

# 超伝導空洞開発 High-Qの実現と窒素ドーピング

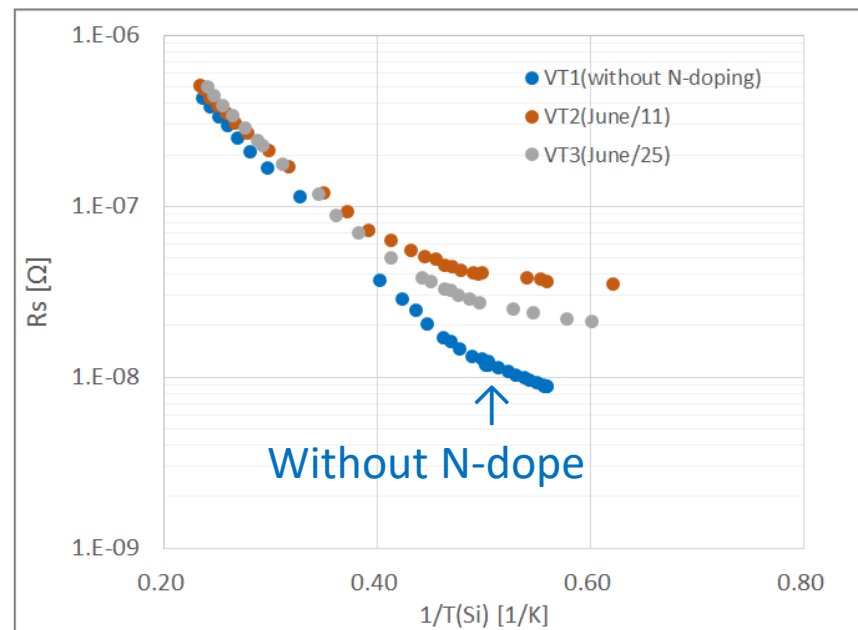
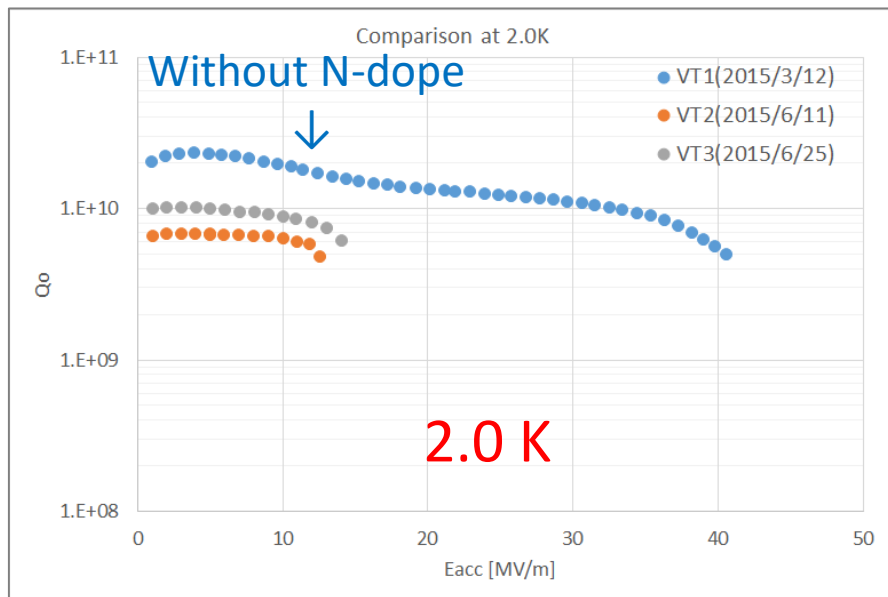
2017年6月8日

ERL検討会

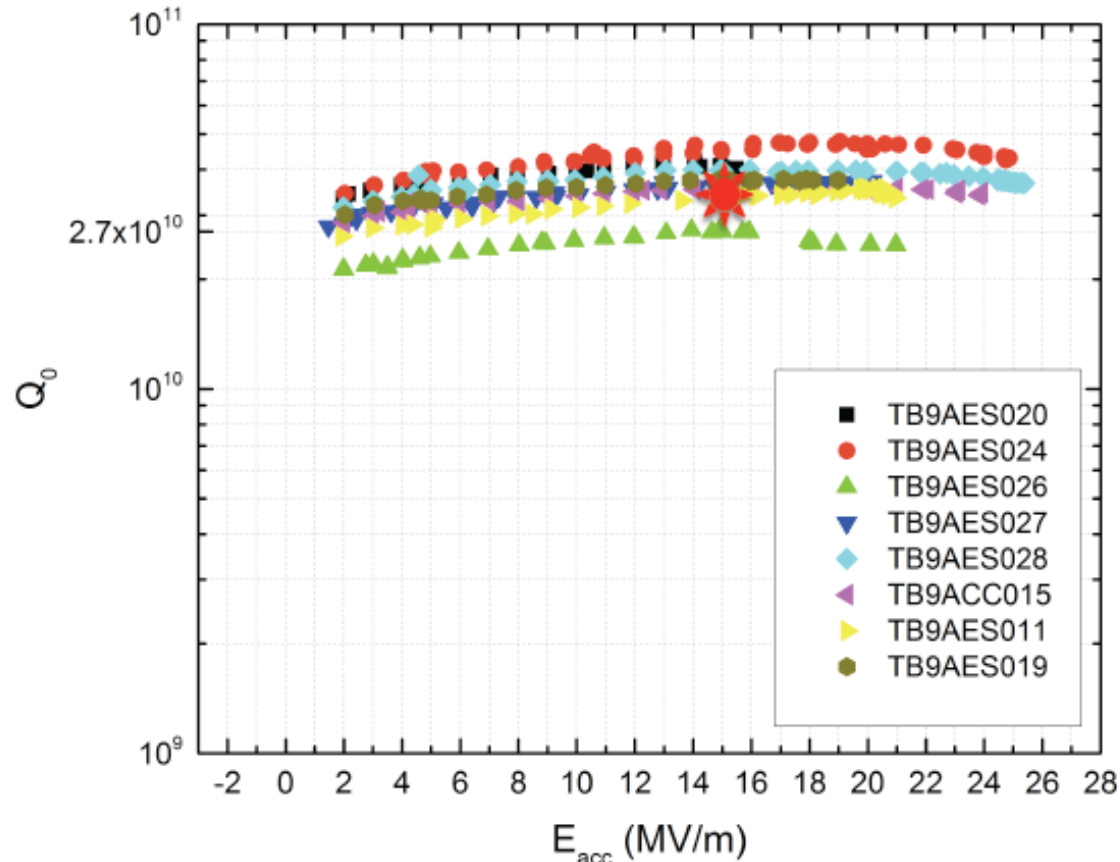
梅森 健成 (SCRFグループ)

# はじめに

- LCLS-IIをはじめ、high-Qでの超伝導空洞の運転にむけた計画が、世界各国で行われている。
- KEKにおいても、ここ2～3年研究を進めている。
- 以前、2015年9月のERL検討会にて「High-Qを目指した窒素ドーピングの試み」として、KEKでの窒素ドーピングはうまくいっていない旨の報告。



## One cryomodule milestone – avg Q (2K, 16 MV/m)~3.75e10, avg quench field ~22 MV/m



**Best N doping recipe so far (high Q and highest gradients achieved on nine cells) is the one known as “Fermilab 2/6”: 800C 3 hrs in HV, then 2 min @ 800C with ~ 25 mTorr N2, then 6 min @ 800C in HV + 5 microns EP**

# Why N-doping does not work?

Possible reason of bad results are followings.

## 1. Nb surface was not N-doped correctly.

真空炉??

- Something wrong?
- Difference of vacuum system? (Cryopump or diffusion pump, oil-free?)
- Difference on N-doping system?

## 2. Effect due to remnant field on vertical test cryostat.

- Trapping of magnetic field on N-doped surface is more sensitive to remnant field on vertical test cryostat.(More than a few ~ several times sensitive?)
- KEK's VT cryostat has more than 10 mG.
- Also depend on cooling procedure.

縦測定での磁場環境??

## 3. Cavity or material is wrong?

- Cavity is made at KEK-CFF.
- Nb supplier is ULVAC and Tokyo-Denkai.

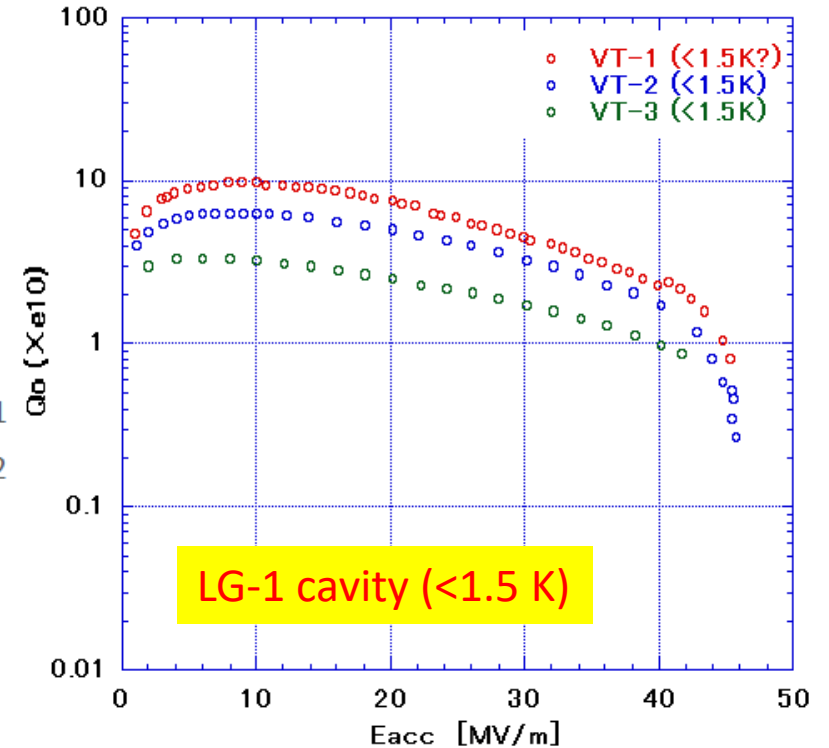
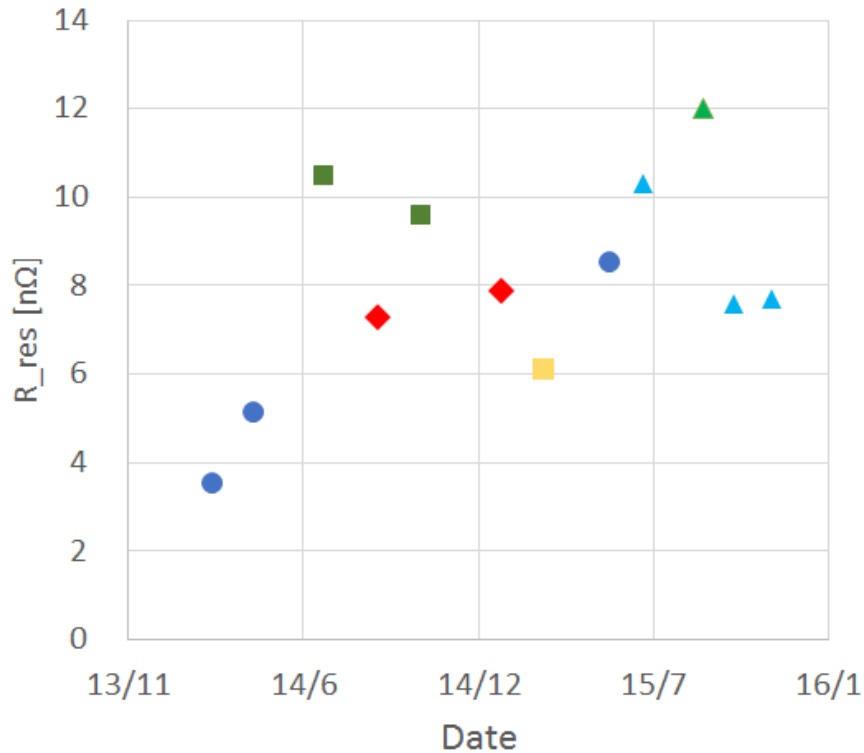
材料または製法??

