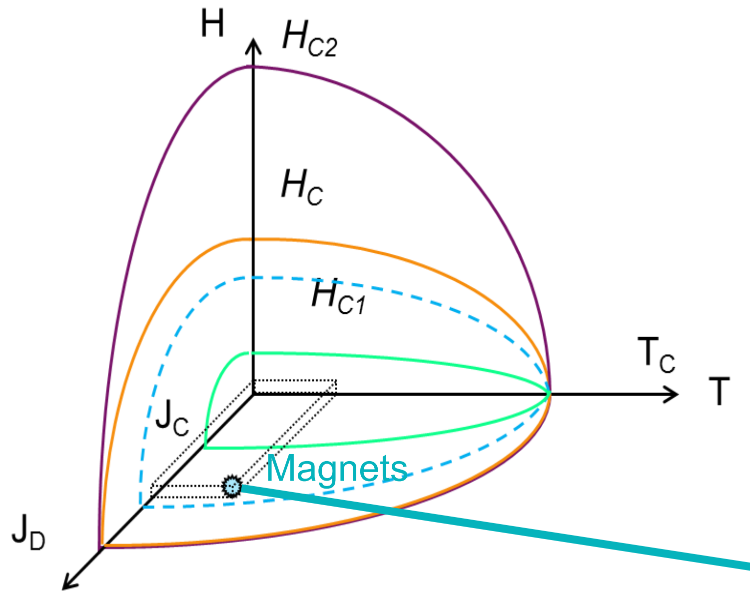
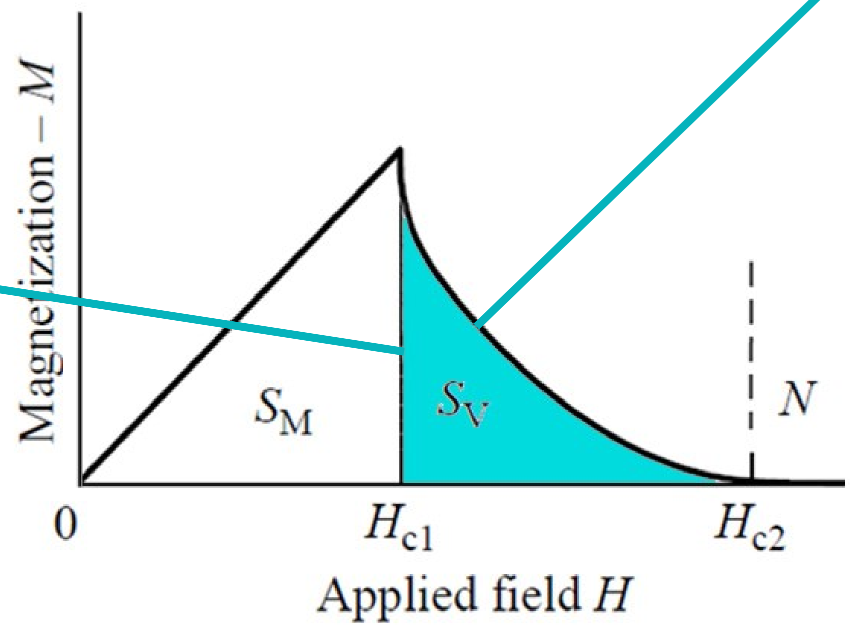
Superconductive Materials

Part 12

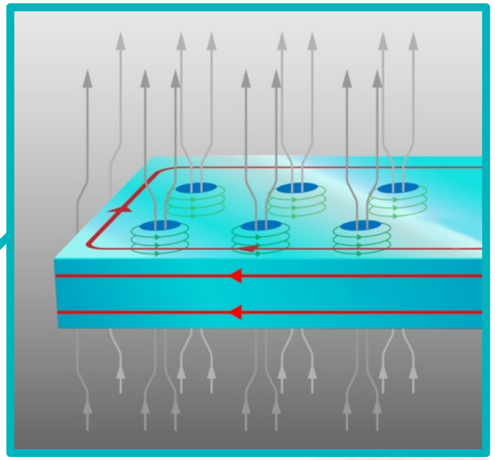
Materials for SRF - Bulk Nb

Superconducting Magnets





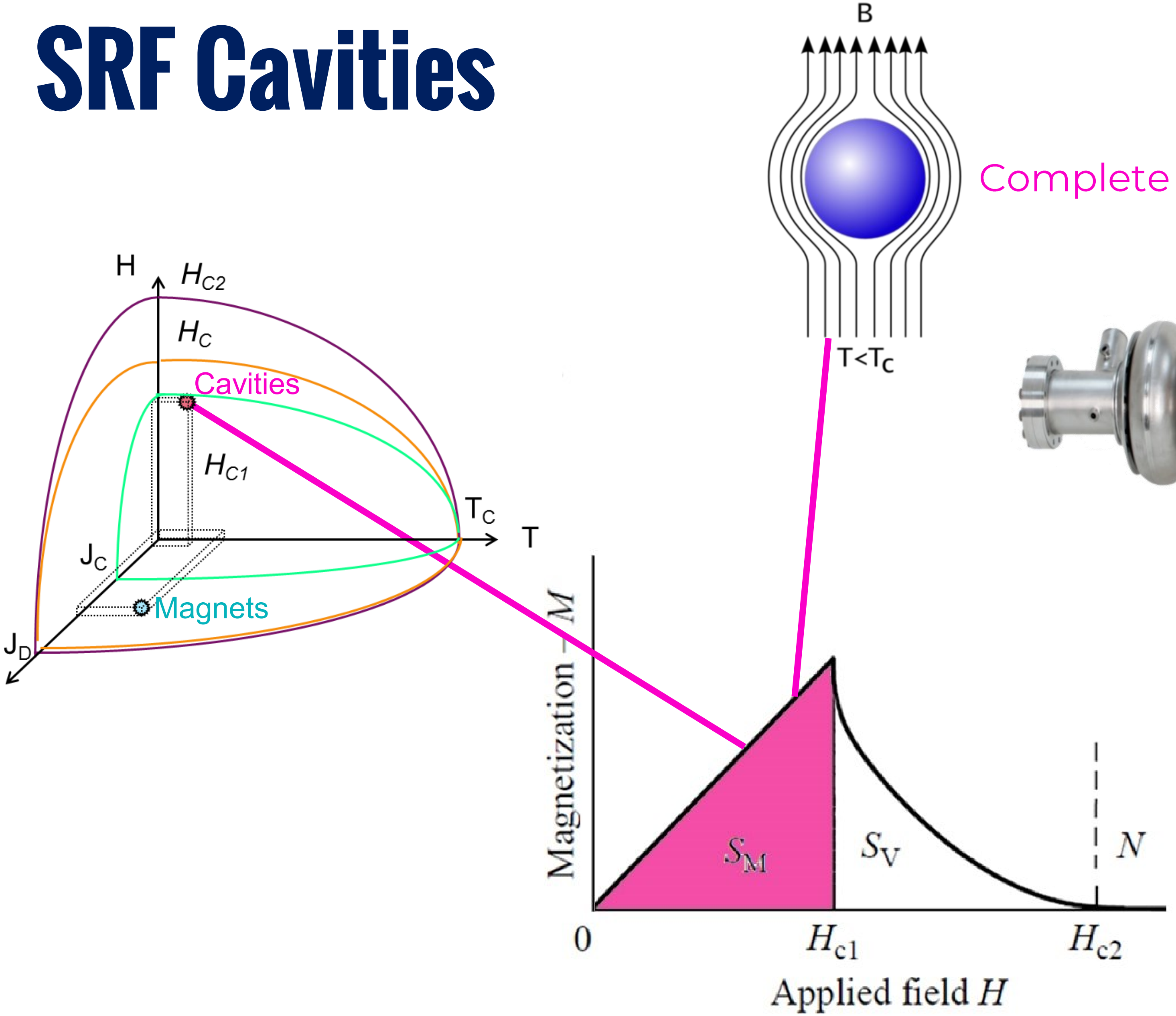
Mixed State, Vortex



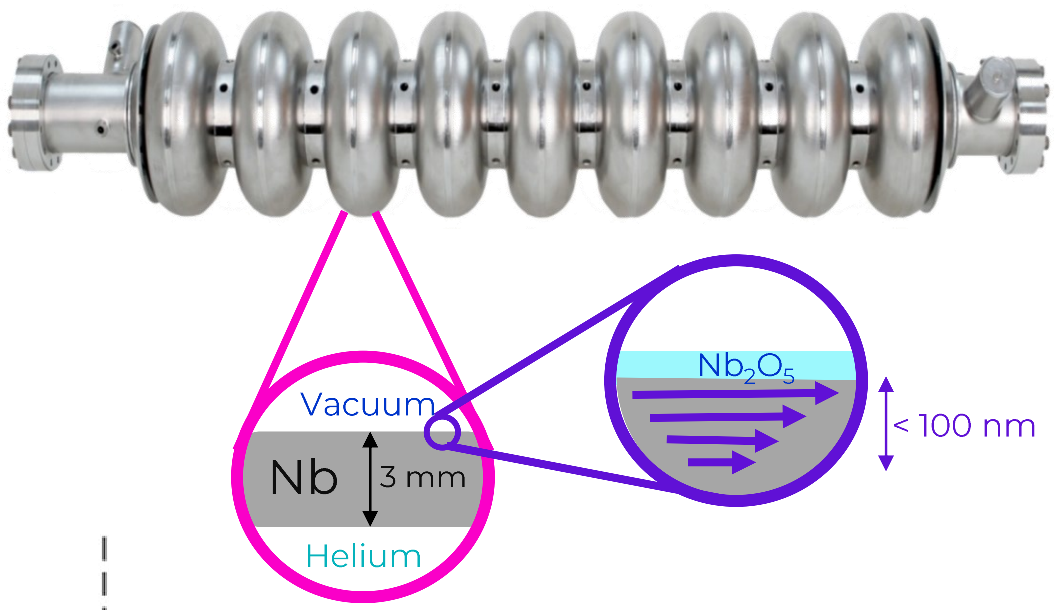
Nb_3Sn ITER cable

REBCO cable prototype, CERN

SRF Cavities



Complete Fux Expulsion



RF interact only with 100s of nm on the **surface**

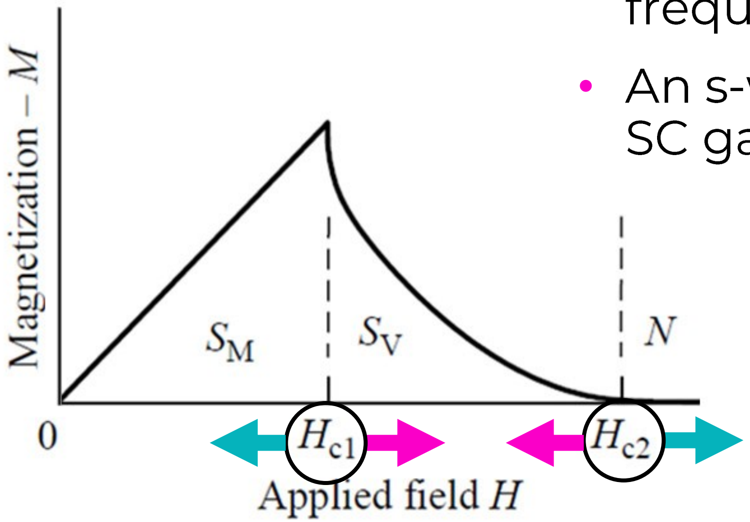
Different regime means different materials

Good SC for magnet application are bad for cavities!

Magnets – DC

- High current densities with 0 resistance
- Mixed State

Defects are voluntarily introduced to **enhance pinning**



Cavities – RF

- very high field with minimal dissipation (10-20 nΩ @1.3 GHz)
- Vortices cannot keep pinned at this frequency → **Meissner State**
- An s-wave Cooper pairing state with a full SC gap on the entire Fermi surface

Emphasis is placed on **reducing** the number of **defects**

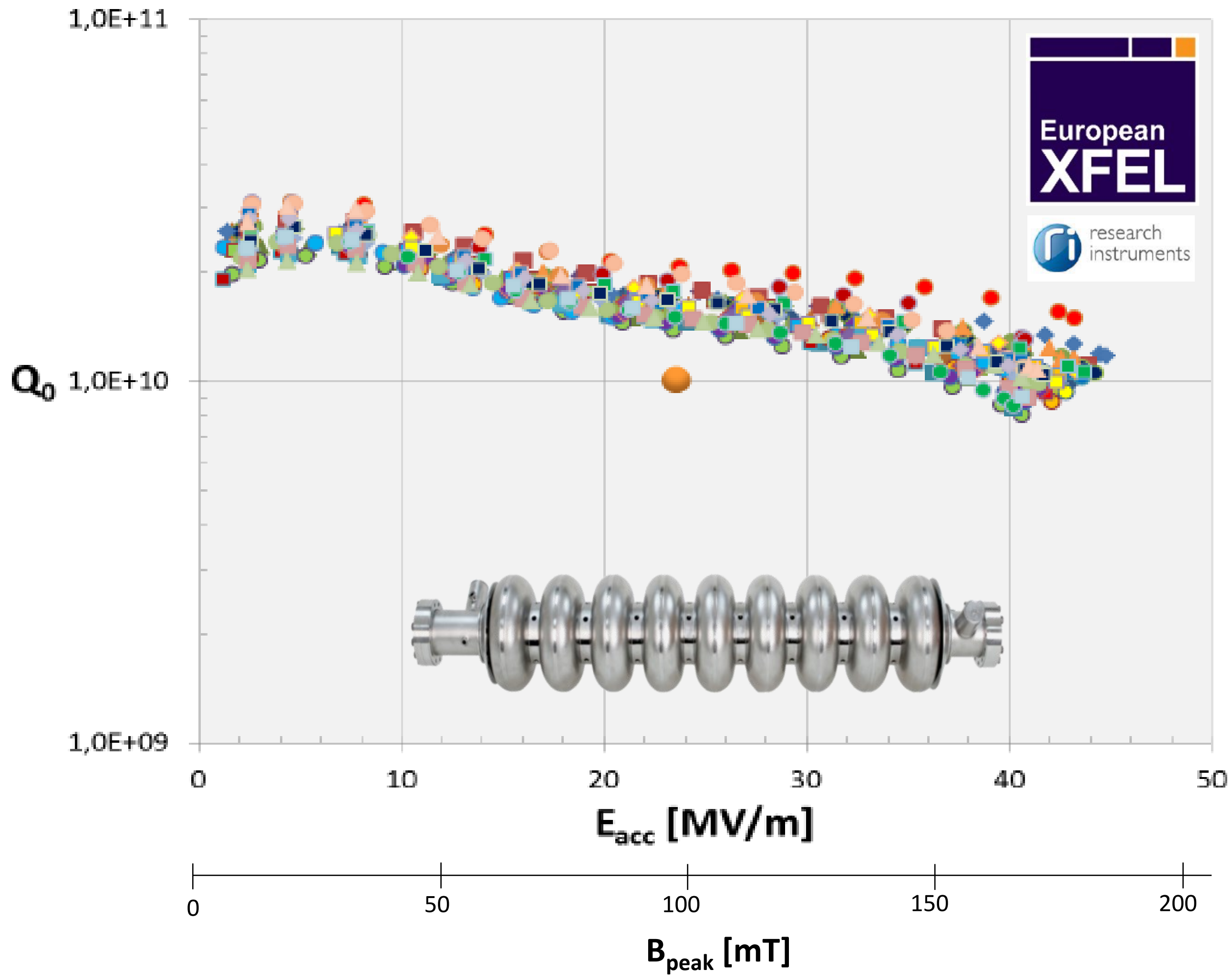
State of the art: Bulk Nb

Performances closer to Nb theoretical limits

$H_{SH} \sim 200$ mT

Because **Tc of Nb is 9.2 K**

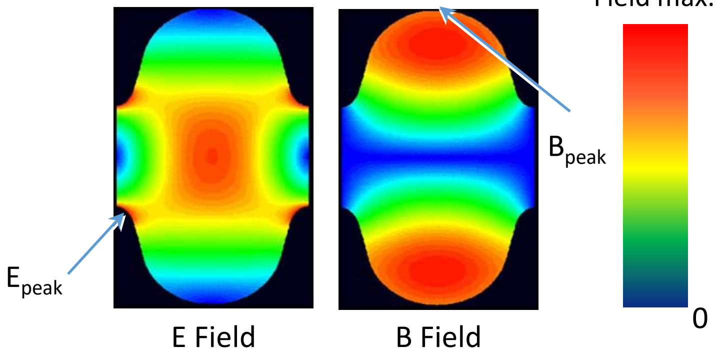
SRF cavities are operated at **2 K for High Q**

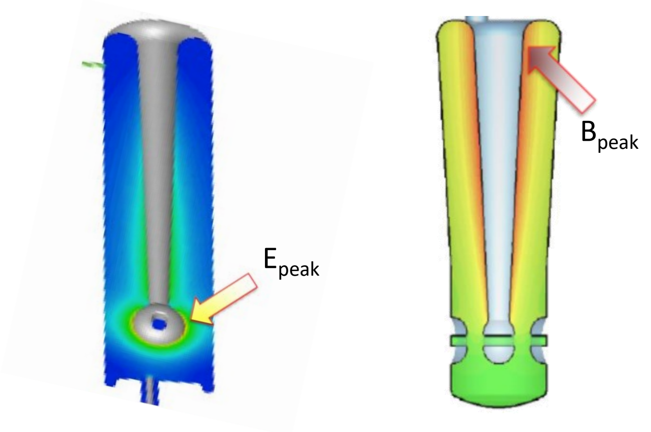


Niobium Elliptical SRF Cavities

Bulk Nb: monopoly since > 30 years

Nb/Cu: applications at low accelerating field only





Figures of merit:

$E_{acc} \propto B_{PEAK}$ *limitation : magnetic transition*

$Q_0 \propto 1/R_S$ *limitation : thermal transition*

Duty cycle (=> 100%) *limitation : cryogenic power*

$\beta = \frac{v}{c}$ *particle speed /light speed (influences design)*

At ω < 3 GHz: cavities are mainly limited by B_{PEAK}!!!

C. Antoine, CEA Saclay

The right SRF material should provide:

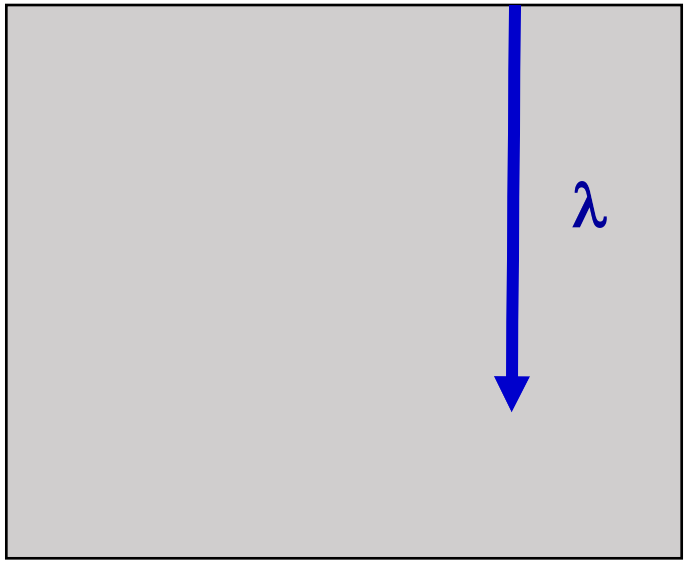
1. **Low surface resistance**, including low residual resistance at T → 0;
2. An **s-wave Cooper pairing state** with a full superconducting gap on the entire Fermi surface;
3. A **high lower critical magnetic field H_{c1}** at which the weakly dissipative Meissner state is destroyed due to penetration of vortices;
4. A **high superheating magnetic field** which defines the theoretical limit of the SRF breakdown;
5. **High thermal conductivity** to transfer the rf dissipated power through the cavity wall;
6. **Grain boundaries transparent to high rf** screening currents in polycrystalline cavities;
7. Comparatively **simple chemical composition**, so that the material is not contaminated by nonsuperconducting second phases, and the superconducting properties are not degraded by local chemical nonstoichiometry;
8. **Good mechanical properties and malleability** to minimize crack formation during cavity manufacturing (forging, deep drawing, spinning, etc.)

Superconductors for SRF

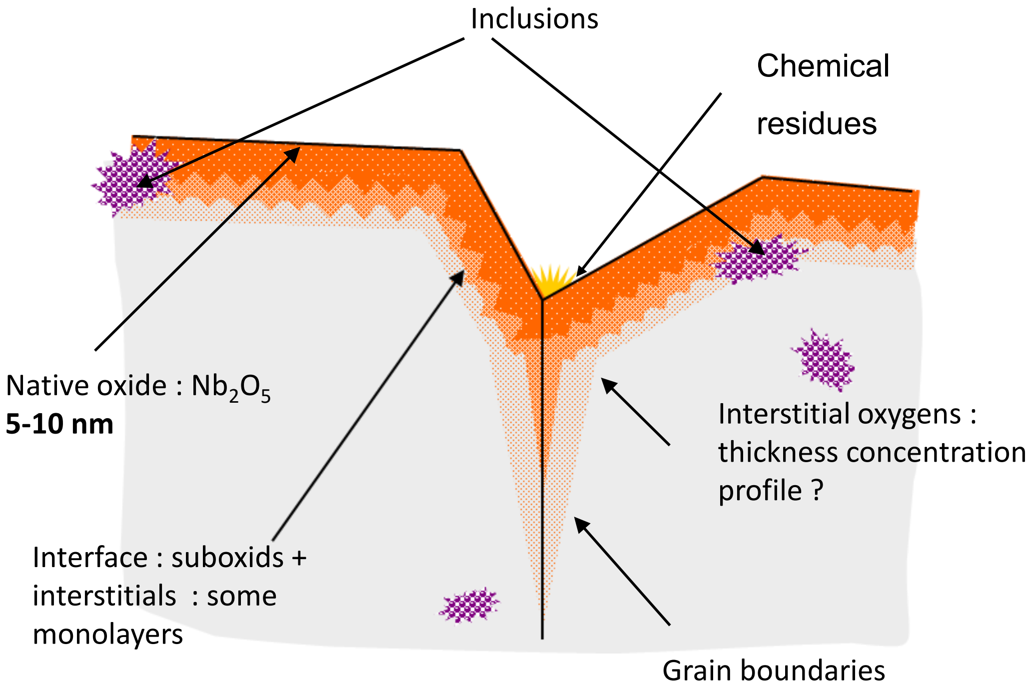
Material	T_C (K)	$\mu_0 H_{SH}$ (mT)@ 0 K
Pb	7,1	100
Nb	**9,2**	**219,0**
NbN	17,1	214,0
Nb_3Sn	18,3	425,0
MgB_2	39,0	170,0
Pnictides $Ba_{0.6}K_{0.4}Fe_2As_2$	38,0	756,0
Cuprates YBaCuO	93,0	1050,0

We need a quasi perfect material on the surface…

Surface as seen by theoretician…



Real Surface



Even after best surface treatments they are **still a lot of defects at the µm and nm level** that have a **strong influence on SC properties** but are difficult to include in models

➡ **always trust experiments rather than models…**

C. Antoine, CEA Saclay

Nb bulk cavities

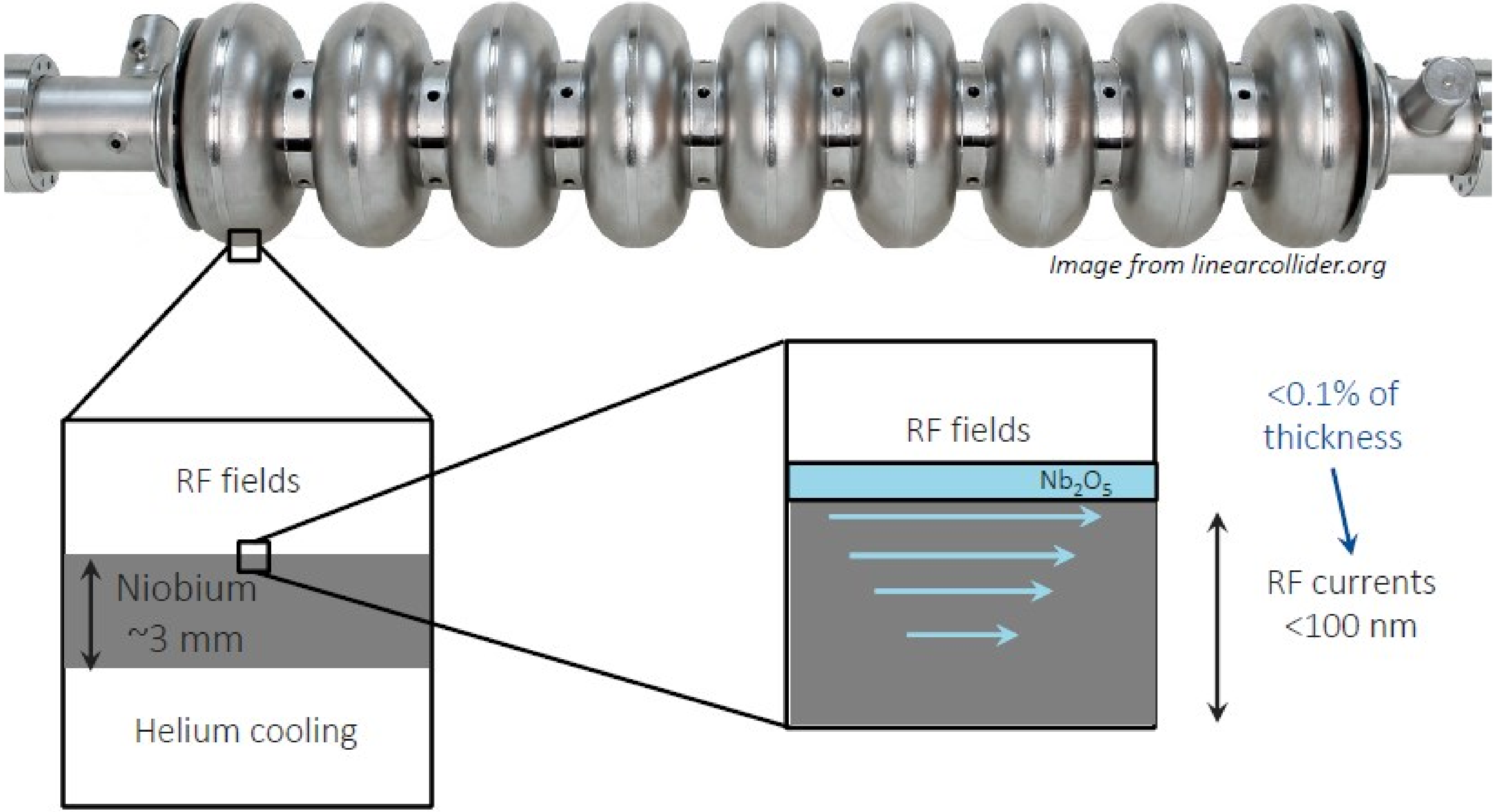
Nb bulk Cavities

State of the art in superconductive resonant cavities

Maximum accelerating field of 50 MV/m close to theoretical limit

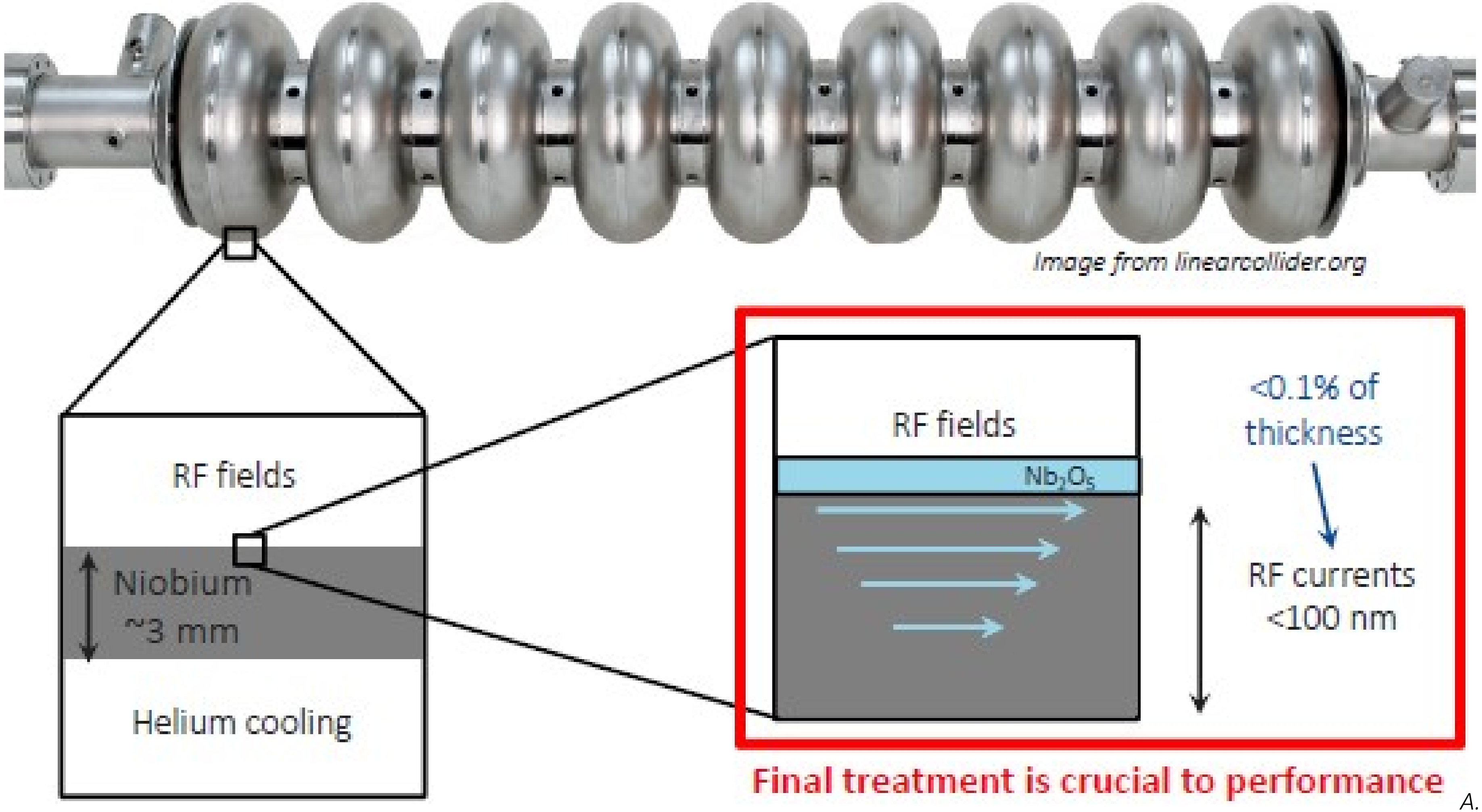
Cost reduction is an issue for the future accelerators

Performance are determined by nanometer scale structure of inner surface



Grassellino, SRF2019 Tutorials

Performance are determined by nanometer scale structure of inner surface



A. Grassellino, SRF2019 Tutorials

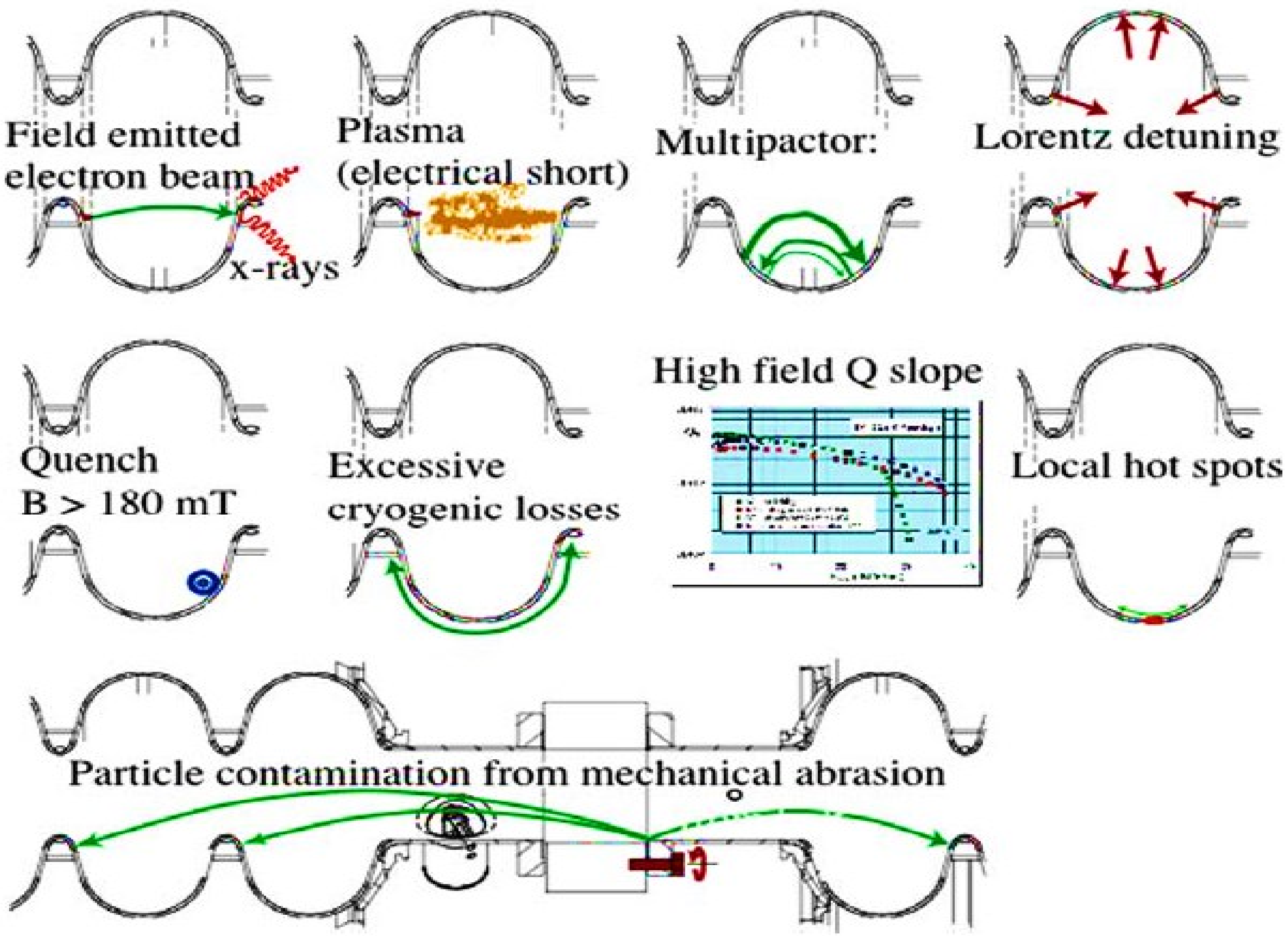
SC cavities production: a long chain, but…

> A chain is as strong as its weakest link !!!

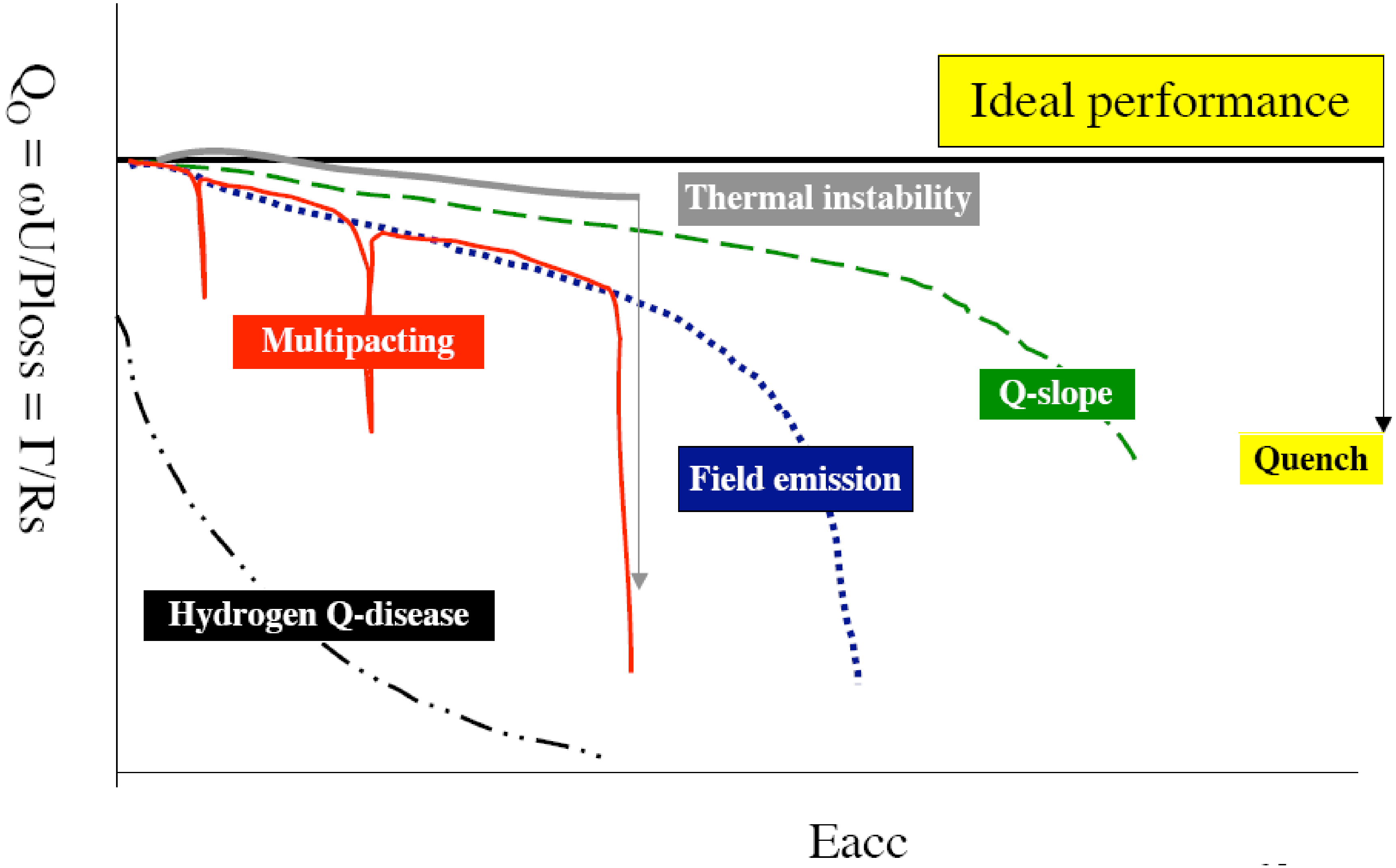
- Chain of
  - Material
  - Fabrication
  - Surface Preparation
    incl. cleanroom, media, procedures, human factor
  - Vacuum
  - Quality assurance
- For high gradient / low loss SRF cavities all aspects have to be fulfilled

D. Sertore, SRF Cavity Fabrication, EASIschool 3 Genoa, 2020

SC cavities may have various “illness”



Anomalous loss mechanism

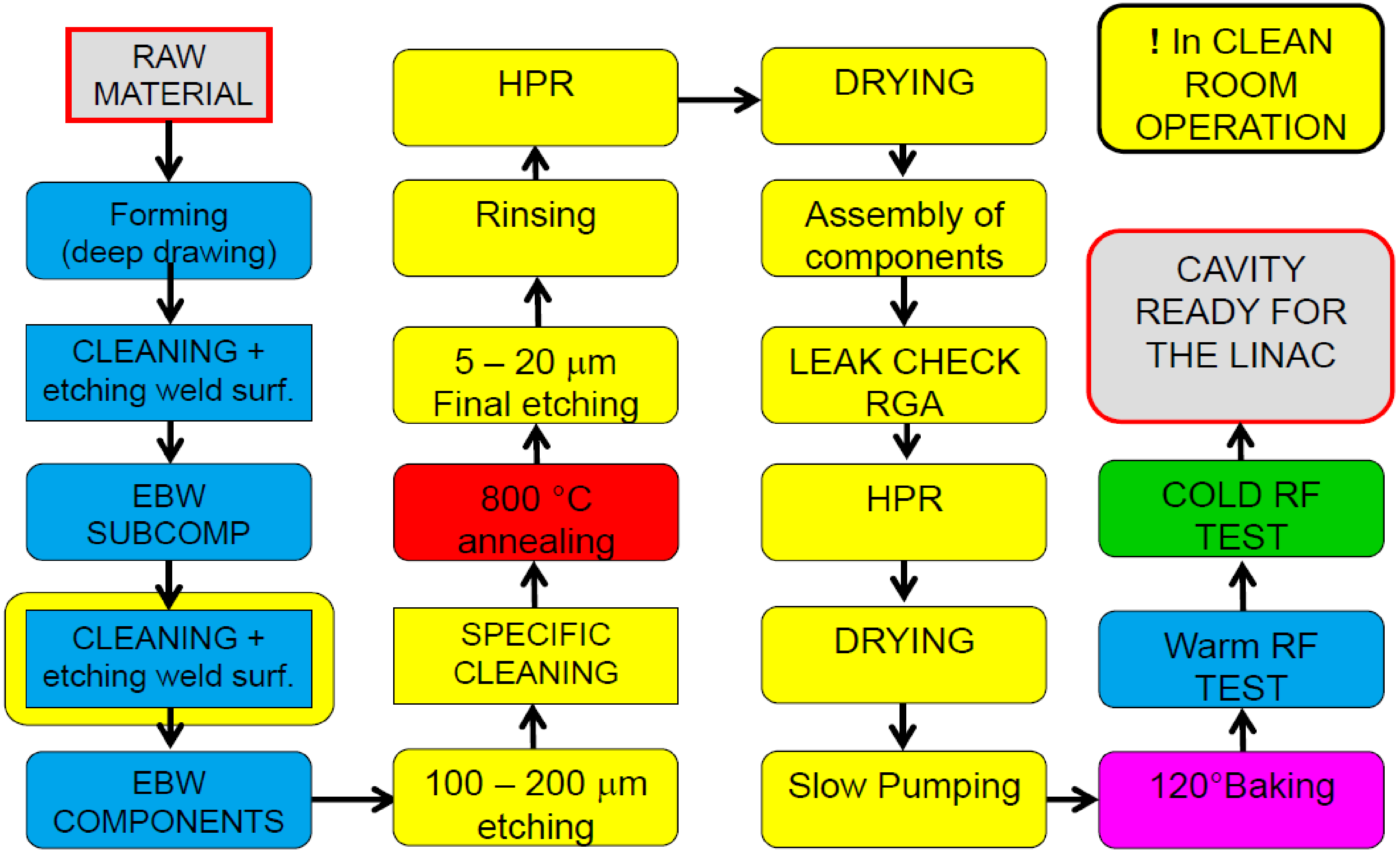


Some general statements

- Anomalous loss mechanisms:
  - Quench (local thermal instability)
    => material + fabrication (=> cleanliness)
  - Field emission
    => Cleanliness of surface treatment, assembly, handling + vacuum
  - Q-drop (without field emission) + Q-slope => ?
  - Multipacting
    => Cavity shape + RF surface condition
  - Hydrogen Q-disease
    => Chemical surface treatment
  - Increased residual surface resistance
    => Cleanliness of surface treatment, assembly, handling + vacuum

D. Sertore, SRF Cavity Fabrication, EASIschool 3 Genoa, 2020

From raw material to RF cavity for EXFEL



Paolo Michelato, SRF2013 tutorials, September 2013

Large scale specialized infrastructure is required to make/study high Q cavities and assemble/test full accelerating cryomodules


TRIUMF
SLAC
FRIB
Argonne
Fermilab
Cornell University
BROOKHAVEN NATIONAL LABORATORY
Jefferson Lab
Science & Technology Facilities Council
HZB Helmholtz Zentrum Berlin
DESY
CERN
cea
INFN
IMP
Institute of High Energy Physics
Chinese Academy of Sciences
KEK

Nb bulk cavities: fabrication cycle

From EM Design to Fabrication: few considerations

- From the EM point of view, the cavity is **design at the operative conditions** (usually 2 K, in vacuum, with tuner).
- When you **fabricate** the cavity, you are at **room temperature, in air** and the cavity needs to be **treated** before being operational
- You need then to consider
  - Thermal shrinkage from 2 K to 300 K (geometry, frequency)
  - Pressure effect (frequency)
  - Dielectric constant effect (frequency)
  - Over-metal for chemical treatment (geometry, frequency)
  - Pre-tuning (frequency)

P.S. When you fabricate a cavity, be sure that the design couples with the feasibility of the processes you are going to apply

D. Sertore, SRF Cavity Fabrication, EASIschool 3 Genoa, 2020

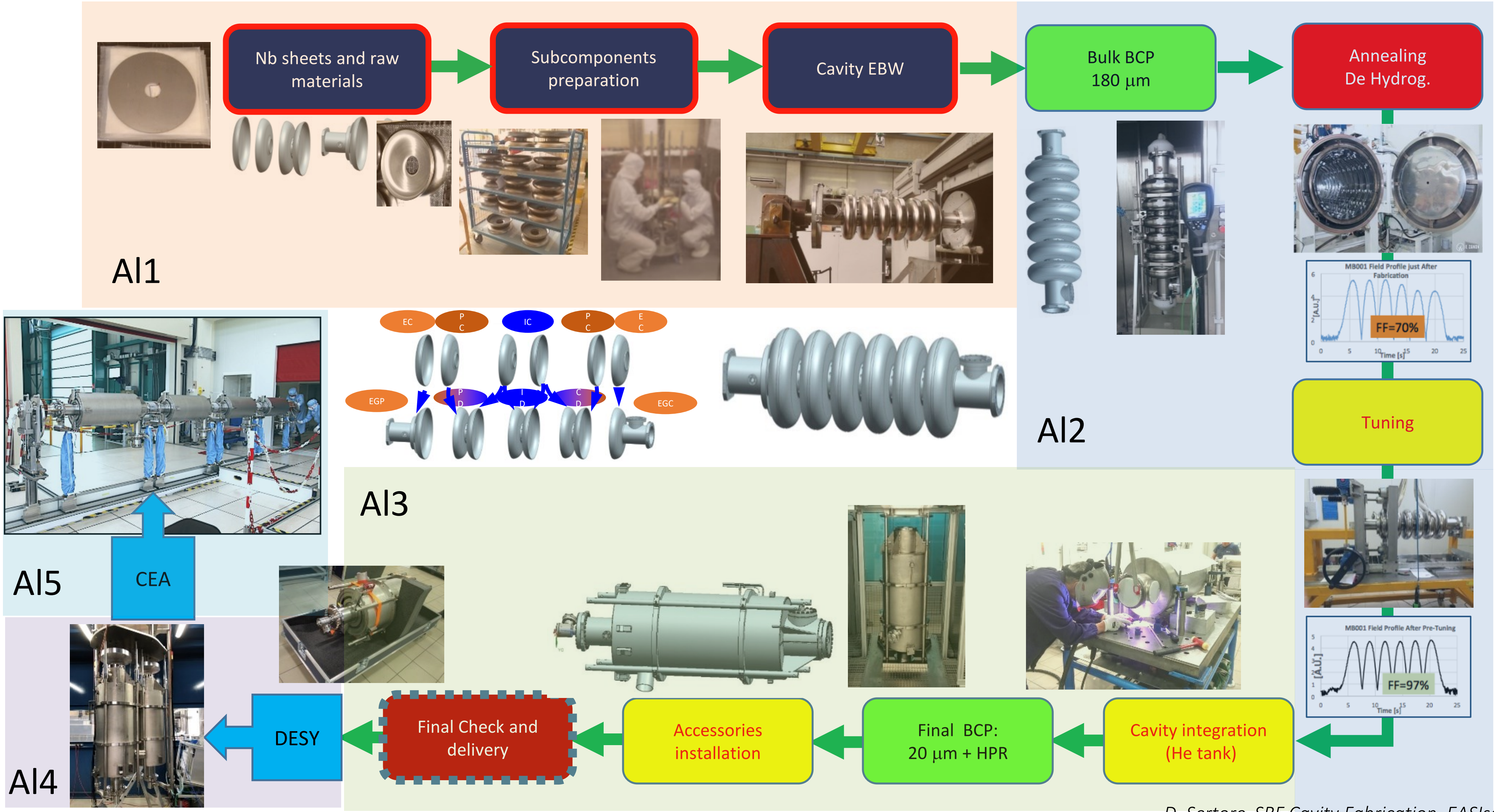
The ESS example

Step	Df [MHz]	Cavity Frequency [MHz]	Comment
Goal Frequency		704.420 MHz	2 K in vacuum
Pre-load for tuner	-0.100 MHz	704.320 MHz	Unloaded cavity at cold
Room Temperature	-1.028 MHz	703.292 MHz	Shrinkage from 2 K to 300 K [a]
In Air	-0.234 MHz	703.058 MHz	Dielectric constant [b]
Etching	+0.480 MHz	703.538 MHz	Before Chemistry (150 μm) [c]
Weld Seam	+0.000 MHz	703.538 MHz	Weld Seam perturbation [d]

[a] Integral shrinkage from 2 K to 300 K tabulated
[b] Inversely proportional to square root of dielectric constant
[c] Estimated from Slater's perturbation on cavity inner surface
[d] Estimated from direct measurement. More significant for high frequency cavities

D. Sertore, SRF Cavity Fabrication, EASIschool 3 Genoa, 2020

The ESS fabrication cycle



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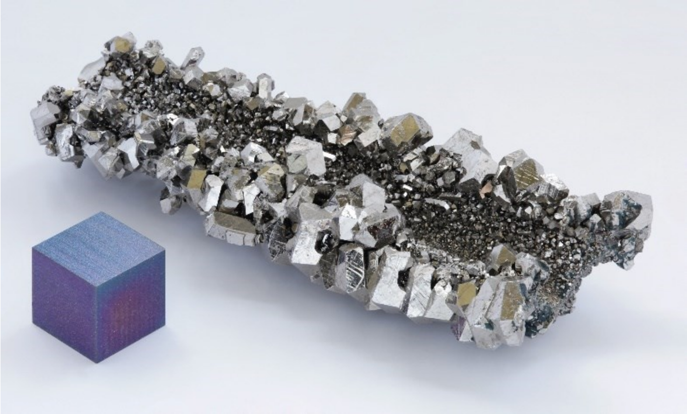
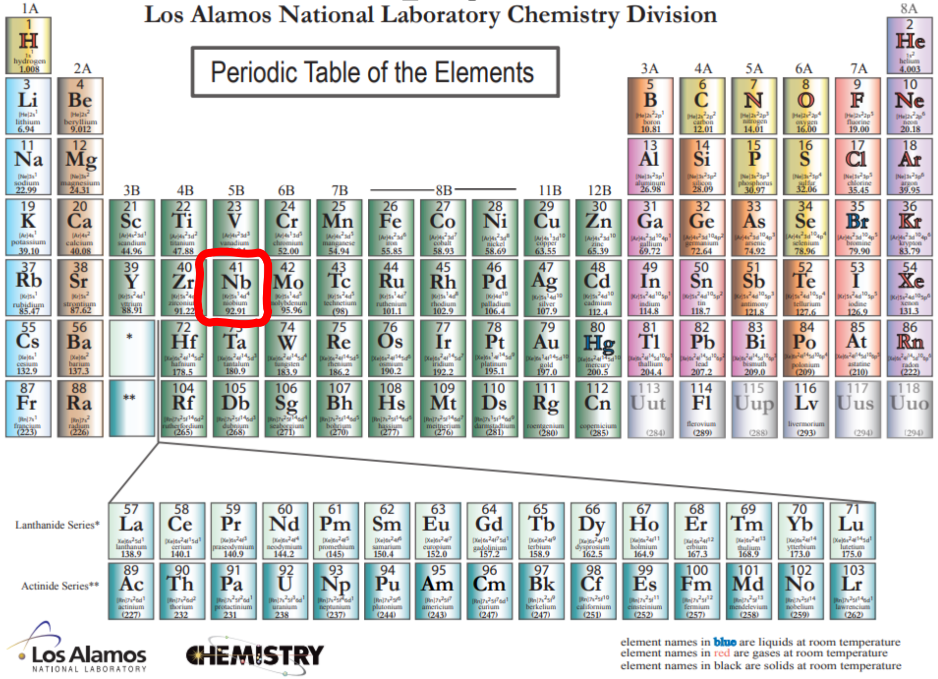
Nb bulk cavities: from raw Nb to sheets

Niobium

- Niobium is ***THE*** material for fabrication of superconducting cavities
  - Critical temperature T_c = 9.25 K
  - High critical field (H_c(0 K) ≅ 240 mT)

    } Highest between pure metals
  - Chemically inert (surface covered by Niobium pentoxide Nb_2O_5)
  - Easily machined and deep drawn
  - Available as bulk and sheets of any size and different shapes





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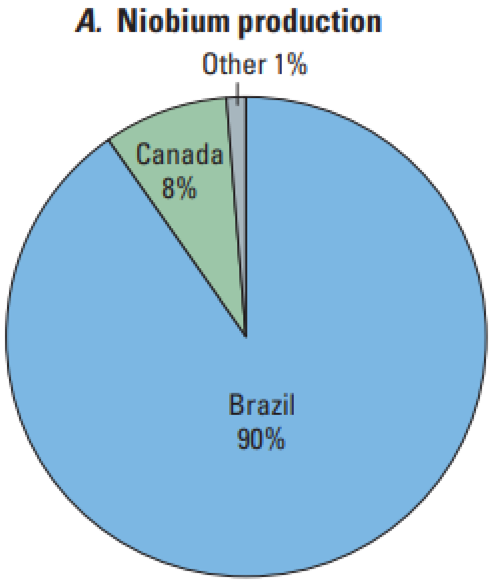
Niobium Production



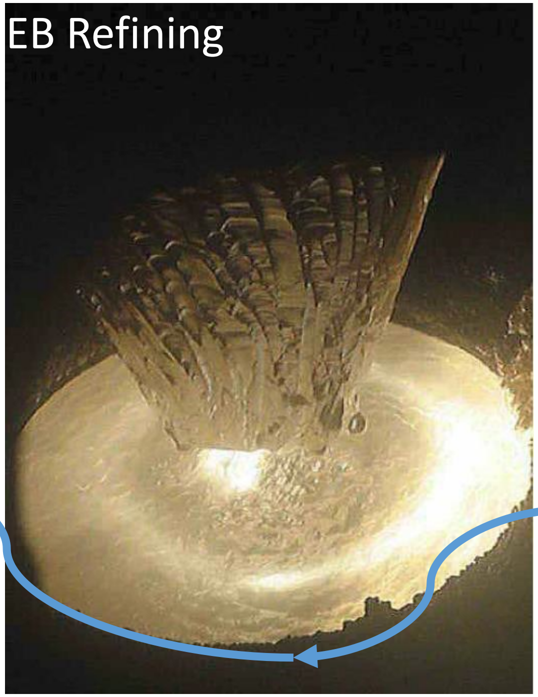
Niobium Production

- The leading use of niobium (about 75 %) is in the production of **high strength steel** alloys used in pipelines, transportation infrastructure, and structural applications.
- Niobium is primarily **derived from the complex oxide minerals** of the pyrochlore group and carbonatites, **usually together with Tantalum**
- The **estimated global reserves** appear more than sufficient to meet global demand for the foreseeable future, possibly the **next 500 years**

Niobium Prodution Process

- The ore (Pyroclore in this case) is treated and **refined** at different stages untill it reaches the **purity necessary** for the Electron Beam Refining.
- The **Electron Beam** process **reduces the impurites** present in the incoming Niobium

2.75 m



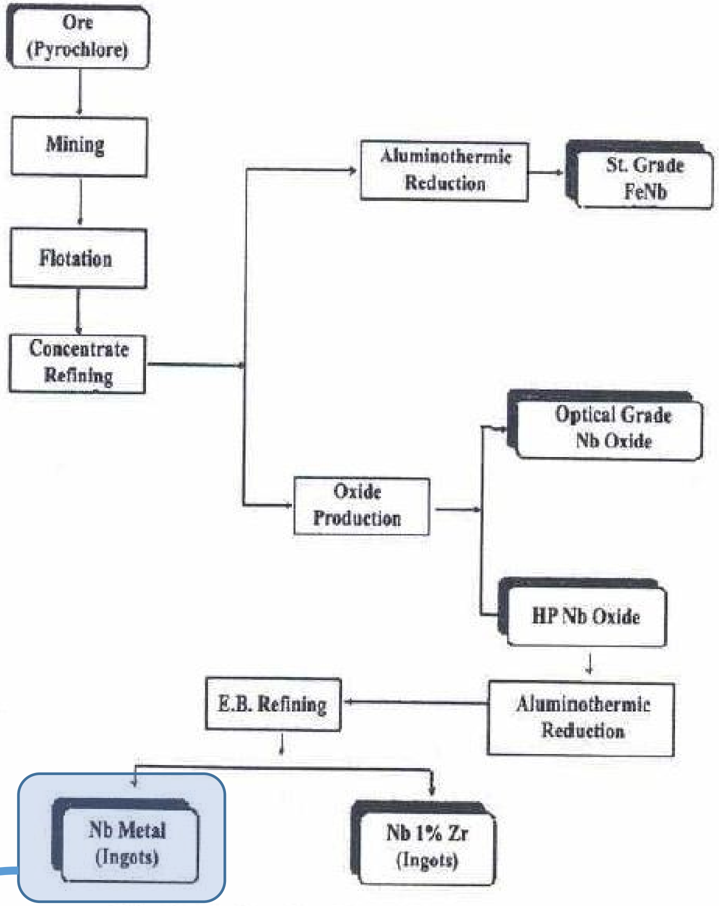
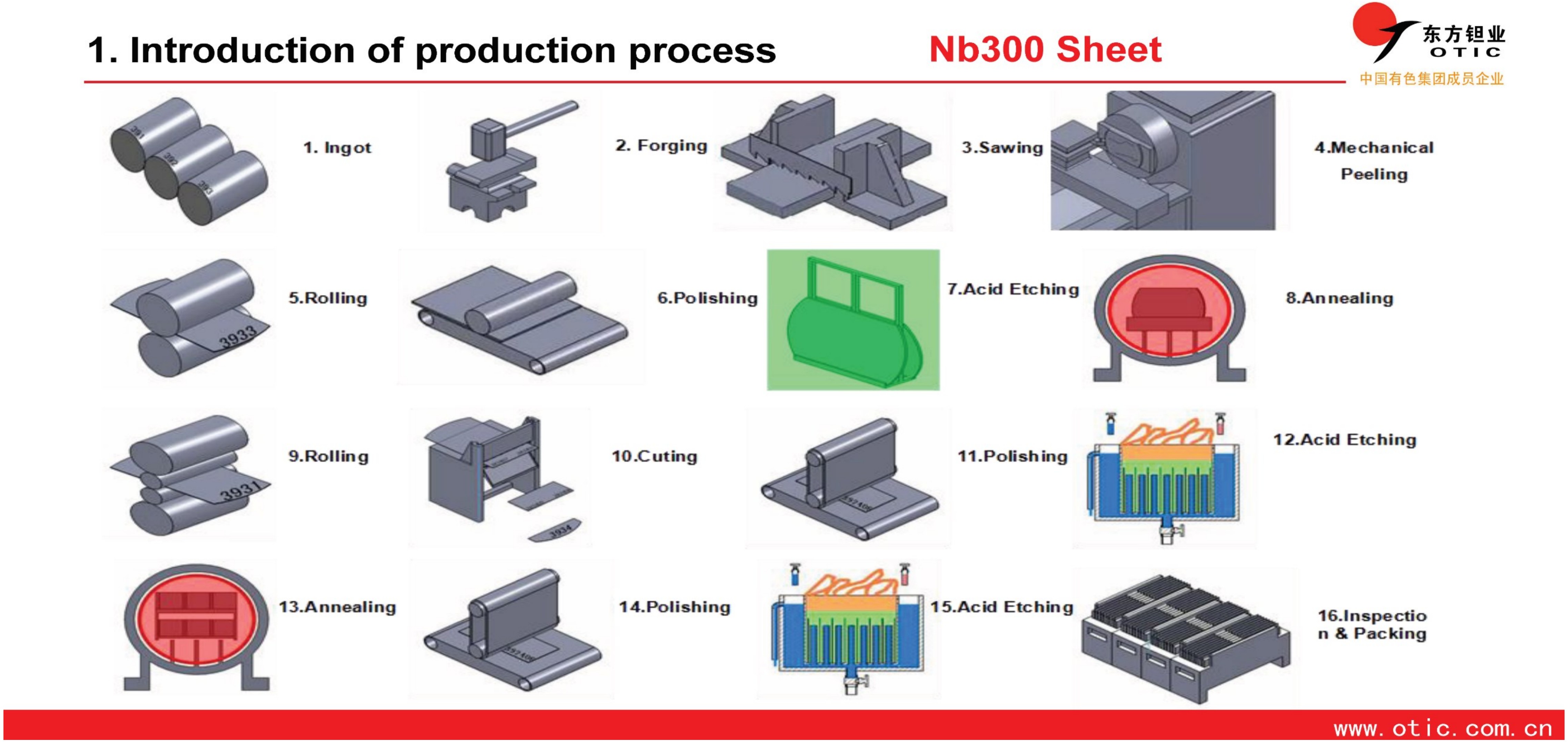


Fig. 3: Production flow chart at CBMM.

