Tutorial on Cavity Preparation
SRF 2007 Workshop Beijing

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Subtitle how to come from a Niobium Cavity
Some background information to understand what is the difference?

1) Surface preparation methods

2) Infrastructure for Cavity preparation

3) Handling and Know how on cavity preparation
1) Surface preparation methods
Virtual micro cut of a Niobium sheet

Niob purity  99.9999999

“Damage layer”
~100 µm thick
Removal of “damage” layer

Methods

Mechanically by
- Grinding
- barrel polishing (tumbling)

Chemically by
- chemical etching (buffered chemical polishing [BCP])
- Chemical polishing (electro polishing [EP])
- + + different new solution under development
For instance
see EU JARY1 program WP 5
Accel / Poligrad
Electro bright
Mechanical removal of damage layer

Grinding

+ Simple handling

+ low cost standard mechanic

+ Mostly in use for removal of local defects non uniform abrasion!

- Abrasives need to be qualified on s.c. Cavties!!

- Remain of C; Si; glue; scratch size

- Produces a new damage layer of about 40 µm thickness!!
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**Tumbling**

Material: “Stones” made in different shape and material

Application: Global
Effect: Smoothening and removal of local enhancement (Sparcs from EB welding weld in area)

Removal: Non uniform contact pressure ➔

For optimum removal you need to design machines that make use of centrifugal forces to uniform the forces (Complicated design)

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Example of a tumbling machine
Designed and manufactured by DESY group MPL Waldemar Singer
Chemical removal of “demage” layer
Two types chemical removal are most commonly in use

**BCP**
*Buffered chemical polishing*
(Mixture of Hydrofluoric acid; Nitric acid and Phosphoric acid)

**EP**
*Electropolishing*
(Mixture of Hydrofluoric acid and Sulfuric acid)
Receipt:

**BCP Acid**
Mixed by volume from
1:1 HF(49%) /HNO3(70%)
to (1:1:2 HF(49%):HNO3(70%) :H3PO485%)
removal rate
1:1 at 20C >20 µm/min Removal Rate
1:1:2 t 20 C 1µm/min

**Mixture is self exiting ! Spontaneous reaction with Nb!!**

**EP Acid**
Mixed by volume from
1:8   HF(45%) /H2SO4 (96%)
to 1:10 (HF( 45%)/H2SO4 (96%)
(+ H2O due to hygroscopic reaction of H2SO4!)
removal rate with 17 V applied
1:9 at 20C 0,3-0,5 µm/min
1:10 a t 20 C 0,3 – 0,4 µm/min

**No reaction on Niobium without voltage applied!**
Removal of niobium by chemical reactions

\[
\text{Niob Oxid Nb}_2\text{O}_5 + \text{XXX} \\
\text{Niob purity 99.9999999}
\]

\[
\text{Niob Oxid Nb}_2\text{O}_5 + \text{XXX} \rightarrow \text{Nb}_2\text{O}_5 + \text{HF} \rightarrow \text{H}_2\text{NbOF}_2 + \text{NbO}_2\text{F}
\]

\[
\text{Niob Purity 99.9999999}
\]

\[
\text{Oxidizer} \quad \text{[H}_2\text{SO}_4 + \text{voltage} = \text{EP}] \text{ or } [\text{HNO}_3 = \text{BCP}]
\]

\[
\text{Niob Oxid Nb}_2\text{O}_5 \\
\text{Niob Reinst 99.9999999}
\]

\[
\text{Niob Oxid Nb}_2\text{O}_5
\]
Chemistry

**BCP (buffered chemical polishing)**
**Mixture by volume** 1/1/2 HF/HNO3/H3PO4

\[
2 \text{Nb} + 5 \text{NO}_{\frac{1}{3}} \Rightarrow \text{Nb}_2\text{O}_5 + 5 \text{NO} + 5 e^{-}
\]

\[
\text{Nb}_2\text{O}_5 + 6 \text{HF} \Rightarrow \text{H}_2\text{NbOF}_5 (l) + \text{NbOF}_2 + 0.5 \text{H}_2\text{O} (\text{unl.}) + 1.5 \text{H}_2\text{O}
\]

