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Surface Processing for High Performance Superconducting Cavities

SCRF Series: Talk II.

The Beams and Applications Seminar Series

June 8, 2007

SRF Group: Joel Fuerst, Scott Gerbick, Mike Kelly, Zack Conway, Ken Shepard

Speaker: Mike Kelly

U.S. Department of Energy

UChicago Argonne LLC

Office of Science
U.S. DEPARTMENT OF ENERGY

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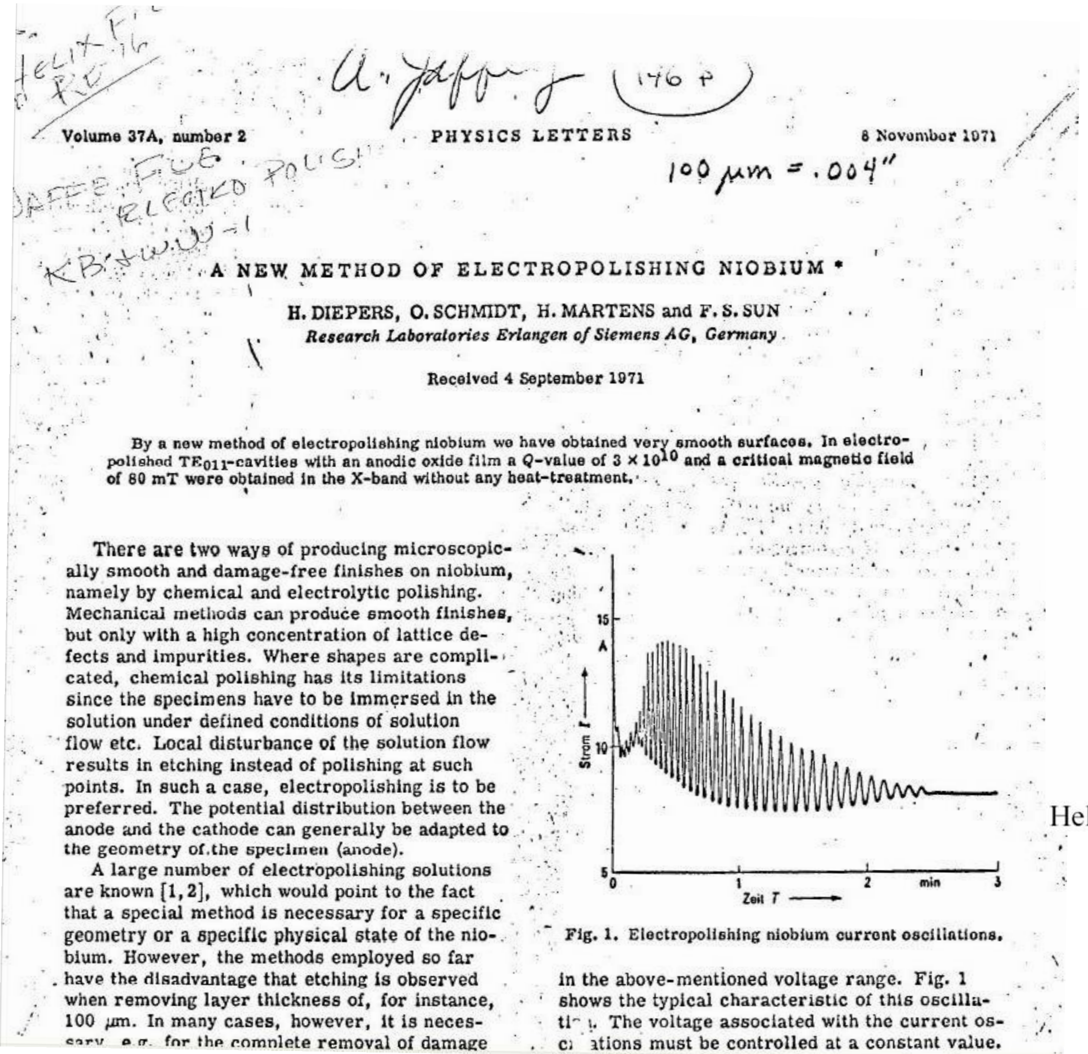
Four Decades of Superconducting RF Cavities



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100 μm = .004"

A NEW METHOD OF ELECTROPOLISHING NIOBIUM *

H. DIEPERS, O. SCHMIDT, H. MARTENS and F. S. SUN
Research Laboratories Erlangen of Siemens AG, Germany

Received 4 September 1971

By a new method of electropolishing niobium we have obtained very smooth surfaces. In electropolished TE_{011}-cavities with an anodic oxide film a Q-value of 3×10^{10} and a critical magnetic field of 80 mT were obtained in the X-band without any heat-treatment.

There are two ways of producing microscopically smooth and damage-free finishes on niobium, namely by chemical and electrolytic polishing. Mechanical methods can produce smooth finishes, but only with a high concentration of lattice defects and impurities. Where shapes are complicated, chemical polishing has its limitations since the specimens have to be immersed in the solution under defined conditions of solution flow etc. Local disturbance of the solution flow results in etching instead of polishing at such points. In such a case, electropolishing is to be preferred. The potential distribution between the anode and the cathode can generally be adapted to the geometry of the specimen (anode).

A large number of electropolishing solutions are known [1, 2], which would point to the fact that a special method is necessary for a specific geometry or a specific physical state of the niobium. However, the methods employed so far have the disadvantage that etching is observed when removing layer thickness of, for instance, 100 μm. In many cases, however, it is necessary, e.g. for the complete removal of damage

Fig. 1. Electropolishing niobium current oscillations.

in the above-mentioned voltage range. Fig. 1 shows the typical characteristic of this oscillation. The voltage associated with the current oscillations must be controlled at a constant value.

(EP collaboration between ANL and Karlsruhe)

Paul Markovich – ANL chemistry

Helical Nb resonator developed at ANL for a heavy-ion linac.

Four Decades of Superconducting RF Cavities

97 MHz β=0.1 ANL

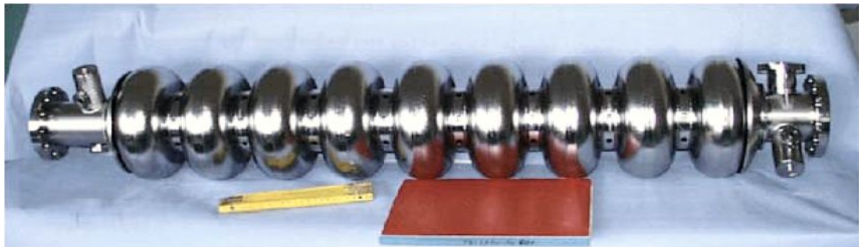
1st SC spoke 1991 (funded through SDI)

850 MHz β=0.28 ANL

High-Beta~1.0

805 MHz β=0.61 JLAB/SNS

345 MHz β=0.63 ANL



1.3 GHz β=1 DESY

- Recent convergence of interest in SCRF community; similar techniques now required for all cavities
- Bulk niobium is the material of choice for today’s high-performance SC cavities

