

# Processing of Superconducting RF Cavities



### S. Raghavendra Raja Ramanna Centre for Advanced Technology

# Why is PROCESSING required for cavity ?



- The performance of a SCRF cavity depends on the *quality of* the RF surface.
- The surface purity gets affected by oil, grease, finger-prints, etc. during forming, machining & RF measurement
- The internal surface of the cavity may also have micron-size surface defects like pits, dents, weld spatter etc.



**Pits outside HAZ** Diameter: 400μm,Depth: 60μm





Pits at Iris Diameter: 400μm, Depth: 37.5μm

Quench location on equator 120µm deep, 700µm wide on top

# Why is PROCESSING required for cavity ?

- Around 100 micron layer on the surface gets damaged & contaminated during fabrication which needs to be removed.
- The main parameters limiting the performance of a superconducting cavity are:
   a) Residual Resistance
   b) Field Emission
   c) Q-Slope
   d) Quench

e) Multipacting

Processing of the SCRF cavity refers to surface preparation of the cavity by improving its surface purity & quality for optimum cavity performance.



Accelerating Field





- Inspection of internal surface of the cavity
- Ultrasonic Cleaning degreasing of surfaces to remove contaminates
- Polishing of internal surface to remove bulk material (~ 120-150 μm)
- Thermal processing removal of hydrogen from bulk niobium
- Mechanical tuning
- Light electropolishing Final polishing step (10-20 μm)
- High Pressure Rinsing (HPR) to remove particulates from internal surfaces (incurred during polishing and handling)
- Drying of cavity for assembly in cleanroom
- Clean assembly and evacuation
- Low-temperature baking
- Preparation for low power RF test at 2 K VTS testing



### **Optical Inspection System**

- An optical inspection of the inner surface, leads to a better understanding of limitations of SCRF performance with respect to surface quality. This provides a qualitative analysis of the surface.
- An optical inspection system was developed at KEK and Kyoto University, better known as the "Kyoto camera system". It is an expensive system costing more than Rs. 50 lakhs. The Kyoto camera system is used at KEK (Japan), DESY (Germany), Fermilab (USA), Jlab (USA), etc.
- An optical inspection system has been indigenously developed at RRCAT, which is costing less than 5% of the Kyoto system.



### **Optical Inspection System**



Illumination System

Digital CCD Camera

Positioning system



### **Optical Inspection System**



Inspection of nine-cell 1.3 GHz cavity



Inspection of single-cell 650 MHz cavity





Images from optical bench (650 MHz cavity)





Inspection system at KEK, Japan



Inspection system at Cornell University



Inspection system at FNAL, USA



Inspection system at LANL, USA



### **Replica Molding Technique**

- The technique is use in quantifying the defects on the surface of cavity
- Replica casted at the location of defect identified by optical inspection
- Replica casted using Room Temperature Vulcanized Silicon
- The mold studied using 3D Laser Scanning Confocal Microscope.



Laser Scanning Confocal Microscope



Silicon Mold

Confocal image of replica



#### Measurement of bead profile



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# **Degreasing of components & cavity**



### **Requirement of degreasing at various stages**

- After forming + machining and welding of components
  - To remove grease, oil and finger prints from cavity surfaces
  - To remove surface contamination due to handling, RF measurements and QA inspection
- After polishing processes
  - To remove traces of chemicals and other residues

### **General method of degreasing – Ultrasonic Cleaning**

- Ultrasonic cleaning is a process that uses ultrasound and an appropriate cleaning solvent to clean items.
- □ Piezo transducers are used to produce standing waves in the solution.
- Wave energy forms microscopic bubbles on component surfaces. Bubbles collapse (cavitation) on surface loosening particulate matter. These bubbles collapse with enormous energy

# **Degreasing of components & cavity**



- Ultrasonic cleaning is carried out in mixture of ultra-pure water and special Phosphorous and Sulphur free detergent like Micro-90, Liqui-Nox (1%-2% concentration at 45-50°C.
- The cleaning should be preferably performed in a clean environment.
- Ultra pure water has a high level of purity; Resistivity – 18 MΩ-cm, TOC < 30 ppb</li>



Ultrasonic cleaner for small components & single-cell cavities at RRCAT



Ultrasonic cleaner for five-cell 650 MHz cavity at RRCAT

### **Degreasing of components & cavity**







#### Pictures of degreasing facilities at few international labs



### **Megasonic Cleaning**

- Ultrasonic operate at lower frequency and random cavitation. Longer duration may damage components to be cleaned.
- Megasonics is like ultrasonics but operates at much higher frequency (typically 0.8–2 MHz). As a result, the cavitation that occurs is gentler and on a much smaller scale, less likely to cause damage
- With ultrasonics, cavitation occurs throughout the tank, and all sides of submerged parts are cleaned. Whereas, with megasonics, the acoustic wave is found only in a line of sight from the transducer surface.
- Hence, more transducers are required for similar volume and also, the process is much slower. This makes it more expensive.
- The efficiency to remove particles of size < 5 microns is lower for this process.