**EP (electrochemical polishing)**
**Mixture by volume** 1/9 HF/H2SO4

\[
2 \text{Nb} + 5 \text{SO}_{4}^{2-} + 5 \text{H}_2\text{O} \Rightarrow \text{Nb}_2\text{O}_5 + 10 \text{H}^+ + 5 \text{SO}_{4}^{2-} + 10 e^{-}
\]

\[
\text{Nb}_2\text{O}_5 + 6\text{HF} \Rightarrow \text{H}_2\text{NbOF}_5 (l) + \text{NbOF}_2 + 0.5\text{H}_2\text{O} (\text{unl.}) + 1.5\text{H}_2\text{O}
\]

\[
\text{NbOF}_2 + 0.5\text{H}_2\text{O} + 4\text{HF} \Rightarrow \text{H}_2\text{NbOF}_5 + 1.5\text{H}_2\text{O}
\]
2) Infrastructure for Cavity Preparation

Super conduction surface will be covered by normal conducting material after standard wet surface treatments (residues from chemical reaction and particulates)

- Large areas of n. c. Material reduce the Q value
- Particulates are origin of field emission

› Need of particle and residue free
  › surface cleaning; Ultra pure water (UPW)
  › particle free storage and working atmosphere (Cleanroom)
  › and particle free handling adapted tools and processes
Particulates on surfaces

Are there

Typical materials?
Non!
All air born substances of the world perform particulates

Typical size?
No!
Every size from ideal ball geometry to multi complex geometries

Common behavior?
Yes!
transportation in air make them flow everywhere
well known forces making them stick on surfaces

Other sources of particles and particle motion

Mechanical vibration

** Hitting, banging, “tapping”
(tightening bolts with wrench!!!)
** Closing a valve quickly

Thermal cycling.
One cannot avoid cycling to 2 K!
Particles can be dislodged by thermal stress differences.
Example:
Particles found in Tristan reactor:
Environment (rock, concrete, paint)
Aluminum alloys (6063, 2219)
Pumps: Ion—TiO, TiN; NEG—ZrO
Forces that make attractive potential to particulates facing a surface

Coulomb forces

\[ F = \frac{g^2}{16 \pi \varepsilon_0 h^2} \]

Van der Waals forces

\[ F \sim \frac{d}{Z_0^2} \]

Electrostatic forces

\[ F \sim \frac{d (\Phi_{W1} - \Phi_{W2})^2}{Z_0} \]

Knudsen forces

\[ F \sim d \]
How to introduce forces (energy) to the particles on a surface?

- Ultrasonic cleaning
- Discharge of static loads ➔ Ionized Gases (air N2)
- Reduce surface tension ➔ Alcohol / Detergents
- Rinsing ➔ High Pressure Rinsing
- Enforced gas flow ➔ air guns (N2 ; Ar)

Problem speed on surface = 0
oder aufgrund der Distanz von der Plattenkante \( x \)

\[
Re_x = \frac{U_0 x}{v}
\]

(7.16b)

Abb. 7.5: Grenzschichtverhalten entlang einer dünnen Platte ohne Druckgradienten, \( dp/dx = 0 \).
But what to do when particulates already rest on a superconduction surface? Apply forces larger than the active ones (static; v.d. Waals etc.) to remove them.
Surface cleaning with sound waves in ultra pure water
Preparation of flange surface:

a) - 20 Min US cleaning within DESY US basin
   - Automatic rinsing with DESY UPW rinsing basin
     (R >= 12 MOhmcm)
   - Drying in class 100

b) - cleaning with hand held megasonic cell®₂ at optimized
distance of 20 cm between Object and Cell (Nozzle)
   Drying in class 100

c) - 20 Min US cleaning within DESY US basin
   - Automatic rinsing with DESY UPW rinsing basin
     (R >= 12 MOhmcm)
   - Drying in class 100
   Blowing of surface by ionized nitrogen of the pulsed gun
Surface scanning to measure particle concentrations on surfaces
Studies on most efficient cleaning

Counts

Particle size [µm]

0.3 µm  0.5 µm  1.0 µm  3.0 µm  5.0 µm  10.0 µm

- ultrasonic cleaning/fine rinsing
- SONOSYS Megasonic
- blown off with nitrogen

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### Particulates in the air

Which particles do we find in our normal air?