Outline

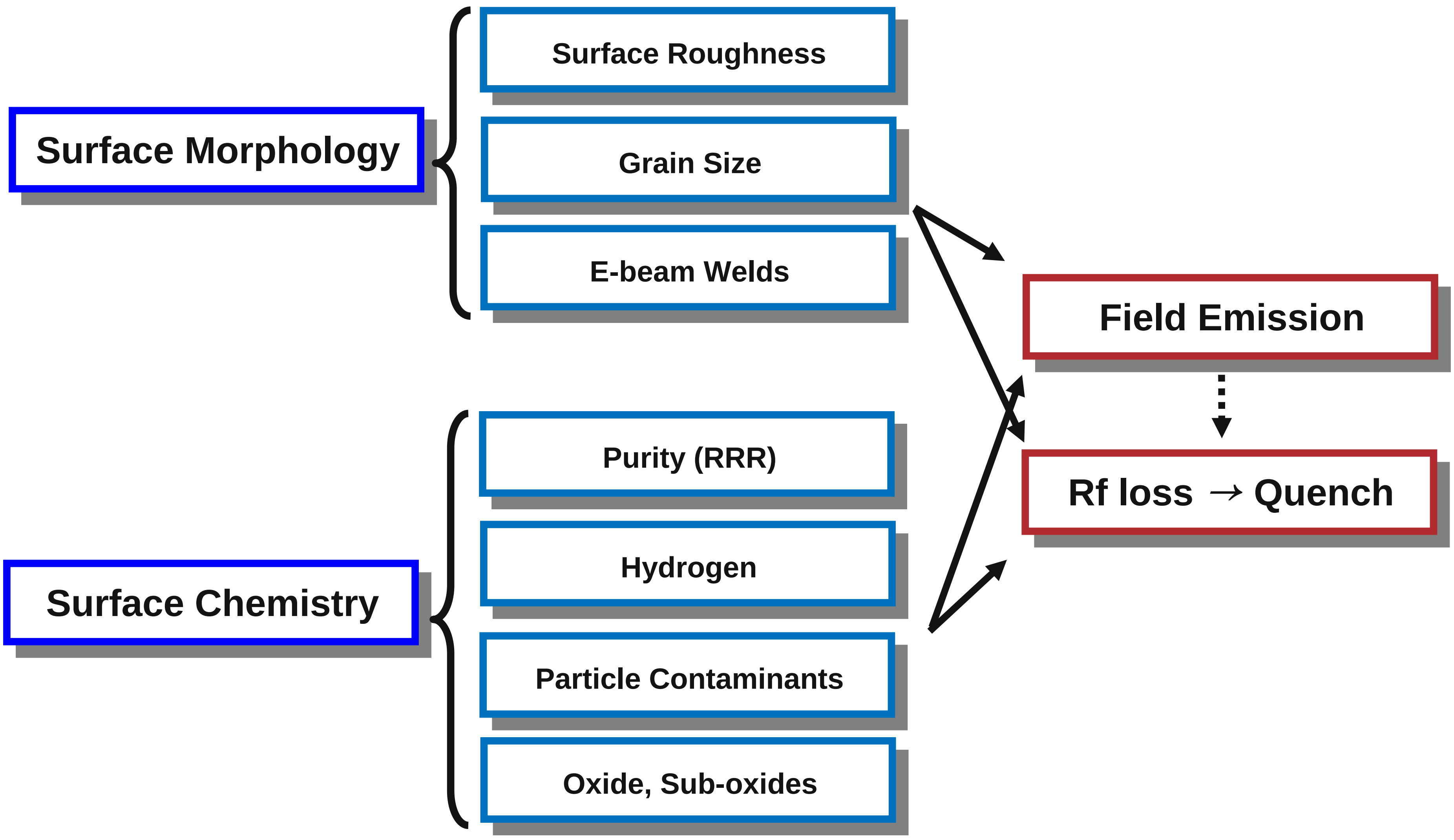
I. RF surface

e.g. roughness, oxygen, hydrogen

II. Practical Considerations (The current state-of-the-art)

e.g. electropolishing, high-pressure rinsing, facilities

I. RF Surface: Properties in Bulk Niobium Cavities



I. RF Surface: Surface Resistance, RF losses, Quality Factor

$$R_S = R_{BCS}(T,\omega) + R_{RES}\left[n\Omega\right]$$

Surface resistance modeled as T, ω-dependent term plus everything else

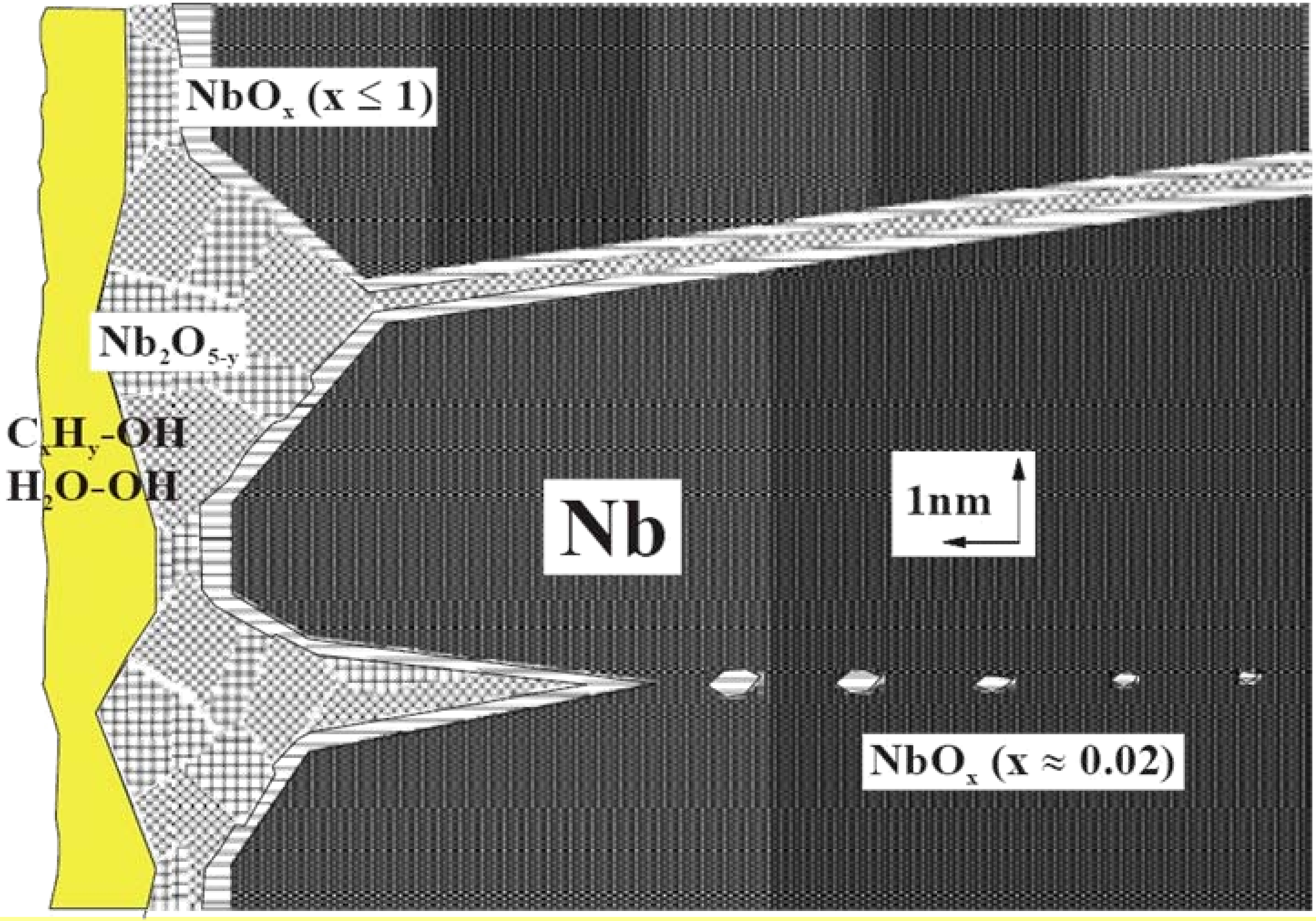
$$P_{IN} = \frac{1}{2}\oint R_S \left|H\right|^2 dA\left[Watts\right]$$

Power dissipated in the cavity walls is product of local R_s and the magnetic field squared over the cavity surface

$$Q_{Int} = \frac{U}{\Delta U} = \frac{U_o E_{ACC}^2}{P_{IN}} 2\pi f$$

Quality factor as for classical damped oscillator; stored energy divided by fractional energy loss per cycle

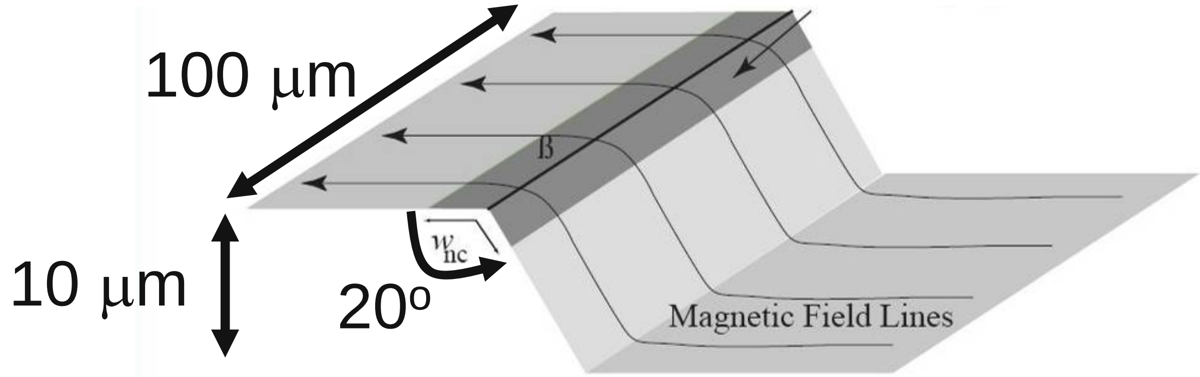
I. RF Surface: (Simplified) Niobium Surface

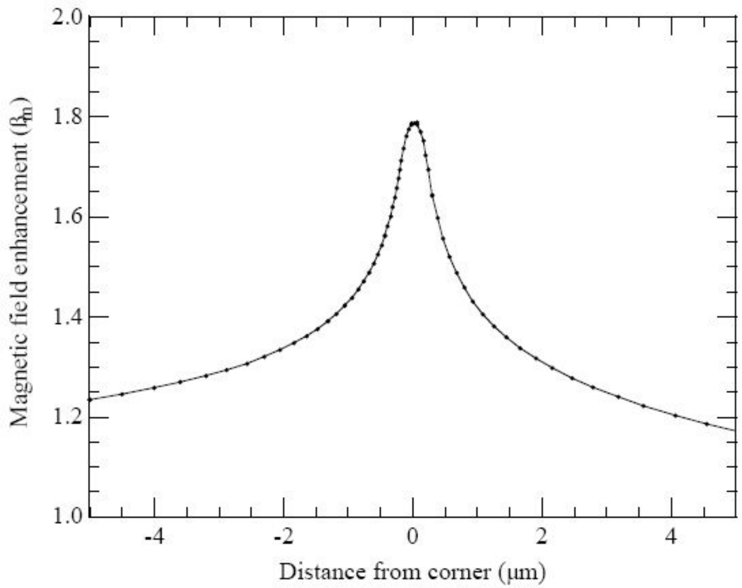


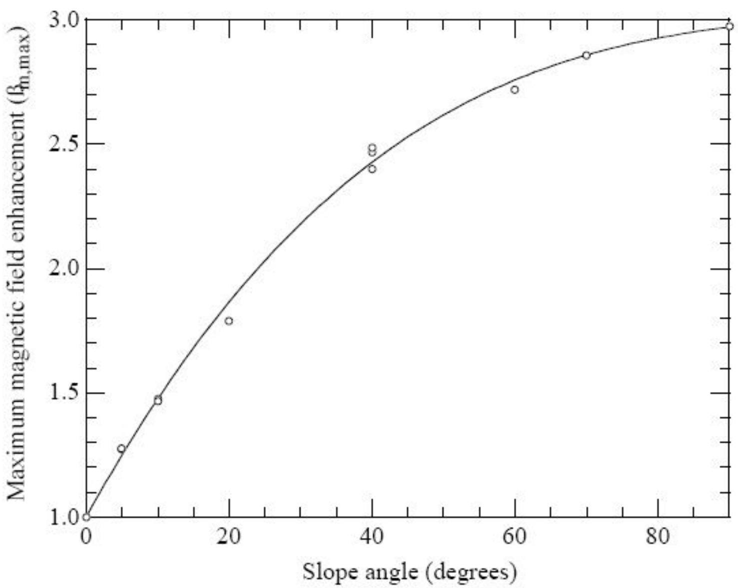
- Water, hydrocarbons adsorbed to the surface
- Several nm of Nb_2O_5 reforms rapidly even for low partial pressures of O_2
- Metallic NbO_x clusters

Reference[1]

I. RF Surface: Surface Roughness



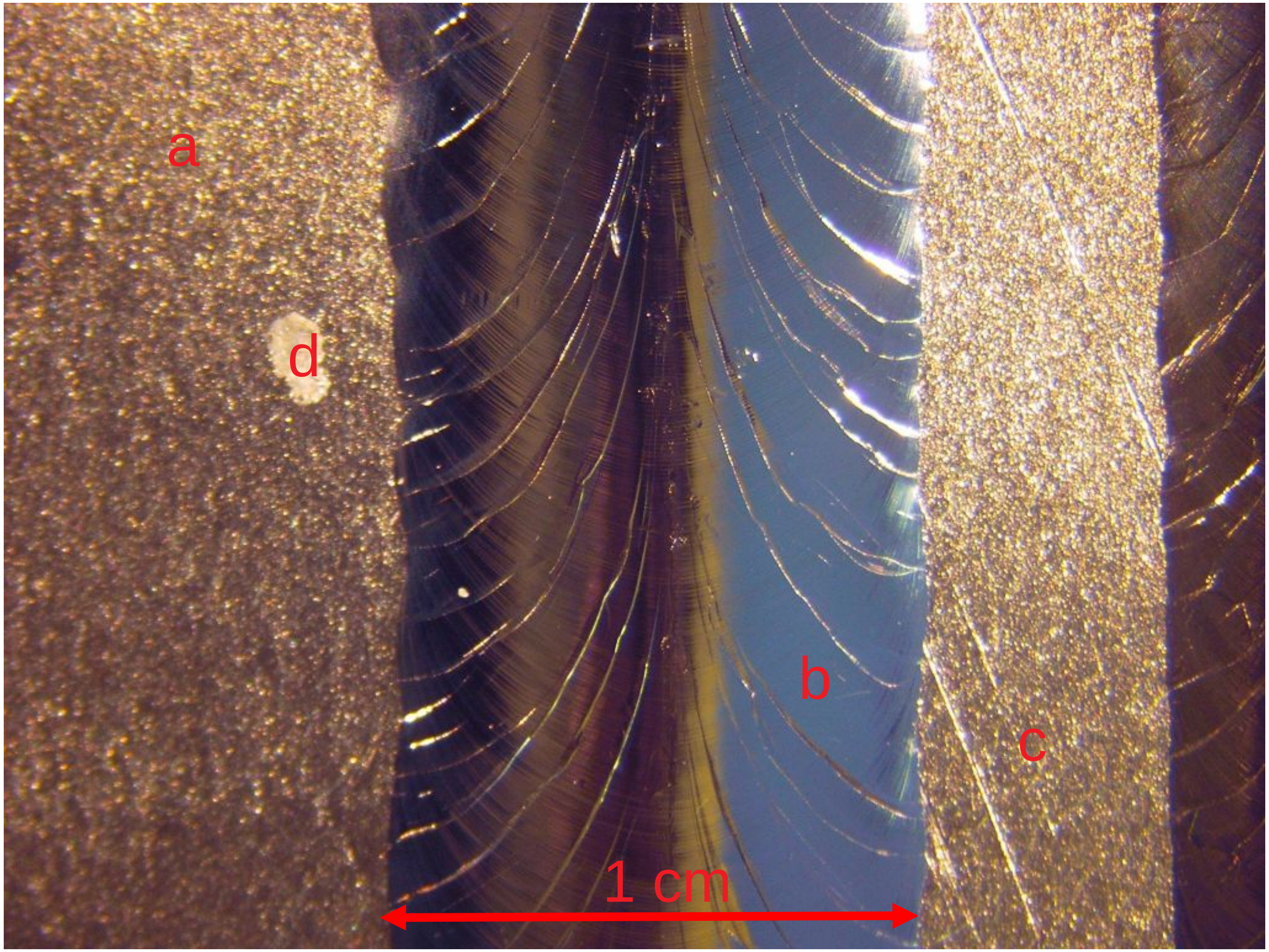




- Surface magnetic fields are enhanced when current runs along a (grain boundary) step
- Thermally stable regions of enhanced losses lead to a lowering of observed Q
- Low surface roughness likely to be key to achieving very high Q

Reference[2]