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- As received high purity niobium material is an "Ideal" surface. It has defect-free niobium crystals with only ~ 5 nm Nb<sub>2</sub>O<sub>5</sub> layer.
- After cavity fabrication stages like forming, machining and welding, the real surface is "damaged" and "contaminated".
- Generally full penetration welding is performed from outside of cavity. This produces prominent weld bead of few hundreds of micron on the inside of cavity. This bead needs to be polished to avoid pre-mature quenching and deteriorating quality factor.
- In the process of removal of material, it is also necessary to produce a highly finished (mirror finish) surface with surface roughness of few tens of nano-meters
- The naturally-forming  $Nb_2O_5$  is a very stable oxide weak acids don't touch it



Hydrofluoric acid (HF), which is a very strong acid is required in processing of niobium cavities



 Empirically found that >100 μm removal is typically required to reliably expose "good" bulk Nb material, i.e. predictable SRF performance.



Residual resistance & Peak electric field v/s material removed from equator Accelerating gradient v/s material removed from equator







Chemical polishing/ etching is required at various stages of cavity development :

### Sub-components require

 Pre-weld etch : Removal of contaminants which come from fabrication steps to reduce the losses and improve cavity welding.

#### **Cavities require**:

- Internal cleaning/polishing 100-200 μm
- External cleaning 20-30 μm material is etched from the outer surface of the cavity by BCP to remove "dirty" layer after fabrication in order to improve the heat transfer at the Nb/LHe interface (Kapitza resistance)



### Etching solution used for BCP : $HF (48\%) + HNO_3 (65\%) + H_3PO4 (85\%)$ Mixture ratio 1:1:1 - used for etching of subcomponents (etch rates up to 5 µm/min) – still used at some labs and 1:1:2 - used for most cavity treatments (etch rates of 1- 3 µm/min) Chemical Reaction Oxidation

 $2Nb + 5HNO_3 -> Nb_2O_5 + 5NO_2$ 

Reduction  $Nb_2O_5 + 6HF \rightarrow H2NbOF5 + NbO_2F 0.5H_2O + 1.5H_2O$  $NbO_2F 0.5H_2O + 4HF \rightarrow H_2NbOF_5 + 1.5H_2O$ 

- Reaction exothermic
- $\succ$  H<sub>3</sub>PO<sub>4</sub> used to "buffer" the reaction rate
- Agitation by stirring/ rotation/ continuous flow
- Acid is usually cooled to 10-15 C to control the reaction rate



• At RRCAT, BCP is presently used for cleaning of cavity components & pre-weld etch only.



• The present processing recipe for HB 650 cavities does not include BCP





BCP Cabinet at Jlab, USA



BCP of single-cell cavity in fume hood





# TI T Y YI B

### Why electropolishing ?

- Electropolished cavities have quality factor better than chemical polished (BCPed) cavities
- The best accelerating gradients have been achieved with electropolishing
- Better surface finish achieved with electropolishing. An order better !
- Better control over the process.





# EP is an electro-chemical method of removing thin layers of metal from a surface by anodic dissolution

- Both electrodes are immersed in electrolyte
- A voltage is applied between Nb (anode) and counter electrode (cathode, Al)

### Basic reactions:

Oxidation 2Nb +5SO<sub>4</sub>2- + 5H2O  $\rightarrow$  Nb2O5 +10H+ +5SO42- 10e-

Reduction

 $\begin{array}{ll} Nb_2O_5 + 6HF \rightarrow & H_2NbOF_5 + NbO_2F \ 0.5H_2O + 1.5H_2O \\ NbO_2F \ 0.5H_2O + 4HF \rightarrow & H_2NbOF_5 + 1.5H_2O \end{array}$ 

Therefore the overall chemical reaction is:  $2Nb + 10HF + 2H_2O \rightarrow 2H_2NbOF_5 + 5H_2$ 







The limiting concentration of dissolved niobium for effective EP is 10 g/l. The acid needs to be changed after the limiting dissolution of niobium

Adding F- in the existing solution is difficult and complicated.



