

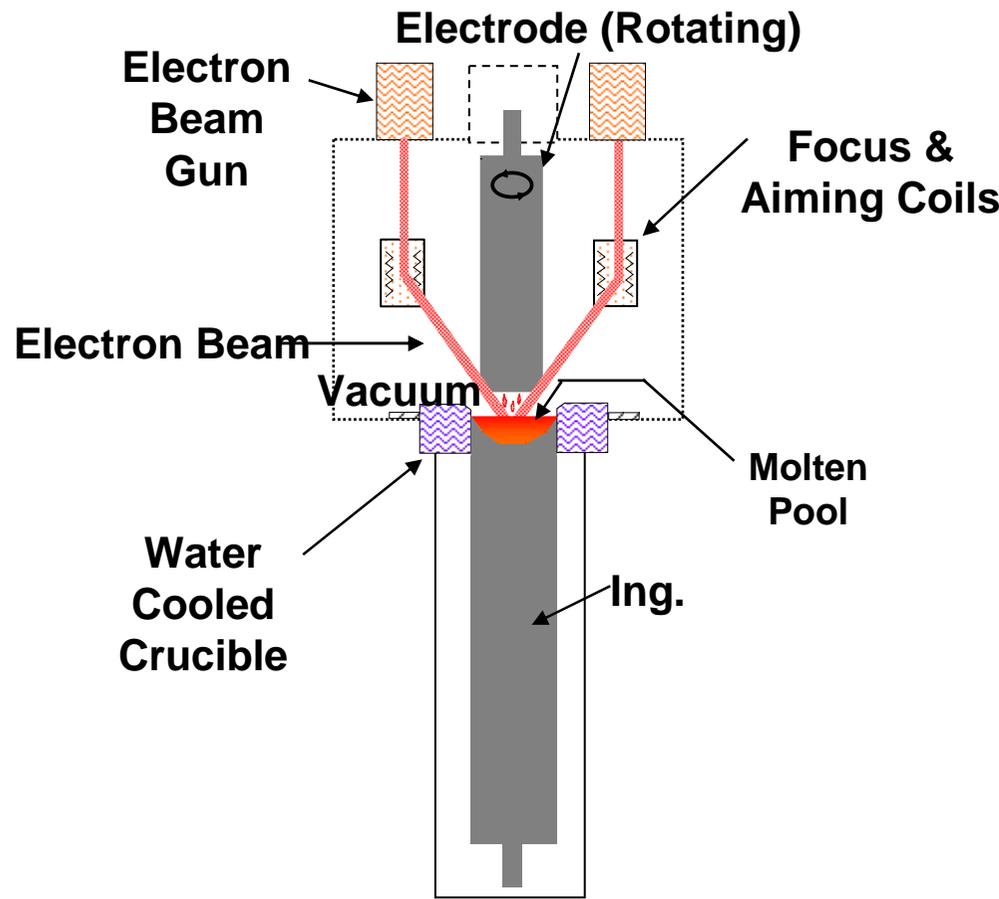
SC Cavities; Material, Fabrication and QA

W. Singer
DESY

Material

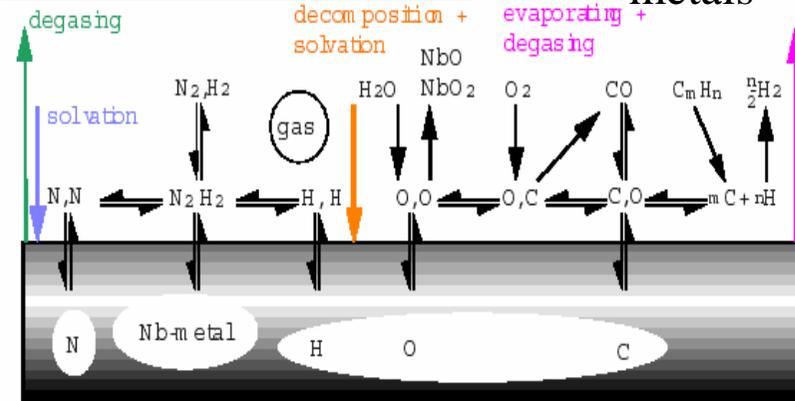
- **Niobium (Nb)** ($T_c=9.2$ K; superheating field of approx. 240 mT) is the **favorite material** for the fabrication of superconducting RF cavities.
- chemically inert (pentoxide layer)
- easily machined and deep drawn
- available as bulk and sheet material
- majority of s.c. RF cavities worldwide are formed from **Nb sheet material**

Mass production of high purity Nb for RF cavities



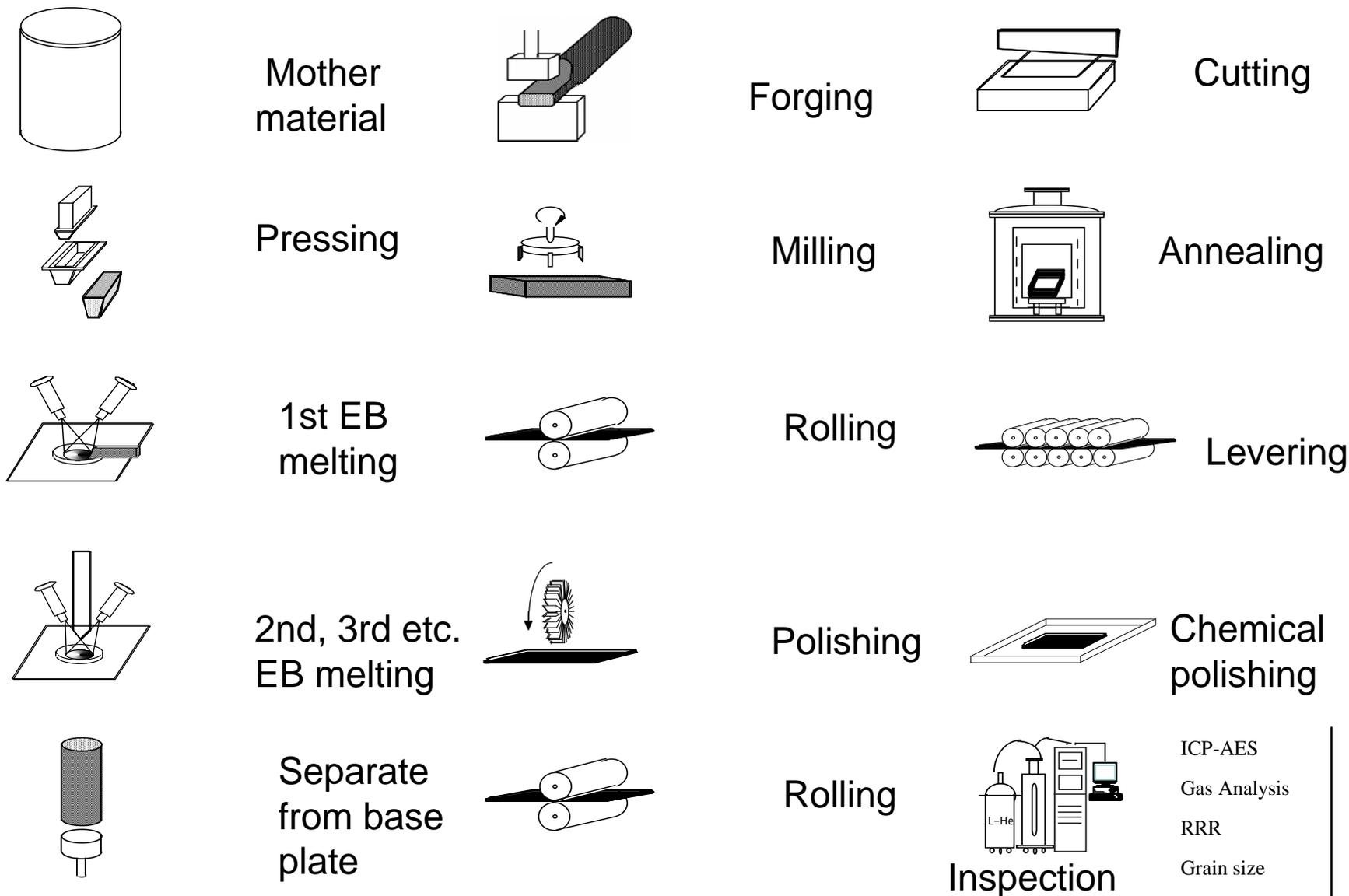
EB furnace for melting of refractory metals

Electron Beam melting of Nb



The melting temperature is a compromise between the maximization of purification and minimization of the material losses by evaporation.

Fabrication of Nb sheets at Tokyo Denkai



- ICP-AES
- Gas Analysis
- RRR
- Grain size
- Hardness
- Tensile tests

In the final sheet the purity of niobium should be not inferior as in the ingot

TD equipment for the quality control of high purity niobium

RRR measurement



東京電解 (株)



東京電解 (株)

Tensile test

Gas analysis



Gas analysis (Hydrogen, Oxygen, Nitrogen) : HORIBA



Could you please test the sugar content in my coffee

Technical Specification to Niobium Sheets for XFEL Cavities.

Concentration of impurities in ppm (weight)				Mechanical properties	
Ta	≤ 500	H	≤ 2	RRR	≥ 300
W	≤ 70	N	≤ 10	Grain size	$\approx 50 \mu\text{m}$
Ti	≤ 50	O	≤ 10	Yield strength, $\sigma_{0,2}$	$50 < \sigma_{0,2} < 100$ N/mm² (Mpa)
Fe	≤ 30	C	≤ 10	Tensile strength	$> 100 \text{ N/mm}^2$ (Mpa)
Mo	≤ 50			Elongation at break	30 %
Ni	≤ 30			Vickers hardness HV 10	≤ 60

No texture: The difference in mechanical properties (Rm, Rp0,2, AL30) orthogonal and parallel to main rolling direction < 20% (cross rolling).



EB melting of ingot
Depending on RRR Quality 4-6 cycles
HERAEUS



Ingot

Ingot
Diameter
90 - 280

Forging

Rectangular
bar
- 60 x 200 x
Length

Milling and
Sawing

Cleaning

Rolling to
rough plate

Cleaning

Heat
Treatment

Rough plate

Rolling to
intermediate size

Cleaning

Rolling to final
thickness

Cleaning

Final Heat
Treatment

Cutting to final
size

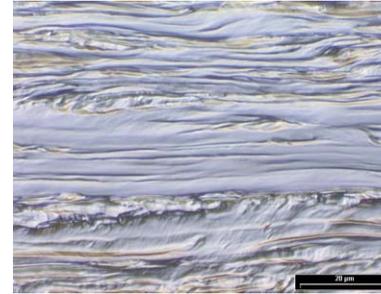
Finishing

Testing procedure

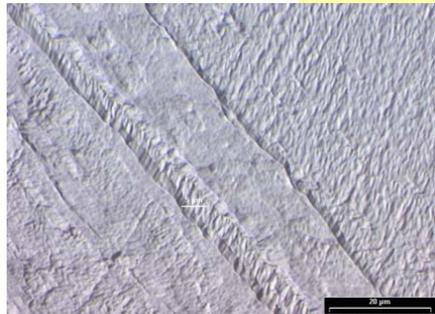
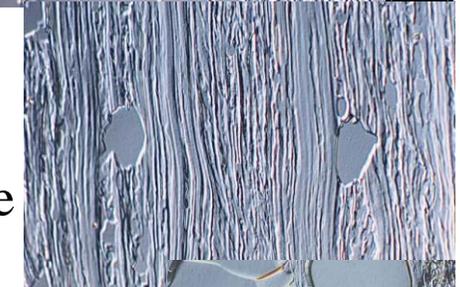
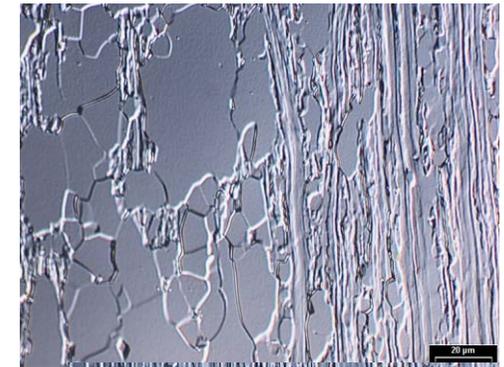
Sheet

Disc

Structure of Nb on different stages of sheet production



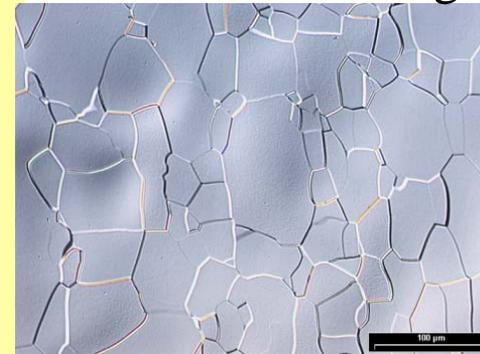
Rolled Nb sheet before final annealing



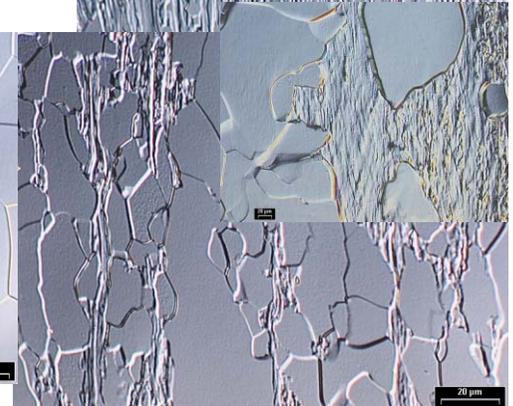
After forging



Rolled rough plate

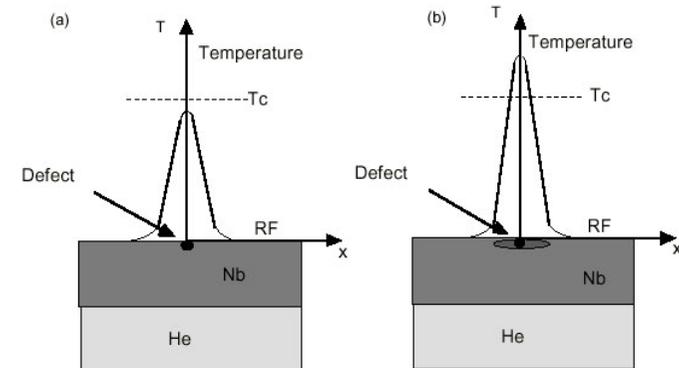
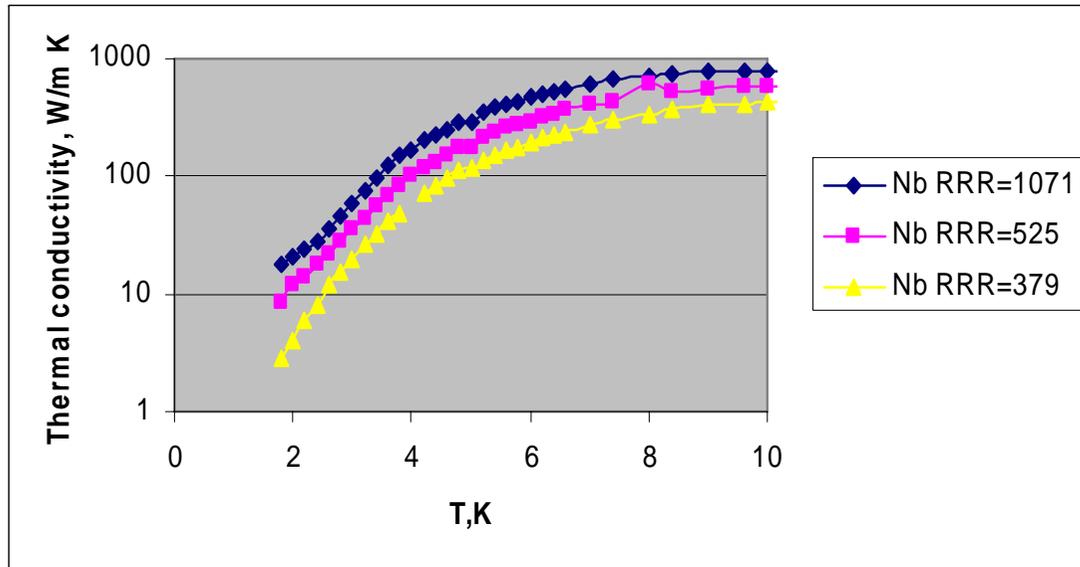


Recrystallized niobium sheet



Examples of bad recrystallization

Task: starting with cm size grain of ingot finish with ca. 50 μm uniform grain without contamination of Nb



