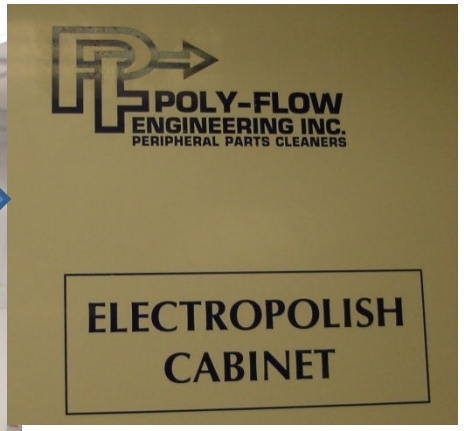
- 5 researchers visited JLab from KEK during 16 (Mon) – 19 (Thu) Nov. 2009 [4 days]

  Shigeki KATO (Surface analysis) ,Michiru NISHIWAKI (Surface analysis), Puneer Veer TYAGI (Surface analysis), Motoaki SAWABE (EP facility), Takayuki SAEKI (SRF / EP facility)

- 7 researchers, 1 post-doctor, 2 engineers, etc. joined the discussions about EP and other surface treatments.

  Rong-Li Geng(SRF), Bob Rimmer (SRF), Charlie Reece (SRF), Peter Kneisel (SRF), Larry Phillips (SRF), Andy Wu (Surface analysis, EP), Xin Zhao (Surface, post-doctor), H Tian (SRF), Byron Golden (EP engineer), Jim(EP engineer from FNAL), Tony Reilly (Head of facility operation/maintenance)

EP facility at JLab



Sleeve design
Nomura plating

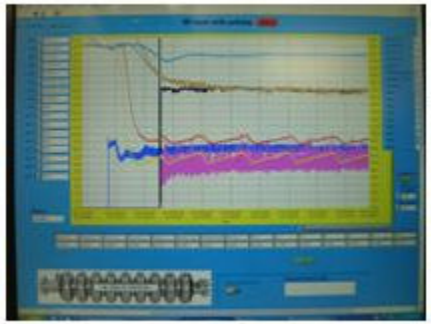
System design
J. Mammosser and Poly Flow Engineering