I. RF Surface: Grain Size and E-beam Welds

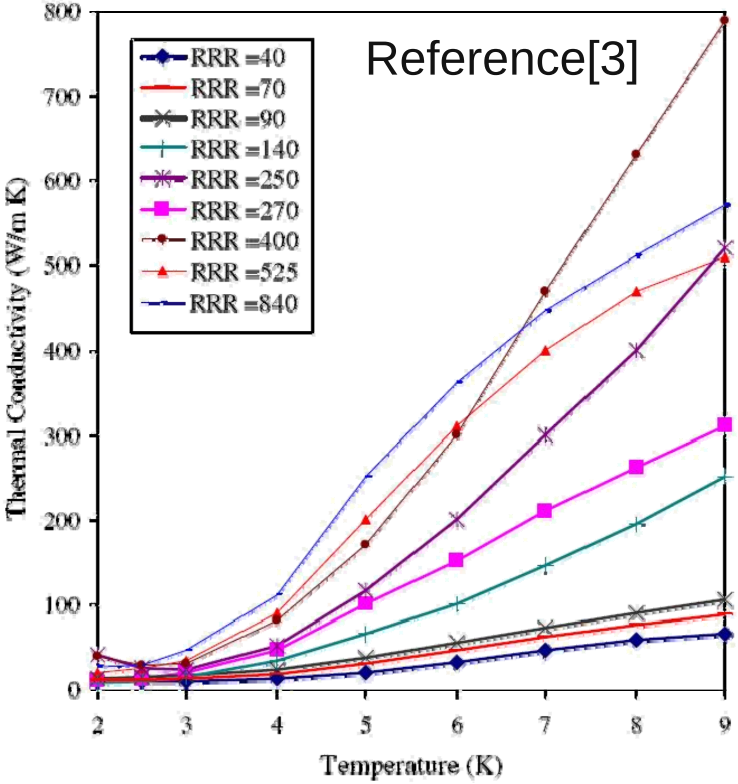


- Shown is a (typical) electron beam weld through 3 mm niobium sheet
- Surface is before final chemistry
- Visible features: Fine grains[a] (50 μm rms), large grain[b], scratches[c], defect[d]

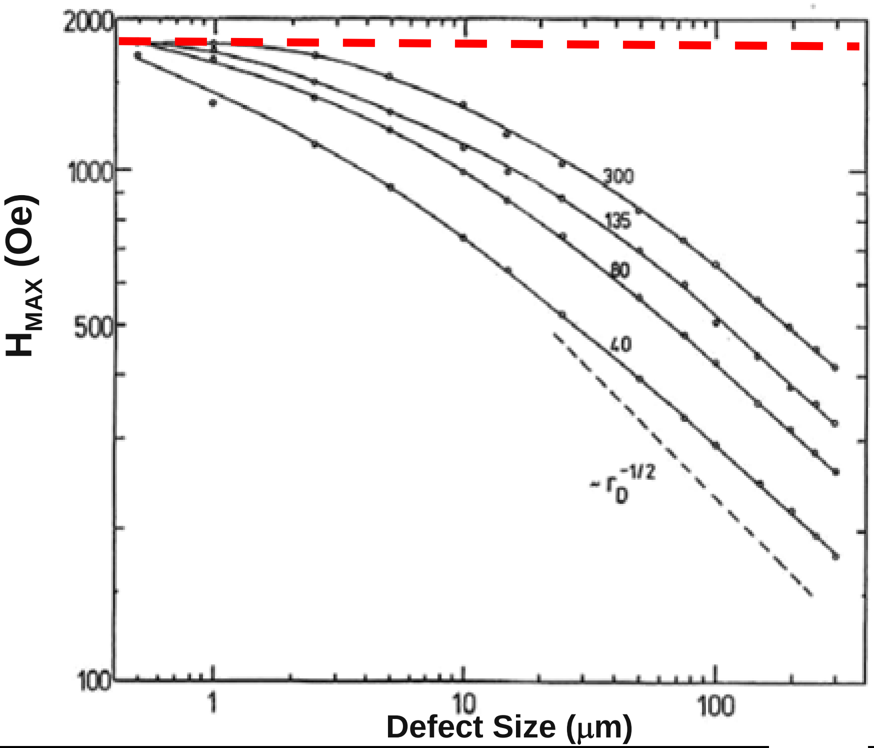
I. RF Surface: High Purity (RRR) as a method to increase the quench field

- Early SC cavities used RRR~40 (reactor grade)

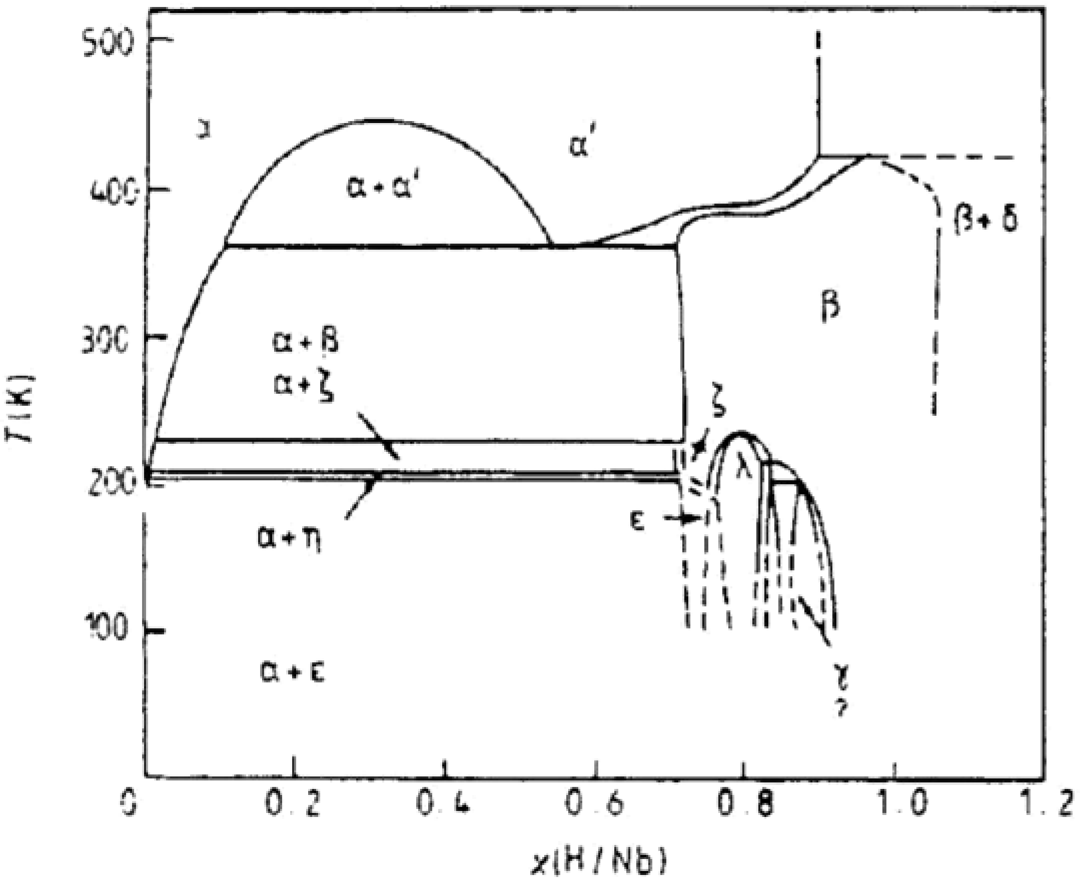
- Today SC cavities use RRR~200 or higher
- Carbon, Nitrogen, Oxygen – 10 ppm
- Titanium, Hafnium, Zirconium, Tungsten – 50 ppm
- Tantalum, Molybdenum – 500 ppm
- Hydrogen – 1 ppm



Quench Field vs Defect size



I. RF Surface: Hydrogen and “Q-disease”

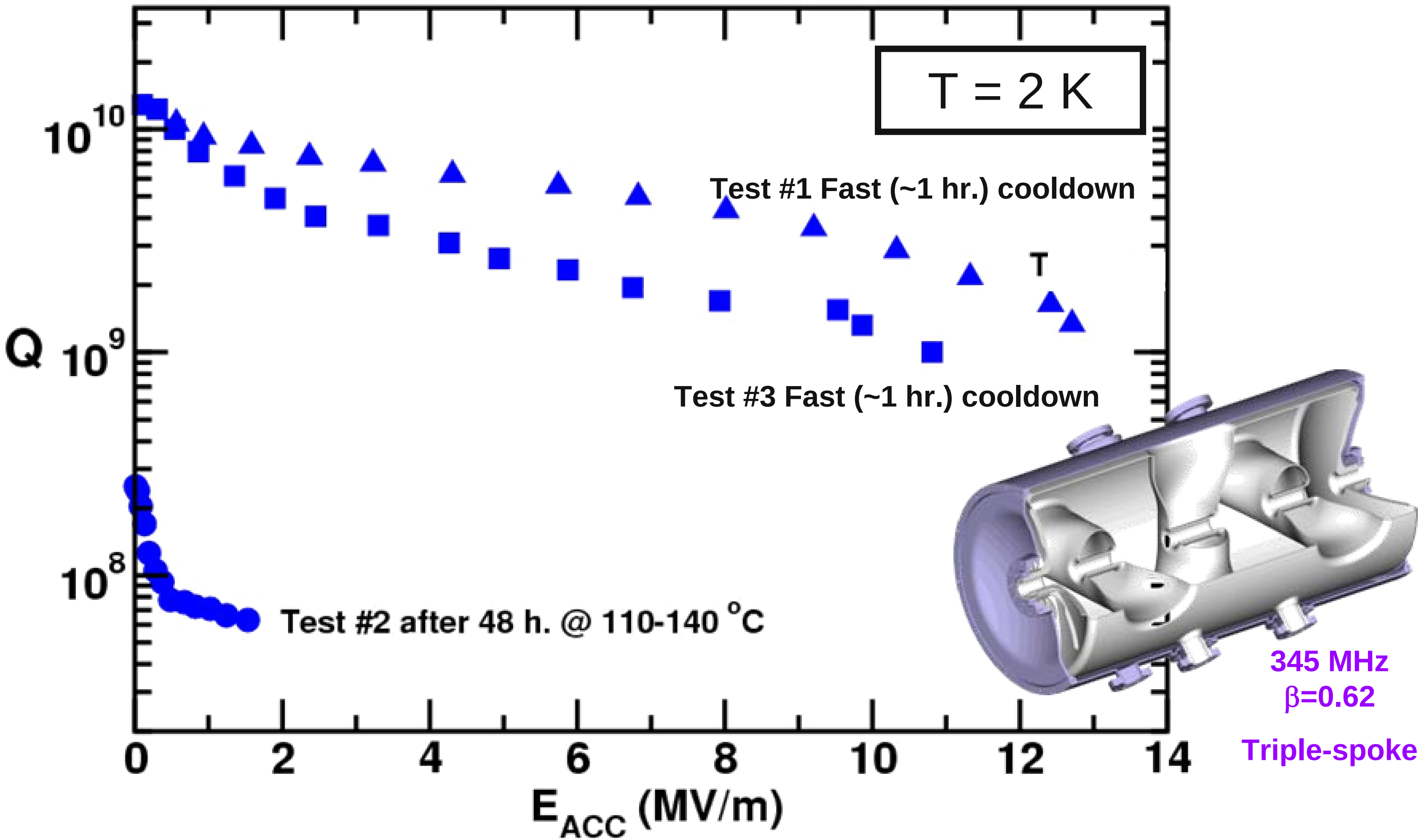


Phase diagram for the Nb-H system (Schober and Wenzl 1978)

- Niobium readily picks up hydrogen
- At room temperature hydrogen distributed throughout (Nb in α-phase fcc)
- At moderately low temperatures (50-150 K) hydrogen in niobium forms hydrides → Rf loss
- Below 50 K hydrogen is immobile

Reference[4]