Thermal conductivity of high purity Nb at low temperatures

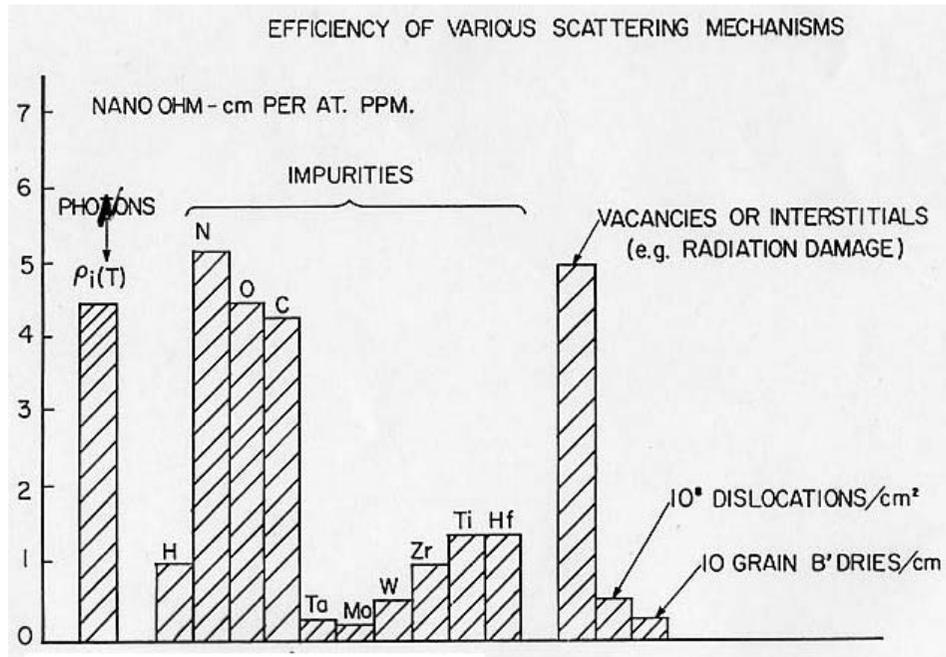
Rule of thumb

$$\lambda(4,2K) = C \cdot \left(\frac{W}{m \cdot K} \right) \cdot RRR$$

$$C \approx 0,25 \div 0,14$$

Normal conducting cluster triggers the quench, if the temperature exceeds T_c

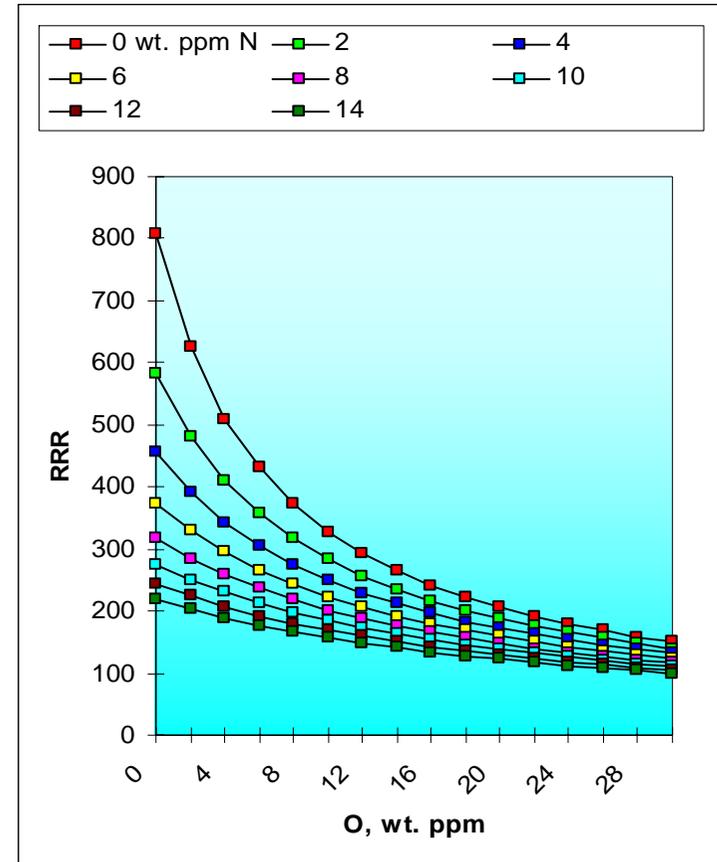
Influence of impurities on RRR



Contribution of different defects in the scattering mechanism (Schulze)

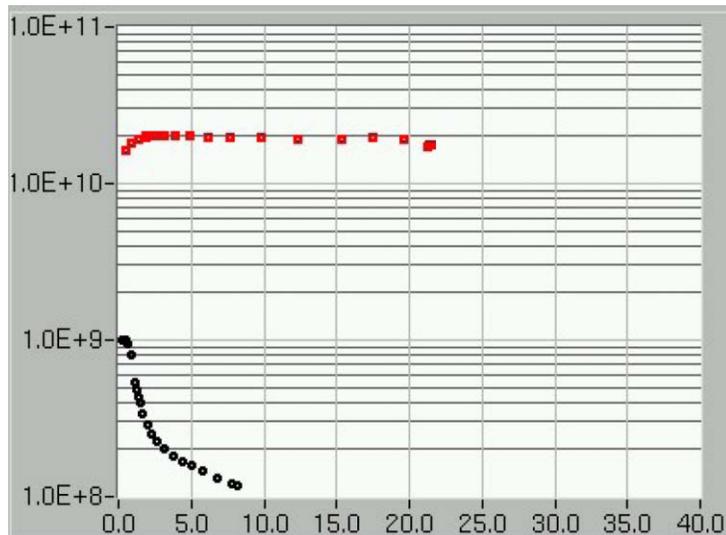
$$RRR = \frac{R(300K)}{R(10K) + \sum_{i=1}^4 \frac{\partial R_i}{\partial C_i} C_i}$$

$R(300K) = 1,46 \cdot 10^{-5} \Omega \text{ cm}$, $R(10K) = 8,7 \cdot 10^{-9} \Omega \text{ cm}$, $C = 1 \text{ wt. ppm}$

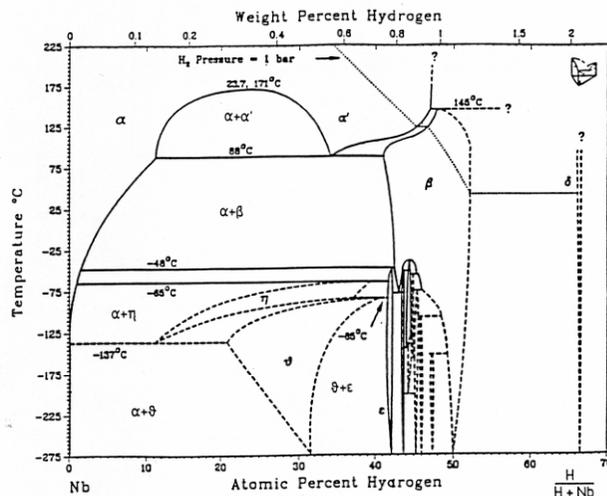


Calculated RRR versus oxygen and nitrogen content.

Hydrogen Q decease



Q(Eacc) of Single crystal cavity 1AC6 before and after 800° C annealing



NbH phase diagram

A slow cool down through 75 – 150 K caused the Qo disease. A fast cool down usually prevents the Qo disease

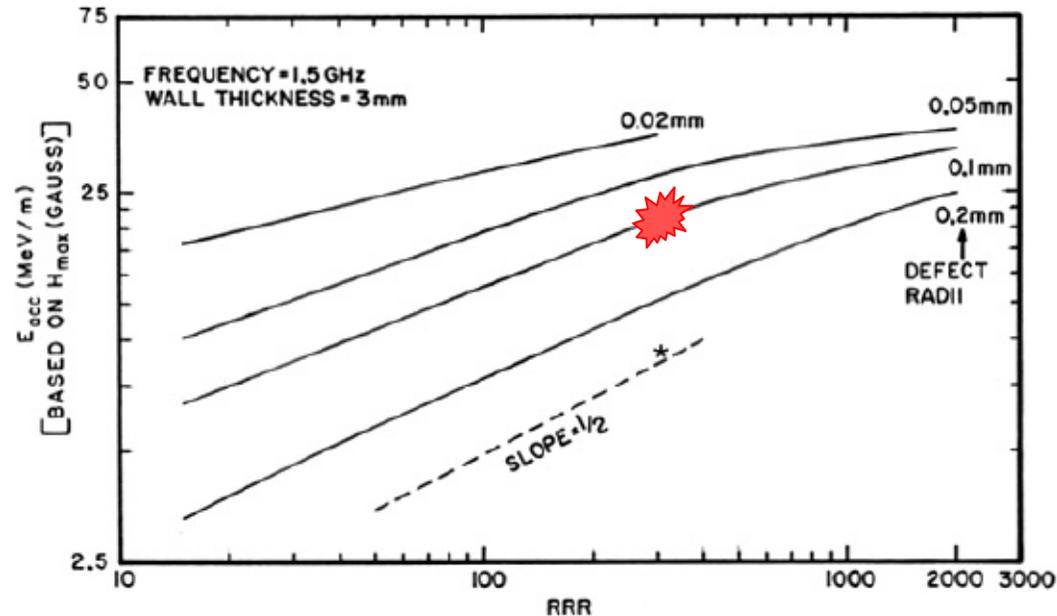
The longer the system is parked in the danger zone the worse the Q degradation
Warm up restored each time the maximal quality factor Qo

Reason: Hydrogen is very mobile at room temperature and even at 100K.

During cool down the former dissolved hydrogen creates the Nb hydrides at the surface and produces high RF losses. (Nb - hydride has $T_c=2,8$ K, $H_c= 60$ Oersted)

A fast cool down prevents hydride formation. At low temperature diffusion is slower and phase transition takes time

Quench field caused by local defect



Simulation of the thermal break down (Quench)

$$H_{tb} = \sqrt{\frac{4\kappa_T(T_c - T_b)}{r_d R_d}}$$

Scanning allow to avoid rough errors

κ_T : Thermal conductivity Nb

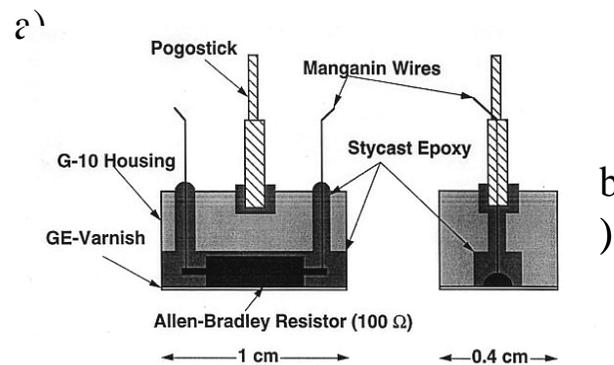
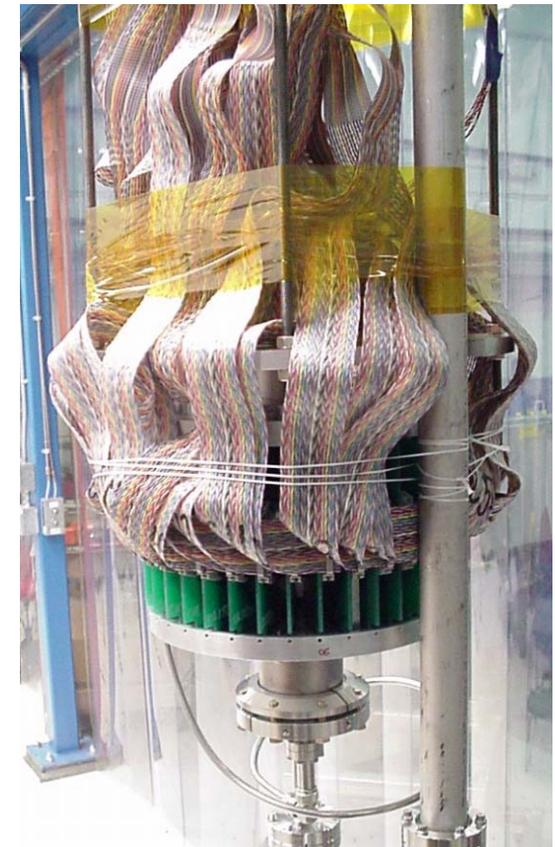
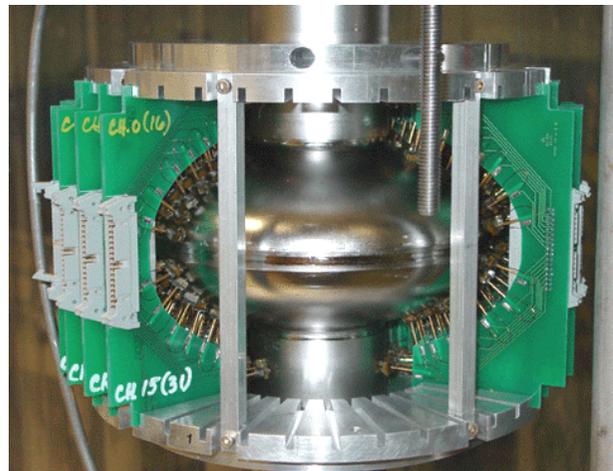
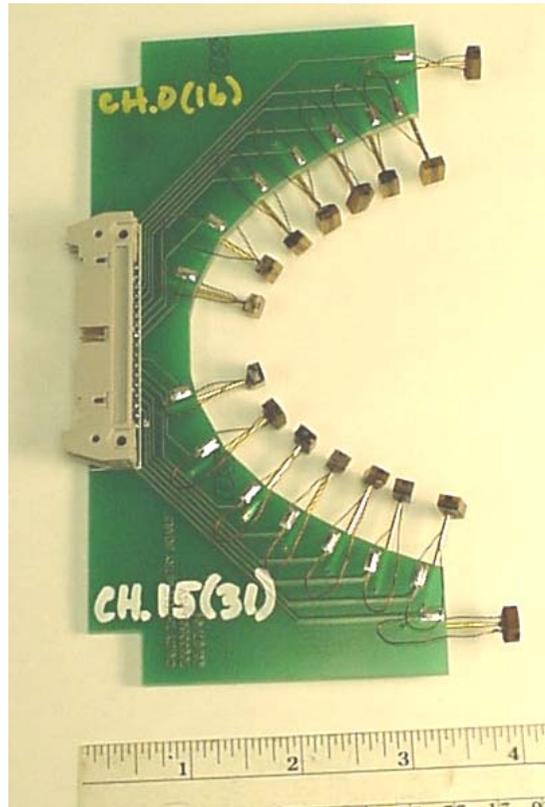
R_d : Surface resistance of the defect

T_c : Critical temperature of Nb

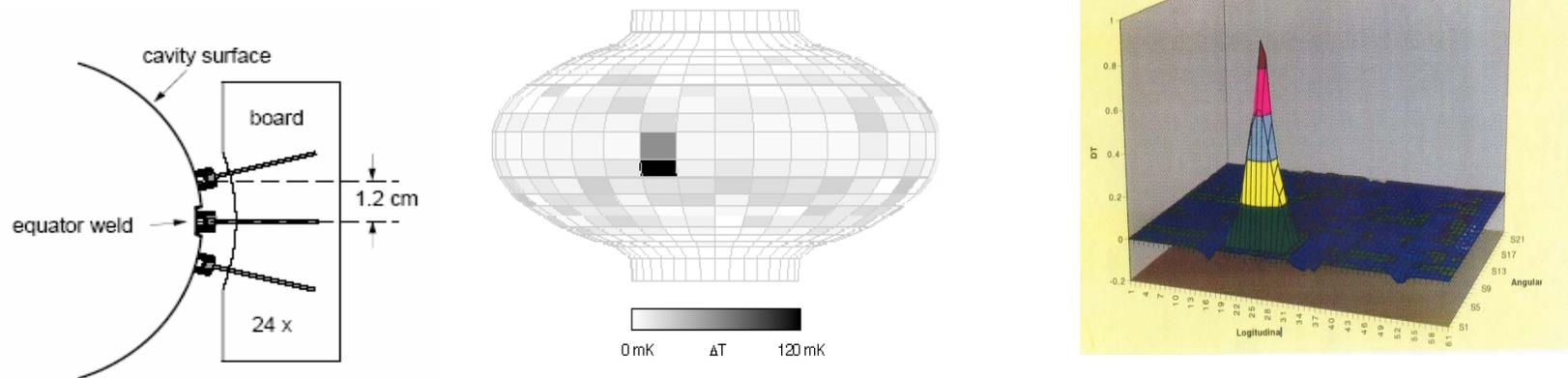
T_b : Bath temperature

r_d : Radius of defects

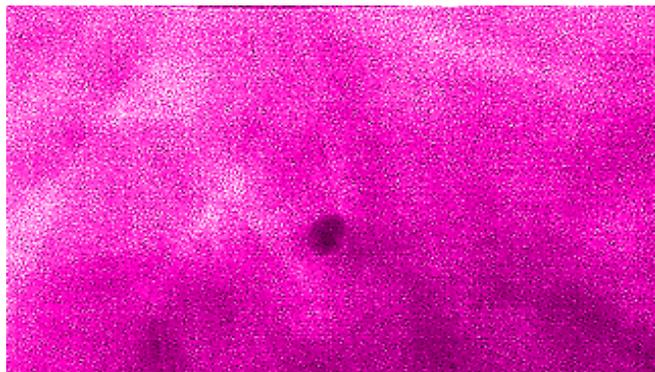
T-Mapping



Foreign material inclusions



Temperature mapping: Cavity D6 with $E_{acc}=13$ MV/m shows excessive heating at a localized spot

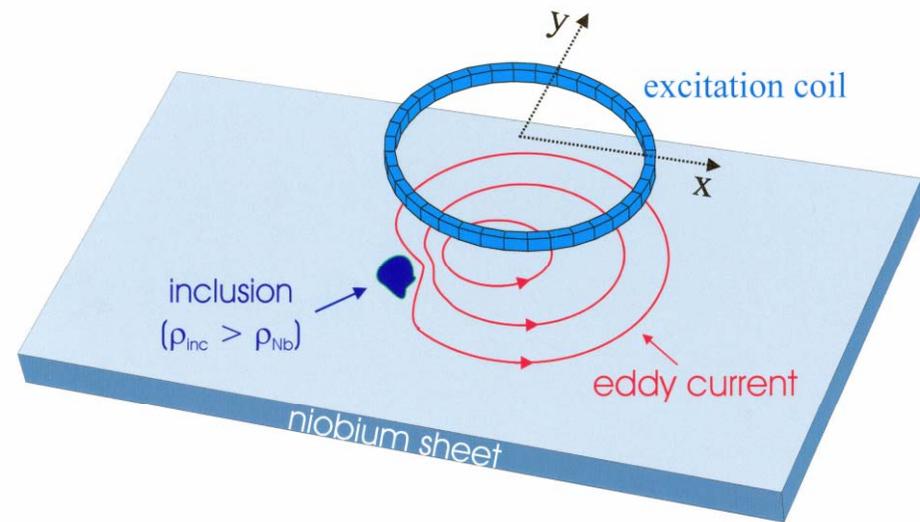


Positive print of a X-ray radiograph showing the “hot spot” as a dark point (0.2 mm large Ta inclusion).

