Large Grain, Single Crystal, NbCu Material for Superconducting RF Cavities

Presented by Waldemar Singer

DESY

Large Grain LG:

Fabrication issues

Main results

Summary and outlook

Single Crystal SC:

Fabrication issues

Main results

Summary and outlook

NbCu (optional)

Fabrication issues

Main results

Summary and outlook

Large grain LG

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
What has been done at JLab

(P.Kneisel, Single Crystal Niobium Technology Workshop, Araxá, Brazil
October 30 – November 1, 2006.)

- Cavities have been fabricated and tested from 4 different manufacturers:

  CBMM (4 different ingots)

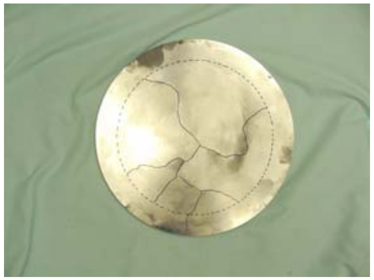
  Ningxia (3 different ingots)

  W.C. Heraeus (2 different ingots, 1 used for single crystal-DESY)

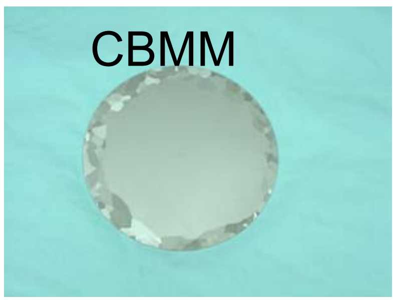
  Wah Chang (1 ingot)
- The material has been cut by wire EDM, saw cutting + machining, and wire saw cutting
- Single cell cavities ranging in frequency from 1300 MHz to 2300 MHz of different shapes and beta values (TESLA, LL_ILC,OC,HG,LL, PD) have been fabricated and tested
- Multi-cell cavities (2 HG (7-cell), LL_ILC(7 cell) have been fabricated and tested or are under test
- In total fabricated and tested 17 single cell cavities and 3 multi-cell cavities and carried out close to 100 tests

Large Grain Niobium (JLab)

CBMM



Ingot “D”,800 ppm Ta



Ingot “A”, 800 ppm Ta

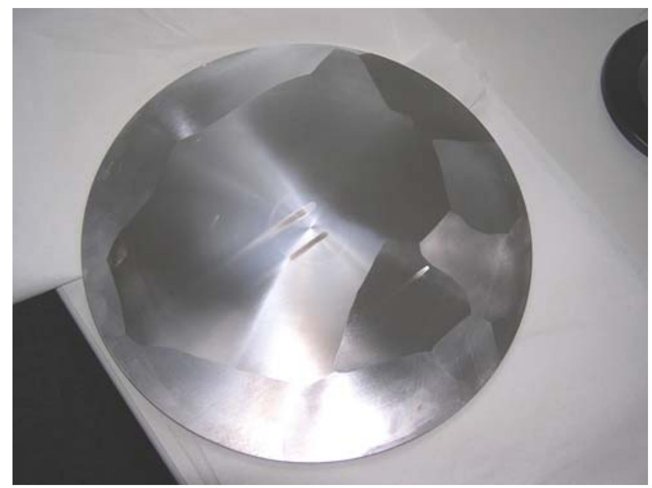
Ingot “B”, 800 ppm Ta

Ningxia

Ingot “C”, 1500 ppm Ta

CBMM

Wah Chang



Heraeus

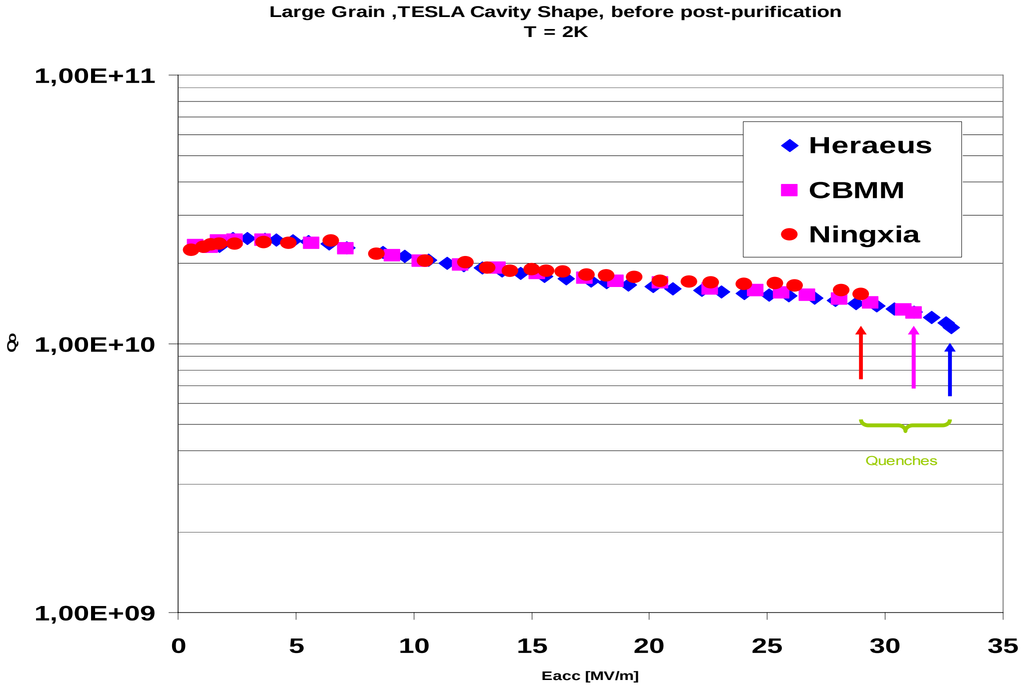
Fabrication and Treatment

Fabrication

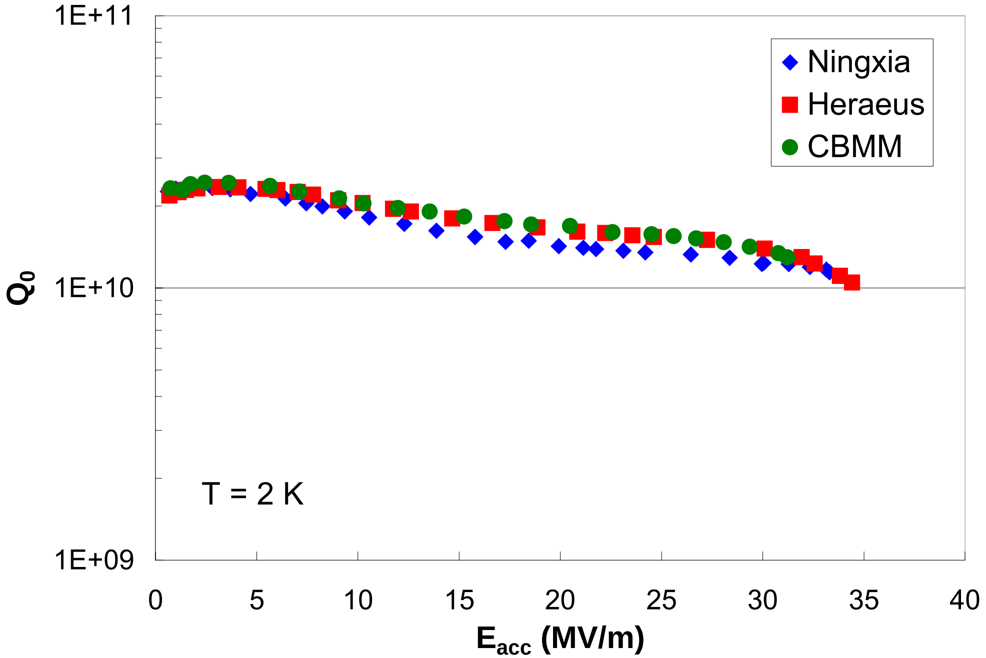
- Standard deep drawing after cutting of sheets (wire EDM/CBMM, saw cut/Ningxia, diamond saw/Heraeus)
- Machining
- Welding of beam pipes to half cell
- Mechanical grinding
- Equator weld

Surface Treatment

- ~ 50 micron bcp
- Hydrogen degassing at 600C for 10 hrs
- ~ 50 micron bcp, Test #1
- 12 hrs “in situ” baking at 120C, Test #2
- 1200 C, 3 hrs post-purification with Ti
- ~ 50 micron bcp, Test #3
- 12 hrs “in situ” baking at 120C,Test #4



Tests 2 summary (before post-purification)



Tests 4 summary (after Post-Purification)

W. Singer, Cavities-COM, September 19-21, 2007

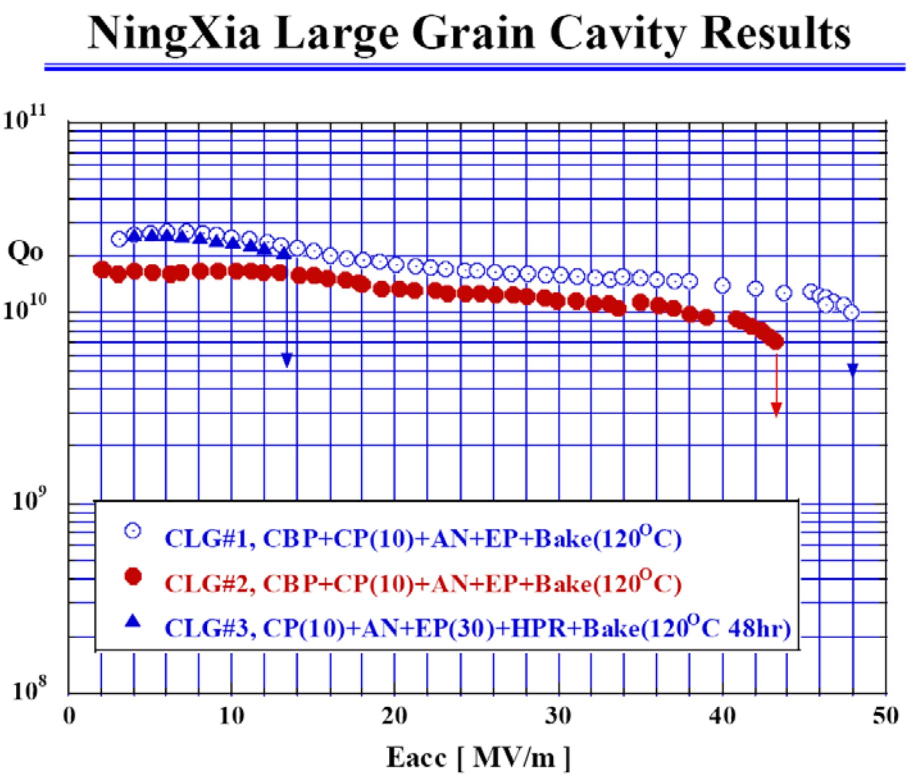
Current activities at JLab (in work)

- Fabrication of 5 single cell cavities each from Ningxia and W.C.Heraeus material to get some statistics
- The 9-cell cavities for FNAL are being fabricated from CBMM large grain niobium. Both cavity received bulk chemistry, heat-treated at 600°C for 10h, followed by final chemistry.