- In the electropolishing process for SCRF cavity, the niobium cavity is the anode.
- The cathode is made from pure aluminium (1100 series).
- The electrolyte is a mixture of hydrofluoric (HF) and sulphuric acid (H2SO4) in a volume ratio of 1:9, using typical commercial strengths HF (48%) and H2SO4 (98%).



Horizontal electropolishing setup

# Hydrogen is released at cathode, which is absorbed by niobium leading to Q-decease





Setup to understand the EP process on single-cell cavity





• Depending on applied voltage and sample geometry, the electropolishing process follows a typical I-V curve, which decides the optimum voltage for a "good" EP:





- The current density (30-100 mA/cm2) in the plateau region:
  - -decreases linearly with lower HF/H<sub>2</sub>SO<sub>4</sub> ratio
  - -increases with increasing temperature
- Temperature during the process is maintained between 25 35  $^\circ$ 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.

Current density	10-150 mA/cm <sup>2</sup>
Voltage	8-25 V
Removal rate	0.1 to 0.4 µm/min
Rotation speed	1-2 rpm
Acid flow rate	2-15 L/min



#### **Electropolishing Setup at RRCAT**



Horizontal electropolishing setup for polishing of 1.3 GHz and 650 MHz SCRF cavities.



Cavity moved to vertical position for draining of electrolyte, rinsing of cavity and For insertion/removal of cathode

### **Cavity Processing Facility**



### **Electropolishing setup for 1.3 GHz & 650 MHz Cavities**





Electropolishing of nine-cell 1.3 GHz cavity



Electropolishing of single-cell 650 MHz cavity





Electropolishing setup at KEK



Electropolishing setup at JLab



Electropolishing setup at DESY



Electropolishing setup at ANL





#### **Vertical Electropolishing Setup**





Pros:

- No rotary acid seals
- Twice removal rate than horizontally rotating EP
- No sliding electrical contacts

#### Cons :

- Acid aging is must faster.
- Repeatability of performance of cavity is lower
- $\blacktriangleright$  Difficult to remove hydrogen  $\rightarrow$  Q-disease

Setup at Cornell university, USA

# **Effluent Treatment of Used EP acid and vapors**



- The gaseous waste like hydrogen, vapours of HF, Sulphuric acid, SO<sub>2</sub>, H<sub>2</sub>S etc. shall be sucked through the suction blowers and passed through a wet scrubber system.
- Aqueous NaOH (40%) shall be used for scrubbing the acidic gases before discharging them to air through a stack. The gases shall be diluted with fresh air before the scrubbing.
- The scrubbing liquor, rinsed dilute acid waste and used acid shall be treated in an effluent treatment plant (ETP).









cel

direction

### **Working Principle**







### **Typical Polishing Media for Barrel Polishing**



**Ceramic Media** Cutting time depends on the material to be removed.



**Plastic/ Polyester Media** *Typical cutting time : 24 - 48 hours* 



**Colloidal Silica & Corn Cob** Typical cutting time ~ 75 -100 hours



- Very useful process to remove the surface defects.
- Benefit of the process is reducing the chemical polishing process, which reduces handling of large amount of toxic & corrosive acids.



### **Barrell Polishing Facility at RRCAT**



#### CBP Machine for single-cell 1.3 GHz cavity



CBP Machine for five-cell 650 MHz cavity

The CBP machine for five-cell 650 MHz cavity is the only operating machine in the world for this size of cavity

- Controlled temperature
- Can polish 4 five-cell 650 MHz cavities simultaneously





At Jlab, USA



#### At Fermilab, USA



At KEK, Japan



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### Thermal processing Hydrogen Absorption and Q-Decease



- Niobium has a natural 5 nm thick passive oxide film .
- This oxide film on the niobium protects it from chemical contamination and it is protective against hydrogen absorption
- This layer is dissolved by F- ions during electropolishing and during barrel polishing
- Hydrogen is absorbed by niobium when it comes in contact with un-protected niobium surface; during electropolishing and barrel polishing
- This increases the chance of hydride formation, which is a normal conducting impurity at 4 K/ 2 K
- When cooling down a cavity, precipitation of dissolved hydrogen (hydrides) occurs around 100 K. The possibility of hydride formation increases multiple times when called around 100 K
- Hydrides increase the surface resistance of the cavity (Q-disease!)
- The cavity quality factor drops suddenly as the field increases due to presence of hydrides, This phenomenon is called Q-Decease