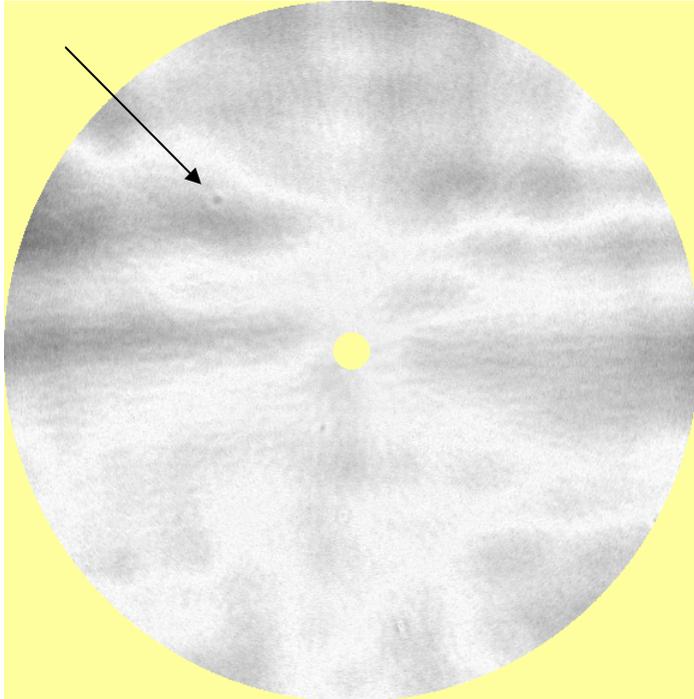
Search for clusters in Nb sheets.
Eddy current system.



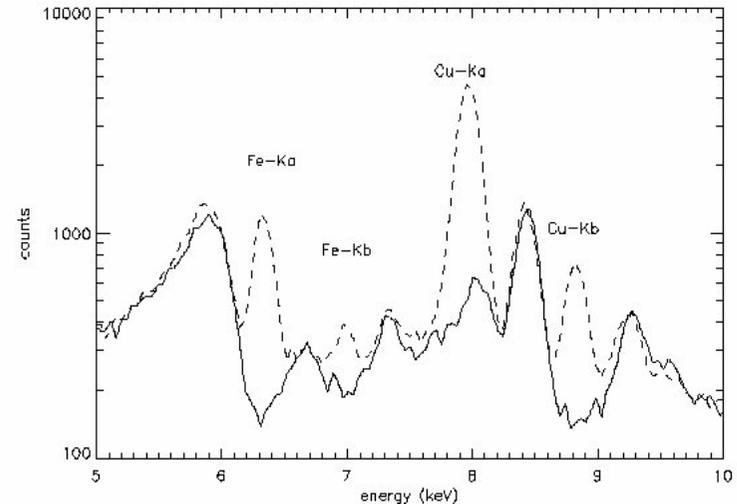
DESY eddy current scanning
apparatus for niobium discs.
100% Nb sheets for TTF
scanned and sorted out



Principle of eddy current
measurement



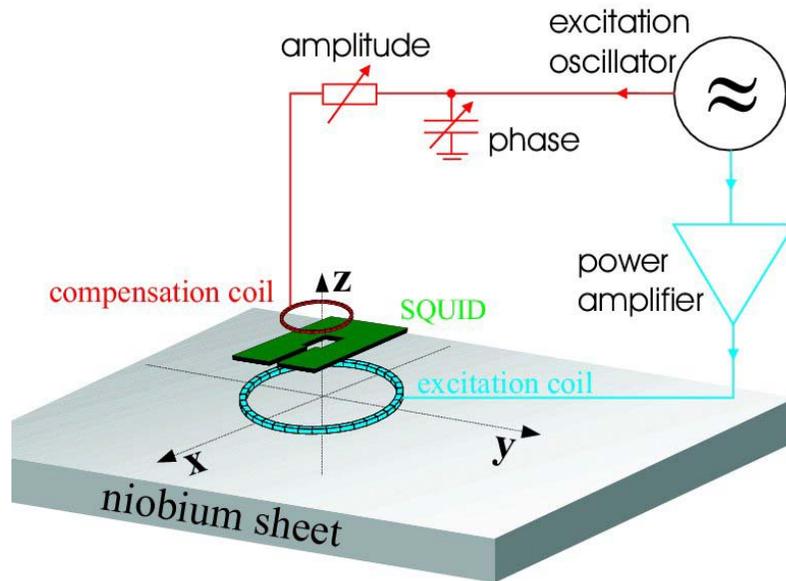
Example of the Nb sheet eddy current scanning test. Arrow indicates the suspicious spot.



SURFA (Synchrotron Radiation Fluorescence Analysis). Spectrum of K-lines at the spot area (dashed line) in comparison with spot free area (full line).

The spot was identified as an inclusion of foreign material. Cu and Fe signal has been observed in the SURFA spectrum in the spot area.

Development of SQUID based scanning system for testing of niobium sheets



An excitation coil produces eddy currents in the sample, whose magnetic field is detected by the SQUID.

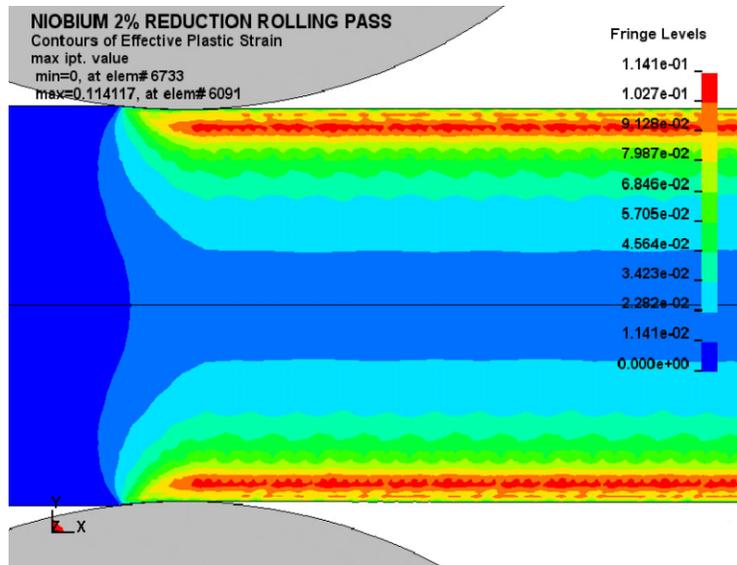
Prototype of SQUID based scanning system for niobium sheets (in work)

Final rolling

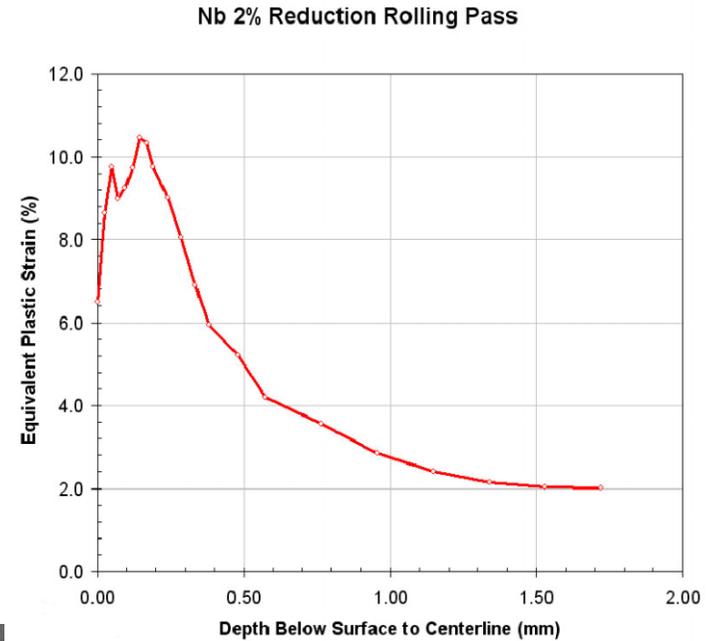


Example of the companies feed back : Tokyo Denkai improved the cleanness around of the rolling equipment

Damage layer by rolling (R. Crooks)

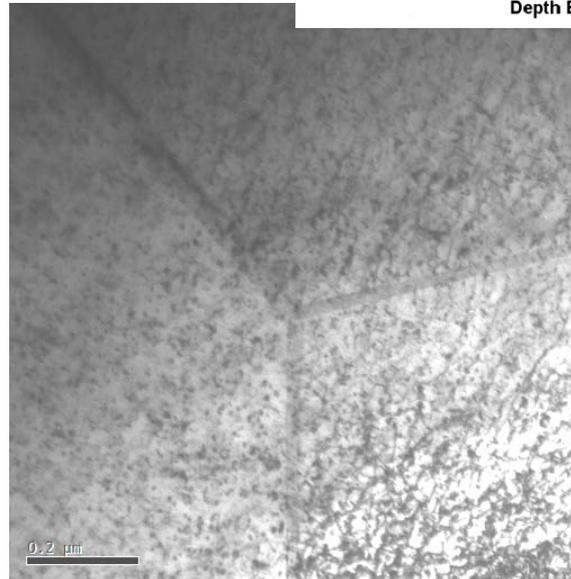


Finite element simulation of 2% reduction of 3.5 mm sheet with 1 cm diameter rolls. Strain is concentrated in the near-surface region



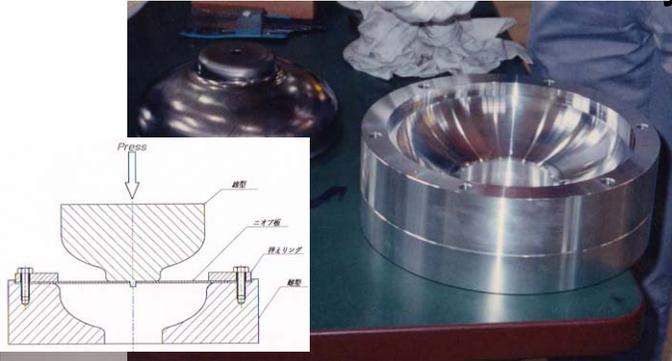
As-received RRR Nb Sheet,
20 μm below surface
(ion milled thin foil)

High dislocation density.



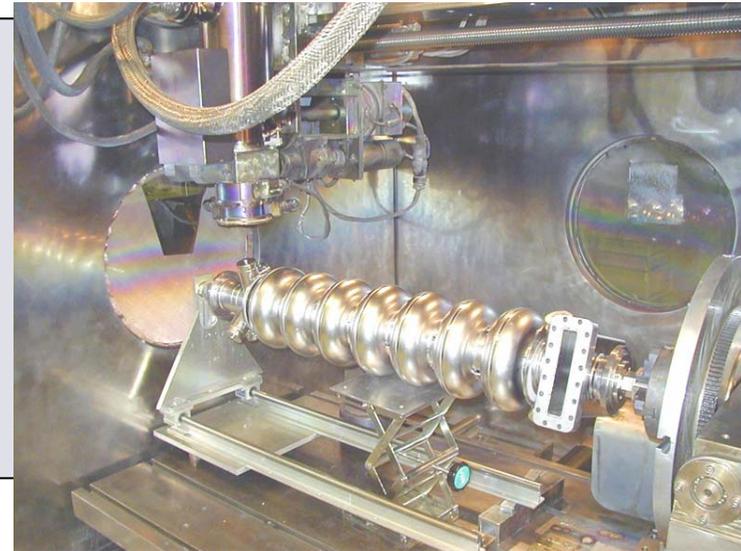
Transmission
electron
microscopy
image
(BFTEM)

Fabrication: Conventional fabrication (deep drawing and EB welding of fine grain Nb). Experiences of ca. 20 years of industrial cavity fabrication are available



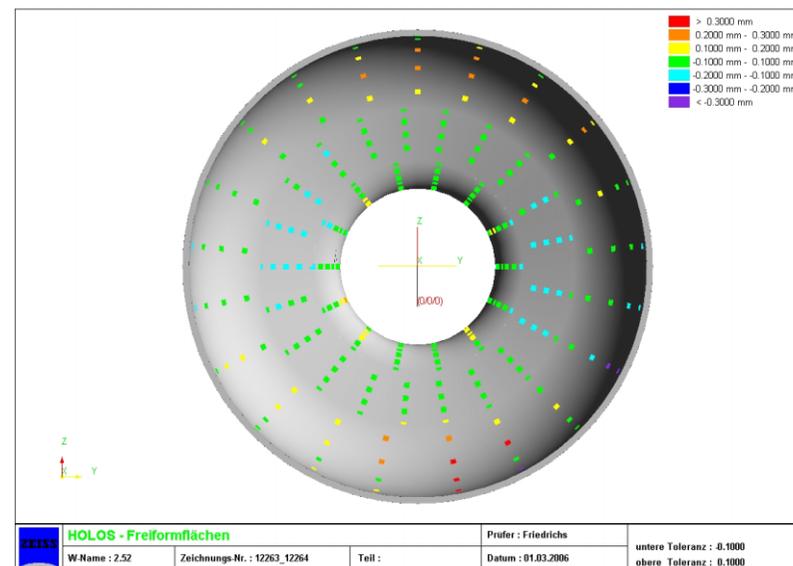
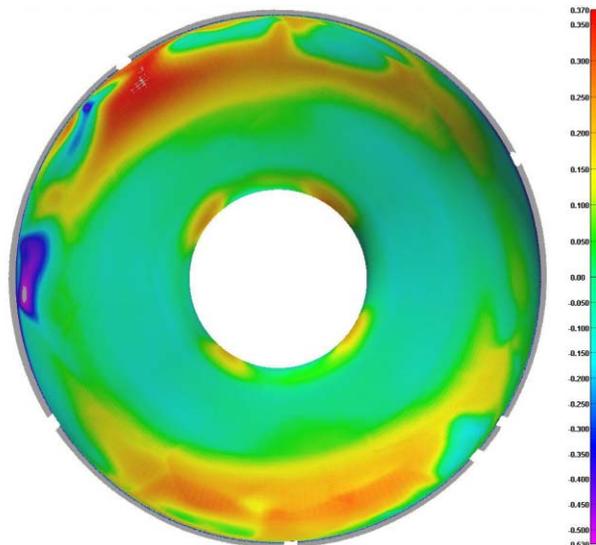
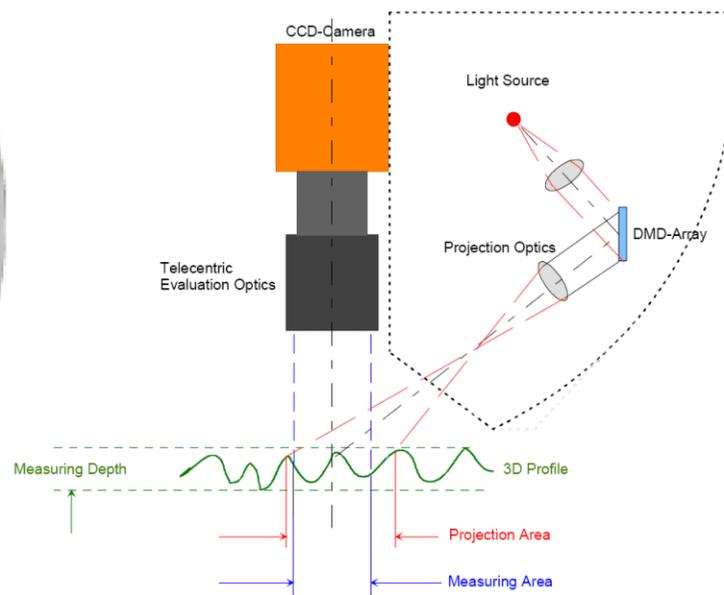
Half cells are produced by deep drawing.