Niobium sheets production



Nb Technical Specifications (typical)

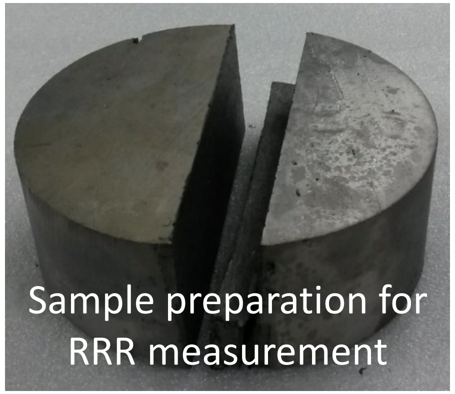
Concentration of impurities in wt.ppm				Mechanical properties	
Ta*	≤ 500	H*	≤ 2	Yield strength**, $\sigma_{0,2}$	$50<\sigma_{0,2}<100$ N/mm² (Mpa)
W*	≤ 70	N*	≤ 10	Tensile strength**	> 100 N/mm² (Mpa)
Ti*	≤ 50	O*	≤ 10	Elongation at break**	30 %
Fe*	≤ 30	C*	≤ 10	Vickers hardness** HV 10	≤ 60
Mo*	≤ 50	RRR*	≥ 300	Absence of foreign material inclusions*	Proven by scanning
Ni*	≤ 30	Recrystal. degree. Grain size* ,** ?	≈ 50 µm	Texture *, ** ?	

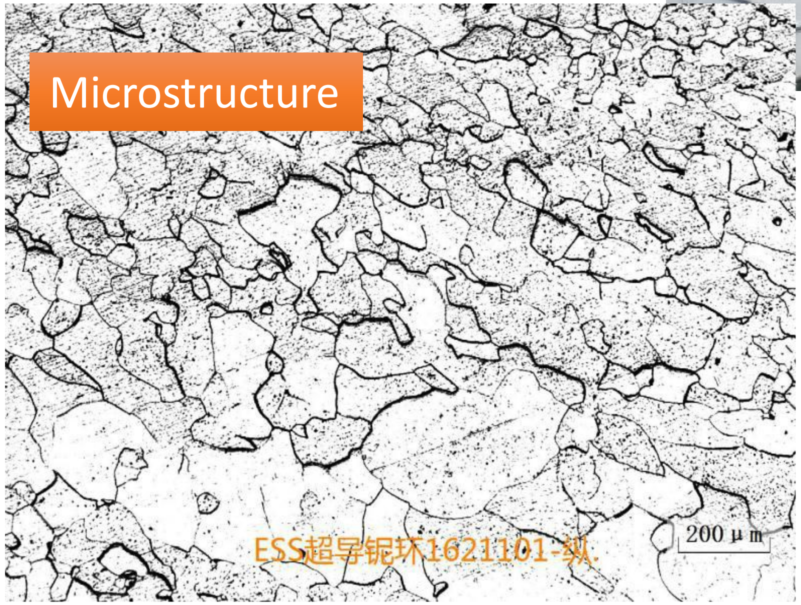
* - relevant for performance

** - relevant for successful fabrication

Nb sheets QC at productor premises

- **Ingot**
  - RRR
  - Mechanical Properties
  - Gas analysis
  - Crystallography
- **All sheets**
  - Visual Inspection
    - Defects (i.e. scratches)
    - Delamination
  - “Rust” test
    - Coarse check for Fe inclusions





Final Report

Dimension and tolerances	Drawing number		DWG-MB-FG-Materials-WOC Pos.1
	Size(mm)		OD160(+2/0)×ID130(0/-2)×40(+2/0)
Item No.	1	Annealing Charge No.	162-171019-5
Ingot No.	ENT-162	RRR value of the ingot	360/385

Chemical Composition I: Ingot Analysis P Product Analysis

Content	Ta	W	Ti	Fe	Si	Mo	Ni	H_2	N_2	O_2	C
guaranteed	≤0.05%	≤0.007%	≤0.005%	≤0.003%	≤0.003%	≤0.005%	≤0.003%	≤2ppm	≤10ppm	≤10ppm	≤10ppm
Result (I)	0.023	<0.0005	<0.0005	0.0005	0.0010	<0.0005	<0.0005	2	10	10	5
Specimen No 1621101			-	-	-	-	-	1	8	8	5
Specimen No P								-	-	-	-
Specimen No 1623101								2	10	10	5

Mechanical tests

Method of test	Hardness test (DIN EN ISO 6507)
Shape of the test piece	Rectangle specimen
Specimen condition	Annealed

Specimen No	Dim.of specimen (mm) L	W	H	Sampling Location	Hardness, HV (min. load 10 N)	
					Requirement	≤60
1621101	10	10	8	*1	Min	54.1
					Max	59.0
1623101	10	10	8	*1	Min	53.8
					Max	58.0

Method of test	RRR values test
Shape of the test piece	Cuboid specimen
Specimen condition	Annealed

Specimen No	Dim.of specimen (mm) L	W	H	Sampling Location	RRR values Requirement	≥300
1621101 (A-B)	85	3	3.0	*1	-	288/309
1623101 (1#-2#)	85	3	3.0	*1	-	281/313

Residual Resistance Ratio (RRR)

- Electrical resistivity of metals at low temperatures is related to the impurity concentrations. The residual resistivity at T = 0 K is caused mainly by scattering of electrons by impurities.

Residual Resistivity Ratio

$$RRR = \frac{\rho(295\ K)}{\rho(4.2\ K)}$$

- **RRR** depends on **impurity content** in the material (typical RRR for cavity is around 300)
- **RRR** is linked to the Nb **thermal conductivity** by

$$\lambda(4.2\ K) \approx 0.25\ RRR\ [\frac{W}{m\ K}]$$

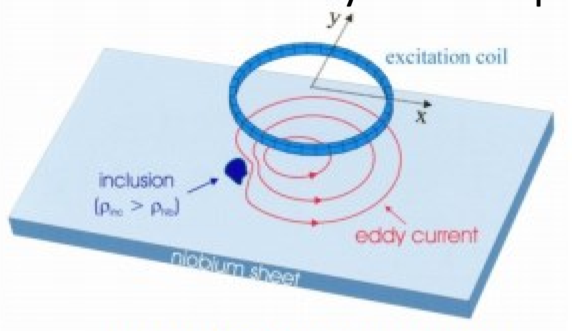
Nb Eddy Current Scanning

When an **AC current flows** in a coil in close **proximity** to a **conducting surface** the magnetic field of the coil will **induce circulating (eddy) currents** in that surface.

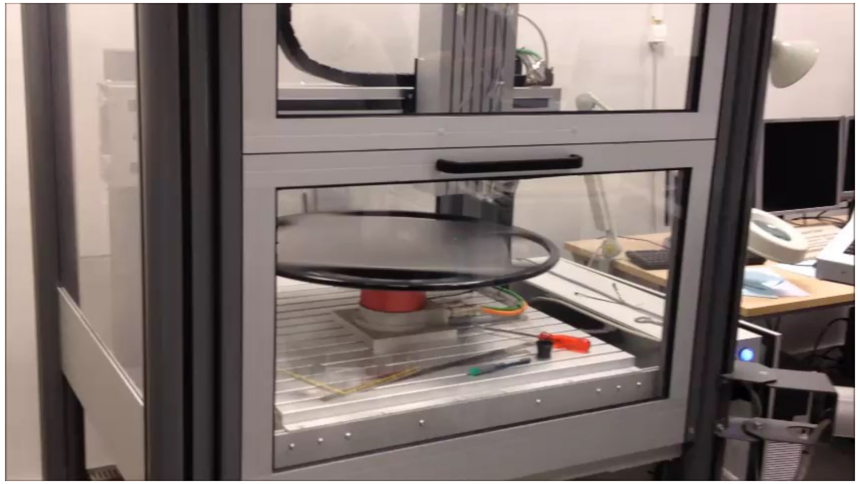
The **magnitude and phase** of the eddy currents will **affect the loading** on the coil and thus its impedance. If there is **a deep crack in the surface** immediately underneath the coil, it will **interrupt or reduce the eddy current** flow, thus decreasing the loading on the coil and increasing its effective impedance.

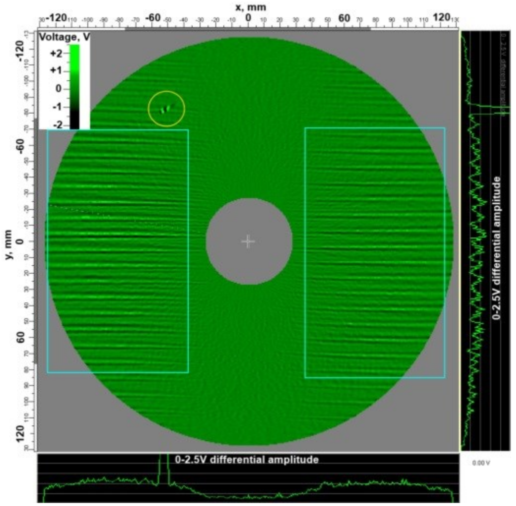
The operating frequency is between 100 kHz to few MHz and can span from the surface down into the materials for **some hundreds of microns**.

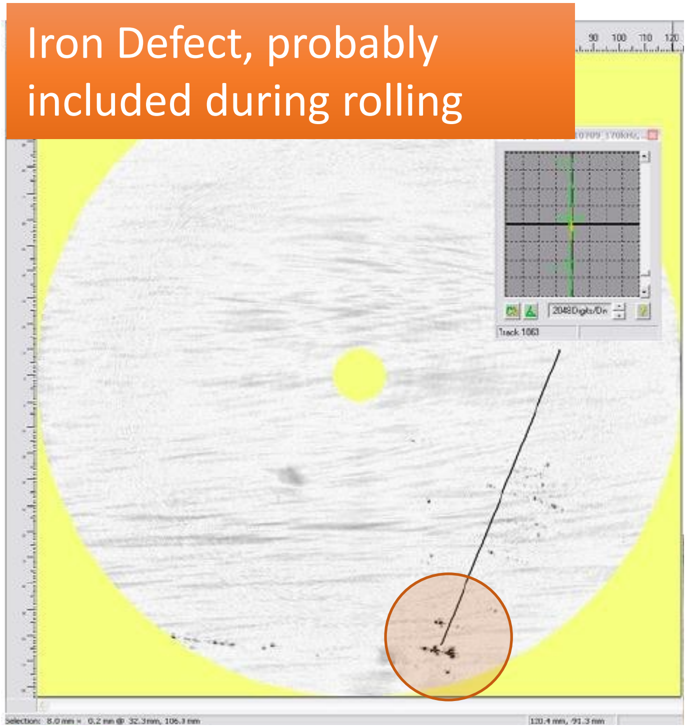
DESY Eddy Current principle



Principle of eddy current measurement

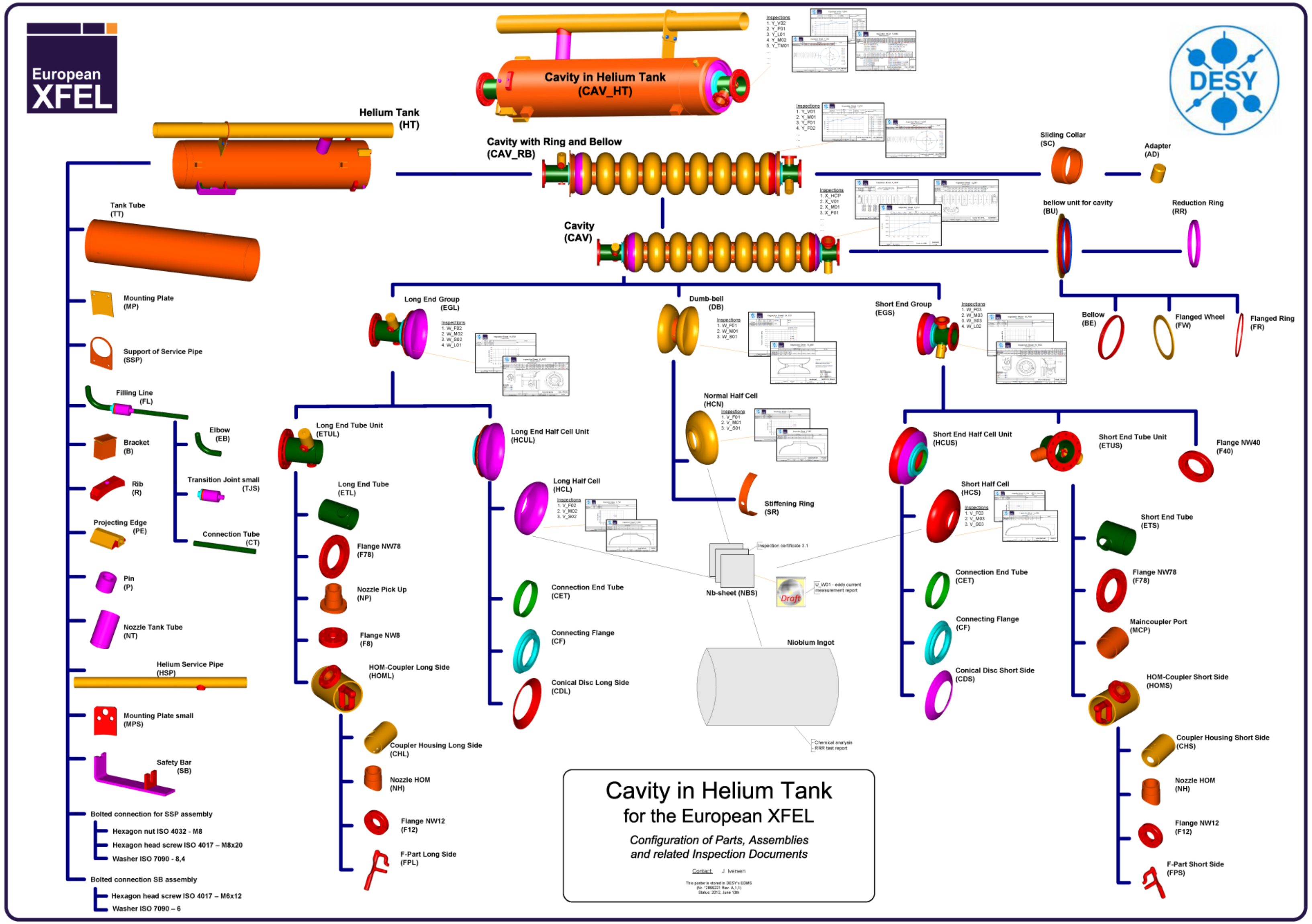




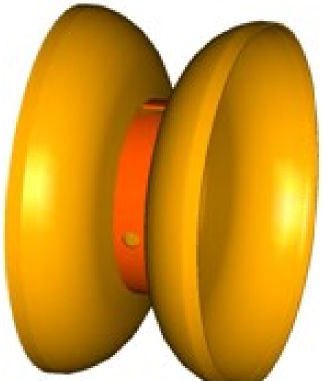


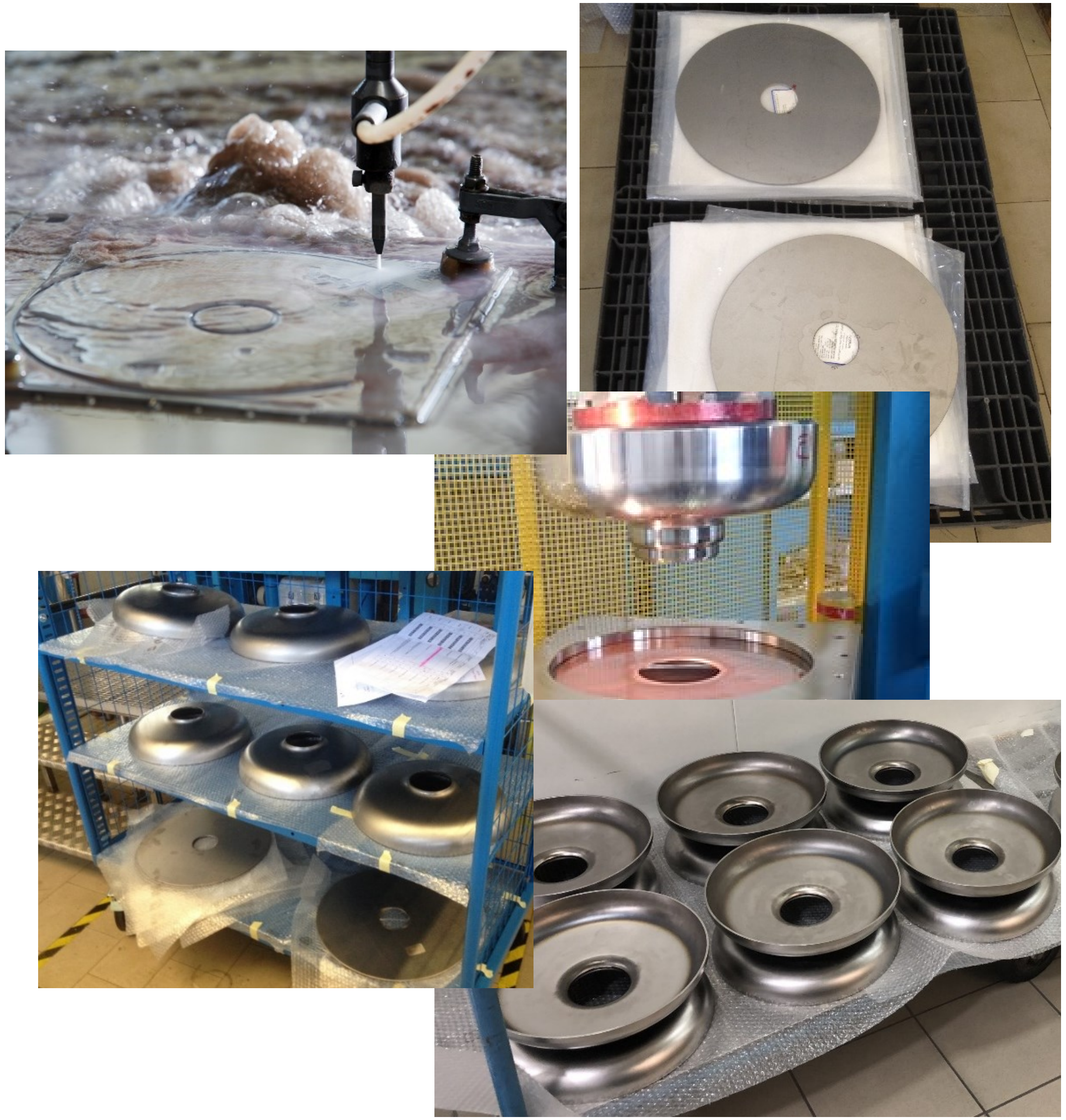
Nb bulk cavities: from sheets to cavity

Cavity components



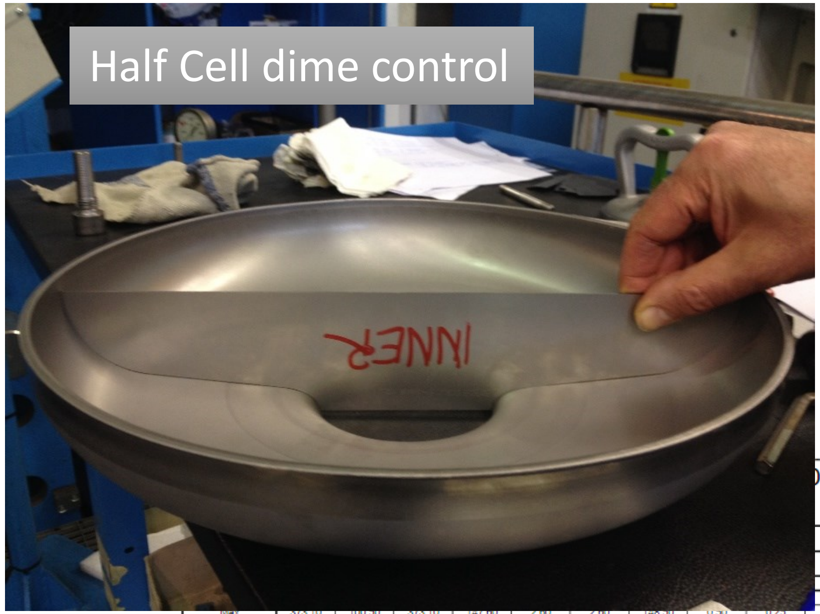
Dumb-bell Fabrication (an example)

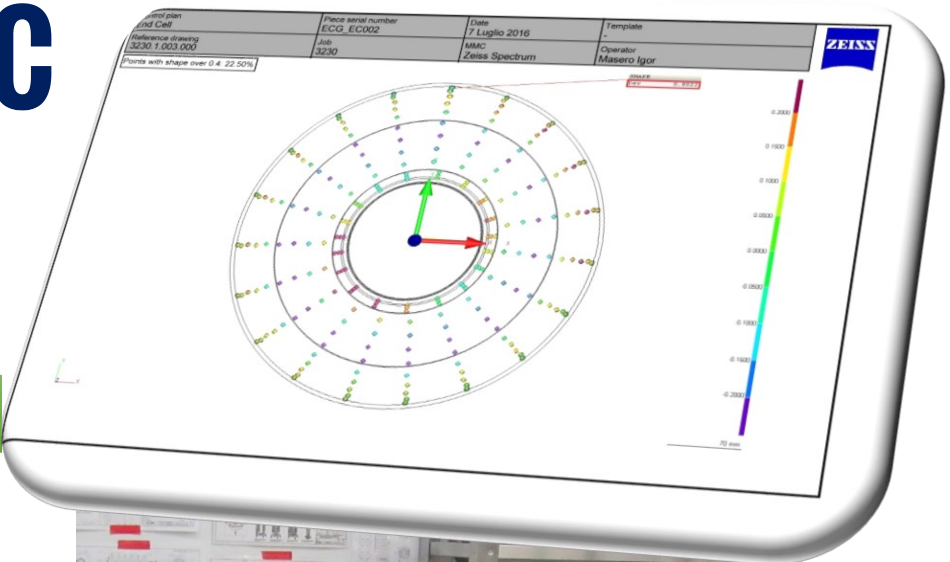




1. Nb sheets cutting
2. Deep drawing
3. Mechanical measurement
4. Cleaning (by ultrasonic cleaning +rinsing)
5. Trimming of iris region and reshaping of cups if needed
6. Cleaning
7. Rf measurement of cups
8. Buffered chemical polishing + rinsing (for welding of Iris)
9. Welding of Iris
10. Welding of stiffening rings
11. Mechanical measurement of dumb-bells
12. Reshaping of dumb bell if needed
13. Cleaning
14. Rf measurement of dumb-bell
15. Trimming of dumb-bells (Equator regions)
16. Cleaning
17. Intermediate chemical etching (BCP /20- 40 µm) + rinsing
18. Visual Inspection of the inner surface of the dumb-bell
19. Local grinding if needed + (second chemical treatment + inspection)

Mechanical QC





3D profile

E. ZANON

Doc No / N° Doc.	180929

										M10	M11	Remarks
										0.00	0.00	
Max	373.10	100.50	373.10	147.60	2.60	2.60	148.50	0.50	0.25	0.60	0.60	
0°	372.11	99.81	372.36	147.23	2.55	2.52	147.58	0.46	0.17	0.68	0.45	
90°	372.28	99.88	372.30	146.98	2.48	2.59	148.06					
180°				146.77	2.52	2.42						
270°				147.14	2.41	2.40						
Result	not good	good	not good	good	good	good	good	good	good	good	good	
Average	372.20	99.85	372.33	147.03	2.49	2.48	147.82	0.46	0.17	0.68	0.45	
Min-Max	0.17	0.07	0.06	0.46	0.14	0.19	0.48	0.00	0.00	0.00	0.00	

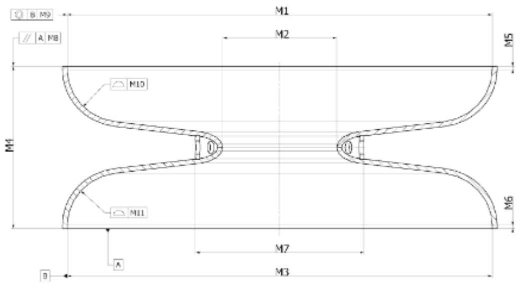
Instructions

Rose cells indicates measurements to be taken for each dimensions.

Keep the IC with the lower serial number always in the bottom position!

If shape 3D measurements are not done insert n.a. for M10 or M11

Shape 2D measurements: good or not good



Remarks on shape

M10 - points with shape between 0,6 and 0,8 (max 10%) [%]:

1.32

M11 - points with shape between 0,6 and 0,8 (max 10%) [%]:

0

Inner shape in tolerance?

	shape 2D	shape 3D
M10	good	good
M11	good	good

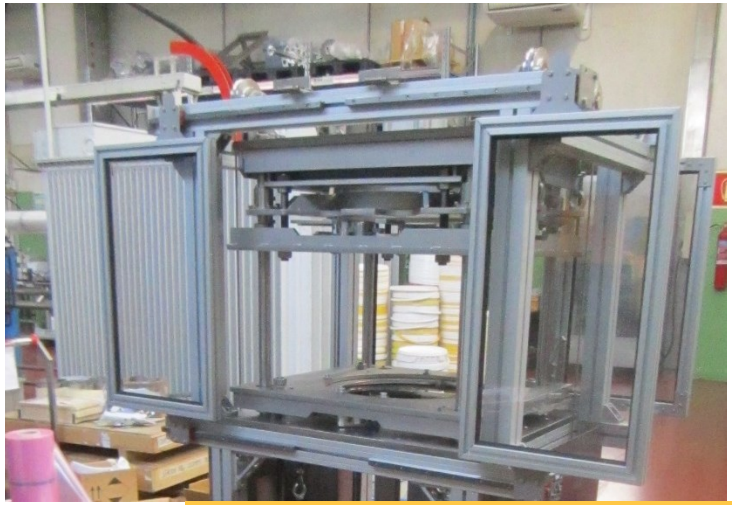
Inspection	10/1/2019 11:07	MARAGNO NICOLA
Description / Notes	Date	Name

Mechanical measurements

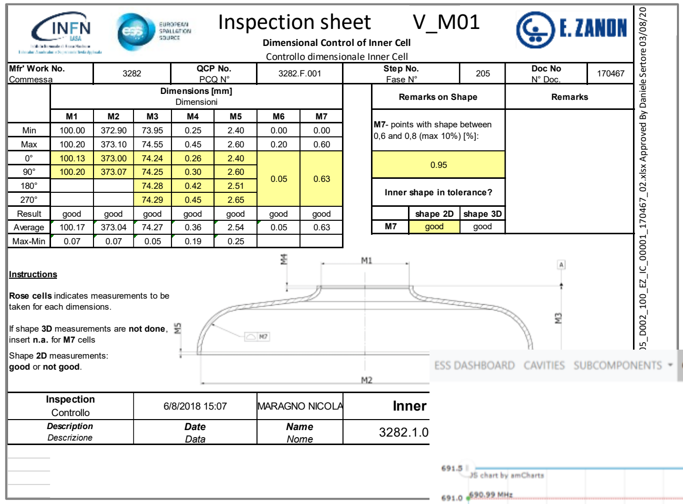
Visual Inspection

Frequency QC

Measure frequency and length to determine how much to **trim** at the equator to obtain **target frequency and length** of the cavity fully welded



Co.Co.De for
HC and DB RF measurements



Inspection sheet V_M01

Dimensional Control of Inner Cell

Controllo dimensionale Inner Cell

Mfr' Work No. Commessa	3282	QCP No. PCQ N°	3282.F.001	Step No. Fase N°	205	Doc No N° Doc.	170467

	M1	M2	M3	M4	M5	M6	M7
Min	100.00	372.90	73.95	0.25	2.40	0.00	0.00
Max	100.20	373.10	74.55	0.45	2.60	0.20	0.60
0°	100.13	373.00	74.24	0.26	2.40	0.05	0.63
90°	100.20	373.07	74.25	0.30	2.60		
180°			74.28	0.42	2.51		
270°			74.29	0.45	2.65		
Result	good	good	good	good	good	good	good
Average	100.17	373.04	74.27	0.36	2.54	0.05	0.63
Max-Min	0.07	0.07	0.05	0.19	0.25		

Dimensions [mm] Dimensioni

Remarks on Shape

M7- points with shape between 0,6 and 0,8 (max 10%) [%]: 0.95

Inner shape in tolerance?

	shape 2D	shape 3D
M7	good	good

Remarks

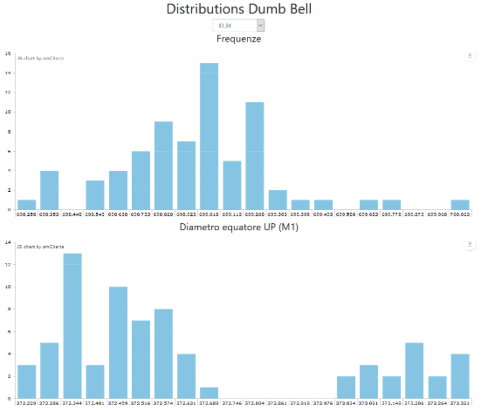
Instructions

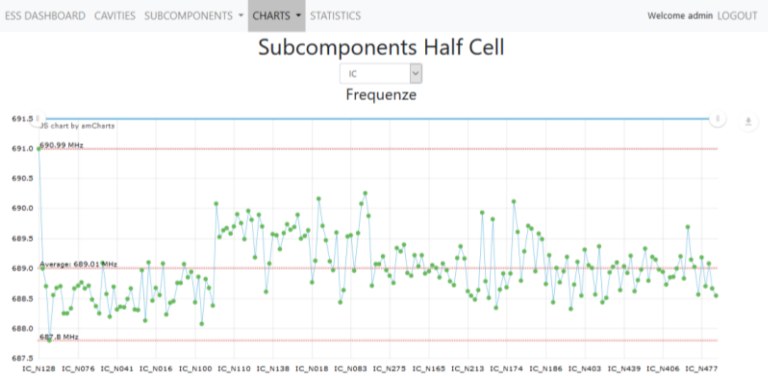
Rose cells indicates measurements to be taken for each dimensions.

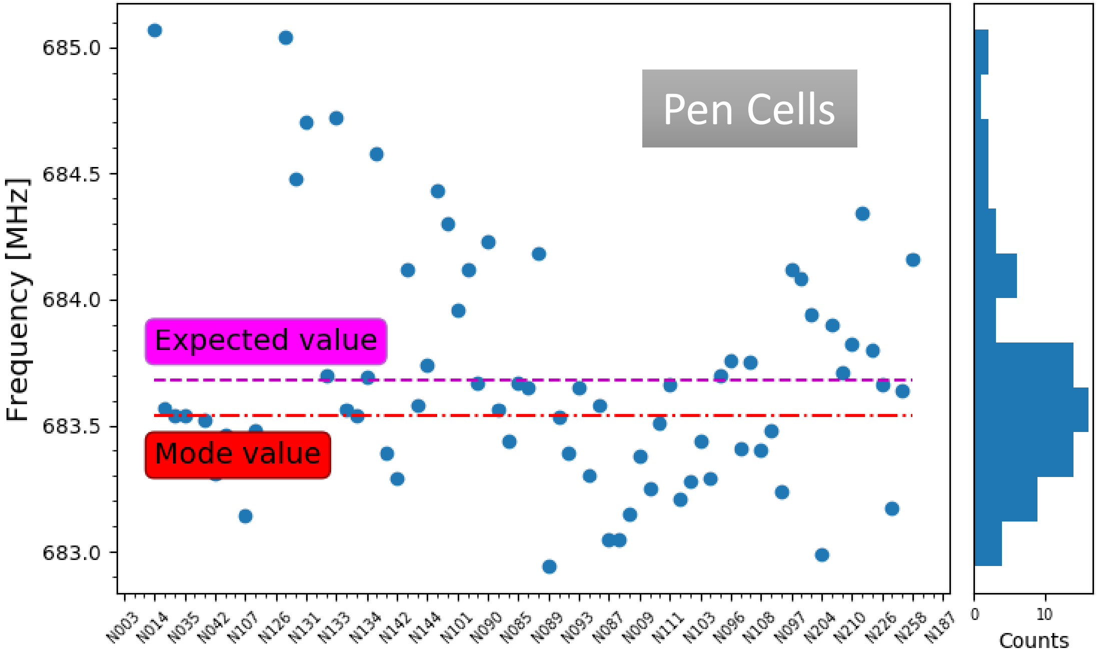
If shape **3D** measurements are **not done**, insert **n.a.** for **M7** cells

Shape 2D measurements: **good** or **not good**.

Inspection Controllo	6/8/2018 15:07	MARAGNO NICOLA	Inner
Description Descrizione	Date Data	Name Nome	3282.1.0

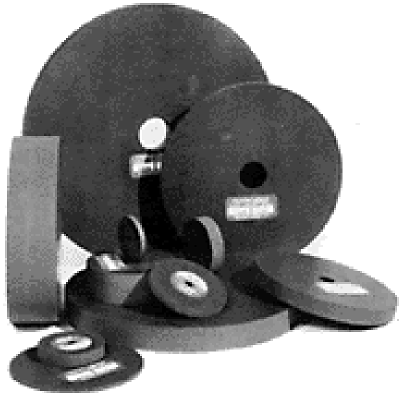






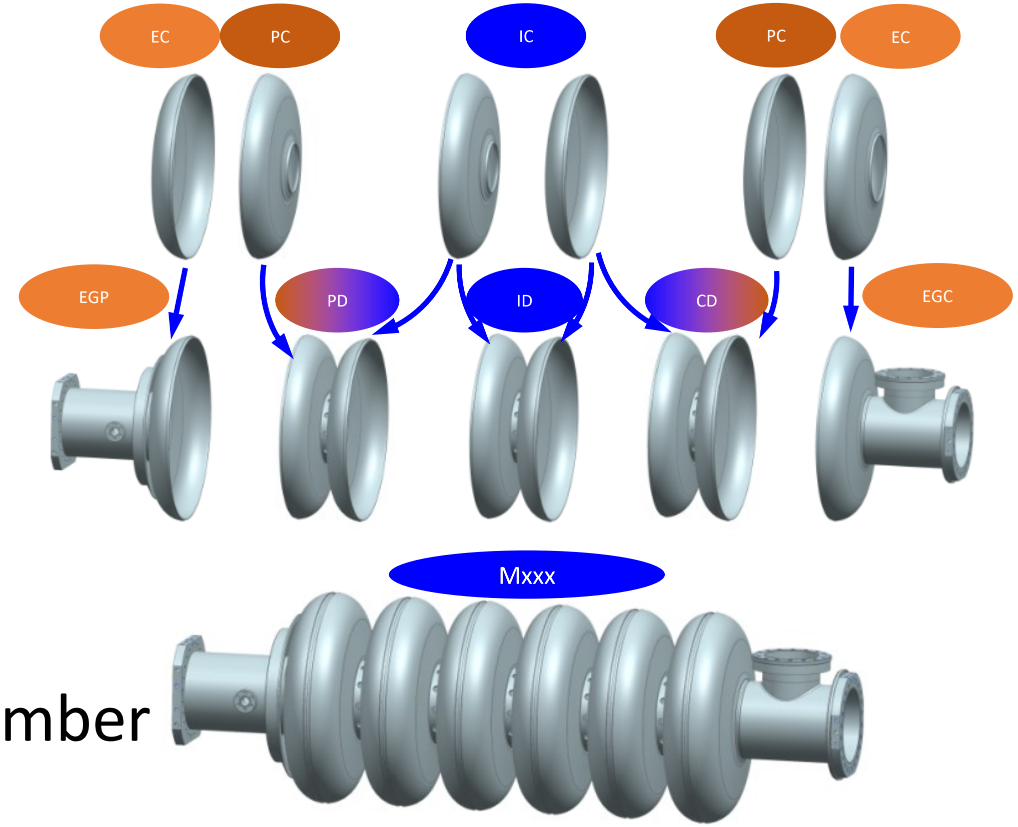
Mechanical Grinding

Mechanical grinding of visible **local defects** (deeper than 15 μm for EXFEL) with aluminum oxide grinding discs or rubberized abrasive (CRATEX®)



Rubberized abrasive

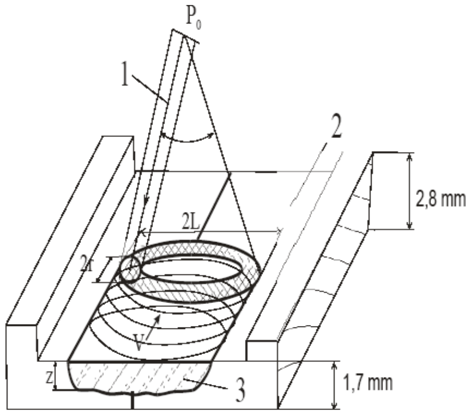
Cavity parts EB welding



- Degreasing and rinsing of parts
- Drying under clean condition
- Chemical etching at the welding area (equator)
- Careful and intensive rinsing with Ultra Pure Water
- Dry under clean conditions
- Install parts to fixture under clean conditions
- Install parts into Electron Beam (EB) welding chamber (**no contamination on the weld area allowed**)
- Pump down to vacuum in the EBW chamber in the 10^{-5} mbar range
- Welding and cool down of Nb to T < 150 °C, then venting with N_2
- Leak check of welds

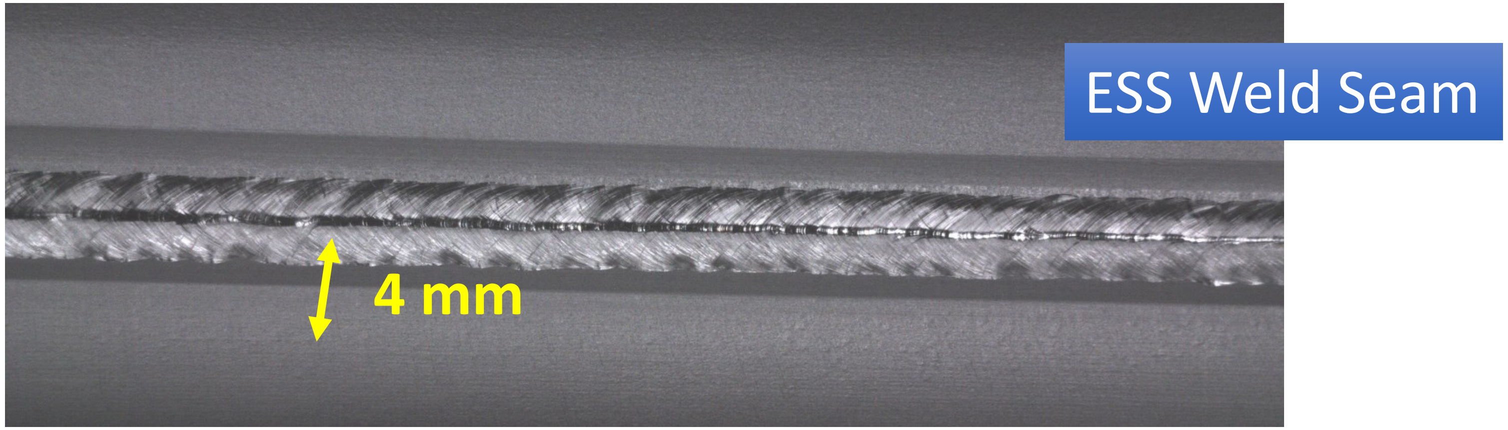
Electron Beam Welding

- **Welding under good vacuum**, 10^{-5} mbar range
- Broad welding seam
  - Operate with defocussed beam
  - Smooth underbead
- **Overlap at end of welding** to avoid accumulation of impurities
- **Wait to cool down before opening chamber**



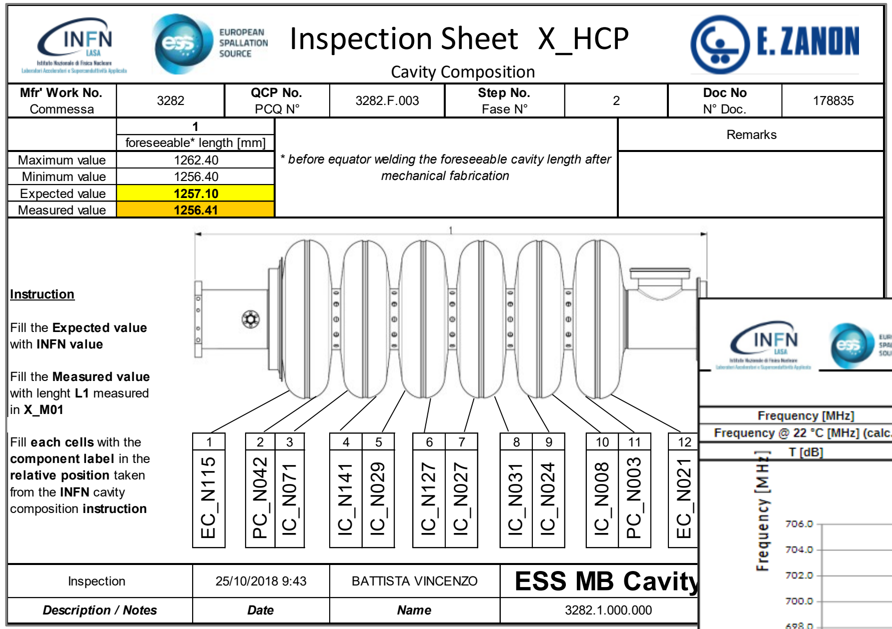
Welding Scheme (circular raster)

1. Electron beam (P_0-power of the beam, r-spot radius on the surface, L-scanning amplitude, V-velocity of the beam movement)
2. Nb sheet
3. melting zone (z-depth of the melting zone)



Mechanical and frequency QC

After EBW, the cavity is mechanical measured, inner inspected and the frequency is controlled


Inspection Sheet X_HCP

Cavity Composition

Mfr' Work No. Commessa	3282	QCP No. PCQ N°	3282.F.003	Step No. Fase N°	2	Doc No N° Doc.	178835

	1 foreseeable* length [mm]		Remarks
Maximum value	1262.40	* before equator welding the foreseeable cavity length after mechanical fabrication	
Minimum value	1256.40		
Expected value	1257.10		
Measured value	1256.41		

Instruction

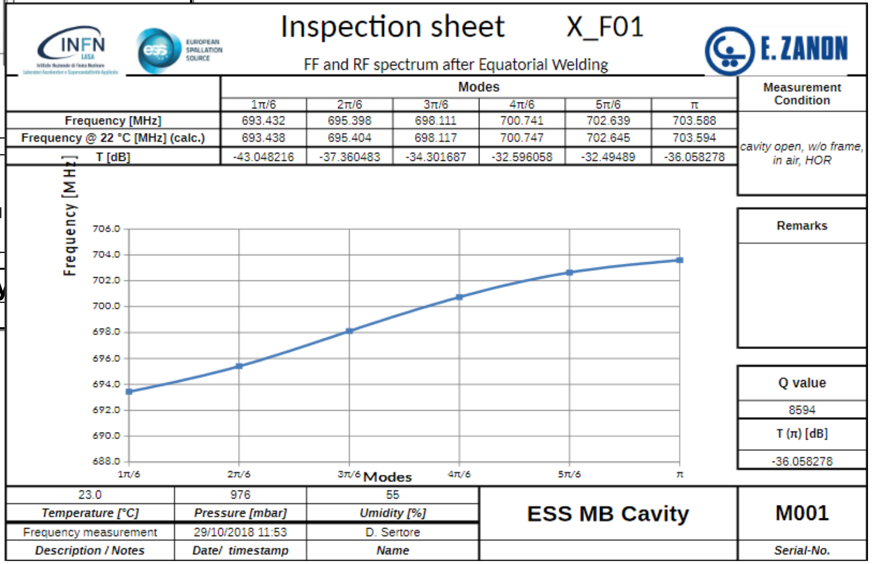
Fill the **Expected value** with **INFN value**

Fill the **Measured value** with lenght **L1** measured in **X_M01**

Fill **each cells** with the **component label** in the **relative position** taken from the **INFN** cavity composition **instruction**

1	2	3	4	5	6	7	8	9	10	11	12
EC_N115	PC_N042	IC_N071	IC_N141	IC_N029	IC_N127	IC_N027	IC_N031	IC_N024	IC_N008	PC_N003	EC_N021

Inspection	25/10/2018 9:43	BATTISTA VINCENZO	ESS MB Cavity
Description / Notes	*Date*	*Name*	3282.1.000.000


Inspection sheet X_F01

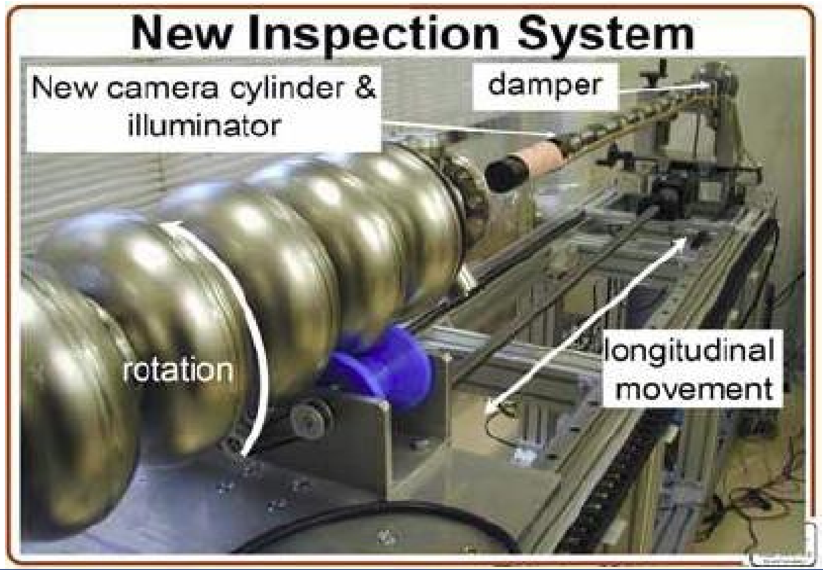
FF and RF spectrum after Equatorial Welding