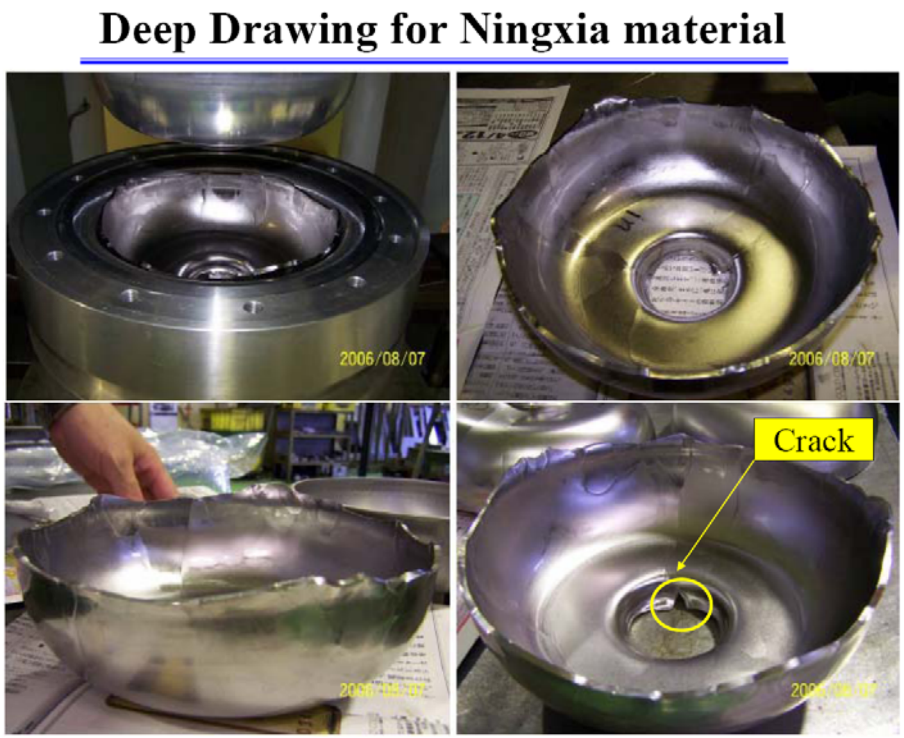
LG Niobium Activities in Asia:

KEK, IHEP (China),

NINGXIA grain size is smaller than CBMM by factor ~2.



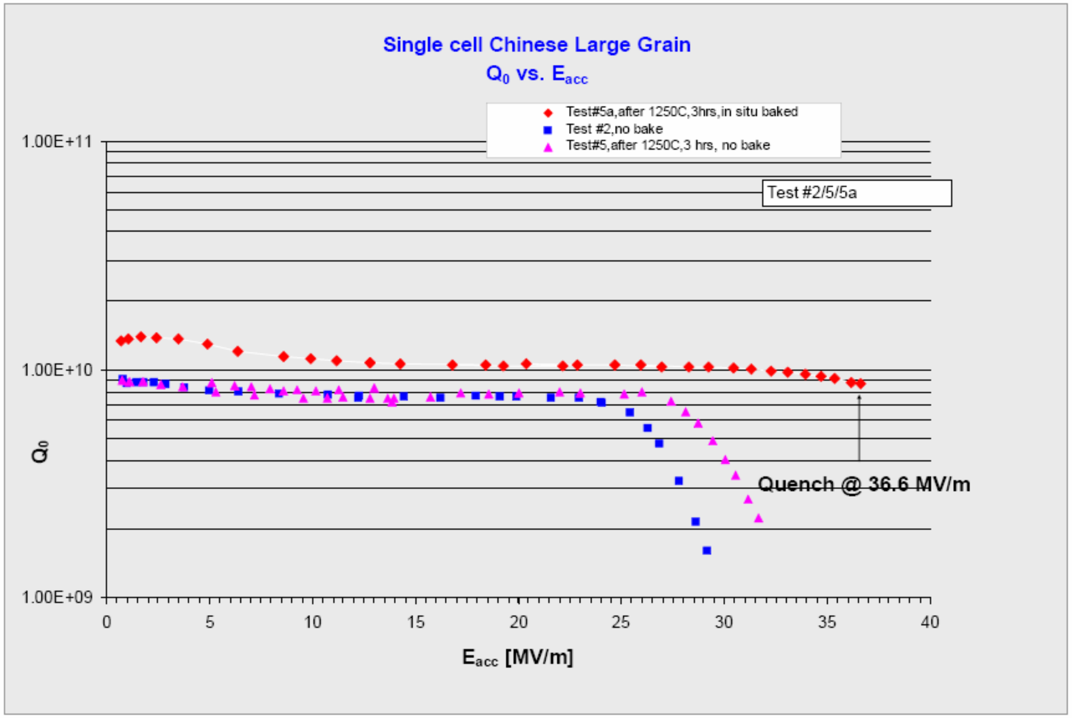
K.Saito



- High gradient performance for EP treated LG cavity is similar to EP treated fine grain cavity.
- Amount of material removal (ca. 100µm after CBP) or baking period is similar to fine grain cavity.

Peking Univ. Large Grain 1 cell cavities

large grain 1 cell cavity



Collaboration with JLAB

LG single cell cav.

LG two cell cav.

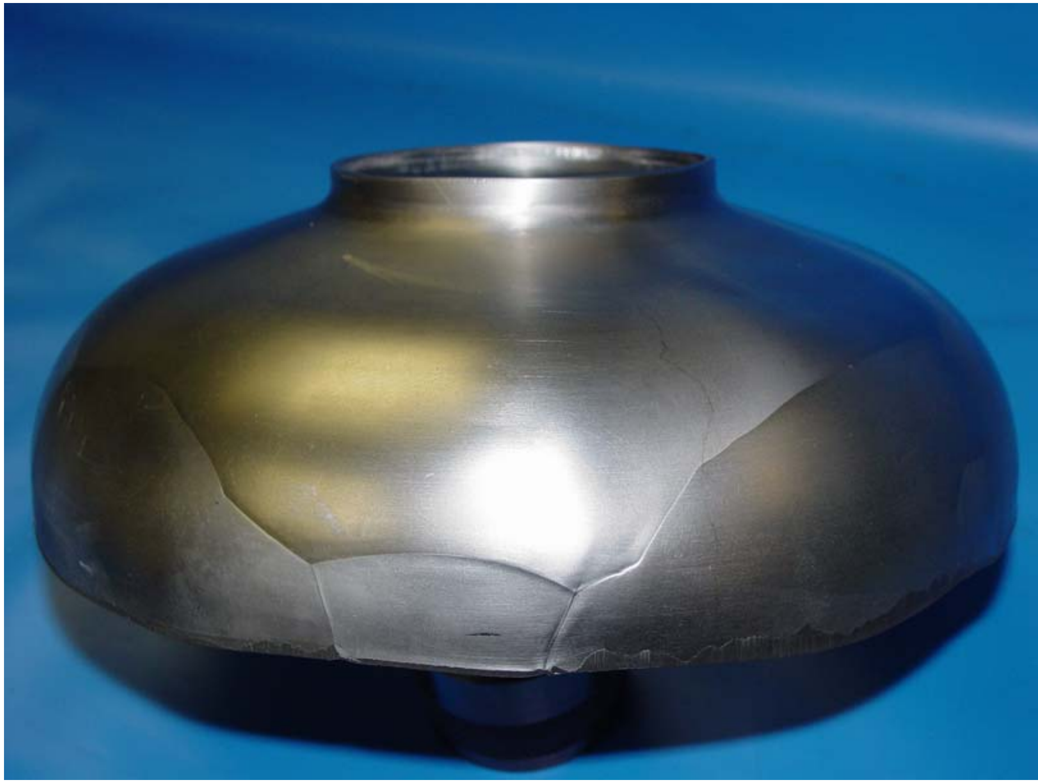
LG DESY: Fabricated several single cell and three LG 9-cell cavities at ACCEL from HERAEUS material (AC112-AC114)

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**

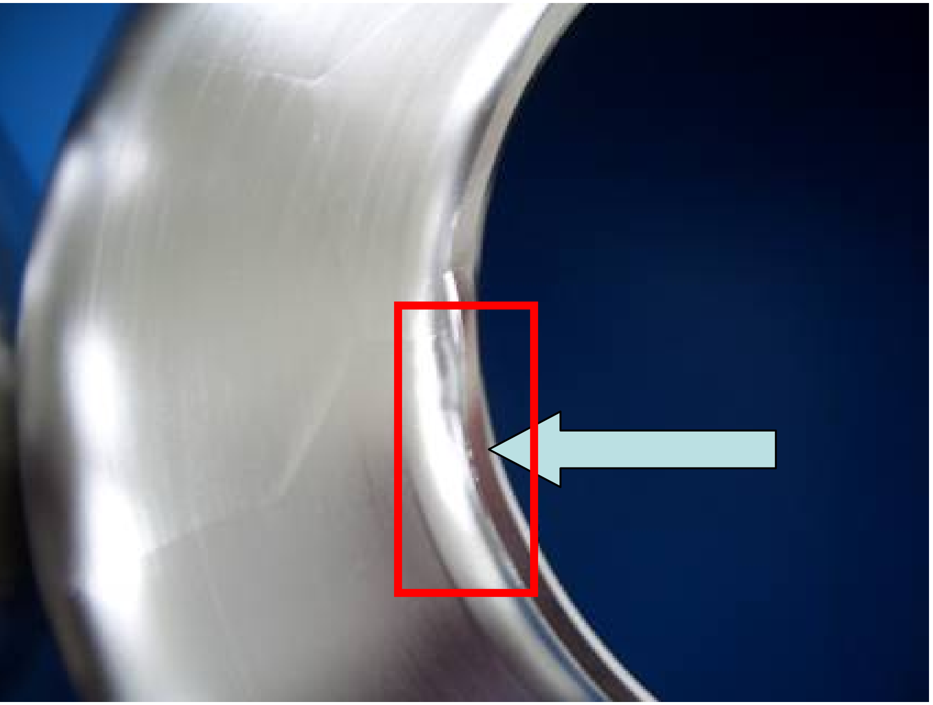
- No problems at EB welding
- Very smooth (shiny) surface in grain areas after BCP;
- the steps at grain boundaries are more pronounced as in polycrystalline material

- Some spring back after the deep drawing, making the half cells “oval”
- The same happens after the trimming for EBW
- Assembly for EBW some more difficult as with fine grain material



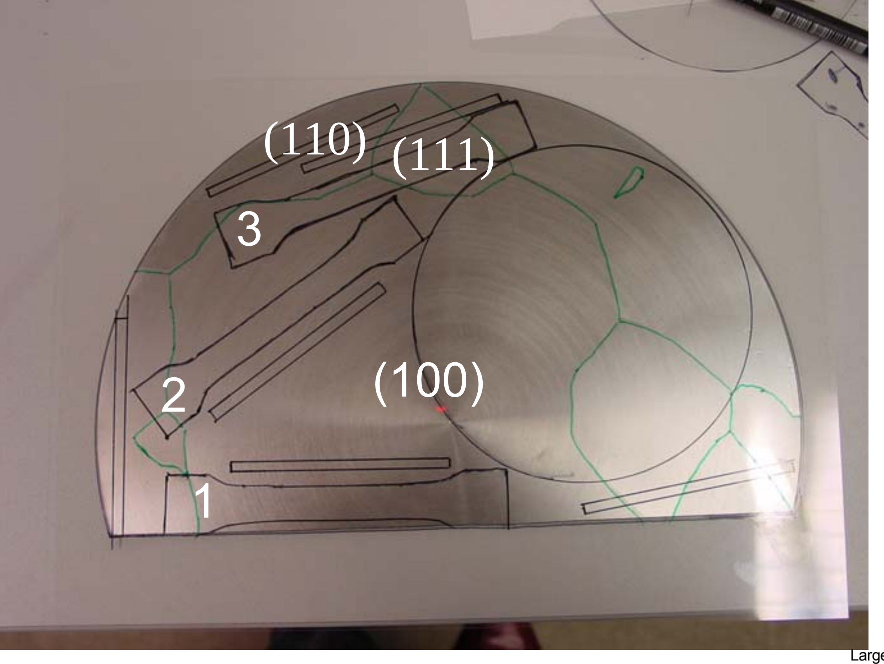
Deep drawn half cell of HERAEUS large grain niobium; Large single crystal at centre, no problems on iris area