# 今回の報告

1. High-Qの測定に向けたKEK-STFの縦測定クライオスタット磁場環境の改善
2. FNALおよびJ-PARCとの協力のもとでの窒素ドーピングの結果

High-Q study

# Rres history of single-cell cavity vertical tests



- $R_{res}$  gradually increase?
- Q-values of LG cavity were gradually decrease.

Degradation of R\_res?



Check magnetization for most of all  
the components of vertical test



# Study on magnetized components (example)

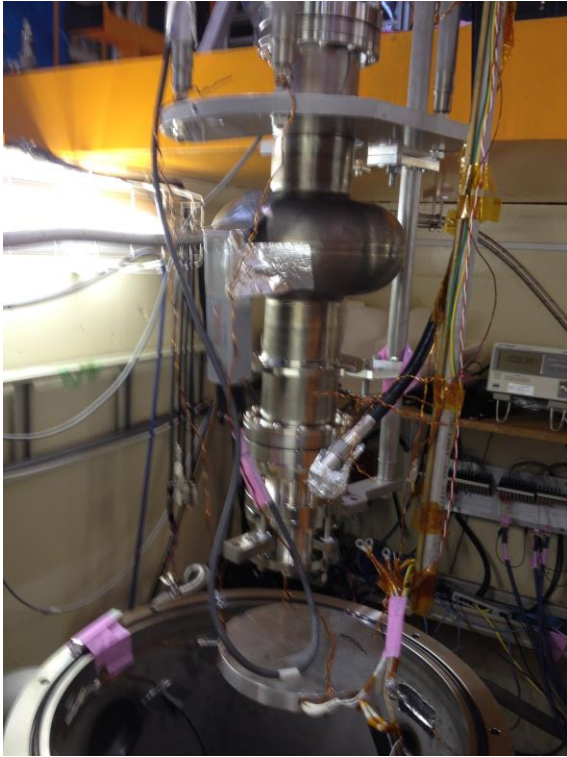
From T. Yanagimachi(9/26)

No.	name	Magnetic field [mG]
14	Φ034 metal valve ①	430
15	Φ034 metal valve (which observed vacuum leak)	80
19	Φ034 metal valve ②	59
25	Volts and washers for support of input coupler shaft	140
28	Nuts and washers for hanging cavity	110
29	Stat-volts, nuts and washers for hanging cavity	300



Measure inside magnetic shield by using 3-axis flux gate sensor.

# Effects of SUS shafts



SUS shafts for variable coupler were highly magnetized.  
More than 1 G!!

Magnetic field with shafts inside vertical test dewar

Angle	Bx [mG]	By[mG]	Bz[mG]	B[mG]
0	-7	-11	-6	15
90	-6	2	-9	11
180	6	-11	-7	15
270	8	130	-49	139

If both shafts were removed  
 $B \leq 2\text{mG}$  for any positions.

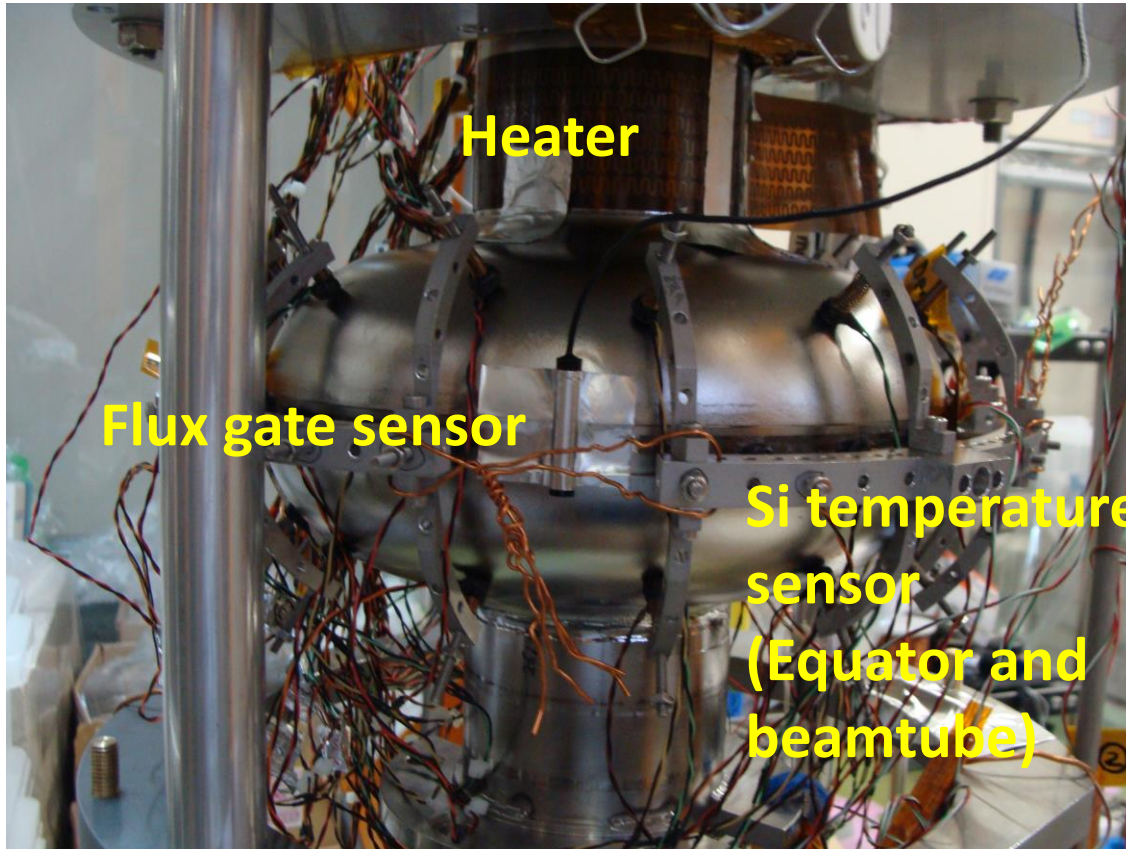
Then...

- ◆ Exchange SUS shafts to Ti
- ◆ Exchange or remove SUS components as much as possible
- ◆ Exchange metal valve to less magnetized one

And vertical tests were carried out.

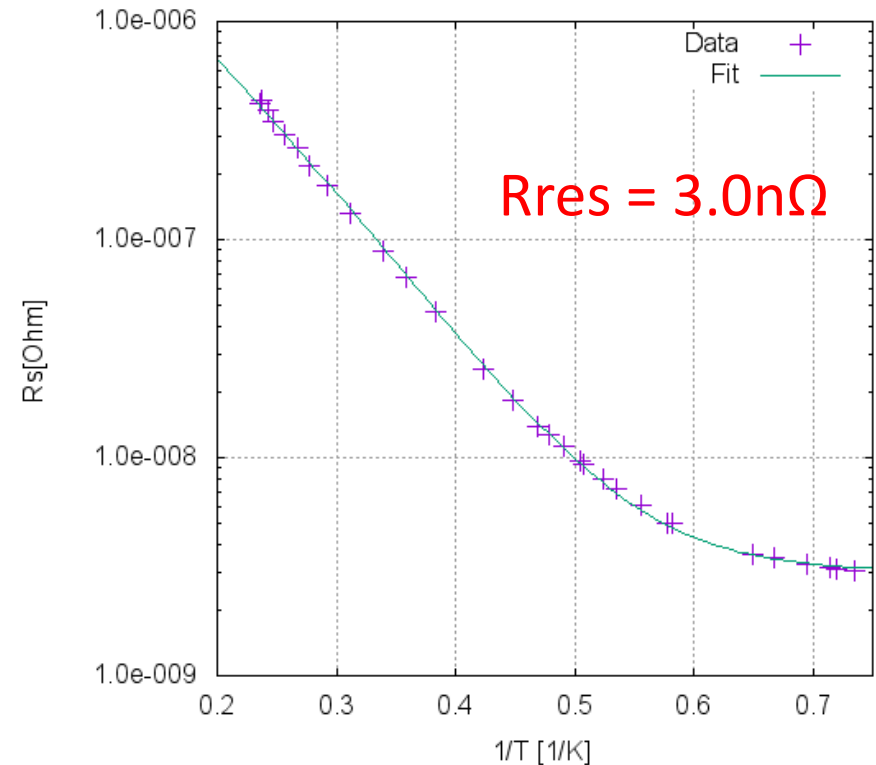
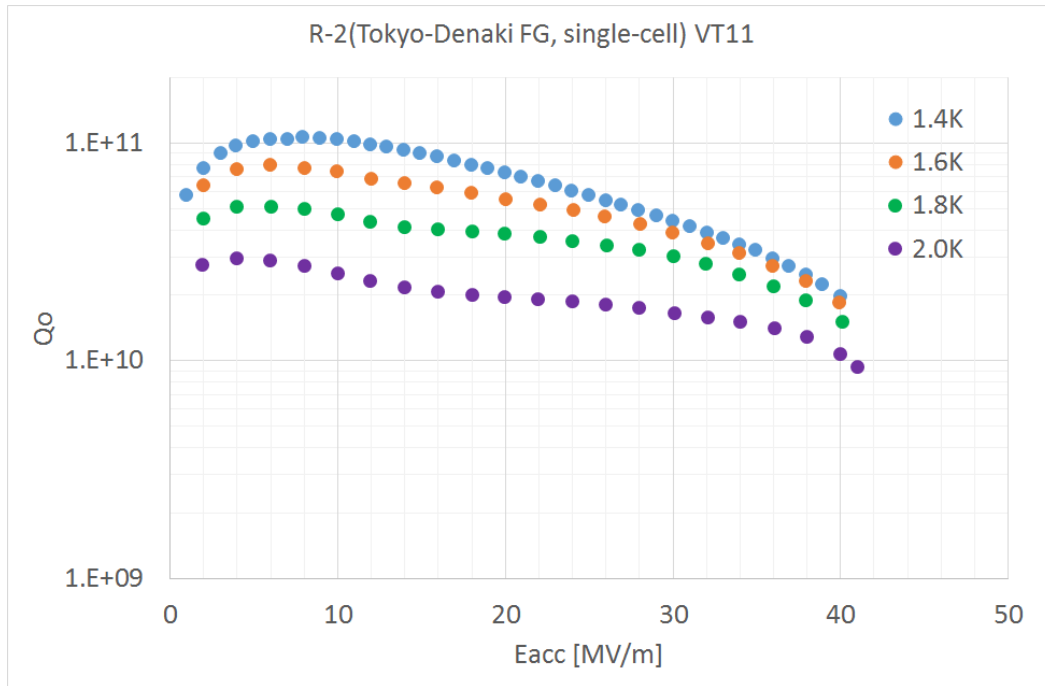
# Typical vertical test setup

- ❌ Pictures are for different measurement.
- ❌ But setup of sensors and coil are same.