	Modes 1π/6	2π/6	3π/6	4π/6	5π/6	π	Measurement Condition
Frequency [MHz]	693.432	695.398	698.111	700.741	702.639	703.588	*cavity open, w/o frame, in air, HOR*
Frequency @ 22 °C [MHz] (calc.)	693.438	695.404	698.117	700.747	702.645	703.594	
T [dB]	-43.048216	-37.360483	-34.301687	-32.596058	-32.49489	-36.058278	

Remarks

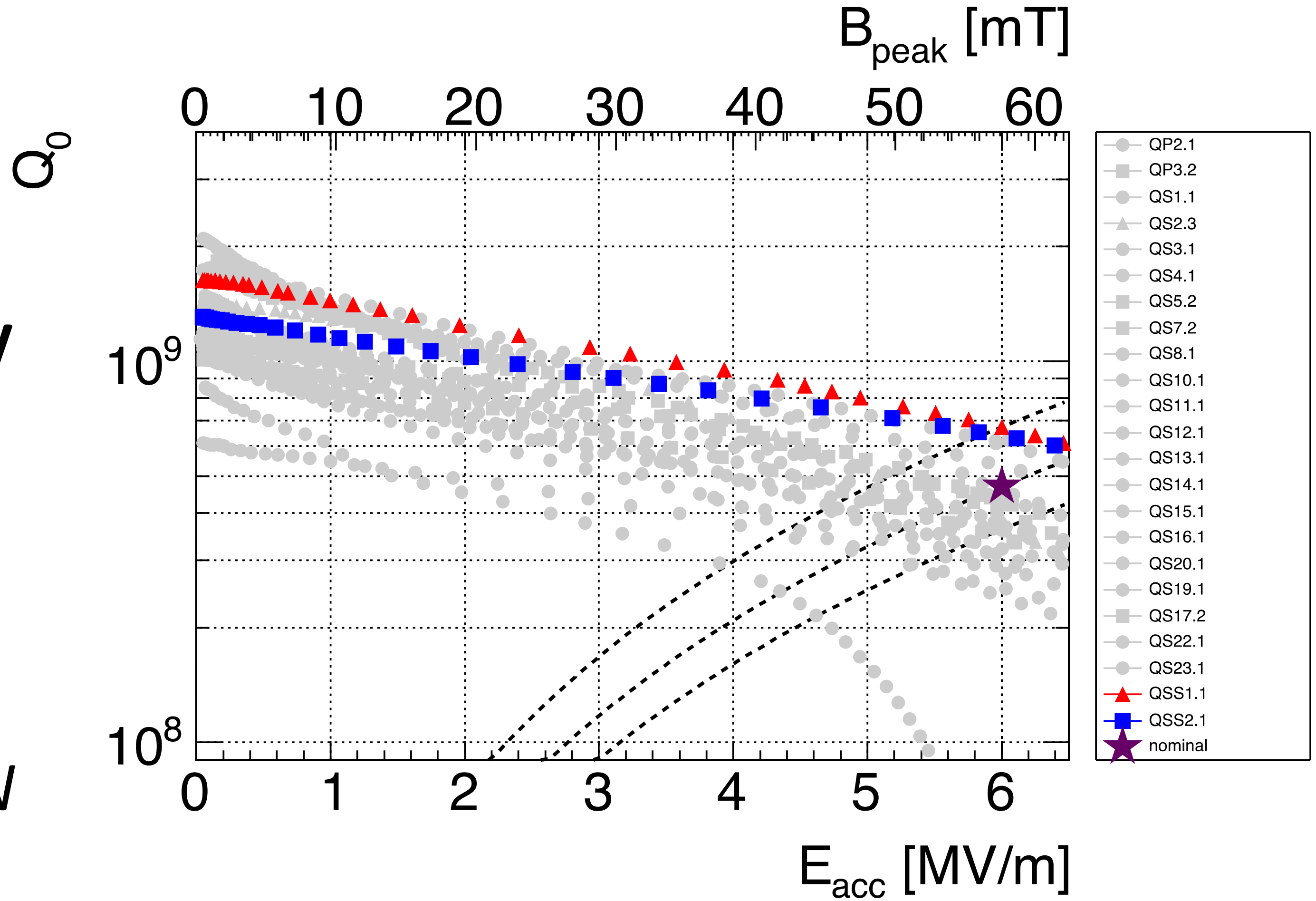
Q value
8594
T (π) [dB]
-36.058278

23.0	976	55	ESS MB Cavity	M001
Temperature [°C]	*Pressure [mbar]*	*Umidity [%]*		
Frequency measurement	29/10/2018 11:53	D. Sertore		
Description / Notes	*Date/ timestamp*	*Name*		*Serial-No.*



Advantages of seamless cavities

- **Cheaper**
- **Avoid defects and irregularity** of welding seams
- **Increase RF performances** *(real examples of ALPI @ INFN and HIE-ISOLDE @ CERN)*



HIE ISOLDE two seamless cavities performance at 4.5 K

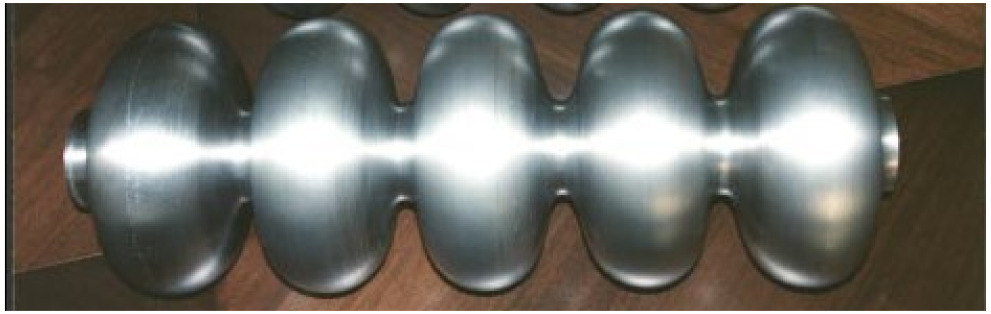
Courtesy of Walter Venturini

Seamless cavities by spinning

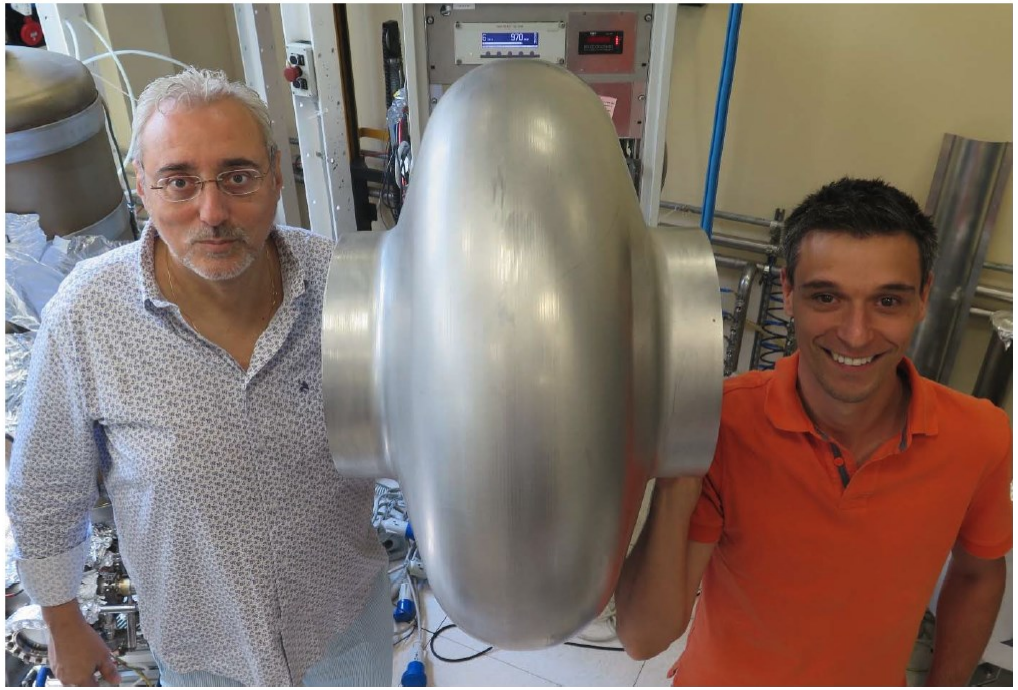
Hydroforming, explosive forming, **electroforming**, **electrodeposition** and **spinning** are the principal techniques explored for the production of seamless elliptical cavities

LNL have a long experience in spinning of 1,3 and 1,5 GHz elliptical cavities

Process invented and developed by Enzo Palmieri in the 90's



First seamless multicell by spinning



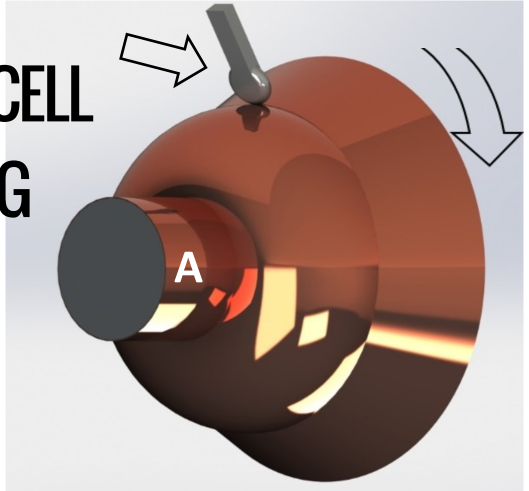
400MHz Seamless Aluminum Cavity prototype (2017)

Spinning production steps

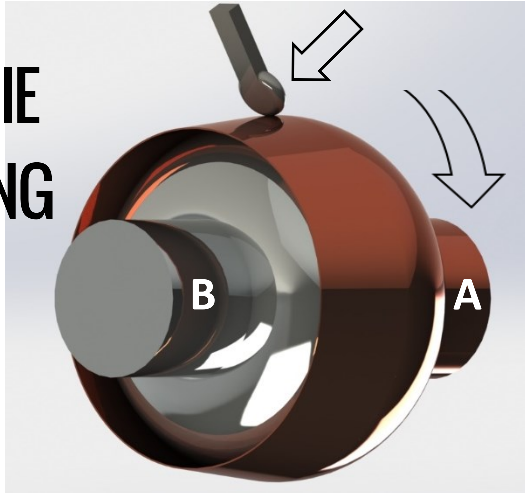
Step 1
COPPER PLATE PREPARATION

Step 2
DEEP DRAWING

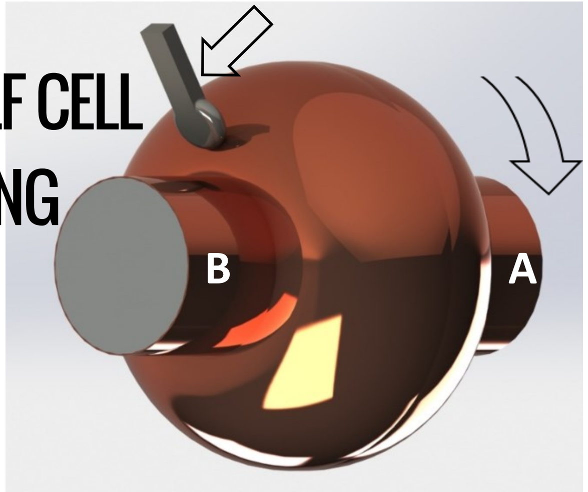
Step 3
1st HALF CELL SPINNING



Step 4
CONE DIE SPINNING

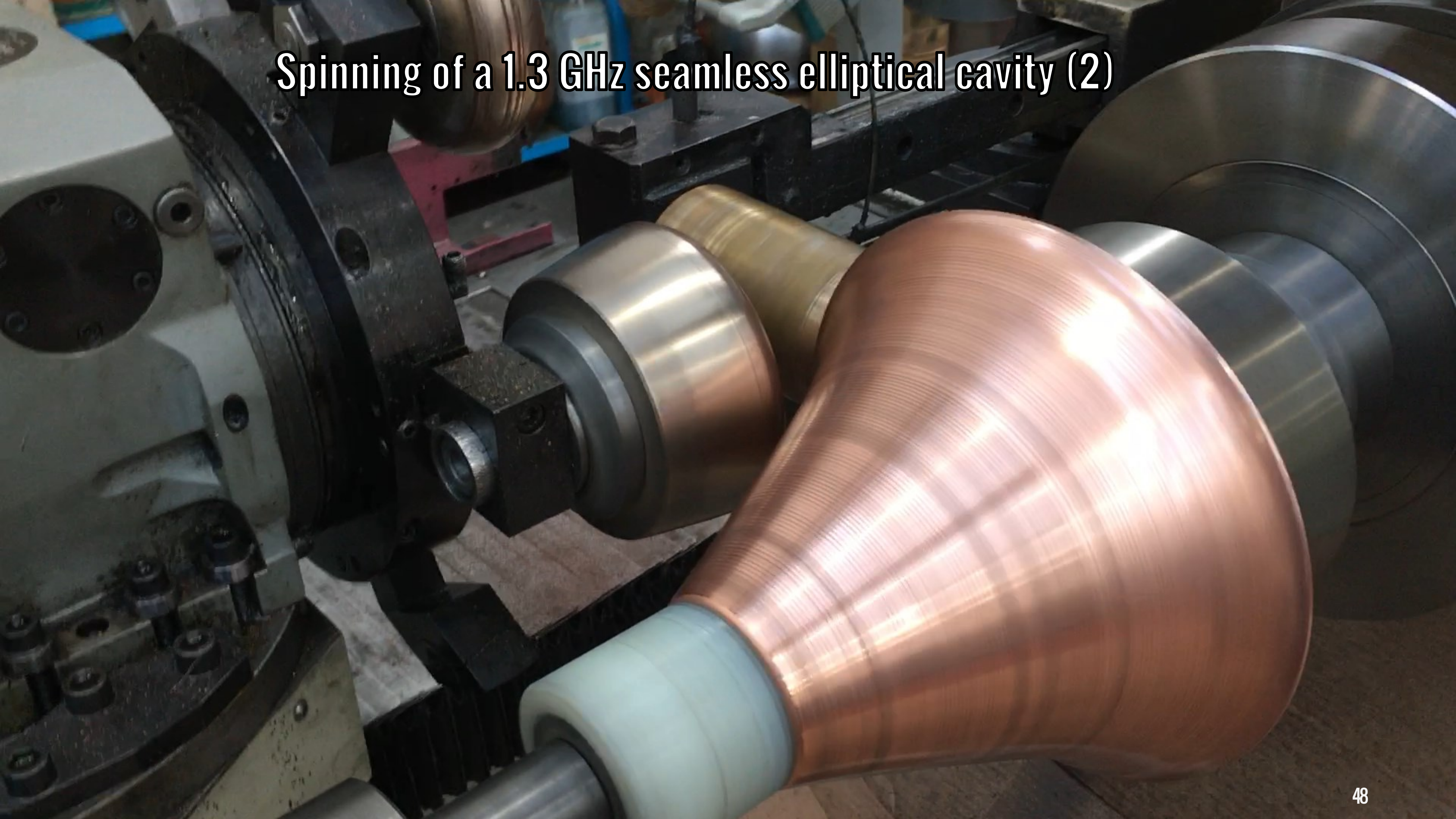


Step 5
2nd HALF CELL SPINNING

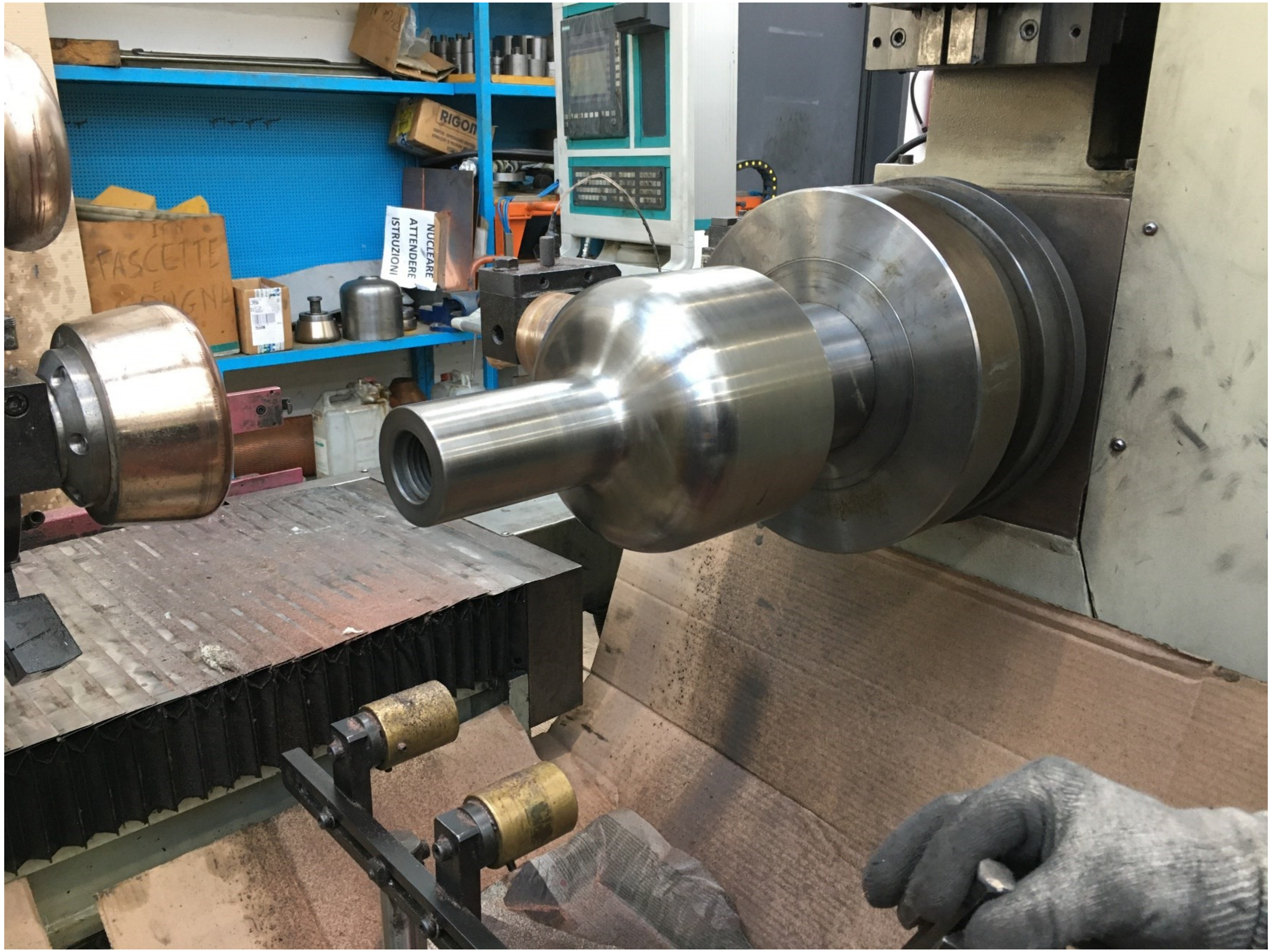


Spinning of a 1.3 GHz seamless elliptical cavity (1)

Spinning of a 1.3 GHz seamless elliptical cavity (2)



Spinning of a 1.3 GHz seamless elliptical cavity (3)

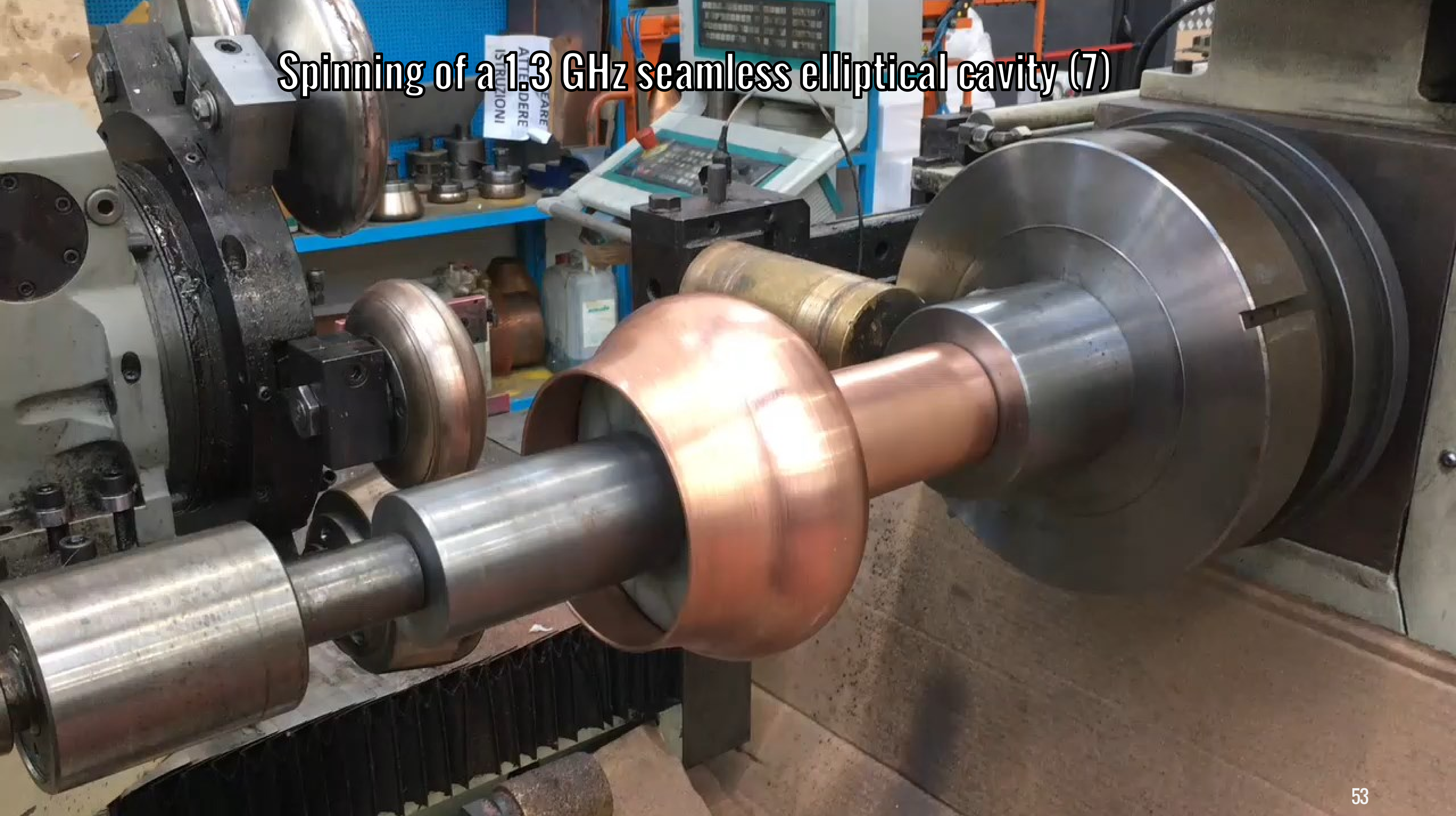


Spinning of a 1.3 GHz seamless elliptical cavity (4)

Spinning of a 1.3 GHz seamless elliptical cavity (5)

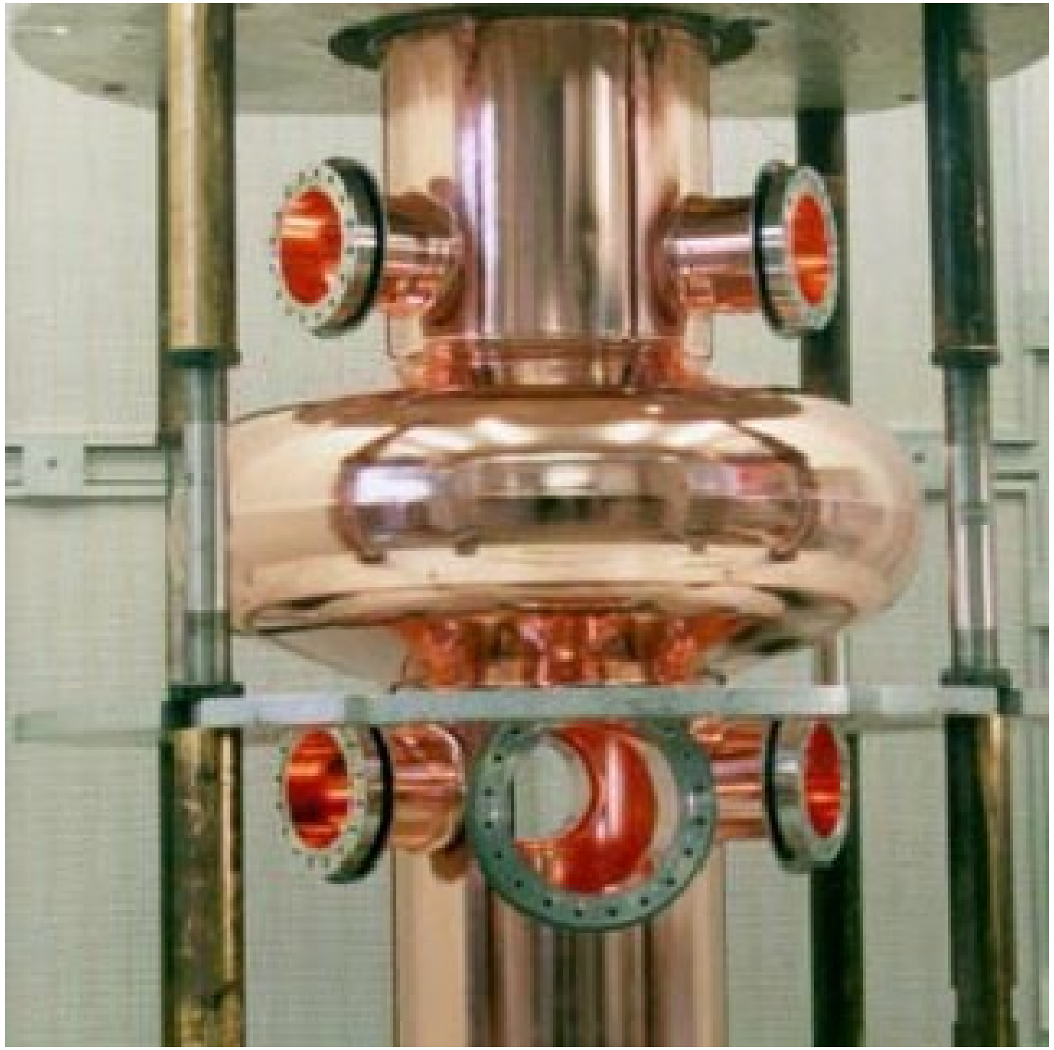
Spinning of a 1.3 GHz seamless elliptical cavity (6)

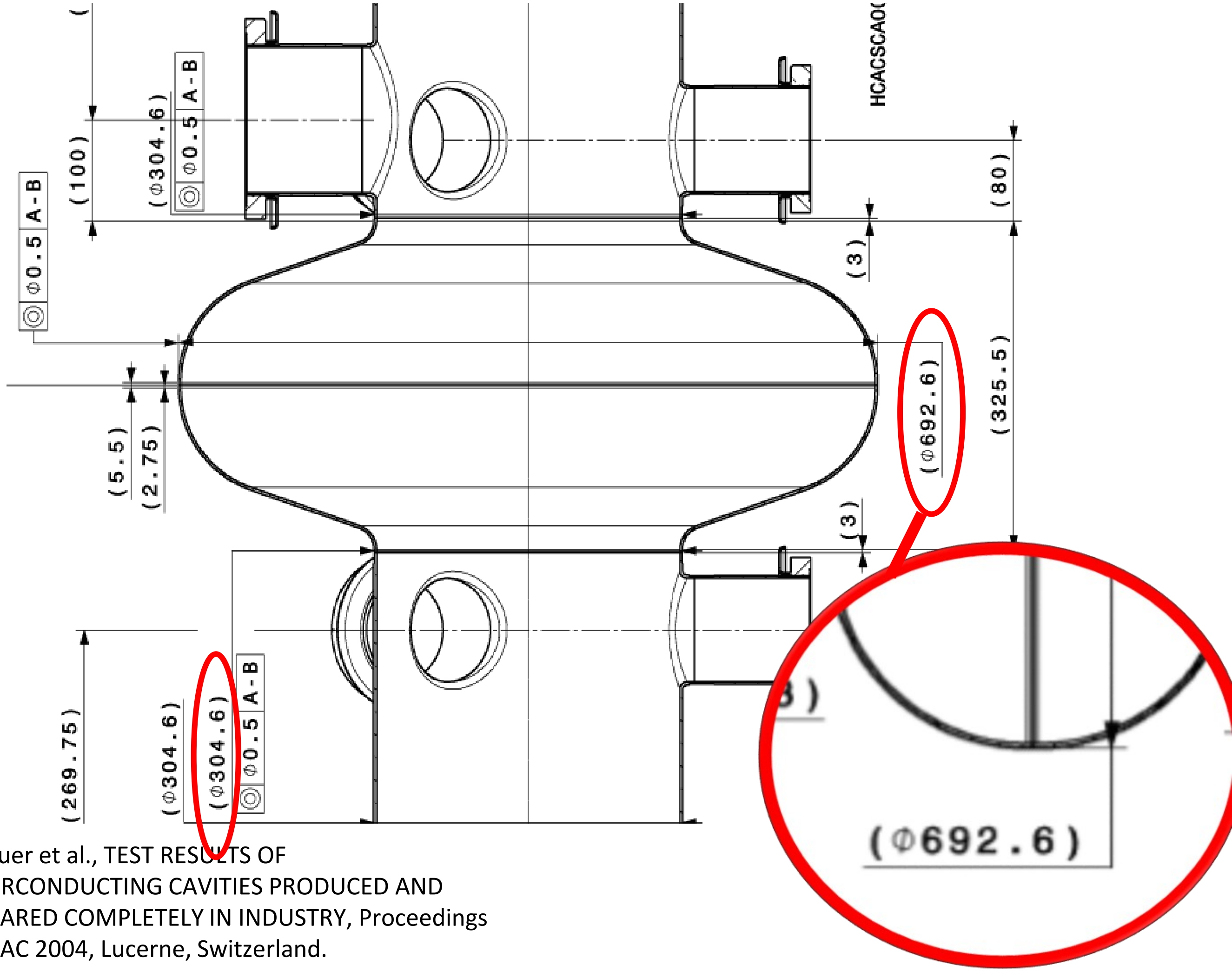
Spinning of a 1.3 GHz seamless elliptical cavity (7)



Spinning steps

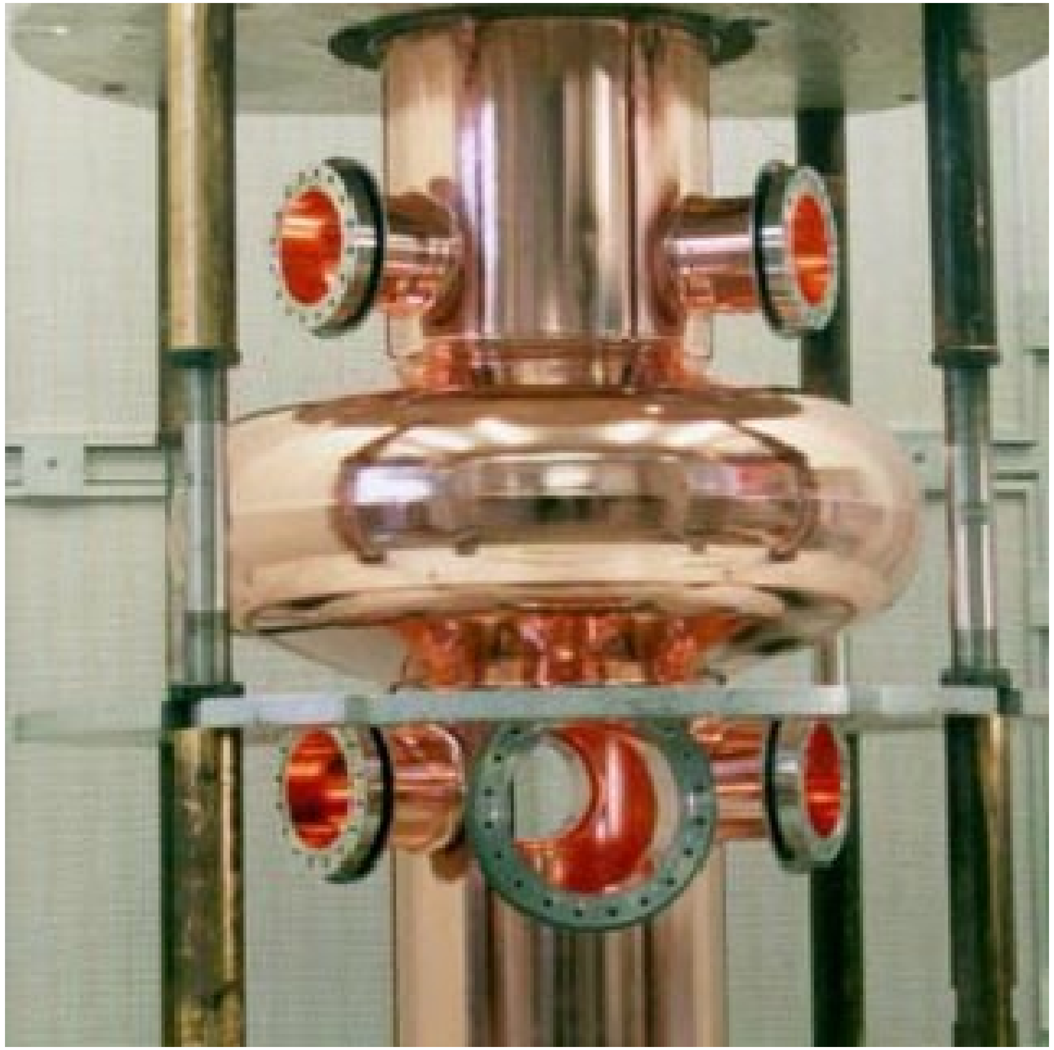
A 400 MHz eliptical cavity challenge (FCC studies)





S. Bauer et al., TEST RESULTS OF SUPERCONDUCTING CAVITIES PRODUCED AND PREPARED COMPLETELY IN INDUSTRY, Proceedings of EPAC 2004, Lucerne, Switzerland.

A 400 MHz eliptical cavity challenge (FCC studies)



STEP 1: OFE Copper sheet

- Dimension out of standard
- Difficulties to provide a cold rolled sheet
- **Thermal treated** (Hardness HV = 46)
- Received @ LNL on June 2018

Abnahmeprüfzeugnis DIN EN 10204-3.1
Inspection Certificate DIN EN 10204-3.1

mkm
we live copper

Besteller / Purchaser:

CERN
ORGANISATION POUR LA RECHERCHE

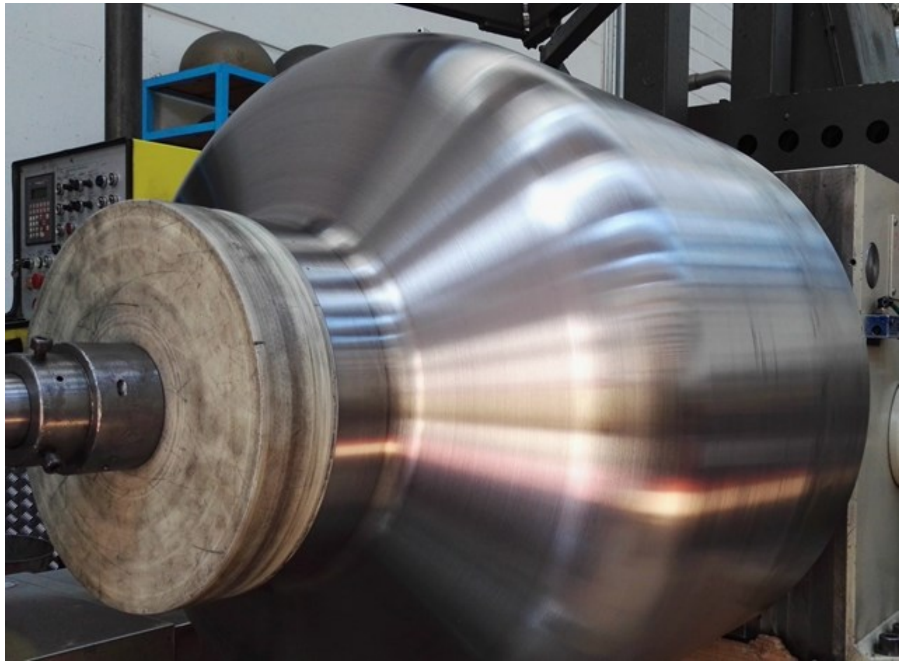
1211 Geneve
CH

MKM Mansfelder Kupfer und Messing GmbH
Lichtlöcherberg 40
06333 Hettstedt / Germany
Produktbereiche Bleche / Bänder
Überprüft nach AD 2000-Merkblatt W0/TRD100
durch den TÜV Hannover/Sachsen-Anhalt e.V.
Zertifiziert nach Richtlinie 97/23/EG durch die
TÜV CERT-Zertifizierungsstelle für Druckgeräte der
TÜV Nord Gruppe, Notifizierte Stelle, Kenn-Nr.: 0045
phone: +493476892473
fax: +493476892403
e-mail: Tom.Burchert@mkm.eu

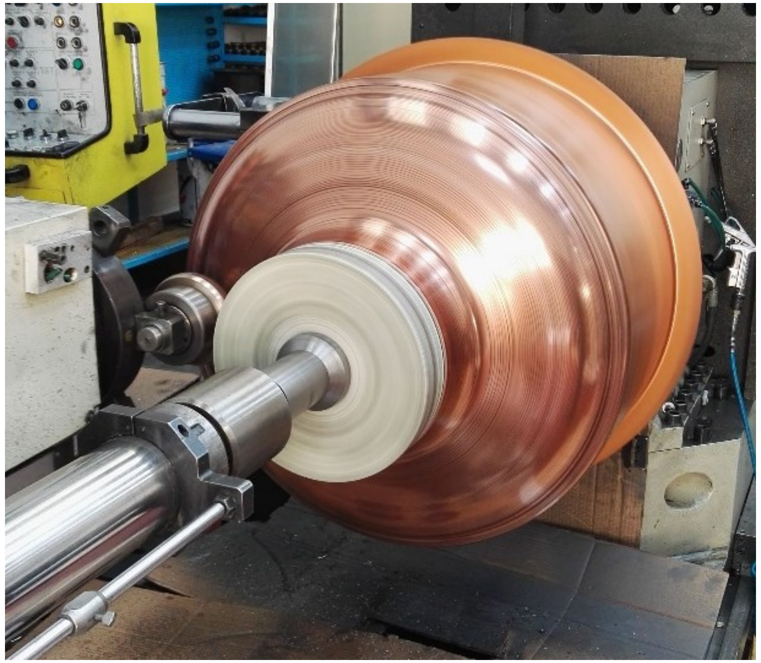
STEP 2 Deep Drawing

IRON DIES

STEP 3 Spinning of 1st Half Cell



IRON DIE



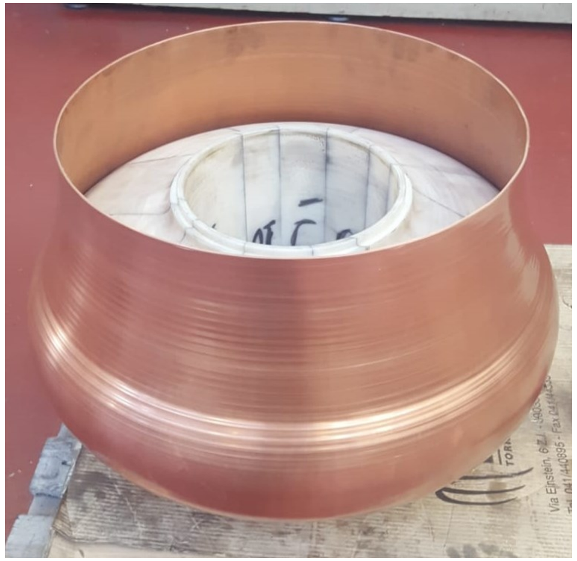
SPINNING OF THE FIRST HALF CELL

FIRST HALF CELL SPUN

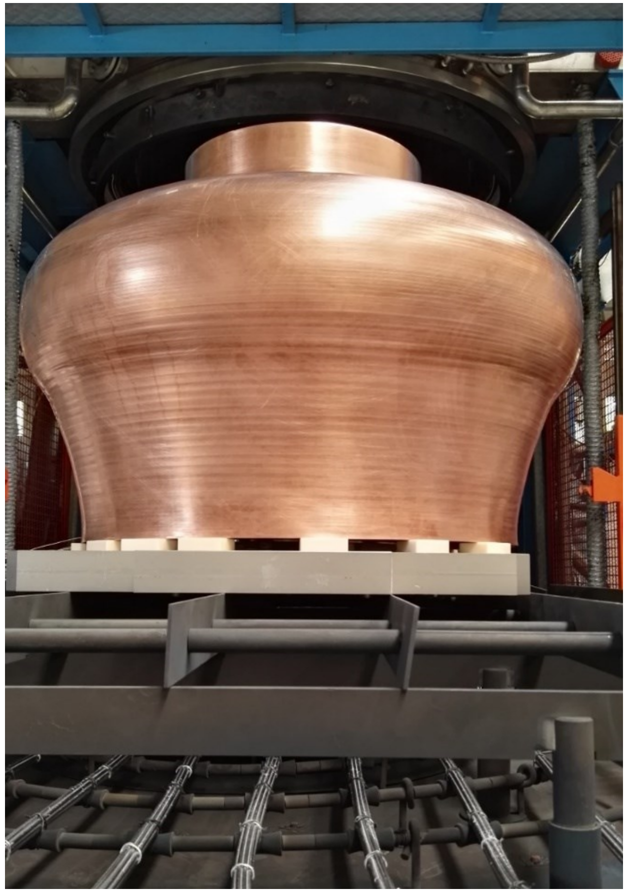
- No thermal treatments are necessary for the spinning of the 1° half cell

STEP 4 Spinning of the intermediate conical die

NYLON CONICAL DIE



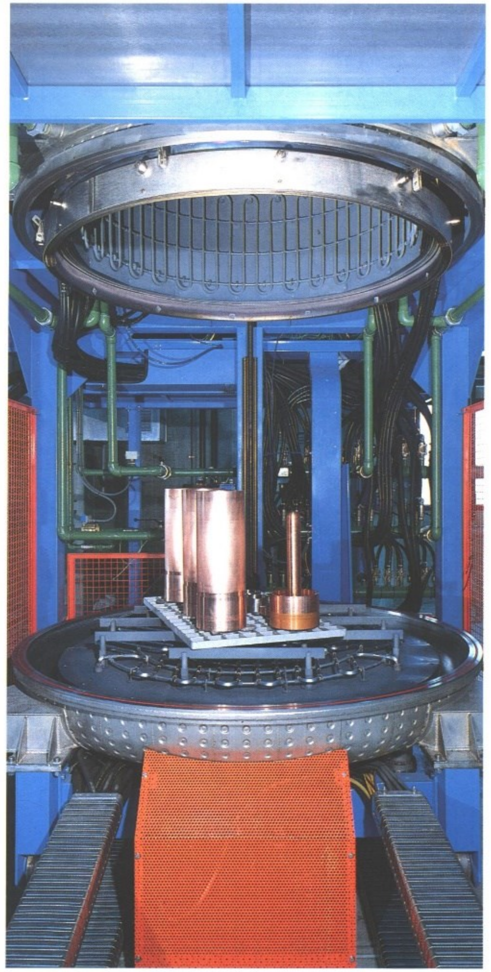
LOW SPINNABILITY



FIRST THERMAL ANNEALING

- A **thermal treatment is necessary** for the spinning of the conical die

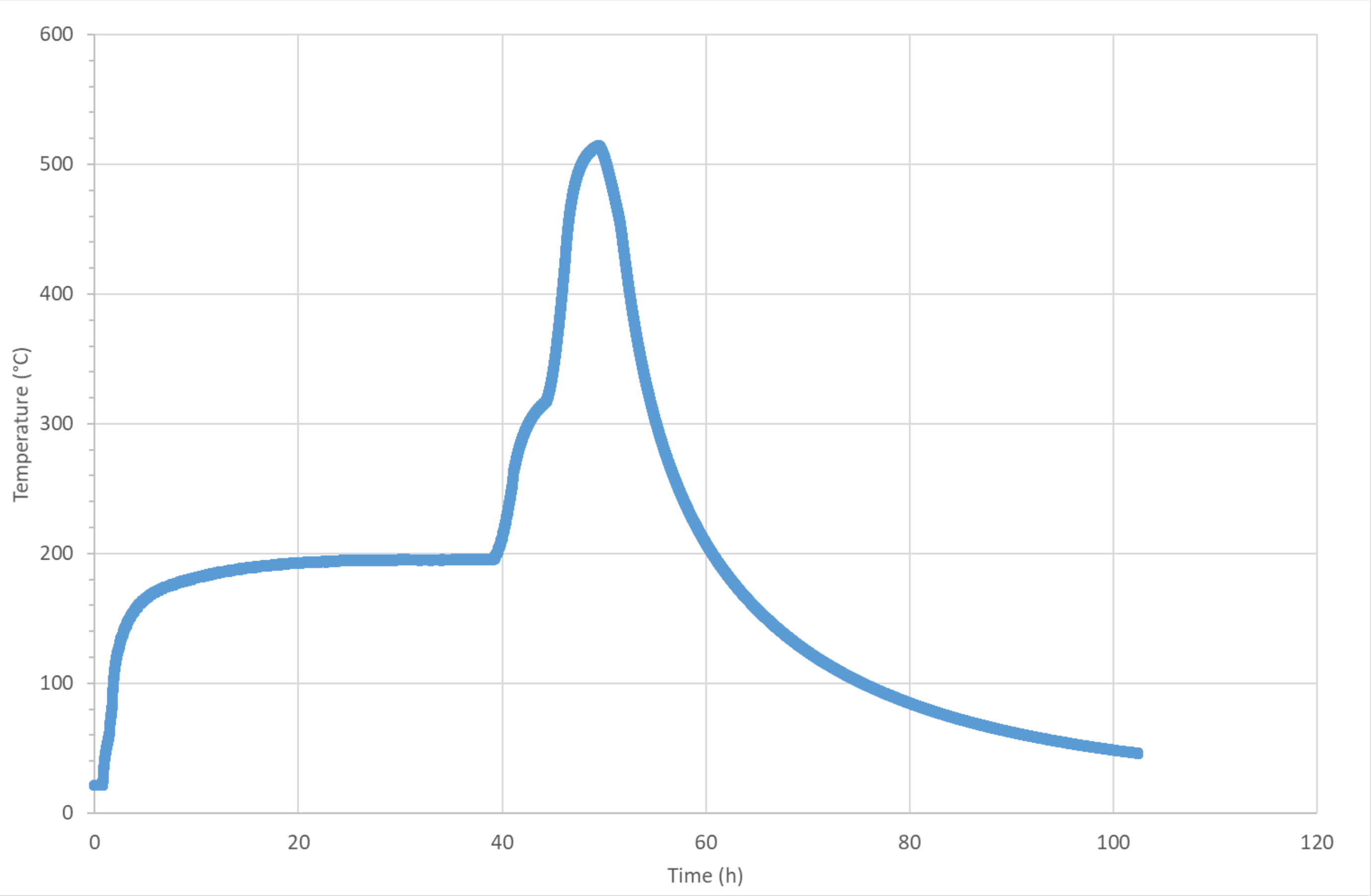
Thermal annealing



UHV Furnace @ LNL

- Maximum mass to be loaded: 1000 kg
- Nominal vacuum conditions for routine treatments: $5x10^{-5}$÷$1x10^{-6}$ mbar
- Lowest achievable base pressure: 10^{-7} mbar
- Maximum operational temperature: 1300°C
- Useful maximum diameter of components to be treated: 1300 mm
- Maximum height of components to be treated: 1600 mm
- Useful volume: around 2 m^3
- With height expansion (option): maximum height 2100 mm, maximum volume ~ m^3
- Access: vertical loading with lifted platform
- Quick load cooling by argon/nitrogen inlet and dedicated heat exchanger

Thermal annealing

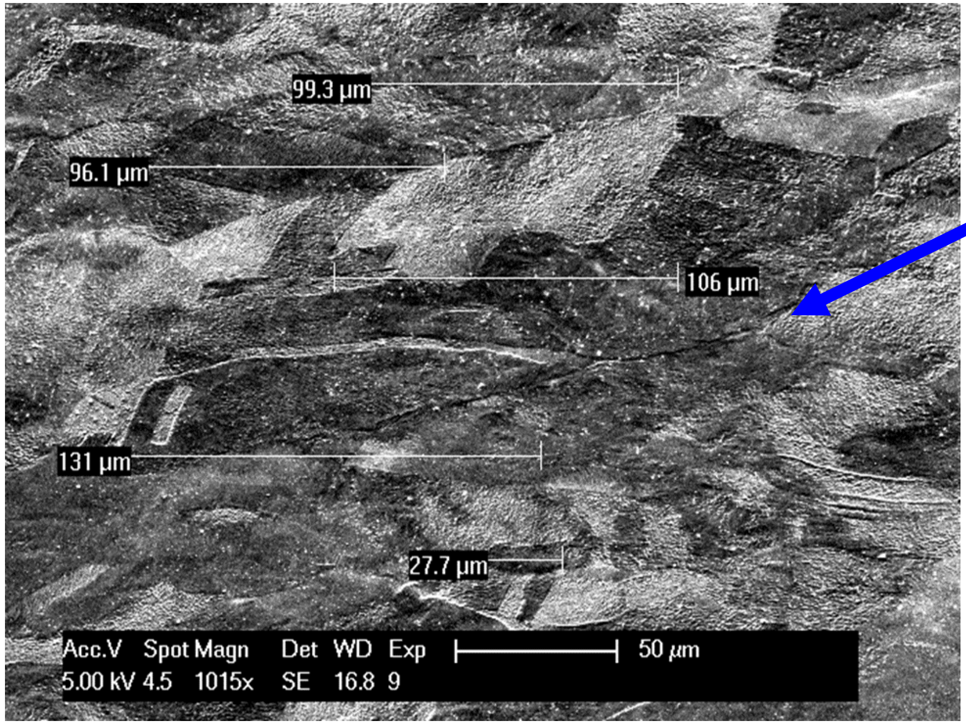


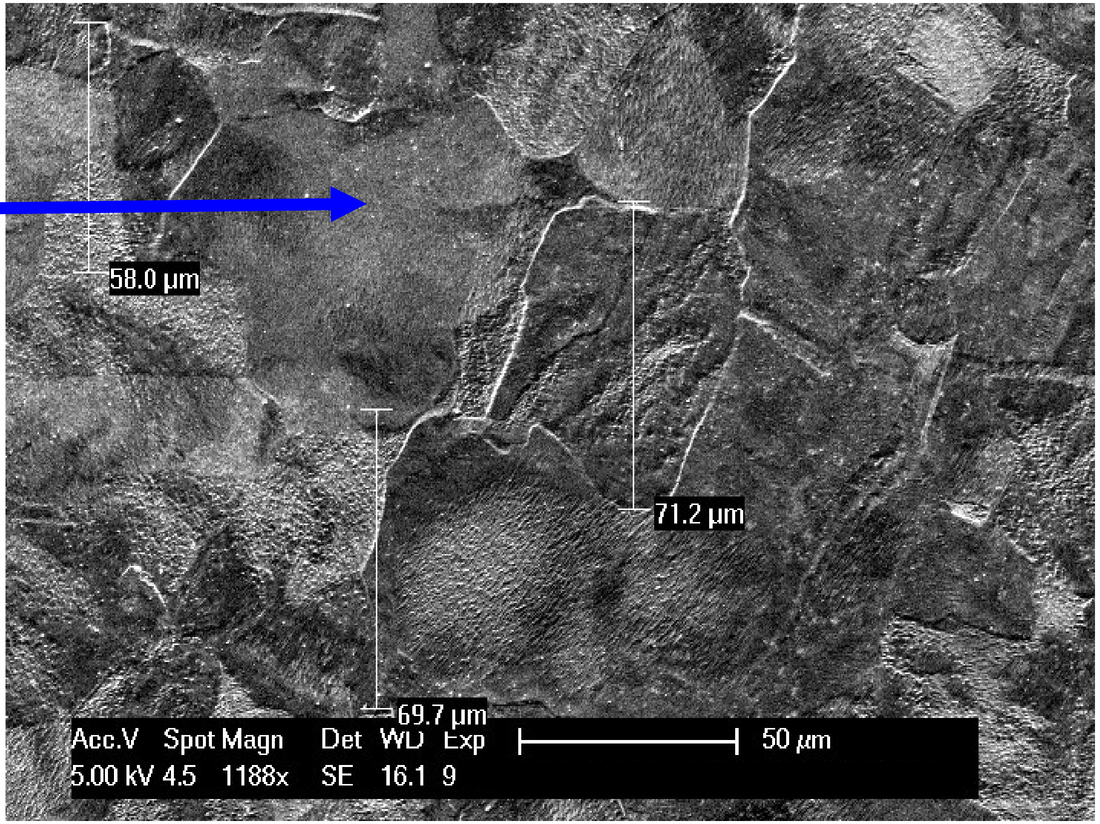
Thermal Cycle (4 Days):