I. RF Surface: Example of Hydrogen Q-disease



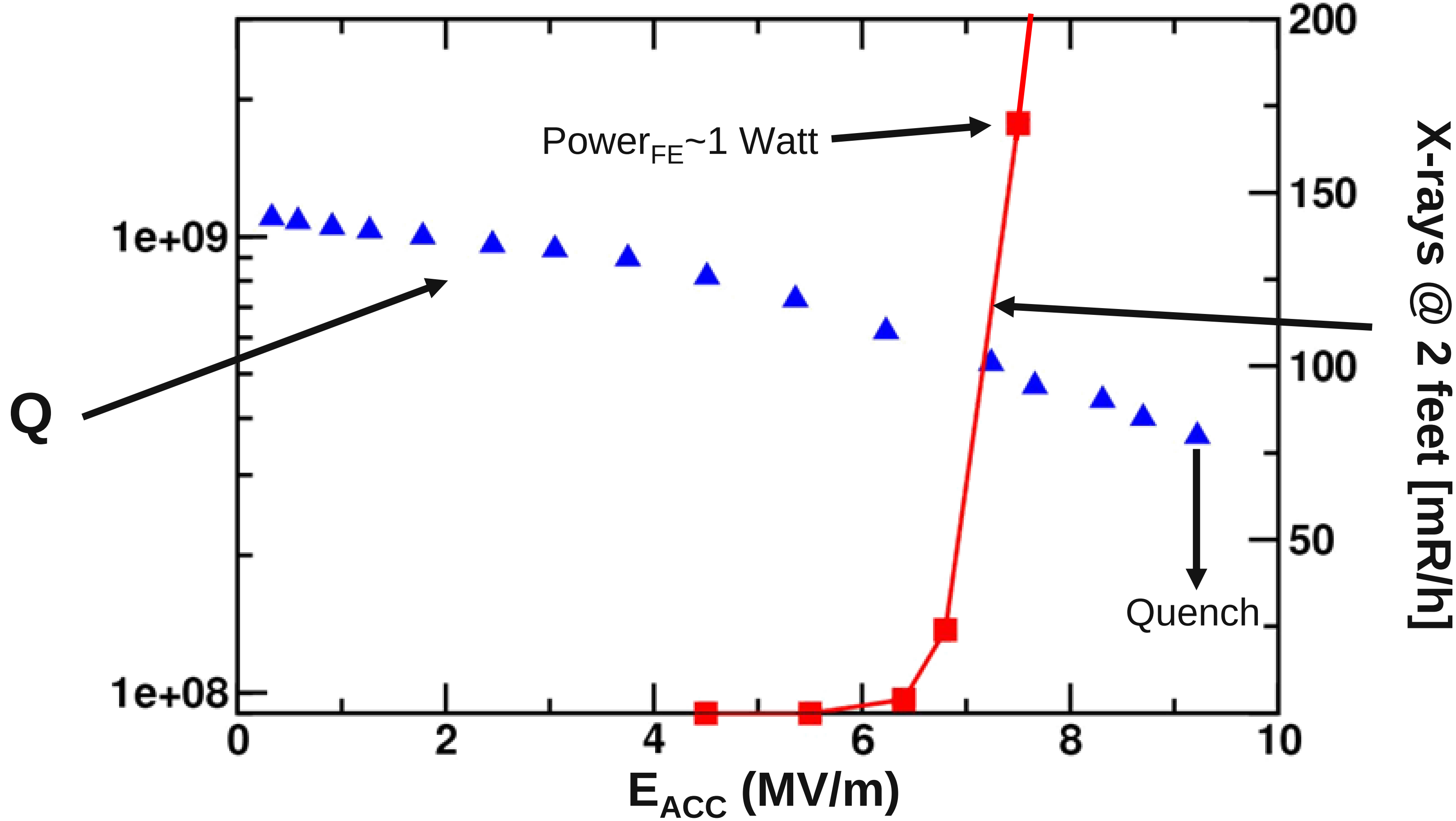
- Dwelling in the hydride formation region leads to increase rf losses
- Performance (mostly) recovers after recycling to room temperature

Reference[5]

I. RF Surface: Field Emission

- Tunneling of electrons at a metal surface through a potential barrier in the presence of an applied electric field
- Fowler-Nordheim tunneling, $J \sim E^2 \exp(-K/E)$
- Manifests in SC cavities by the presence of x-rays, primarily bremsstrahlung from electrons striking the cavity walls
- Experimentally known to be mostly due to loosely attached particulates on the RF surface – *i.e.* dust
- Microscopy studies at emitter sites show Ag, Al, C, Ca, Cl, Cr, Cu, Cs, F, Fe, In, K, Mg, Mn, N, Na, Ni, O, S, Si, Ti, W, Zn Reference[6]
- *Amount of field emission is largely determined by final steps in preparation of the RF surface and does not represent a fundamental limit*

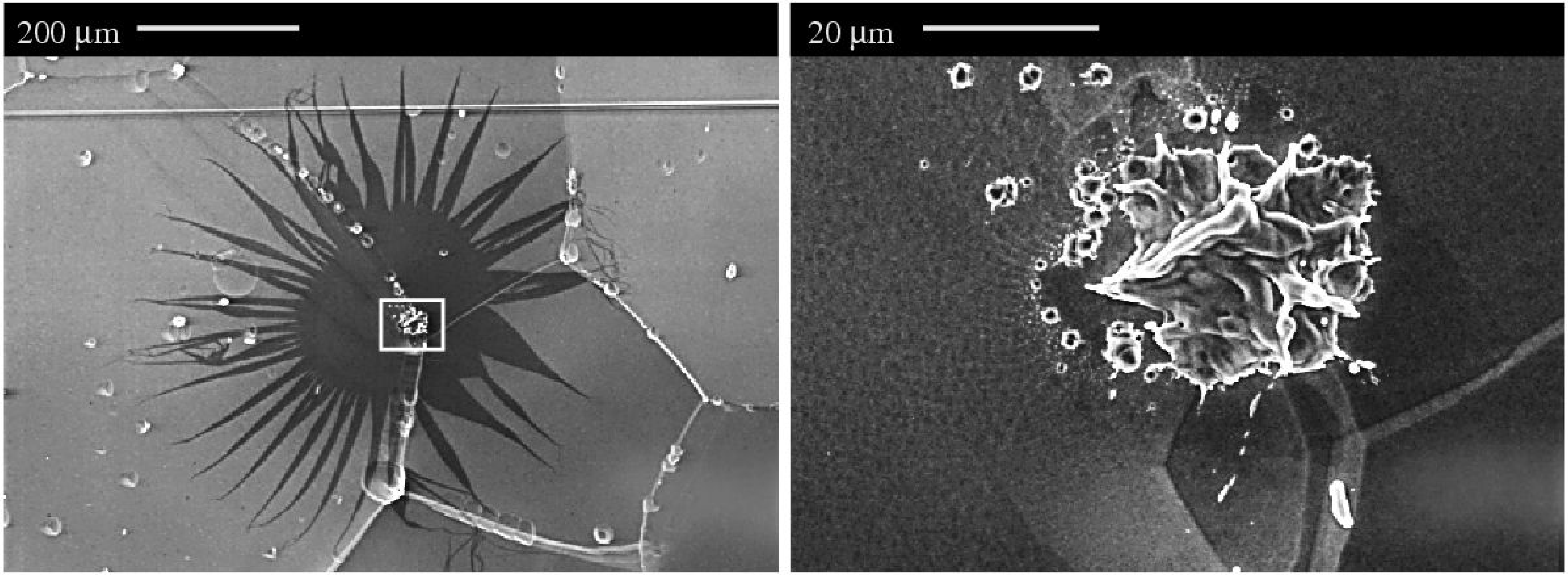
I. RF Surface: Example of Field Emission from Particulate Contamination, spoke cavity @ 4 K



- Field emission turns on rapidly after onset (here @ E_{ACC}~5 MV/m)
- Quench (thermal instability) often induced by field emission

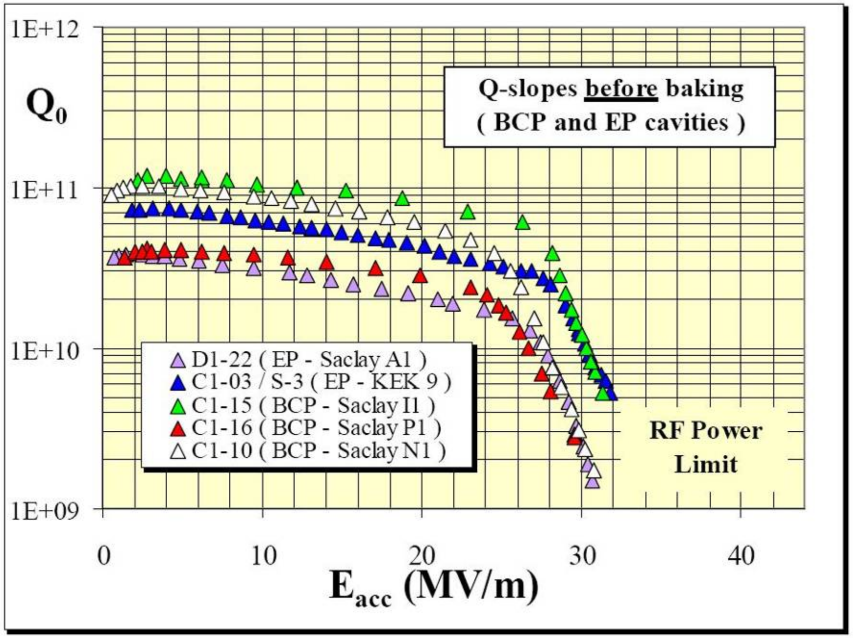
I. RF Surface: Processing of Field Emission

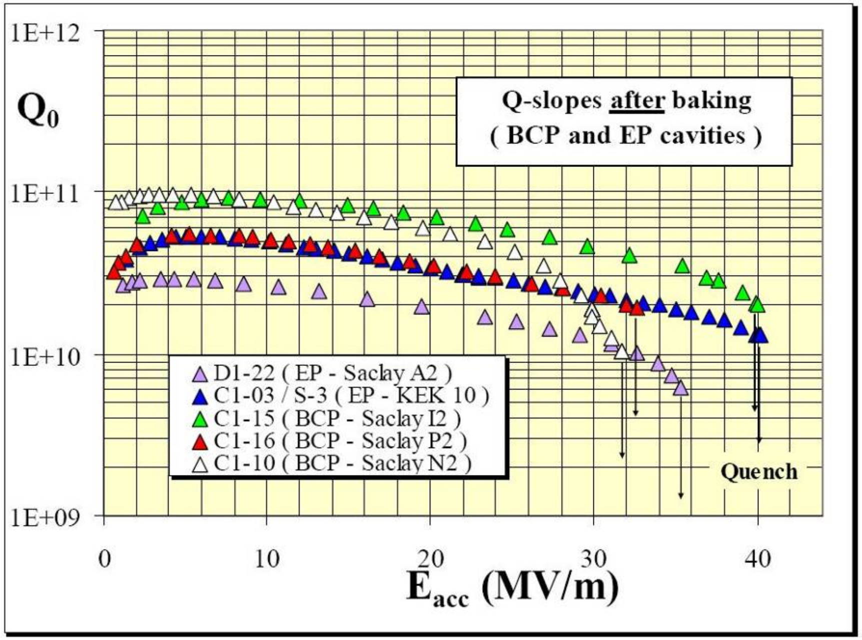
- Emitters may be destroyed using high-power pulsed processing
- RF power of several to hundreds of kilowatts in millisecond pulses
- Processing may be enhanced by the introduction of helium gas (0.1-1x10^{-4} Torr) into the cavity vacuum



Reference[7]

I. The RF Surface: High-field Q-slope





- Theoretical basis not fully understood
- May be due to oxygen diffusion; hydrogen is not the cause
- High-field Q-slope is improved (single-cell 1.3 GHz here) with an in-situ bake
- Bake parameters: 120^{o} C for 48 hours typically in vacuum

Reference[8]