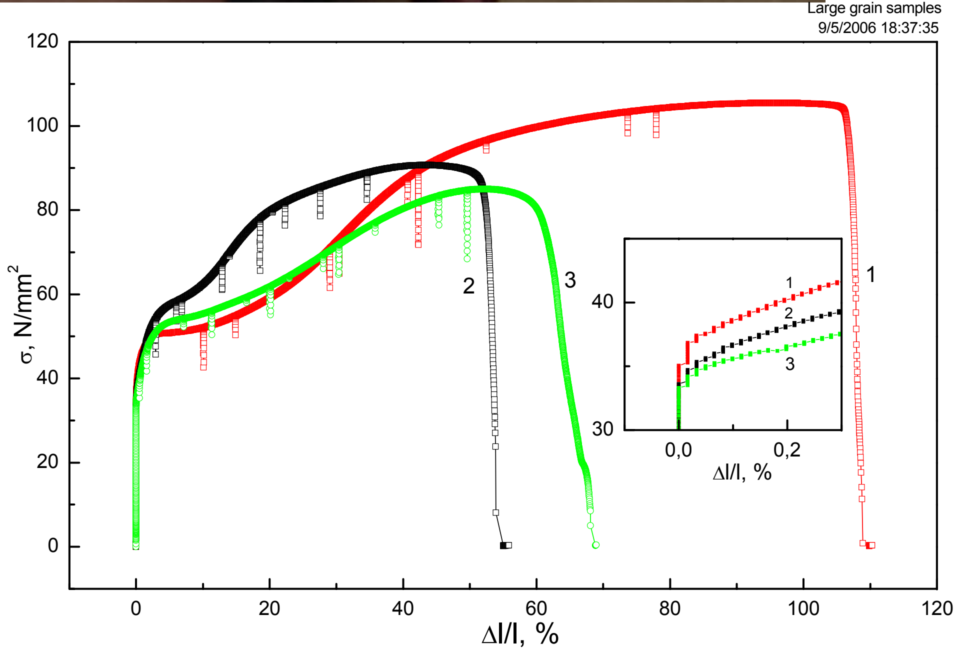
Deep drawn half cells of Ningxia LG Nb;



• thinning or ripping at grain boundary in iris if the grains “meet” in these areas

Safety margin is not big. Large central single crystal is mandatory



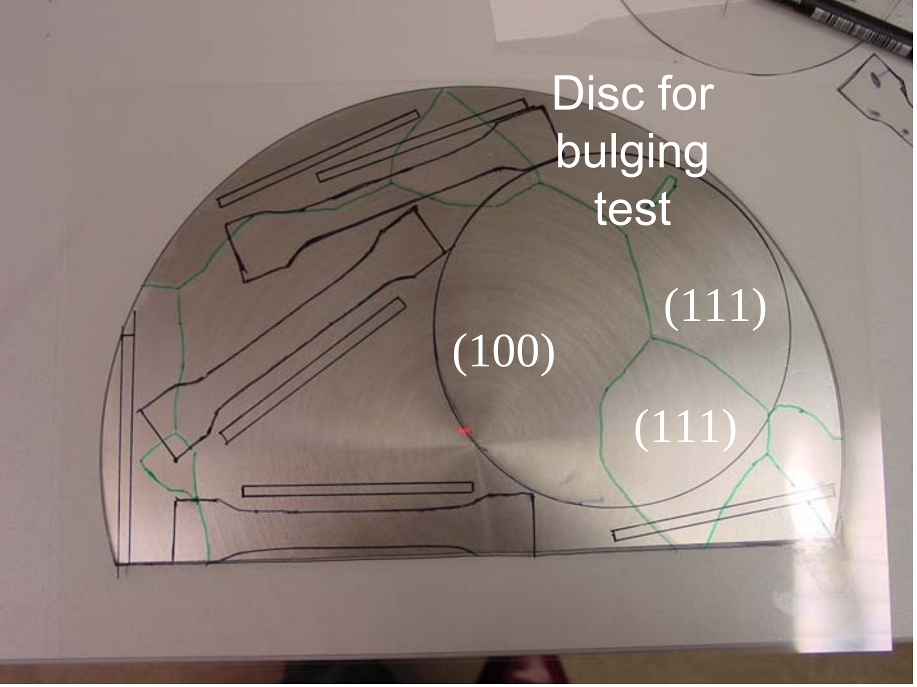


Strain-stress curve of LG/SC niobium

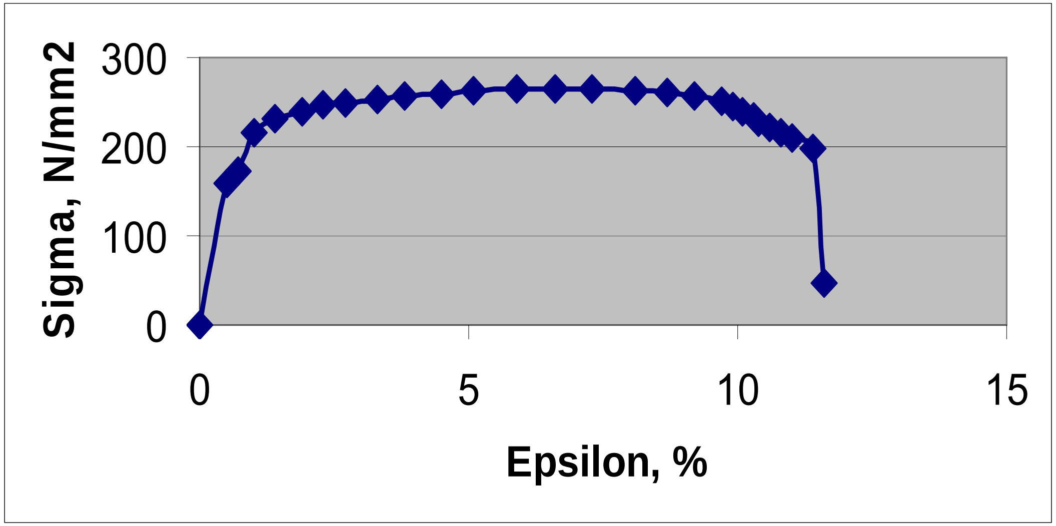
Mechanical properties: Tensile test

Percentage elongation after fracture for LG is rater high despite of grain boundaries.

Percentage elongation after fracture for SCs depends significantly on the load direction



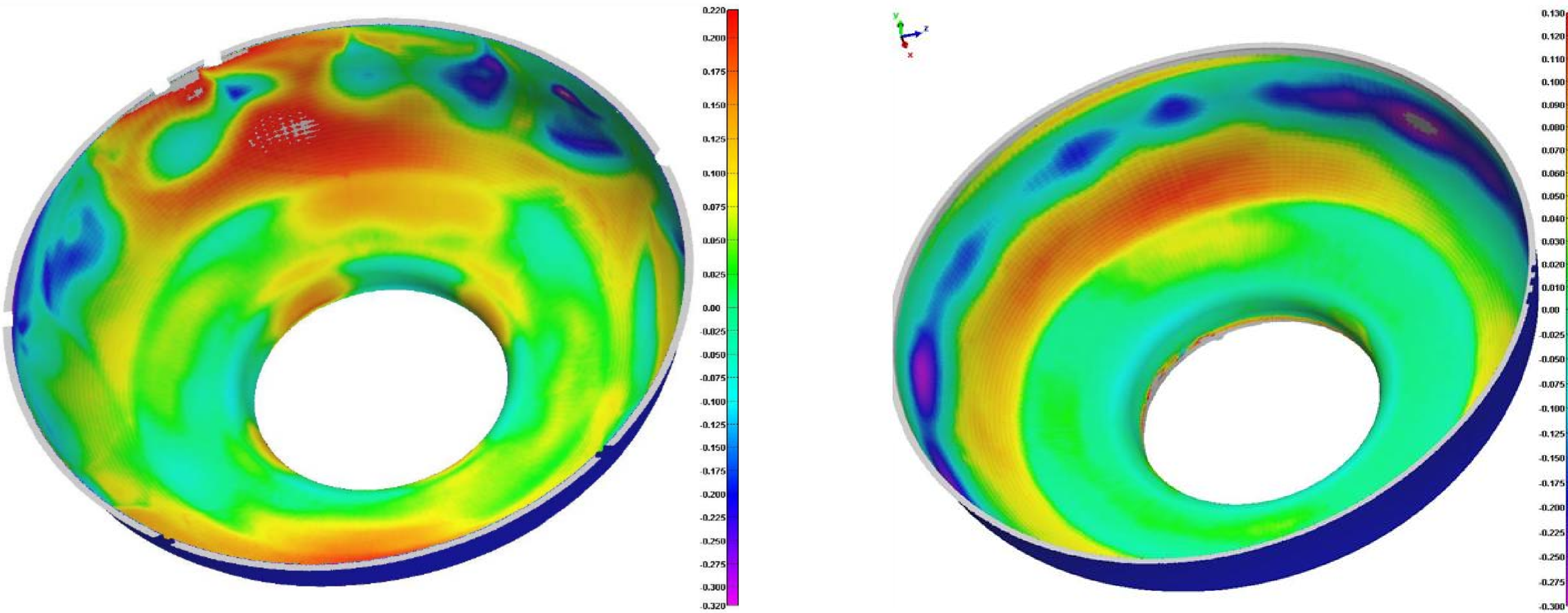
Mechanical properties: Bulging test



Bulging test on large grain niobium.