- Degassing at 200 °C 24h
- Thermal treatment **at 300 °C** for 3h
- Thermal treatment **at 500 °C** for 2h

Grains Before Annealing (After Deep drawing)

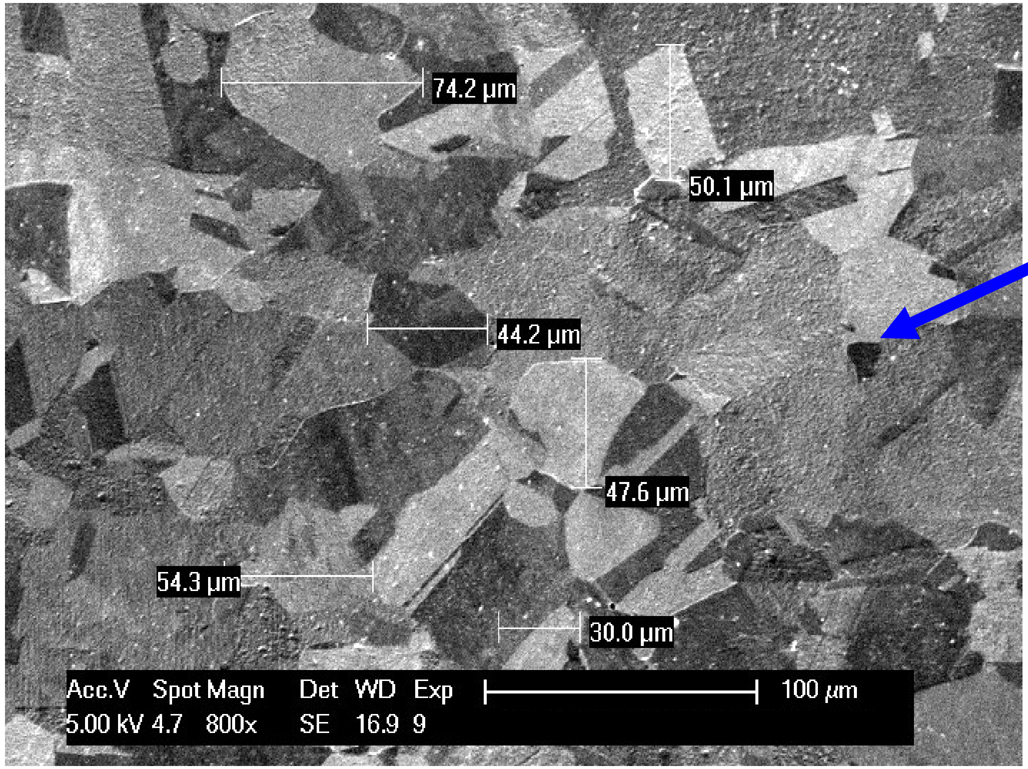
Grains Stretched

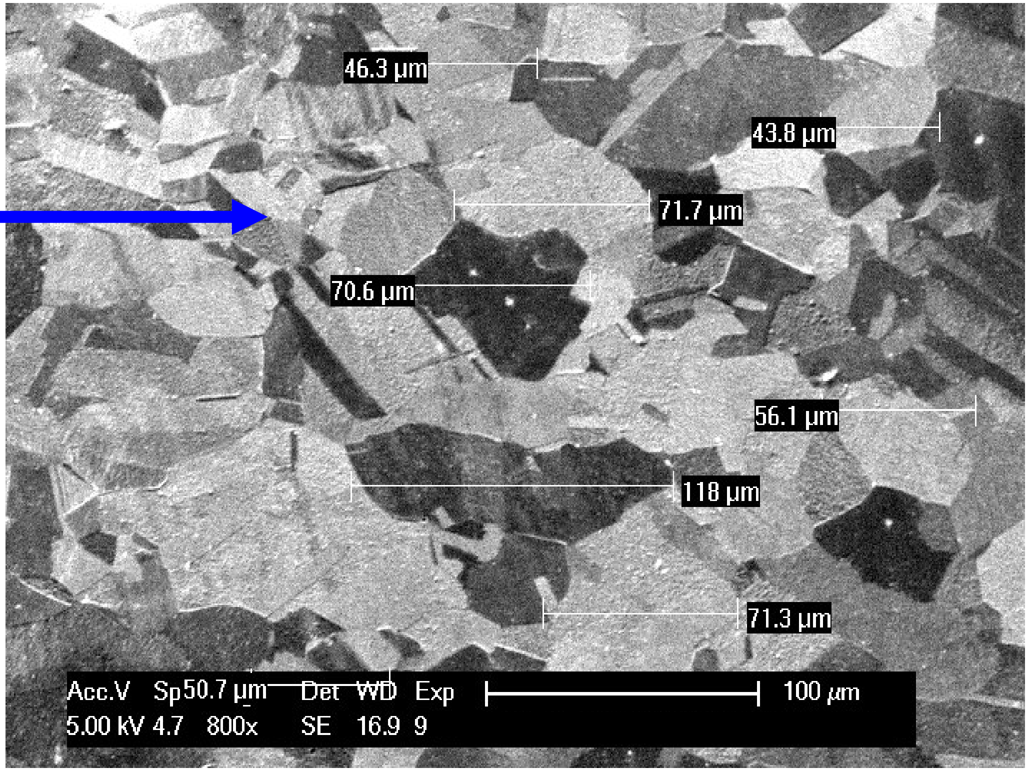




Grains After Annealing

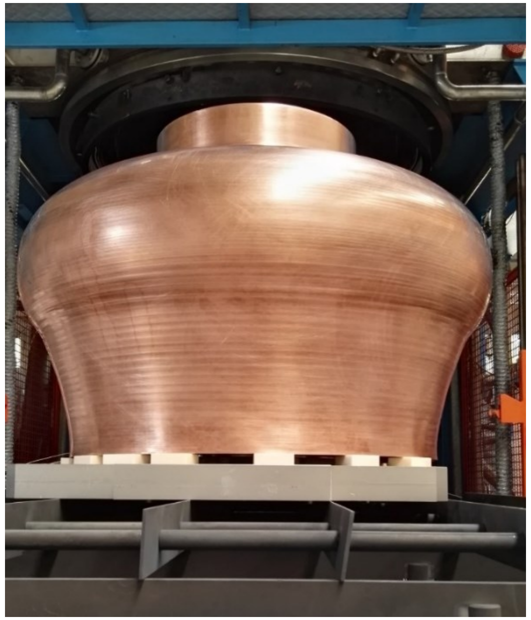
Grains not stretched





STEP 4 Spinning of the intermediate conical die

NYLON CELL DIE +
NYLON CONE DIE

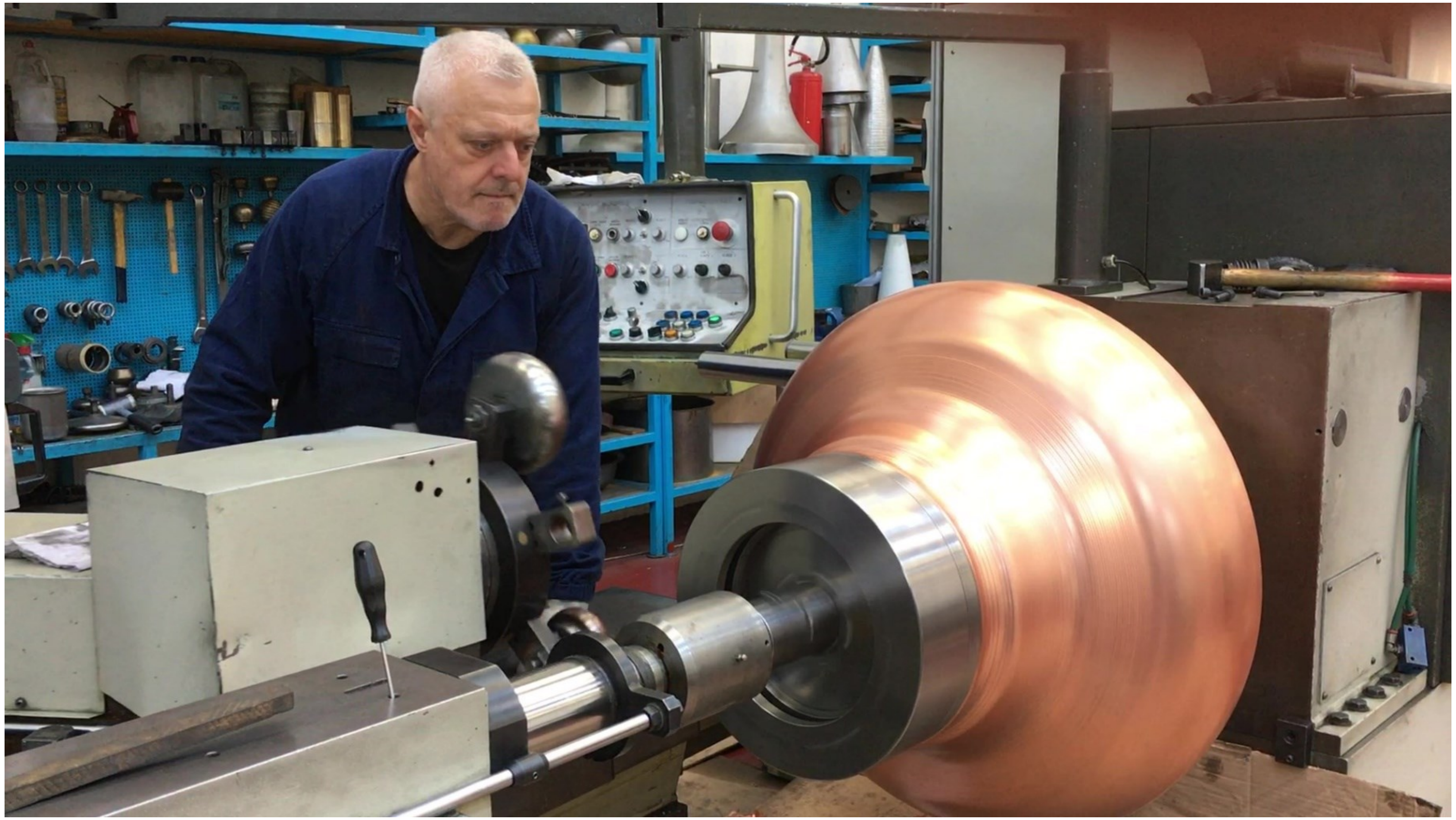


FIRST THERMAL ANNEALING

READY FOR THE SPINNING OF 2° HALF CELL

- **A thermal treatment is mandatory** for the spinning of the conical die

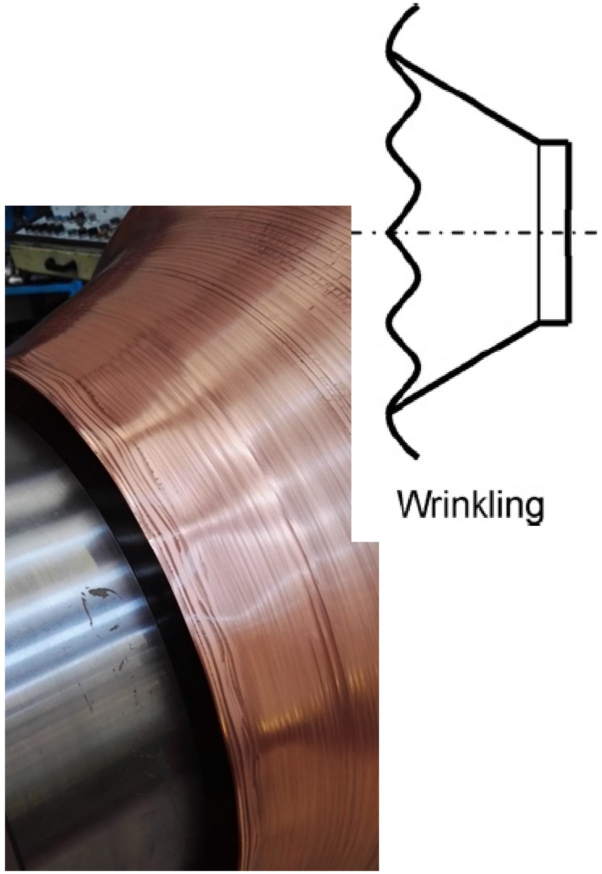
STEP 5 Spinning of the 2nd half cell

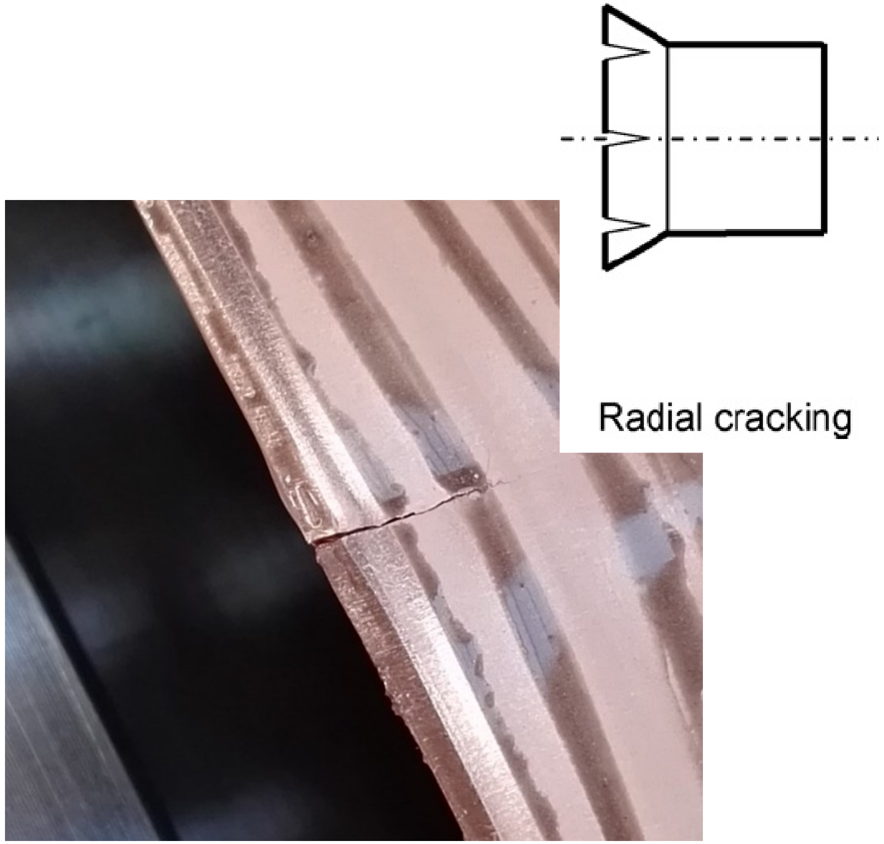


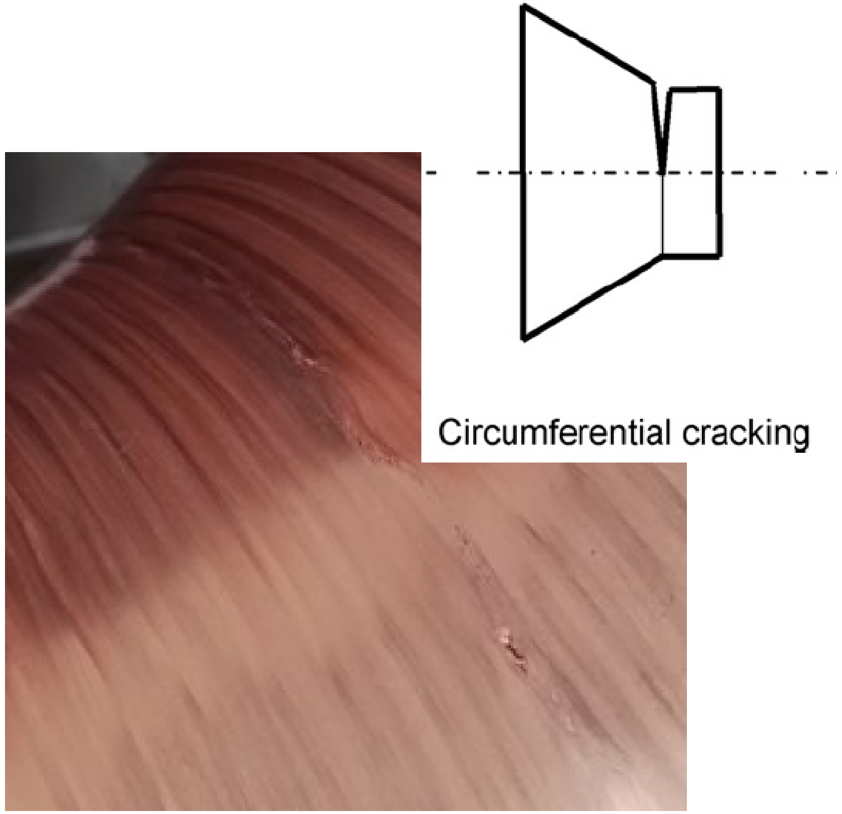
Here comes the troubles

Failure due to Copper Hardening

- The large amount of cold work introduced, produce copper hardening and consequent failures
- The sensibility of technician is fundamental to understand when the material need a thermal annealing







Thermal annealings in Seamless 400 MHz

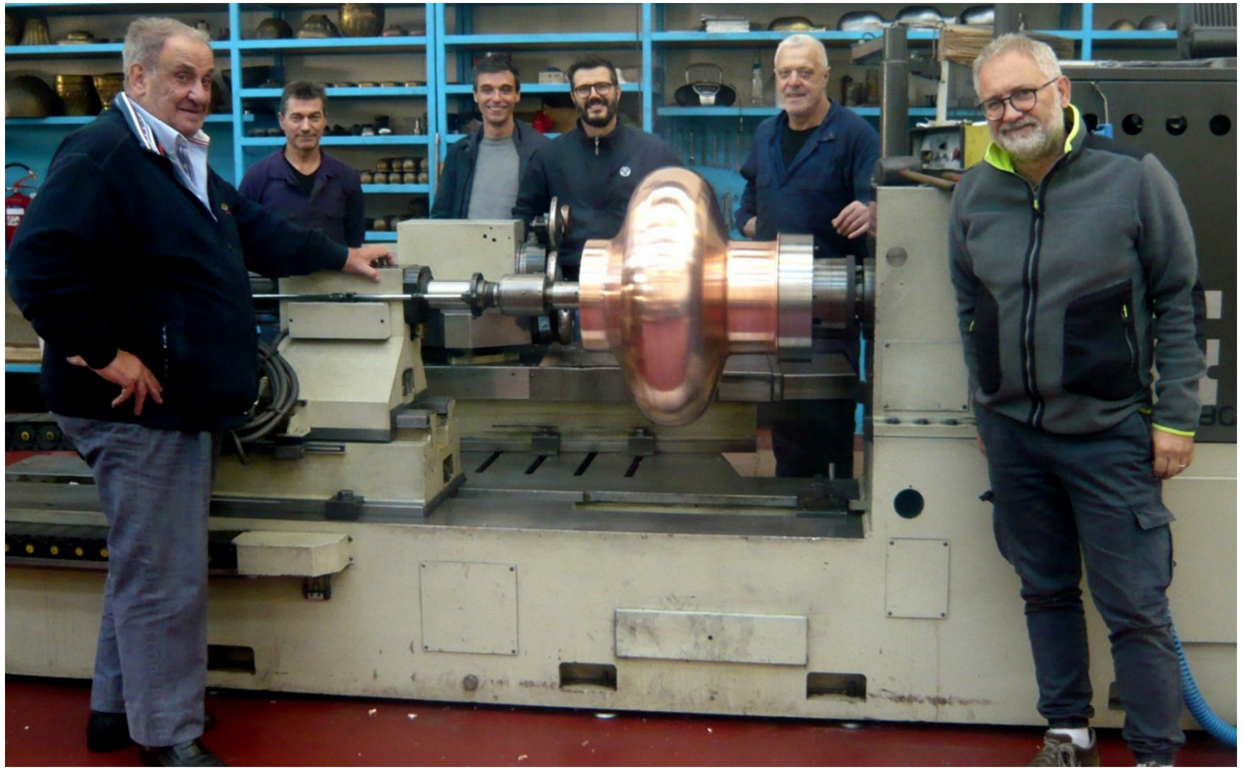
3 annealing necessary!

1st Thermal Annealing

2nd Thermal Annealing

3rd Thermal Annealing

Spinning completed!



The team that realized the first seamless 400 MHz elliptical cavity

Nb bulk cavities: **cavity treatment**

Cavitie's general treatment scheme

Step	WHY	COMMENTS
Forming		
EB Welding	Clean welding	Nb = getter material. If RRR/ 10 @ welding => $Q_0/10$
Ti purification	RRR enhancement	RRR 300-400 now commercially available
Chemical etching 100-200 µm (BCP / EP)	Remove contamination and damage layer	Limitation : BCP ~ 30MV/m; EP => >40 mV/m but lack of reproducibility
Annealing 800°C, 2h (or 600°C, 10h)	Get rid of hydrogen	Source of H: wet processes H segregates near surface in form of hydrides (= bad SC)
Chemical etching 5-20 µm	Remove diffusion layer (O, C, N)	Diffusion layer < ~1µm in bulk, a little higher at Grain Boundaries
Specific rinsing	e.g. remove S particles due to EP	Under evaluation HF, H_2O_2, ethanol, degreasing,…
High pressure rinsing (HPR)	Get rid of dust particles	Not always enough (recontamination during assembly)
Assembling	Ancillaries : antennas, couplers , vacuum ports…	**In clean room, but recontamination still possible**
Baking, 120°C, 48h	Decrease high field losses (Q-drop)	Unknown mechanism, first 10 nm of the surface in concern.
Post processing	Get rid of "re-contamination" ?	Under evaluation: dry ice cleaning, plasma
Test RF	Cavity's performance	First naked cavity in vertical cryostat, then dressed in horizontal cryostat/ accelerating facility
He processing, HPP	Decrease field emission	RF power with/ without He to destroy field emitters (dust particles) NB field emission : principal practical problem in accelerators

A general consideration

Do not make Nb surface worst than before with the next treatment!

- **Do not apply treatments** that affect the Nb surface and **could not be "accepted" by the next step**
- If a **mistake is done**, **go back in the procedure** until the step where contaminant can be removed without contaminating the system
- **Chemical reactions** in many cases can **not be stopped** simply **removing acid (residuals, no cooling, …). Rinsing is needed!**
- **Do not contaminate US bathes with material that can not be diluted**, as for **silicone grease**, oil, etc. Moreover take care of **contaminants** that can **float** over the liquid surface!
- **Wet components are more "sensitive" for collecting particles.**
- Duration limit for a final treated cavity is about 24 hours (XFEL)
  Do not leave open cavities for longer time

Cavity preparation for SRF qualification

- Degreasing surfaces to remove contaminates
- Chemical removal of exterior films incurred from welding
- Removal of damage layer of niobium from fabrication (≈ 150÷200 μm)
- Removal of hydrogen from bulk Nb
- Mechanical tuning
- Chemical removal of internal surface for clean assembly (10-20 μm)
  - – Additional “cleaning” steps if Electropolishing (EP) is used
- High Pressure Rinsing (HPR) to remove particulates from interior surfaces (incurred during chemistry and handling)
- Drying of cavity for assembly in cleanroom (reduce risk of particulate adhesion and reduce wear on vacuum systems)
- Clean assembly
- Clean evacuation
- Low-temperature baking

Cavity preparation for SRF qualification

- **Degreasing surfaces to remove contaminates**
- Chemical removal of exterior films incurred from welding
- Removal of damage layer of niobium from fabrication (≈ 150÷200 μm)
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- Drying of cavity for assembly in cleanroom (reduce risk of particulate adhesion and reduce wear on vacuum systems)
- Clean assembly
- Clean evacuation
- Low-temperature baking

Degreasing and surface preparation @INFN LASA

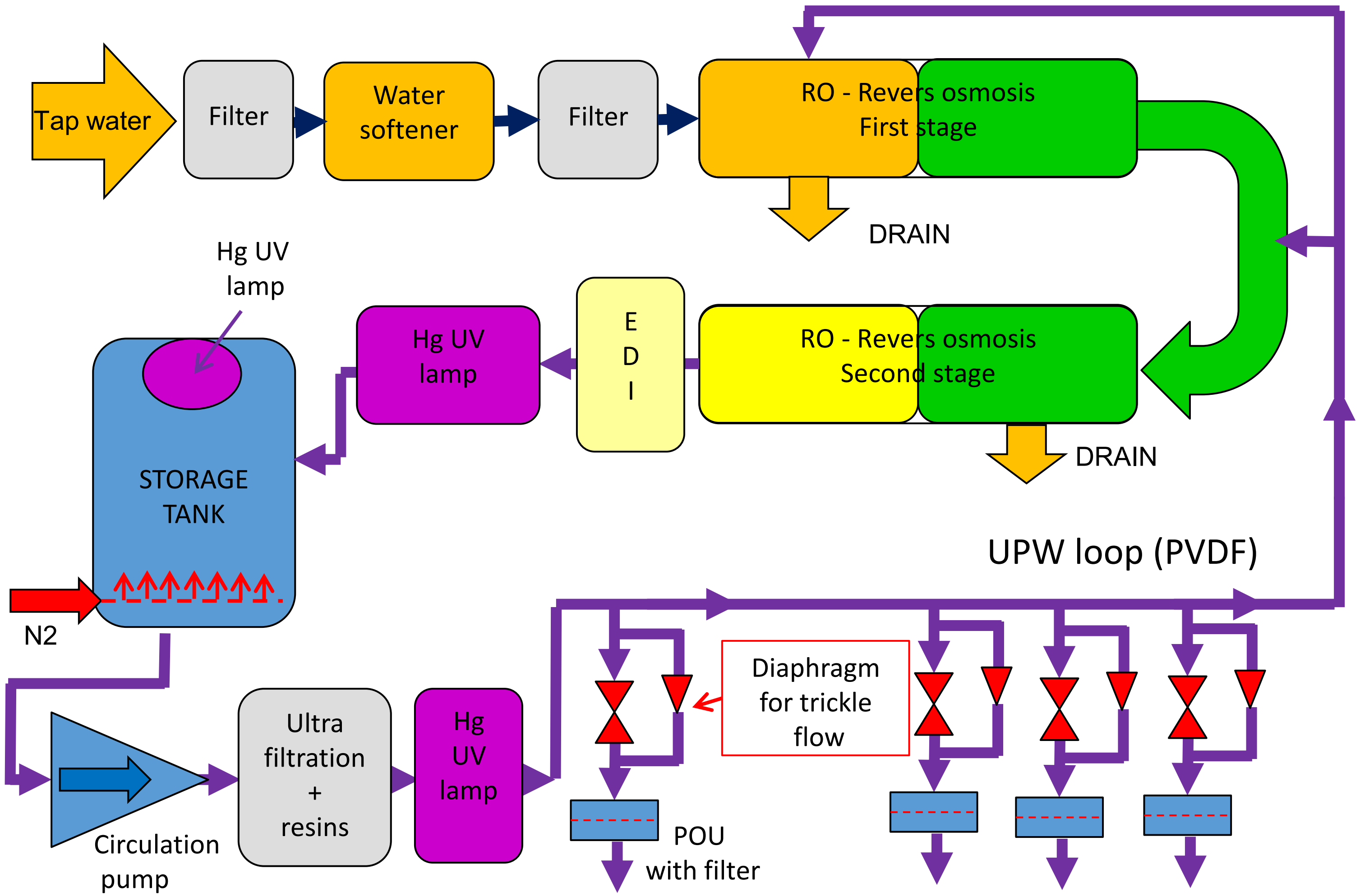
- After mechanical fabrication, **all contaminants** (fingerprints, oil, residuals from machining and QC) **must be removed**, similar to preparation of Ultra High Vacuum components.
- Typical process:
  - Water rinsing with specific detergent (Tikopur TR33, Micro -90, Liqui-Nox) usually 1-3%
    - For "dirty" component, alcohol and acetone could be used before
  - Water is usually **Ultra Pure Water** with 18 MΩ cm and filtered below 200 nm
  - Often in **HEPA filter environment**
- For entering **ISO7 clean room**, dishwashers are used for small components and car-wash for large components
- For entering **ISO4 clean room**, UltraSound is mandatory

Ultra Pure Water

- UPW Specifications
  - **Resistivity:**
    18.2 MΩ cm
  - Total organic carbon (TOC):
    < 5 ppb
  - Particulate counts (> 0.3 μm/l):
    < 10
  - Bacteria counts:
    < 0.1 CFU/100 ml

The water quality is as for the semiconductor industry:
ASTM- D 5127-07 E-1.2

Typical UPW plant



Industrial and laboratory plant

Large system:

- Production: **3000 l/h**
- **Storage: 9000 l**
- Typ. TOC: 3 ppb

Small lab production plant:

- Production: **170 l/h**
- **Storage: 6000 l**
- Typ. TOC: 3 ppb

Ultrasonic Cleaning

- Immersion of components in DI water and detergent medium
- **Wave energy forms microscopic bubbles** on component surfaces
- **Bubbles collapse (cavitation)** on surface loosening particulate matter
- **Transducer** provides **high intensity ultrasonic fields** that set up standing waves. Higher frequencies lowers the distance between nodes which produce less dead zones with no cavitation
- Ultrasonic transducers are available in many different wave frequencies from 18 kHz to 120 kHz, **the higher the frequency the lower the wave intensity**

Cavities and all hardware components (Flanges, nuts & bolts…) have to be degreased with ultrasonic cleaning

Water Break Test on Nb Sample

- It's a standard test for testing cleaning procedure with UPW and US

Not efficient cleaning

After good US cleaning procedure

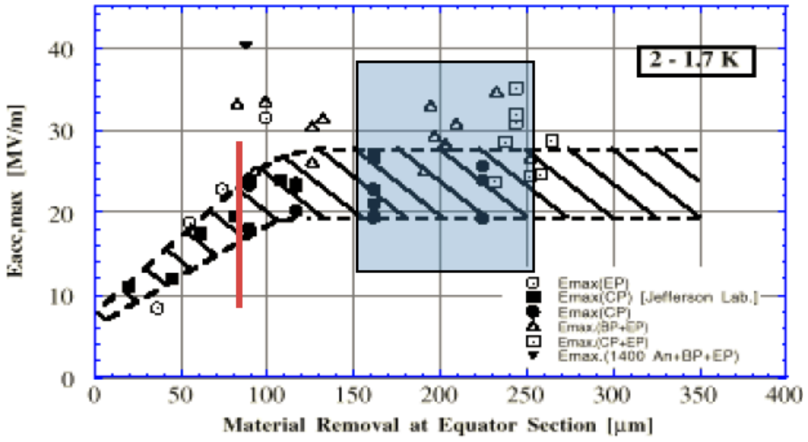
ASTM F22 - 02(2007) Standard Test Method for Hydrophobic Surface Films by the Water-Break Test.

Cavity preparation for SRF qualification

- Degreasing surfaces to remove contaminates
- Chemical removal of exterior films incurred from welding
- **Removal of damage layer of niobium from fabrication (≈ 150÷200 μm)**
- Removal of hydrogen from bulk Nb
- Mechanical tuning
- Chemical removal of internal surface for clean assembly (10-20 μm)
  - – Additional "cleaning" steps if Electropolishing (EP) is used
- High Pressure Rinsing (HPR) to remove particulates from interior surfaces (incurred during chemistry and handling)
- Drying of cavity for assembly in cleanroom (reduce risk of particulate adhesion and reduce wear on vacuum systems)
- Clean assembly
- Clean evacuation
- Low-temperature baking

Removal of damage layer

After all the mechanical operations, a **thin layer of about 200 μm** must be removed



K. Saito

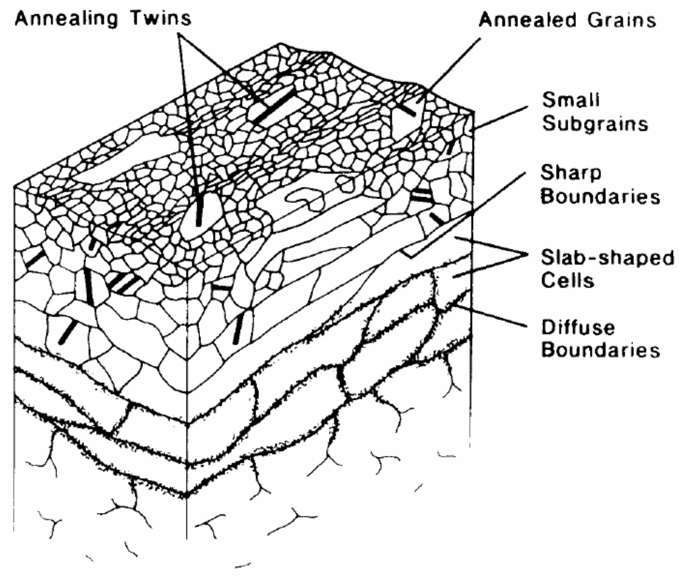
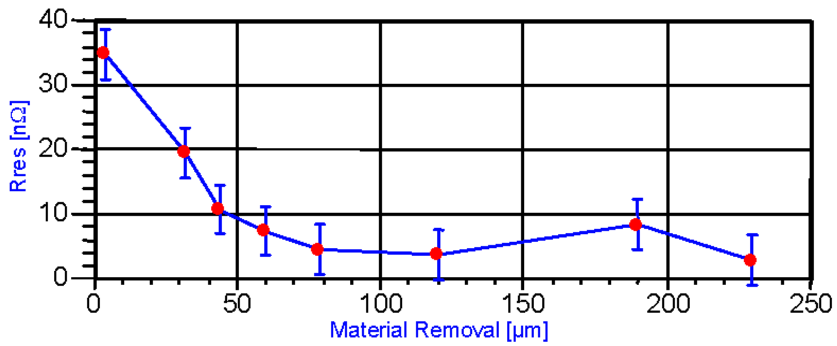
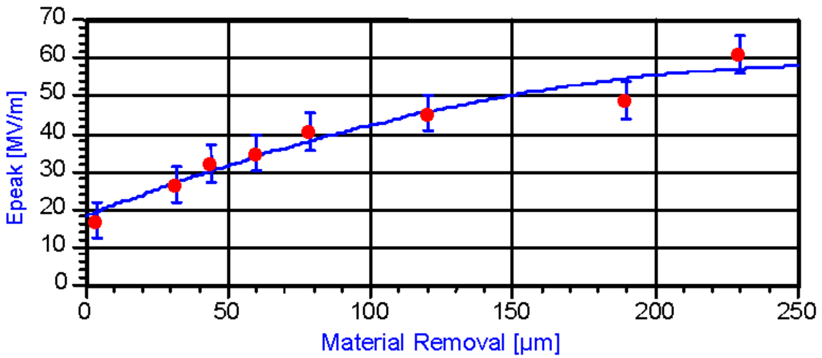


Fig. 1 - Schematic sketch of the structure observed for an abraded OFHC Copper surface.

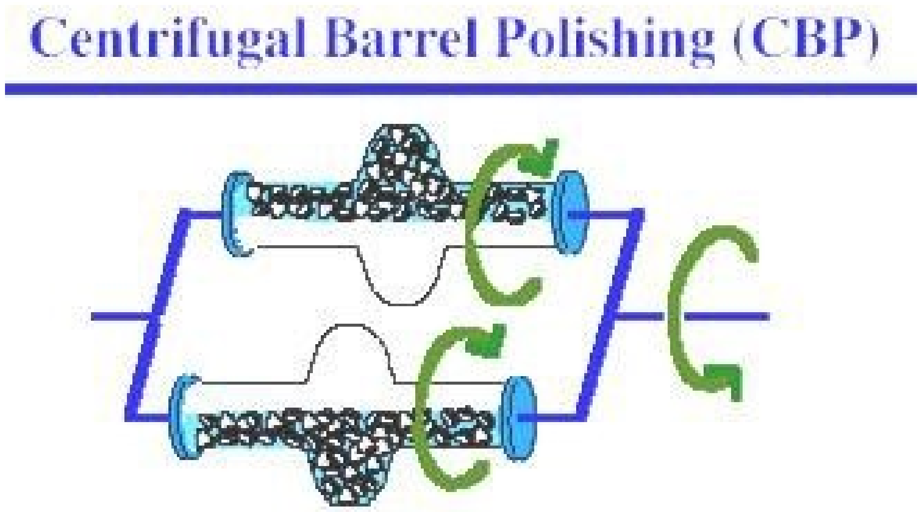
V. Palmieri





P. Kneisel

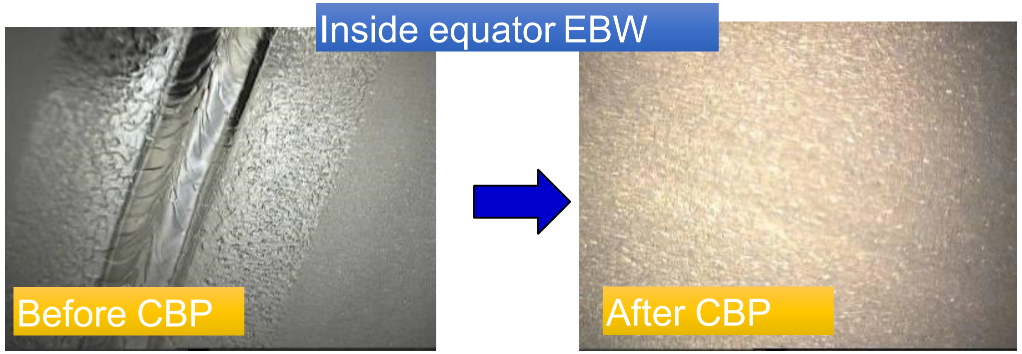
Centrifugal Barrel Polishing (CBP)



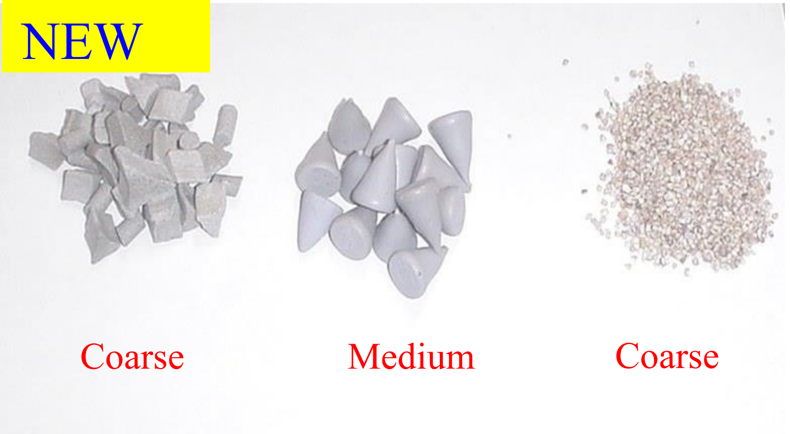
KEK 2001

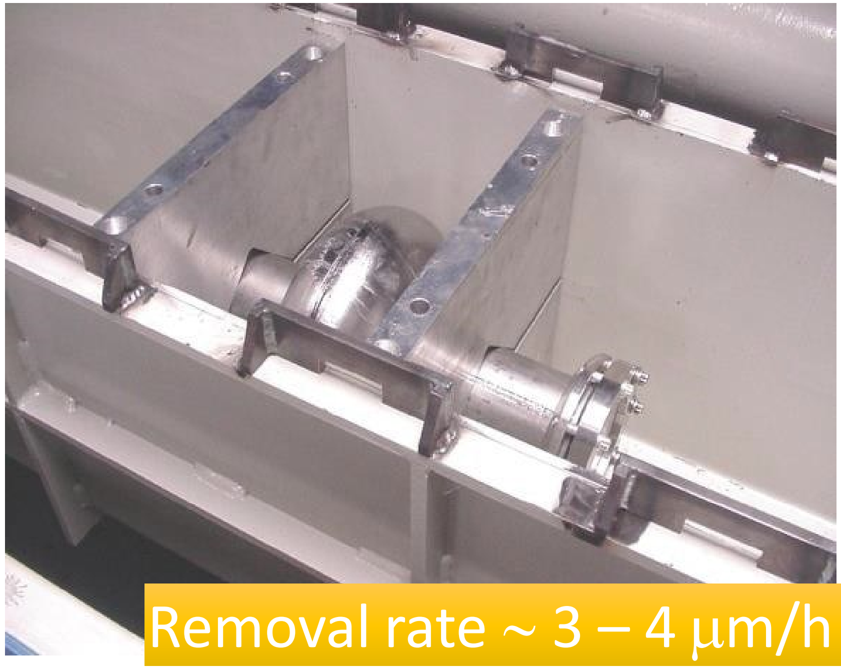
Implementation:

- **Plastic stones and liquid abrasive** added inside cavity and rotated
- **Stones rubbing on surface removes material** thus smoothing the surfaces (including weld areas)
- Benefit is **less overall chemistry** needed (80 μm) and smooth weld areas
- Removal of material **2x on equators** then irises. Average removal rate ≈ 5 μm/h



Barrel Polishing Machine @ JLAB





Removal rate ~ 3 – 4 μm/h

(Electro-)Chemical Nb removal

Nb is resistant to chemical attack

- **HNO_3:** oxidation of Nb surface and **passivation**, i.e. no more corrosion of the metal.
- **HF**: **dissolve only Nb oxides**, but doesn't attack Nb itself
- **HCl**: no attack
- **H_2SO_4**: no attack
- Strong alkaline solution (**NaOK, KOH, NH_4OH**): no attack

Two effects have to be coupled: Nb oxidation (e.g. HNO_3) and Nb oxides dissolution (HF).

Buffer Chemical Polishing and Electro Polishing

Buffer Chemical Polishing (BCP)

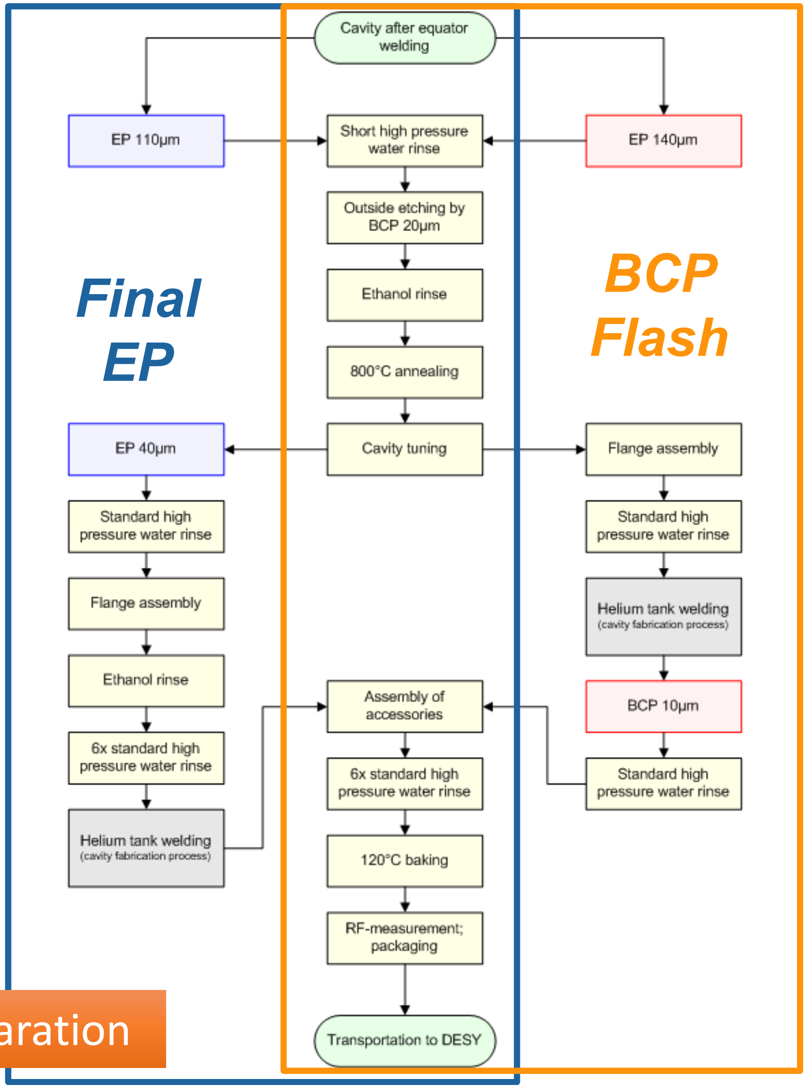
- A mixture of Hydrofluoric (**HF**), Nitric Acid (**HNO_3**) and Phosphoric acid (**H_3PO_4**), usually in 1:1:1 or 1:1:2 ratio in volume

Electro Polishing (EP)

- A mixture of Hydrofluoric Acid (**HF**) and Sulfuric Acid (**H_2SO_4**) + electric current

Sometimes, the **two processes** are used **together** to achieve better surface polishing (see EXFEL final steps)

EXFEL Cavity preparation



Buffered Chemical Process (BCP)

- Mixture of concentrated Hydrofluoric Acid (**HF, 40%**), Nitric acid (**HNO_3**, 70%) and Phosphoric Acid (**H_3PO_4, 85%**)
- H_3PO_4 doesn't participate the reaction: it act like a buffer slowing down the speed of the **exothermic reaction (self exiting!)**.
- **1:1:2, generally used, 1 μm/min @ 20 °C**

Oxidation

$$2Nb + 5NO_3^- \Rightarrow Nb_2O_5 + 5NO_2 + 5e^-$$

Brownish gas

$$2NO_2 \Rightarrow N_2O_4$$

Dissolve

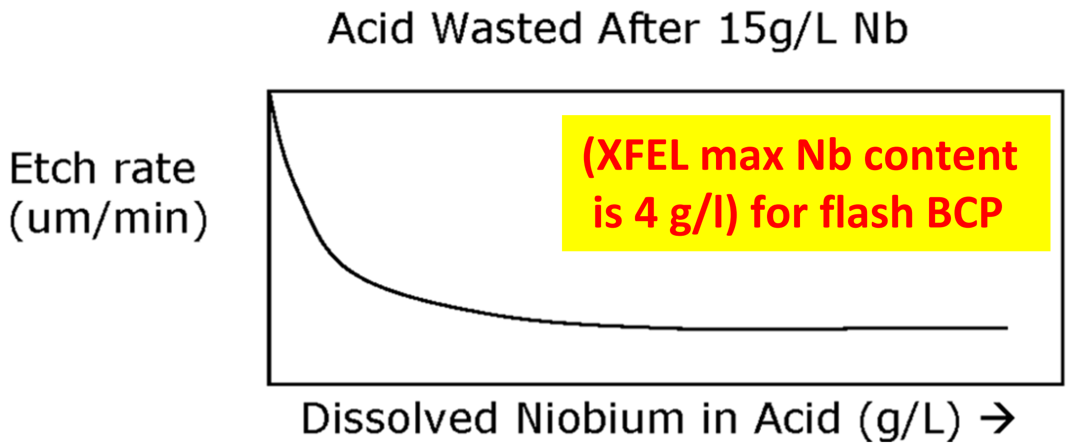
$$Nb_2O_5 + 6HF \Rightarrow H_2NbOF_5(\text{soluble}) + NbO_2F \cdot 0.5H_2O\ (\text{not soluble}) + 1.5H_2O$$

$$NbO_2F \cdot 0.5H_2O + 4HF \Rightarrow H_2NbOF_5 + 1.5H_2O$$

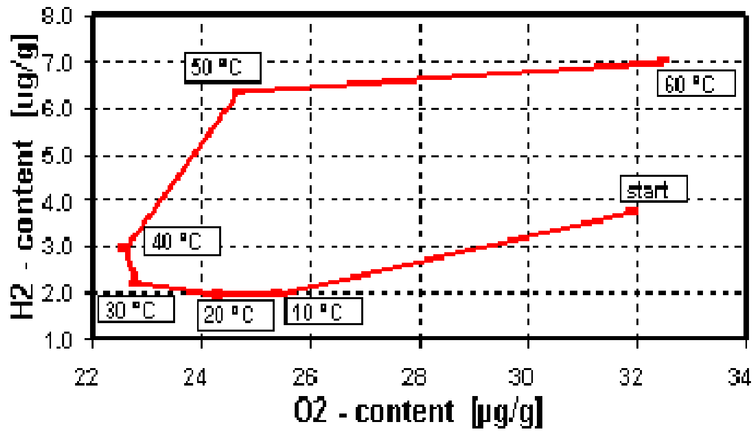
Use of BCP Process

- **1:1:1** still used for subcomponents due to high etching rate (~ 8 μm/min)
- **1:1:2** used for cavity treatment (~ 1 μm/min)
- BCP must **mixed before used** because it stratifies
- BCP is usually **cool down before and during etching** to mitigate temperature increase and hydrogen content (starts at 3-5 °C and ends around 20 °C)

Etching rate versus dissolved Nb



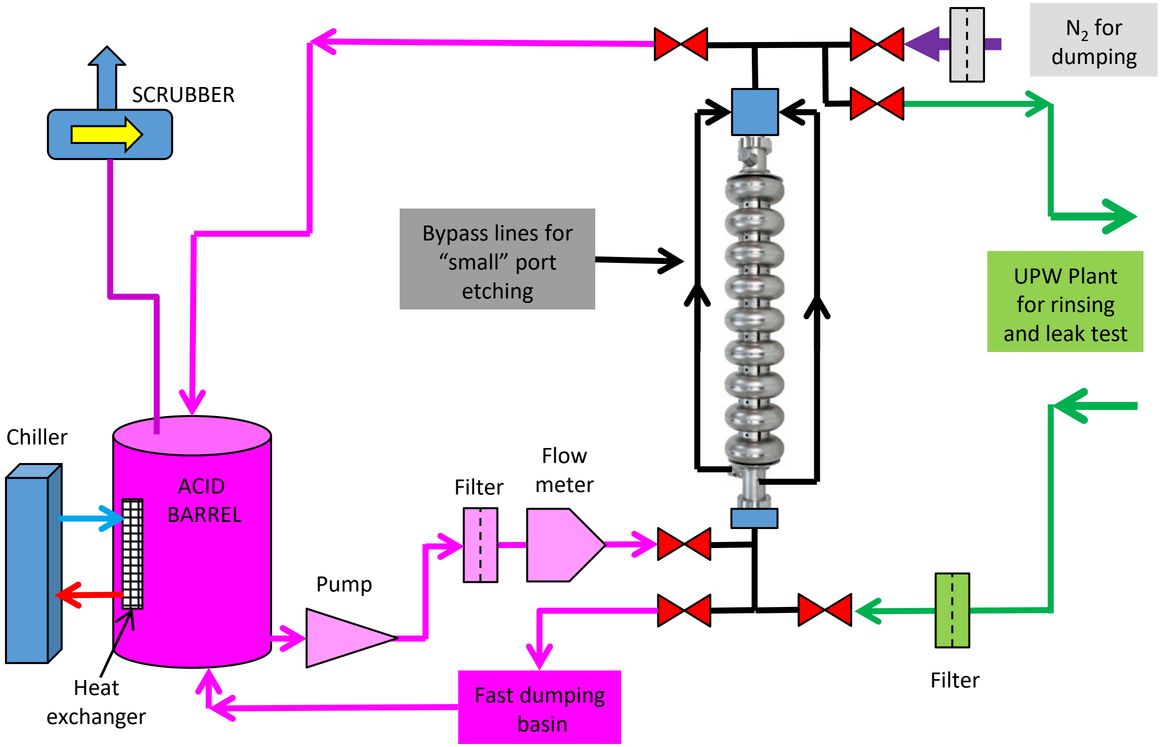
Hydrogen content vs BCP Temperature



Gas content of Nb at different etching temperatures (Schölz). Etching time 20 min, BCP 1:1:4

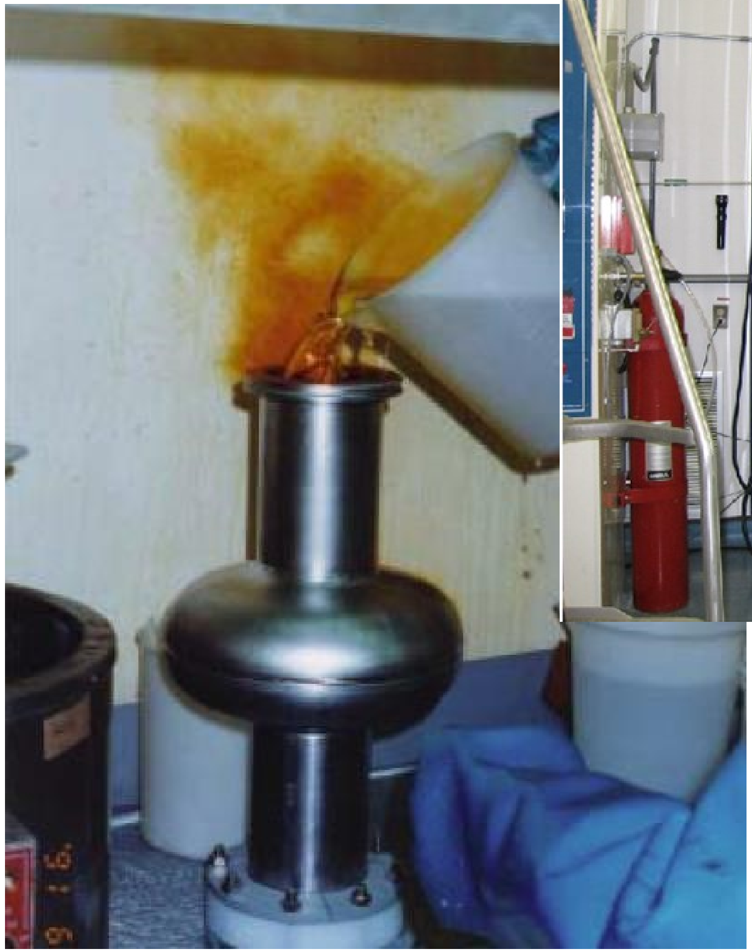
BCP Plant Layout

- **All components** in the acid mixture circuit **MUST be resistant** to acid attack
- Operative **temperature**: **below 20 °C**, to reduce hydrogen diffusion in Nb. Usually treatment starts at about **5°C ÷ 6 °C**
- **Exothermic reaction**: heat exchanger or cooled barrel is needed
- **Cavity held in vertical position**, acid flow from the bottom part



- **Temperature gradient** causes **increased** etching from one end to the other
- **Usually etching rate on iris is 2 x the equator one**
- Used both for **bulk removal and final etching**: for **EXFEL** only for **final etching of half of cavities**

BCP Plant in Operation in Labs



BCP Cabinet JLAB

DESY BCP

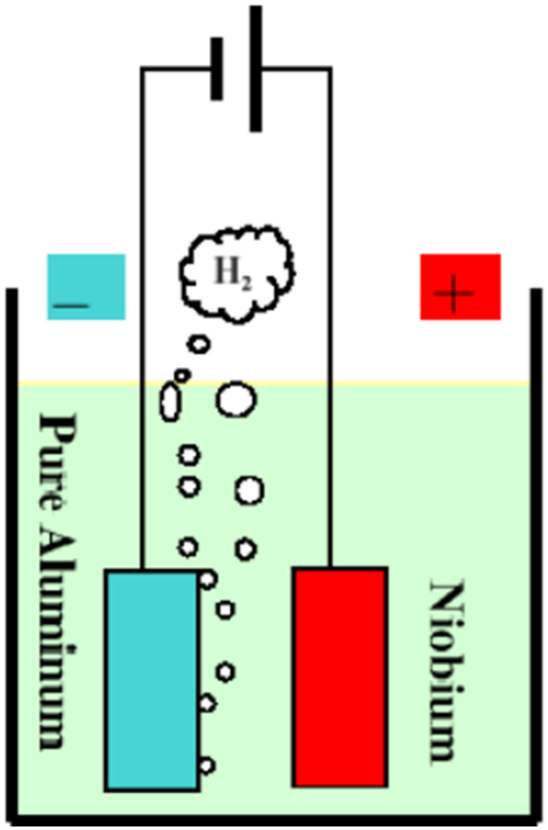
BCP Plant in Operation at Qualified Vendors

Automatic BCP system for subcomponents @ Ettore Zanon for EXFEL (etching + rinsing)

Electropolishing (EP)

A **constant voltage** is kept between an Aluminum electrodes and the cavity immerse in a mixture of Hydrofluoric Acid (**HF**, 49%) and Sulfuric Acid (**H_2SO_4**, 96%) in a ratio 1:9 (typical) in volume.