Dumb bells are formed by electron beam welding.



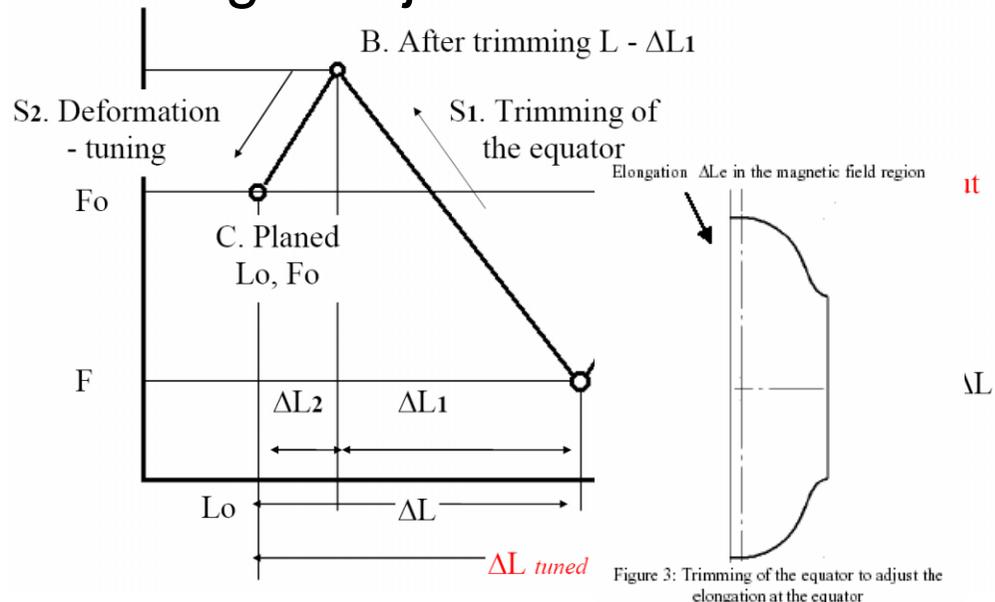
After proper cleaning eight dumb bells and two end group sections welded by electron beam together

Important: clean conditions on all steps
shape accuracy, preparation and EB welding

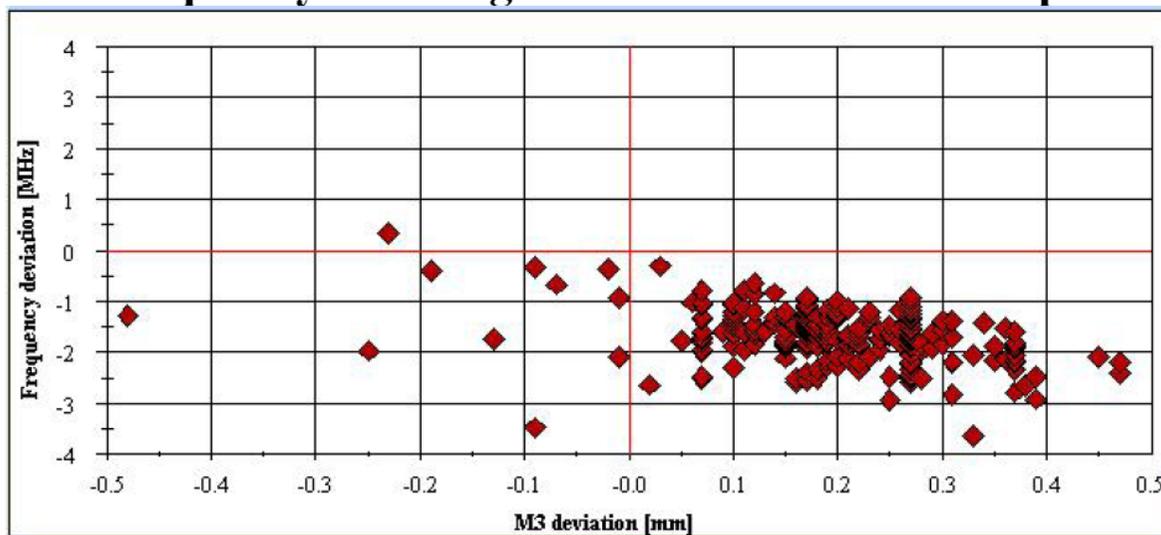


Optical and mechanical 3D measurement measurements of the HO52 half cell shape

Frequency and the length adjustment



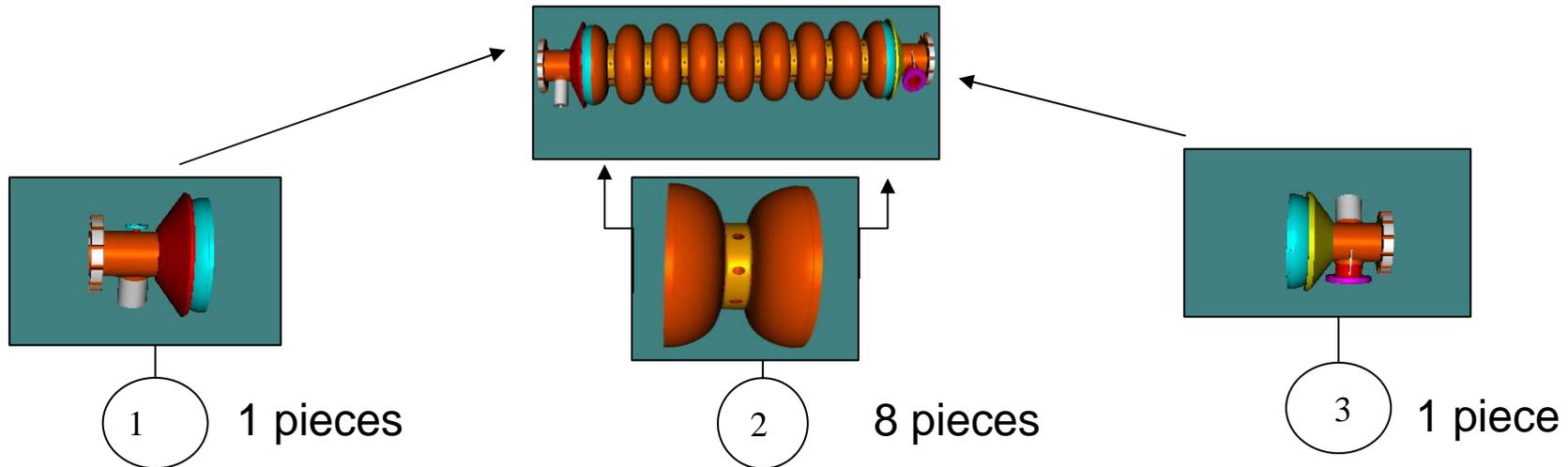
Frequency and length deviation of middle cups



Wah Chang (EDMS-DB)

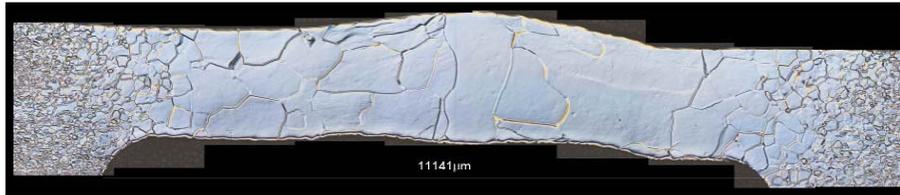
Cavity welding: the general way

There are differences of welding processes in industry

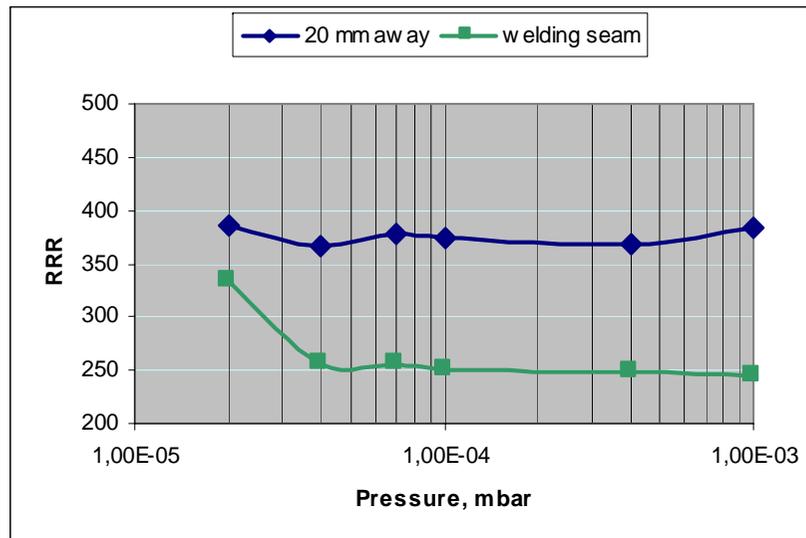


1. Degreasing and rinsing of parts
2. Drying under clean condition
3. Chemical etching at the welding area (Equator)
4. Careful and intensive rinsing with ultra pure water
5. Dry under clean conditions
6. Install parts to fixture under clean conditions
7. Install parts into electron beam (eb) welding chamber
(no contamination on the weld area allowed)
8. Install vacuum in the eb welding chamber $\leq 1E-5$ mbar
9. Welding and cool down of Nb to $T < 60$ C before venting
10. Leak check of weld

Electron beam welding of niobium

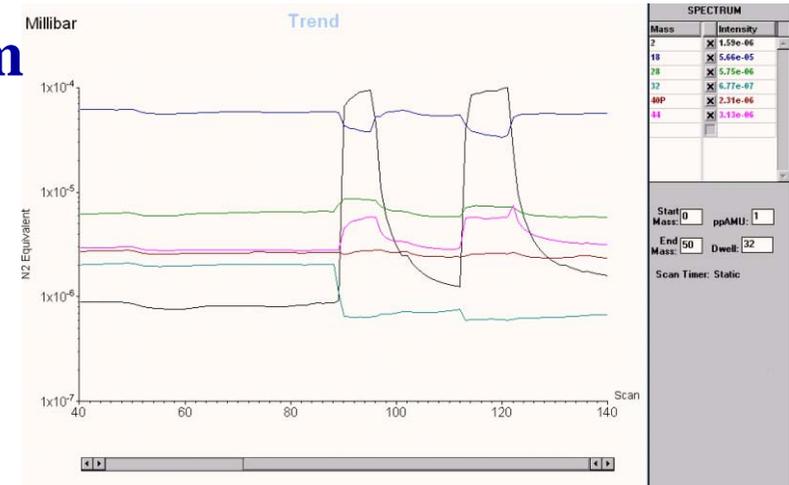


Microstructure of the EB welding area.
The grain size $G=50 \div 2000 \mu\text{m}$

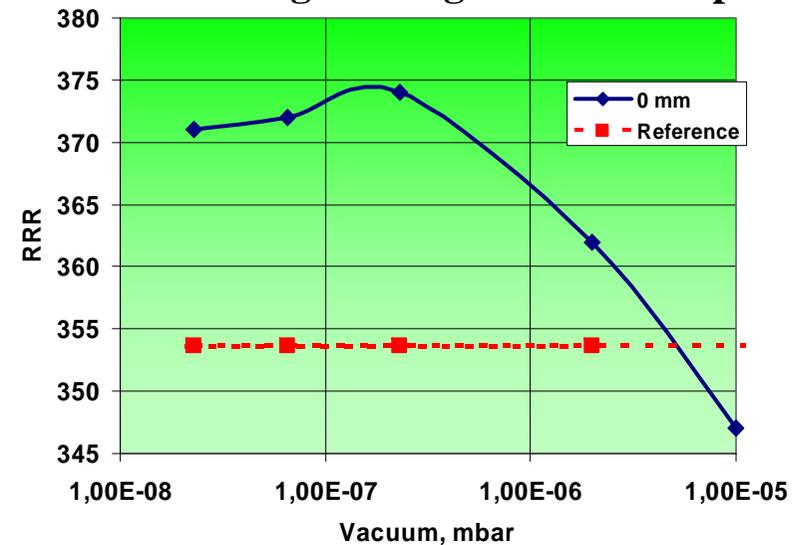


RRR in the welding seam and 20 mm away versus pressure in the chamber during EB welding

The RRR degradation at welding seam started since pressure of ca. 10^{-5} mbar.



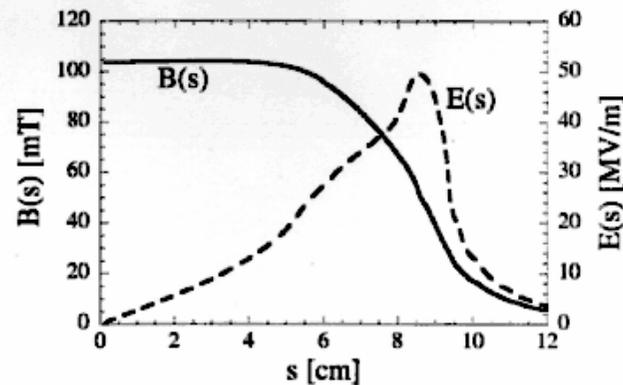
Partial pressure in the EB chamber (CERCA) during welding of Nb300sample



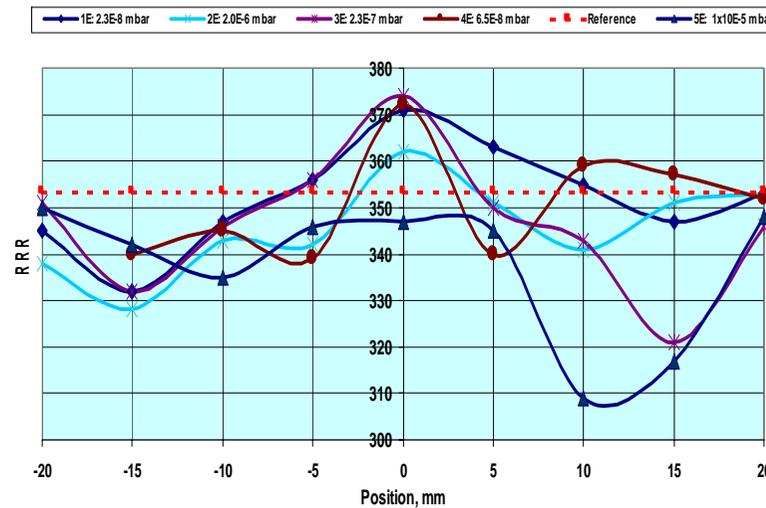
RRR in the welding seam versus pressure in the welding chamber

RRR degradation

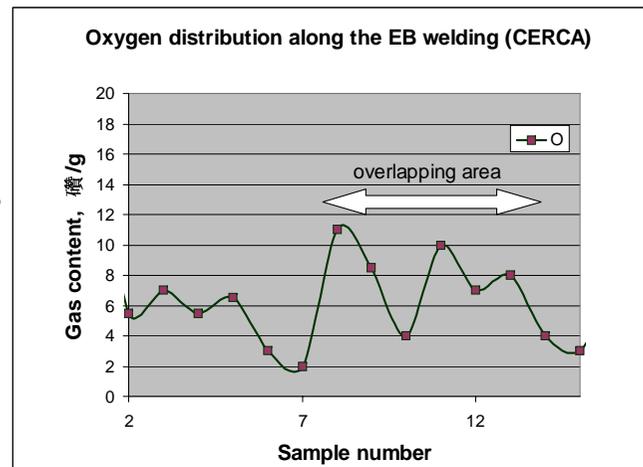
The RRR degradation can take place in the welding seam itself, but also in the thermally affected area and overlapping



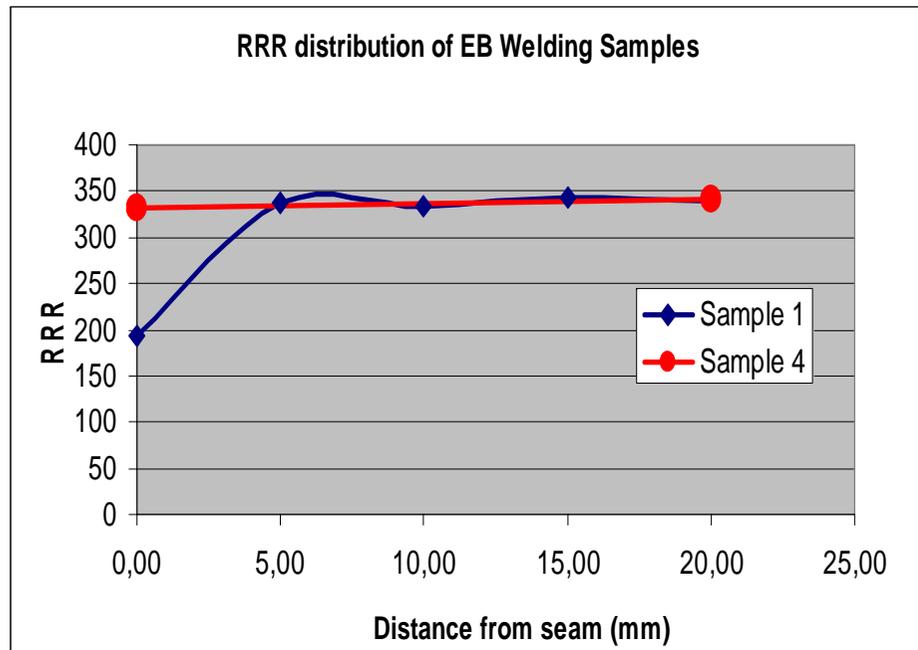
Distribution of the magnetic and electrical field from equator to the iris on the surface of the TESLA cavity
Thermally affected area of the welding seam can be critical for break down.