EP & Rinsing Process of 5-cell cavity

- **Nov.17,2009 Tuesday**

EP Room

EP process



Water rinse at vertical Position

Disassembly of setup and water washing/rinsing

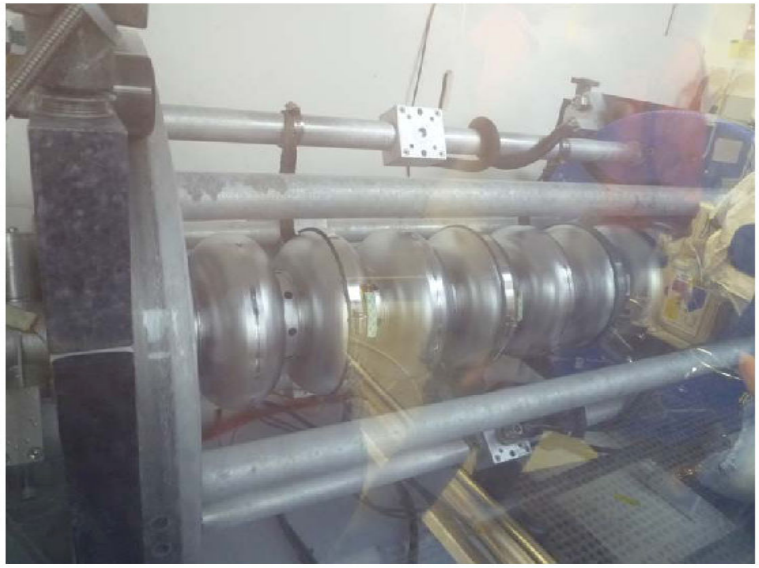
Water washing/rinsing

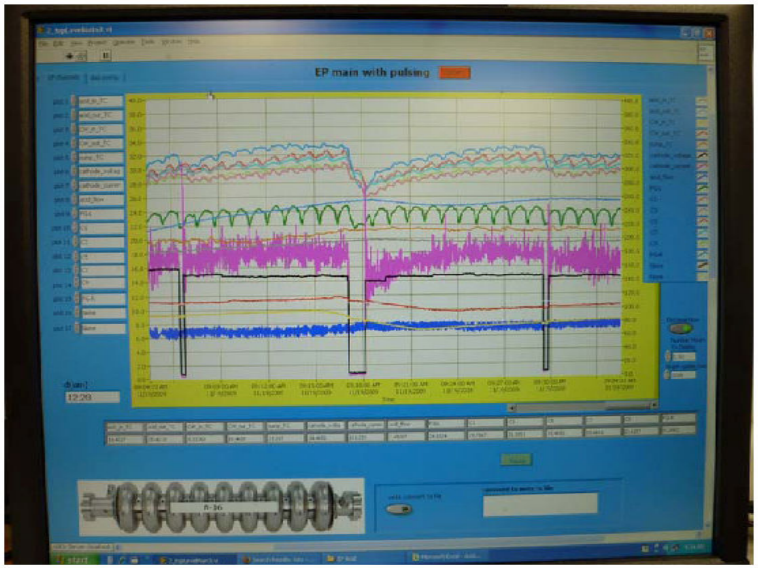
EP facility at JLab / RI-16 9-cell cavity

Fourth Day 09/11/19

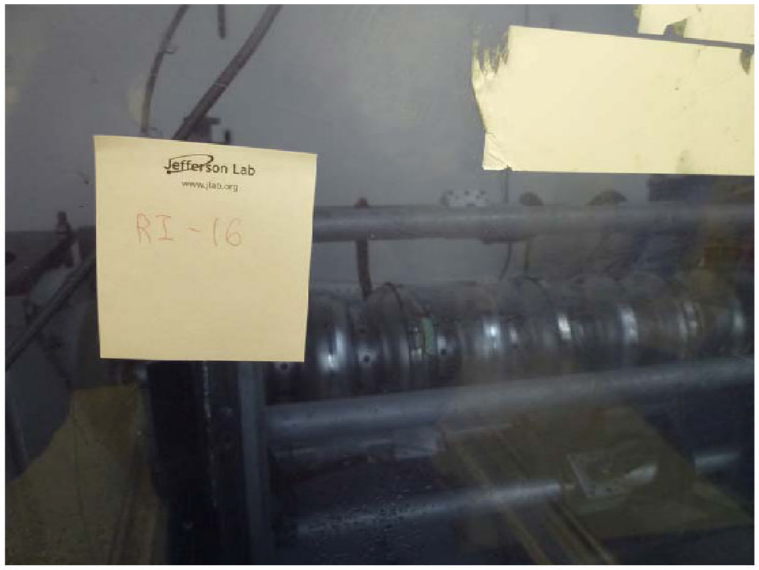
- We saw 9-cell Cavity EP follwed ultrasonic rinsing with liquinox and then cavity was moved for High Pressure Rinsing.