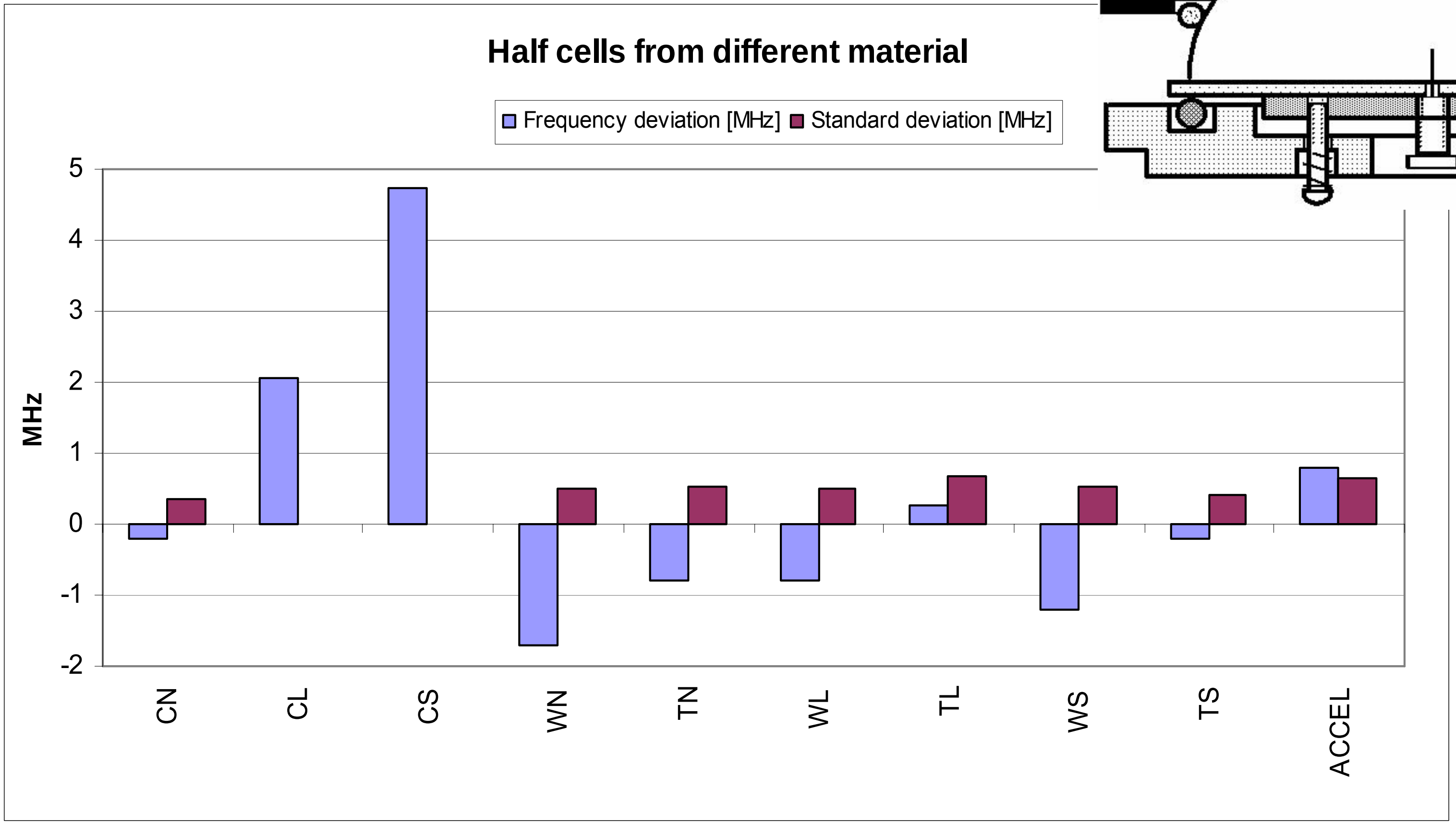
Percentage elongation after fracture by two dimensional deformation for LG is low (<15%). The rupture takes place close to grain boundary

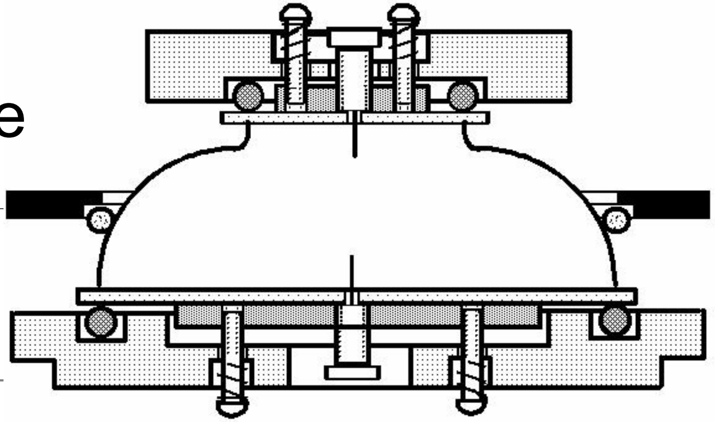
Shape accuracy: 3D profile measurement



3D Image of the optical measurement of the shape on large grain half cell (left; realized accuracy +0,22 / -0,32 mm) in comparison with a fine grain half cell (right; realized accuracy +0,13 / -0,30 mm). The large grains are fractionally pronounced. The variation of the large grain half cell shape is somewhat larger

RF measurement:
LG has better reproducibility in the shape



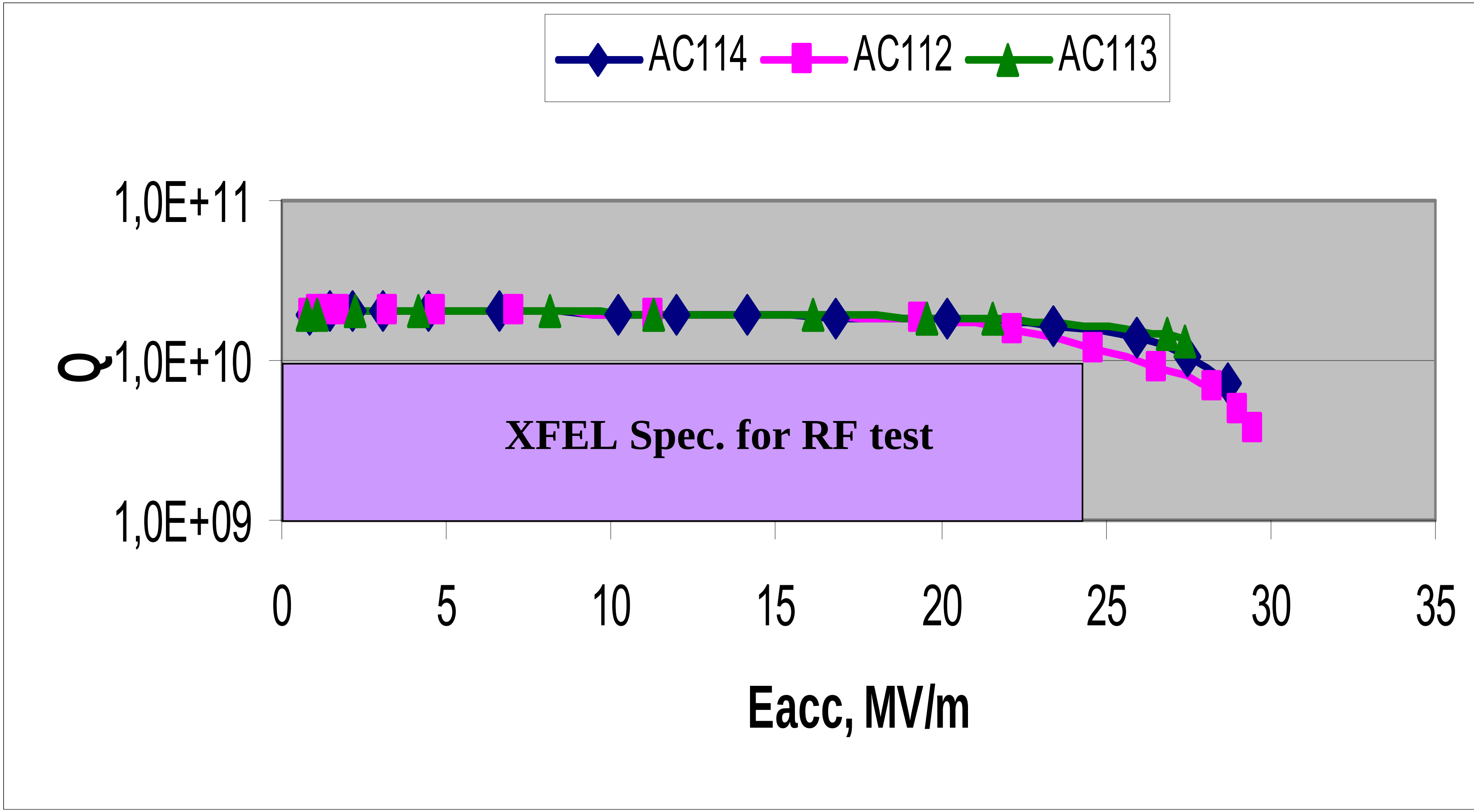


Frequency measurement of 6 end half cells (L and S) and 48 middle half cells (N) for cavities AC112-AC114. C - large crystal, W - Wah Chang, T - Tokyo Denkai. The shape conformity of half cells from large grain material is lower as of conventional fine grain (could be improved by correction of the tools), the uniformity of the half cells from large grain material is better.

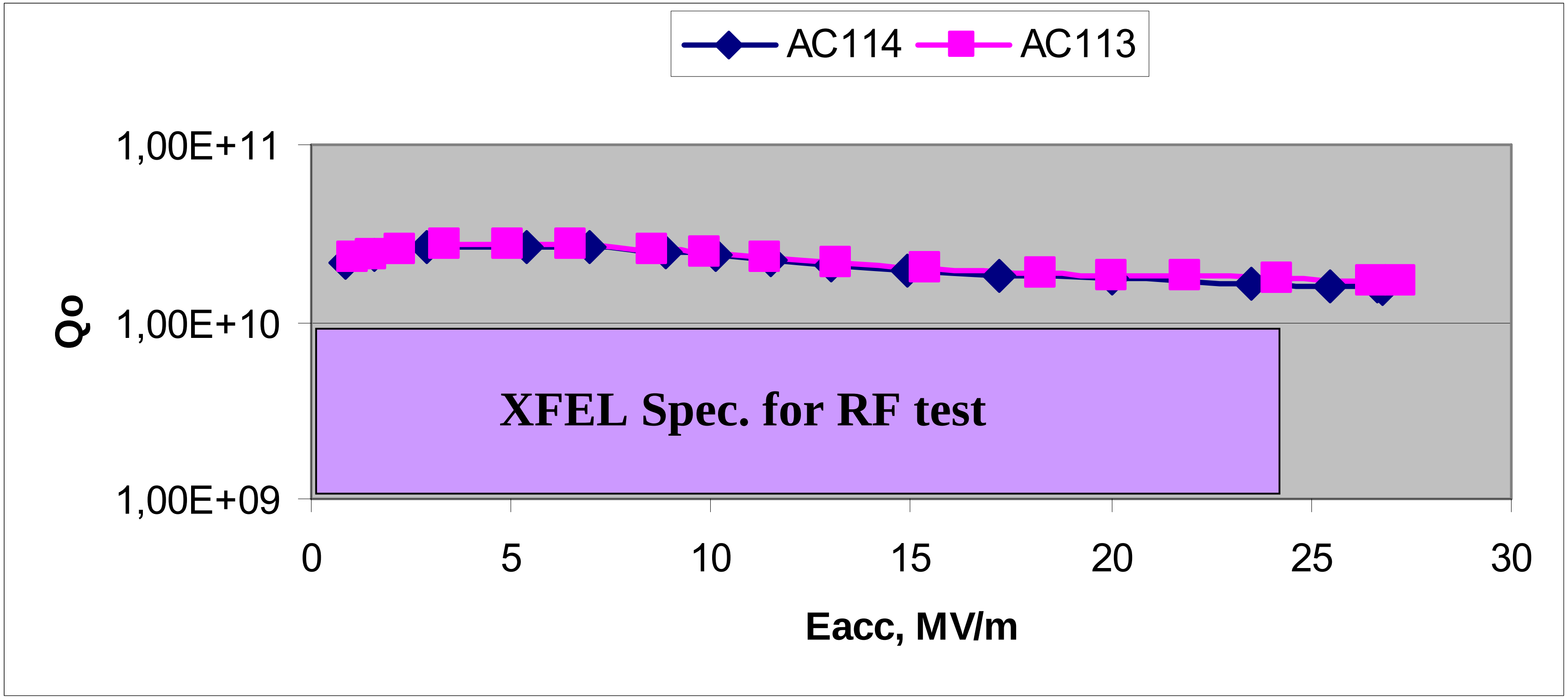
W. Singer, Cavities-COM, September 19-21, 2007