Reaction is not self sustained: no current - no reaction



Oxidation

$$2Nb + 5SO_4^{2-} + 5H_2O \Rightarrow Nb_2O_5 + 10H^+ + 5SO_4^{2-} + 10e^-$$

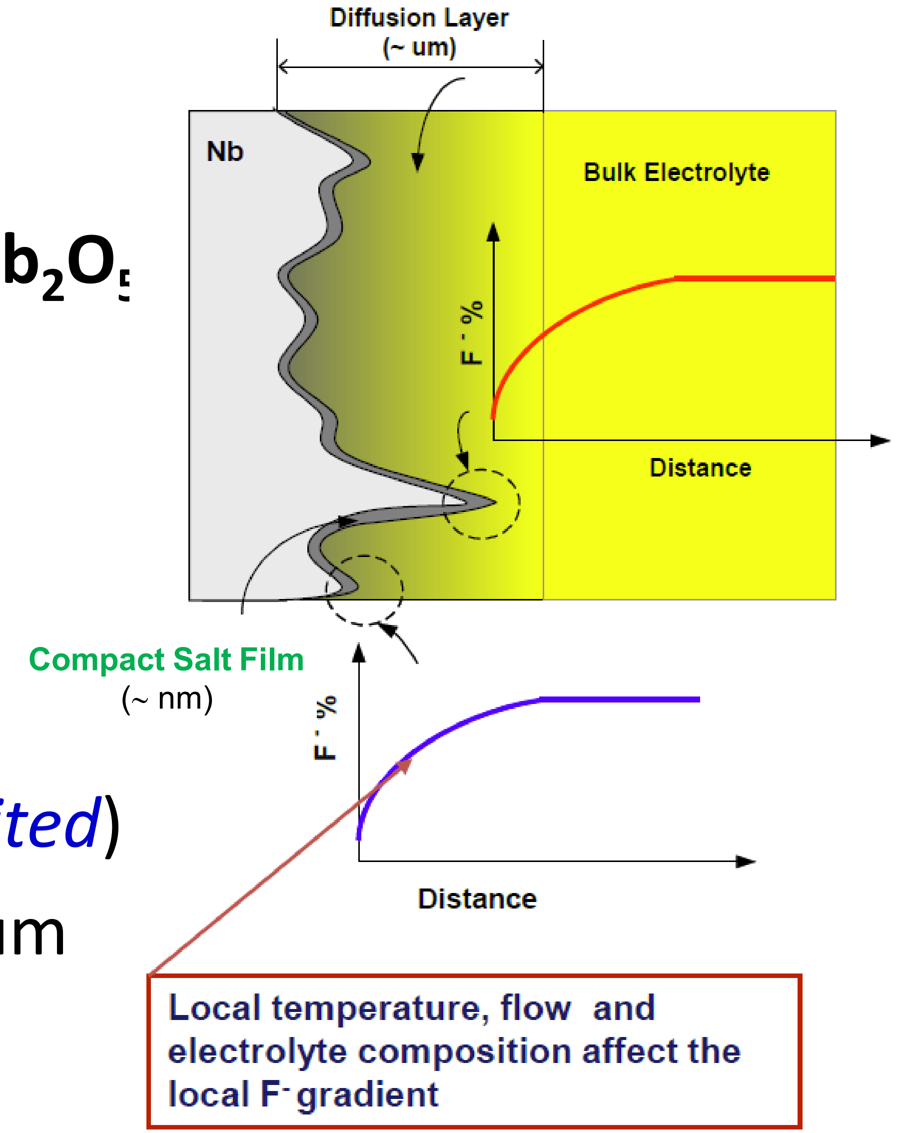
Dissolve

$$Nb_2O_5 + 6HF \Rightarrow H_2NbOF_5 (soluble) + NbO_2F \cdot 0.5H_2O \ (not\ soluble) + 1.5H_2O$$

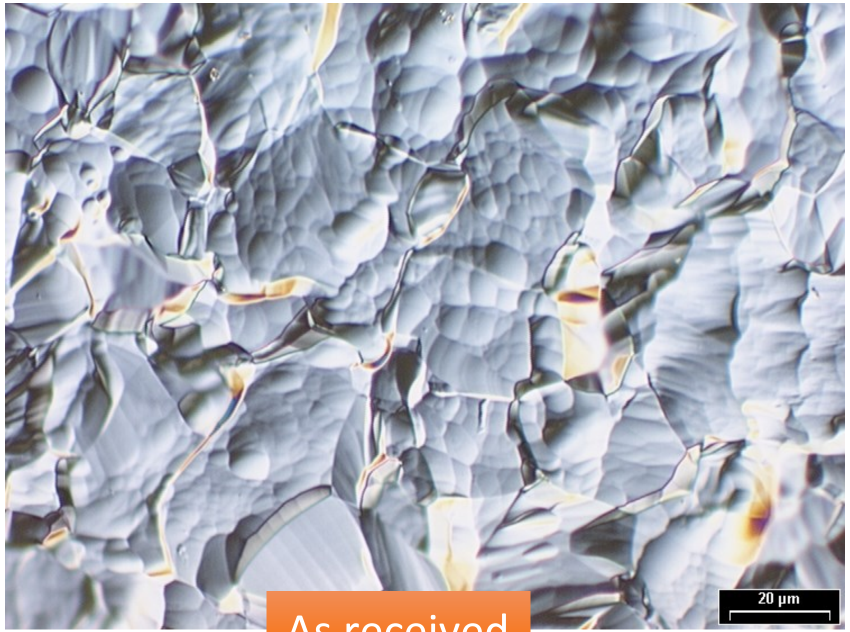
$$NbO_2F \cdot 0.5H_2O + 4HF \Rightarrow H_2NbOF_5 + 1.5H_2O$$

EP Mechanism

- Anodization of **Nb** in **H_2SO_4** forces growth of **Nb_2O_5**
- **F^-** dissolves **Nb_2O_5**
- These competing processes result in current flow and material removal
- Above a certain anodization potential, the **reaction rate plateaus**, limited by how fast fresh **F^-** can arrive at the surface (*diffusion-limited*)
- The diffusion coefficient sets a scale for optimum leveling effects

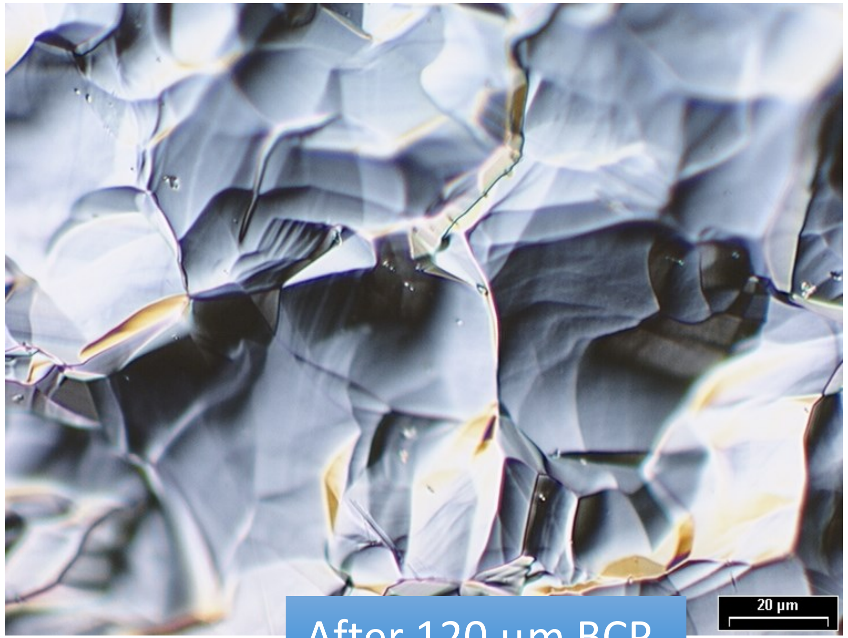


Surface Polishing BCP vs EP

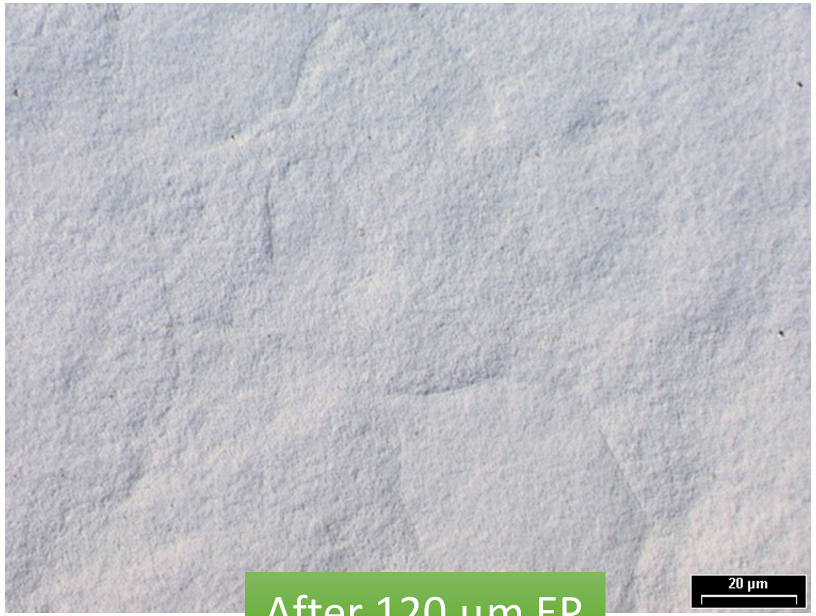


As received

The main **difference** between **BCP** and **EP** is **smoothening of grain boundaries**

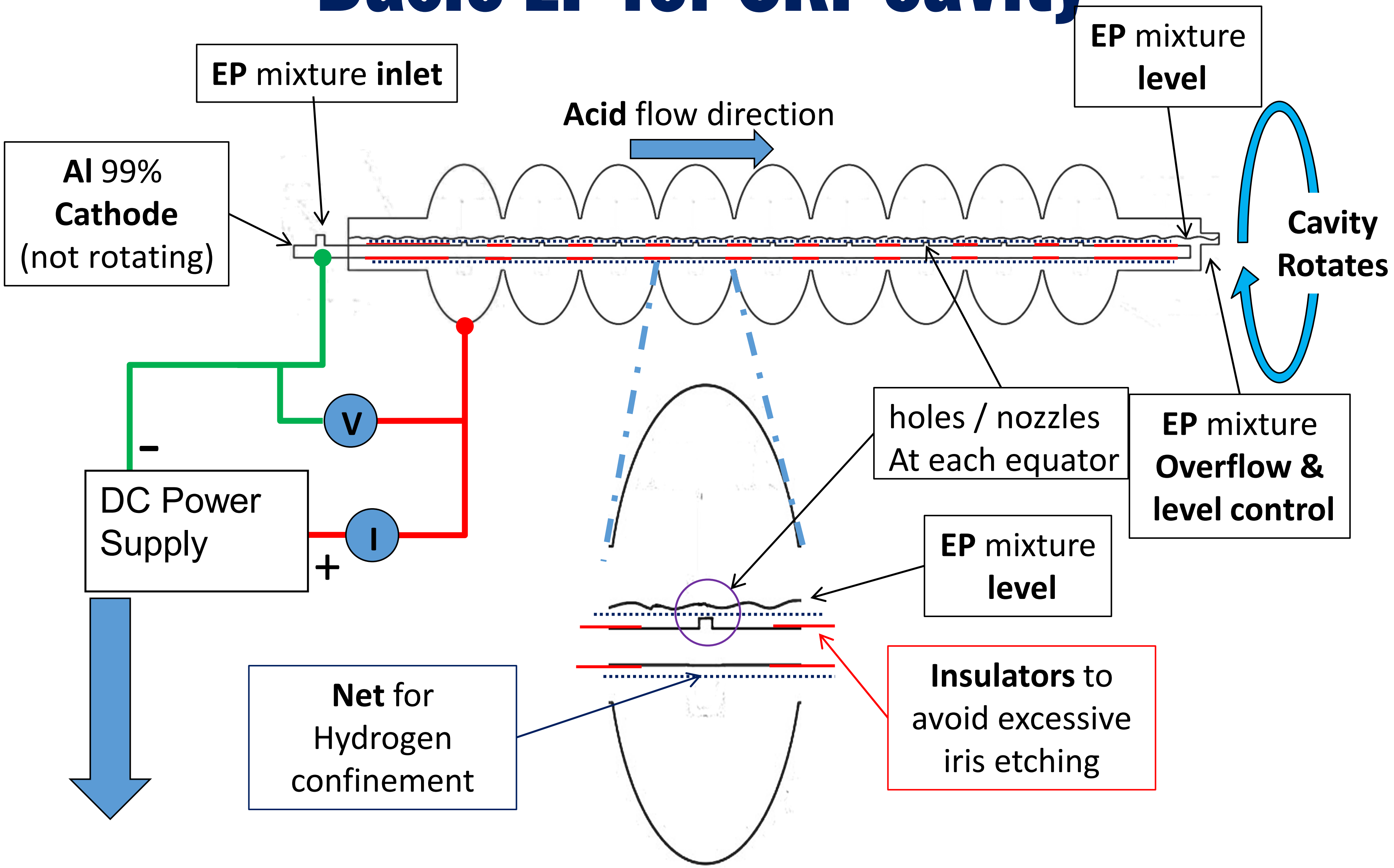


After 120 μm BCP



After 120 μm EP

Basic EP for SRF cavity



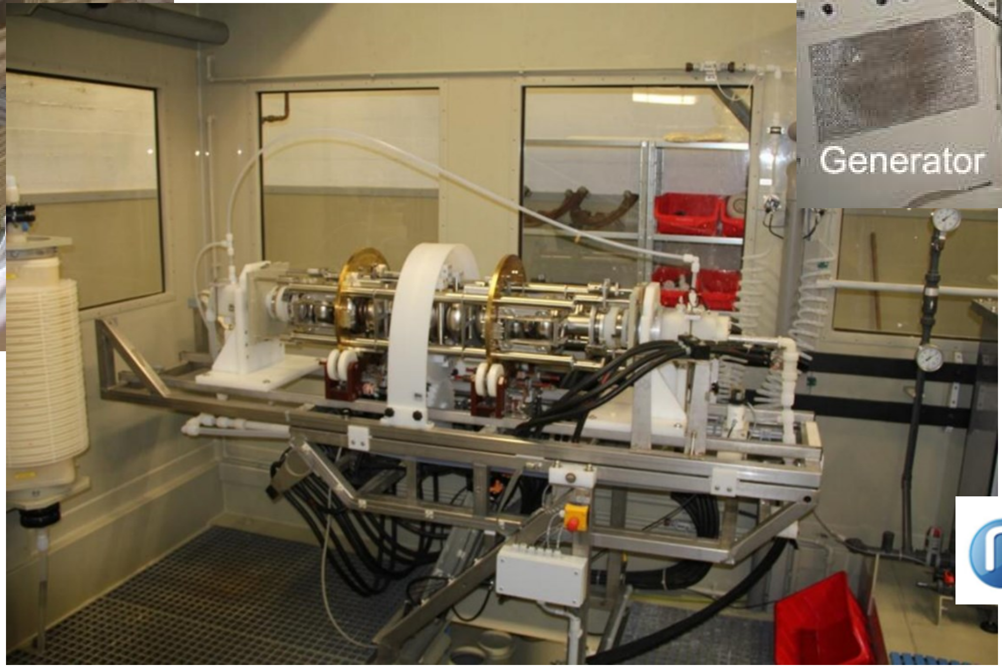
Electropolishing of SRF cavities

- Etching rate typical **0.3 ÷ 0.4 μm/min**
- Cavity (or electrode) is **rotating**
- It requires **ethanol rinsing** to remove Sulphur
- The current density (30-100 mA/cm^2) in the plateau region:
  - decreases linearly with lower HF/H_2SO_4 ratio
  - increases with increasing temperature
- **Temperature** during the process is maintained **between 25 – 35 °C**
- **Current oscillations often observed during polishing** (dynamic balance between oxide formation and dissolution). It's not a necessary condition for good surface finishing but indication of good processing parameters (temperature, voltage, agitation, HF concentration)

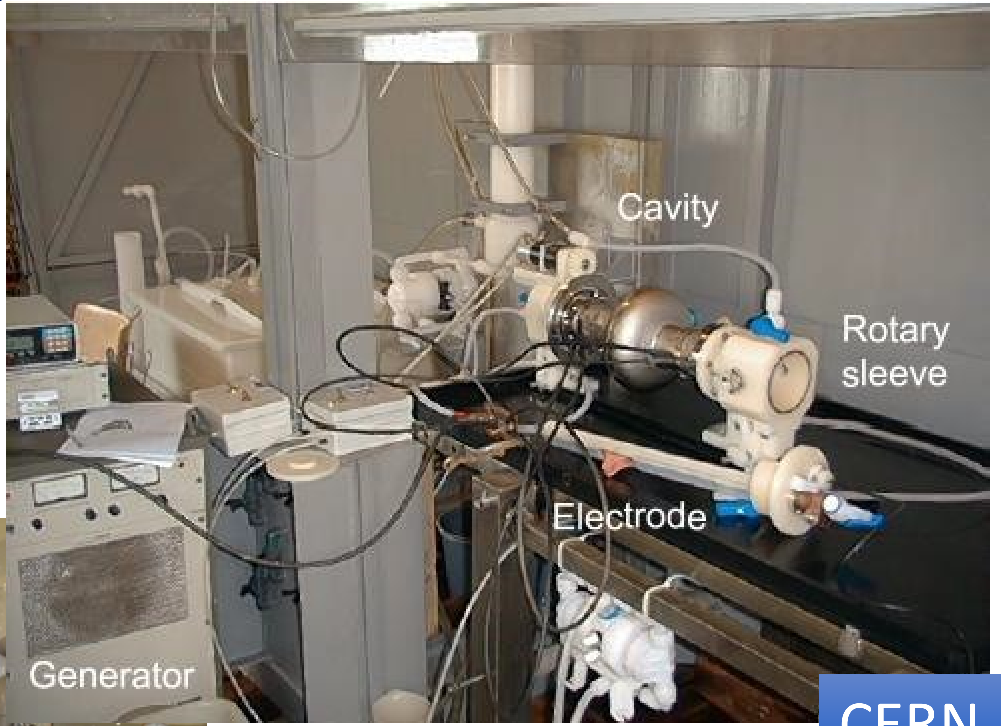
Finding the right balance among the processing parameters becomes complicated when polishing multi-cell cavities!

EP Systems

DESY



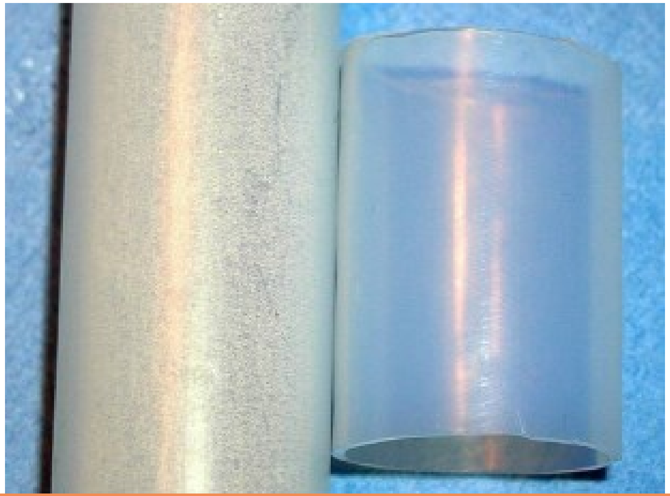
research instruments



CERN

EP Ethanol Rinse

- **Motivation: during EP process sulfur is produced and can cause field emission**
- Sulfur segregates out of the acid as a **reaction with the Al electrode**, and is **deposited all over the system**, and also on the **Nb surface**
- Risk of **reaction with Nb during 800 °C** heat treatment: S must be removed before this step
- **Sulfur is insoluble in water**, but (slightly) soluble in ethanol
- Either ethanol rinse or cleaning with detergent + US necessary



PVDF tube before and after ethanol cleaning

Sulfur removed from a PVDF tube

BCP vs EP

BCP

- 2 Volumes of **H_3PO_4** (**buffer**, very viscous)
- 1 Volume of **HNO_3** (**oxidant**, transforms Nb into Nb^{5+})
- 1 Volume of **HF** (**complexant** of Nb^{5+}, dissolves the oxide layer formed by HNO_3 into NbF_5)

Pros

- **Easy to handle**, middle stirring necessary
- **Fast etching rate**
- **Very reproducible**

Cons

- **This is not "polishing" but "etching"**: all crystalline defects are preferentially attacked (etching pits, etching figures)
- Grains with various orientations are not etched at the same rate, which **induced roughness**!
- Except for a few cases E_{acc}^{max}~25-30 MV/

Caution!

- Do not process at temperatures higher than 25 °C
- Risk of runaway

BCP vs EP

EP

- 9 Volumes of **H_2SO_4** (**buffer**, very viscous)
- 1 Volume of **HF** (**complexant** of Nb^{5+}, dissolves the oxide layer formed due to the high potential applied to Nb)

Pros (Ideal condition, i.e. viscous layer present)

- **This is really "polishing"**, not sensitive to crystallographic defects – it produces a smooth surface
- Should not be sensitive to the cathode-anode distance - the **same etching rate everywhere**
- It gives (but not always) **the best ever E_{acc}^{max}** ~ 45 MV/m (TESLA shape -> ~180 mT)

Cons

- It is **not possible to reach an ideal state** in most of our processing conditions
- **Very sensitive** to stirring condition, temperature, and aging of the mixture
- **Not very reproducible**
- **Safety issues** (acid mixture sensitive to water, H2 evolution, etc.)

Caution!

- If T increases: the etching rate increases but there is also a risk of pitting, H loading and HF evolution
- It V increases: the etching rate increases but there is also a risk of pitting, the generation of Sulphur particles and sensitivity to the cathode-anode distance

BCP vs EP

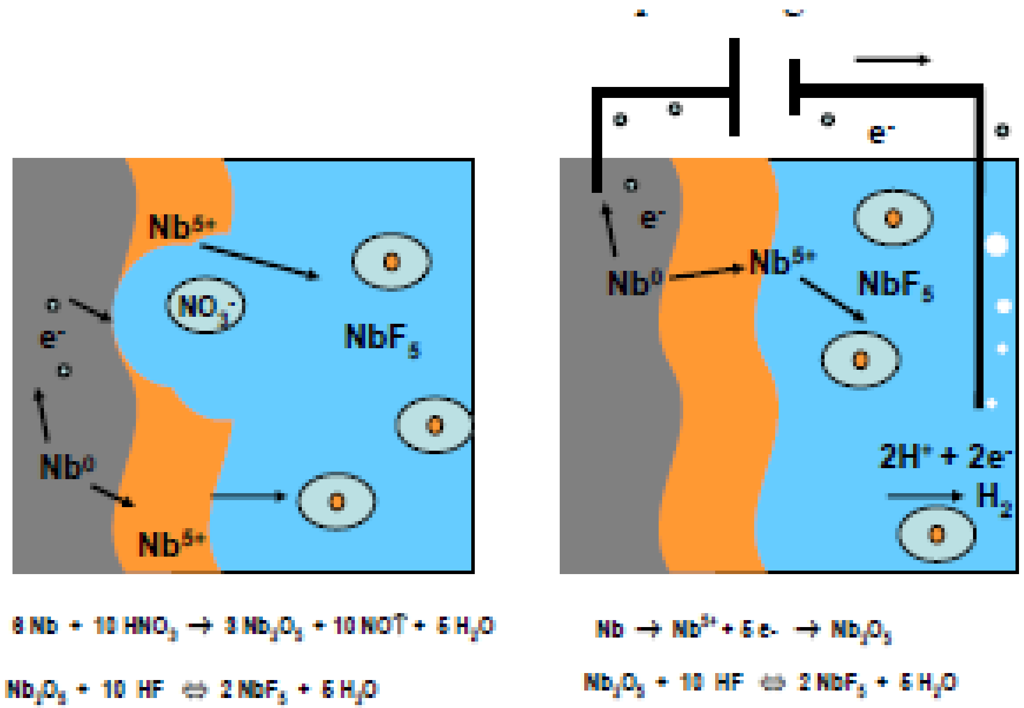


Fig. 6: A comparison between chemical polishing (left) and electropolishing (right). In both cases, niobium is oxidized into Nb^{5+}. In the case of chemical polishing, oxidation occurs because of the presence of a strong oxidant (NO_3^-) in the solution, while in electropolishing oxidation occurs because of the bias applied to the anode. Because of the presence of water, the stable form of Nb is Nb_2O_5; but HF decomposes the oxides into NbF_5, which is soluble in the solution.

Cavity preparation for SRF qualification

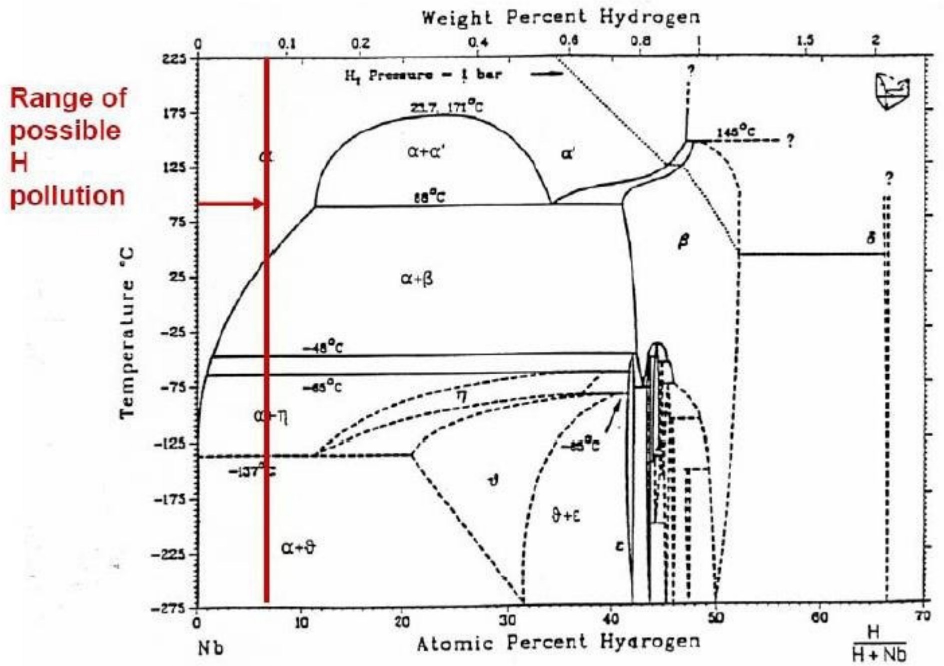
- Degreasing surfaces to remove contaminates
- Chemical removal of exterior films incurred from welding
- Removal of damage layer of niobium from fabrication (≈ 150÷200 μm)
- **Removal of hydrogen from bulk Nb**
- Mechanical tuning
- Chemical removal of internal surface for clean assembly (10-20 μm)
  - – Additional "cleaning" steps if Electropolishing (EP) is used
- High Pressure Rinsing (HPR) to remove particulates from interior surfaces (incurred during chemistry and handling)
- Drying of cavity for assembly in cleanroom (reduce risk of particulate adhesion and reduce wear on vacuum systems)
- Clean assembly
- Clean evacuation
- Low-temperature baking

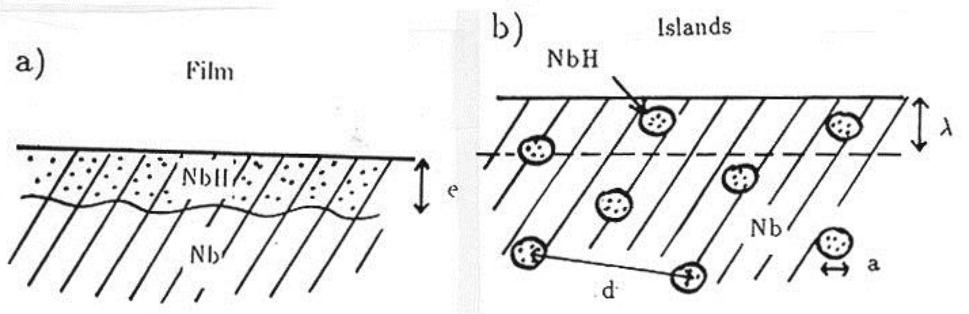
Annealing - Cavity firing at high temperature

- H diffuses in the bulk during the various etching treatments.
  See **R. E. Ricker and G. R. Myneni,** J. Res. Natl. Inst. Stand. Technol. **115**, 353-371 (2010), Evaluation of the Propensity of Niobium to Absorb Hydrogen During Fabrication of Superconducting Radio Frequency Cavities for Particle Accelerators.
- Nb is an active metal with respect to various gases: it acts like a getter.
- Hydrogen makes a solid solution in Nb, H_2 equilibrium pressure is driven by Sievert Law
- Equilibrium pressure is **temperature dependent** and **increasing the temperature**, maintaining a **low H_2 partial pressure**, **H_2 is desorbed from the bulk (Nb)**

Hydrogen in Niobium Q "disease"

- Cavities that remain at **70-150 K for several hours** (or slow cool-down, < 1 K/min) experience **a sharp increase of residual resistance**
- **More severe** in cavities which have been heavily **chemically etched**
- **H is readily absorbed into Nb** where the oxide layer is removed (during chemical etching or mechanical grinding)
- **H has high diffusion rate in Nb**, even at low temperatures.
- **H precipitates** to form a hydride phase **with poor superconducting properties**: T_c=2.8 K, H_c=60 G
- At room temperature the required concentration to form a hydride is 10^3- 10^4 wppm
- At 150 K it is < 10 wppm





Annealining

Hydrogen outgassing
=> most efficient at 750°C – 800°C, **2h under good vacuum**

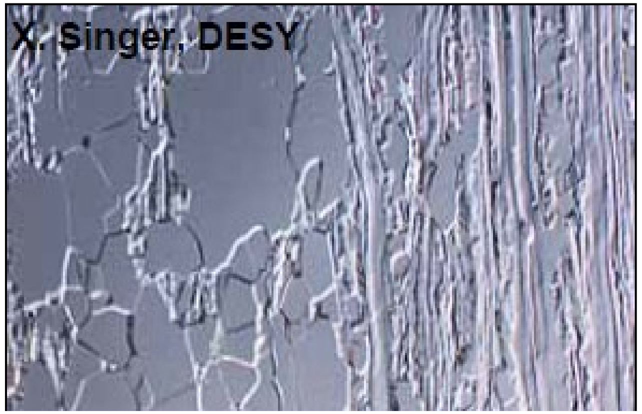
Recrystallization (goal is close to 100% with highest RRR)

- **Removing of defects and curing of dislocations**
- **Nucleation of new grains and growing of new crystals**
- Grain growth (depending on temperature and purity)

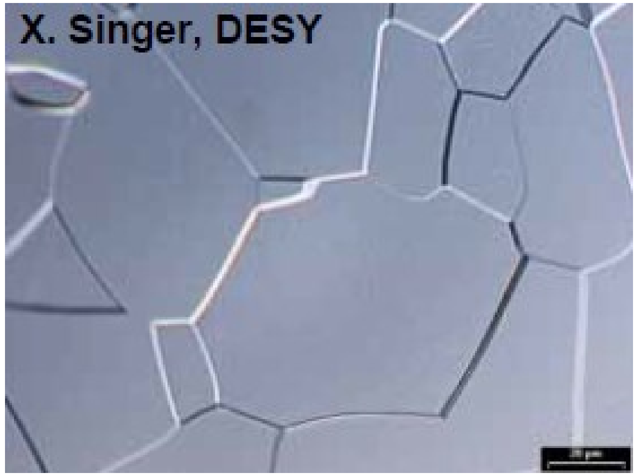
Nb becomes softer and this facilitate the cavity tuning process

Different parameters at different labs:

- 600 °C/10 h at Jlab
- 800 °C/2 h at DESY
- 750 °C/3 h at KEK

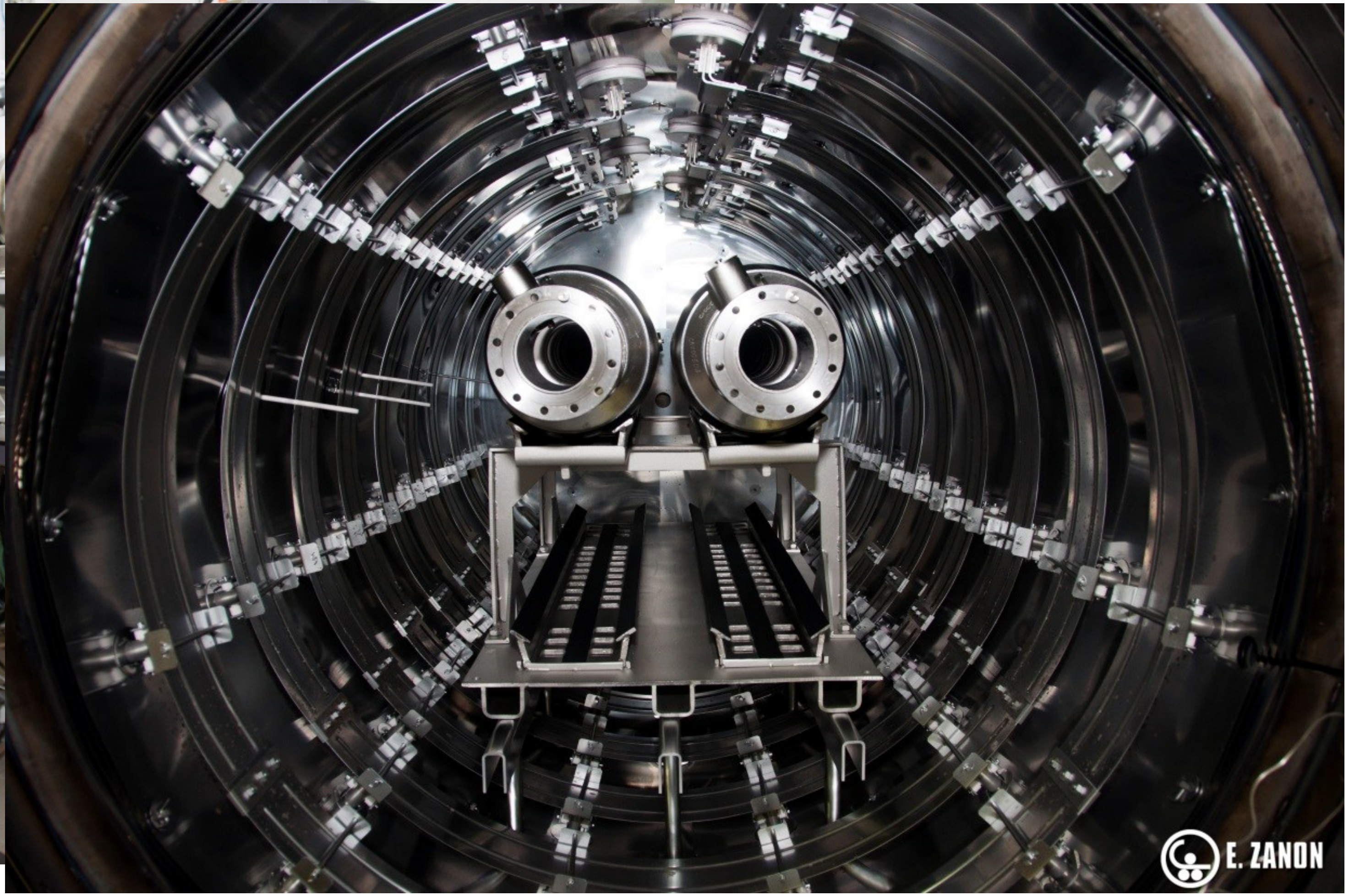


No completely recrystallized Nb



Completely recrystallized Nb

EZ Furnace



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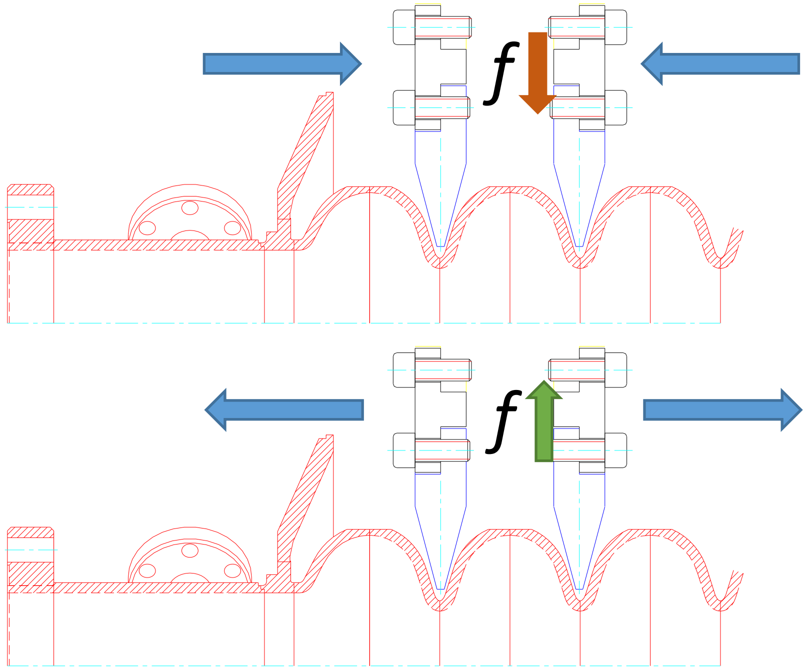
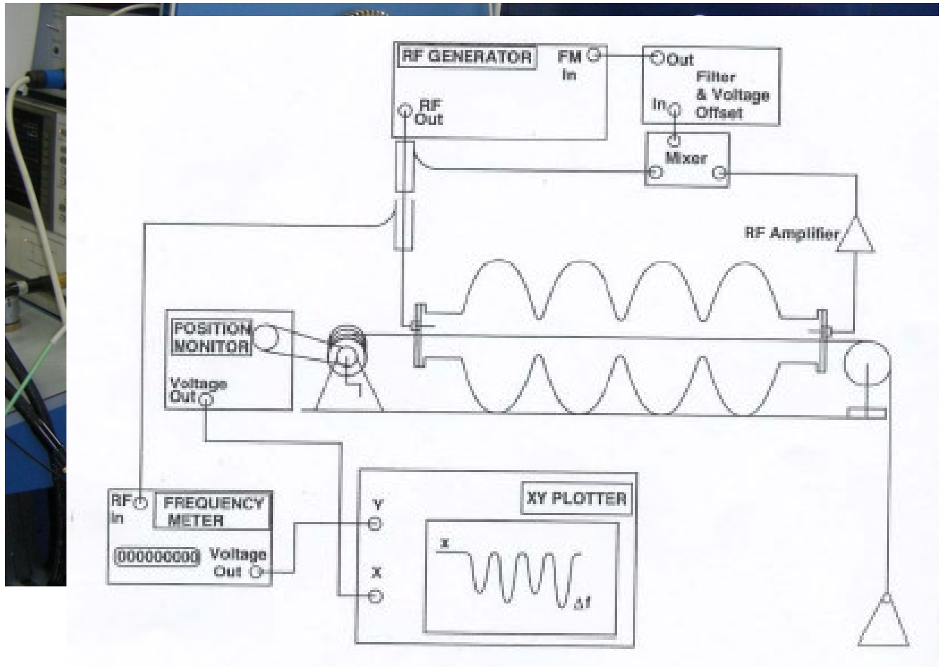
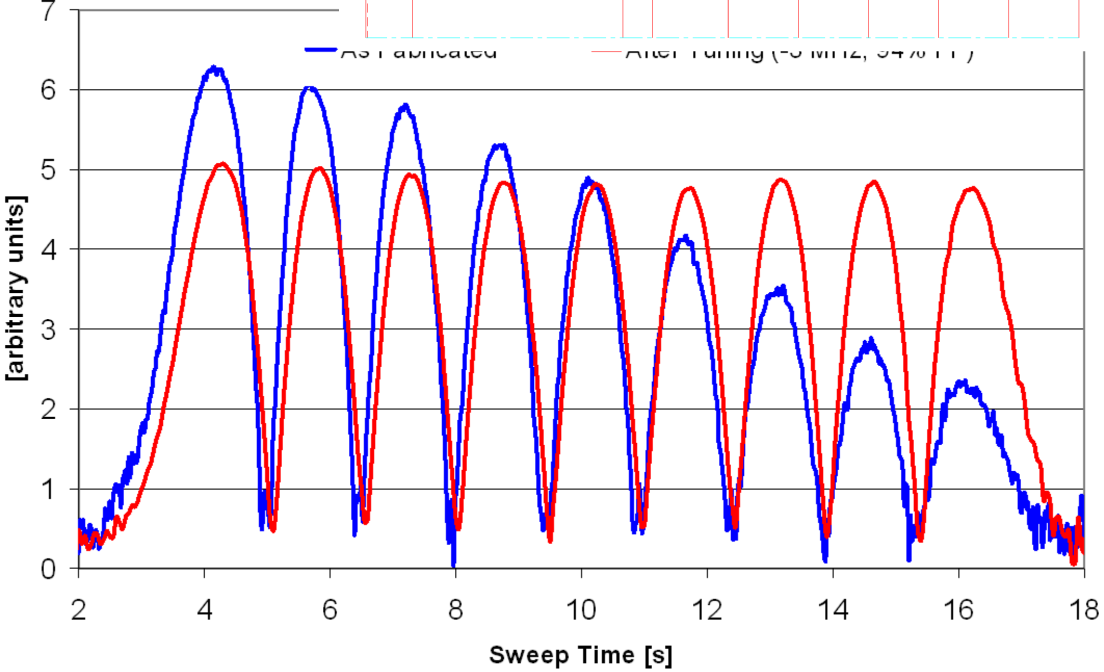
- Low-temperature baking

Frequency tuning



- After the treatments, the cavity needs to be tuned to the **right frequency** and **field flatness**.
- This operation is done by **tuning each single cell** to achieve proper field distribution





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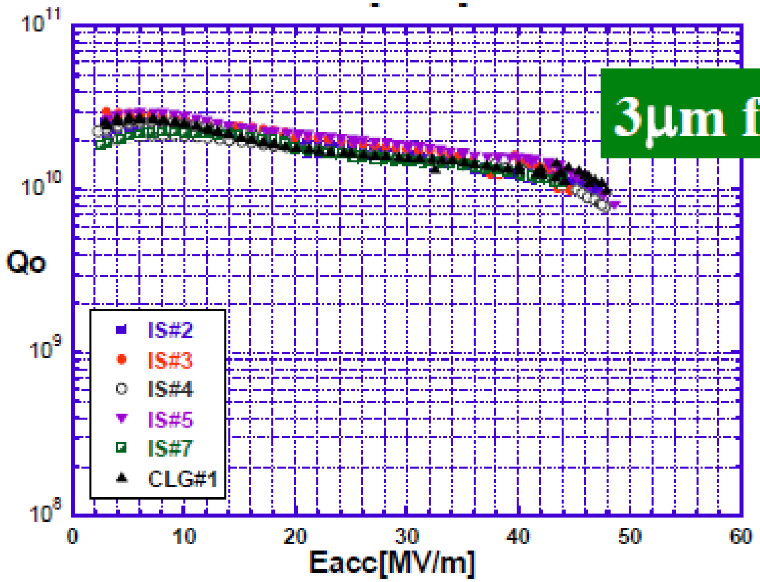
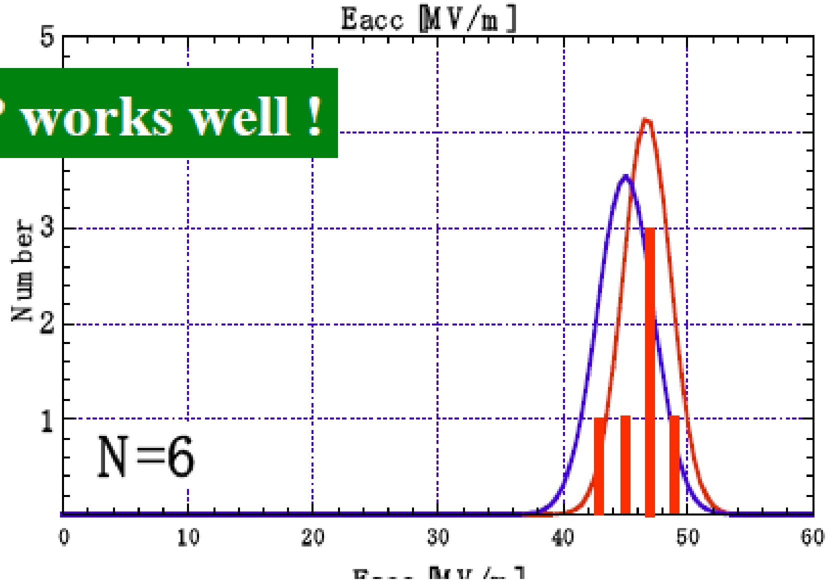
- Low-temperature baking

Post EP treatment

- Ethanol Rinse (DESY)
- "Flash" BCP (10 μm) (DESY)
- "Flash" EP (3 μm, fresh acid, no re-circulation) (KEK)
- Ultrasonic Degreasing with Micro-90 and hot water (JLab)



3μm fresh EP works well !



Courtesy G. Ciovati

Cavity preparation for SRF qualification

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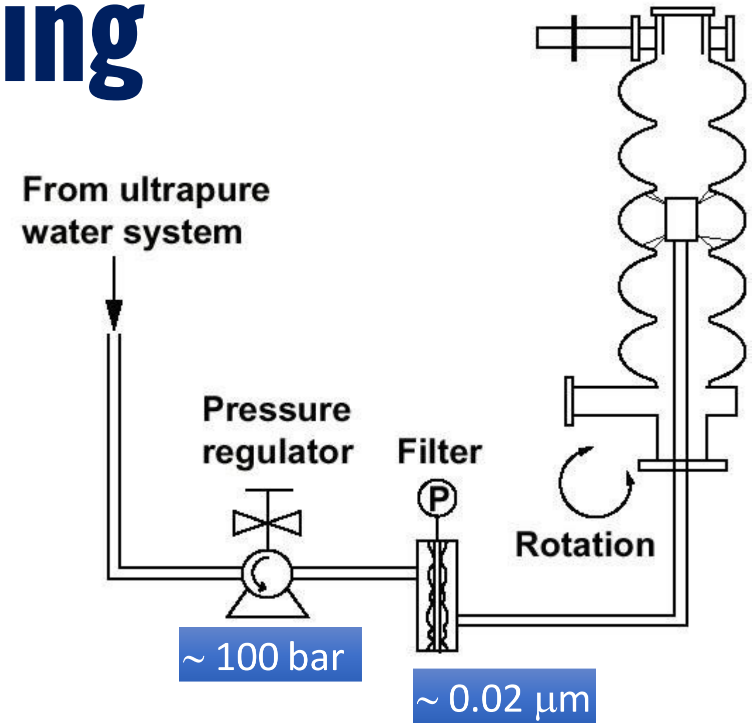
High Pressure Rinsing

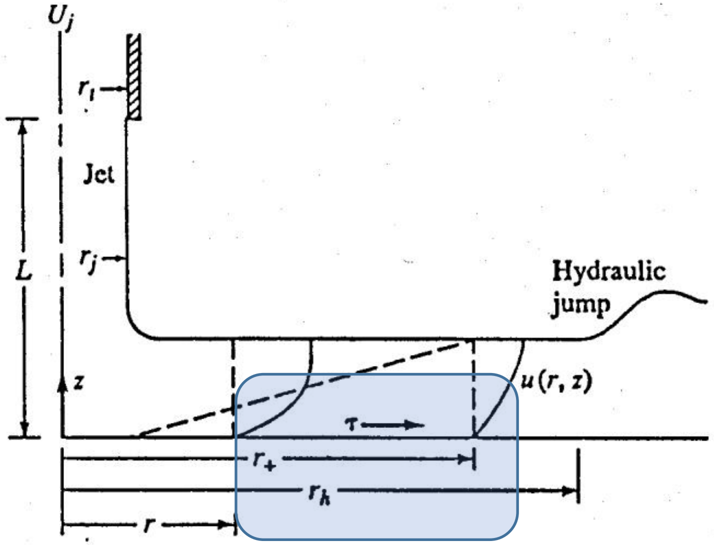
- The **final step** in cavity assembly is the **Rinsing with a High Pressure UPW** jet to remove particulate from the handling and residual from chemical treatments
- Water jet **must be moved continuously**: if jet impacts stably in one-point Nb surface can be damaged
- Continuous **motion of the cavity respect jets** (drawing a spiral behavior that cover completely the Nb surface)
- Ultra pure (6.0) filtered (40 nm) **nitrogen protection** gas injection **coaxial** with water to reduce risk of particles entering
- **Cavity** must be **grounded** otherwise it will be electrically charged



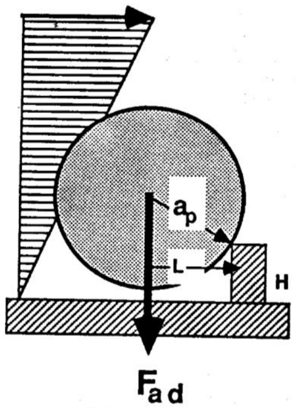
Oxidation induced by fixed HPR jets

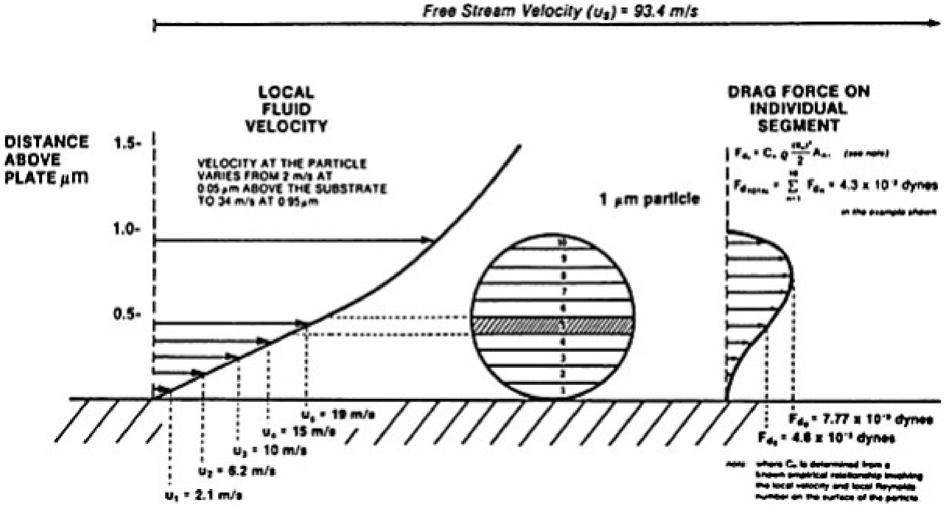
HPR Process

- Hydrodynamic model allows estimating the **shear stress τ of the water jet**, which depends on flow rate and pressure
- **Particle removal by rolling** if the water shear stress is greater than a critical shear stress τ_0, related to the particle size, adhesion force and surface roughness



$$\tau_0 = \frac{F_{ad}}{44\, a_p^2}\sqrt{2\frac{H}{a_p} + \left(\frac{H}{a_p}\right)^2}$$



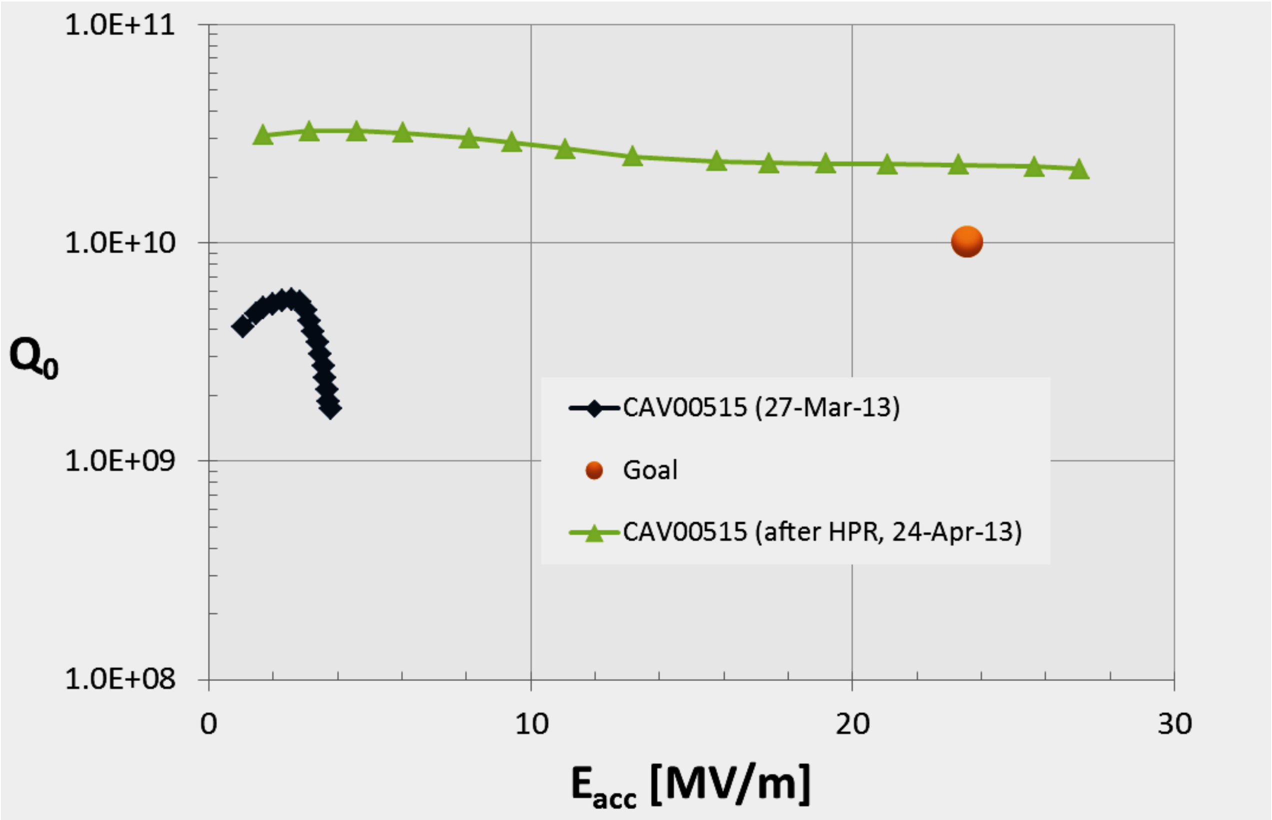


https://doi.org/10.1063/1.494924

HPR Effect

Cavity had a **problem** in the 120°C treatment (vacuum system power failure) that **produced a rapid change of pressure (a bump) in the cavity during last pump-down**, with particle movement in the system. Consequences are clearly visible (**dark blue curve**).