II. Practical Considerations:

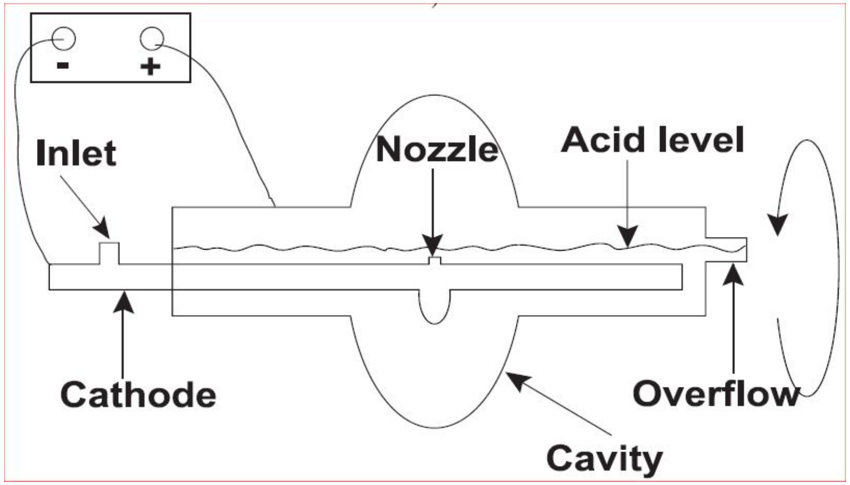
- There are dozens of primary steps and hundreds of minor steps in the fabrication of a real SC cavity – almost any one of these has the potential to destroy the SC performance

II. Practical Considerations: A Summary of the State-of-the-Art in Cavity Processing

- Specification, procurement, QA of (high RRR) niobium
- Fabrication
  - Die forming
  - Milling
  - Wire EDM trimming
  - Electron beam welding
- Electropolish to remove surface damaged layer (100 μm)
- Bakeout at high temperature (600-800^{o} C) to degass hydrogen
- High-pressure rinsing with ultra-pure water
- Clean room drying and assembly

- Many other possible intermediate steps determined by cavity type, performance goals, technical capabilities
  - Final light chemistry
  - Ultrasonic cleaning
  - Methanol rinsing
  - In-situ 120^{o}C bake

II. Practical Considerations: Recipe for EP

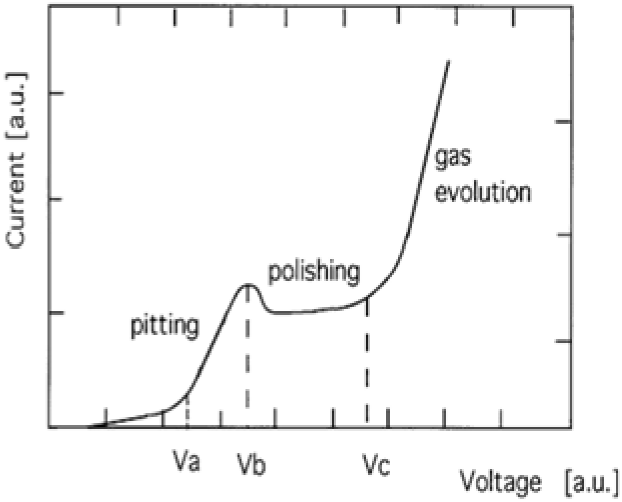


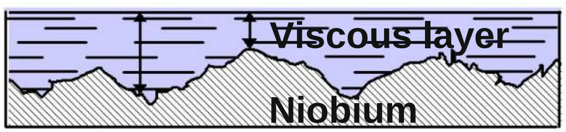
Single cell cavity EP

- Process: Developed by Siemens for SC cavities in the early 1970's
- Anode: Niobium cavity
- Cathode: High purity Al (1100, 3000 series or similar) roughly tailored to the cavity shape; cathode area 10% or more surface area of Nb
- Anode-cathode potential ~ 12-18 Volts
- Acid composition 85:10 mixture of 96% H_2SO_4, 40% HF, reagent
- Temperature 25-35^o C (*e.g.* chilled water through a hollow cathode)
- Duty cycle: Continuous for e-cell geometries, intermittent (1 min on 1 min off)
- Average anode current density ~40 mA/cm^2
- Acid sheer velocity at niobium surface ~ 1 cm/s

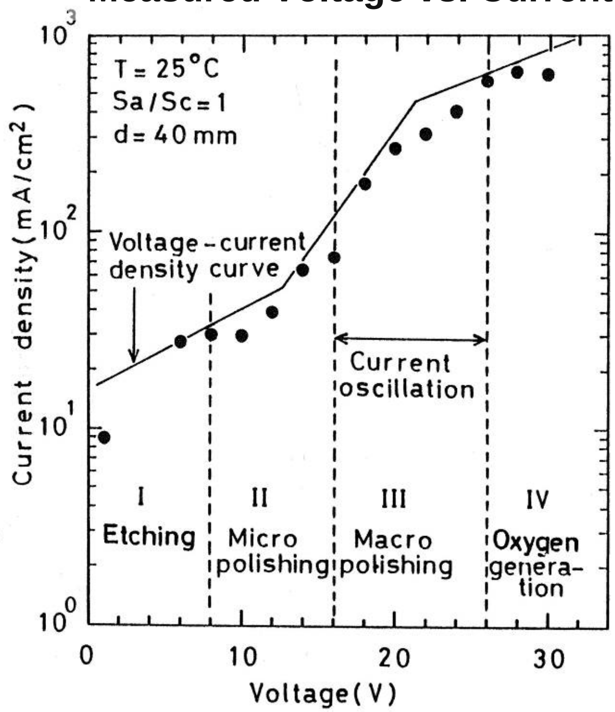
II. Practical Considerations: Fundamental Aspects of EP

Canonical Voltage vs. Current

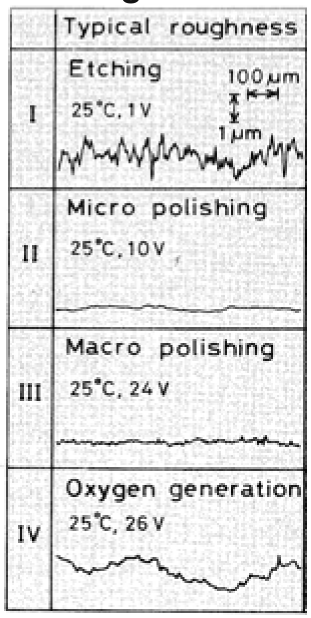




Measured Voltage vs. Current



Characteristic roughness



- A plateau in the I-V curve corresponds to the formation of a viscous layer
- High points are preferentially removed when in the EP regime ⇨ smoothing

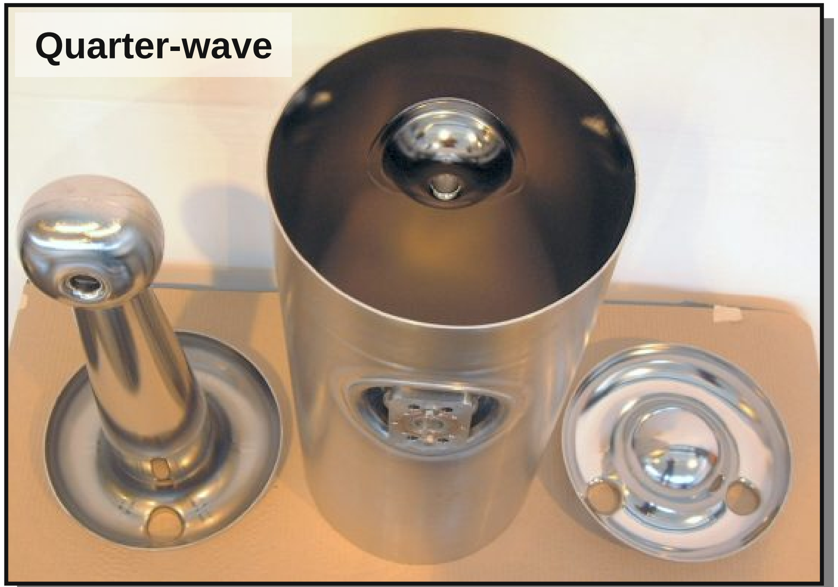
$$2Nb + 5SO_4^{--} + 5H_2O \rightarrow Nb_2O_5 + 5SO_4^{--} + 10H^+ + 10e^-$$

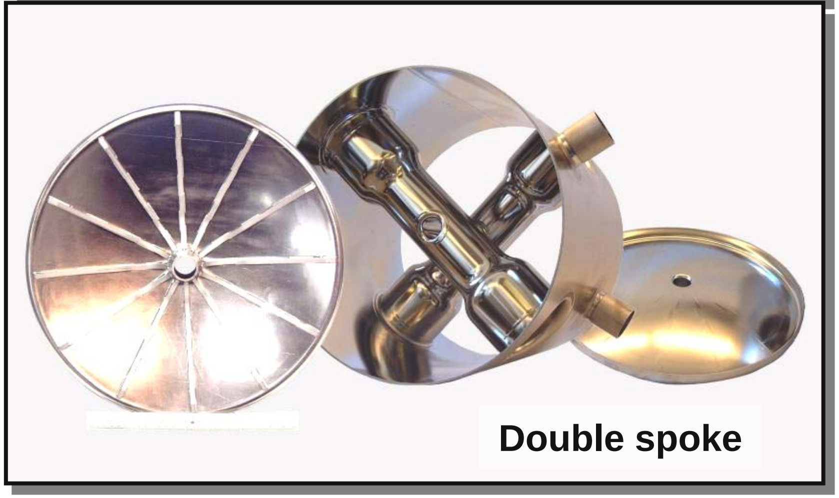
$$Nb_2O_5 + 6HF \rightarrow H_2NbOF_5 + NbO_2F \cdot 0.5H_2O + 1.5H_2O$$

$$NbO_2F \cdot 0.5H_2O + 4HF \rightarrow H_2NbF_5 + 1.5H_2O$$

Reference[9,10]

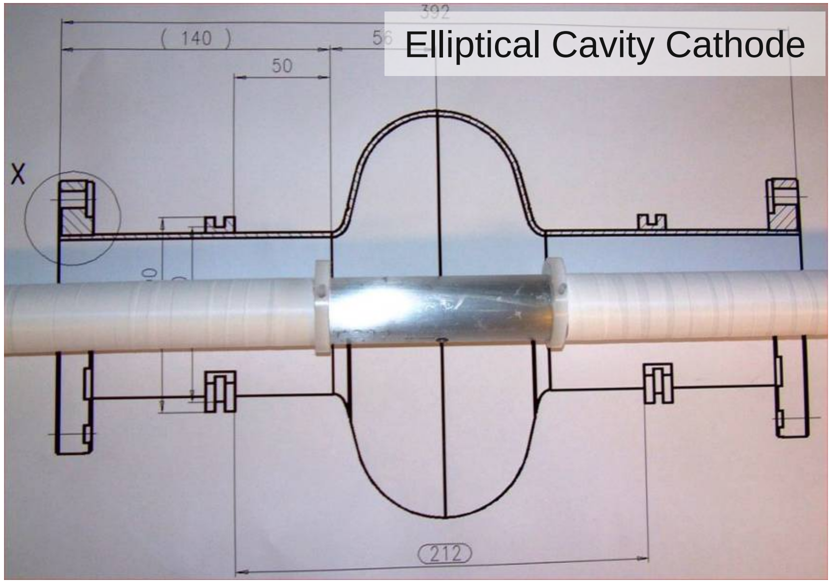
II. Practical Considerations: EP adaptable to various shapes