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

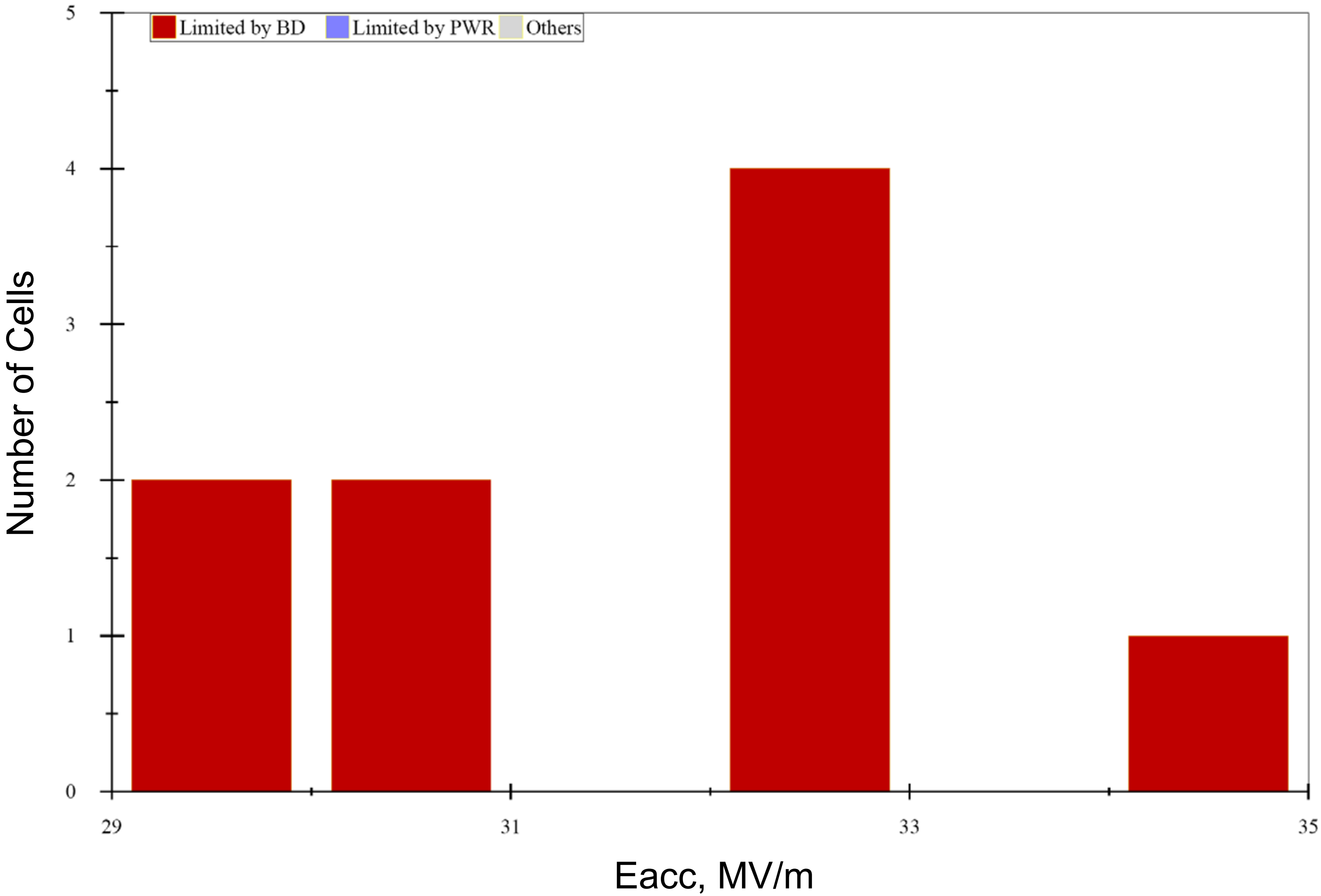


Second test Q(Eacc) curve of the LG nine cell cavities AC113-AC114 at 2K after additional 20μm BCP and 125°C, 50 h baking

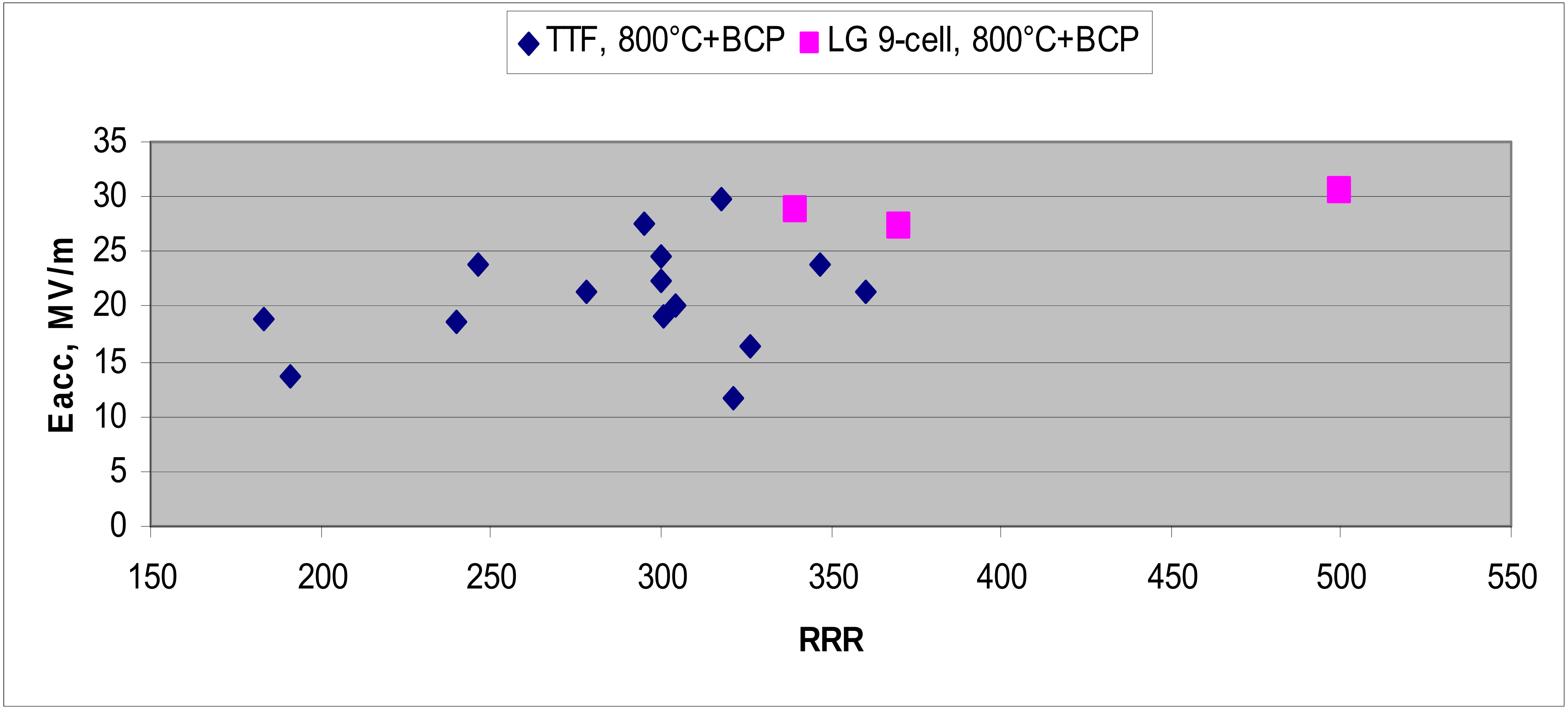


- Very similar behavior, good reproducibility

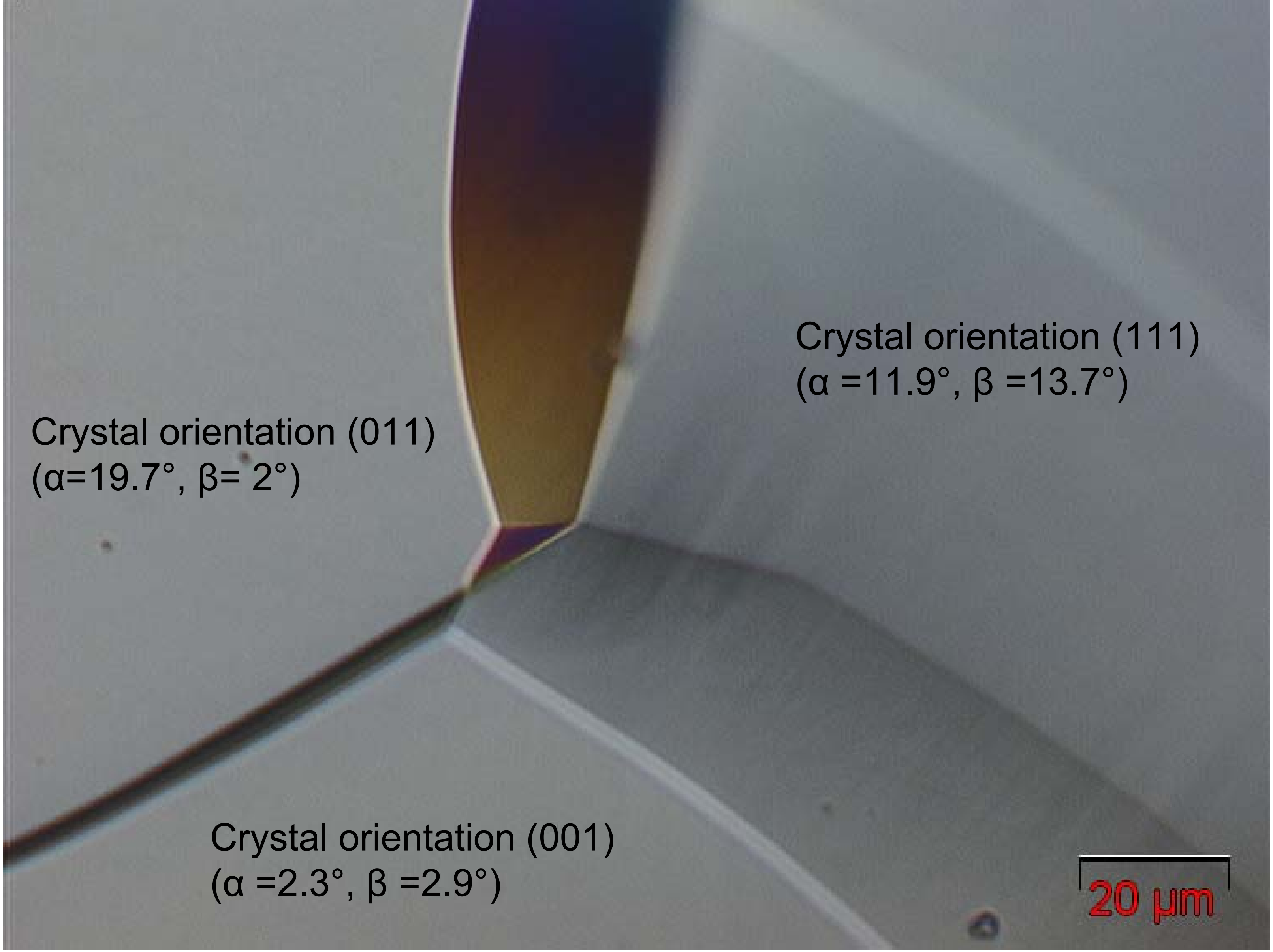
Cavity AC114 after baking; Mode measurement



W. Singer, Cavities-COM, September 19-21, 2007



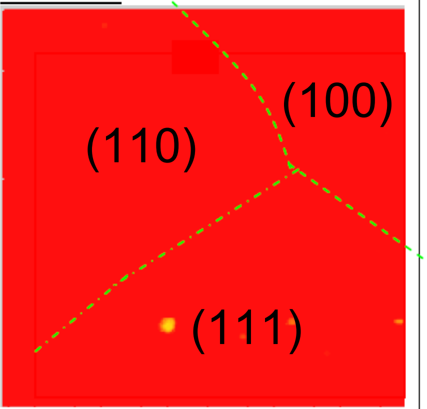
Comparison of the Eacc performance of large grain (LG) 9-cell cavities with similarly treated fine grain TTF cavities