9-cell ILC cavity(RI-16) EP at J-Lab





Control monitor of EP process



Difference between Jlab and KEK

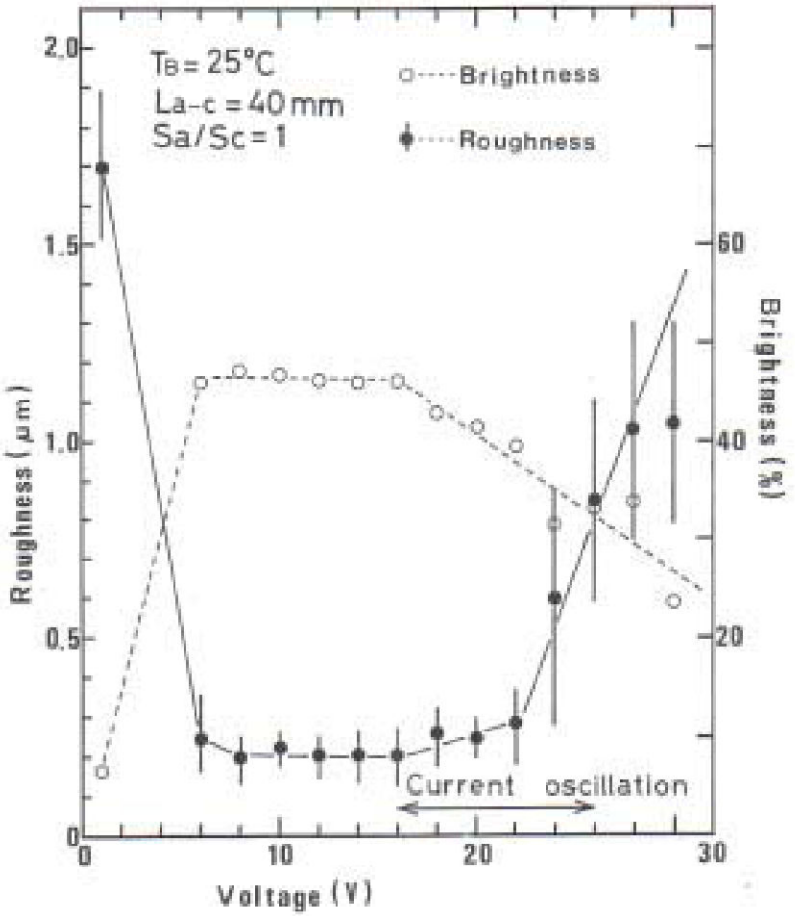
	JLab	KEK
EP Acid Tank	270L (Tank Capacity) 230L (EP acid volume)	2000L (Tank capacity) 1000L (EP acid volume)
EP Acid Flow Rate	5 ~ 10L/min. Over flow pipes 15mmϕ	10 ~ 20L/min. Over flow pipes 25mmϕ
EP and Water Rinsing Atmosphere	Nitrogen During EP: 12L/min. During Rinsing: 13 ~ 20L/min.	Air
EP Acid Temp.	16 ~ 26℃ EP acid (Return pipe) 21 ~ 34℃ Outside cavity	17 ~ 32℃ EP acid (Center of Cell) 28 ~ 50℃ Outside cavity
EP Voltage and Current	14 – 17 V Current density = 20 – 30 mA/cm2	19 ~ 21V Current density = 50 mA/cm2

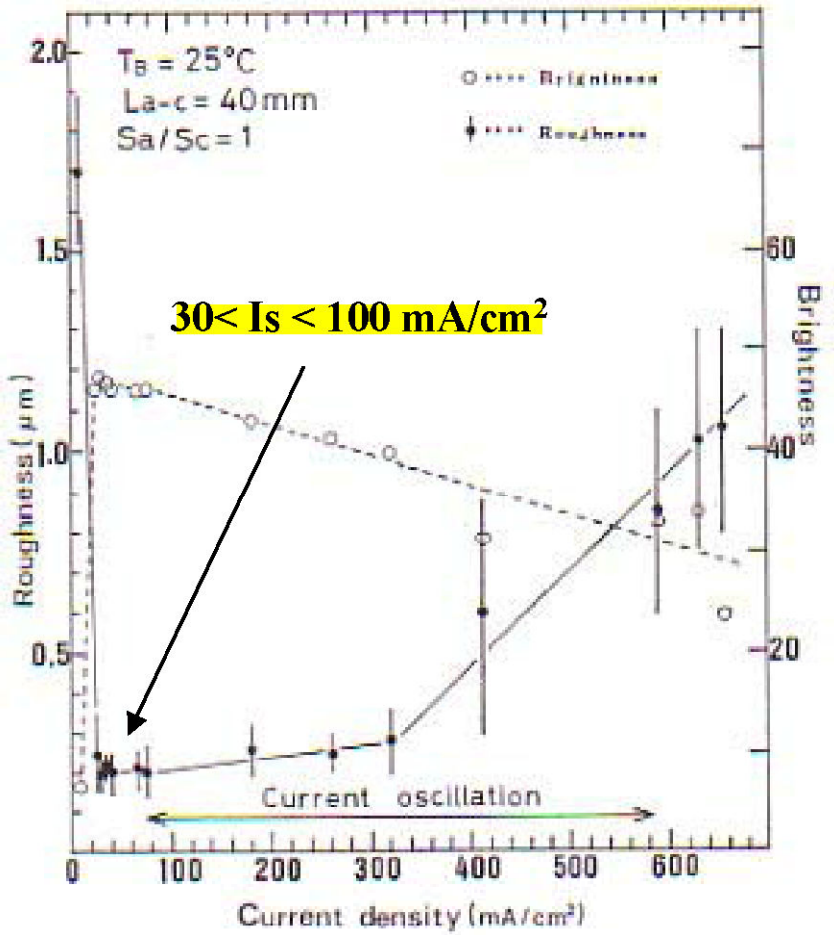
Related to stain problem?