Flux gate sensor, Si temperature sensor, heater and solenoid coil were used.

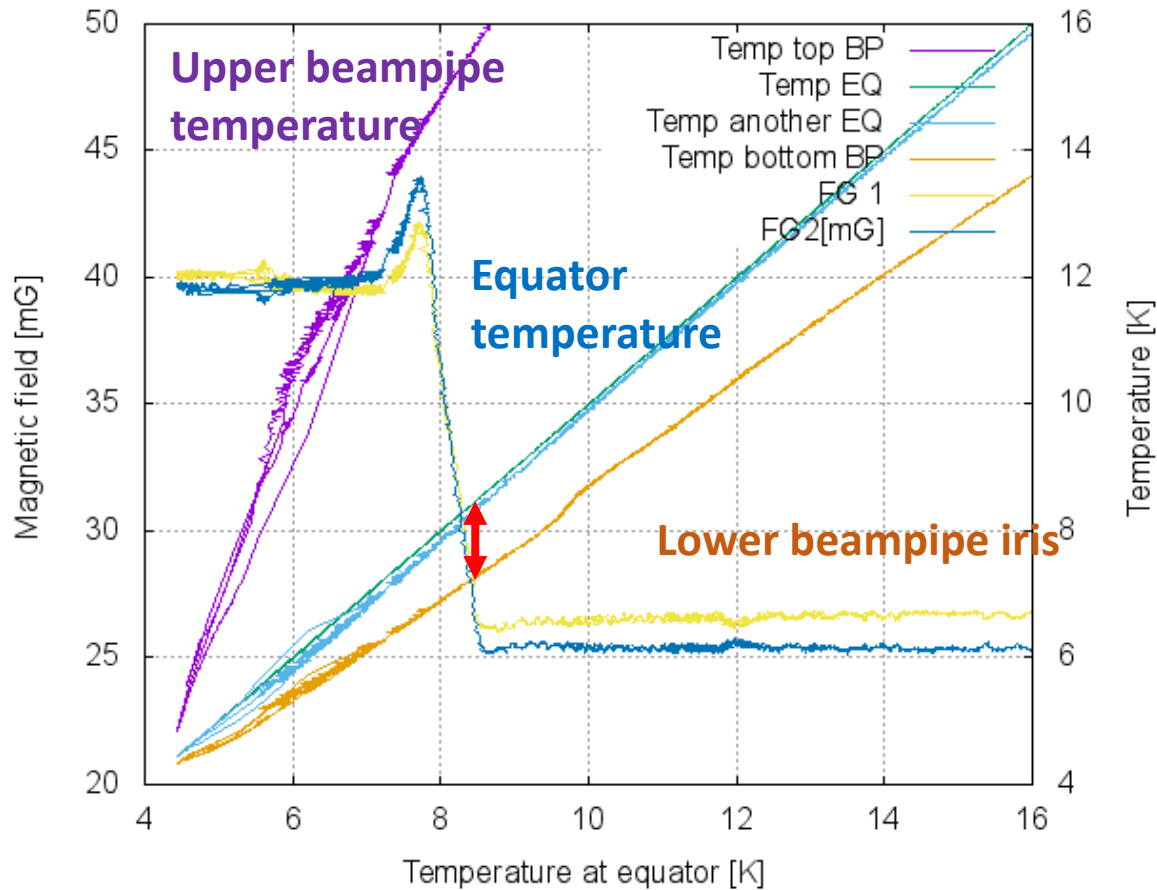
# Vertical test results after demagnetization effort



- FG single-cell cavity (Tokyo-Denkai)
- Nominal recipe (Not N-doping)
- With cancelling coil
- With thermal gradient by heater

Very high-Q was observed after the effort for demagnetization

# Flux expulsion during cool-down(add 16mG with coil)



- FG single-cell cavity (ULVAC)
- Nominal recipe (Not N-doping)
- 900 degree heat treatment applied
- Add +16mG with coil (Total 9 + 16 = 25mG)
- With thermal gradient by heater

- Clear flux expulsion (~90%) can be observed.
- Temperature gradient of more than 1 degree between equator and lower beampipe iris.

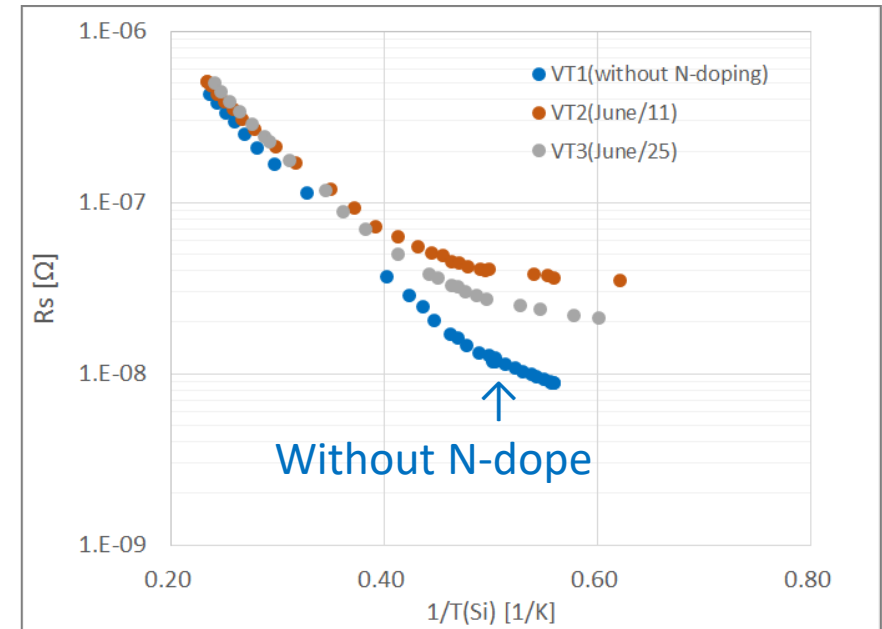
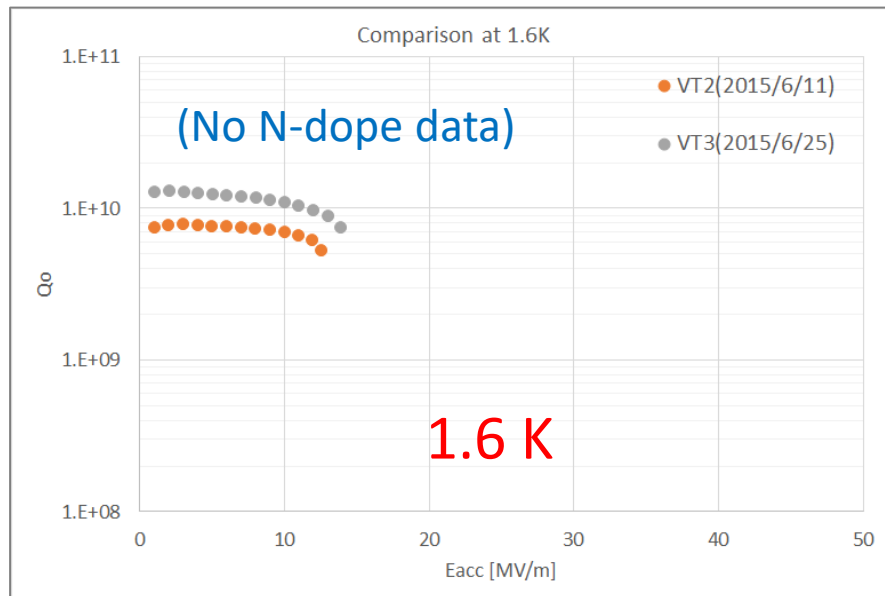
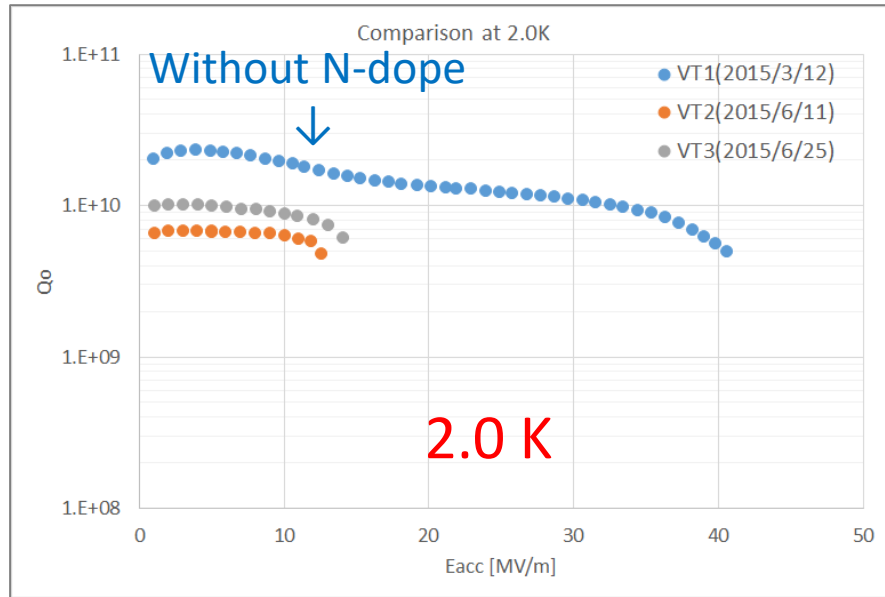
## Summary (High-Q study)

- It was difficult to achieve high-Q at KEK-STF vertical test.
- Magnetization was investigated for each components of vertical tests.
- Some components were highly magnetized. One of highest was shaft for variable coupler.
- Magnetized components were removed or exchanged. Also solenoid coil was prepared.
- After these effort, high-Q could be measured and clear flux expulsion signal was observed.