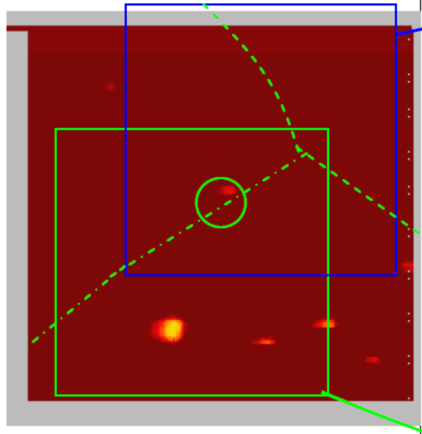
Light microscope image of LGs sample after 100 µm BCP.Mirror like surface of grains and steps on grain boundaries, dependent on crystallographic orientation of neighbor grains

W. Singer, Cavities-COM, September 19-21, 2007

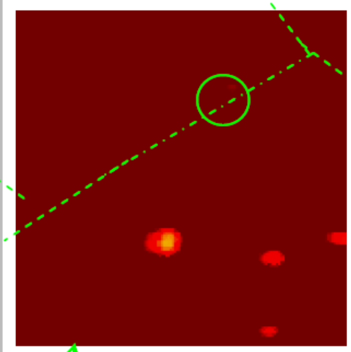
After HT



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

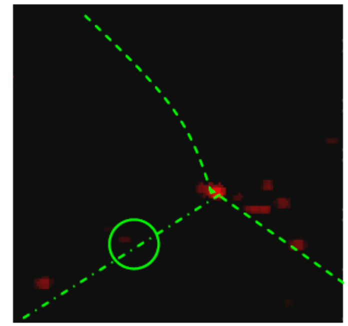


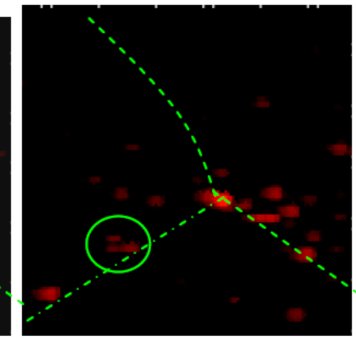
@ 150 MV/m, $(7.5\ mm)^2$



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

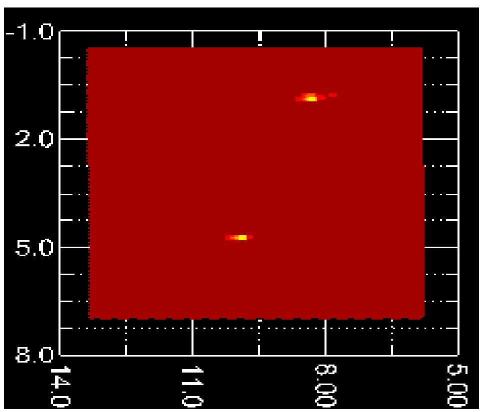
Encircled emitter is the one activated before HT, dotted lines in the scan show grain boundaries (GB)

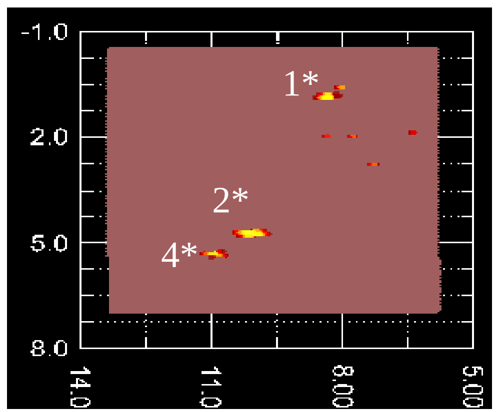




@ 250 MV/m, $(5\ mm)^2$ @ 300 MV/m, $(5\ mm)^2$
Increased number density after HT, all emitting sites appear near GB. Strongest emission observed at the intersection of three grain boundaries.

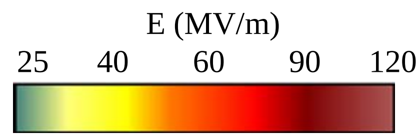
FE scans on three LG after 100 µm BCP and baking 150°C, 14hs





Dangwal, G.Mueller

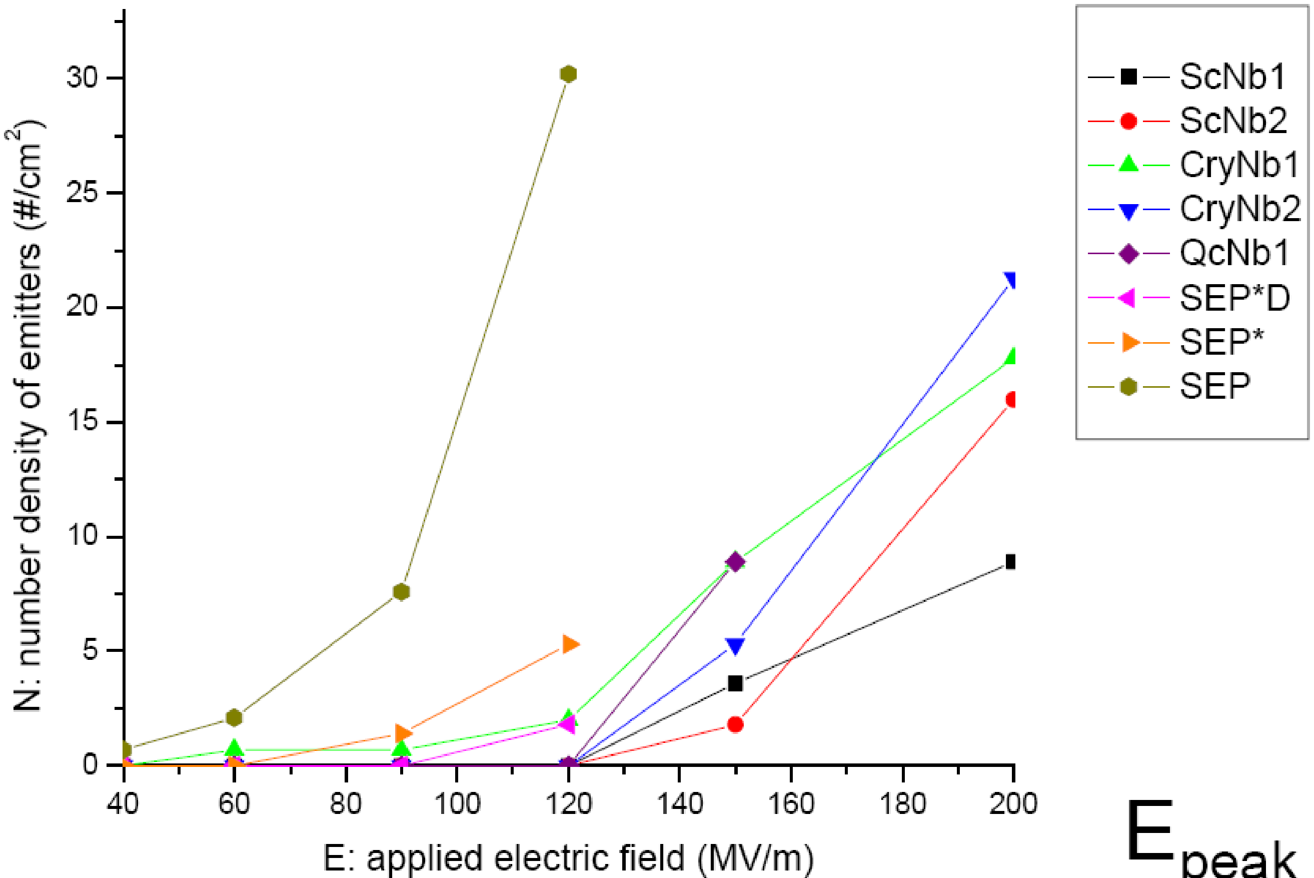
Wuppertal Univ.



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.

Emitter statistics for various types of Nb samples



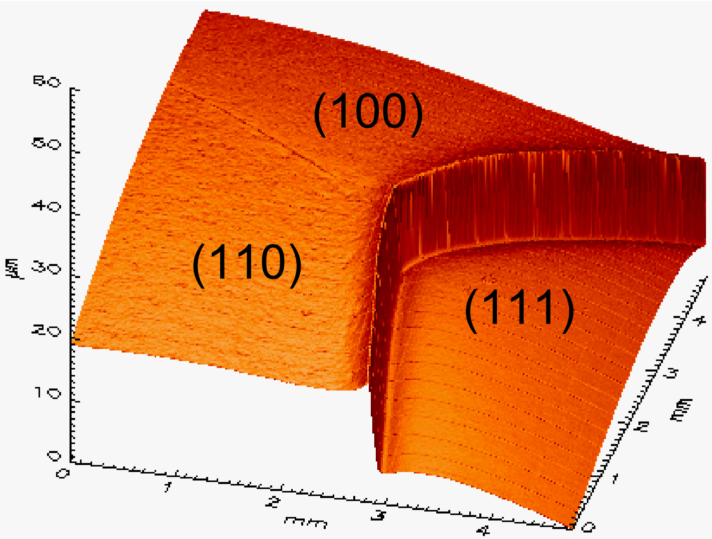
single crystal #1
& #2, BCP30HPR
3 large grains #2
& #1, BCP30HPR
EP+HPR at DESY
EP+HPR+DIC
EP+HPR
EP at Saclay

$$E_{peak} = 2 \times E_{acc}$$

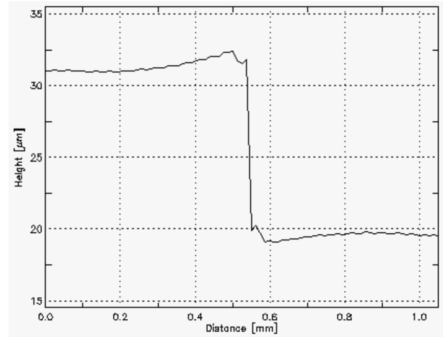
Systematically reduced FE by EP+HPR, DIC and large crystal Nb

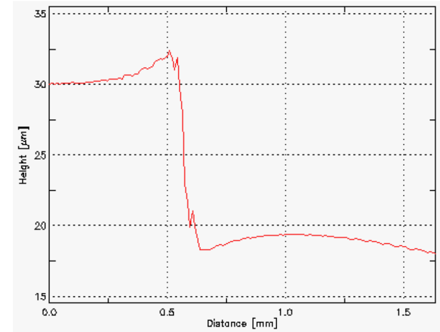
BCP+HPR of large crystal Nb is probably sufficient

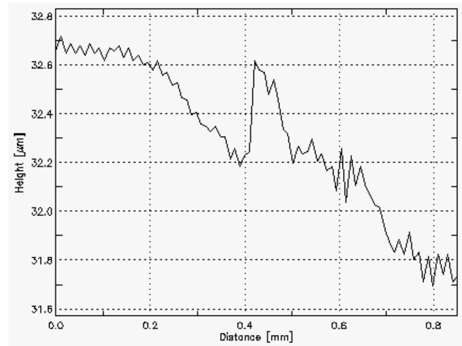
- Is large grain good enough, or do we have to have single grain?