After HPR, with **no further chemical etching**, cavity performances are completely recovered.



HPR Systems


Rinsing cabinet of
“old“ DESY HPR system


Rinsing cabinet of “new“ DESY
HPR system with “plastic” cavity


CEA HPR system

HPR QA

- **Examples of QC at HPR systems**
  (DESY, EXFEL cavities production@ companies)

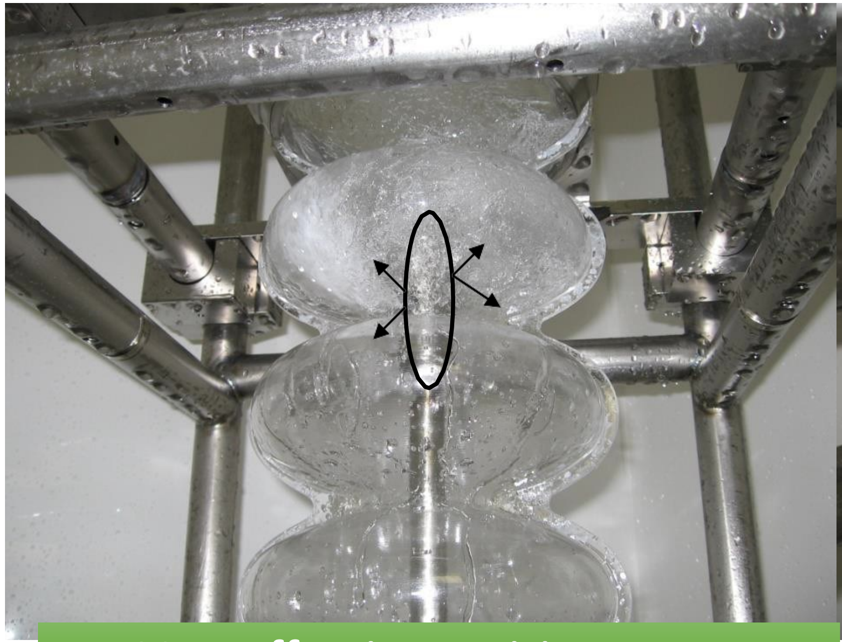
- Check of Point-of-use supply water quality:
  - **UPW conductivity**
  - **Particles**: online particle counter
  - Particles: off-line sampling & identification (SEM optical microscope)
  - **TOC: online monitoring**
  - **TOC drain line: sampling, after maintenance**
  - **Bacteria (=> offline)**

HPR Head QA

HPR Water Collector

HPR Spray Head Optimization

- For a given pump displacement **the nozzle opening diameter and number of nozzles sets the system pressure and flow rate**
- The **HPR spray head needs to be optimized for each cell geometry**!



Very effective on irises

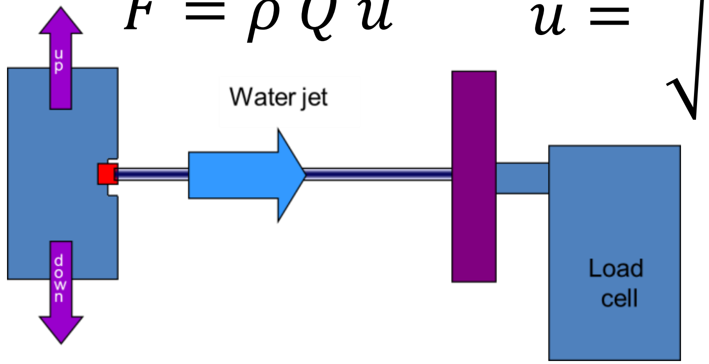
Equator fill with water → too high flow rate

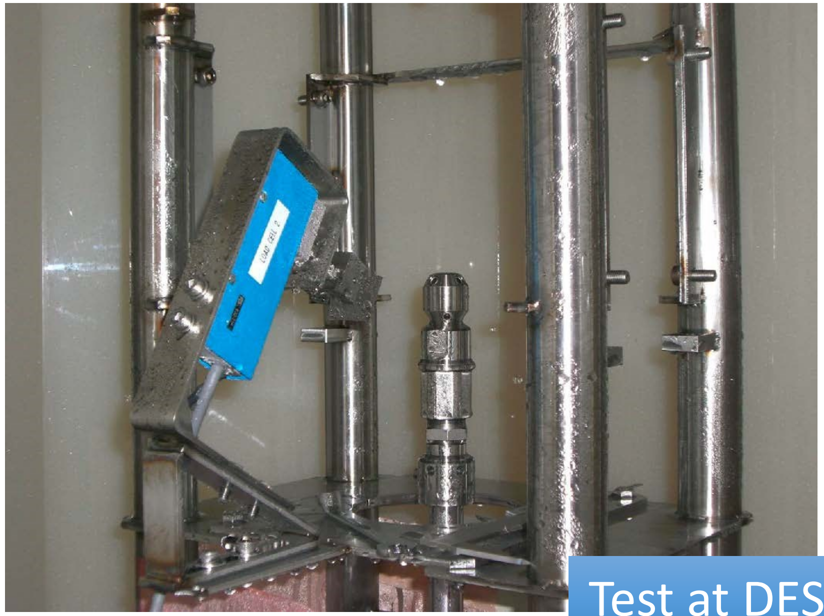
HPR Water Jet Characterization (INFN-LASA)

- Use a **load-cell to measure the jet force**

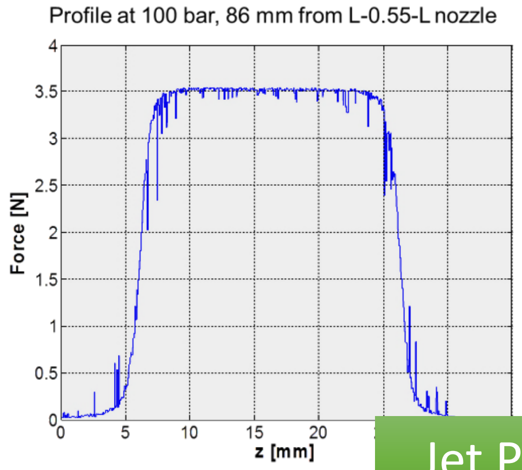
$$F = \rho\, Q\, u \qquad u = \sqrt{\frac{2\,p}{\rho}}$$

u = velocity
Q = flow
p = pressure
ρ = density

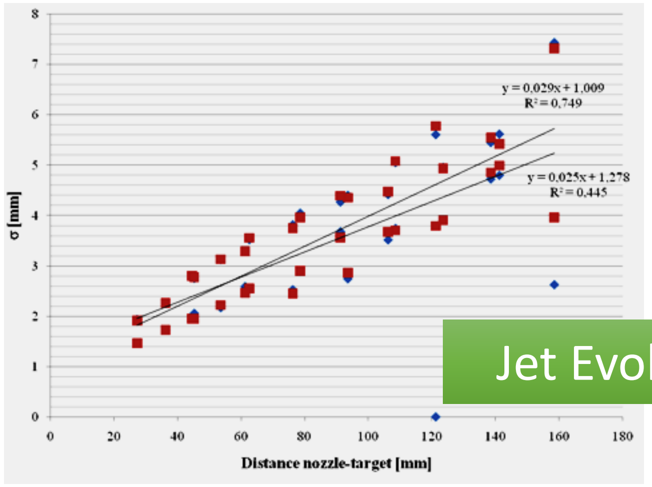




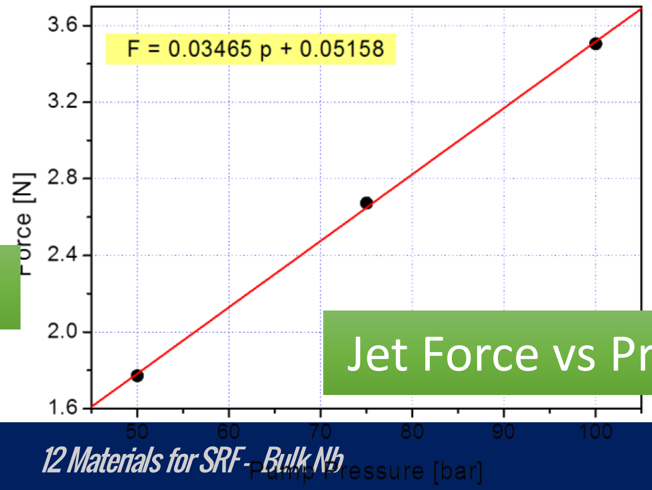
Test at DESY



Jet Profile



Jet Evolution



Jet Force vs Pressure

Cavity preparation for SRF qualification

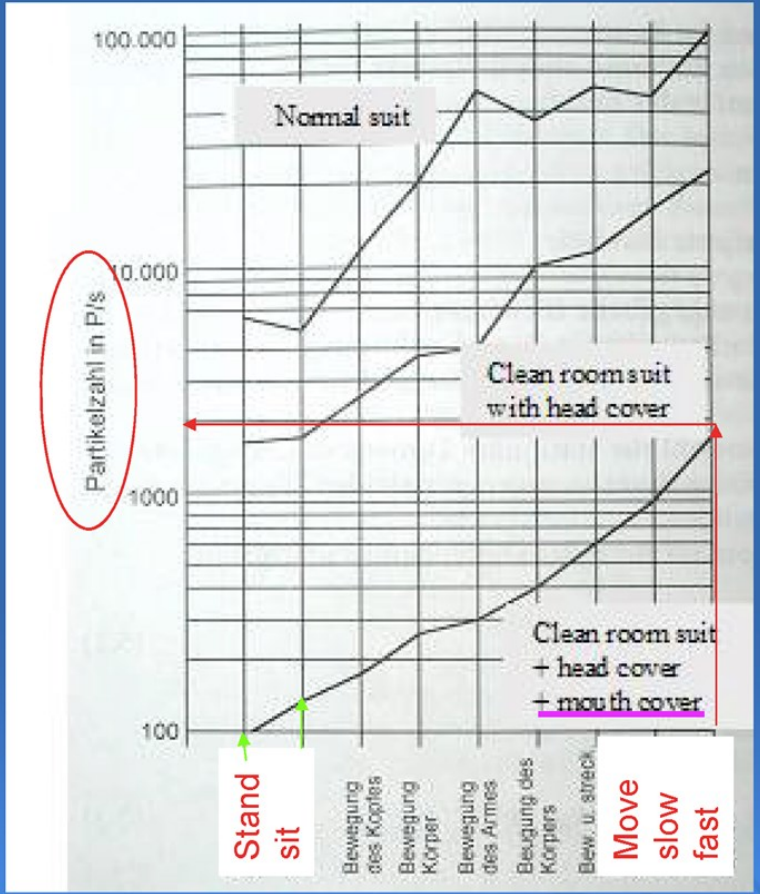
- Degreasing surfaces to remove contaminates
- Chemical removal of exterior films incurred from welding
- Removal of damage layer of niobium from fabrication (≈ 150÷200 μm)
- Removal of hydrogen from bulk Nb
- Mechanical tuning
- Chemical removal of internal surface for clean assembly (10-20 μm)
    - Additional "cleaning" steps if Electropolishing (EP) is used
- High Pressure Rinsing (HPR) to remove particulates from interior surfaces (incurred during chemistry and handling)
- **Drying of cavity for assembly in cleanroom (reduce risk of particulate adhesion and reduce wear on vacuum systems)**
- **Clean assembly**
- Clean evacuation
- Low-temperature baking

What is a cleanroom? The ISO 44644 definition

- "A room in which the **concentration of airborne particles** is **controlled**, and which is constructed and used in a manner to **minimize the introduction, generation and retention of particles** inside the room and in which other relevant **particles** inside the room and in which other relevant parameters, e.g. **temperature, humidity and pressure**, are **controlled as necessary**."
- **A cleanroom** is likely to have between **some tens of air changes per hour up to many hundreds of them.**
- A cleanroom uses filters that would normally be 99.97 % and more efficient in removing particles greater than 0.3 μm from the room air supply. These filters are known as **High Efficiency Particle Air (HEPA) filters, although Ultra Low Particle Air (ULPA) filters**, which have a higher efficiency, are used in microelectronic fabrication areas.

Human generated particle

One major part inside a cleanroom is PERSONAL



1st Dress code

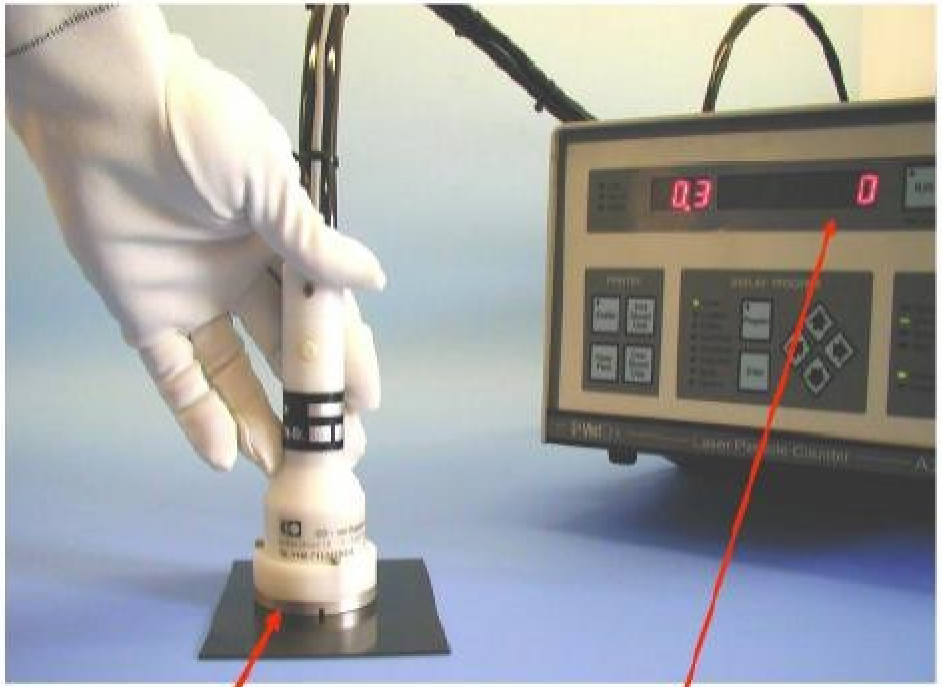
A.Matheisen SRF workshop 2007
Bejing China October 2007

Clean room «dress» code

- **People are a major source of particulate contamination** inside a clean room through:
  - Body Regenerative Processes - Skin flakes, oils, perspiration and hair.
  - Behavior - Rate of movement, sneezing and coughing.
  - Attitude - Work habits and communication between workers.

Particle counters

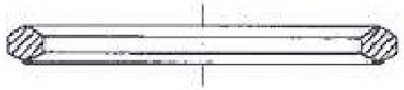
- To ensure the proper cleanness, **all components** before installation need to be **washed, rinsed and particle counted**



Cavity Assembly

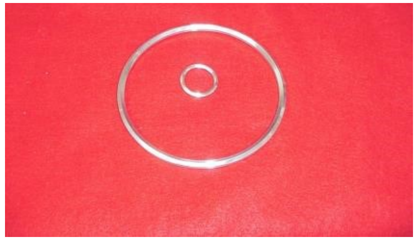
- The cavity strings have to be **vacuum tight to a leak rate of < 1 10^{-10} mbar l/sec**
- The **sealing gaskets and hardware** have to be **reliable and particulate-free**
- The clamping hardware should minimize the space needed for connecting the beamlines

AlMg-Gasket



- UHV Gasket
  - Present choice for SRF cavities:

    diamond-shaped $AlMg_3$ –gaskets + NbTi flanges + bolts



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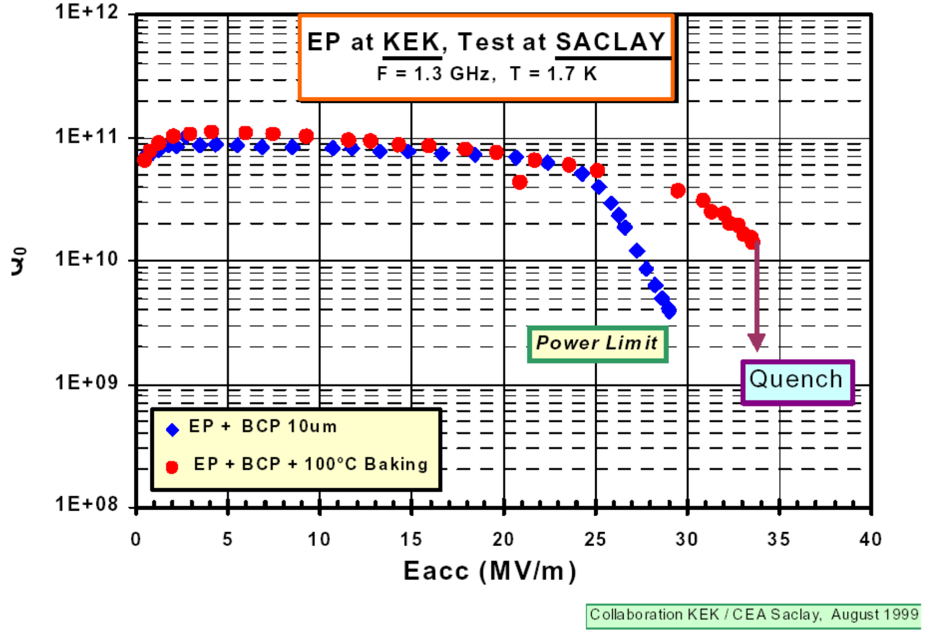
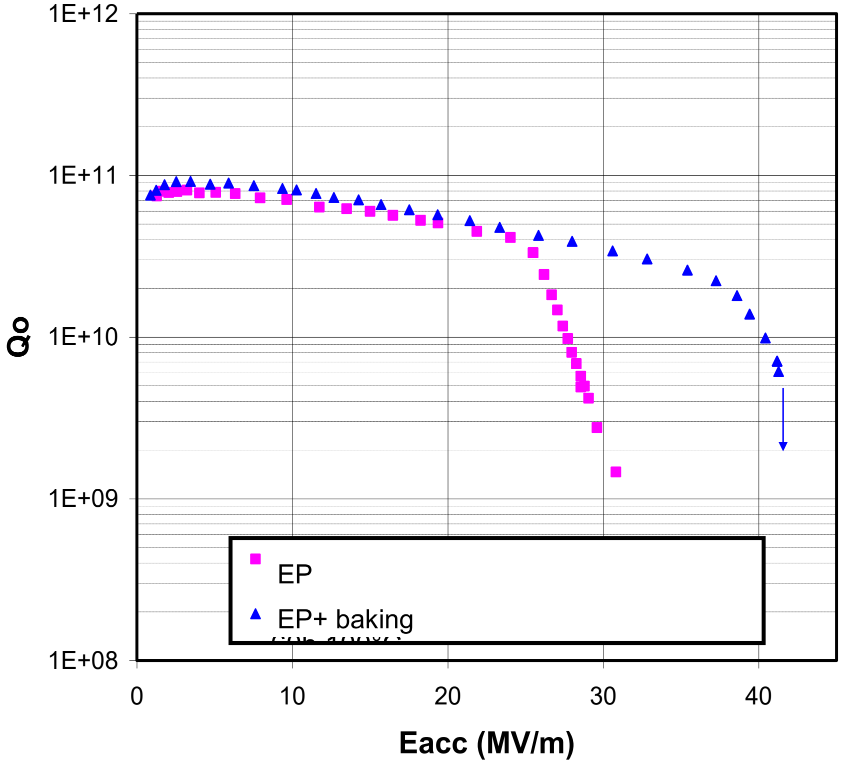
- Drying of cavity for assembly in cleanroom (reduce risk of particulate adhesion and reduce wear on vacuum systems)
- Clean assembly
- Clean evacuation
- **Low-temperature baking**

Why low temperature baking (120 °C)?

- **Baking:** shifts high field dissipation to higher field
  - Discovered at Saclay in 1998 (B. Visentin)
  - Low temperature treatment : 110-120°C, 48 H : few changes expected
  - **Dramatic effect on performances**
  - **Still resists full explanation**

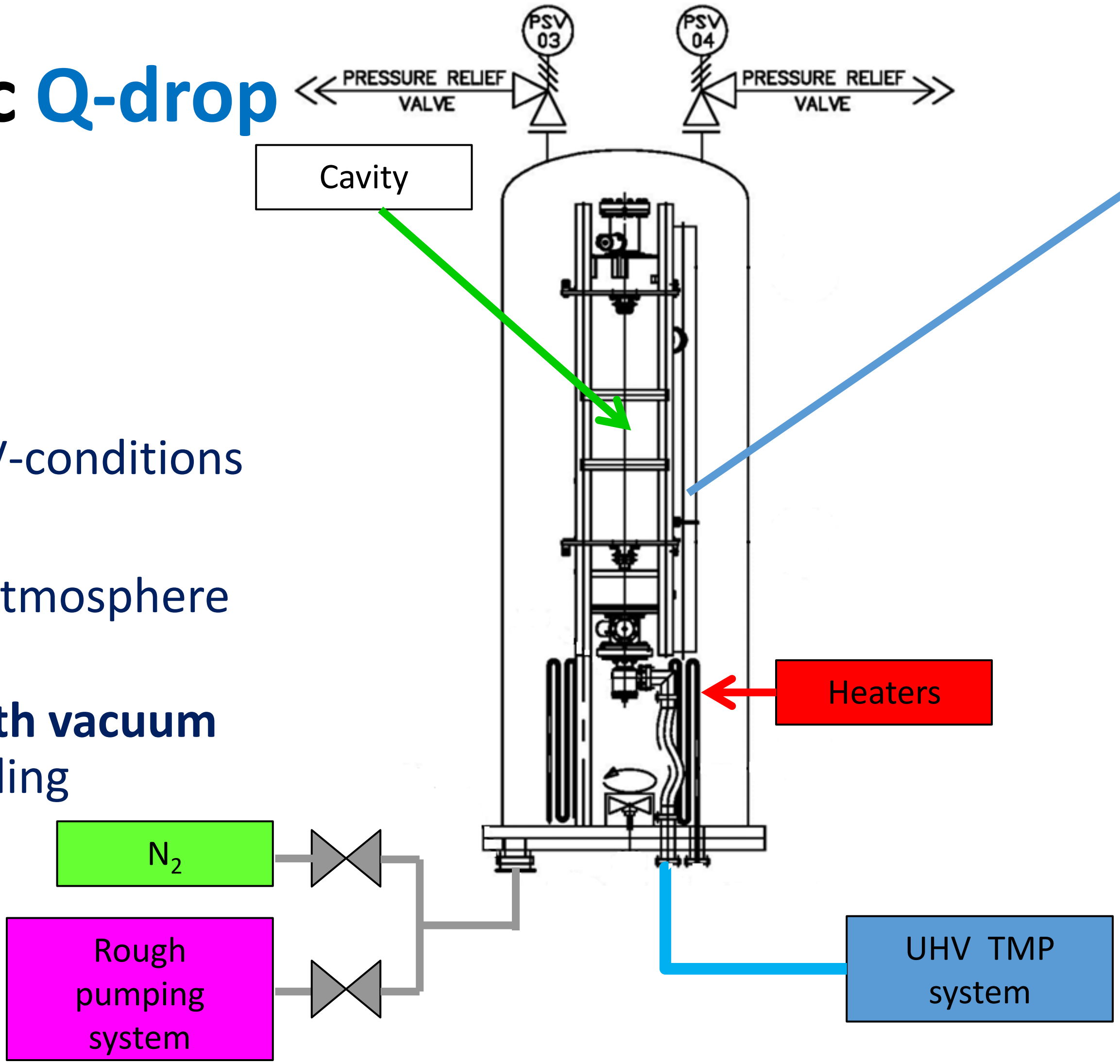


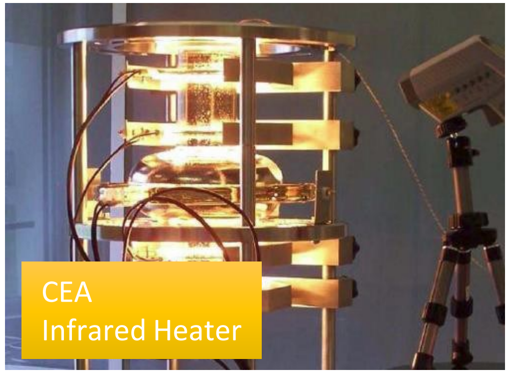


120 °C baking

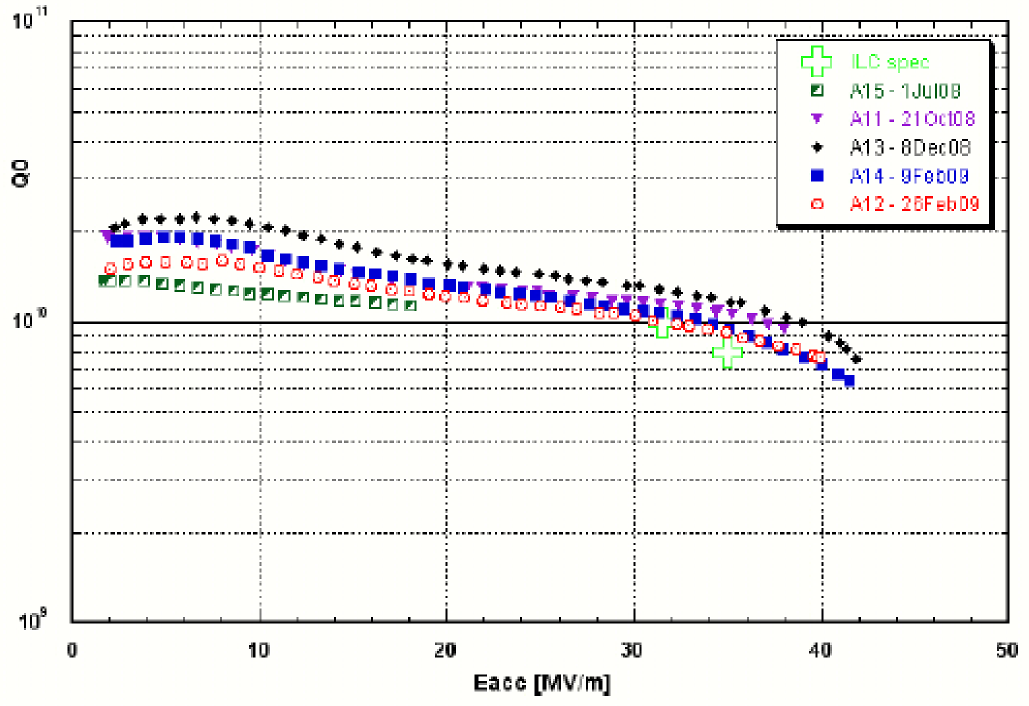
Cure of characteristic Q-drop

- **Standard recipe:**
  - **T = 110 - 125°C for 48h**
  - **Active pumping** (TMP)
  - Oil free vacuum system, UHV-conditions
  - Fully assembled cavity
  - **Nitrogen or argon** external atmosphere to avoid oxidation
  - External volume: **purging with vacuum before** inert gas (N_2 or Ar) filling

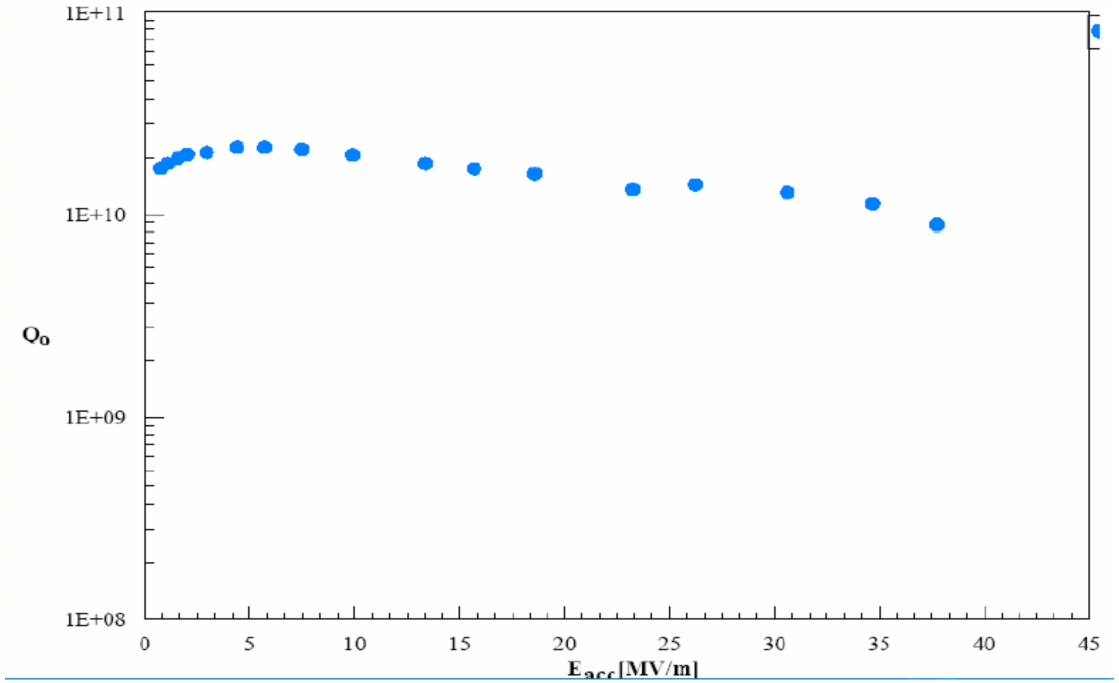




If everything went well you will get …



JLAB

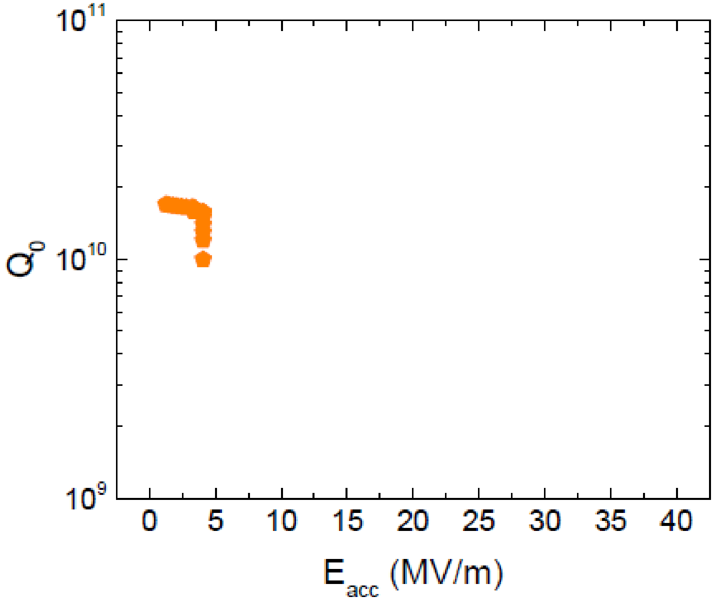


DESY

$E_p \cong$ 80 MV/m, $B_p \cong$ 170 mT can be achieved in the vertical test of 9-cell ILC cavities (~ 1 m² of Nb surface)

Nb bulk cavities: performance evolution

SRF Performance Evolution



1.3 GHz, 2K

3 – 4 MV/m
Multipacting

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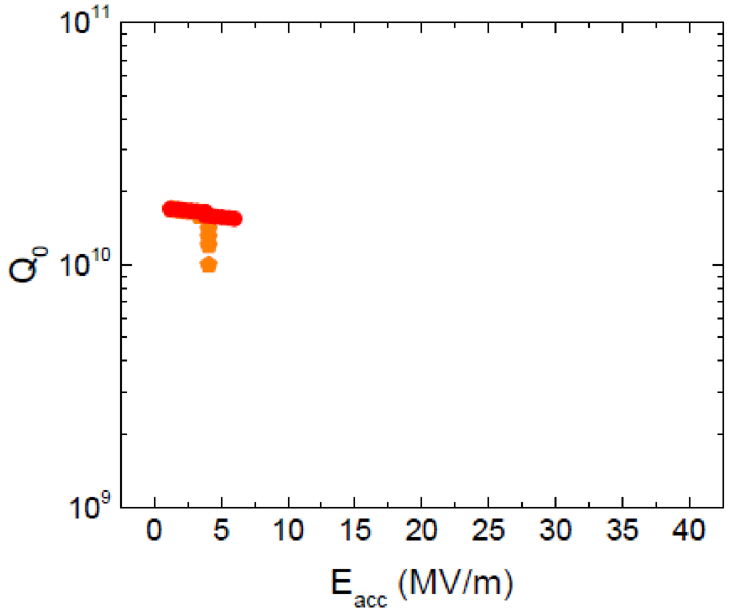
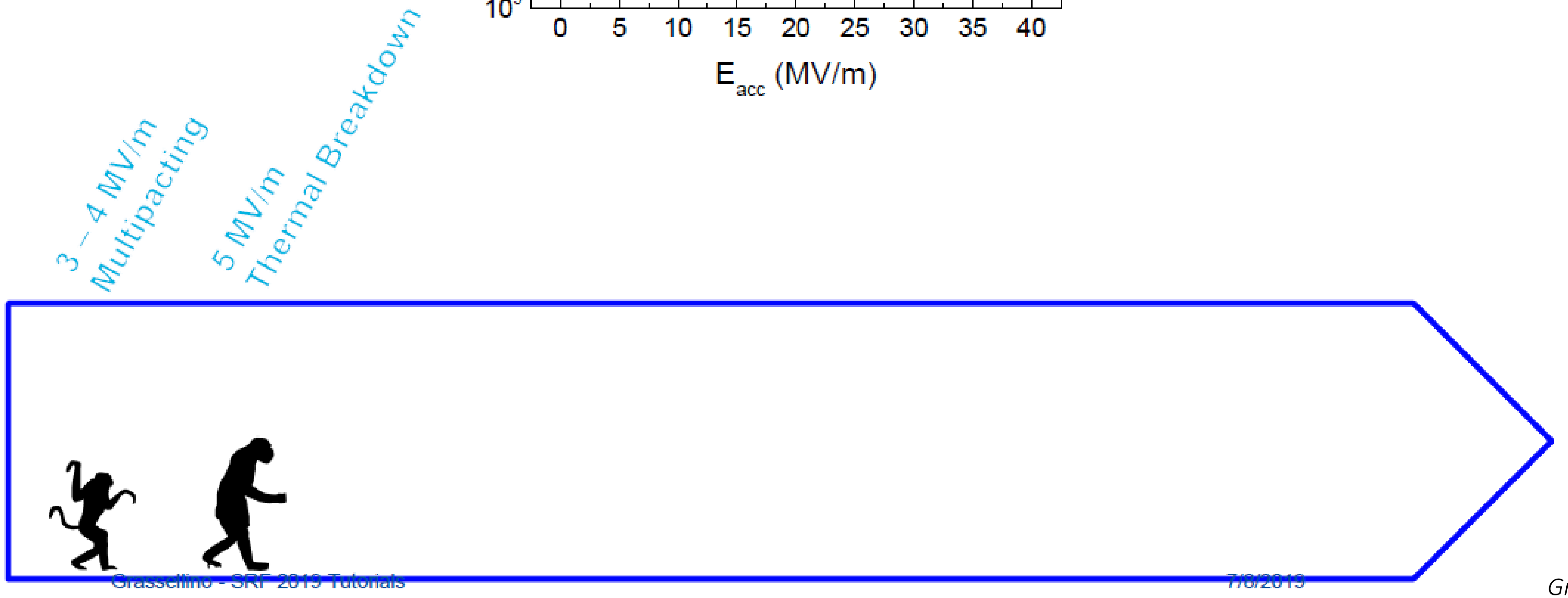
SRF Performance Evolution



1.3 GHz, 2K



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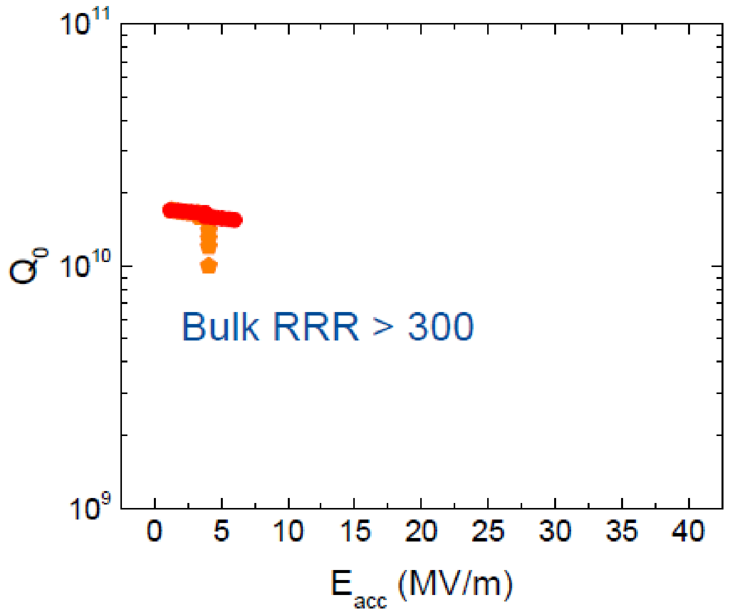
SRF Performance Evolution



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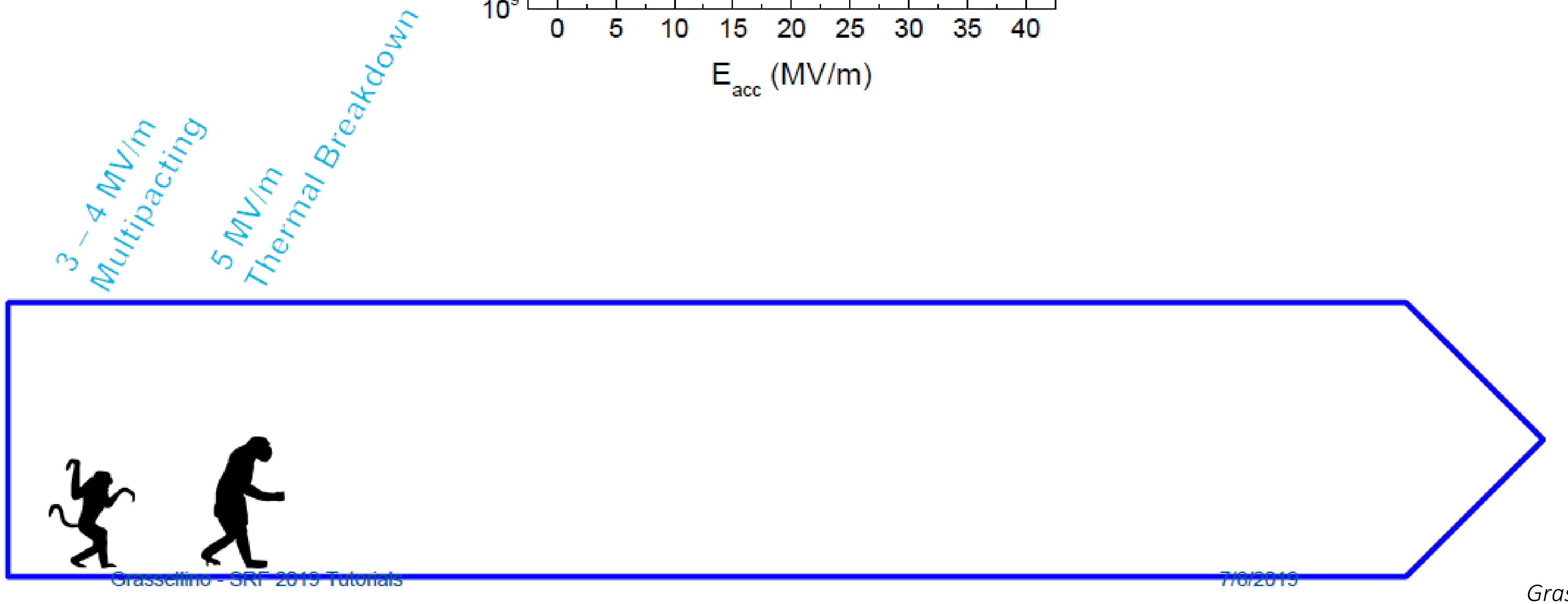

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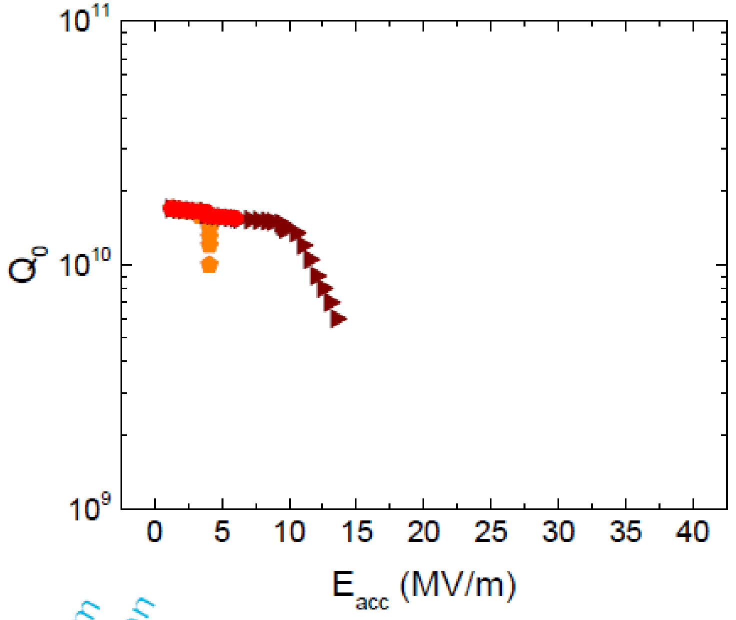
SRF Performance Evolution



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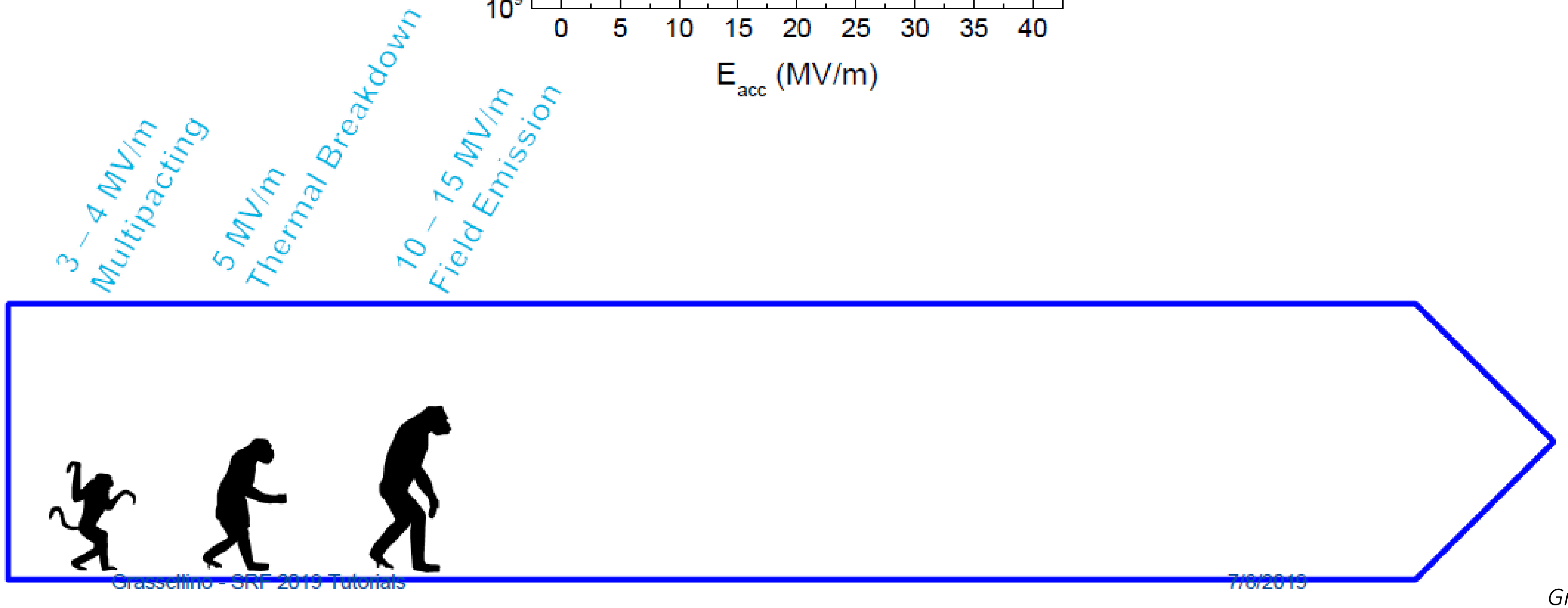

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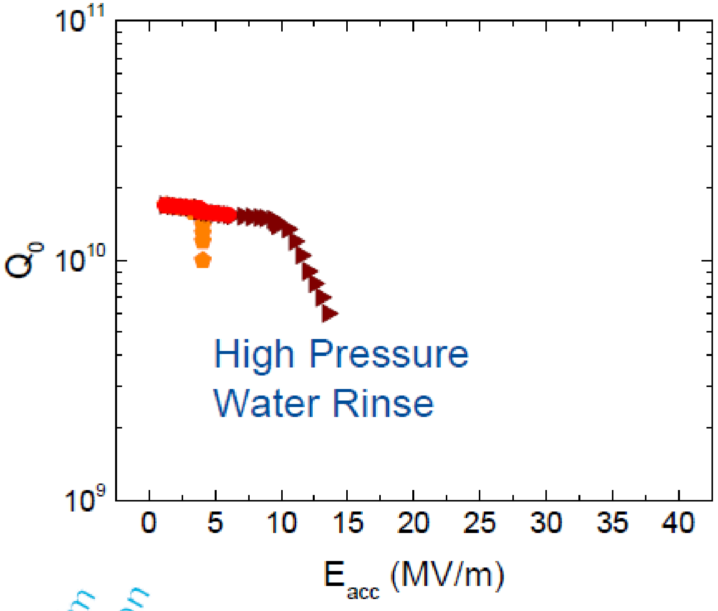
SRF Performance Evolution



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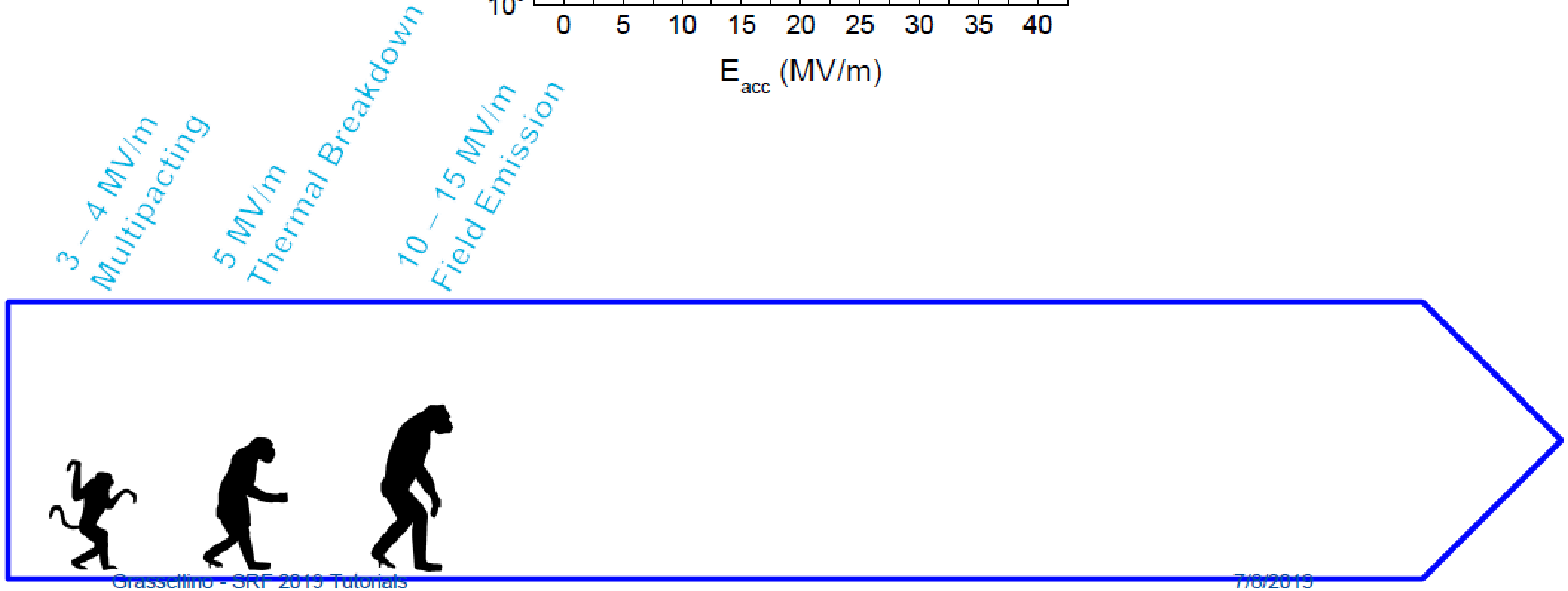