# Nitrogen doping



## 縦測定結果



- ・ 窒素ドーピングの後、15 $\mu$ m + 15 $\mu$ mのEP-2をして2回の縦測定を行った。
- ・ 今回は更にひどくQ値が劣化。1 $\times 10^{10}$ に届くか届かないか。
- ・ Quench fieldは13MV/m。
- ・ 2回目のEPで、Q値、quench fieldともに少しだけ回復。
- ・ Quenchの場所は、今回はほぼ同じ。

# Why N-doping does not work?

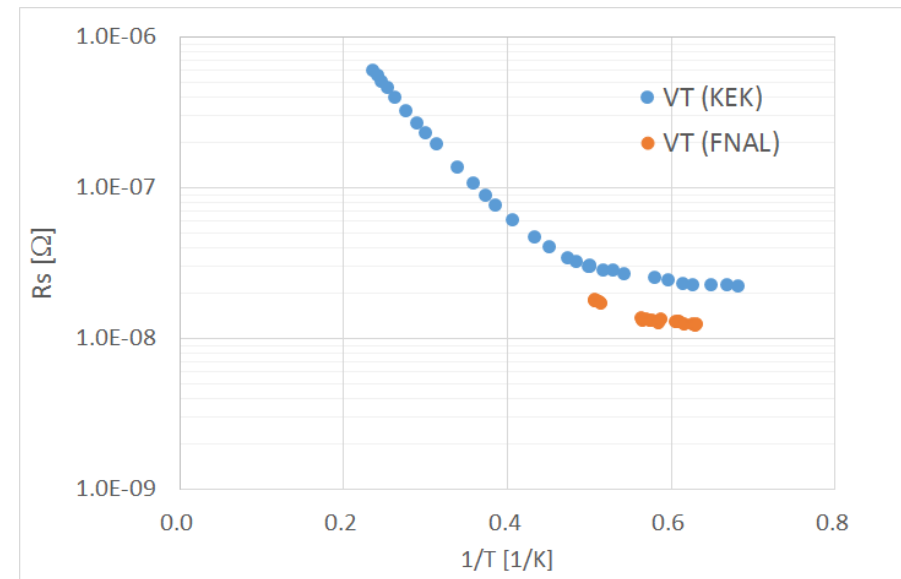
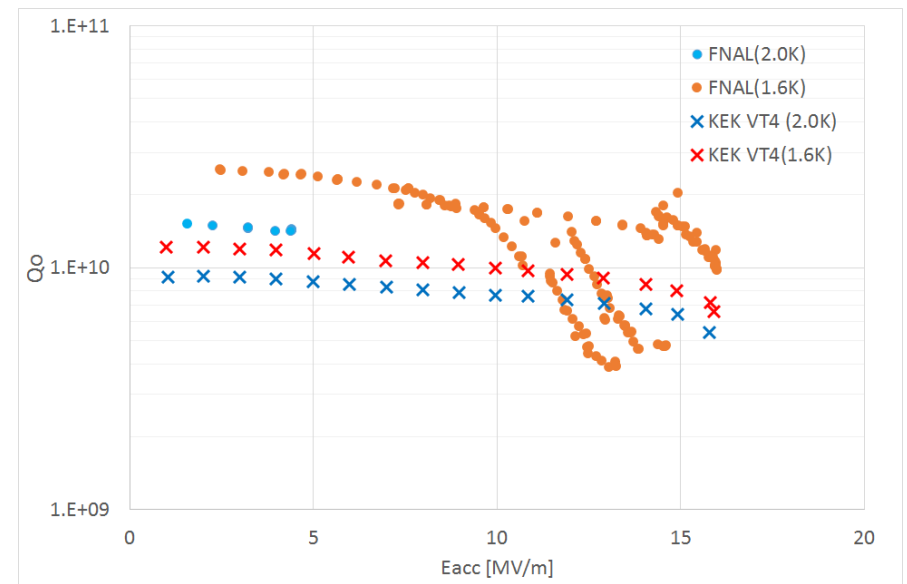
Possible reason of bad results are followings.

1. Nb surface was not N-doped correctly. ⇒ N-dope@FNAL and J-PARC
  - Something wrong? 真空炉??
  - Difference of vacuum system? (Cryopump or diffusion pump, oil-free?)
  - Difference on N-doping system?
2. Effect due to remnant field on vertical test cryostat. ⇒ VT@FNAL(March)
  - Trapping of magnetic field on N-doped surface is more sensitive to remnant field on vertical test cryostat.(More than a few ~ several times sensitive?)
  - KEK's VT cryostat has more than 10 mG. 縦測定での磁場環境??
  - Also depend on cooling procedure.
3. Cavity or material is wrong? ⇒ N-dope@FNAL and VT@FNAL(October)
  - Cavity is made at KEK-CFF. 材料または製法??
  - Nb supplier is ULVAC and Tokyo-Denkai.

# Vertical test of KEK doped cavity at FNAL



- Vertical test of KEK N-doped cavity was carried out at FNAL, where magnetic field inside VT dewar is very small.
- However, Q-value was not good as nominal N-doping cavity.

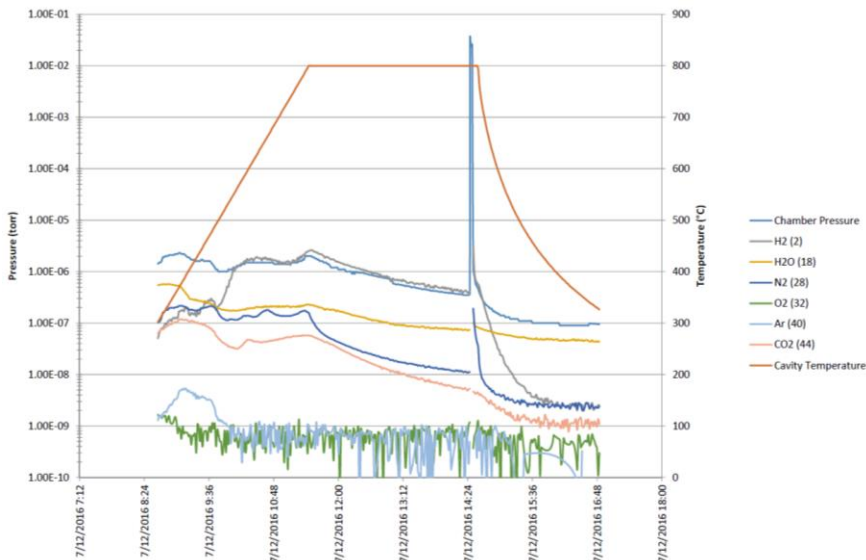
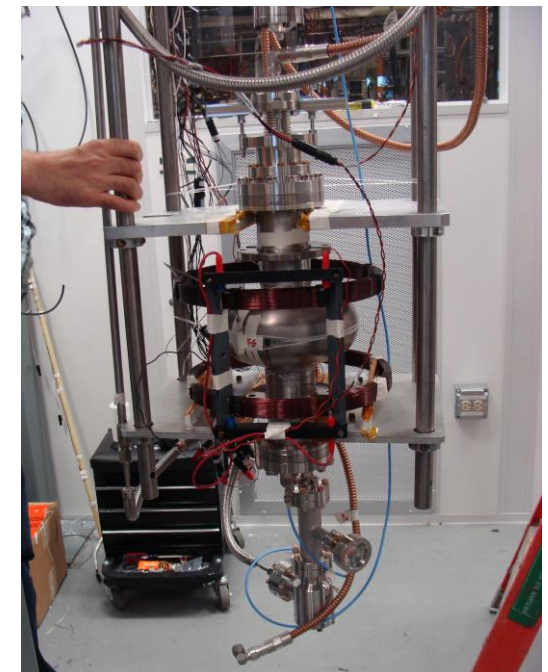


Even in zero magnetic field, still  $R_{res}$  was too large.

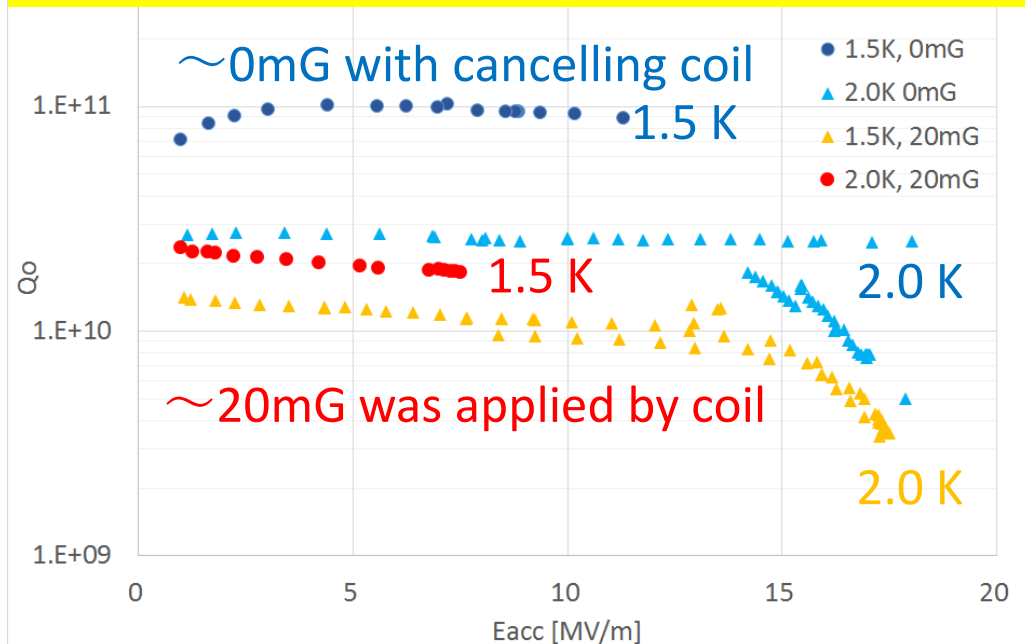
# N-dope and VT of KEK cavity @ FNAL

- 2016/7/9 EP 60um
- 2016/7/12 N-doping (FNAL standard recipe 2/6)
- 2016/9/13 EP 6um
- 2016/10/25, 26 VT

- KEK cavity was doped at FNAL and also tested.
- It showed successful doped performance.



N-doping successful !! Thanks for FNAL-SRF group!!



# Why N-doping does not work?

Possible reason of bad results are followings.

1. Nb surface was not N-doped correctly. ⇒ N-dope@FNAL and J-PARC

➤ Something wrong?

真空炉??

➤ Difference of vacuum system? (Cryopump or diffusion pump, oil-free?)