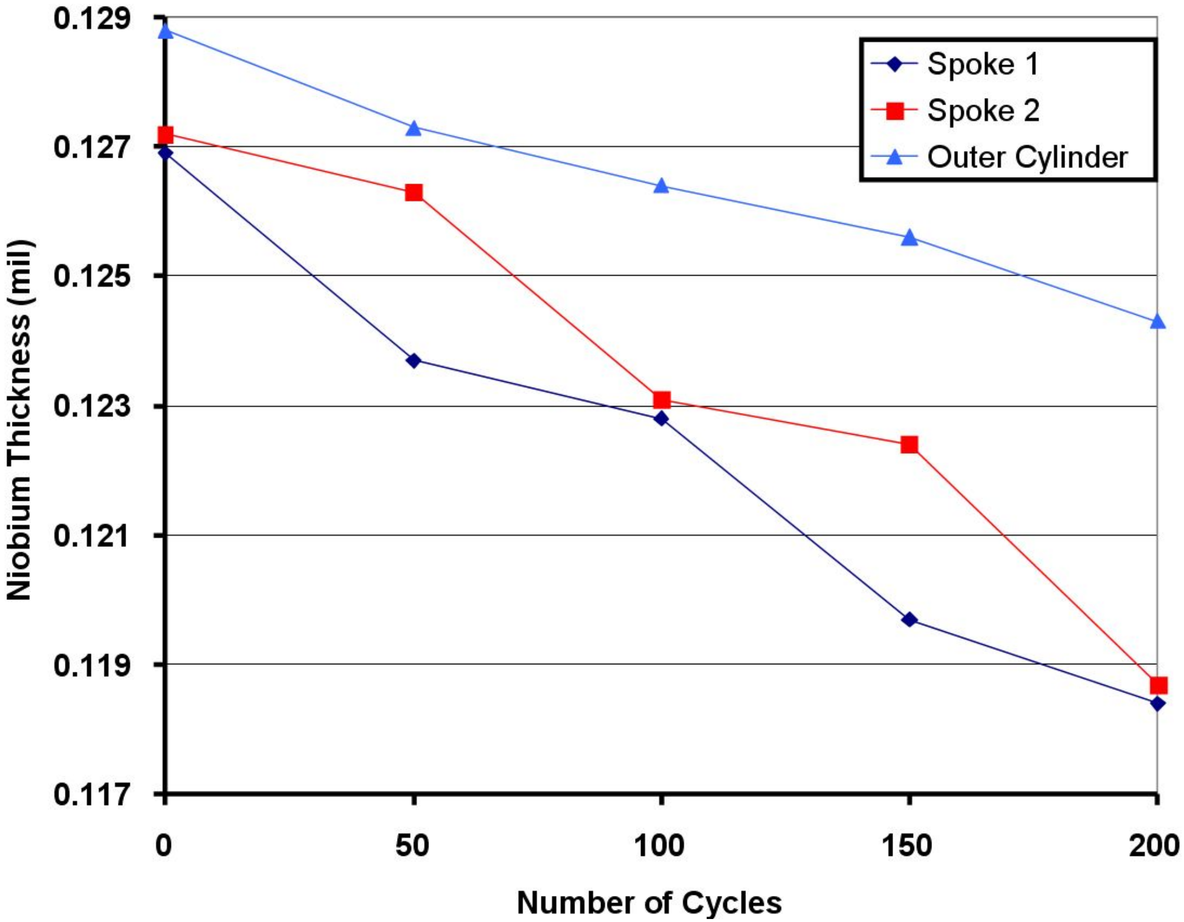
II. Practical Considerations: Electropolished Triple-Spoke Resonator: RF surface area ~1.5 m^2

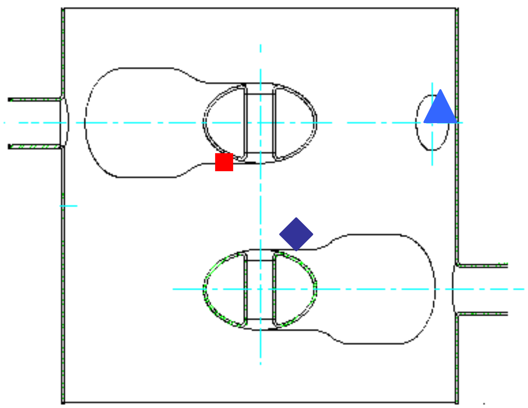
II. Practical Considerations: EP cathodes



- Hand wound cathodes from ½” 3003 Al tubing (left, middle)
- Single 1.3” OD high-purity aluminum tube (right)

II. Practical Considerations: Differential electropolishing due to orientation of niobium surface

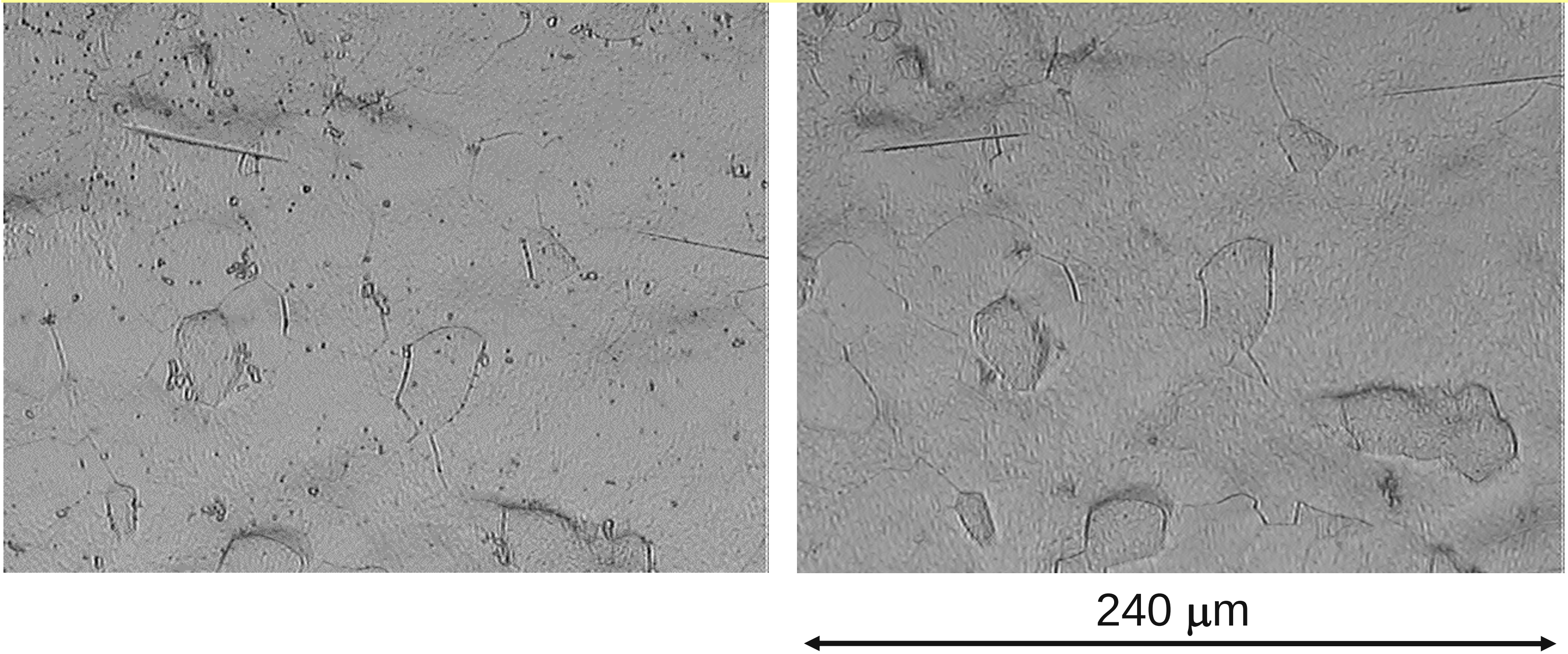




- Cavity surface removal measured using a ultrasonic thickness gauge
- Cavity flipped after each 50 cycles (1 cycle = 1 minute)
- Twice the surface removal for downward facing surfaces as for upward facing surfaces

II. Practical Considerations: HPR to Remove Particulates From an Electropolished Niobium Surface (1750 PSI)

- Particulates are the most important cause of field emission

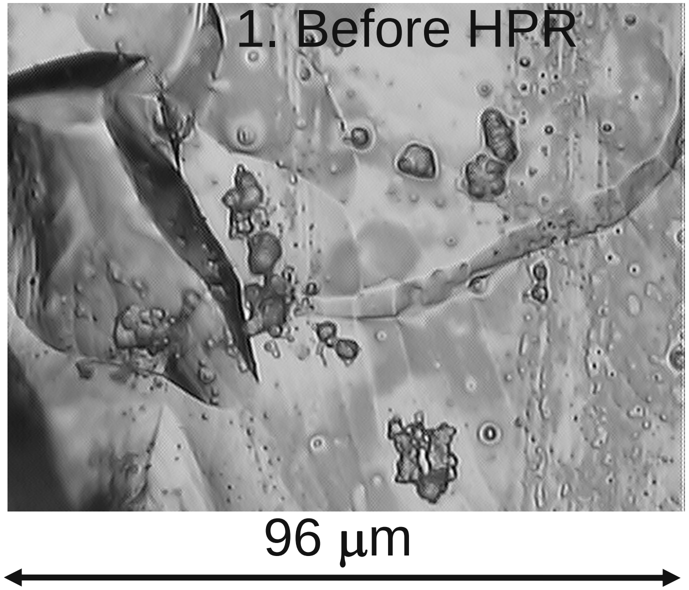


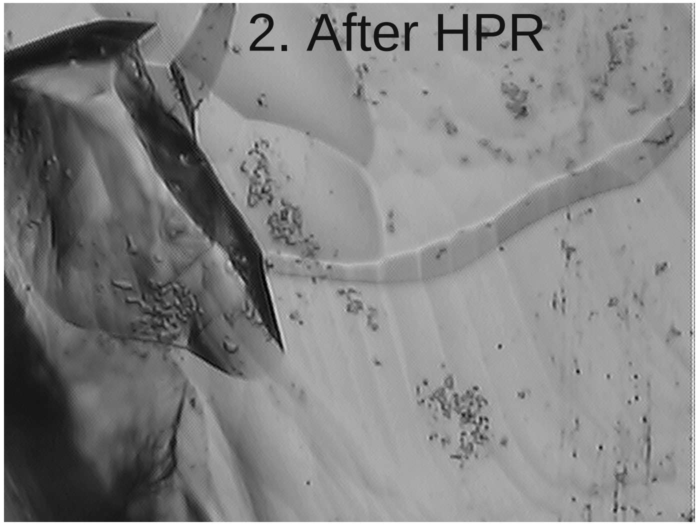
- Adhesion forces bind particulates to the cavity surface
- A high velocity water jet (150 m/s) effectively remove particulates
- Practical limit ~ 1 μm

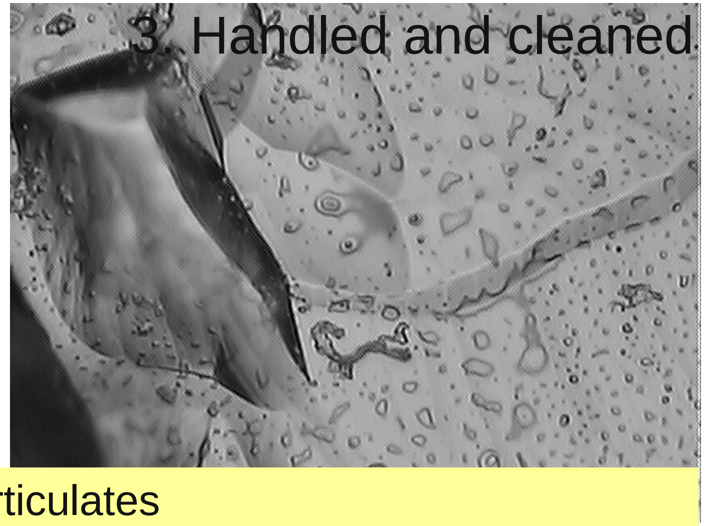
  -adhesion forces scale as particle diameter, mechanical force scales as particle area

Reference[11]