RRR in the EB welding area versus distance from the welding seam at different pressures of DESY EB facility



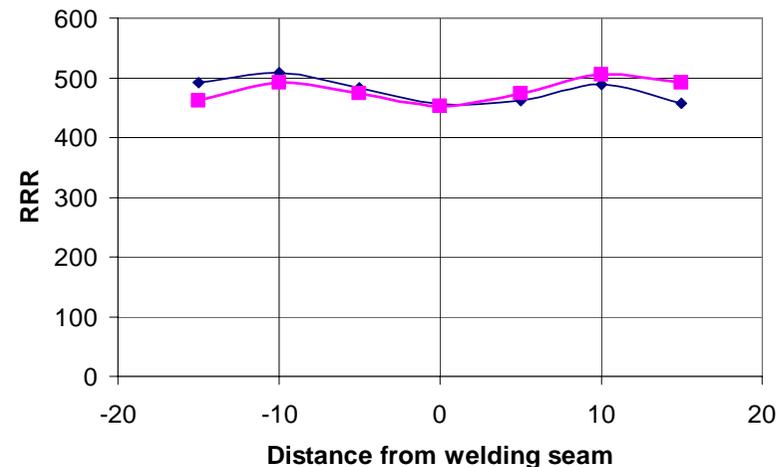
Oxygen distribution along the welding seam. RRR= 280 in the welding seam and RRR= 207 in the overlapping.



RRR in the welding seam of the welded sample RRR=485

RRR in the welding seam area with (sample 4) and without (sample 1) of Nb evaporation in the EB chamber (Julich)

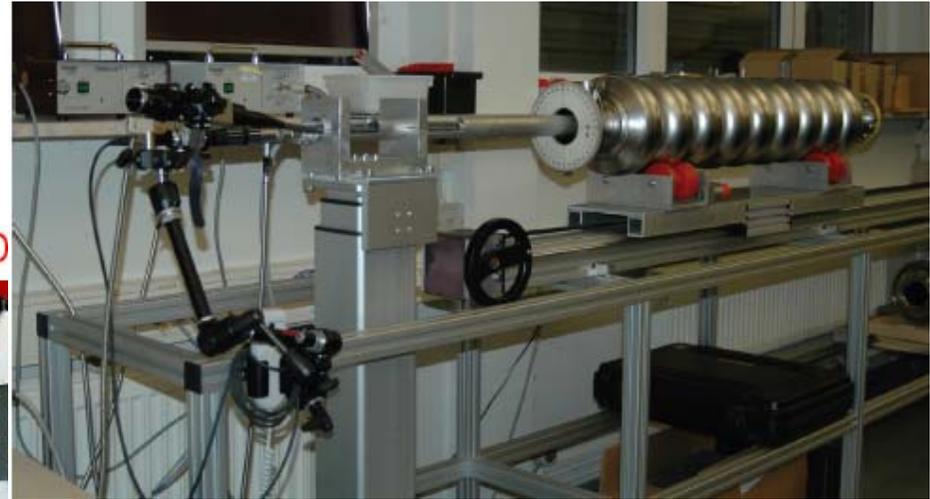
Zanon EB welding sample E-part



The RRR degradation at welding seam started since pressure of ca. 10^{-5} mbar. The vacuum conditions for EB welding of cavities are specified.



Eccentricity measurement



Optical control of the inside surface

Dimensions check



Fabrication of large grain LG cavities

Possible advantages (hope):

- Cost effective
- Higher purity. RRR=600 of ingot is achievable
- No danger that during many steps from ingot to sheet the material will be polluted.
- Simplified quality control (reduced number of measurements: grain size, eddy current scanning etc.)
- Higher thermal conductivity at low temperatures (phonon peak)
- Less RF losses on grain boundaries. Fine grain Nb sheet corresponds to length ~ 3000 m, LG Nb disc corresponds to length ~ 3 m (B. Spaniol, Linac2006)
- Seems to be less susceptible to field emission
- Seems that the baking at 120° C works better after BCP (compare to fine grain BCP)



LG DESY: Fabricated several single cell and three LG 9-cell cavities at ACCEL from HERAEUS material (AC112-AC114)

Fabrication:

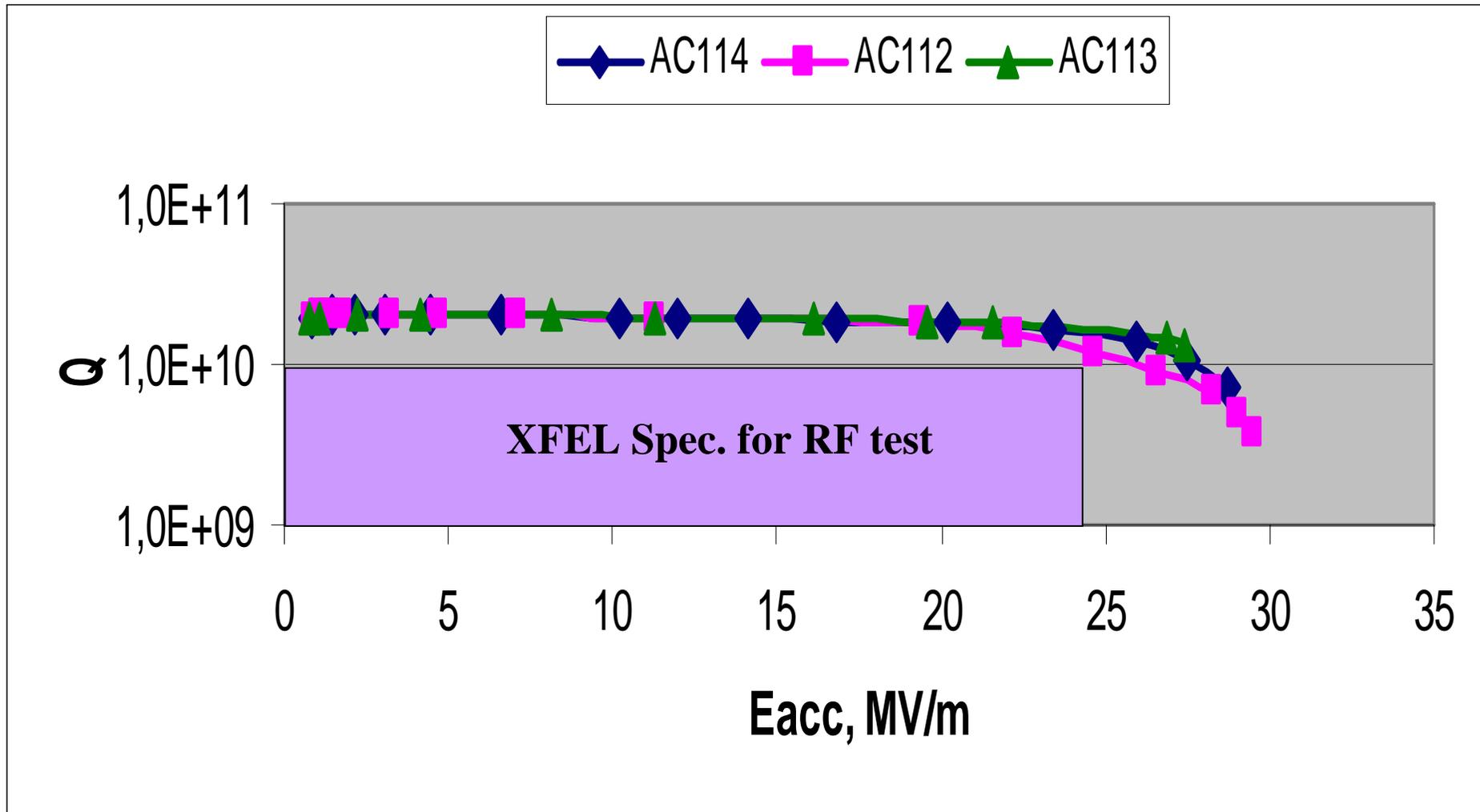
- disc of HERAEUS cut by diamond saw (B.Spaniol, LINAC 2006, TUP024)
- Discs scanned only for two cavities.
- Deep drawing
- Machining
- EB welding
- No grinding of grain boundaries

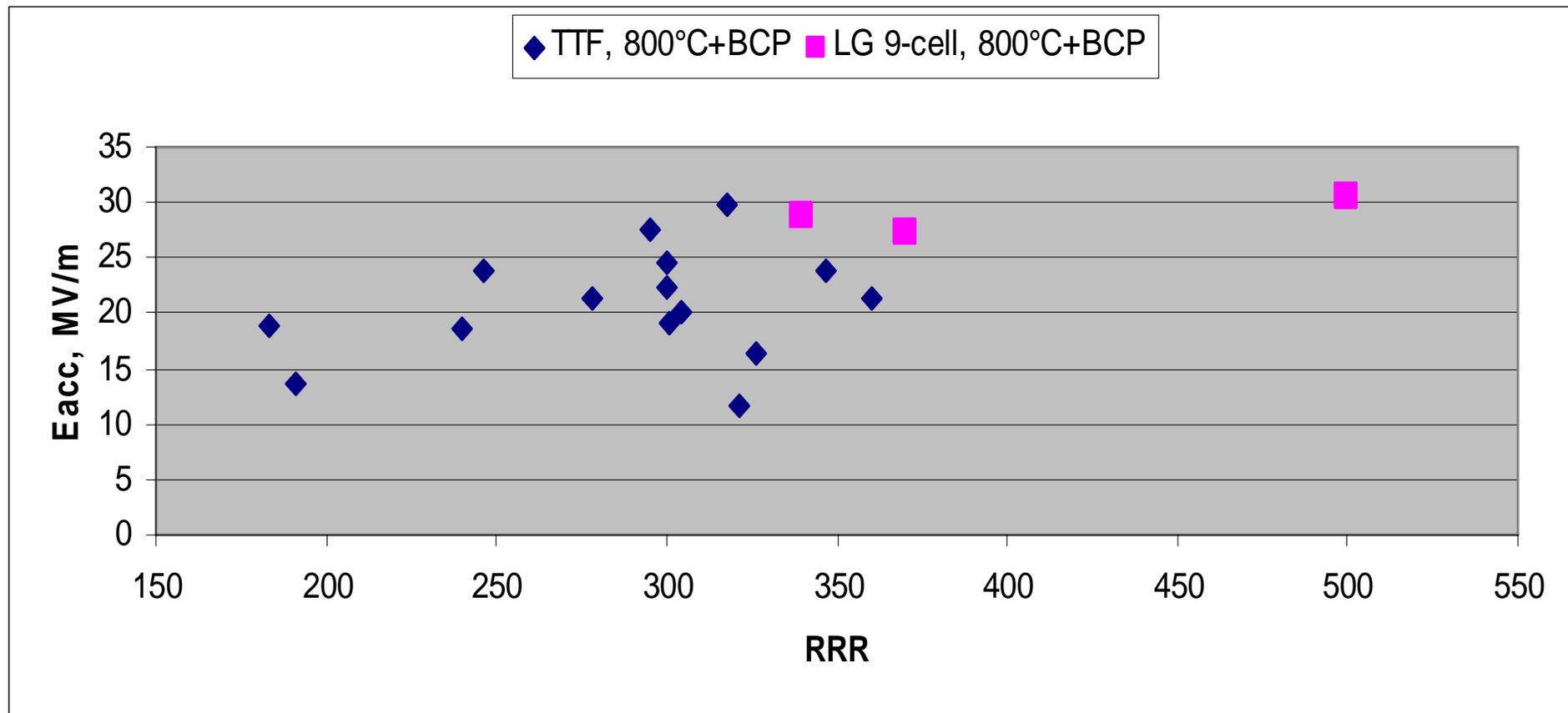


- No problems at EB welding
- Very smooth (shiny) surface in grain areas after BCP;
- the steps at grain boundaries are more pronounced as in polycrystalline material

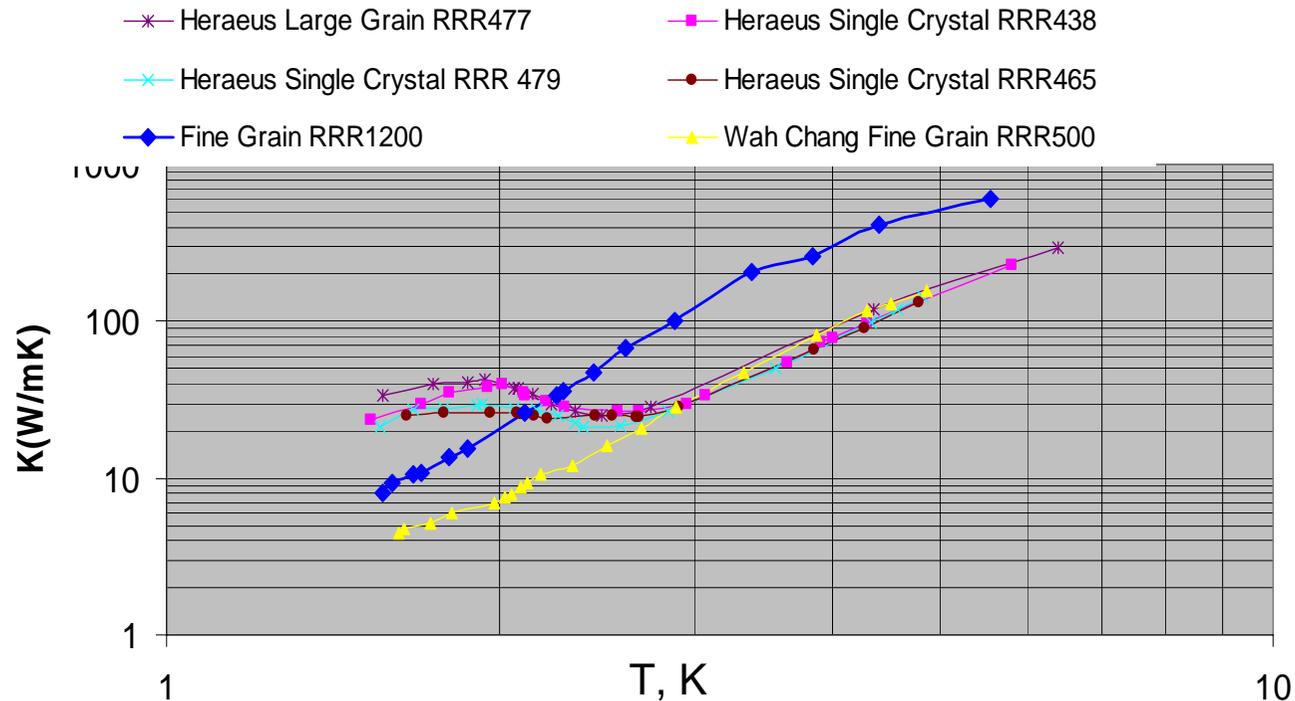
Preparation and RF tests

First test Q(Eacc) curve of the LG nine cell cavities AC112-AC114 at 2K after 100 μm BCP, 800° C, 20 μm BCP, HPR





Comparison of the Eacc performance of large grain (LG) 9-cell cavities with similarly treated fine grain TTF cavities

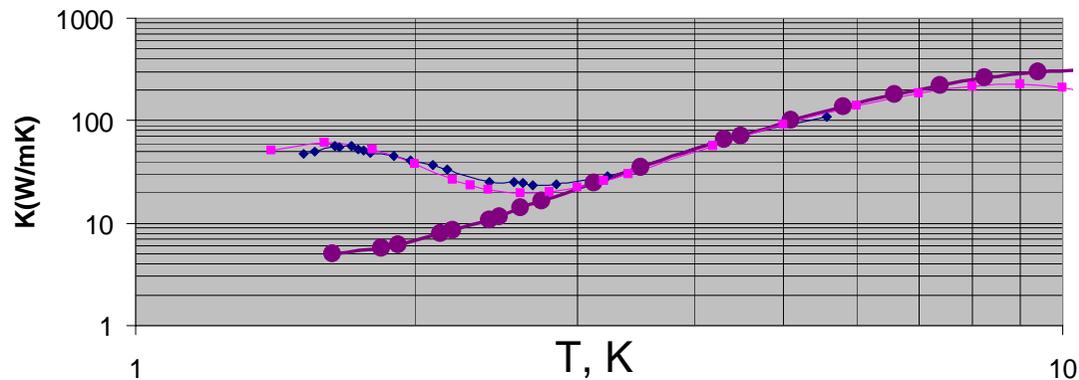


Thermal conductivity of single crystals in comparison with polycrystalline material. Phonon peak is clearly pronounced for single crystals.