<table>
<thead>
<tr>
<th>Particle Size [µm]</th>
<th>Particulates</th>
</tr>
</thead>
<tbody>
<tr>
<td>0,01</td>
<td>Dust, Oil fume, Ash, Smoke of tobacco, Smoke of metal, Gas molecule, Dust of color paint, Soot particle</td>
</tr>
<tr>
<td>0,1</td>
<td>Smoke, Hair</td>
</tr>
<tr>
<td>1</td>
<td>Soot particle, Spore, Pollen</td>
</tr>
<tr>
<td>10</td>
<td>Viruses, Bacteria</td>
</tr>
<tr>
<td>100</td>
<td>Aerosol of sniffing</td>
</tr>
<tr>
<td>1000</td>
<td>Hair</td>
</tr>
</tbody>
</table>

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Particulates in air

Gravitational forces

\[ F = ma \sim d^3 \]

Flow induced forces

\[ F \sim V A_0 \rho c_w \]

Diameter d

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Example from SRF workshop 2007
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Overview talk of John O'Hanlon
vacuum@dakotacom.net

Settling velocity for particles
Gas: Air
Temperature = room temperature
Electric fields: non
airflow: not enforced (normal air)
For super conduction cavities clean water and cleanroom technology is required to prevent air born particulates from settlement on surfaces.

Basics of cleanroom technology

- Media: Gas / liquid
- Filter membrane
- Cleaned media
- Object in clean environment
- Boarder to normal areas
- Exit of media (used for process control)
Basic function of a cleanroom

Fresh air

Fan units

Hepa Filter

Hepa filter ::

Advanced System recirculate 90% of the filtered air

Air velocity 0.35 – 0.5 m/sec
Air flow laminar from cl 100 on

Perforated cleanroom floor

Exit of air for air exchange

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One major part inside a cleanroom is PERSONAL

1st Dress code
3) Handling and know how on cavity preparation

Behavior of personal inside a cleanroom

Wrong !!!

Right !!!

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Open the door
But right!
Be careful where your hands and your body is close to an open cavity.
But from where do we know that all this technology works as designed?

**Quality control during cavity preparation**

1) Air; gasses; liquids
2) surfaces
3) Flow pattern
4) Degreasing and rinsing
Schematic of particle counters

Figure 1-1. Particle Counter Simplified Flow Diagram

Air; gases; liquids
Some example on particle detection in ultra pure water

Particles found after installation of a new filter to the HPR stand, total sampling time 16 hours

Particle concentration of the HPR filter after 72 h of rinsing

Air; gasses; liquids
<table>
<thead>
<tr>
<th>unit</th>
<th>hardness [Deutsche Härte]</th>
<th>Restitvity [Mohm cm]</th>
<th>Bacteria colonies [per liter]</th>
<th>TOC [ppb]</th>
<th>Particulates [Counts &gt;0,3 µm/ liter]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tap water</td>
<td>7-10</td>
<td>&lt;= 0,0001</td>
<td>&gt;= 100 ( @DESY tap connection )</td>
<td>Not defined</td>
<td>Not defined</td>
</tr>
<tr>
<td>Decalcification of tap water</td>
<td>&lt;=1</td>
<td>&lt;= 0,0001</td>
<td>50-100</td>
<td>&lt;10</td>
<td>Not defined</td>
</tr>
<tr>
<td>Reverse osmosis</td>
<td>&lt;=1</td>
<td>R &lt;= 0,2</td>
<td>50-100</td>
<td>&lt; 10</td>
<td>Not defined</td>
</tr>
<tr>
<td>UV light</td>
<td>&lt;=1</td>
<td>R = 18,2</td>
<td>1-5</td>
<td>1-3</td>
<td>Behind filter 20- 100</td>
</tr>
<tr>
<td>Ion exchanger / Polisher</td>
<td>&lt;=1</td>
<td>R = 18,2</td>
<td>1-5</td>
<td>&lt;10</td>
<td>Behind filter 20-100</td>
</tr>
<tr>
<td>UV light</td>
<td>&lt;=1</td>
<td>R = 18,2</td>
<td>1-5</td>
<td>1-3</td>
<td>Behind filter 20-100</td>
</tr>
<tr>
<td>Point of use filtration</td>
<td>&lt;=1</td>
<td>R = 18,2</td>
<td>1-5</td>
<td>1-3</td>
<td>&lt;=10</td>
</tr>
</tbody>
</table>
Particle detection on rinsing water of the HPR system