II. Practical Considerations: Limitations of HPR



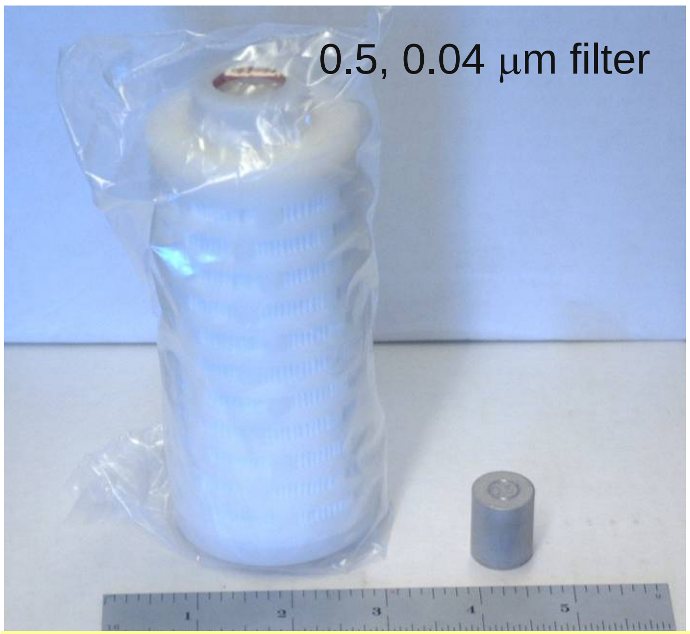




1. A BCP surface showing 10 µm-sized particulates
2. After rinsing at high-pressure water at 1750 PSI; small particulates remain
3. After handling with hands, cleaning with ethanol and drying

II. Practical Considerations: High-Pressure Rinse Hardware

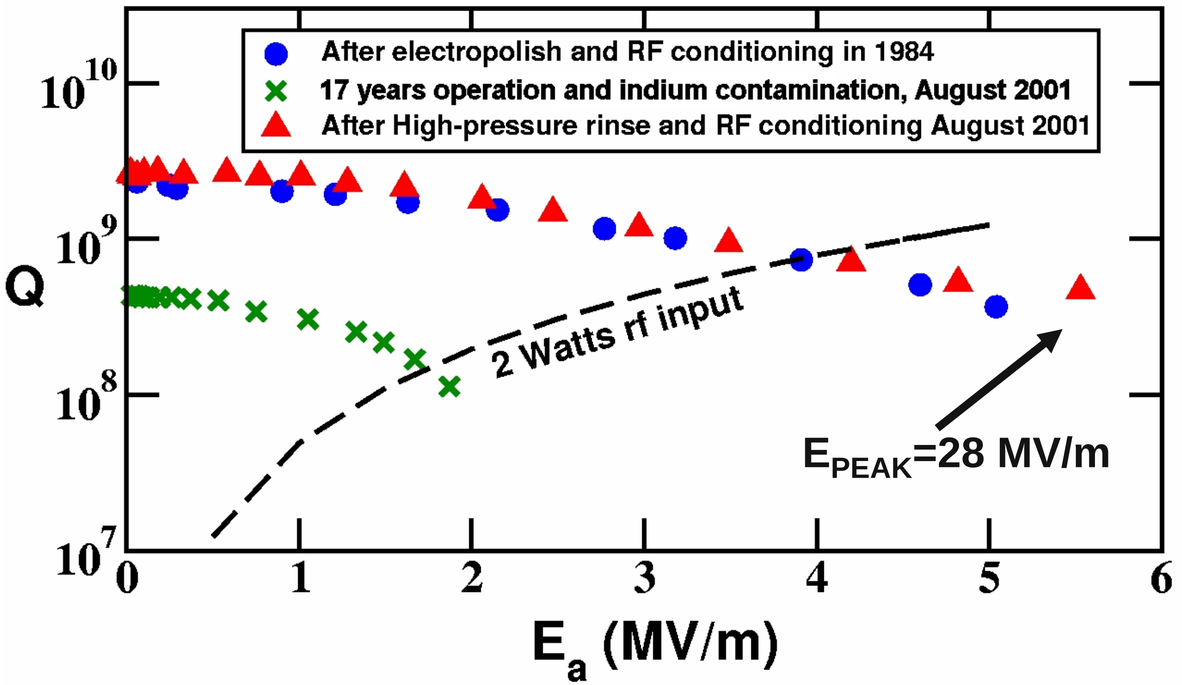
HPR pump

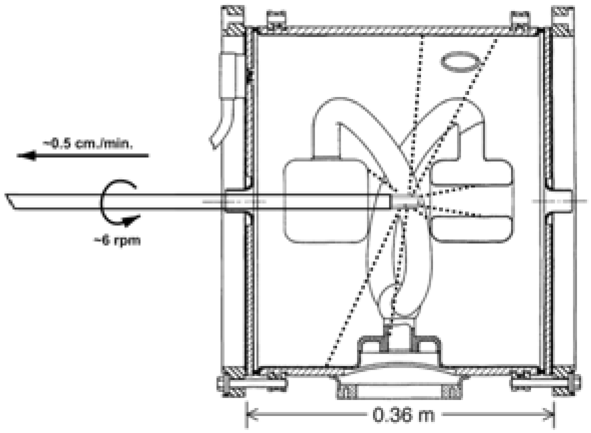


- Deionized water system
- Stainless steel or sapphire tipped nozzles
- Filtration on high-pressure sized of the pump
- Spray wand, clean room area

Reference[12]

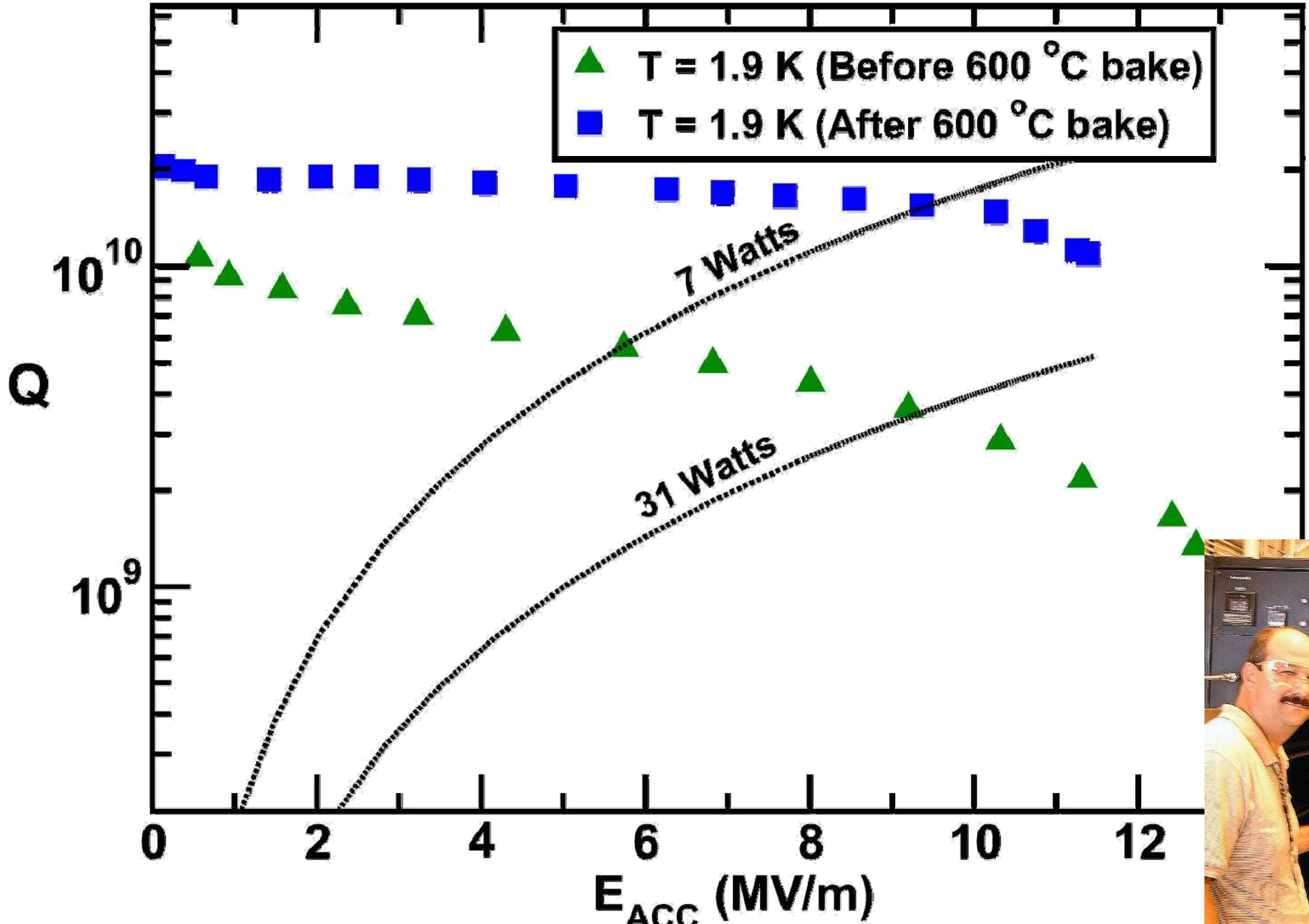
II. Practical Considerations: High-Pressure Rinse of an ATLAS Split-ring





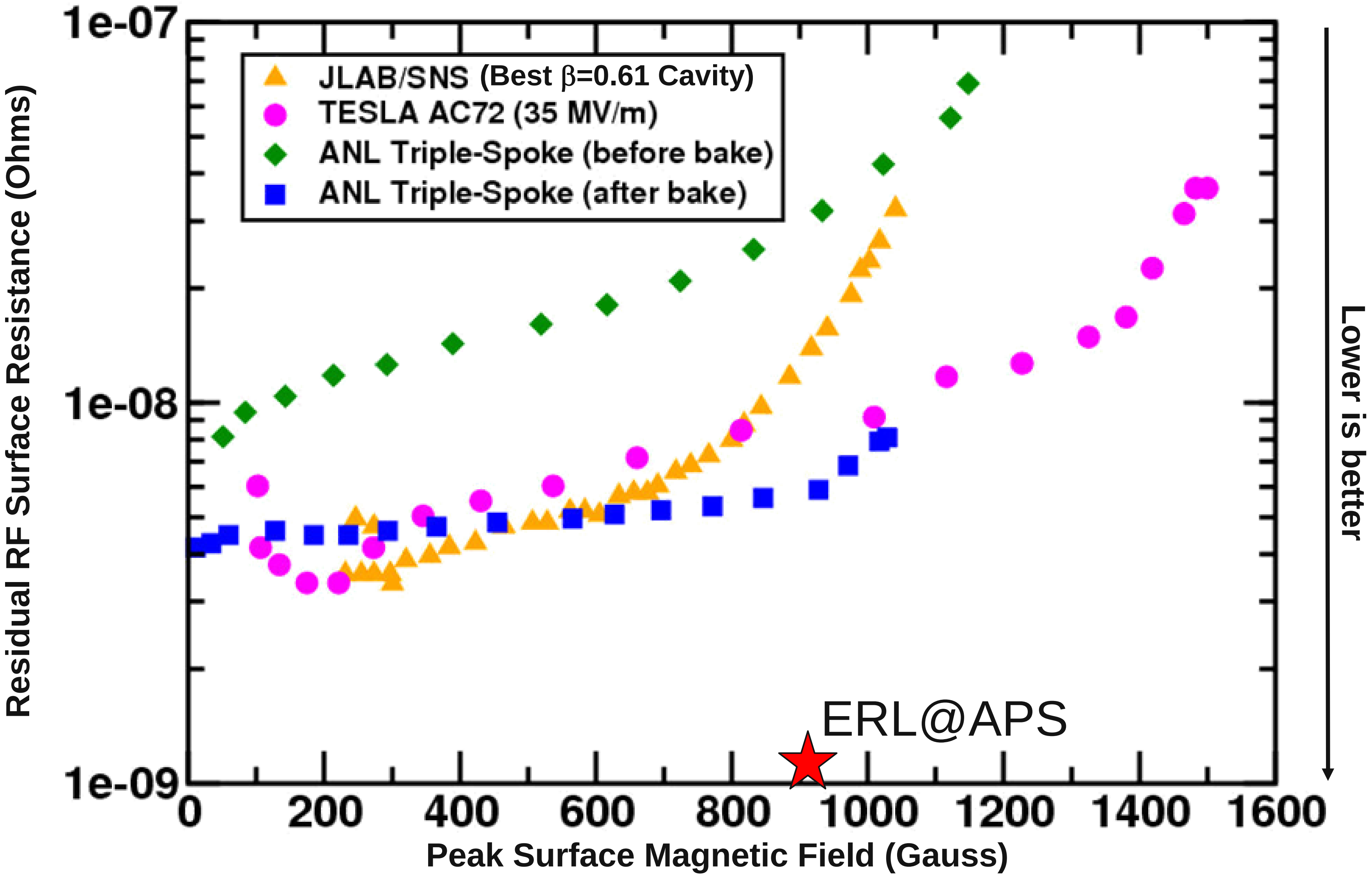
- Robust nature of SRF technology; HPR after 17 years operations
- The highest Q (>6×10^{9} at 2 K)
- Highest cw accelerating fields (6.8 MV/m at 2 K, E_{PEAK}=34 MV/m)
- Lowest surface resistance (R_{RES}=2.7 nΩ)