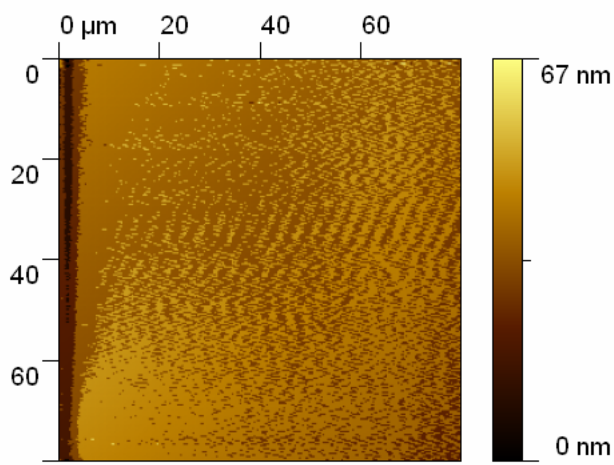
AFM image of LG Nb, BCP etched up to 100 µm

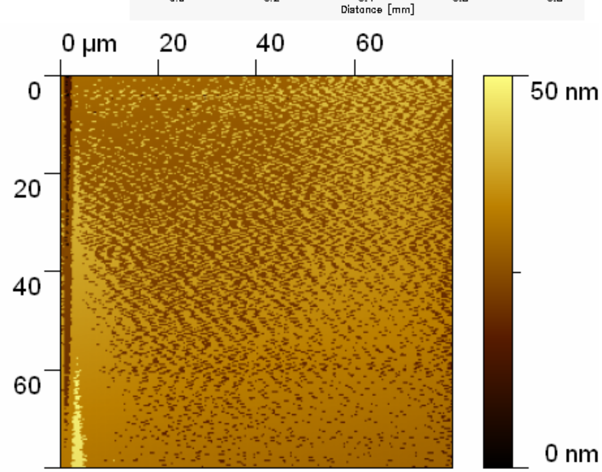


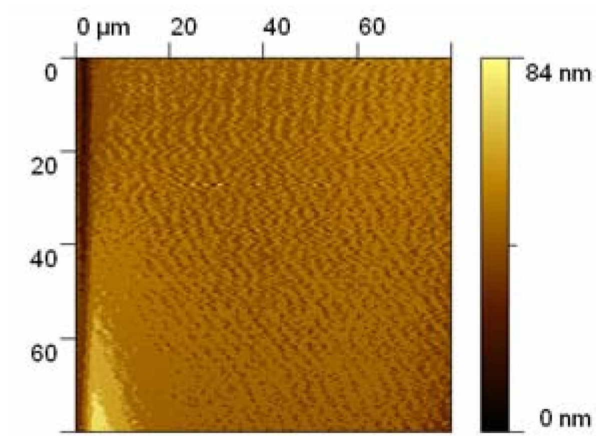




Profile and step heights of the three grain sample at the grain boundary intersection

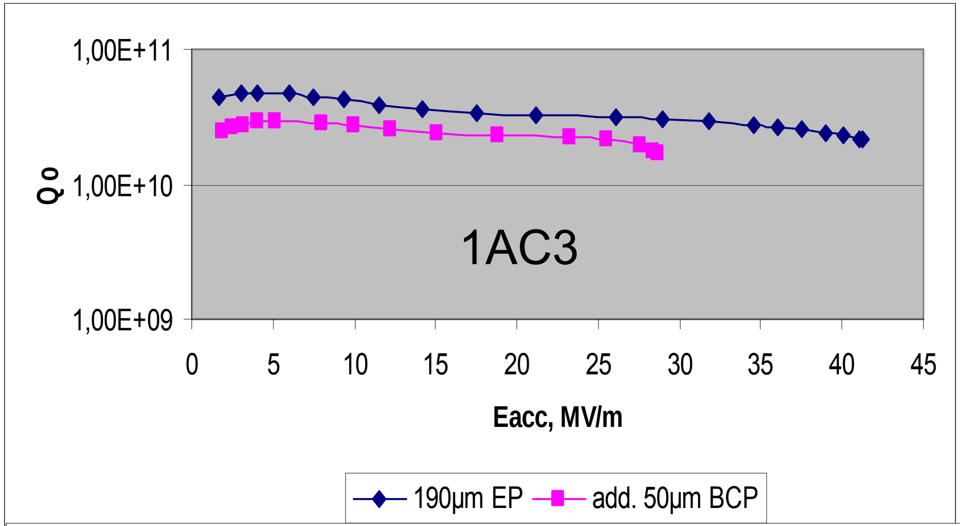


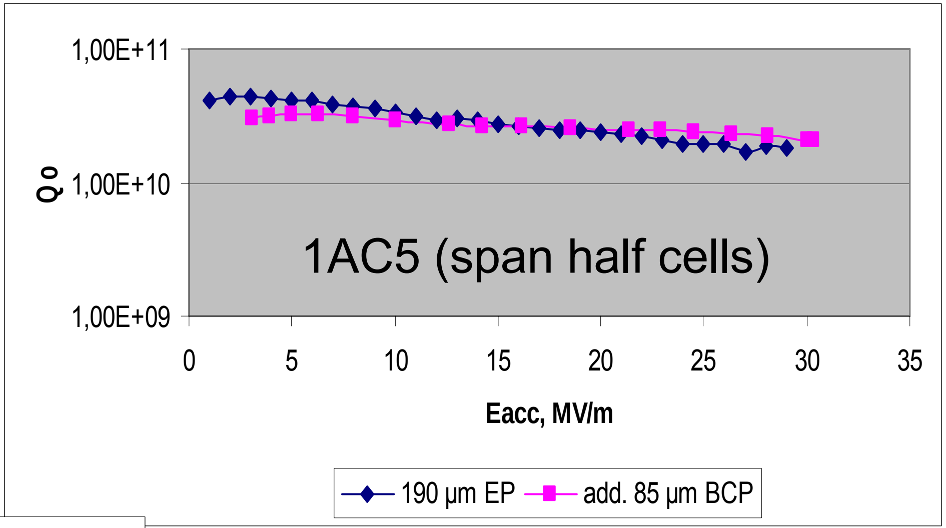


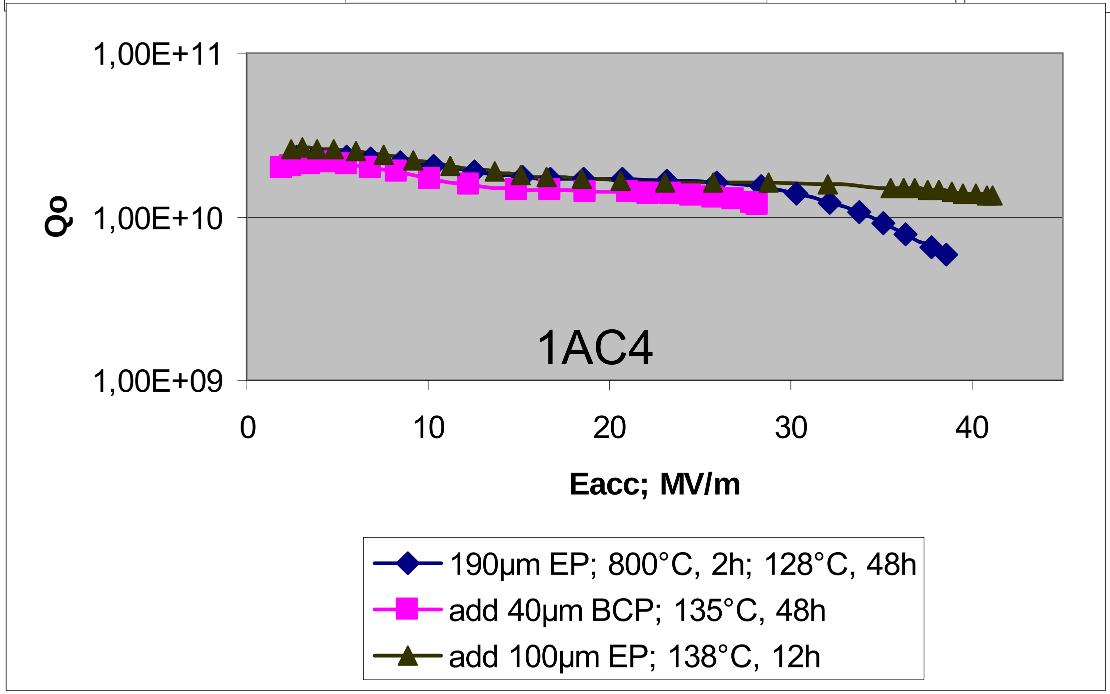


AFM roughness measurement (X. Singer, A. Dangwal-Pandey). Roughness of fine grain Nb after EP is 251 nm (A. Wu) .

- EP is planned as the next treatment step for LG AC112-AC114 cavities
- Experiences on single cell cavities; it seems that EP works better







Q(Eacc) curve of the single cell cavities 1AC3 -1AC5 after EP and BCP treatment

Preparation and RF tests of DESY cta

LG: Conclusions and outlook

- Fabrication of multi cell cavities from large grain niobium by deep drawing and electron beam welding is feasible. DESY produced three 9-cell cavities; order of new 8 LG 9-cell cavities is in work.
- Performance 29 - 35 MV/m can be achieved after BCP treatment only (ca. 5 MV/m more as similar treated fine grain).
- Baking at ca 120°C works for BCP treated LG cavities (better as for BCP treated fine grain cavities)
- Higher onset of field emission for LG compare to EP fine grain is observed
- Materials of different companies with different properties (RRR,Ta..) and prepared by different cutting methods behaved very similar after BCP only (less performance scattering is to expect).
- Scanning of LG discs could be possibly eliminated. AC112 not scanned. More statistic is required
- High gradient performance for EP treated LG cavity is similar to EP treated fine grain cavities. Up to $B_{p,max}$=175 mT (41 MV/m for TESLA shape) measured on a LG single cell cavities after EP.

Drawbacks

- The cut of the discs from ingot by saw cutting or EDM is not a cost effective option.
- Simultaneously cutting of many discs by diamond saw procedure seems to be cost effective. Only one company has it available at the moment.
- Big central single crystal in the disc seems to be mandatory for series production. Only one company is in position produce such ingots on more or less mass production scale. Some companies are working on this, but no real break through can be expected without substantial investment in this option.
- There are some unofficial estimation that LG Nb could be ca. 20% cheaper compare to fine grain Nb. It is not proven.