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~~縦測定での磁場環境??~~

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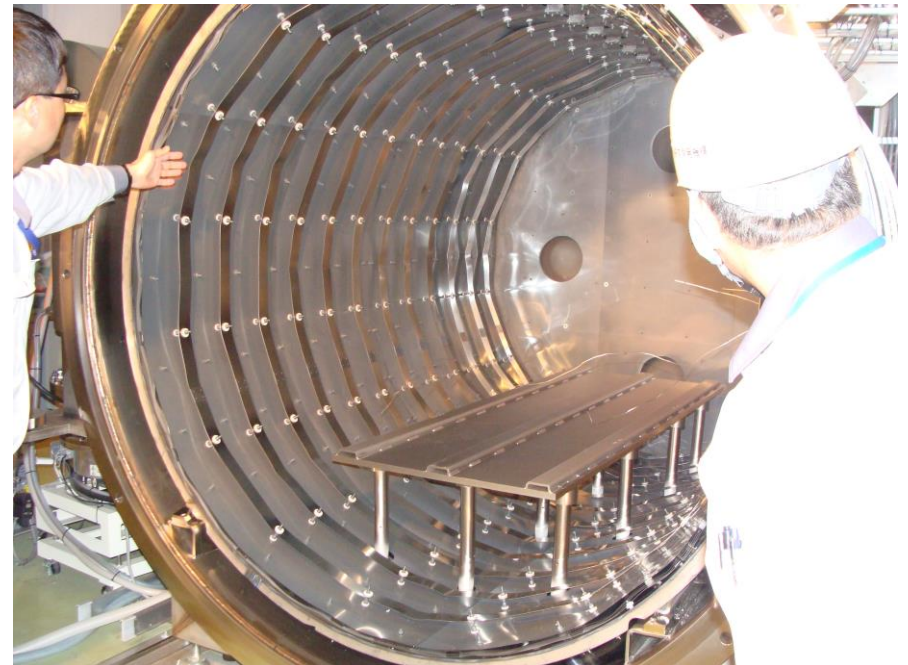
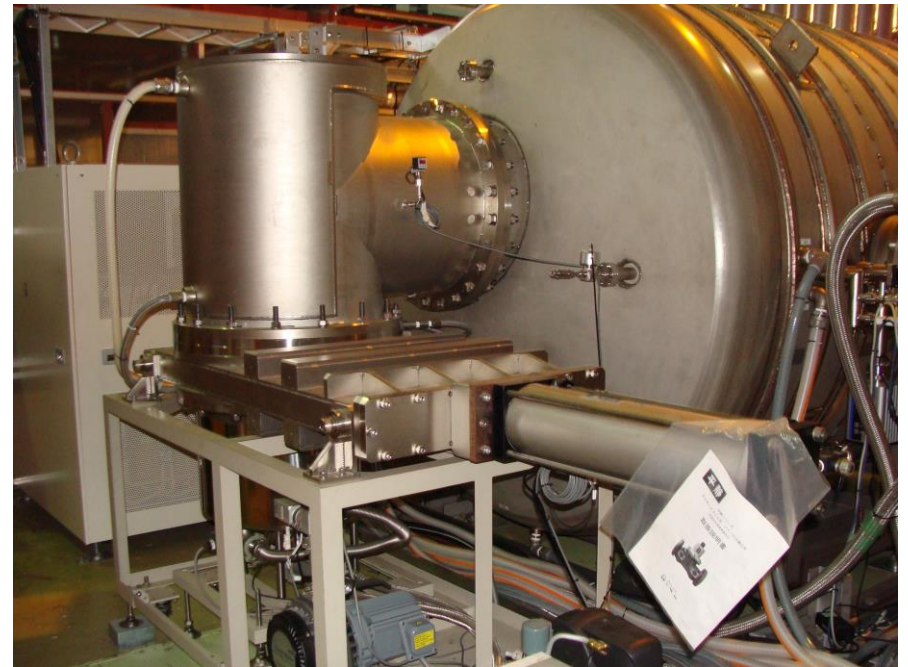
~~材料または製法??~~

~~➤ Nb supplier is ULVAC and Tokyo-Denkai.~~

# Furnace at J-PARC



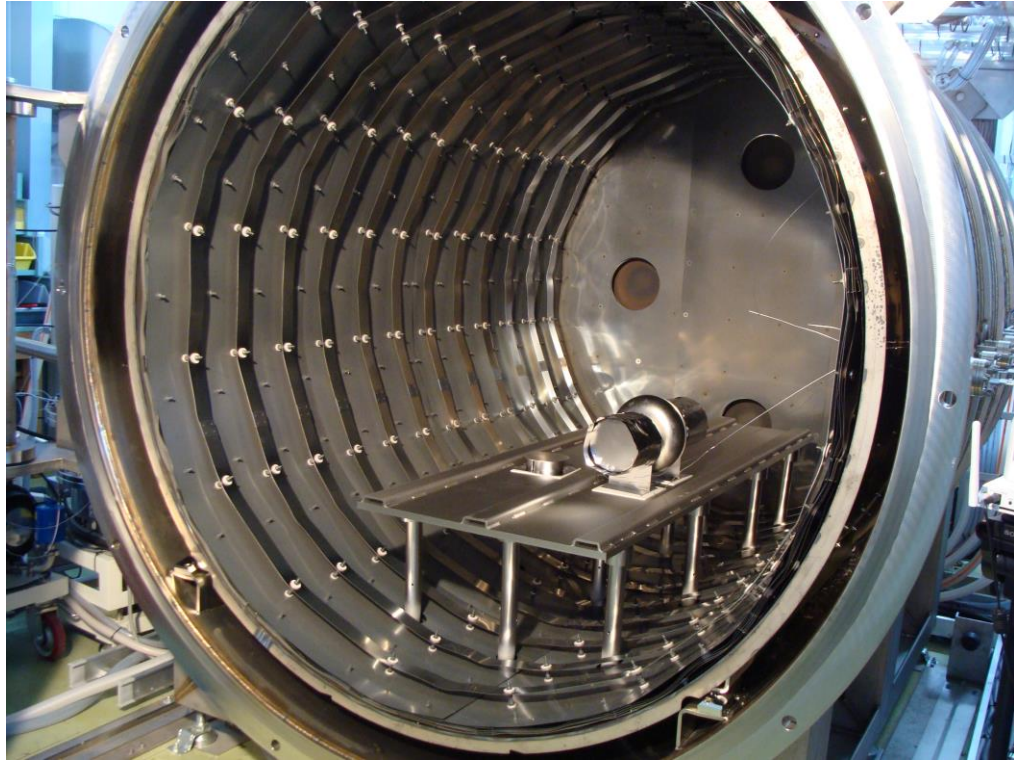
- J-PARC has oil-free furnace with cryo-pump and TMP.
- We try to use it for N-doping / N-infusion.
- Normally used for degassing of beam-ducts and components.



# N-doping@J-PARC furnace

N-doping at 2017/May/11

800 deg, 3 hours  
800 deg, 20 min, N 2.7Pa  
800 deg, 30 min  
Cooldown



Pumping system of J-PARC furnace

- Cryopump + TMP (+ scroll)
- reach to 1e-6 Pa after cool-down

# VT results of N-doping

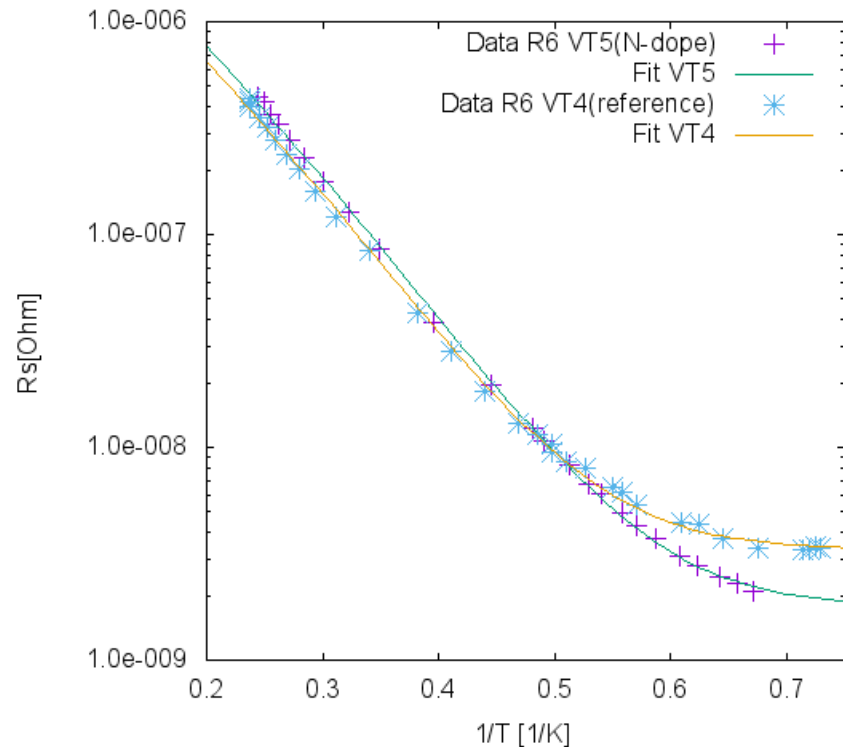
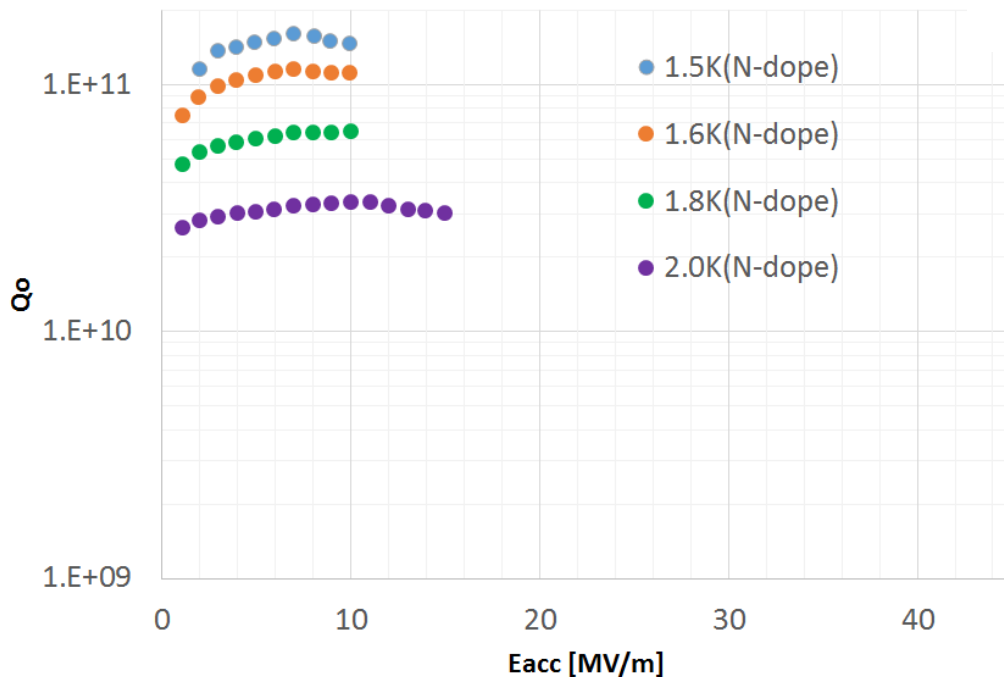
15um EP

HPR (total) 3~4hours

Dry assembly (No baking)

- Magnetic field canceled with solenoid coil. (< 1mG)
- Cooled down with thermal gradient by heater