Related to nice results of JLab?

What is the curtail EP parameter?

The KEK recipe by Kenji SAITO from TRISTAN age. Current density = 50 mA/cm2





We had been keeping the KEK recipe established by Kenji SAITO in TRISTAN age.
Current density = 50 mA/cm2

TEST OF LOW VOLTAGE EP (5V) AT SACLAY TO REDUCE SULFUR CONTAMINATION

= Theoritical way to reduce Sulfur Contamination (experience on going)

Reduction Reactions at the Cathode

Reduction of H^+: $2\ H^+ + 2\ e^- \rightarrow H_2$ (Predominant)

Reduction of SO_4^{2-}: $SO_4^{2-} + 8H^+ + 6e^- \rightarrow S + 4H_2O$

Does a cathodic overpotential lead to an increased sulfur generation?

→Two Niobium samples electro-polished at 5V and 20V for a similar heavy removal with 1-9-1 (HF-H_2SO_4-H_2O) Mixture

Sample A, 20 Volts:

9.18g removed

51 hours EP

Sample B, 5 Volts:

9.11g removed

115 hours EP

Experiment at Saclay

TEST OF LOW VOLTAGE EP (5V) TO REDUCE SULFUR CONTAMINATION

SULFUR FOUND IN THE EP MIXTURE (200mL) AFTER TREATMENTS OF SAMPLES A AND B.

SULFUR EXTRACTED WITH CHLOROFORM:

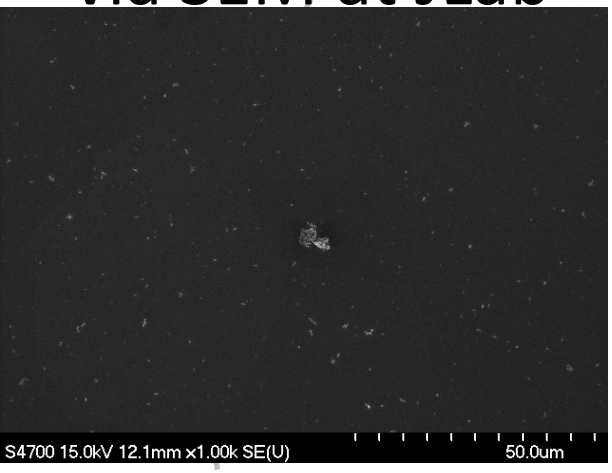
Experiment at Saclay

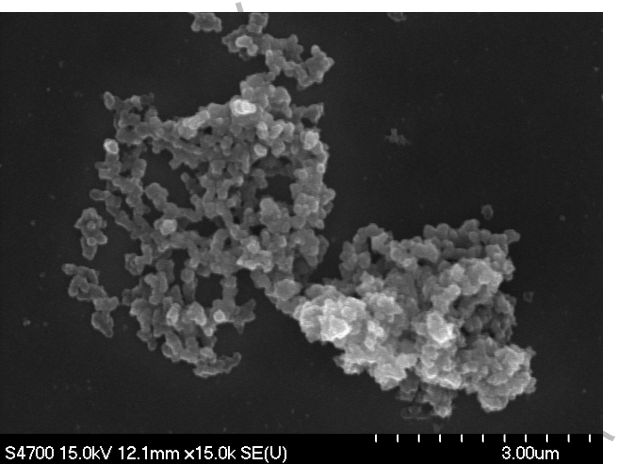
Sample	A (20 V)	B (5V)
1st Rinse	4.5mg	2.6-2.7mg
2d Rinse	1.2-1.3mg	0.9-1.2mg