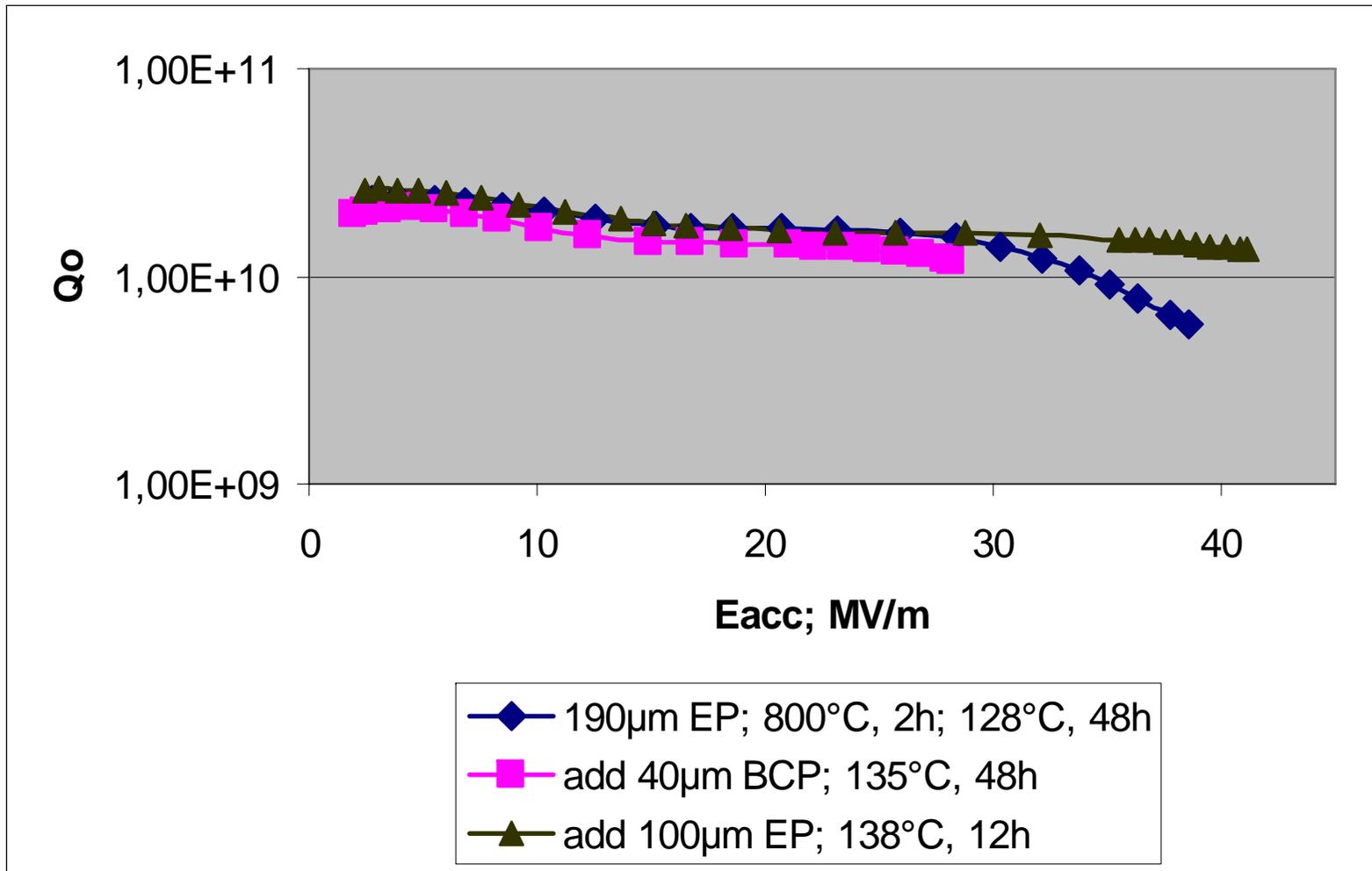
Parameterizations of F. Koechlin and B. Bonin

$$\lambda(T, RRR, G) = R(y) \cdot \left[\frac{\rho_{295K}}{L \cdot RRR T} + a \cdot T^2 \right]^{-1} + \left[\frac{1}{D \cdot \exp(y) \cdot T^2} + \frac{1}{B \cdot G \cdot T^3} \right]^{-1}$$

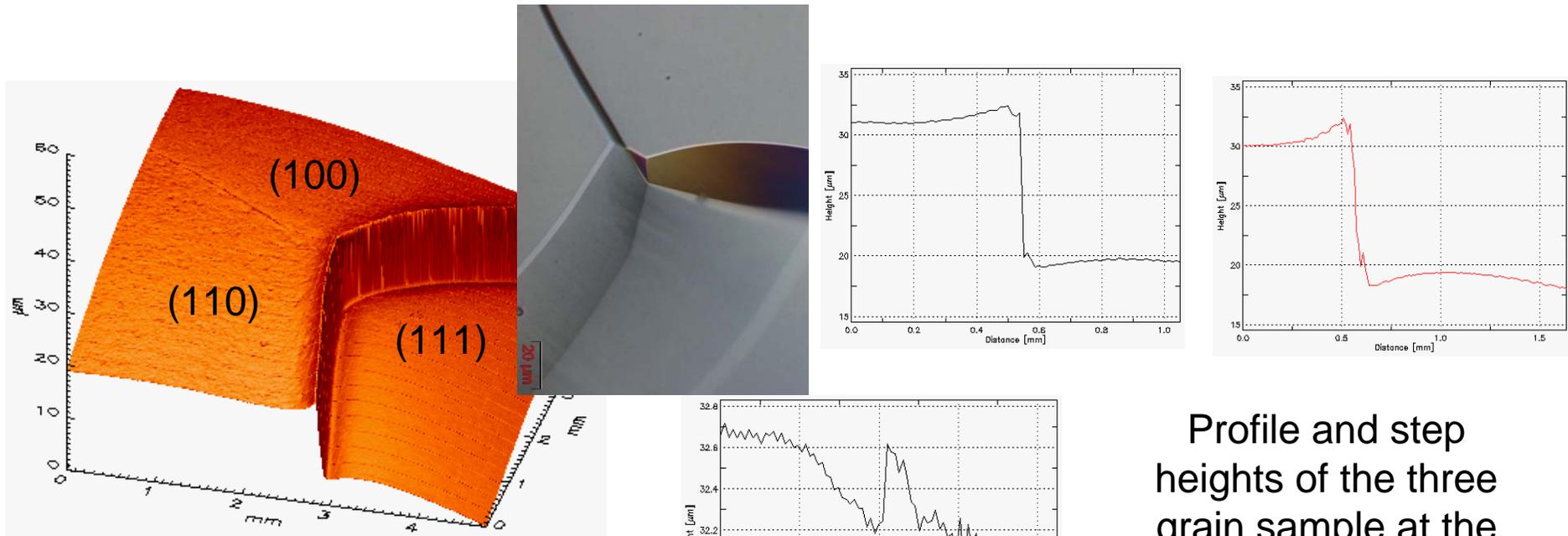
- ◆ Heraeus Single Crystal Before Deformation RRR255
- Heraeus Single Crystal After Deformation (8.5% length extension) RRR



Already small deformation destroy the phonon peak

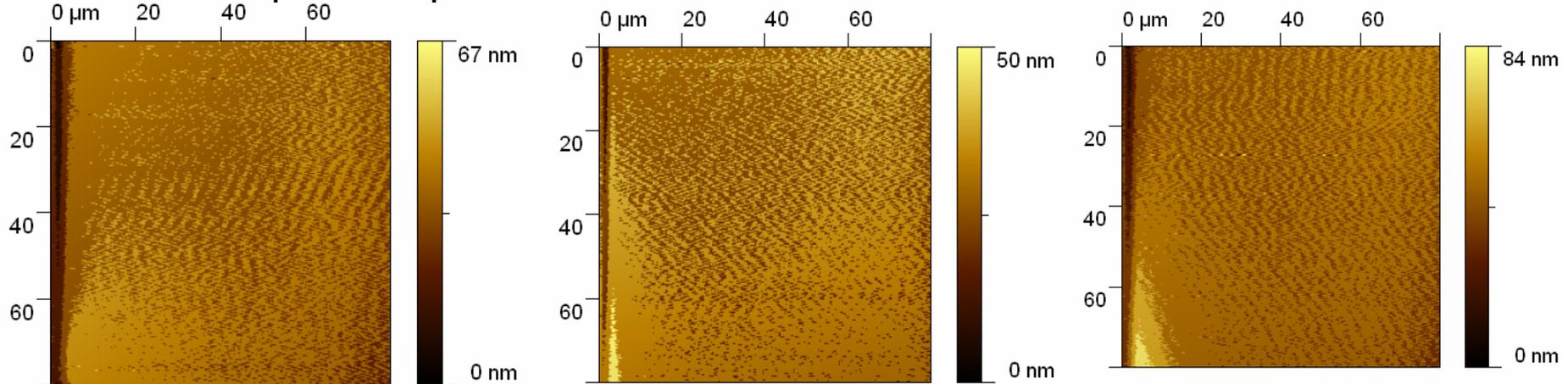


Q(Eacc) curve of the single cell cavity 1AC4
after EP and BCP treatment



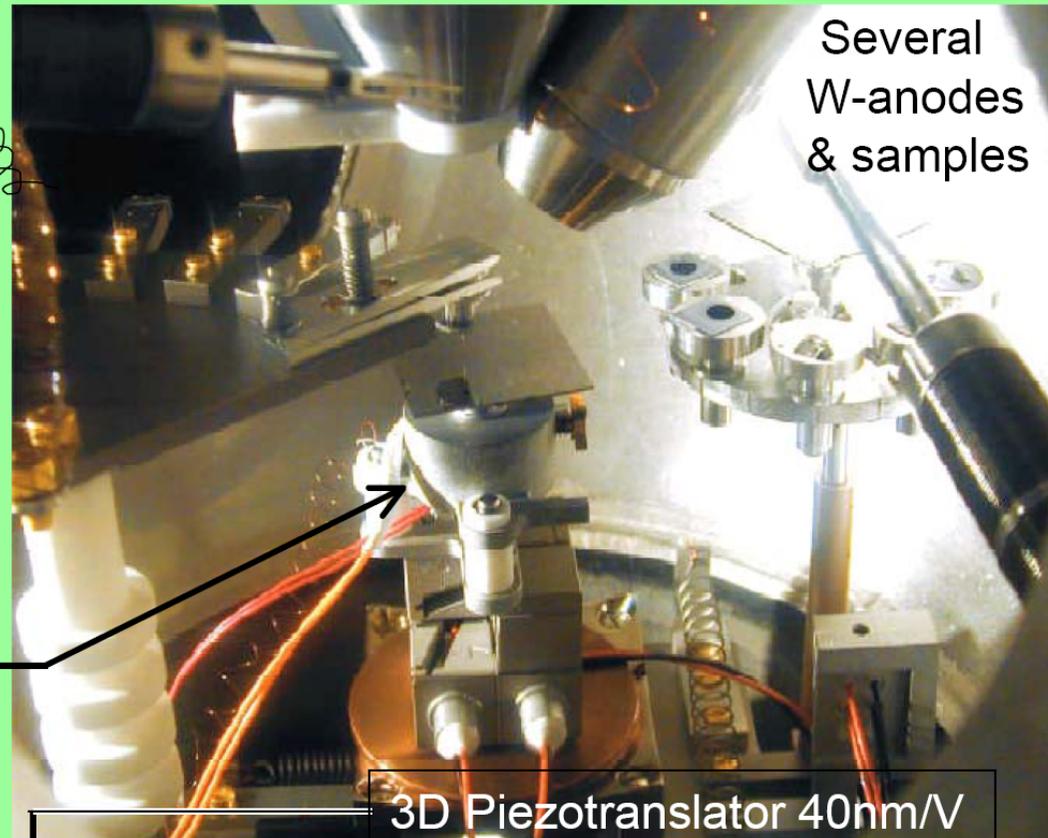
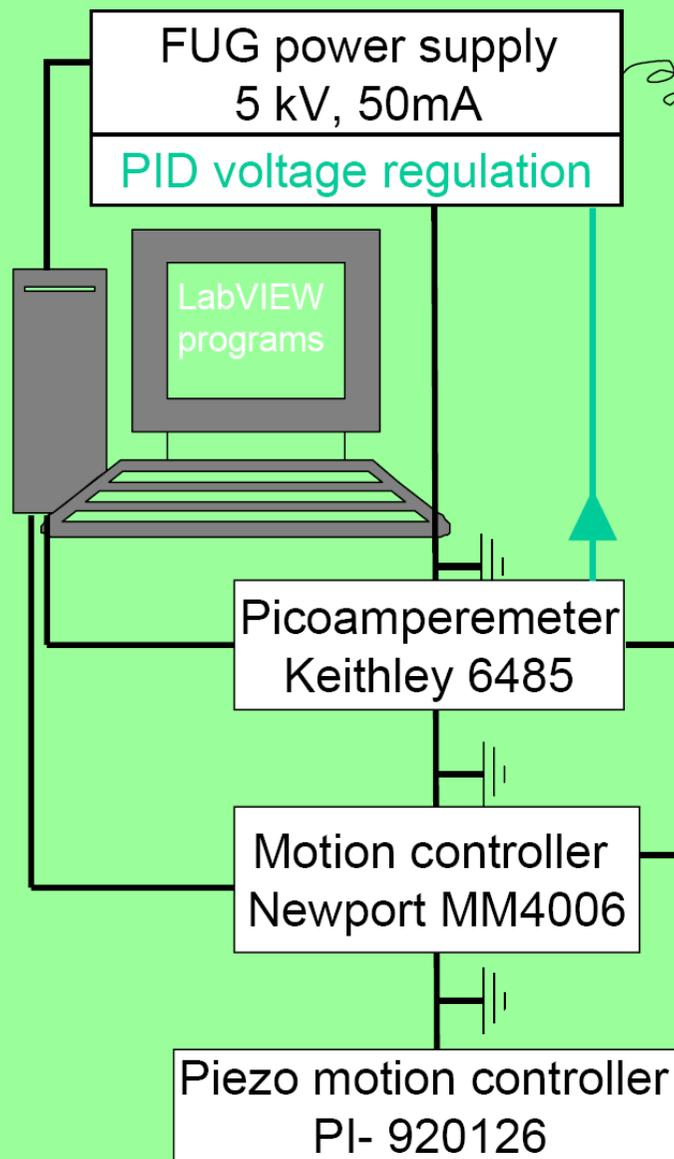
Light microscope and AFM image of LG Nb, BCP etched up to 100 μm

Profile and step heights of the three grain sample at the grain boundary intersection



AFM roughness measurement (X. Singer, A. Dangwal-Pandey). Roughness of fine grain Nb after EP is 251 nm (A. Wu) .