II. Practical Considerations: Effect of Hydrogen Degassing

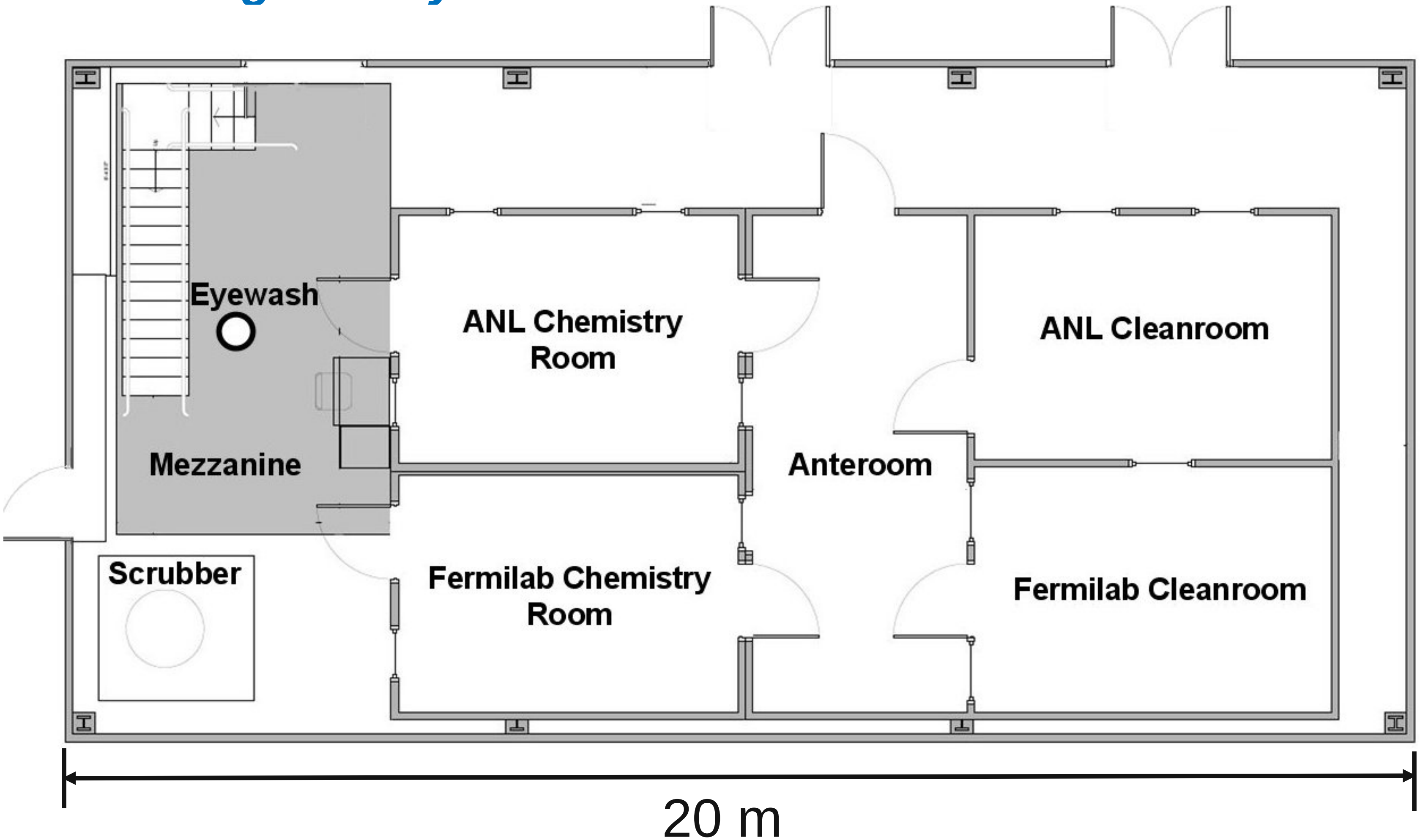


- Baking at 600-800ºC in order to degas hydrogen is required in order to achieve the lowest possible rf losses

II. Practical Considerations: Cavity Performance
Residual Surface Resistance vs. B_{PEAK}



II. Practical Considerations: Joint ANL-FNAL Single Cavity Processing Facility

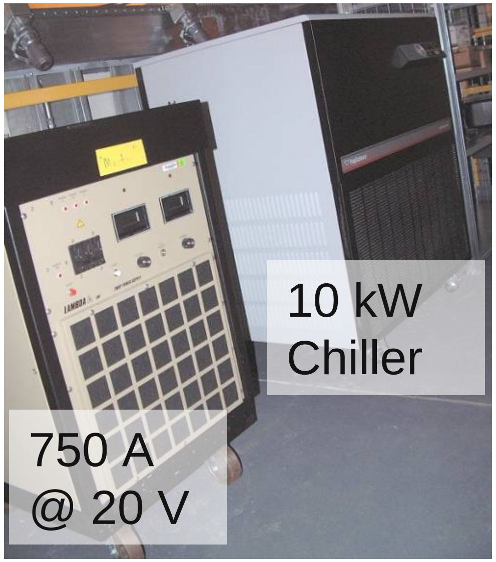


- Total Cost with manpower ~$2 M

II. Practical Considerations: ANL Portion of the Chemistry Facility

II. Practical Considerations: SCSPF Shared Class-1000 Anteroom

II. Practical Considerations: ANL-FNAL Shared Infrastructure

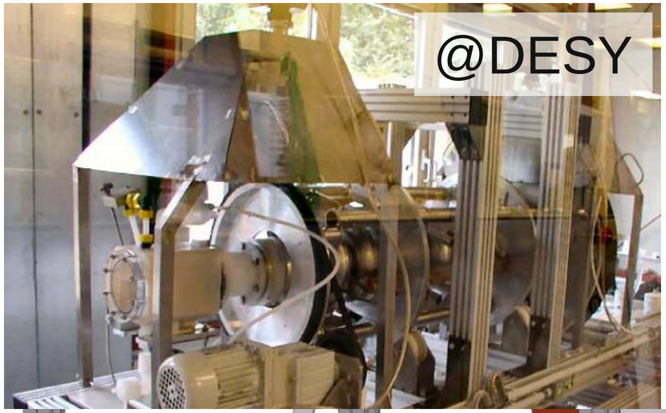


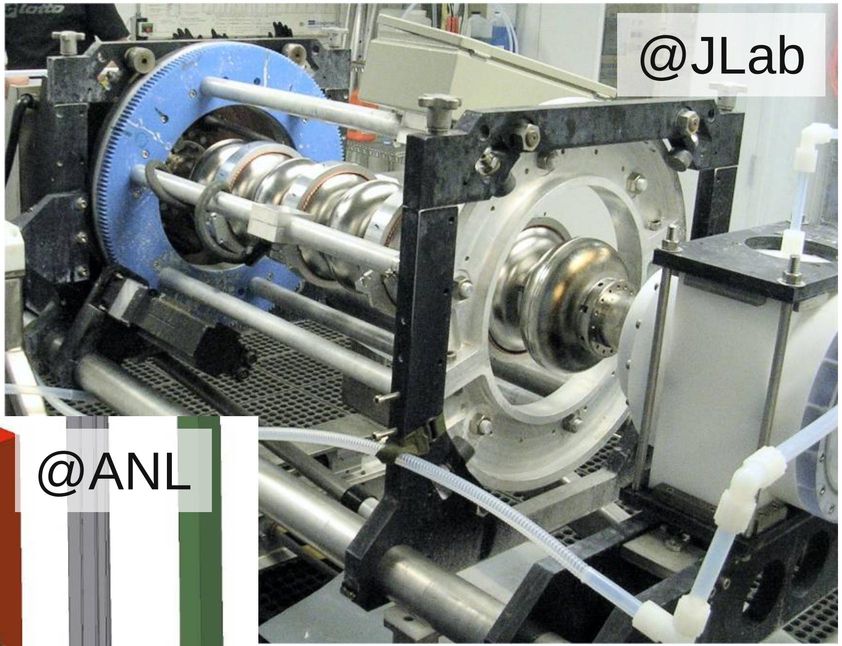
Air Scrubber

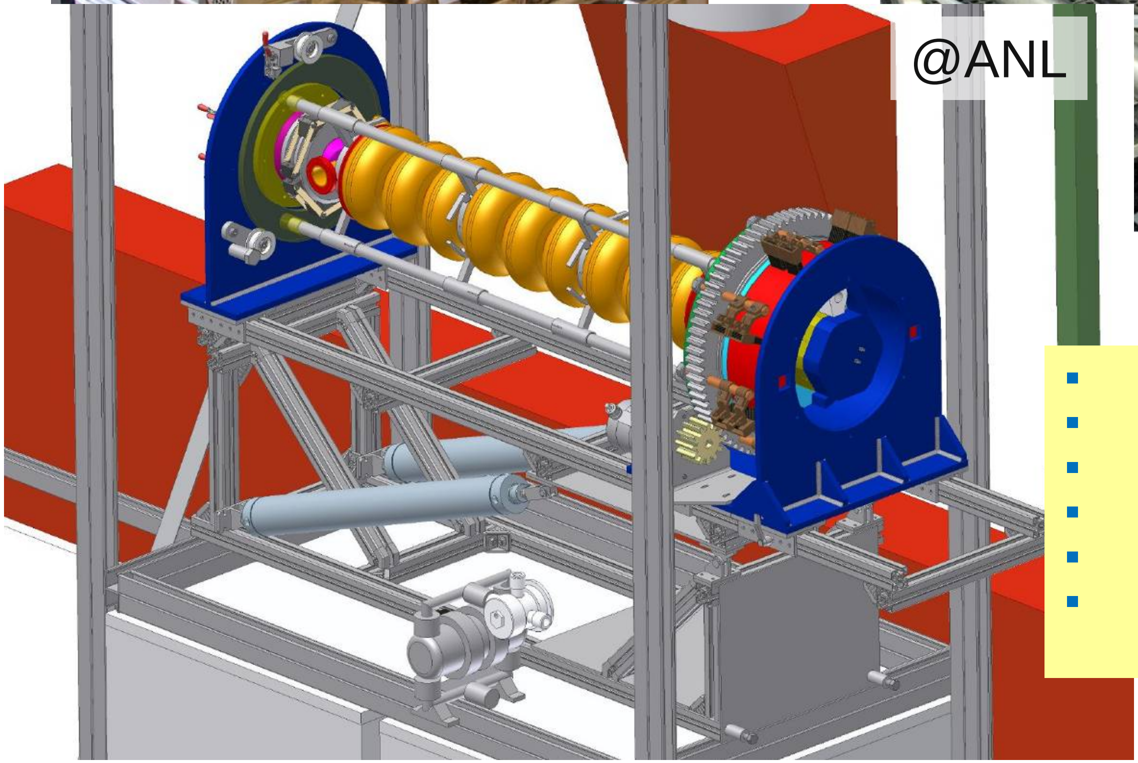
DI Water System

Chiller/EP Supply

II. Practical Considerations: Electropolish Hardware for 9-cell 1.3 GHz Cavities







Features

- **Horizontal EP**
- **Cleanable – no sulfur buildup**
- **Aluminum heat exchanger**
- **Fast fill/empty**
- **Direct Water Cooling (upgrade)**
- ***Direct experience for FNAL/ANL personnel***

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See Also: http://tdserver1.fnal.gov/project/workshops/RF_Materials/agenda.htm