HPR ejection nozzles and QC funnel

Particle filter $\leq 2 \, \mu m$ in HPR draining line

Air; gasses; liquids
Scanning microscope for particle filter counting and visual analysis
Monitoring of the total oxydable carbon (TOC) in ultra pure water

Air; gasses; liquids
TOC-Meßwerte vor RWA Shut down am 20. Januar 2005, ca. 14:00

Größe des Meßwertes [ppb TOC]

Anzeigedatum des Meßgerätes

Example on bacteria contamination in one of the two reverse osmosis units of the cleanwater plant

Air; gases; liquids
Quality control on particulates removed from a surface by ionized air (top gun)

QC of studs before installation to a cavity

QC of washers before installation to a cavity

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Quality control on particle concentration on surfaces

Air particle counter modified for measurement on surfaces

Particle detection on a cleanroom wall

Particle detection on a cleanroom overall
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Particle detection on a cleanroom table
Instrument: modified Air Particle Counter
Add on surface instrumentation: designed by CCI von Kahlden GmbH.

**Definition of particle density on surface**

Particle density:
- **Static application**: \( P = \frac{C}{A} \) [counts per cm\(^2\)]
- **Dynamic application**:
  \[ P = \frac{C}{Ax} \]

Scanned surface:
- \( Ax = A_1 + A_2 \)
- \( A_1 = \frac{(\pi d^2)}{4} \) [mm\(^2\)]
- \( A_2 = W \times d \) [mm\(^2\)]

Sampler speed:
- \( V = \frac{W}{t_1} \)

Setting of counter:
- Sampling time \( t \) [sec]

**Dynamic application**:
- \( P = \frac{Ct}{(V \times t_1 \times d + 1964)} \)

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Particle concentration on:
Table = 67,3 [Particles/mm²]
Wall = 11,5 [Particles/mm²]
Cleanroom overall = 17,4 [Particles/mm²]

related numbers of particle density

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Flow pattern
Degreasing and Rinsing

Test on cleaning procedure/ detergent
Nb sample polluted with grease and oil

Not efficient cleaning

After Ultrasonic cleaning with sufficient detergent and procedure

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New method to control the High pressure rinsing jet (INFN Milano)
One example of Cavity preparation
Preparation of superconducting cavities at DESY

Cavies arrive from Industry and have undergone in comming inspection
After that
A) Main EP
   1) Degreasing and rinsing
   2) Prepare for EP
   3) EP treatment
   4) Clean cavity and rinse for cleanroom
   5) out side etching for 800 C annealing
   6) 800 C annealing
   7) Tuning for vertical test
Ultra sonic rinsing (up to 4KW Power)

Rinsing by ultra pure water

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Degreasing and rinsing
Degreasing and rinsing
Cavity installed in electro polishing apparatus

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1) EP treatment
Ep apparatus during draining of acid

EP treatment
Tuning for vertical test
After that

B) Preparation for vertical test at 2K

1) “Car wash”
2) Degreasing and rinsing
2a) Prepare for EP and **EP** treatment
2b) or **BCP** treatment
4) Rinsing and 1st high pressure rinsing
5) Drying in class 10 (ASTM)
6) Assembly of accessories
7) Alcohol rinsing
8) Six times high pressure rinsing
9) Drying in class 10 (ASTM)
10) Assembly of test antenna
11) 120 C baking
vertical test
Prepare for EP and EP treatment
Cavity installed in BCP bench
(Buffered chemical polishing
Valve box for BCP treatment

or BCP treatment
View into class 10000 cleanroom
“Wet preparation area”
Assembly of accessories
Alcohol rinsing
Six times high pressure rinsing

HPR Stand inside the cleaner room
If ever things!!!! works perfect you will get
From vertical test to module
(Shortened version there are more steps than shown here necessary)

Back to clean room
Install FEM (in situ bead pull measurement)
Tank welding
Remove FEM
Install Antennas
High pressure rinse cavity
Install power coupler
Assemble of module
1) Cavity assembly for vessel weld

Requirements to the FMS:
- Cleanroom class ISO 4 (ASTM: RK10)
- Protect inner surface of cavity from ambient air
- No desorption from FMS (Teflon tube and clamping system)
- Vacuum tightness
谢谢大家

会议愉快