Field emission scanning microscope (FESM)



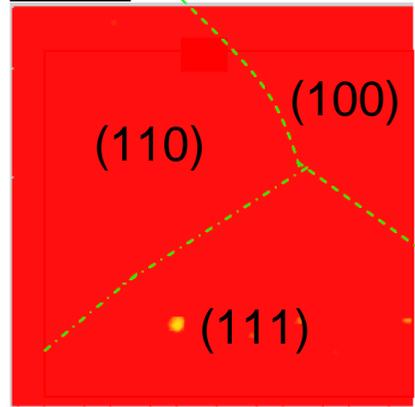
UHV system typically at $2 \cdot 10^{-7}$ Pa

LabVIEW automated scans of $U(x,y)$ for 2 nA

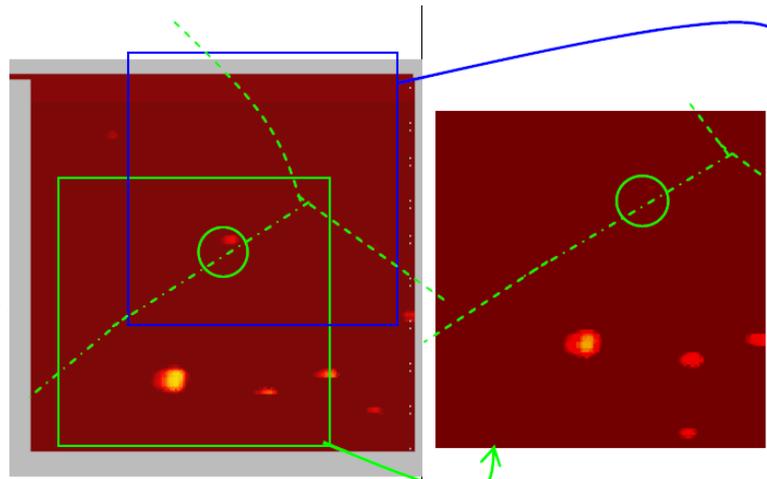
Scanning speed: (100×100) pixels in 1 hr

I/V curves and localization of stable emitters

After HT



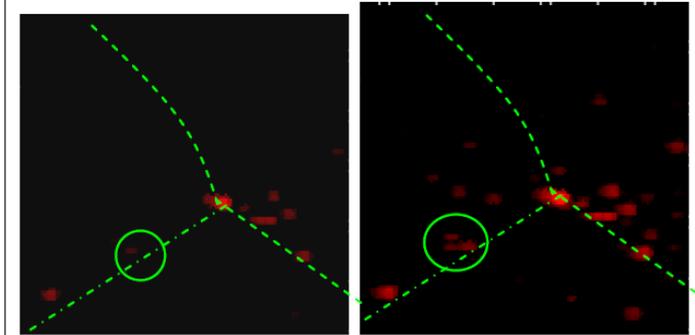
@ 90 MV/m, (10 mm)²
Emitters activated during V-scans before HT.



@ 150 MV/m, (7.5 mm)²

@ 200 MV/m, (5 mm)²
4 old + 1 new emitters observed.

Encircled emitter is the one activated before HT, dotted lines in the scan show grain boundaries (GB)

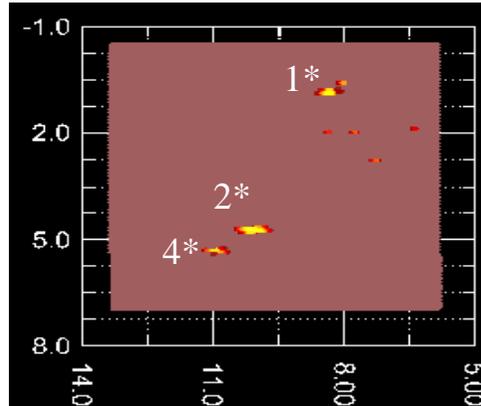
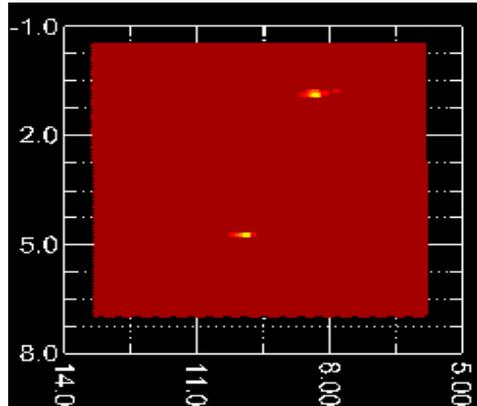


@ 250 MV/m, (5 mm)²

@ 300 MV/m, (5 mm)²

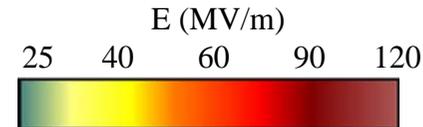
Increased number density after HT, all emitting sites appear near GB. Strongest emission observed at the intersection of three grain boundaries.

FE scans on three LG after 100 μm BCP and baking 150° C, 14hs



A. Dangwal, G.Mueller

Wuppertal Univ.



Example of similar FE scans on fine grain EP Nb sample. (left) E = 90 MV/m, 3 emitters
(right) E = 120 MV/m, 8 emitters

Orientation (111) is the worst one. Another data indicates that the (100) is the best one. A lot of emitters are close to grain boundaries.

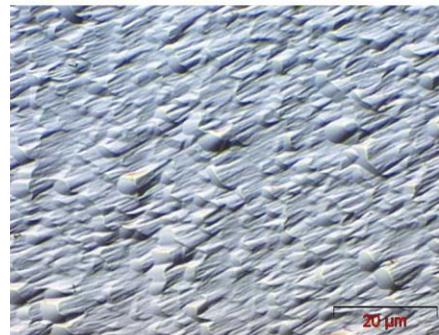
Single Crystal Option

Better not to have the grain boundaries at all

Fabrication of TESLA shape single crystal single cell cavities was proposed at DESY.

Following aspects have been investigated and taken into consideration during cavity fabrication

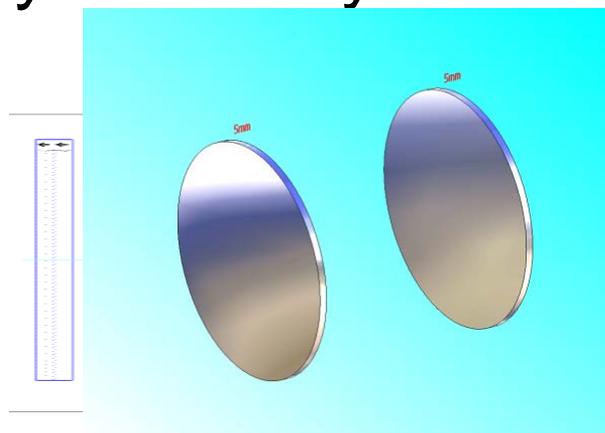
- Definite enlargement of the discs diameter is possible without destroying the single crystal structure in an existing state.
- Appropriate heat treatment will not destroy the deformed single crystal
- The single crystals keep the crystallographic structure and the orientations after deep drawing and annealing at 800°C
- Two single crystals will grow together by EB welding, if the crystal orientations is taken into account.



DESY Single crystal cavity fabrication



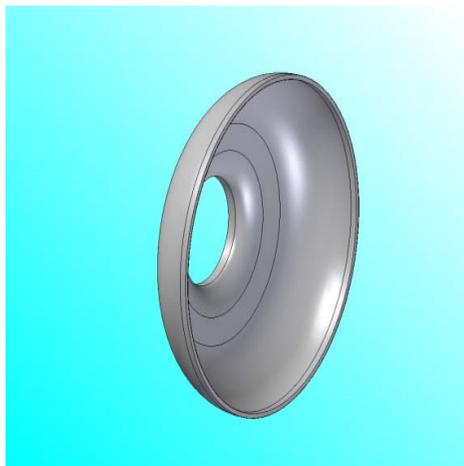
1. Take out central single crystal of definite thickness



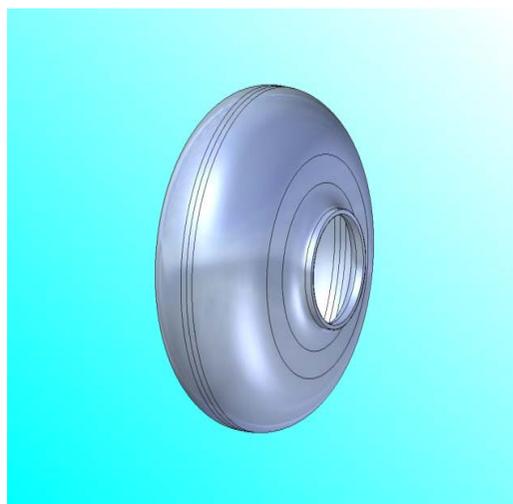
2. Cutting through the disc



3. Increasing of diameter by special rolling with an intermediate annealing



4. Deep drawing

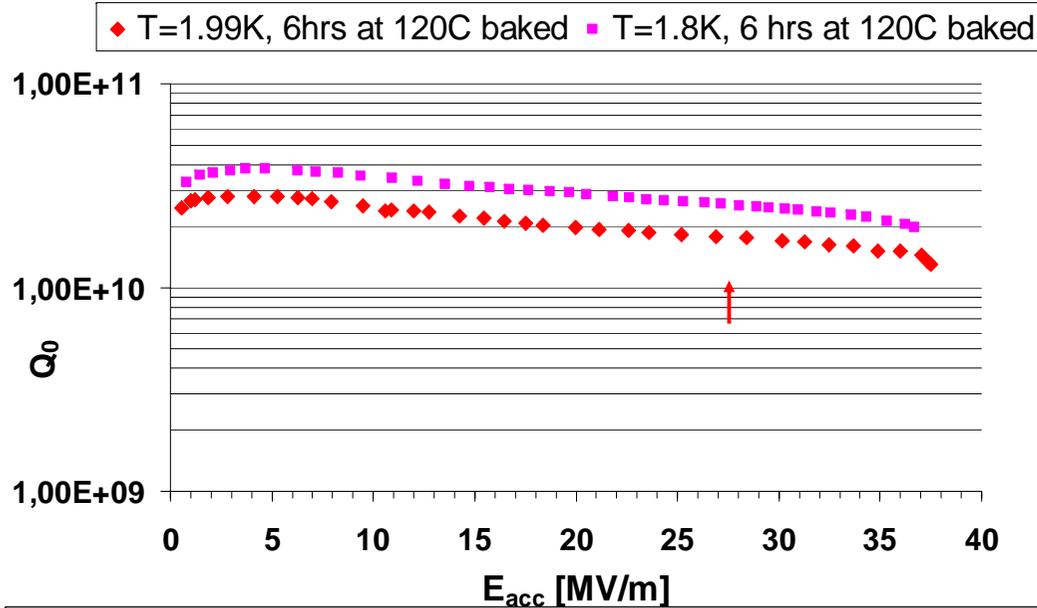


5. EB welding by matching the crystal orientation



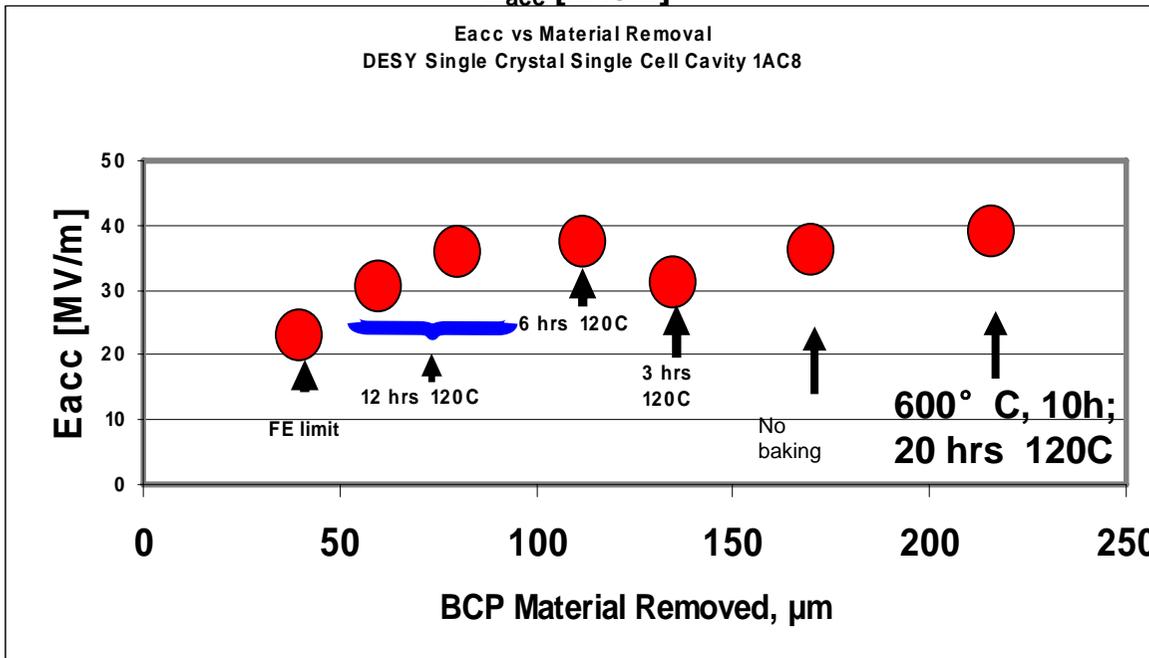
DESY SC cavity 1AC8 (TESLA shape) build from Heraeus disc by rolling at RWTH, deep drawing and EB welding at ACCEL

Single Crystal DESY Cavity, Heraeus Niobium
112 micron bcp 1:1:2



1AC8

Q(Eacc) curve of 1AC8 after only 112 μm BCP and in situ baking 120° C for 6 hrs 37,5 MV/m (equivalent to 160 mT)

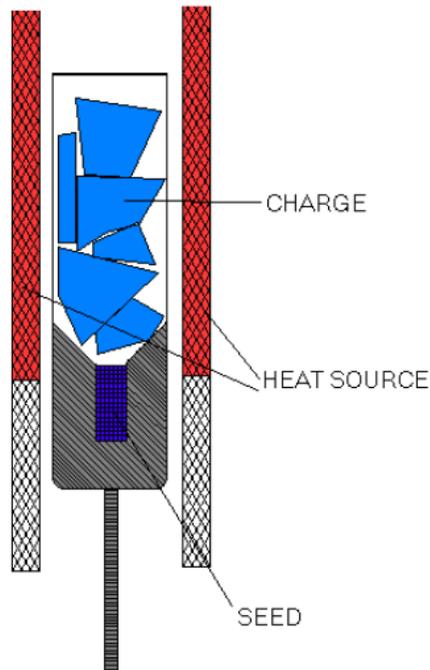


Eacc vs. material removal on single crystal single cell cavity 1AC8. Best Eacc= 38,9 MV/m (equivalent to $B_{p,max} = 166 \text{ mT}$)

Preparation and RF tests of P.Kneisel, JLab

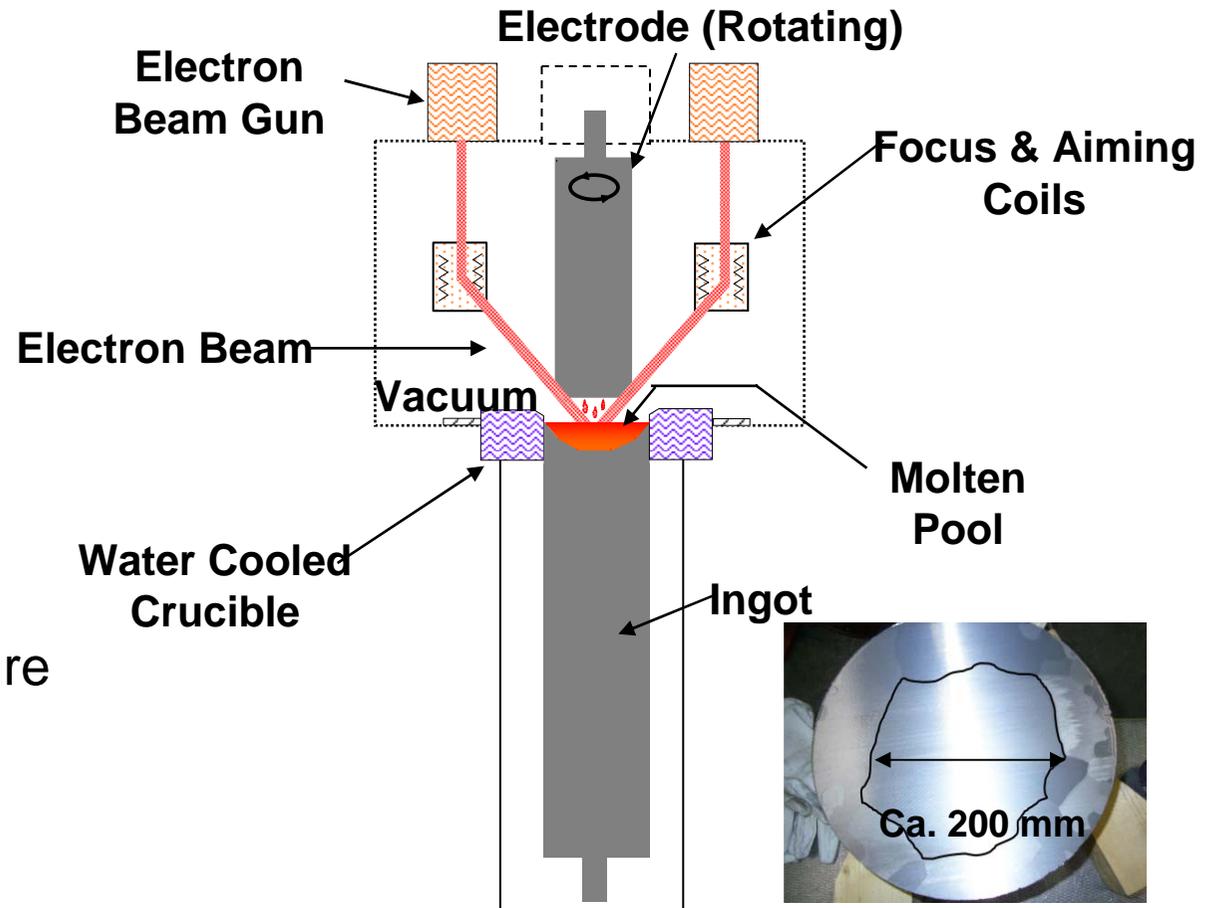
The proposed method can be extended on fabrication of multi cell cavities

Is it realistic produce single crystal cavities of sizes required for ILC?