R-6 cavity : Nitrogen-doping 20min, 2.7Pa/30min -- 15um EP



VT4(reference)  $R_s=3.3n\Omega$

VT5(N-dope)  $R_s=1.8n\Omega$

□ Very high Q up to high field

□  $Q = 1.6e11 @ 7MV/m, 1.47K$

□  $Q = 3.0e10 @ 15MV/m, 2K$

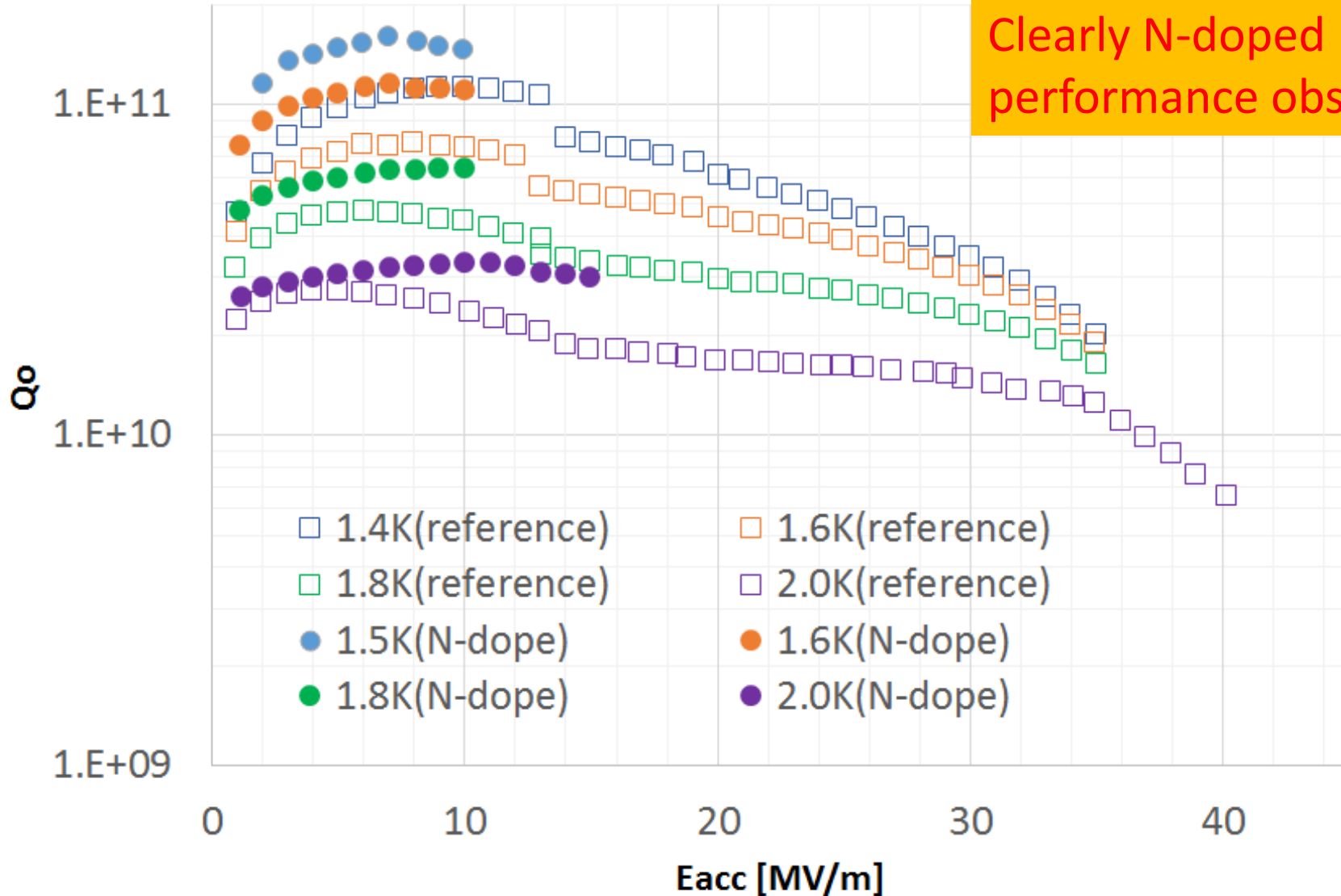
□ Quench at 16MV/m

□ No field emission



# Comparison with previous measurements

R-6(Mirapro, single-cell) VT4:reference / VT5:N-dope



## Summary (N-dope)

- In KEK, study on N-doping have been continued under collaboration with FNAL and J-PARC.
- N-doping was tried using J-PARC furnace, which has oil-free pumping system, consists of a cryopump and TMPs.
- After removable of 15um by EP, vertical test showed good performance:  $1.6e11$  at 7MV/m(@1.47K) and  $3.0e10$  at 15MV/m(@2.0K).
- This is first successful N-doped results in Japan.
- Clean furnace seems to be essential for N-doping. Details of reason is under investigation.

# 物構研・加速器合同セミナーのお知らせ

日時：7月4日（火）15時から16時30分

場所：4号館1階セミナーホール

講師：許斐太郎氏（加速器第六研究系 助教）

題目：**超伝導RF加速空洞のHigh-Q, High-Gの最近の動向とKEKでの展開**

(Recent world wide research and KEK activities of the Superconducting RF cavity for High-Q, High-G)

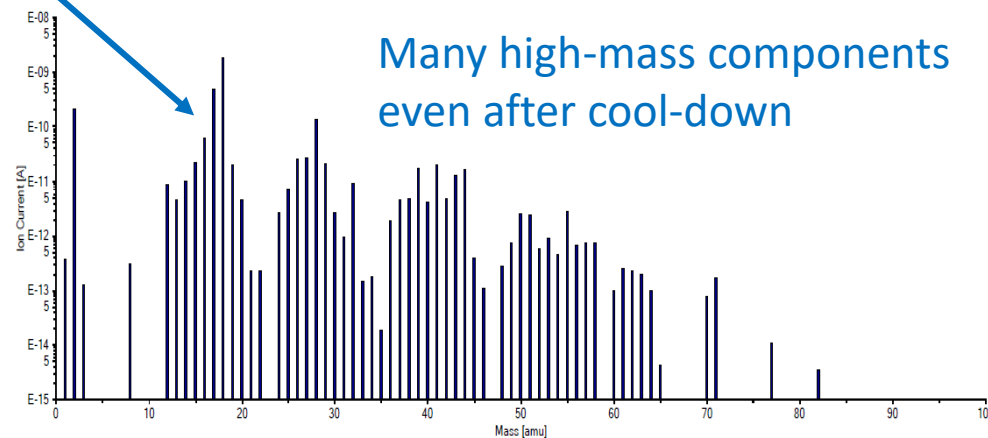
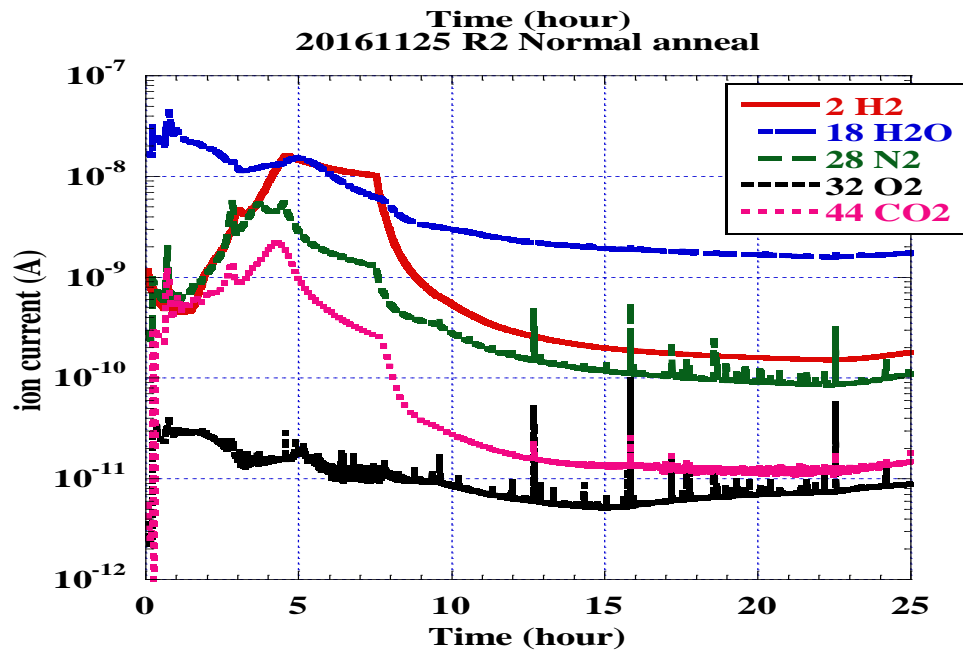
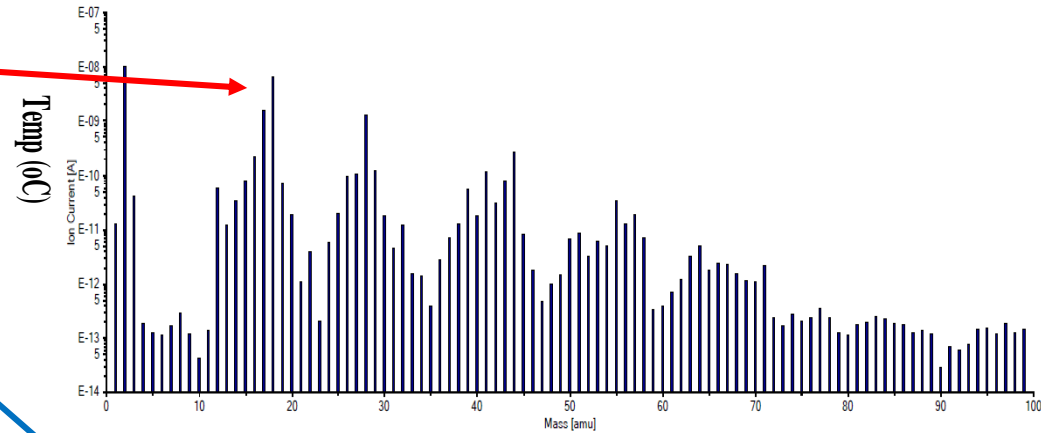
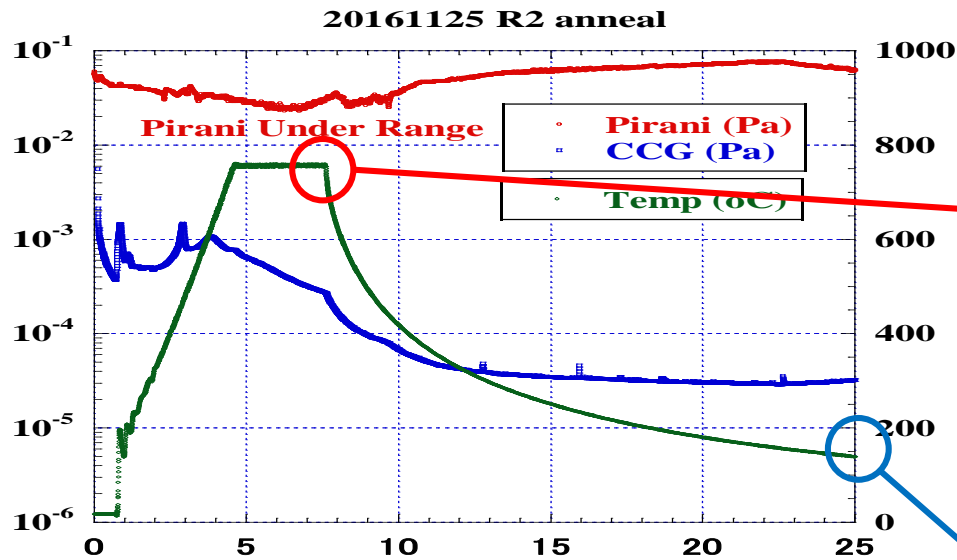
要旨：超伝導RF加速空洞には一般的に加工性が良くかつ超伝導転移温度が比較的  
高い二オブが使用される。近年はニオブの限界性能を越えるためNb3Sn、MgB2、窒素処  
理などによる薄膜コーティングに関する研究も盛んとなってきた。窒素を導入  
することにより空洞の低損失・高電界化（High-Q, High-G）を実現する新技術が開  
発された。ILC（国際リニアコライダー）のコスト削減を目指したR&Dを日米で始  
めることになったが、この低損失・高電界化はR&Dの重要なテーマとなっており、  
KEKでもこの処理についてR&Dを開始しつつある。本セミナーでは、超伝導空洞  
の最近の動向を説明した後、KEKでの展開を報告する。