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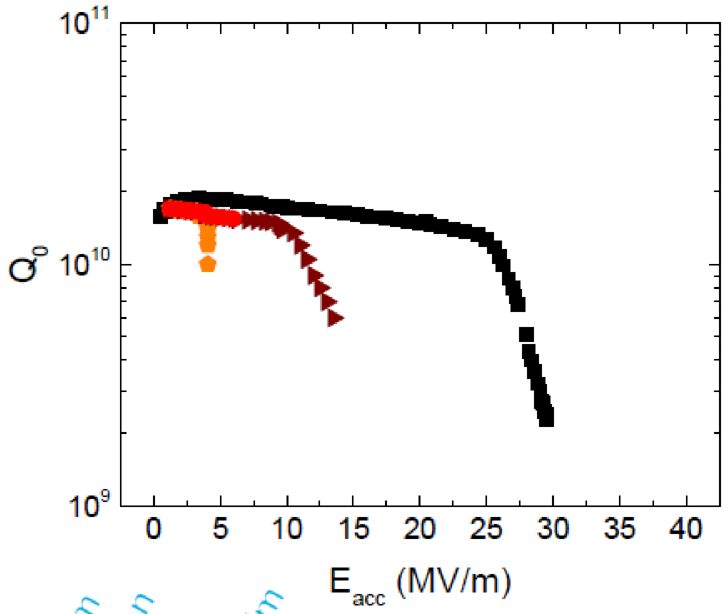
SRF Performance Evolution



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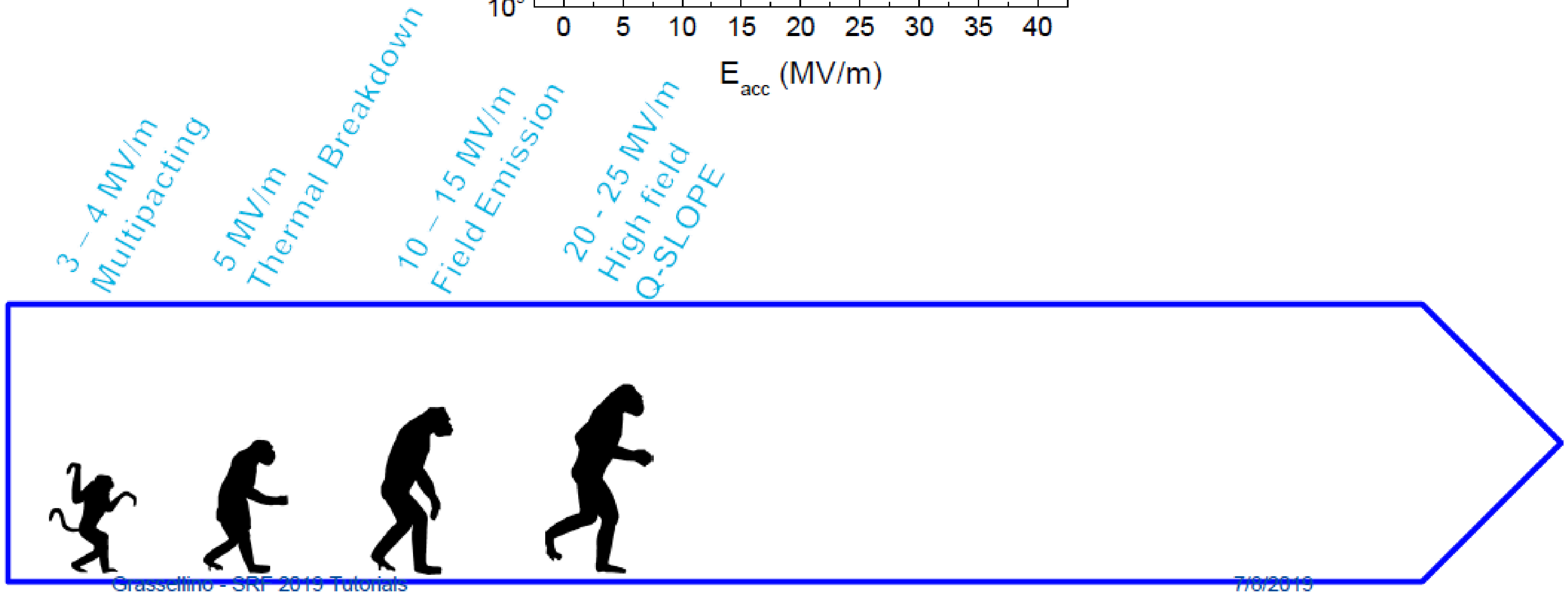

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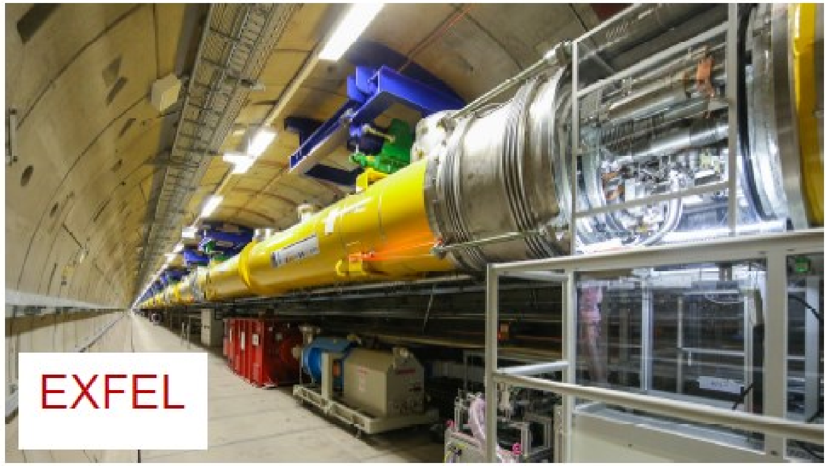
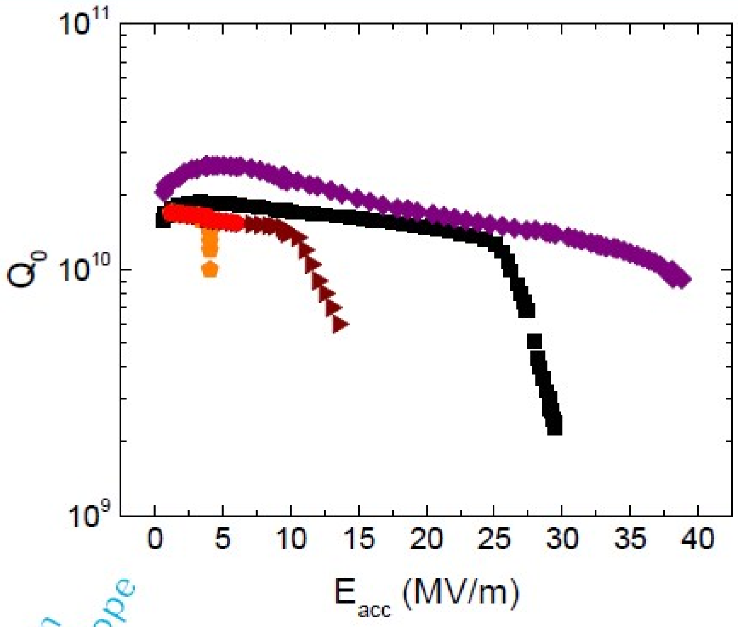
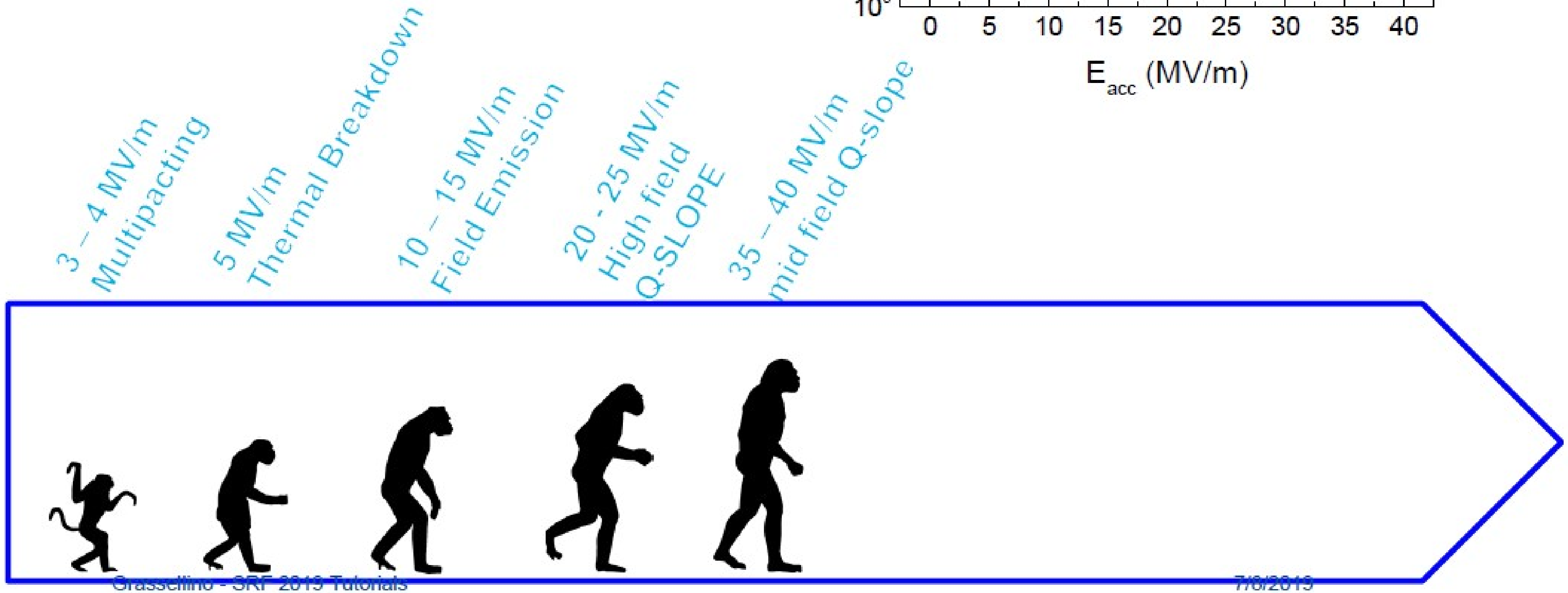
SRF Performance Evolution

1.3 GHz, 2K







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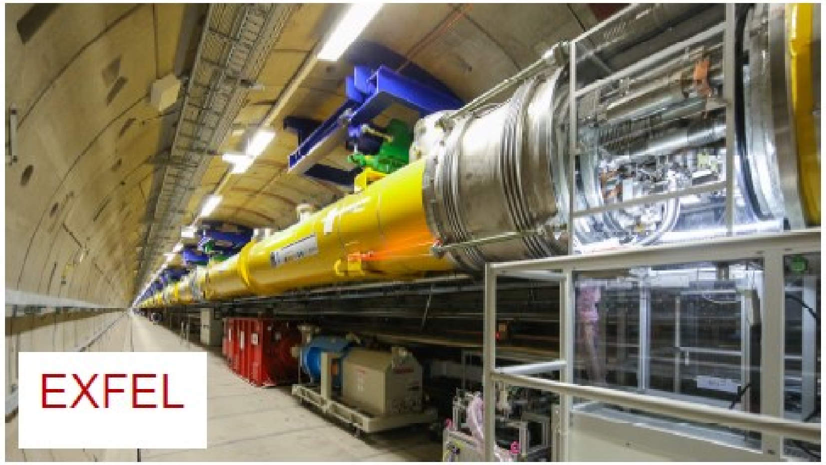

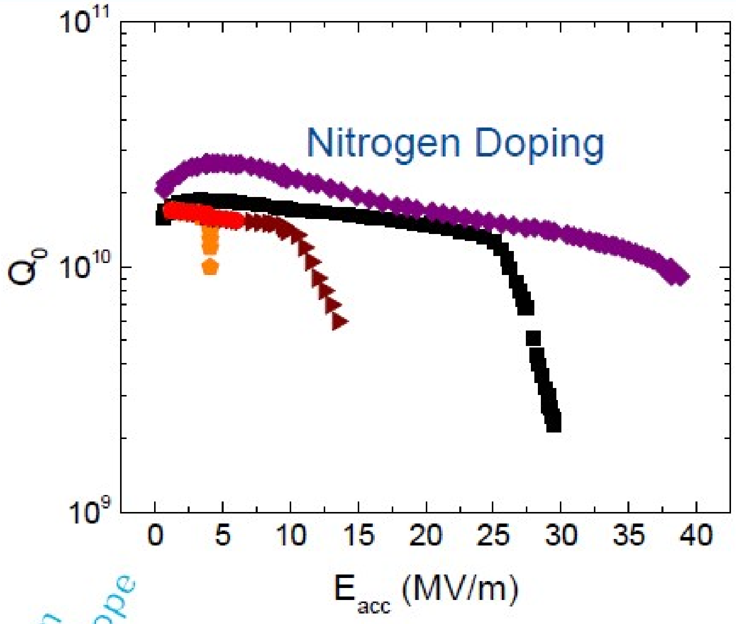

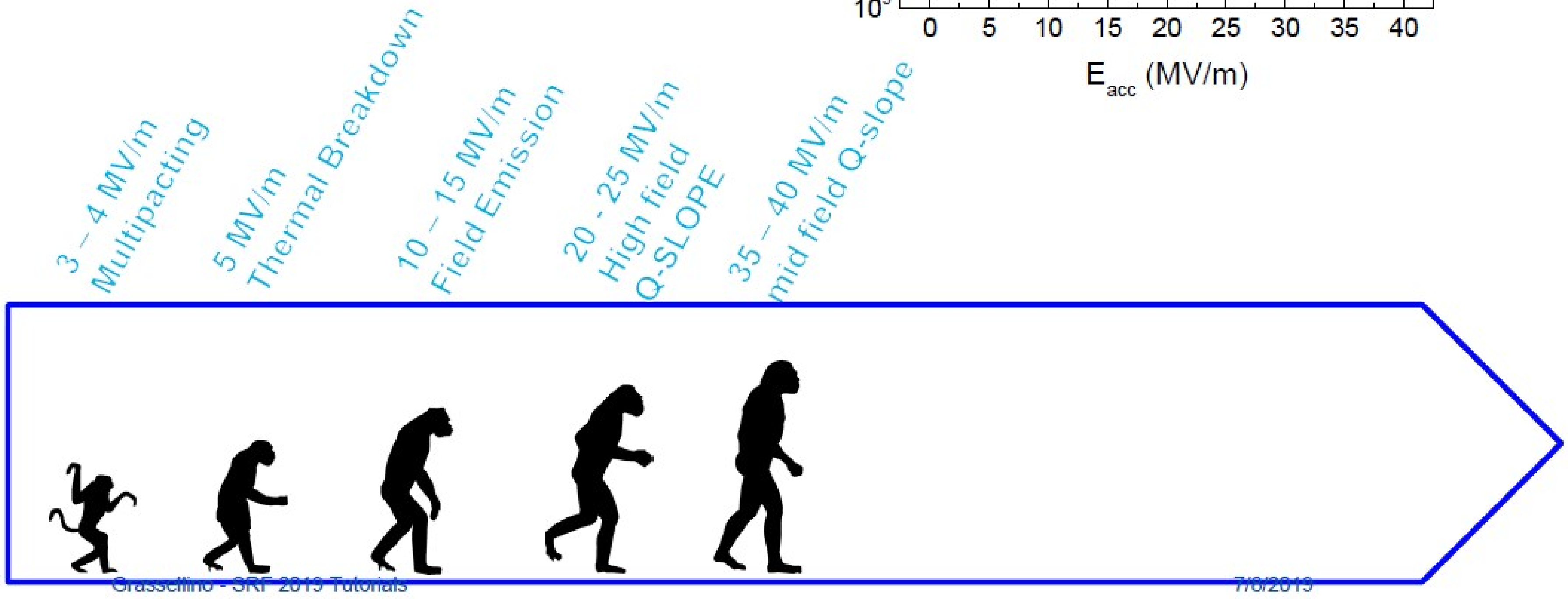

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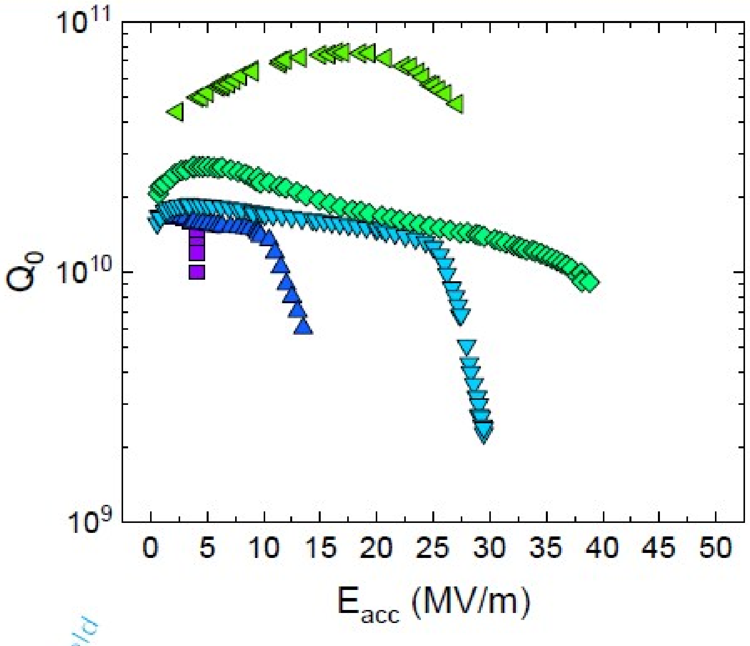
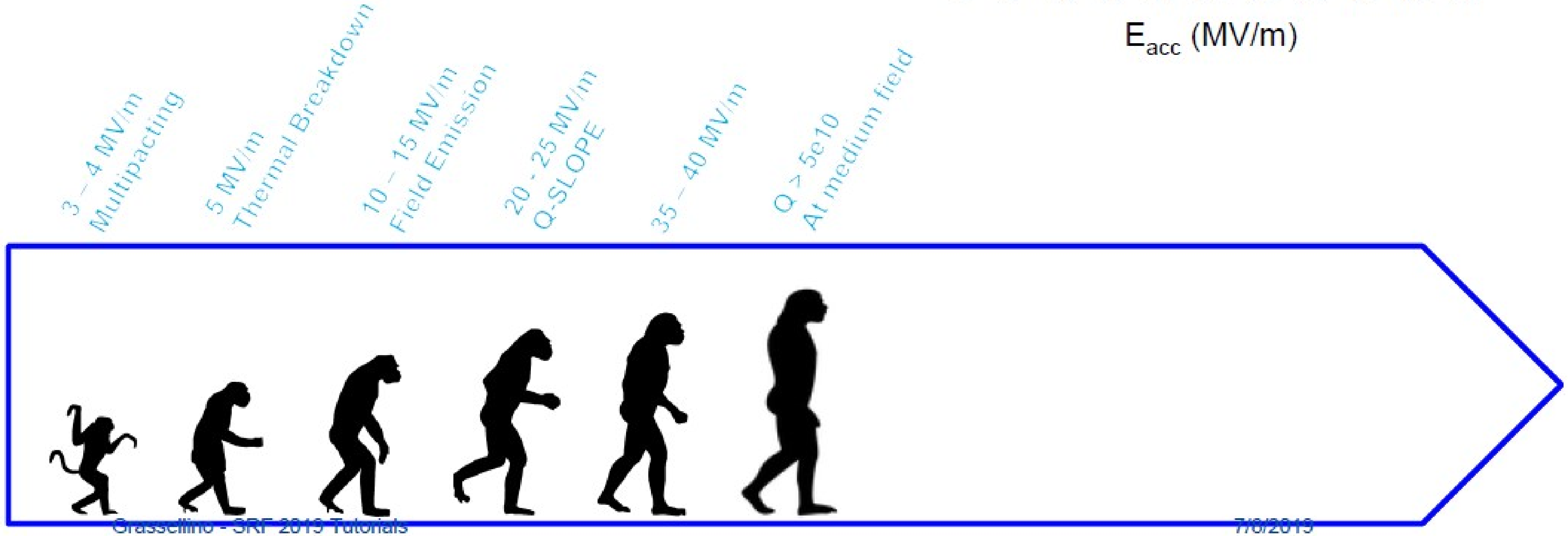
SRF Performance Evolution – 2013: N doping!





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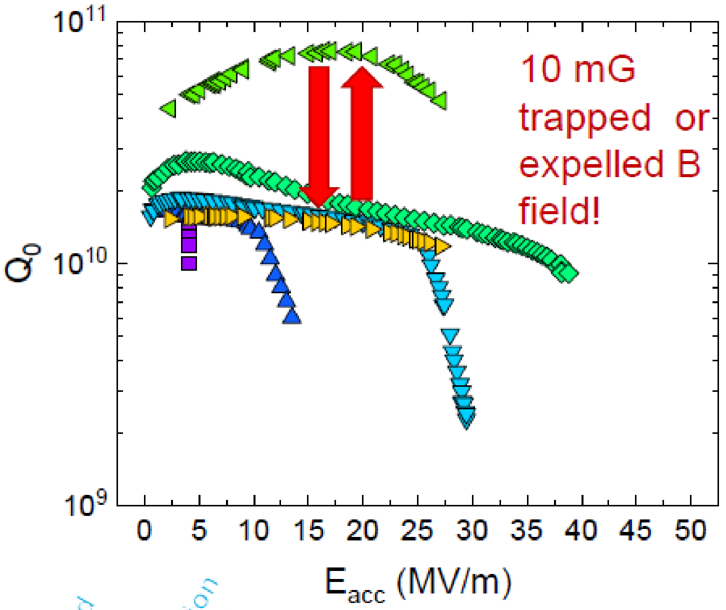
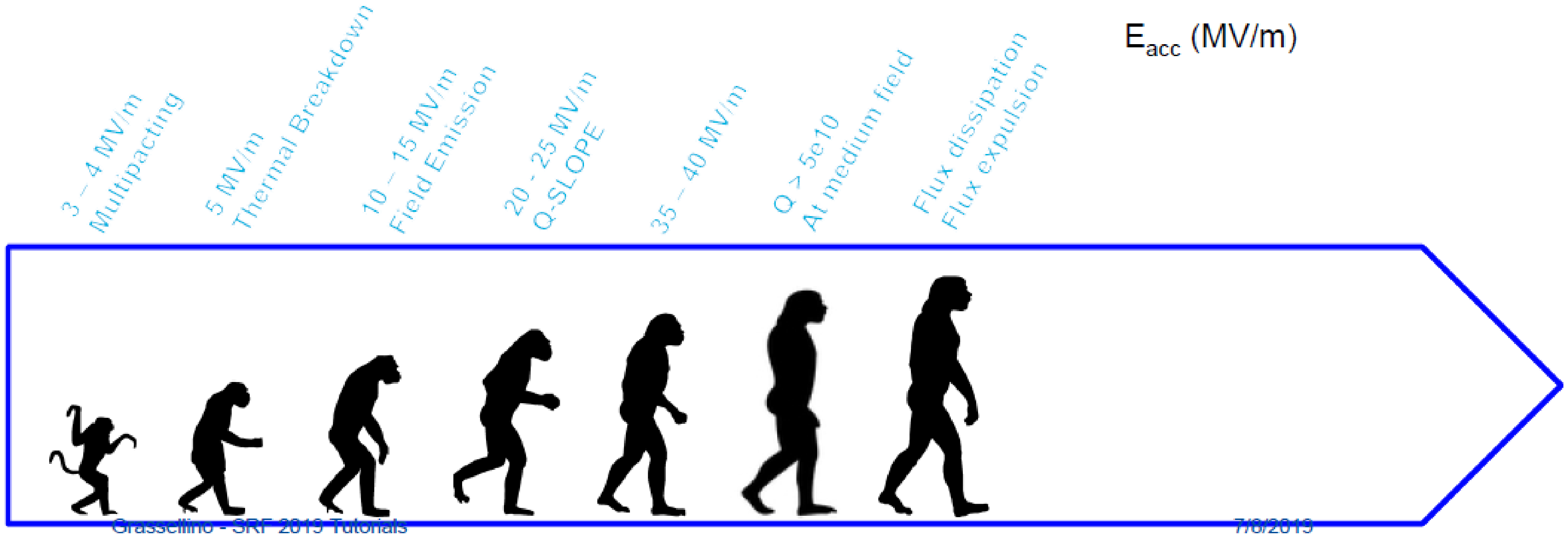
SRF Performance Evolution – 2014: Magnetic flux trapping with slow cooldown/ efficient expulsion with fast





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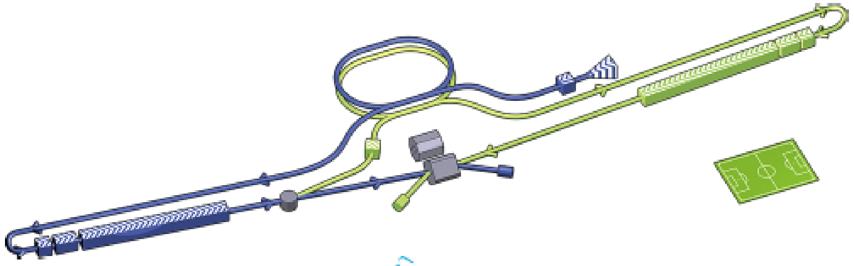
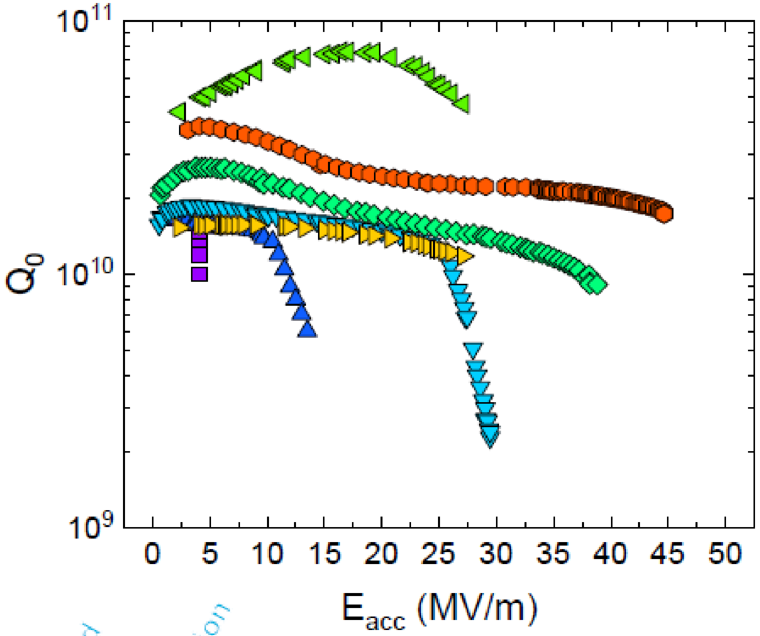
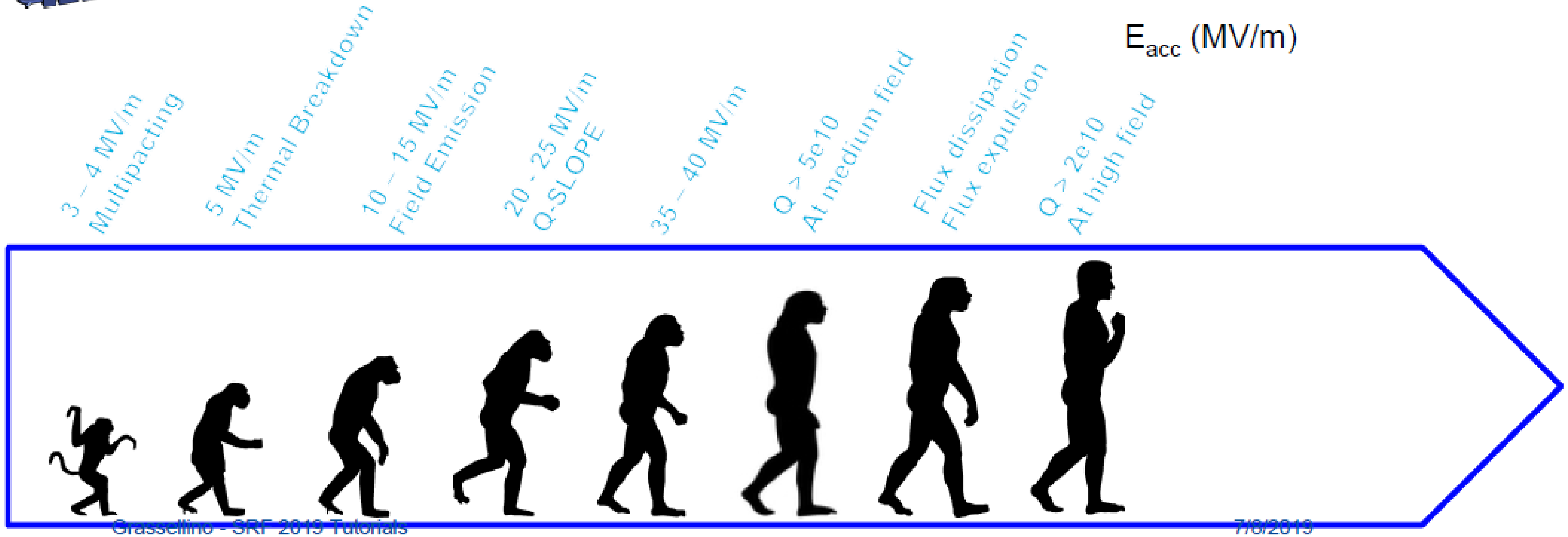
SRF Performance Evolution – 2017: N infusion

ILC cost reduction??







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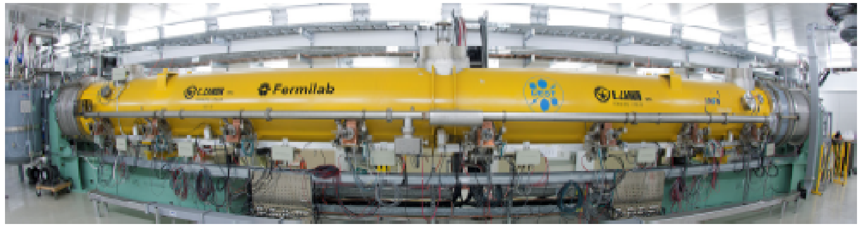
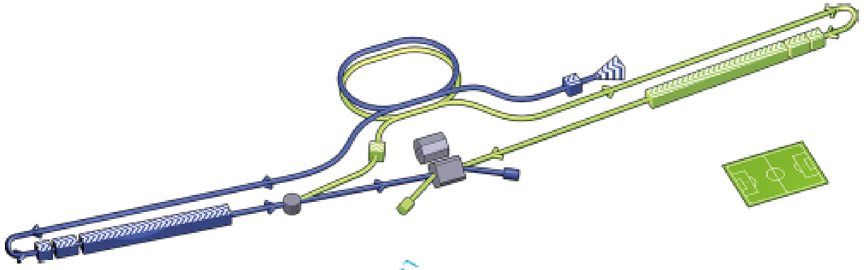
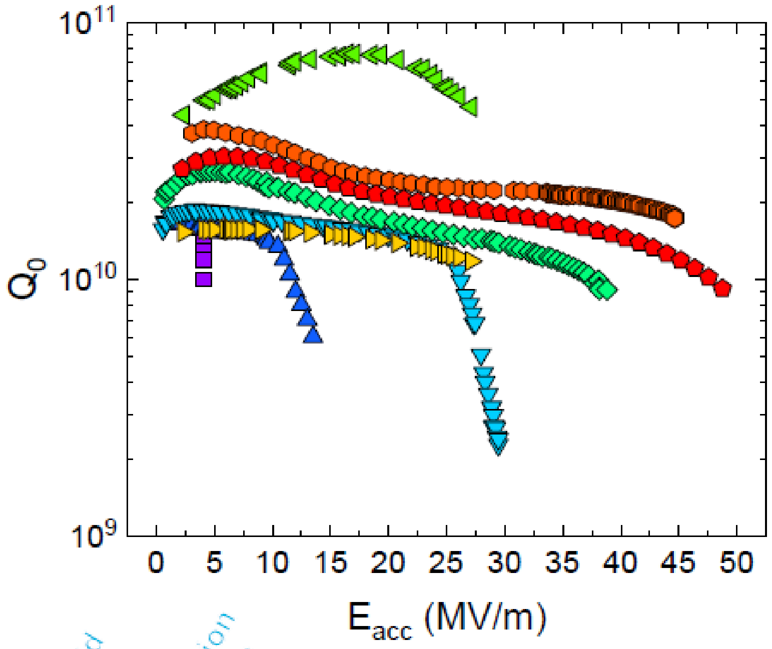
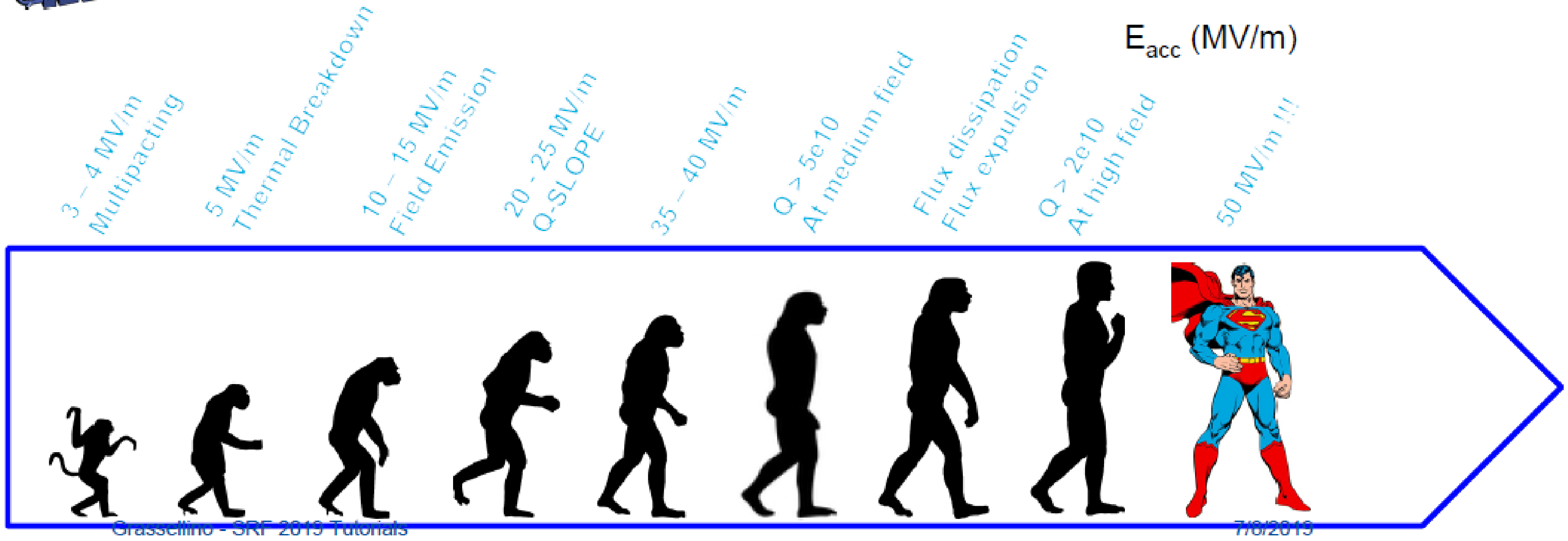
SRF Performance Evolution – 2018: the 75C bake

ILC cost reduction??









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Cavitie's general fabrication scheme

	WHY	COMMENTS
Forming		
EB Welding	Clean welding	Nb = getter material. If RRR/ 10 @ welding => $Q_0/10$
Ti purification	RRR enhancement	RRR 300-400 now commercially available
Chemical etching 100-200 µm (BCP / EP)	Remove contamination and damage layer	Limitation : BCP ~ 30MV/m; EP => >40 mV/m but lack of reproducibility
Annealing 800°C, 2h (or 600°C, 10h)	Get rid of hydrogen	Source of H: wet processes H segregates near surface in form of hydrides (= bad SC)
Chemical etching 5-20 µm	Remove diffusion layer (O, C, N)	Diffusion layer < ~1µm in bulk, a little higher at Grain Boundaries
Specific rinsing	e.g. remove S particles due to EP	Under evaluation HF, H_2O_2, ethanol, degreasing,…
High pressure rinsing (HPR)	Get rid of dust particles	Not always enough (recontamination during assembly)
Assembling	Ancillaries : antennas, couplers , vacuum ports…	**In clean room, but recontamination still possible**
Baking, 120°C, 48h	Decrease high field losses (Q-drop)	Unknown mechanism, first 10 nm of the surface in concern.
Post processing	Get rid of "re-contamination" ?	Under evaluation: dry ice cleaning, plasma
Test RF	Cavity's performance	First naked cavity in vertical cryostat, then dressed in horizontal cryostat/ accelerating facility
He processing, HPP	Decrease field emission	RF power with/ without He to destroy field emitters (dust particles) NB field emission : principal practical problem in accelerators

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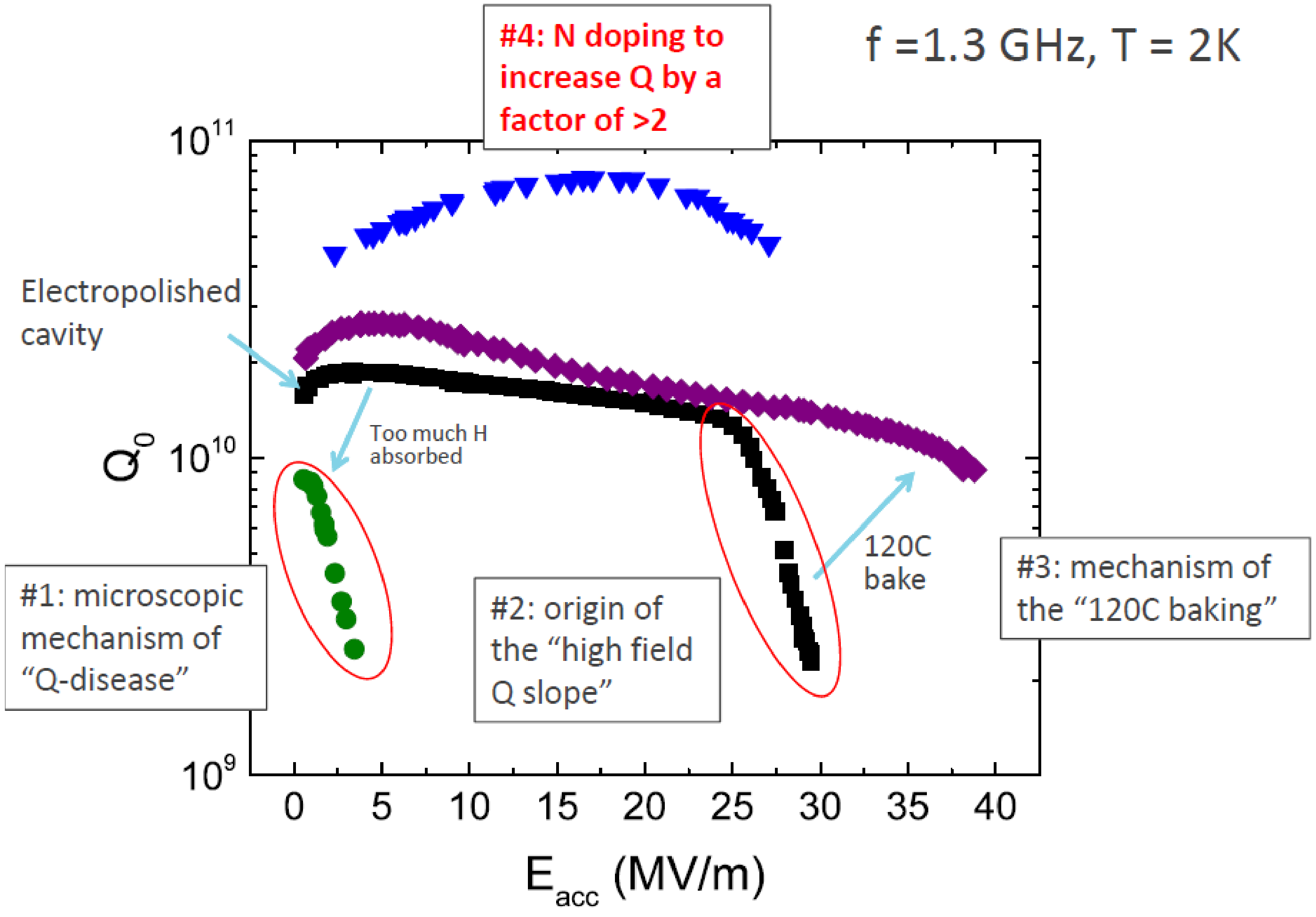
He processing, HPP	Decrease field emission	RF power with/ without He to destroy field emitters (dust particles) NB field emission : principal practical problem in accelerators

Possible explanations to performance limitations and solutions



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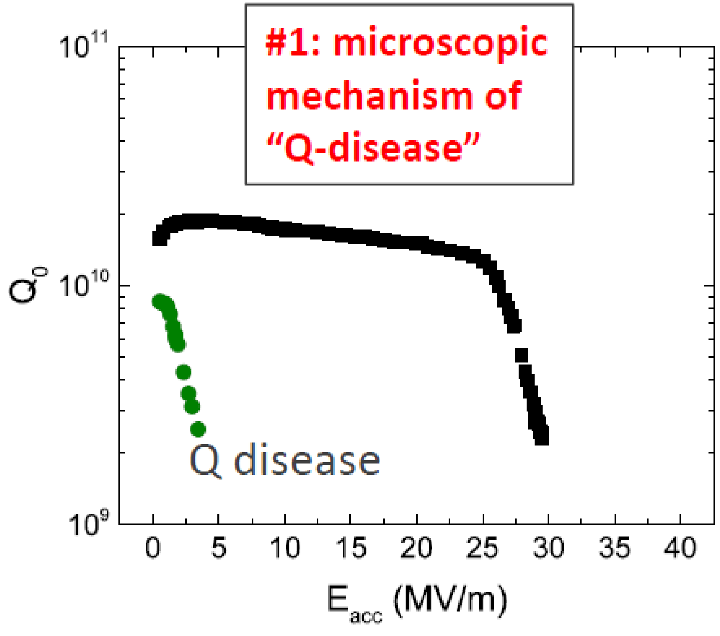
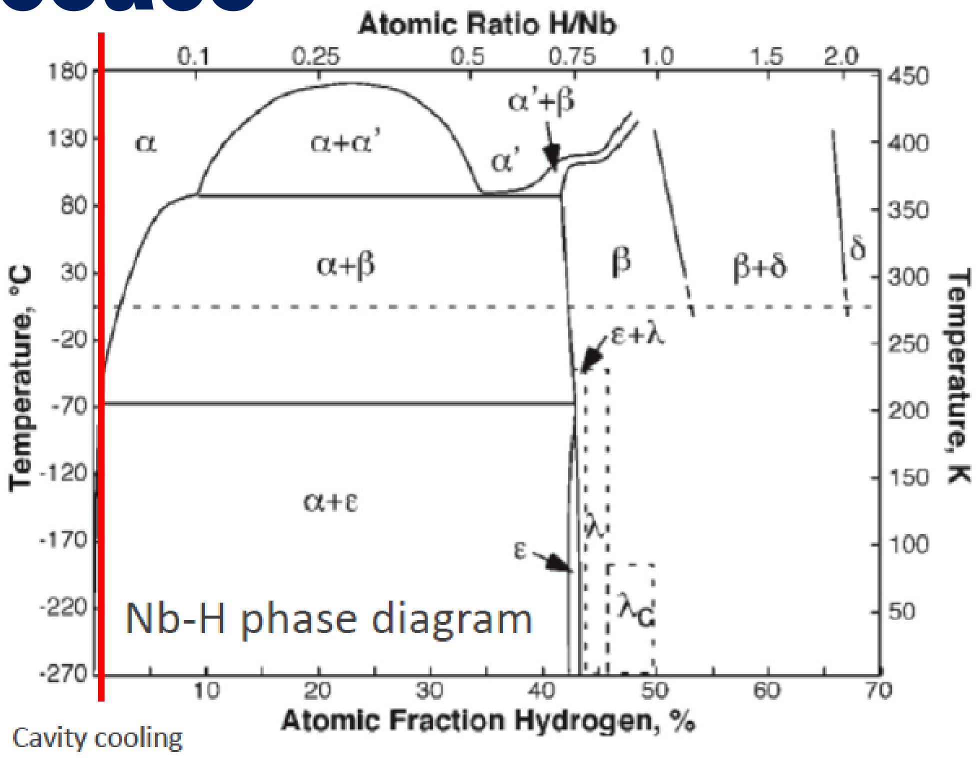
Q-disease





Q-disease is related to the **excess of hydrogen**, which forms non-SC Niobium hydrides upon cooldown

600-800 °C vacuum anneal to degas hydrogen

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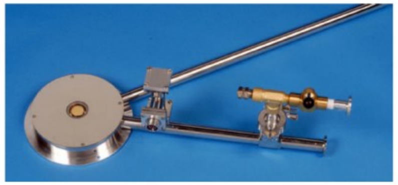
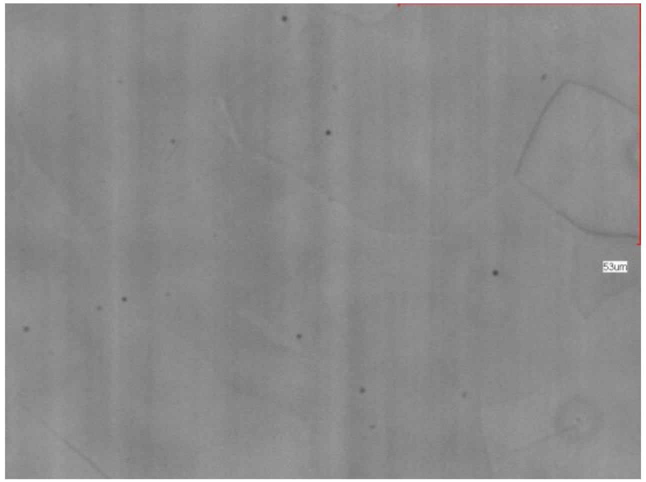
Microscopic Mechanism of Q-disease



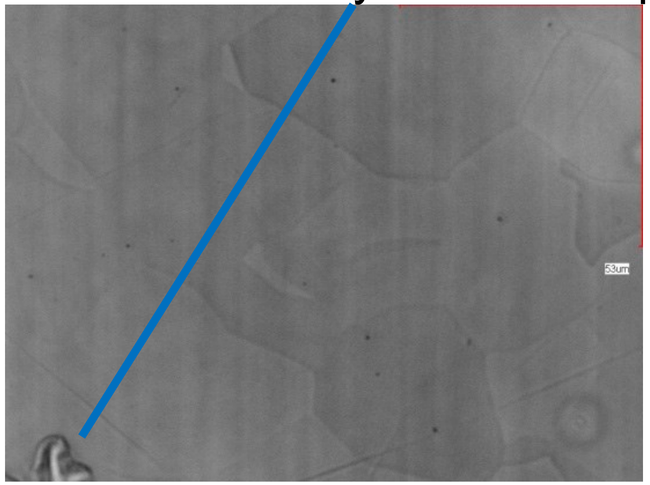
cryostage in the laser confocal scanning microscope

COOLDOWN



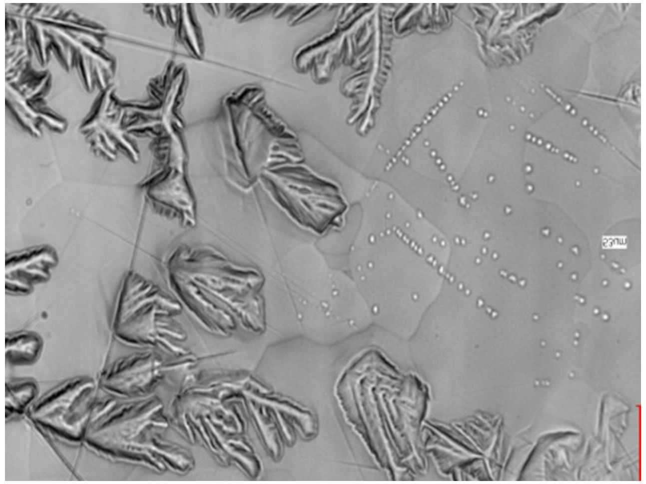
T = 300 K

Hydrides first appear

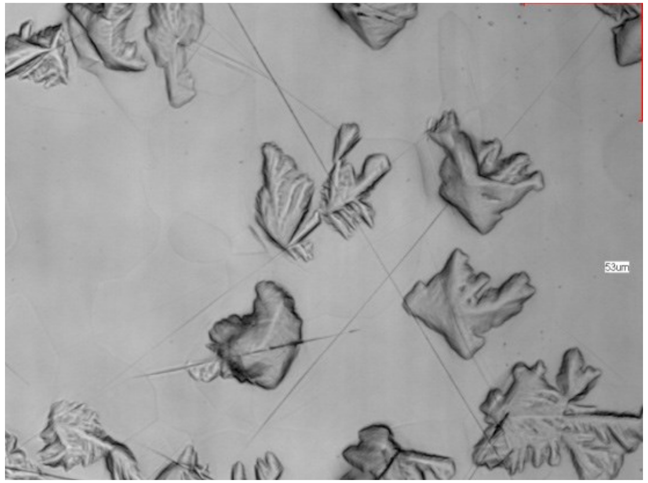


T = 160 K

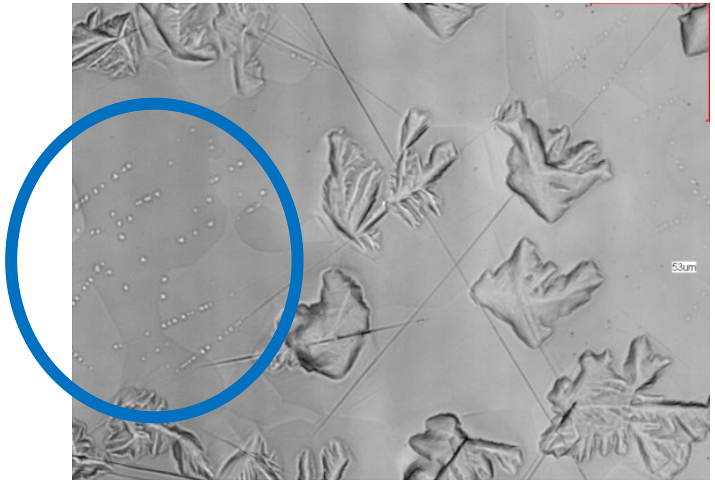
Second (smaller) phase of hydride forms



T = 6 K



T = 100 K



T = 140 K

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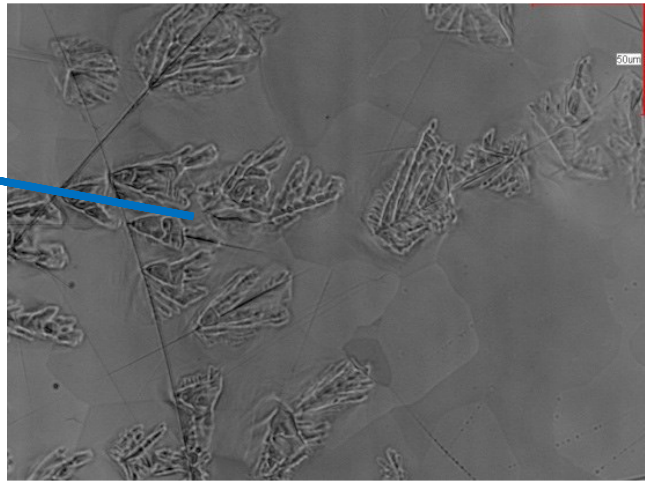
Microscopic Mechanism of Q-disease

cryostage in the laser confocal scanning microscope

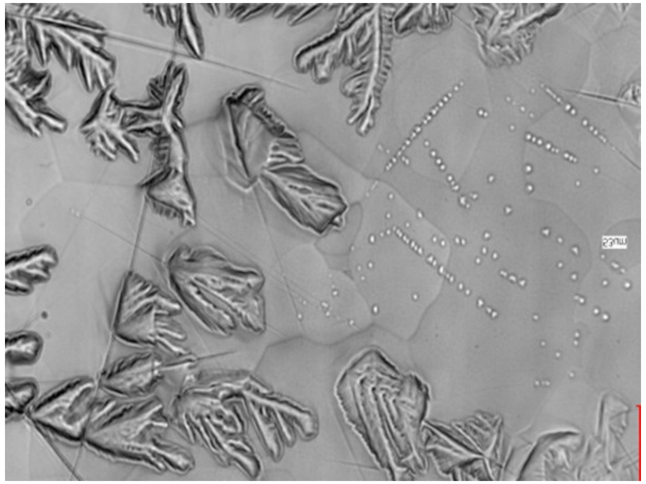
WARM-UP

Hydrides gone, dislocation skeleton (deformation) remains on the surface

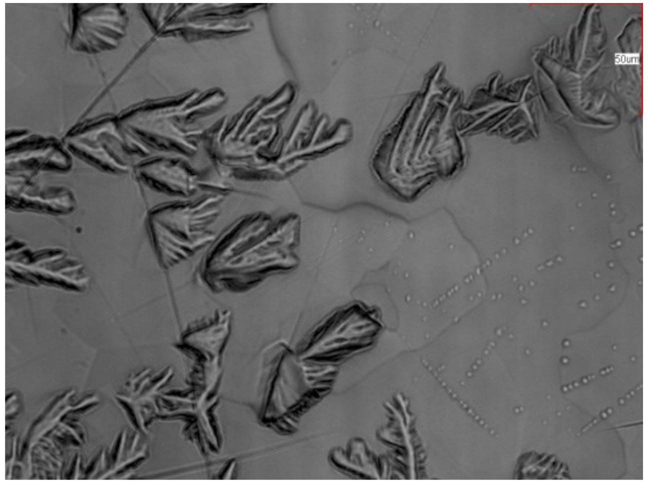
Large phase starts to dissolve



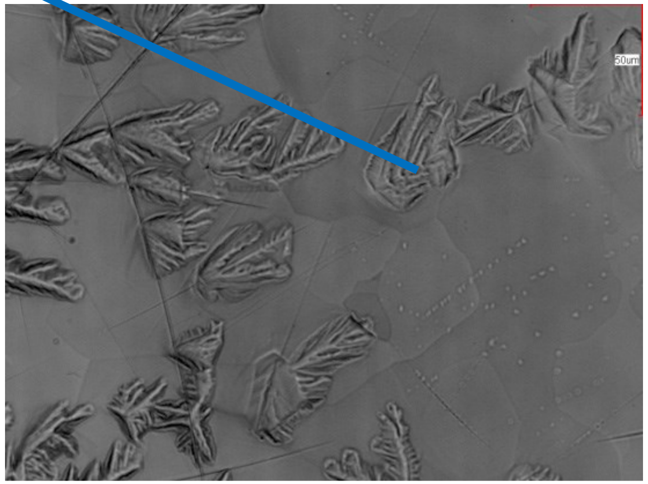
T = 260 K



T = 6 K



T = 180 K



T = 210 K

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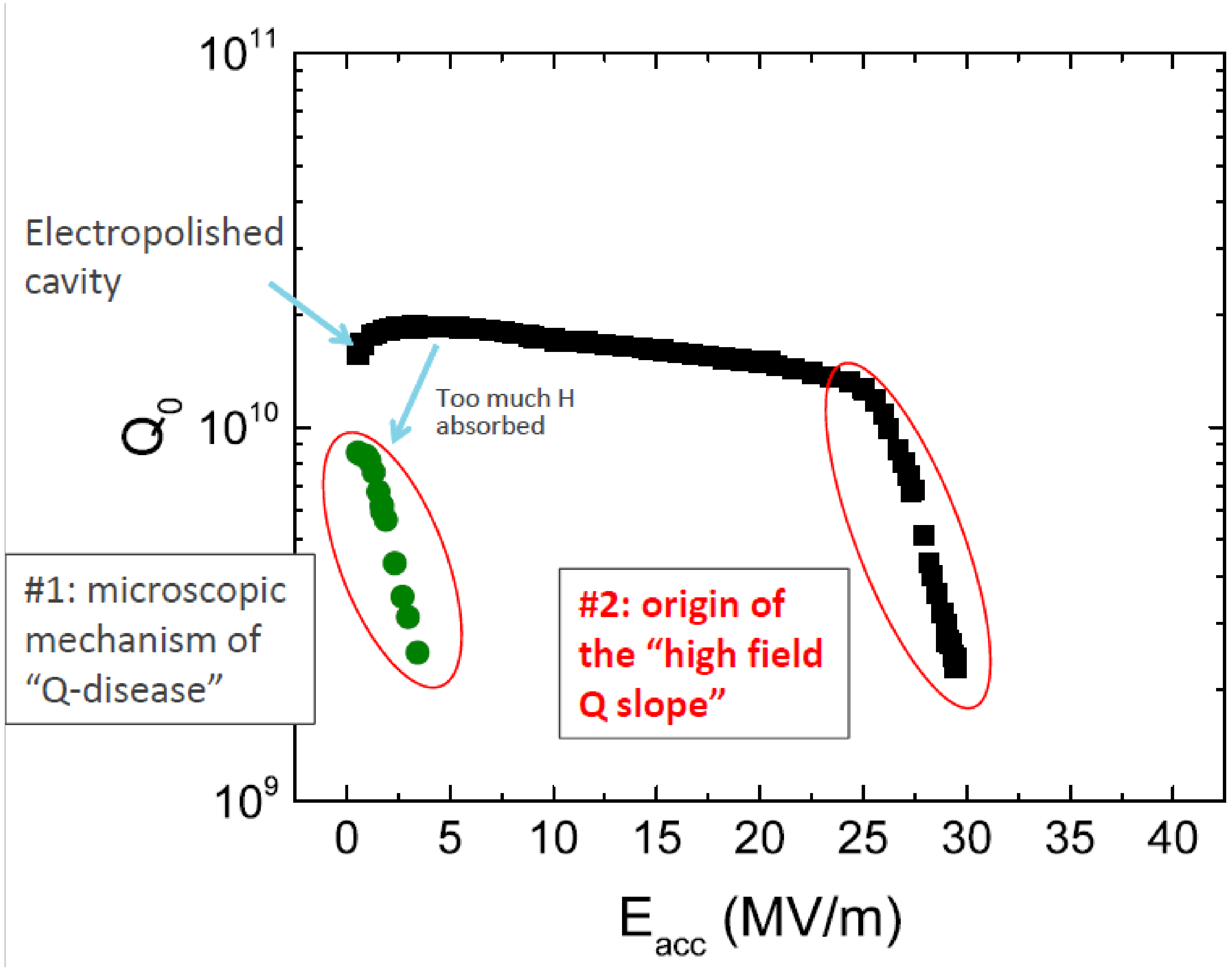
Possible explanation of the high field Q slope



Could be the same mechanism?