#### S Raghavendra | SCRFWS-2017, 18-21 July 2017, RRCAT

### **Thermal processing**

- A high temperature degassing treatment, in the range 600-1000°C, is necessary to prevent poor cavity operational performance due to formation of hydrides precipitates Nb-H.
- Different parameters at different labs:
  - 600°C for 10 hours/ 800°C for 2 hours at RRCAT
  - 800°C for 2-3 hours at Fermilab
  - 600°C/10 h at JLab
  - 800°C/2 h at DESY
  - 750°C/3 h at KEK
- The heat treatment also "stress-relieves" the niobium. This helps in the tuning of cavity.
- The heat treatments are then followed by a 20-40 µm amount of material removal again with EP or BCP, typically done to remove surface 'contaminants' which might be introduced during the furnace treatment.





### **Thermal processing at RRCAT**





**High Vacuum Annealing Furnace at RRCAT** 

Specification of High Vacuum Furnace				
Orientation	Horizontal			
Maximum Temperature	1400°C Max			
Working Vacuum	<1 x 10 <sup>-7</sup> mbar (600°C -1000°C ) <1 x 10 <sup>-6</sup> mbar (> 1000°C)			
Working Volume	Dia : 825 mm Depth :1500 mm			



Nine-cell 1.3 GHz cavity prepared for annealing

### **Thermal processing**





Annealing Facility at Fermilab



#### Annealing Facility at JLab



Annealing Facility at KEK



### **Nitrogen Doping for Q & E Improvement**

- Nitrogen doping at high temperature of 800-1000°C for a short time helps in improving the quality factor of cavity at medium fields.
- This also helps in improving the accelerating gradient of cavity by 10-15%
- The doped nitrogen goes in the niobium as interstitial element, which displaces hydrogen and prevent formation of hydrides.
- The optimized recipe by Fermilab, which is a part of their processing plan for LCLS-II cavities is as below:
  - ➢ 800°C−3h
  - 2 min @ 800°C + 25mTorr N2
  - 6 min @ 800°C vacuum
  - Cool down
  - 5 microns EP



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- Any dust, chemical residue or other contaminants on the interior of cavity causes "Field Emission".
- Jets of high pressure ultra-pure water dislodge surface contaminants that normally resist removal with conventional rinsing procedures, leading to substantial reduction in field emission and better cavity performance
- High pressure rinsing at 80 100 bar pressure can dislodge particles down to about one micron size (# Ref: P. Kinsel and B. Lewis, Proceedings of 1995 Workshop on

RF superconductivity)

• HPR is performed in class 100 or better cleanliness.





Schematic of high pressure rinsing setup





HPR Setup at RRCAT



#### **System Specifications**

Water Pressure Water flow rate Wand rotation Cavity movement Filtration Enclosure Nozzles

- : 80-100 bar
- : 10 l/min
- : 2-10 rpm
- : 5-250 mm/min
- : 0.05  $\mu$ m after swivel
- : Class-100 cleanroom
- :40° Fan jet



#### HPR Setups at different labs



HPR Setup at Jlab, USA



HPR Setup at DESY, Germany



HPR Setup at ANL, USA



### **Ultra-Pure Water**

Ultra-pure water is used for high pressure rinsing and ultrasonic cleaning of SRF cavities:

Ultrapure Water Parameters			
Resistivity	: 18 M-Ohm.cm		
тос	: < 30 ppb		
Bacteria	: < 1 cfu/100ml		

TDS : < 0.03 ppm



Ultra-pure water plants at RRCAT





### **Dry-Ice Cleaning**

- After HPR, the cavity dries in the clean room for 12 to 24 hours before it is prepared for VTS.
- The cavity is left in a WET state after HPR for prolonged time. This increases possibility of oxidation and also moisture
- Complementary method to HPR, developed at DESY
- Liquid CO<sub>2</sub> (Dry Ice) jet is used instead of water, resulting in a snow/gas mixture at a temperature of 194 K
- This removes hydrocarbons and sub-micron particles while keeping the surface dry
- This method has been tried as a R&D effort



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# **Clean assembly and evacuation**



- Cleanroom technology is required to prevent airborne particulates from settling on the surface of SRF cavities
- Cleanroom is a controlled environment in which all incoming air is filtered to meet high standards of purity. Temperature, humidity and pressure are also controlled.
- Humans 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
- Cleanroom of Class 10 to Class 1000 are required for assembly, cleaning, RF measurement, etc. of SCRF cavities