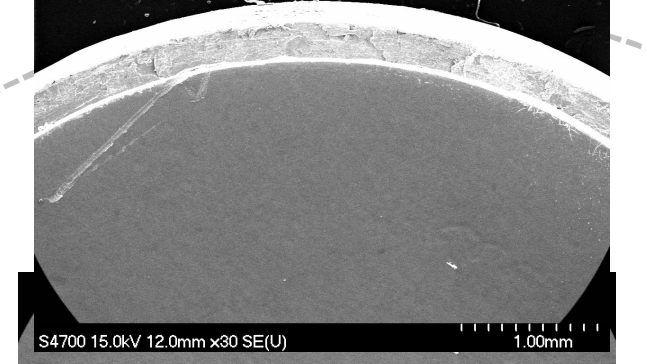
Difference between Jlab and KEK

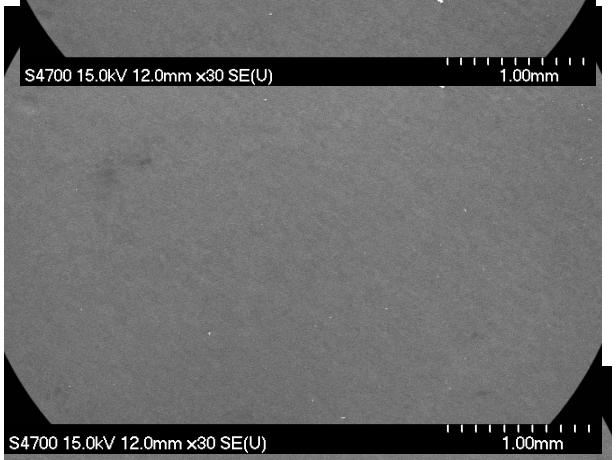
	JLab	KEK
Rotation after EP (HF rinse duration: V=0)	1 r.p.m 30min.	3 r.p.m 5min. Related to less field emission?
Flow rate of water rinsing	DI water/U.P.W., 27L/min.	U.P. W.,13 L/min.
Rinsing Flow Route	Through the cathode pipe (from lower)	Through the cathode pipe (from upper)
Rinsing Time	60min.	90min.
Number of Fill & Dumps	7	-

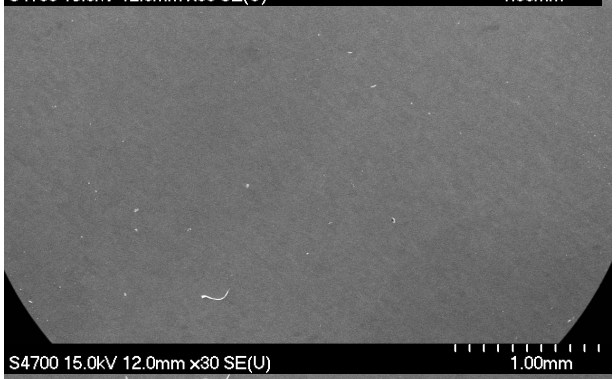
“micro-granules” were found on EP’ed Nb sample via SEM at JLab