But nanohydrides instead of micron-sized one?

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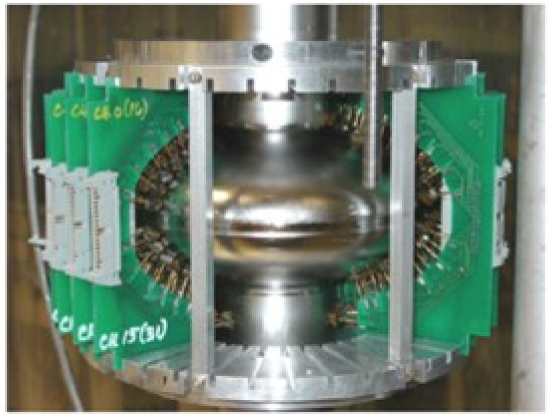
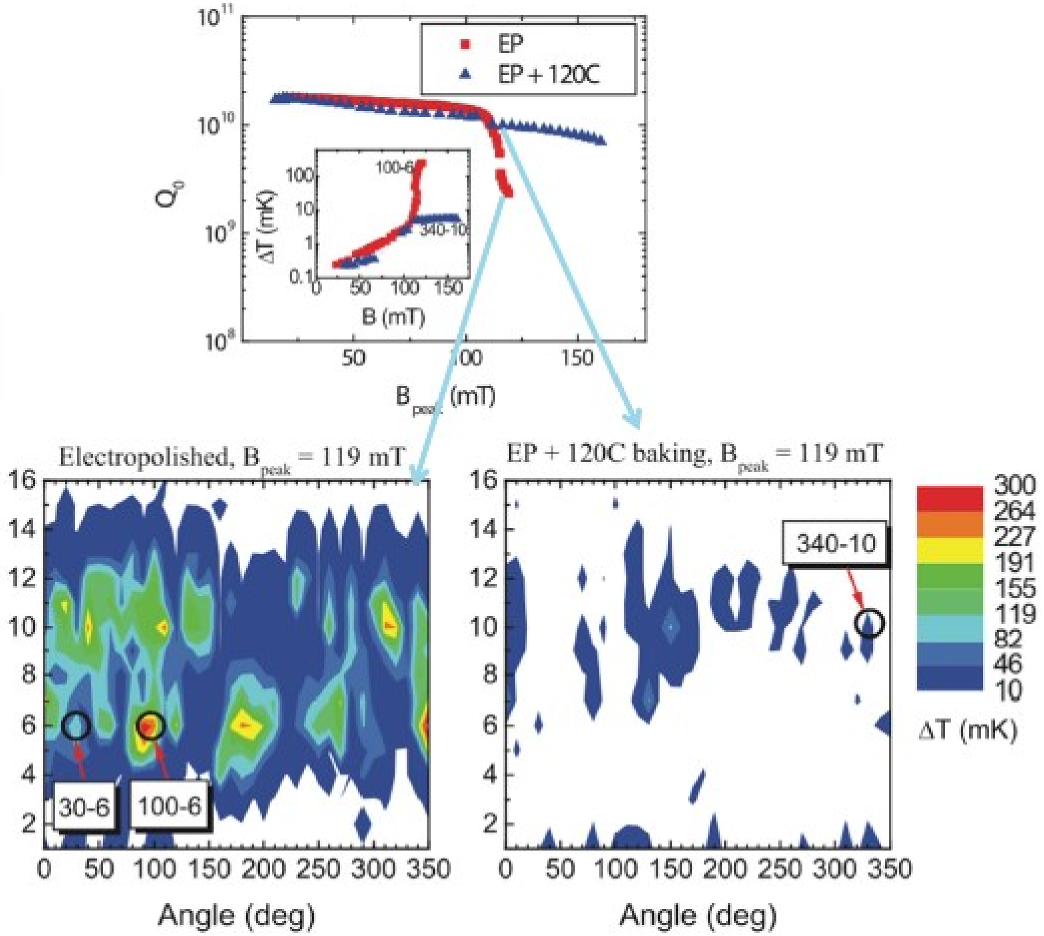
First: find the piece of cavity to characterize



Array of 576 thermometers attached to the outside cavity walls allows mapping wall dissipation



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Cut the cavity

Extract samples from cavity walls locations identified by temperature mapping -> direct correlation of RF losses with material structure

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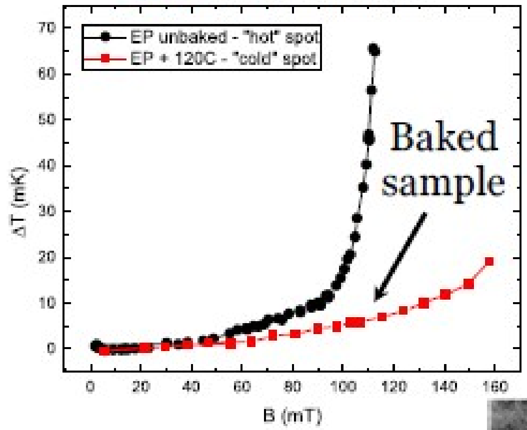
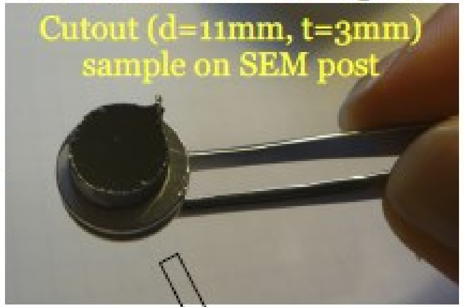
TEM characterization

Cold: 120C in situ bake for 48hours
Hot: no such bake

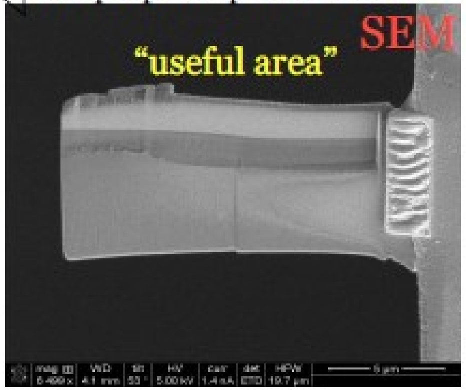


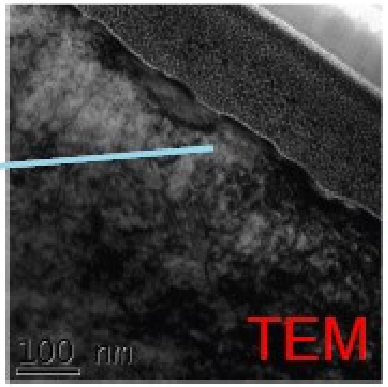
Identify samples based on T-map

Heating: comparison of "cold" and "hot" spots



FIB prep sample for TEM





Look at this area with subnanometer resolution in TEM at room AND T<100K temperatures

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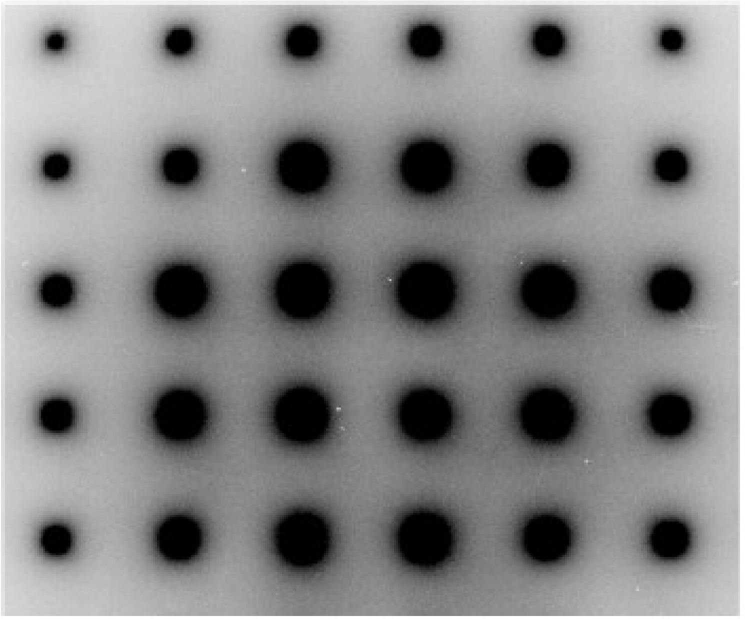
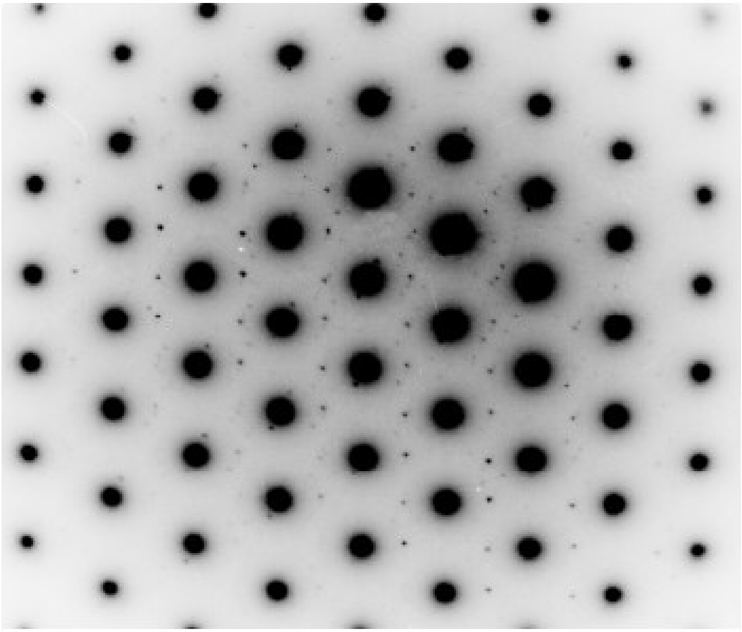
Confirmation of the existence of nanohydrides

TEM diffraction on cavity cutouts confirms the existence of nanohydrides



Room T: BCC Nb patterns, NO additional phases



94K: stoichiometric Nb hydride phases!

Y. Trenikhina, A. Romanenko, J. Zasadzinski, Proceedings of SRF'2013, TUP04

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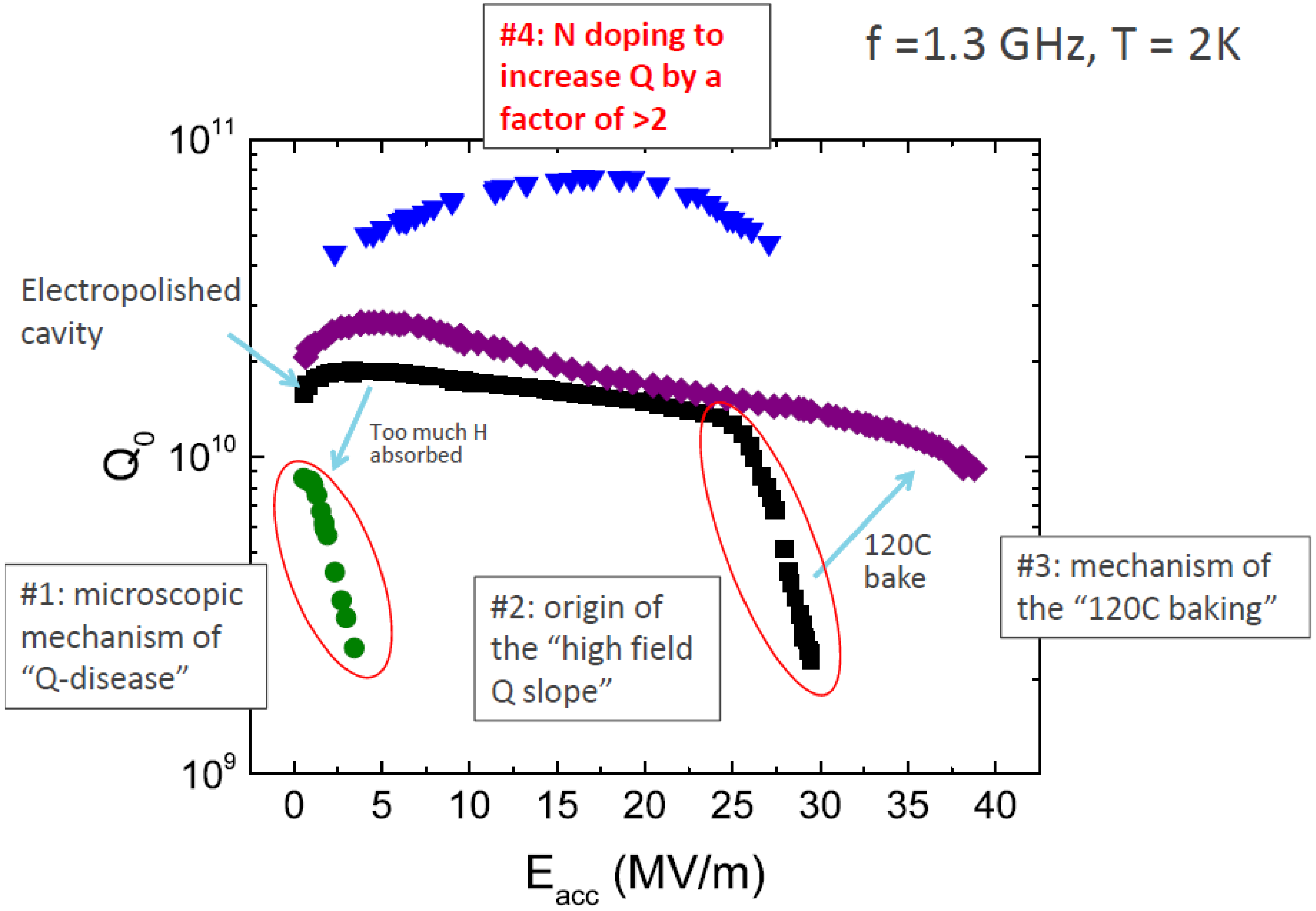
N - doping, a breakthrough for Q



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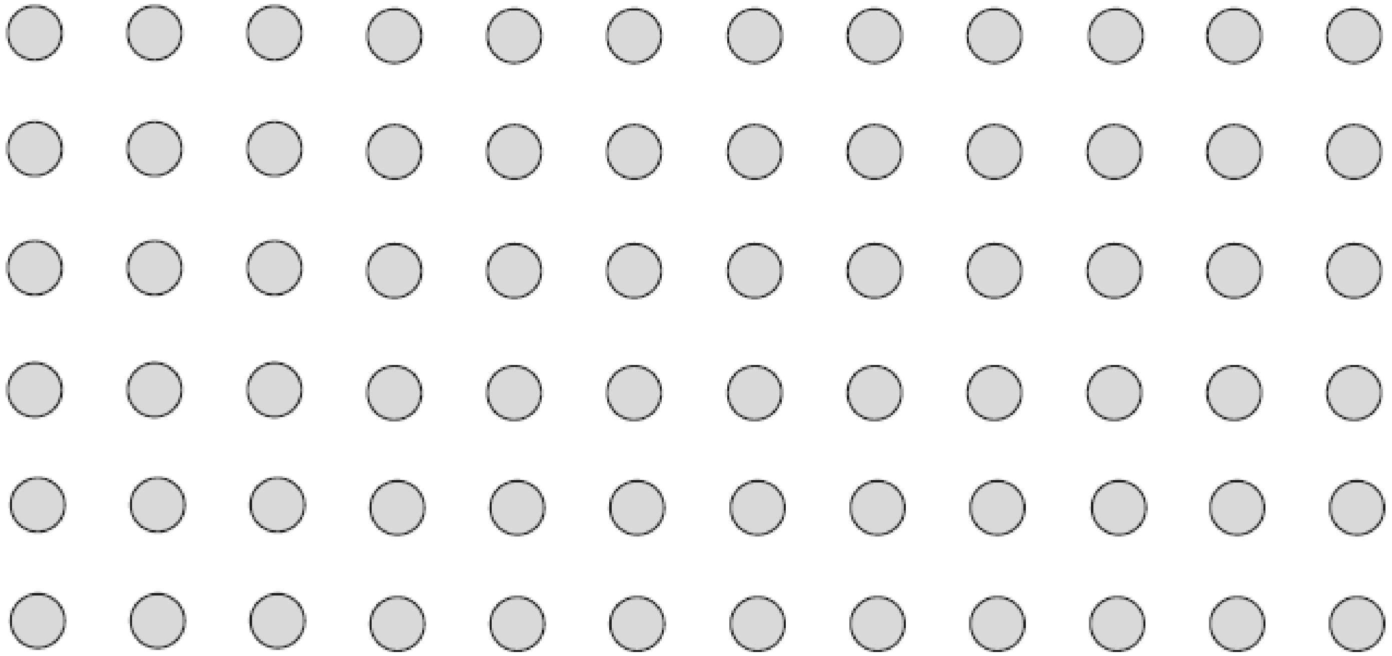
N-doping how it works?

800C UHV, 3 hours → 800C N_2 p = 25 mTorr 2 minutes → 800C UHV, 6 minutes → UHV cooling → 5 um EP



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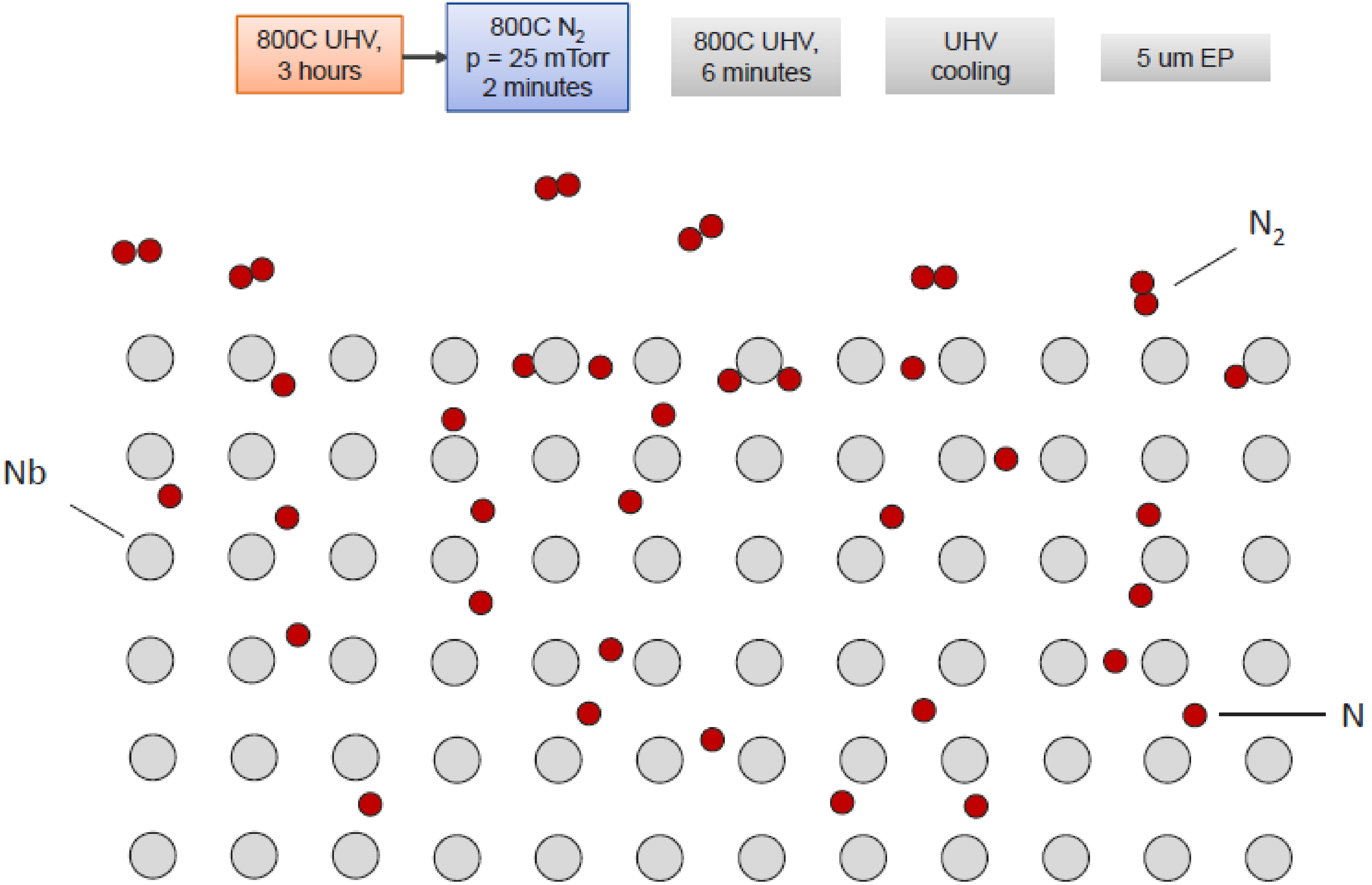
N-doping how it works?



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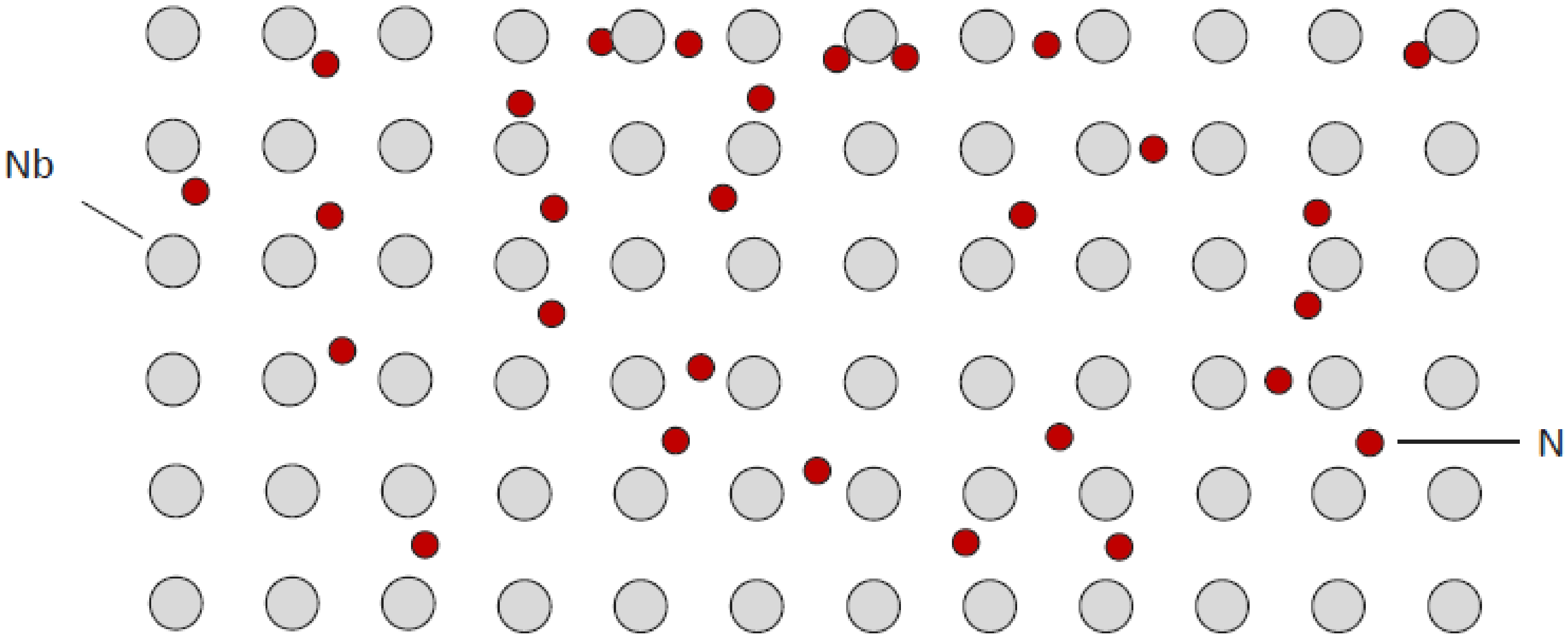

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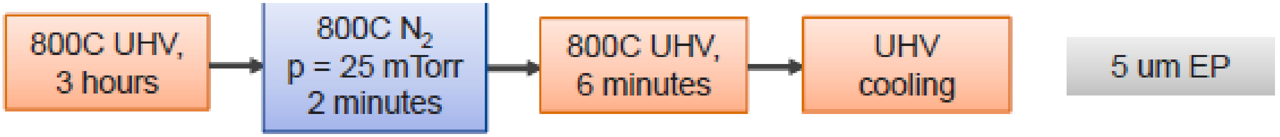

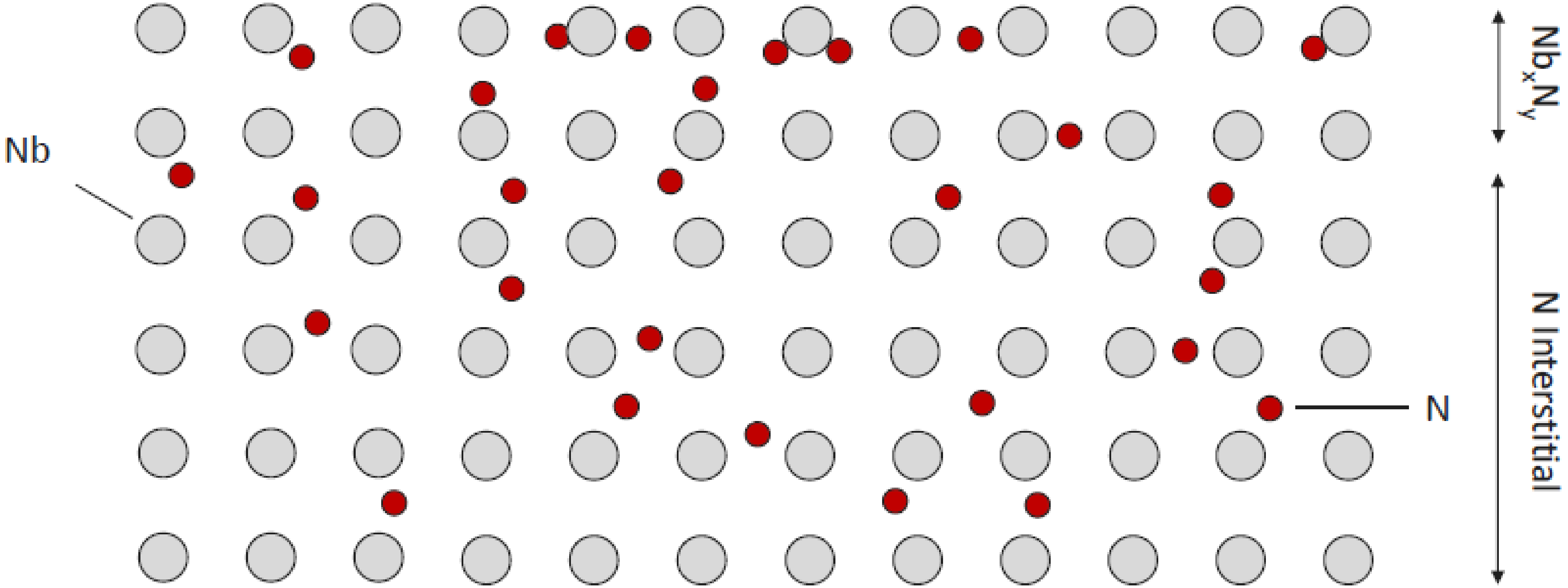

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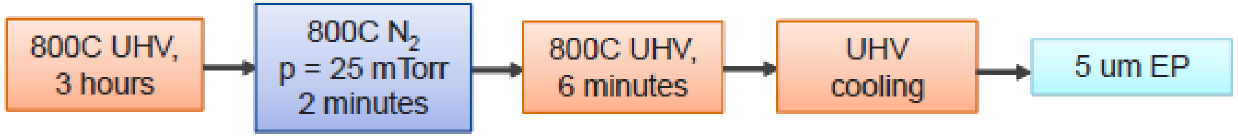

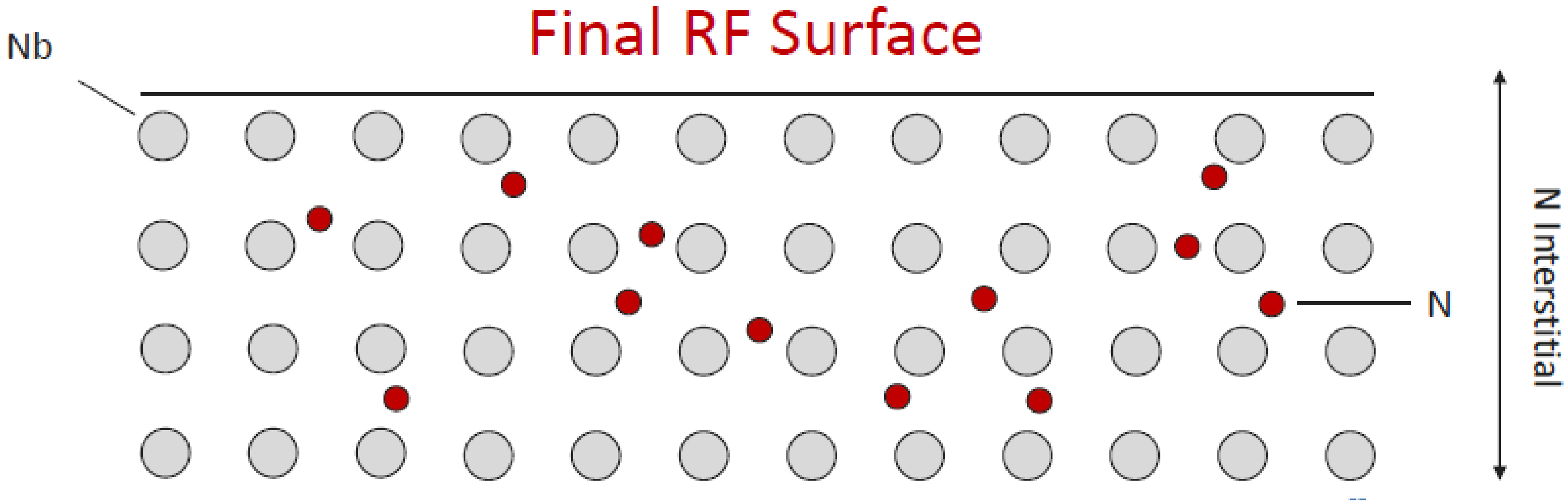

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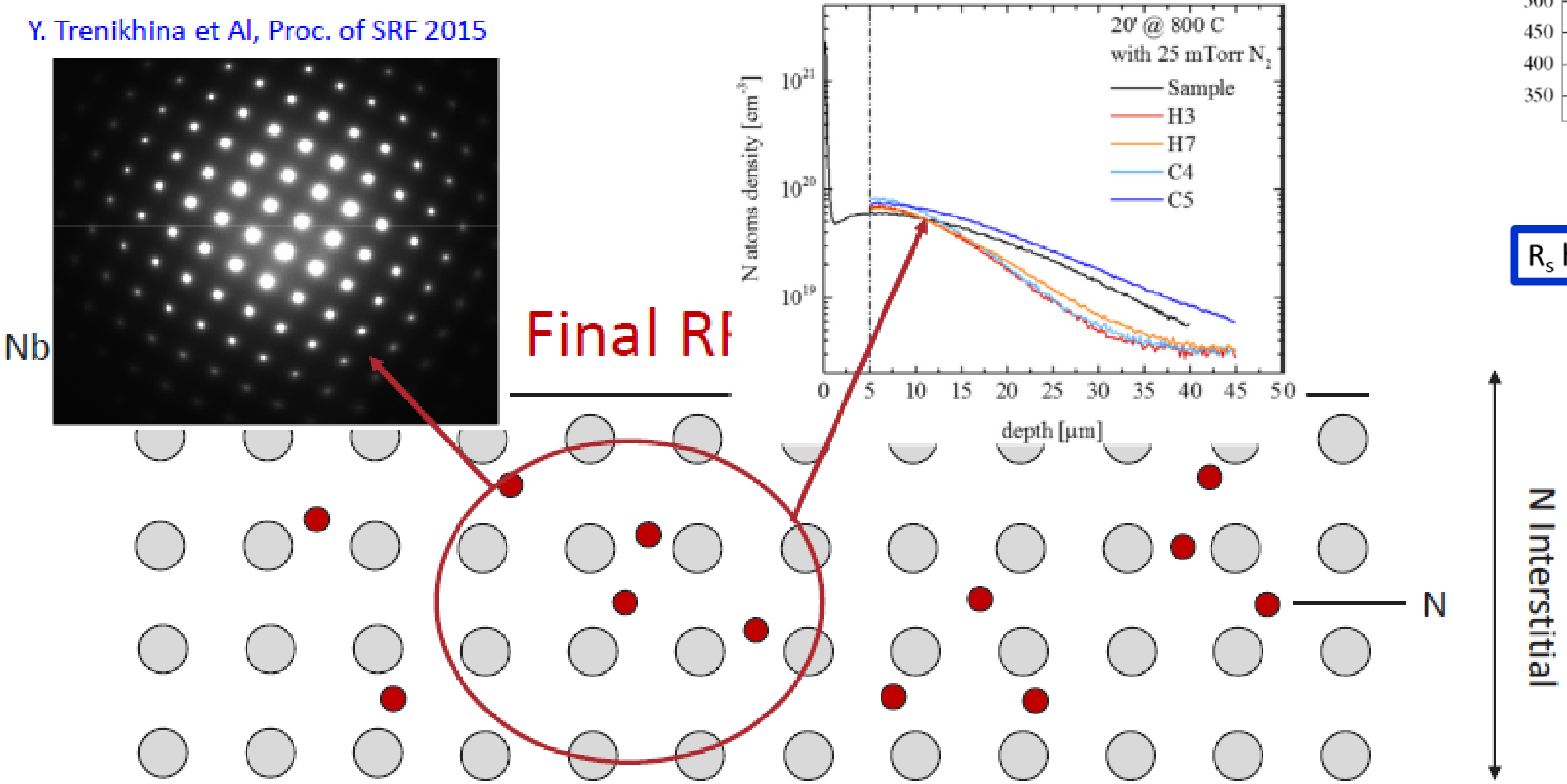
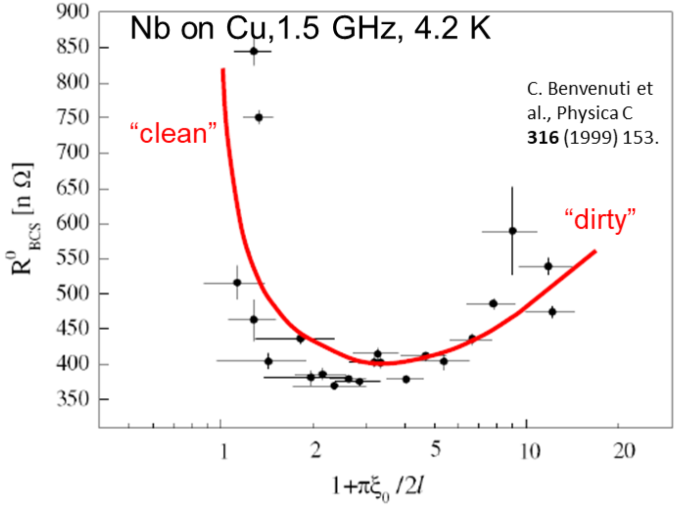
Y. Trenikhina et Al, Proc. of SRF 2015





R_s has a minimum for $\ell = \pi\xi_0/4$

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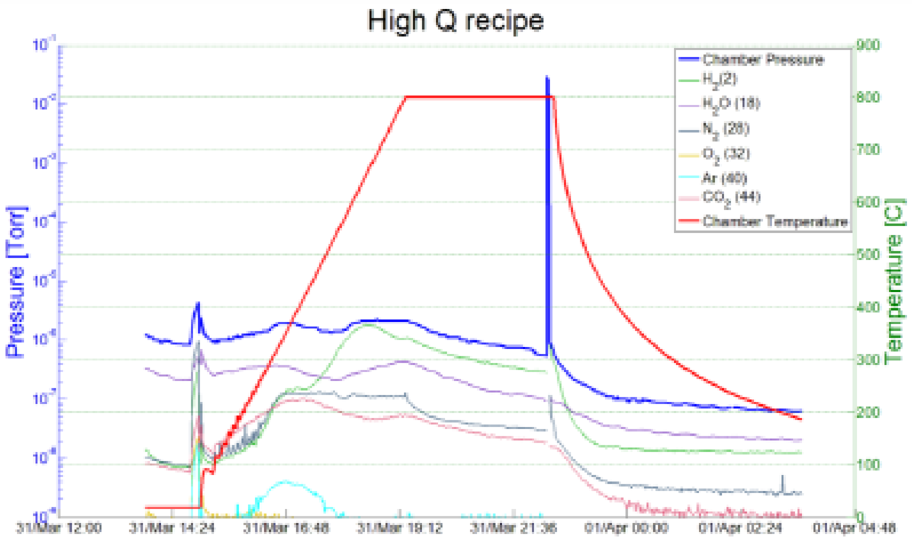
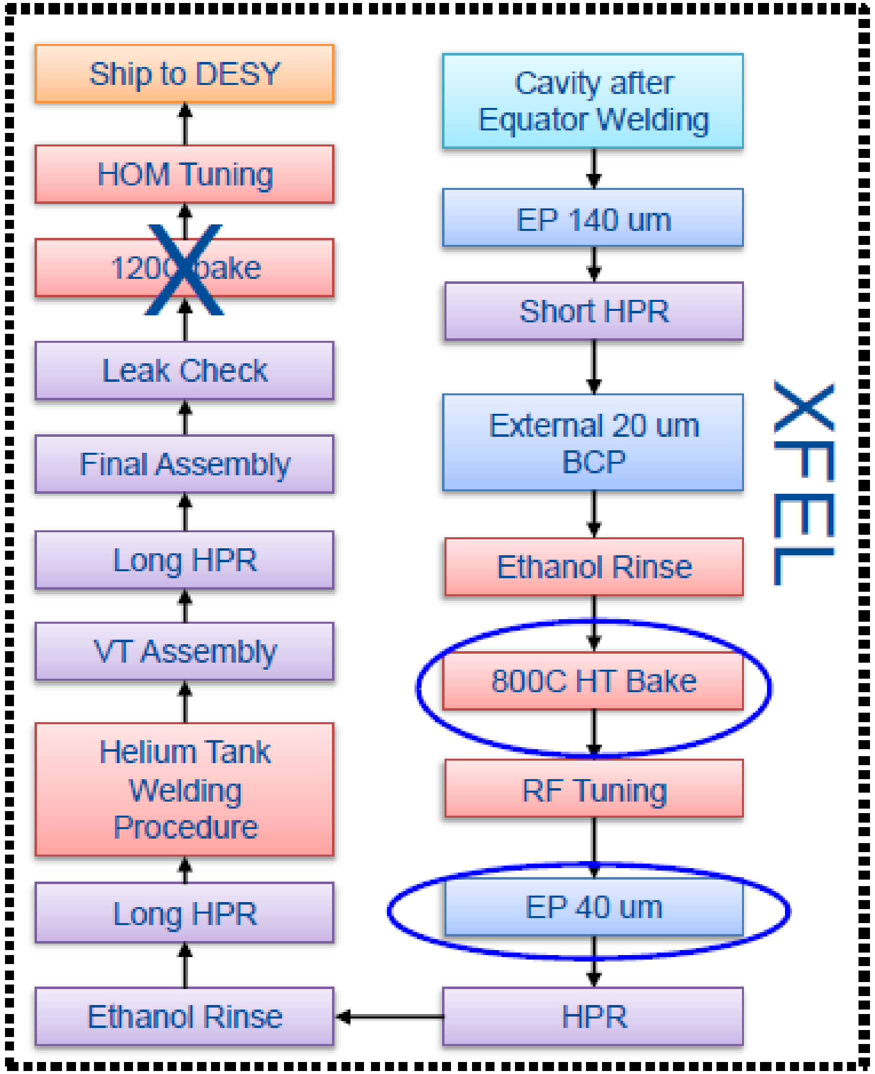
New protocol immediately implement in a real accelerator

Example from a doping process developed for LCLS-2:

- Bulk EP
- 800 C anneal for 3 hours in vacuum
- 2 minutes @ 800C nitrogen diffusion
- 800 C for 6 minutes in vacuum
- Vacuum cooling
- 5 microns EP





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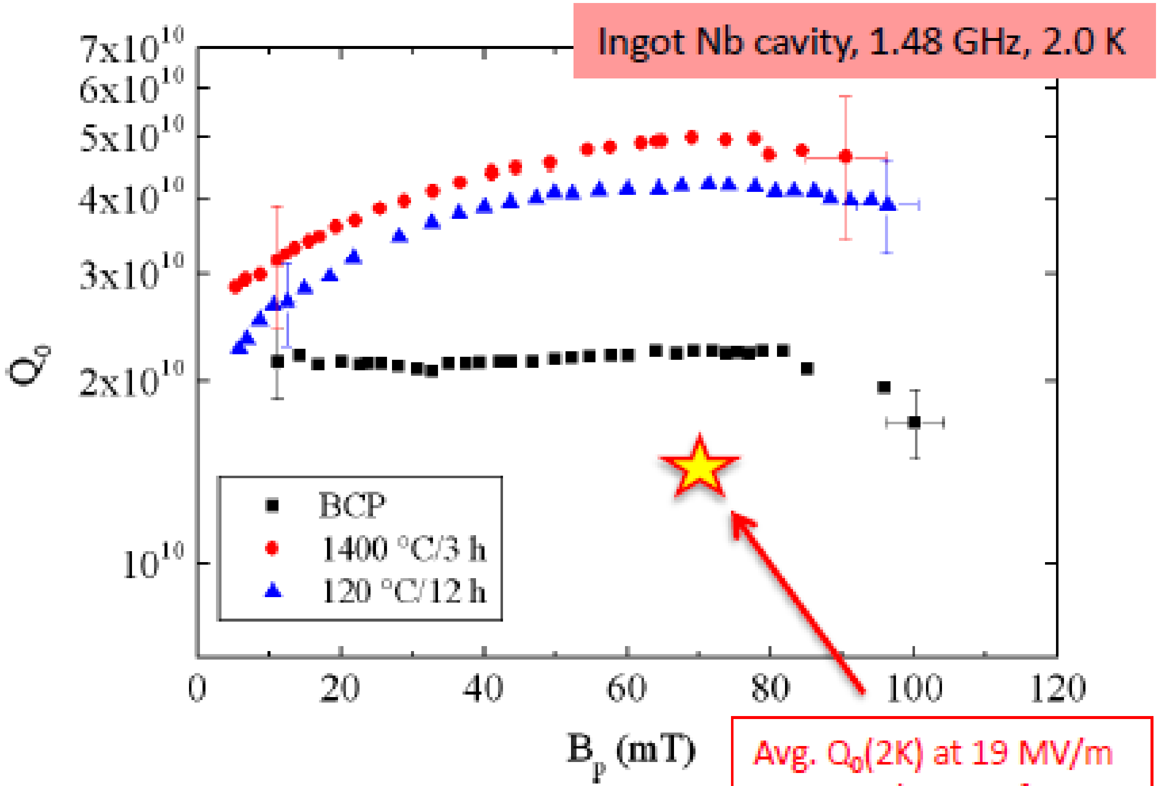
Doping with Ti works as well

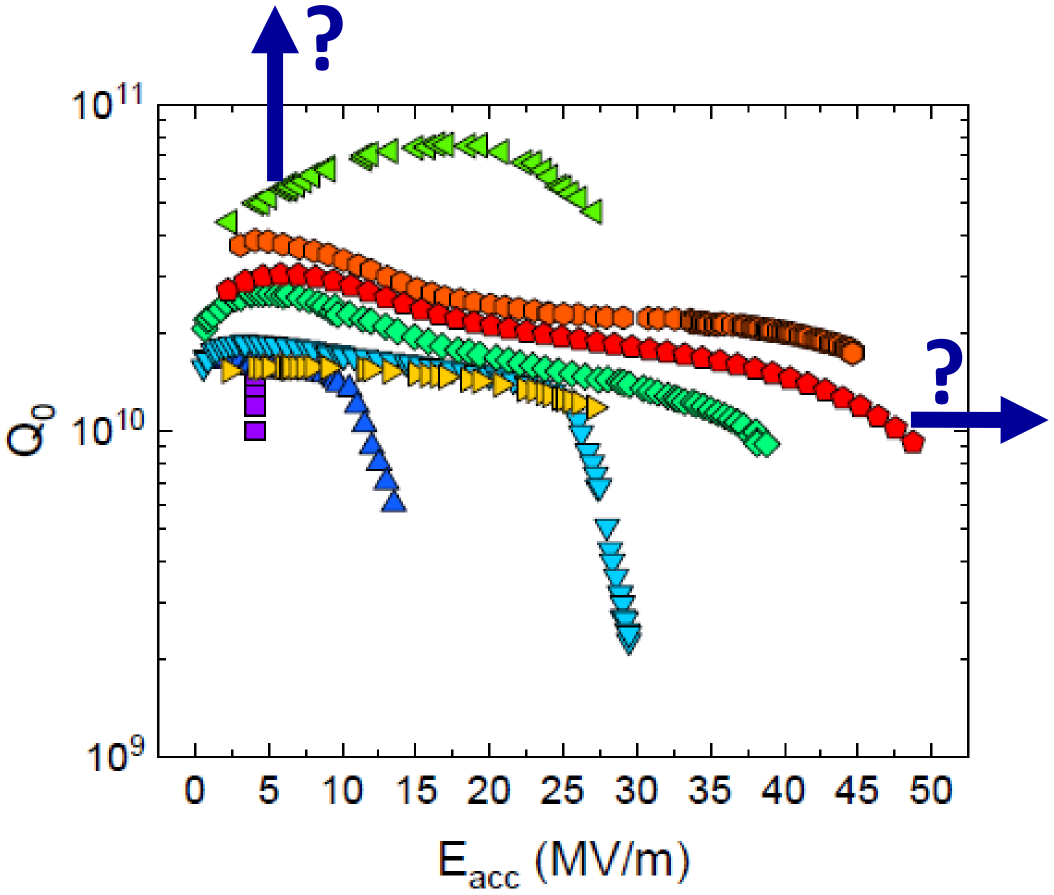


P. Dhakal, Rev. Sci. Inst. 83, 065105 (2012)
P. Dhakal et al., *Phys. Rev. ST Accel. Beams* 16, 042001 (2013)
P. Dhakal et al., IPAC'14, p. 2651

Ciovati

Why looking beyond bulk Nb?

Nb has the highest critical temperature T_c (=9.25k) and the highest lower critical magnetic field H_{c1}(≈180 mT) of any elemental SC



Breakdown fields close to the de-pairing limit of 50 MV/m for Nb have been achieved

Best Nb cavities approaching their intrinsic limit at $H_{max} = H_C$

For further improvement, innovation needed

Possibilities to use higher performance superconductors other than bulk Nb?

A-M Valente, SRF2017 Tutorials

Recommanded Literature

- R. Padamsee, J. Knobloch and T. Hays – « RF Superconductivity for Accelerators », Wiley-VCH, 2008
- J. P. Turneaure, J. Halbritter, and H. A. Schwettman. « The surface impedance of superconductors and normal conductors: The Mattis-Bardeen theory. » Journal of Superconductivity 4.5 (1991): 341-355
- A. Gurevich « Theory of RF superconductivity for resonant cavities. » Superconductor Science and Technology, 30(3), 034004 (2017).
- SRF Tutorials (https://jacow.org/Main/Proceedings?sel=SRF and websites of the SRF conferences)

This slides are mainly based on: D. Sertore, SRF Cavity Fabrication, EASIschool 3 Genoa, 2020