Vertical Bridgman procedure
for single crystal grow

1. Seed, partially melted
2. Axial temperature gradient
3. Interface between solid and liquid phase is shifted by movement of container or temperature gradient

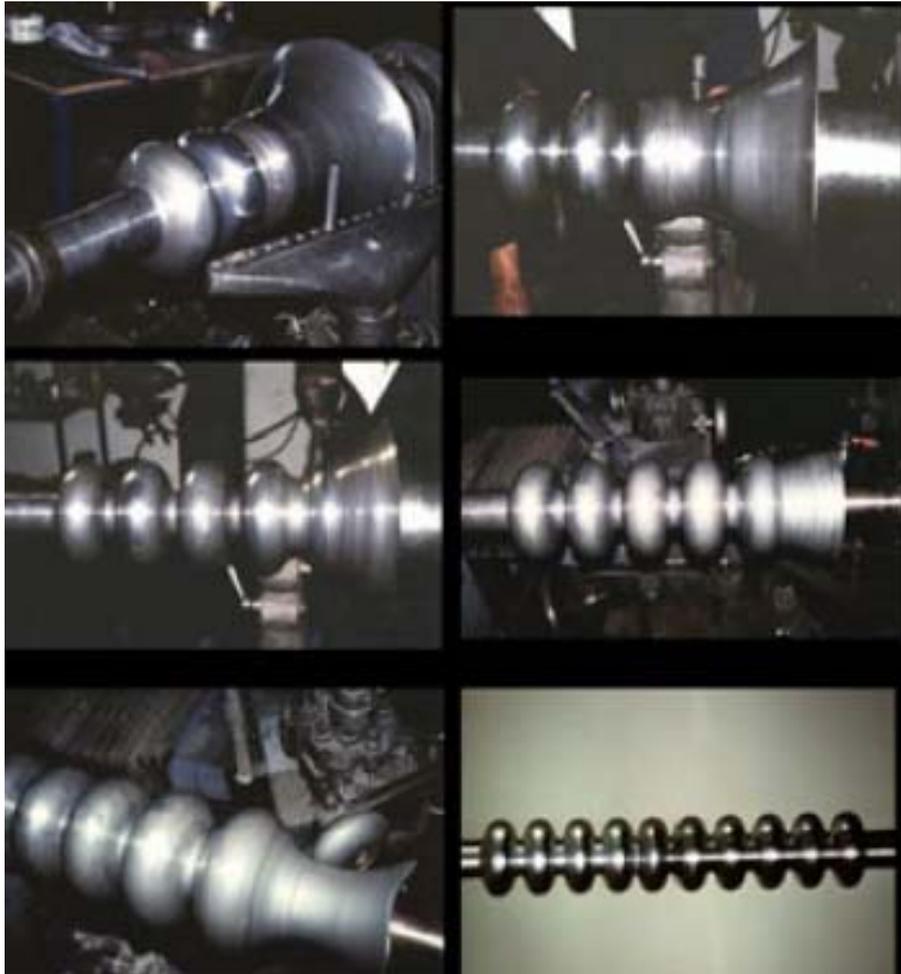


Electron beam melting principle

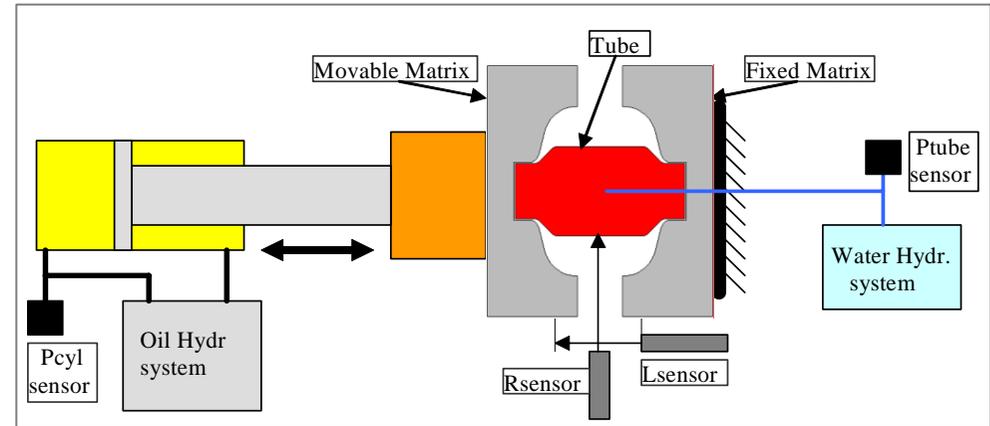
Challenge for the industry

Alternative fabrication methods

Spinning (V.Palmieri, INFN Legnaro)

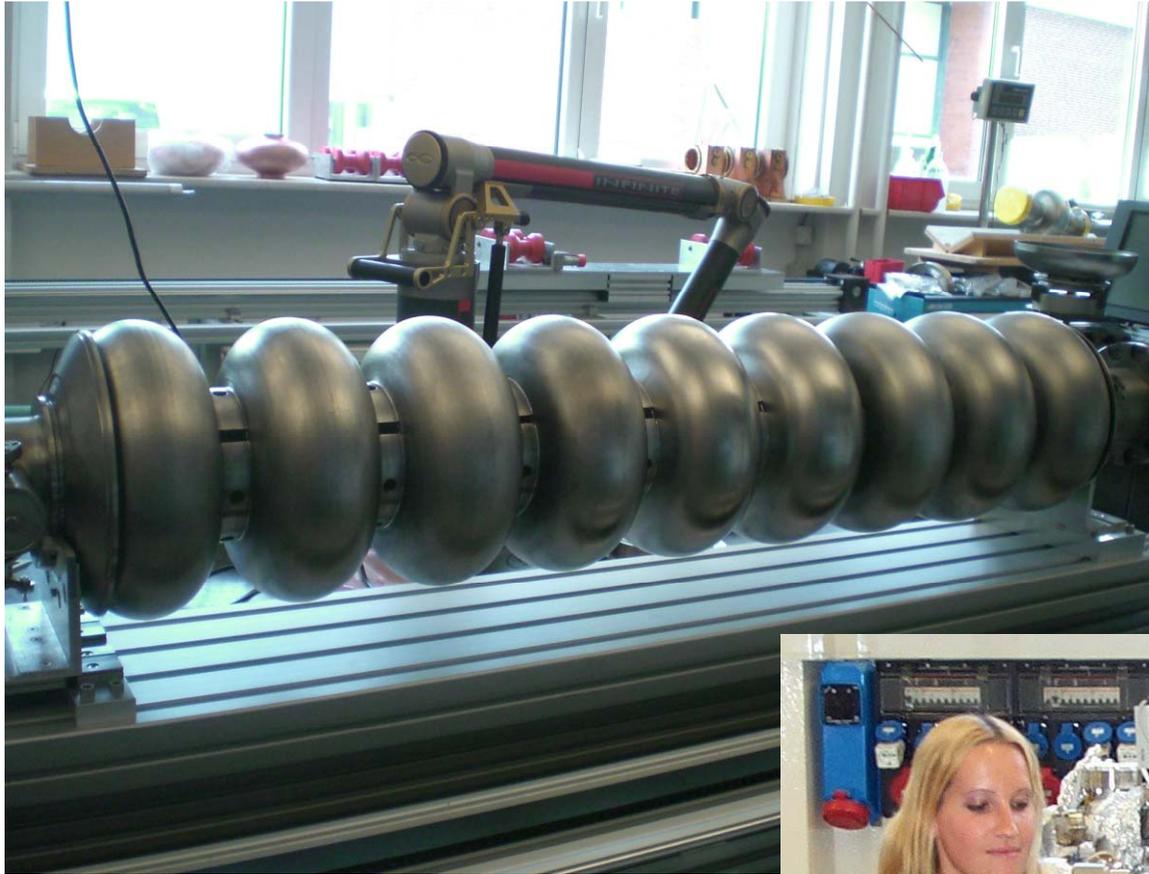


Hydroforming, DESY, KEK



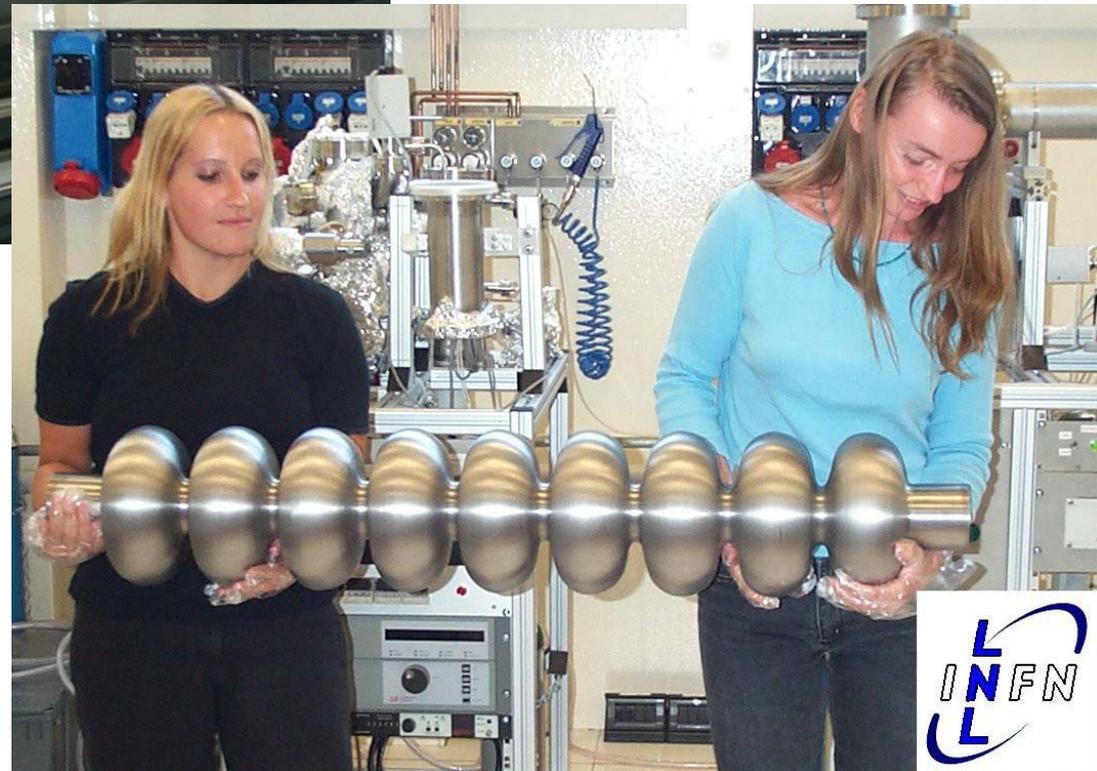
DESY hydroforming machine
HYDROFORMA

Fabrication of
9 cell cavities
became a
reality



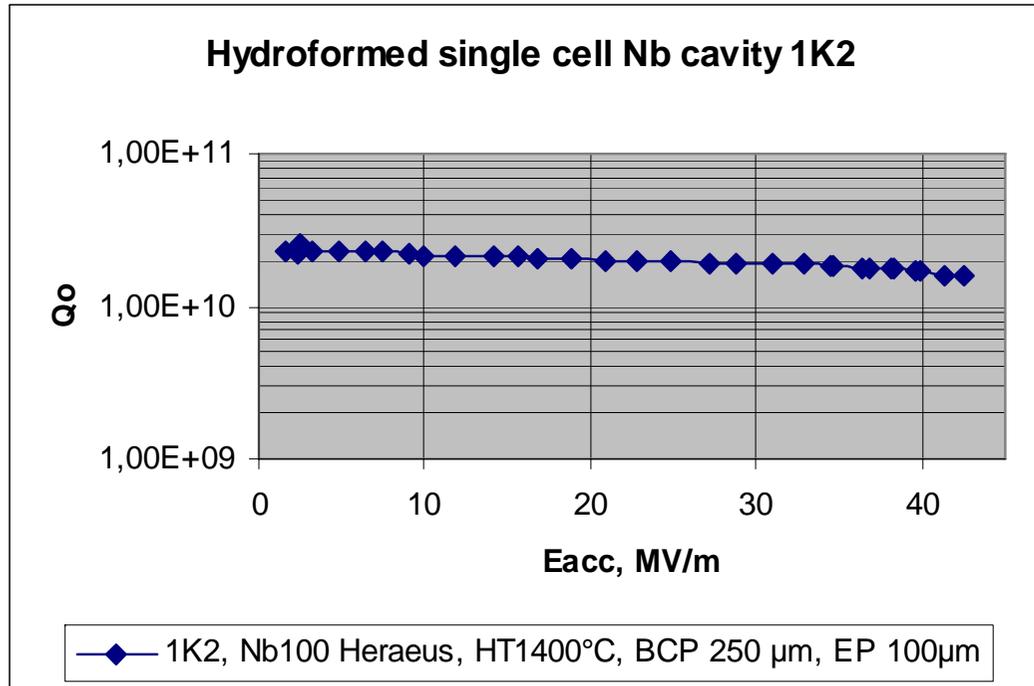
DESY Hydroformed
cavity

INFN
Spun
cavity



Hydroforming, Spinning

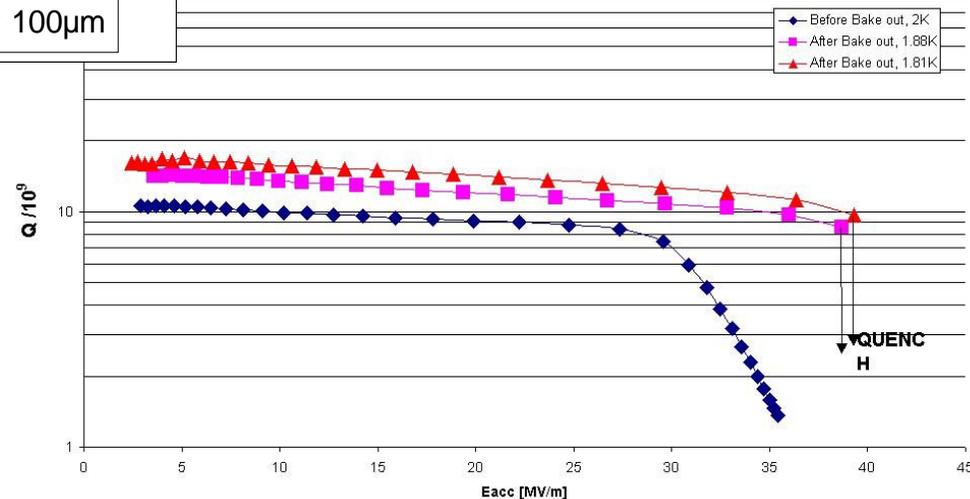
Proof of principle is done



The best Q(Eacc) result of by hydroforming produced single cell cavity

1P5, 100μm EP

The best Q(Eacc) result of by spinning produced single cell cavity



Acknowledgement

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