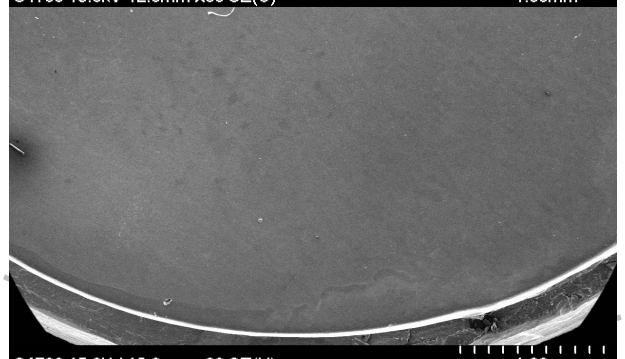


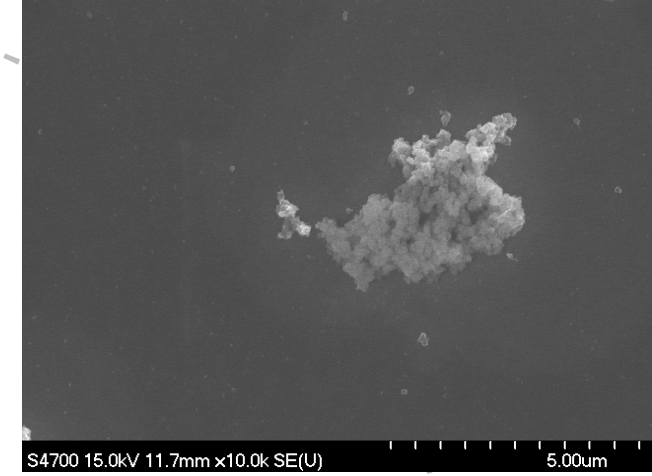


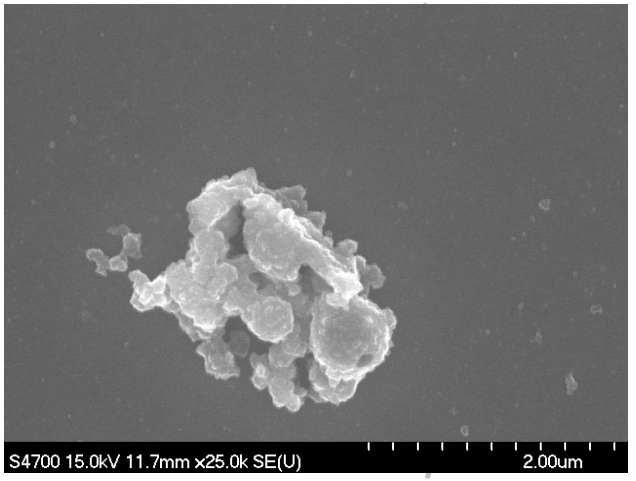












Micro-granules mostly consist only of Nb and Oxigen. This is one of candidates for field emission.