世話人 村上洋一（物構研）、道園真一郎（加速器）

Backup slide

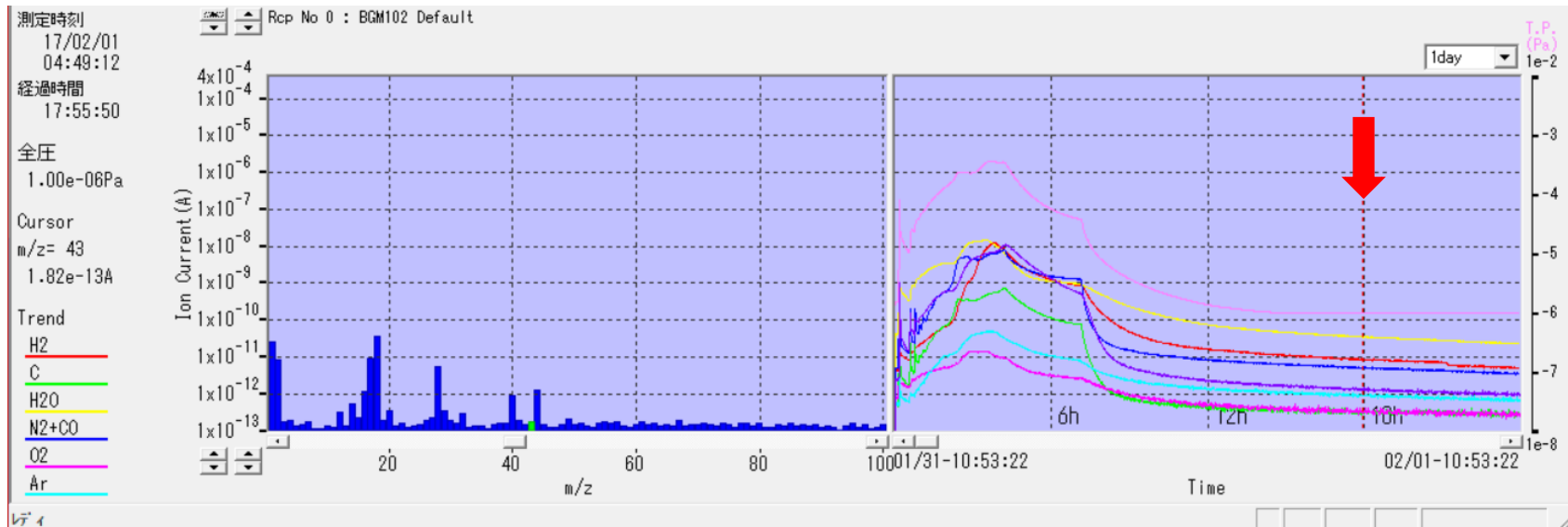
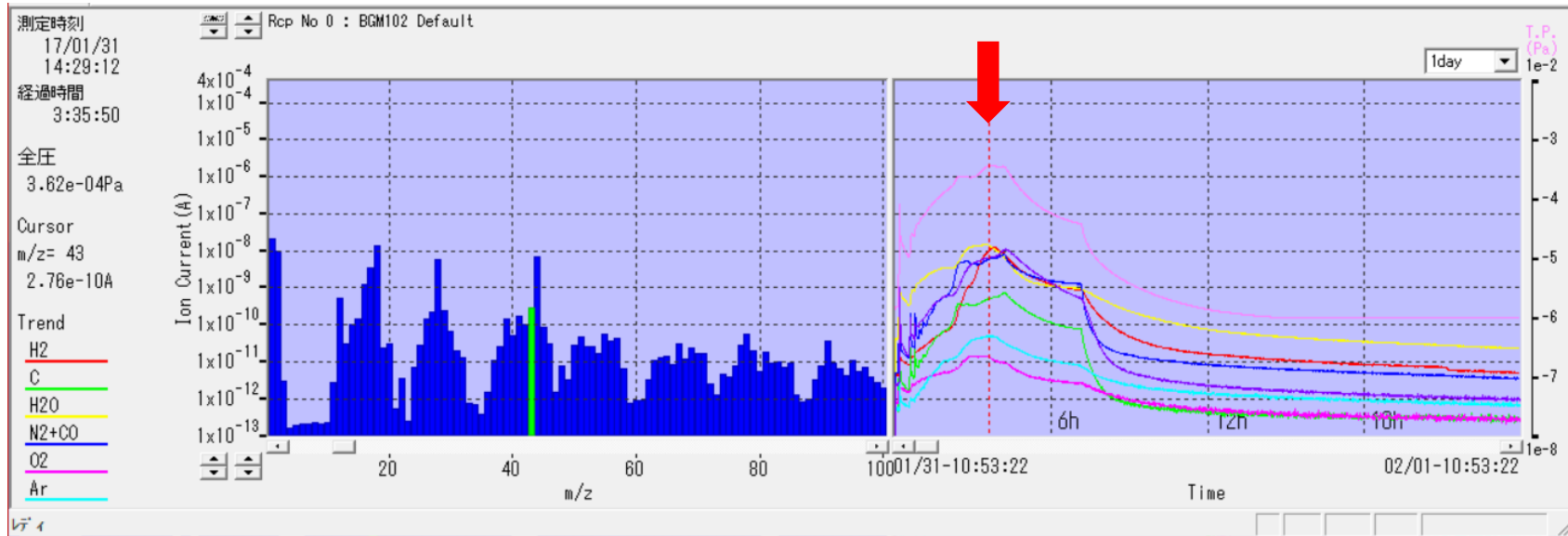
# RGA spectrum of KEK big furnace

No RGA data for KEK small furnace



# RGA spectrum of J-PARC furnace

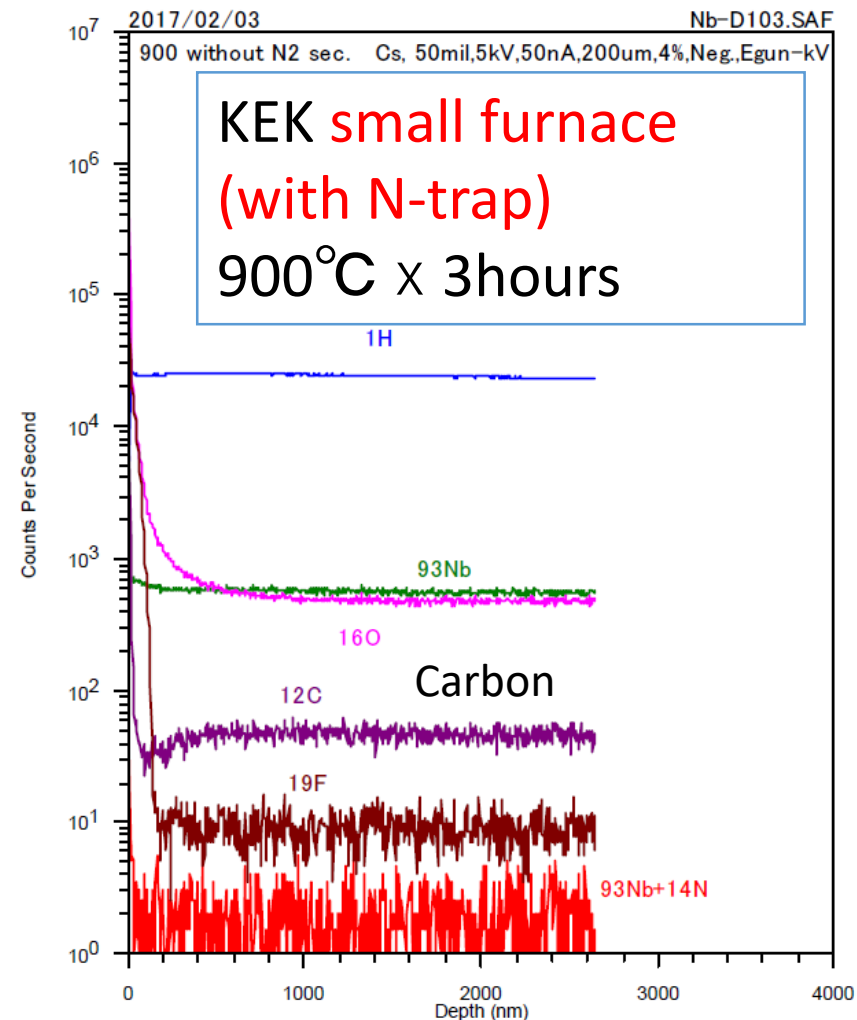
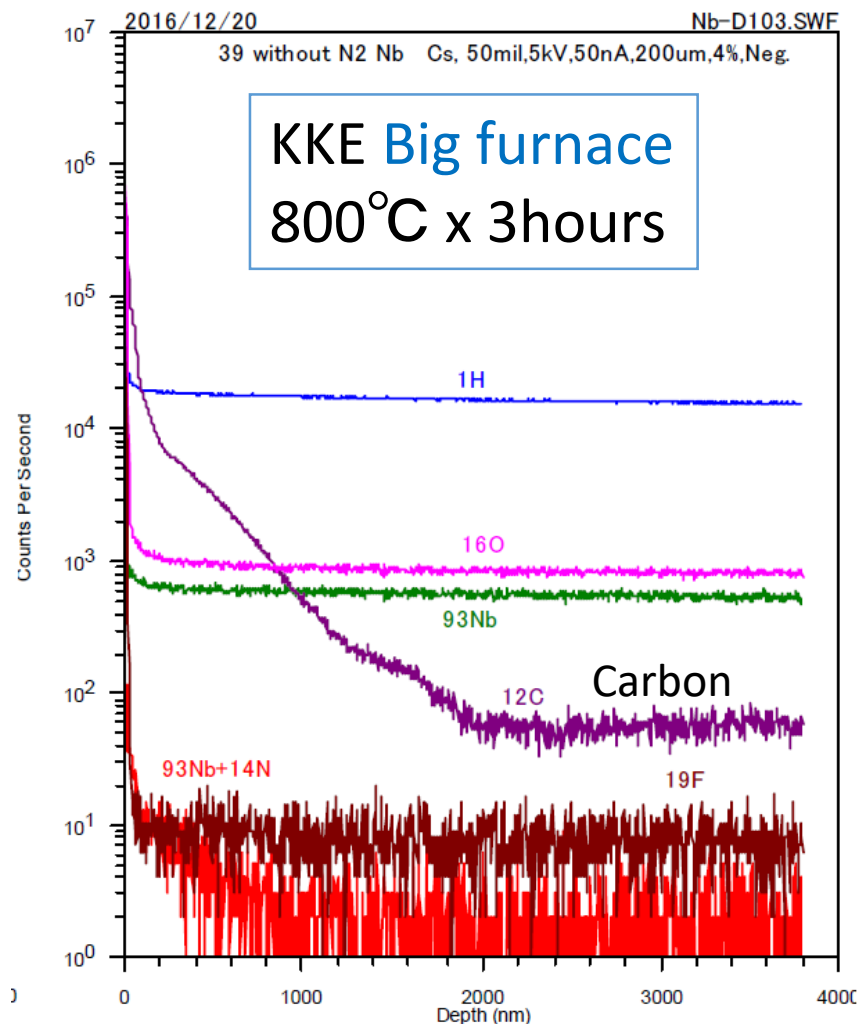
750deg, 3hour annealing (No doping)  
No cavity. Jigs and Nb samples.