# **Clean assembly and evacuation**



ISO Classification number	Maximu and larg	um concentrat ger than the co	tion limit onsidered	s (parti l sizes s	cles/m³ of a hown belo	iir) for j w	particles	s equ	al to
	>=0.1µm	n>=0.2µm	>=0.3µm	>=	0.5µm	>=1µn	n >	>=5.0	μm
ISO Class 1	10	2							
ISO Class 2	100	24	10 4						
ISO Class 3	1 000	237	102	35		8			
ISO Class 4	10 000	2 370	1 020	35	2	83			
ISO Class 5	100 000	23 700	10 200	3 5	3 520		2	29	
ISO Class 6	1 000 000	237 000	102 000	35	200	8 320	2	293	
ISO Class 7				35	2 000	83 200	) 2	2 930	
ISO Class 8				3 5	520 000	832 00	00 2	29 30	0
ISO Class 9				35	200 000	8 320	000	293 00	00
ISO 14644-1 Classes FS 209 Classes	Class 3 Class1	Class 4 Class 10	Cla ) Cla	ass 5 ass 100	Class 6 Class 10	C 100 C	Class 7 Class 10,	000	Class 8 Class 100, 000
		Cavity assembl	CI ly f	eanroo for SR	om F				-

# **Clean assembly and evacuation**



- The processed, clean and dried cavity is assembled with RF couplers, vacuum plumbing, burst disc, etc. in a Class 10 (ISO 4) cleanroom facility.
- The cavity is evacuated and leak tested to prepare it for low power RF performance testing in Vertical Test Stand facility.



Cleanroom assembly of 650 MHz cavity at RRCAT



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### Low-temperature baking



- After the assembly in cleanroom, the cavity is baked at low temperature of 120 C for 48 hours.
- This is the final processing step for the bare cavity before vertical test
- A high vacuum is maintained inside the cavity during the entire baking process.





120 C Hot Air Baking Facility at RRCAT

### Low-temperature baking





Hot nitrogen baking facility (JLAB)



Infrared heaters heating (Scalay)



Baking stations (DESY)



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### **VTS testing**



- A Vertical Test Stand (VTS) is a facility to qualify a SRF cavity for its required performance by measuring *quality factor* and cavity *accelerating gradient* at 2K. After qualifying the test, cavity is dressed for cryomodule assembly.
- A vertical test facility has been set up at RRCAT. The VTS cryostat has been designed for performance testing of variety of SRF cavities at 2K, which may include 325 MHz Spoke resonators, 650 MHz and 1.3 GHz multi-cell SRF cavities for proton and electron accelerators.
- The cryostat is magnetically shielded by two layers of magnetic shields to restrict the magnetic field to less than 1  $\mu$ T at cavity, i.e.
  - External magnetic shield (room-temperature) ,
  - Internal magnetic shield (2K)
- Fabrication of Cryostat was carried out in strict accordance with ASME B&PV code under joint supervision of engineers from RRCAT and Fermilab.

### **Vertical Test Stand (VTS) Facility**



VTS can test following cavities in single cool-down

- ✓ 6 Nine-cell 1.3 GHz cavities or
- 2 Five-cell 650 MHz cavities
- ✓ 2 325 MHz single spoke resonators



Overall outside diameter of Cryostat	1370 mm			
Overall height of Cryostat	5420 mm			
Clear aperture of the Helium Vessel	864 mm			
Height of liquid Helium Vessel	4860 mm			
80K Shield is in between He Vessel & Vacuum Vessel				
Maximum LHe capacity - 2900 liters				



# Processing and testing of 650 MHz beta=0.92 single-cell SCRF cavity





Centrifugal barrel polishing (~ 200 micron)



Cavity loading in high temperature furnace for hydrogen degassing (@ 600 °C for 10 hrs)



Electropolishing of cavity (~ 20 micron)



Low temperature baking (@120°C for 48 hours)



High pressure rinsing (@ 100 bars pressure)



Cavity loaded on VTS insert assembly for 2 K testing)

### VTS Testing of 650 MHz single-cell cavity at RRCAT







 $Q = 3 \times 10^{10}$  $E_{acc} = 12.8 \text{ MV/m}$ 

VTS Insert assembly



- ✓ He-vessel welding
- ✓ Degreasing
- Final material removal (10-20 μm)
- ✓ Final HPR
- ✓ Horizontal assembly into cavity-string
- Evacuation of cavity string