Sample #4

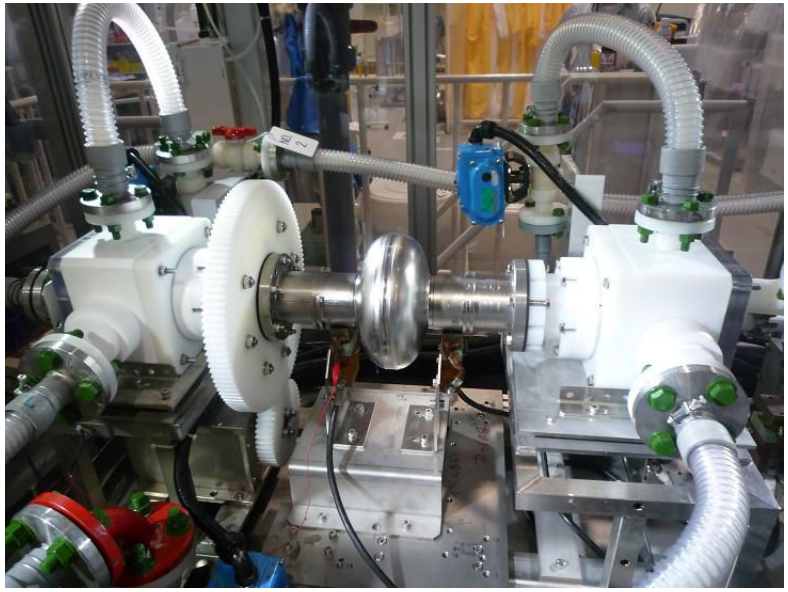
Difference between Jlab and KEK

	JLab	KEK
End criteria of Water Rinsing	By cell temperature	By water conductivity
Replacement criteria of EP Acid (g-Nb/L)	9Cell 20μm×2 (308g) and 9Cell >100μm(>770g) Total >1078g(≒>4.7 g/L)	8 ~ 10 g/L
Cleaning of EP Acid Tank	2 times/year By Micro 90	1 time/year By Ultra Pure Water
Detergent rinse with Ultra Sonic	2% Liqui-Nox at 60 ^{0}C. 1Hr. Inside and outside at once	2% FM-20 at 50℃ 1Hr. Inside only
After Detergent Rinse	U.P.W. rinse by hand	U.P.W. Rinse with Ultrasonic at 50℃ 1Hr.