# Nb sample analysis for KEK big/small furnace (Heat treatment without N-dope)

Analyzed by ULVAC

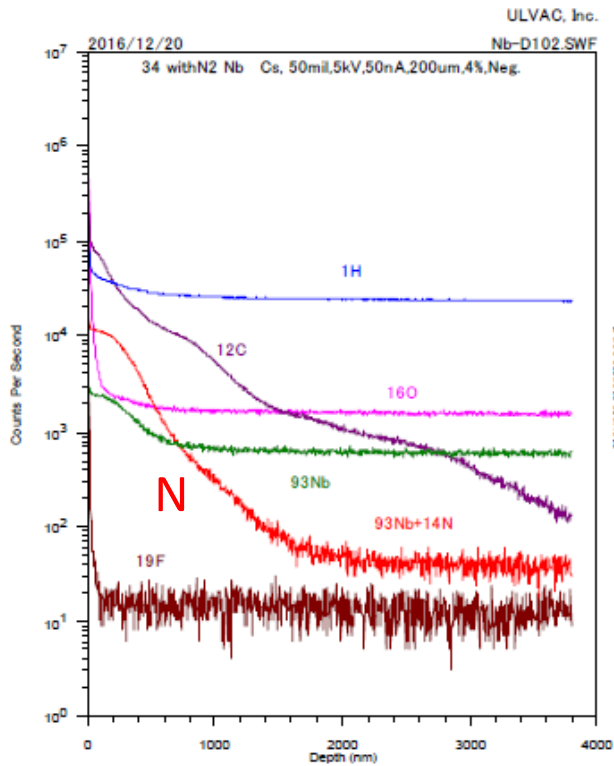
ULVAC, Inc.



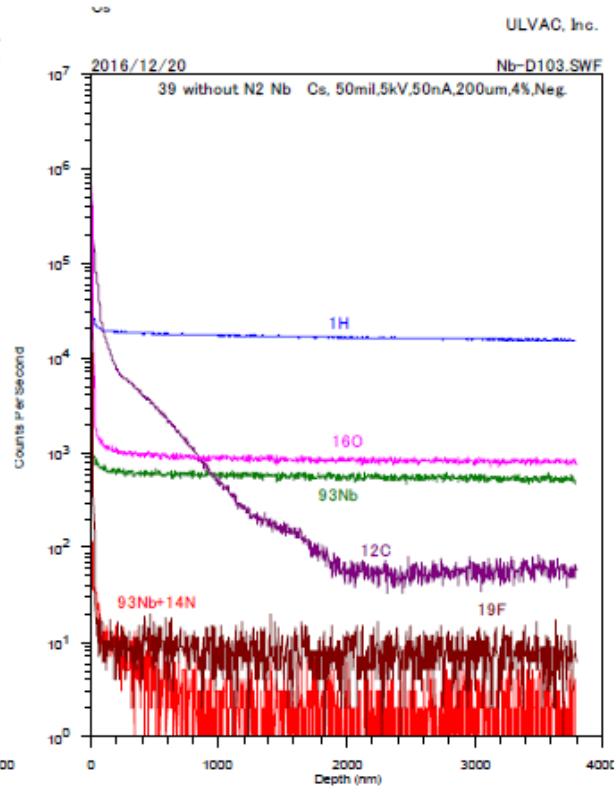
Each figures are up to 4um.

# Nb sample analysis for KEK big furnace Analyzed by ULVAC

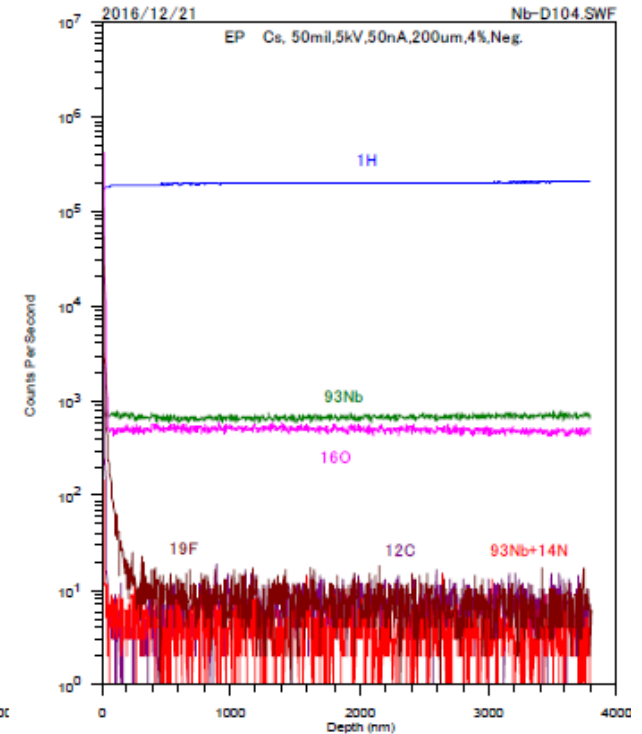
Heat treatment  
with N-doping



Heat treatment  
No N-doping



No heat treatment  
(only EP)



— 93Nb — 1H — 16O  
— 14N+ 93Nb — 12C — 19F

Each figures are up to 4um.

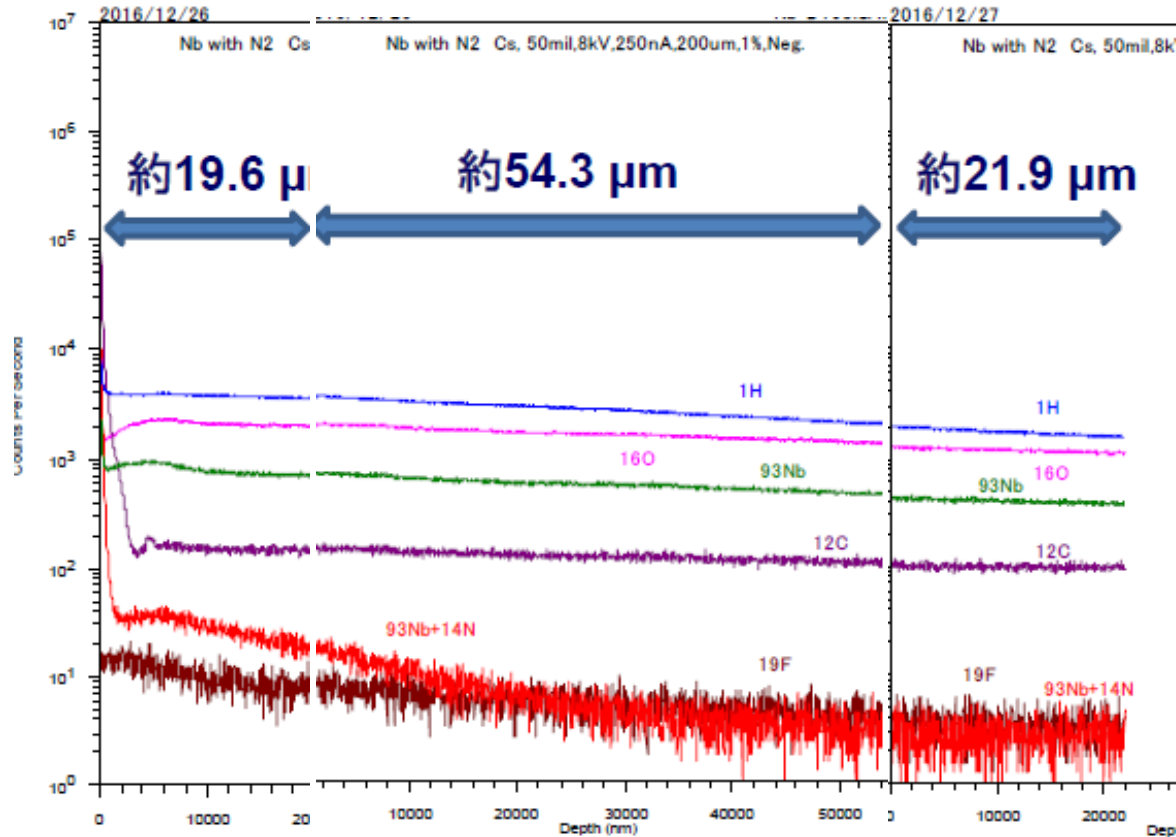
N is observed for N-doped sample  
C is observed for heat treated samples.



# SIMS for N-doped sample (~100um)

Analyzed by ULVAC

Total of three measurements



- Rapidly decrease until ~1um
- Flat up to ~10um
- Then gradually decrease (down to lower limit)
- Nbehavior seems to be similar

What does N treatment do? N depth profiles by SI

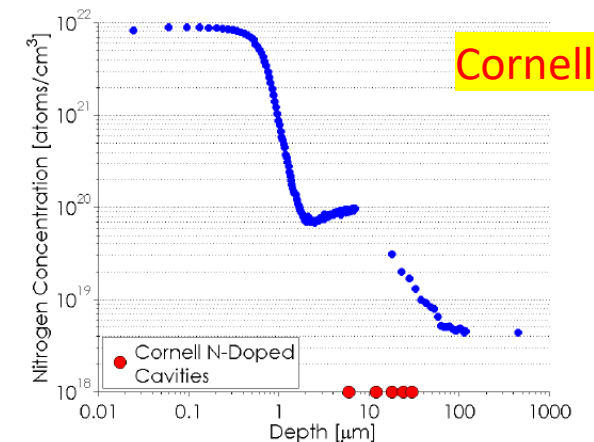
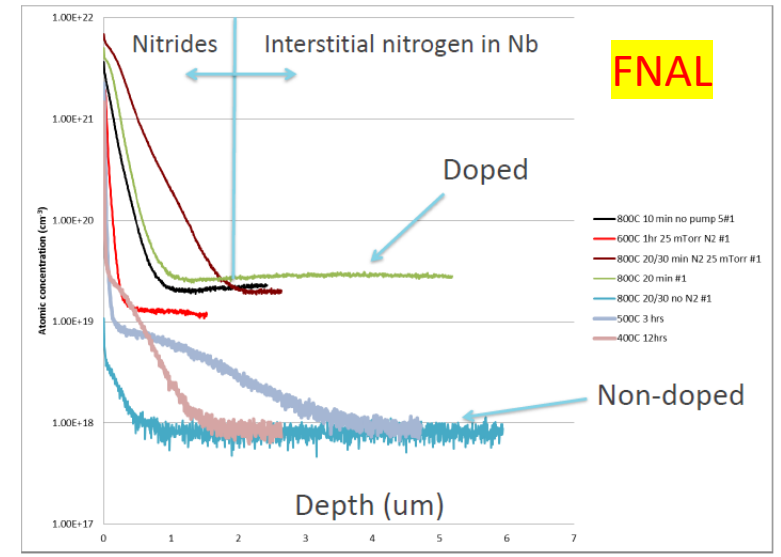


Figure 6: SIMS results from a sample treated with TE1-4 and TE1-5. Single-cell cavities are also included for reference.