EP by low current density at STF/KEK

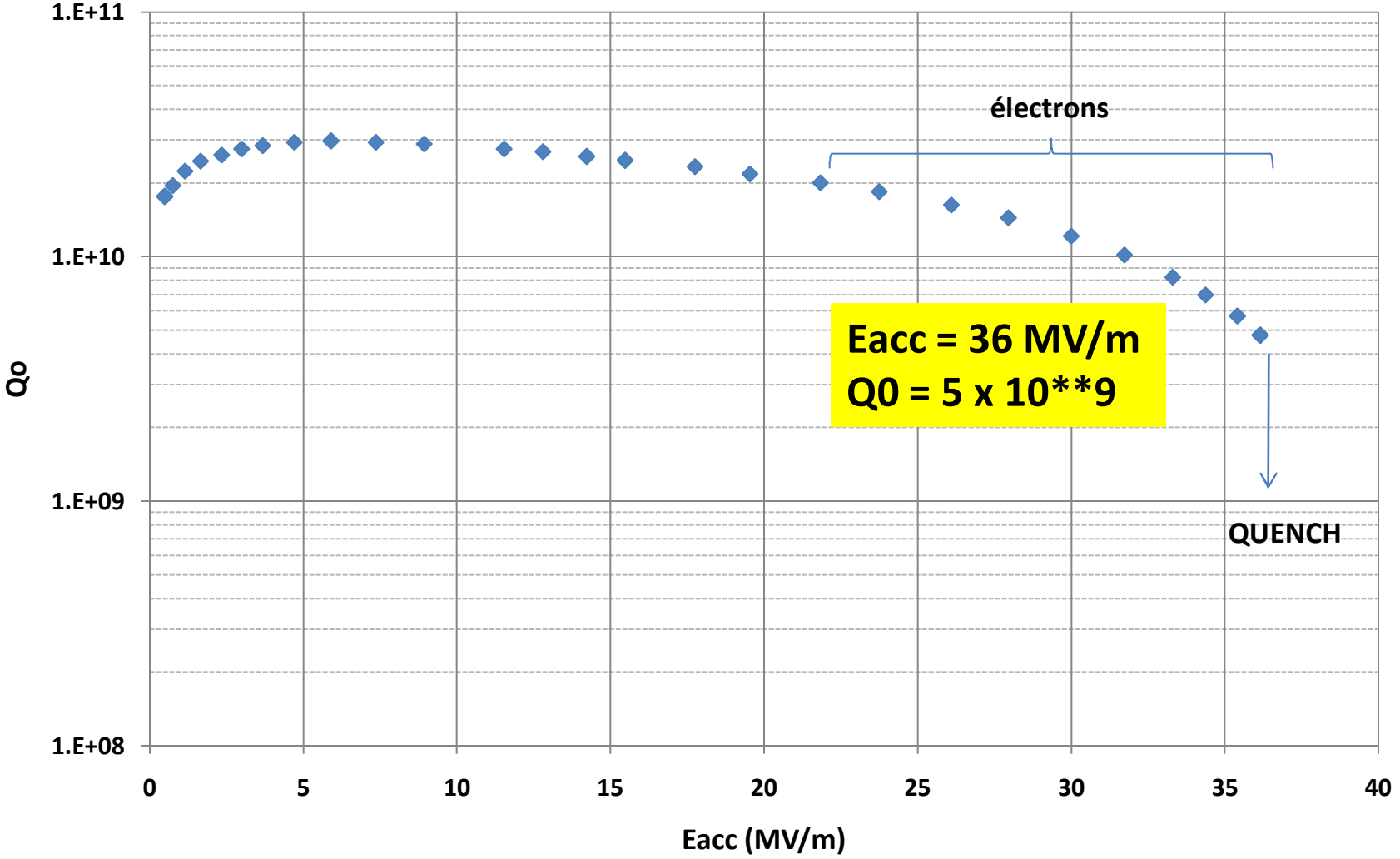
Usually 50mA/cm2

Date 2010/Jan/08
Cavity Name: 1DE1
Removal 50µm
Current density : 30mA/cm2 (1400 cm2×0.6×0.03 A/cm2=25.2 A)
EP acid rinse (3 r.p.m., 20 min.)
U.P.W. rinse 60.min.(Supply 3min. Drain 1min.)
2% FM-20 Rinse 60min. (at 50℃)
Ultra Sonic Rinse 30min. (at 50℃)
H.P.R 1Hr.



VT at CEA/Saclay
On 18 Feb 2010

D1 DE1 I4 T = 1.7 K



Local grinding at iris part + EP(50 um, Id=30mA/cm2) + HPR

Series EP's by low current density

- ERL injector 2-cell cavity #1:

  final EP(20 um) with 40 mA/cm2, with air flow

  on 28 Jan. 2010.

  VT result: 43.7 MV/m

  details → **Presentation by E. Kako / Presentation by K. Watanabe in 21st ILC S0 WebEx cavity meeting**

- 9-cell MHI-#8:

  final EP(20 um) with 30 mA/cm2, with N2 flow

  on 9 Feb 2010

  VT result: 37.8 MV/m

  details → **Presentation by E. Kako / Presentation by Y. Yamamoto in 21st ILC S0 WebEx cavity meeting**

- ERL injector 2-cell cavity #2:

  final EP(20 um) with 30 mA/cm2, with N2 flow

  on 22 Feb 2010.

  VT result: 40.9 MV/m

  details → **Presentation by E. Kako / Presentation by K. Watanabe in 21st ILC S0 WebEx cavity meeting**

TESLA Technology Collaboration
TTC-Report 2008-05

Final Surface Preparation for Superconducting Cavities

An attempt to describe an optimized procedure

Reply to the

Request for Consultancy from TTC

raised by

the ILC R&D Board Task Force on High Gradients (S0/S1)

Comparison of EP parameters between Jlab and KEK looks very successfull.

TTC-Report 2008-05 :
Detailed comparison of surface processes among laboratories.
But this is going to be obsolete in some part.

Natural question:
Should we update the comparison table step by step?

Camille Ginzburg proposed me to visit ANL during the TTC meeting (FNAL, April 2010) to compare the EP parameters among KEK, Jlab and ANL.
I agreed with this proposal.

Summary

- We had stain problem at STF/KEK in summer 2009. We heard DESY and Jlab have no stain problem.
- Jlab produced very nice VT results in 2009.
- KEK researchers visited Jlab in Nov. 2009 to compare the EP parameters between KEK and Jlab.
- We found some differences of EP parameters between Jlab and KEK. Current density: JLab(20 – 30 mA/cm2) vs. KEK (50 mA/cm2). Atmosphere: Jlab(N2 flow) vs. KEK(Air flow), ...and so on.
- We performed series of EP with low current density at STF/KEK. All of these cavities produced very nice VT results (Eacc > 35 MV/m).
- EP parameter comparison looks very successful. Should we update TTC report 2008-05?
- Plan: C. Ginzburg and T.Saeki will visit ANL to compare the EP parameters among ANL, Jlab, and KEK.