Single Crystal Cavities SC

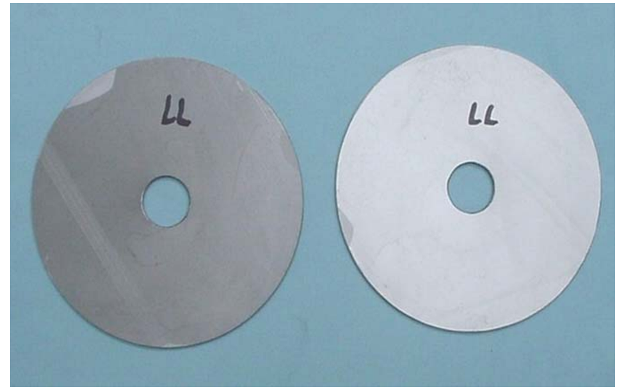
HG Cavity Shape:2.3 GHz

E_{peak}/E_{acc} = 1.674

H_{peak}/E_{acc} = 4.286 mT/MV/m

Single Crystal Cavities (JLab): CBMM Ingot"A" (111)

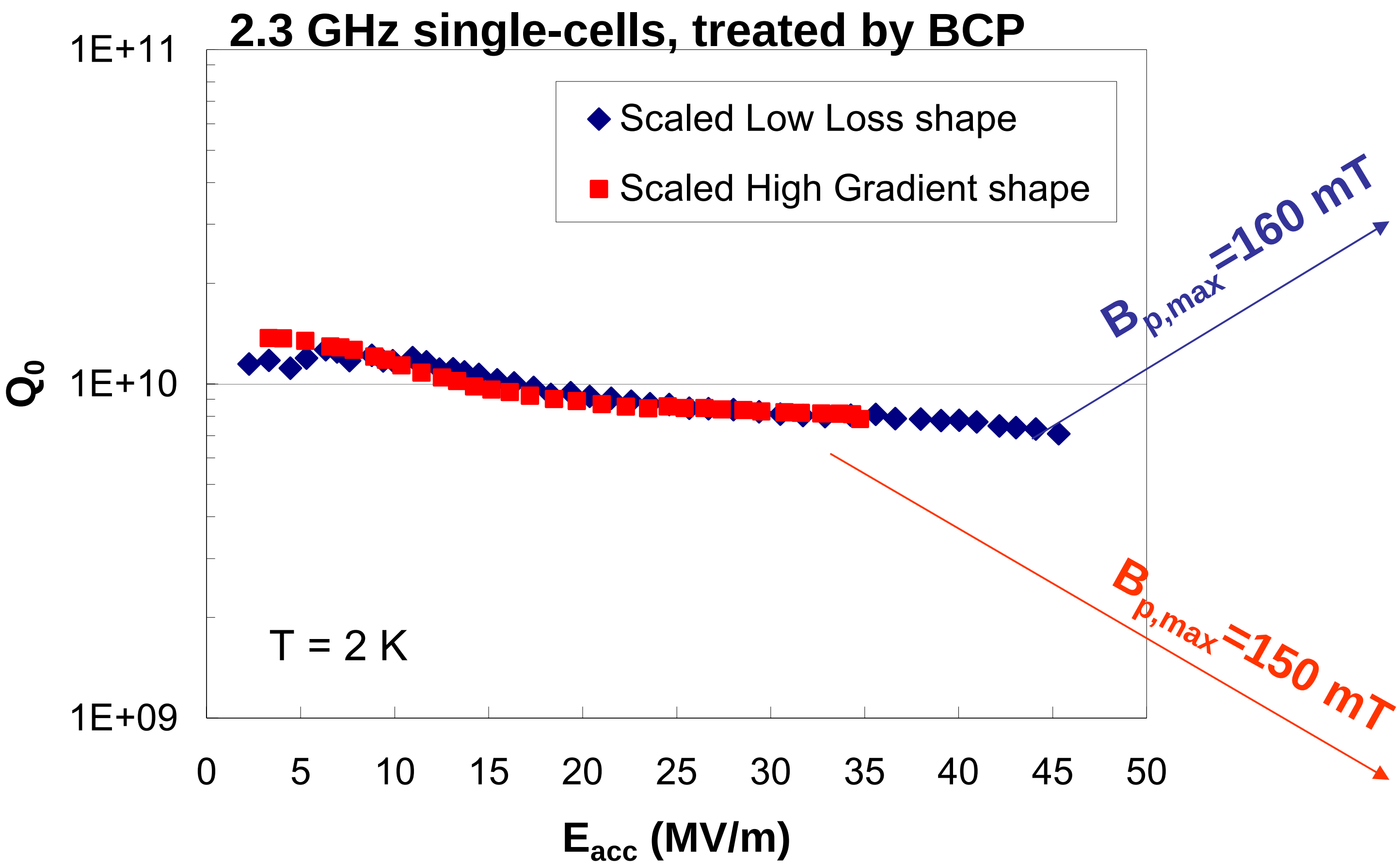
ILC LL cavity Shape:2.3 GHz



E_{peak}/E_{acc} = 2.072

H_{peak}/E_{acc} = 3.56 mT/MV/m

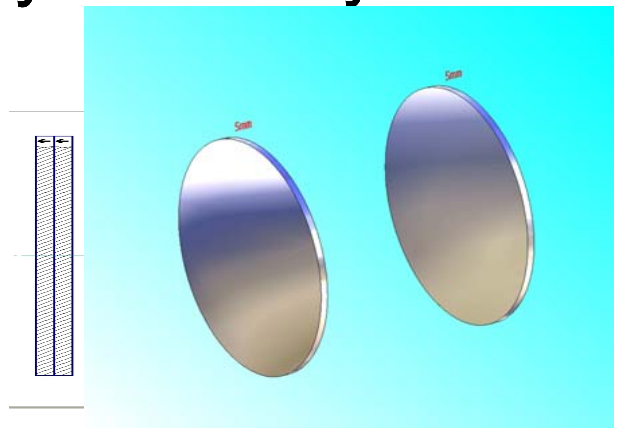
Single Crystal Cavities (JLab): CBMM Ingot"A" (111)



P. Kneisel et al., Proc. of PAC'05, Knoxville, TN, 2005, p. 399

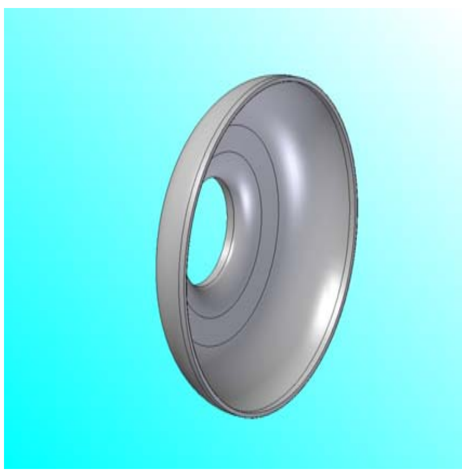
DESY Single crystal cavity fabrication

1. Take out central single crystal of definite thickness

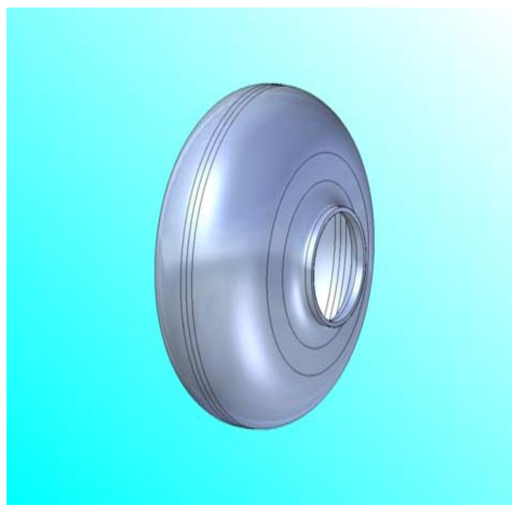


2. Cutting through the disc

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

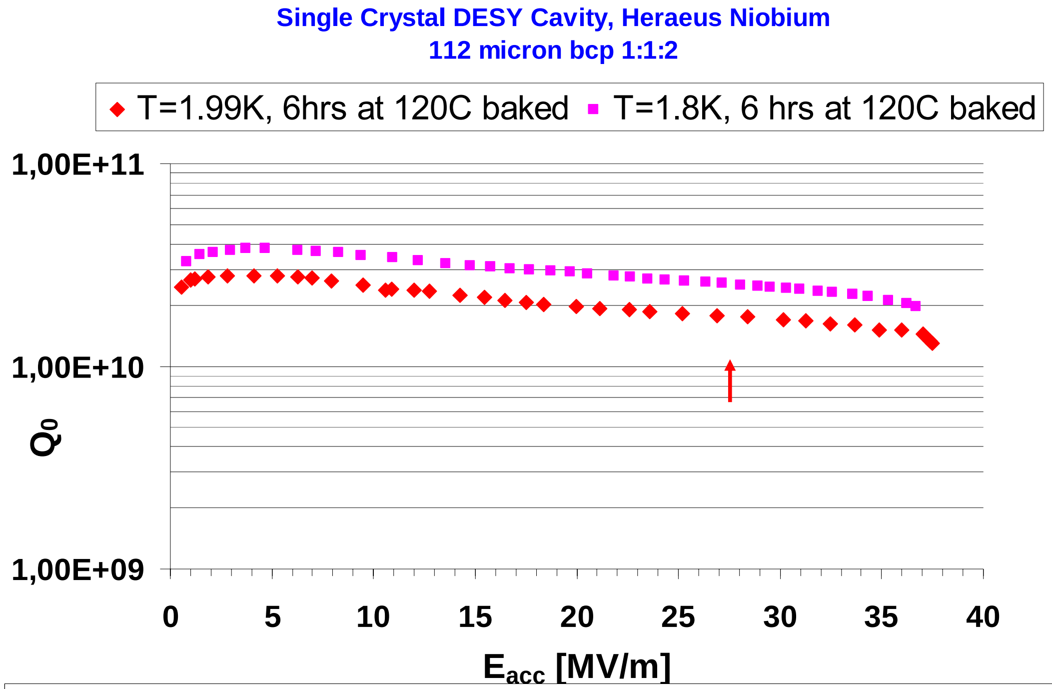


4. Deep drawing



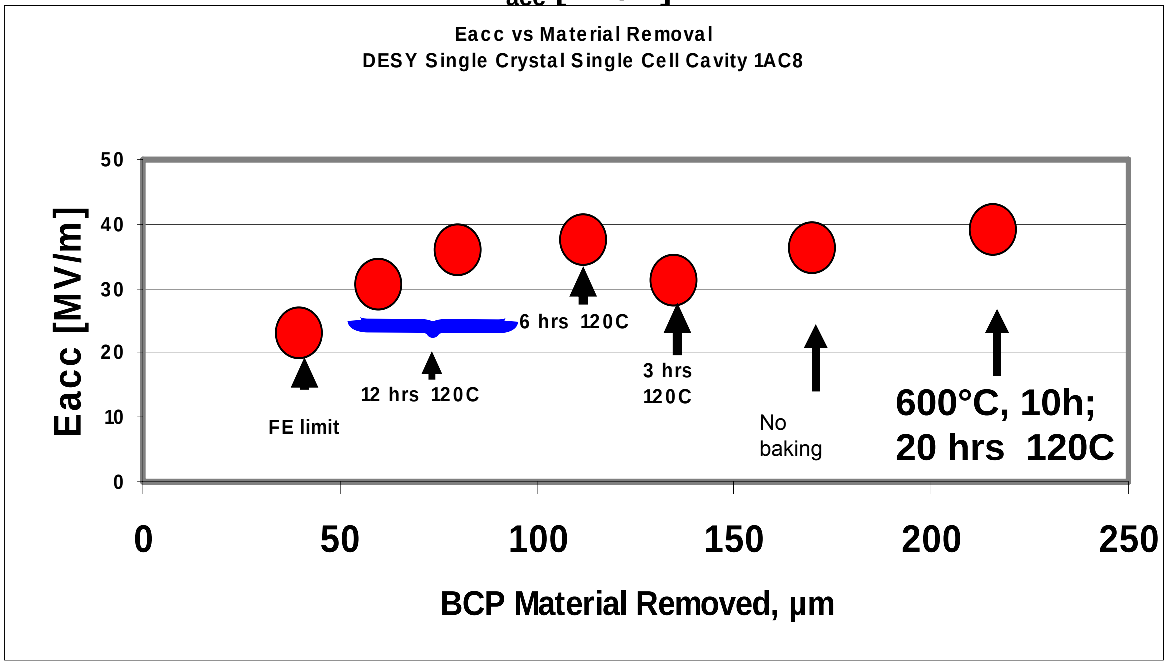
5. EB welding by matching the crystal orientation

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



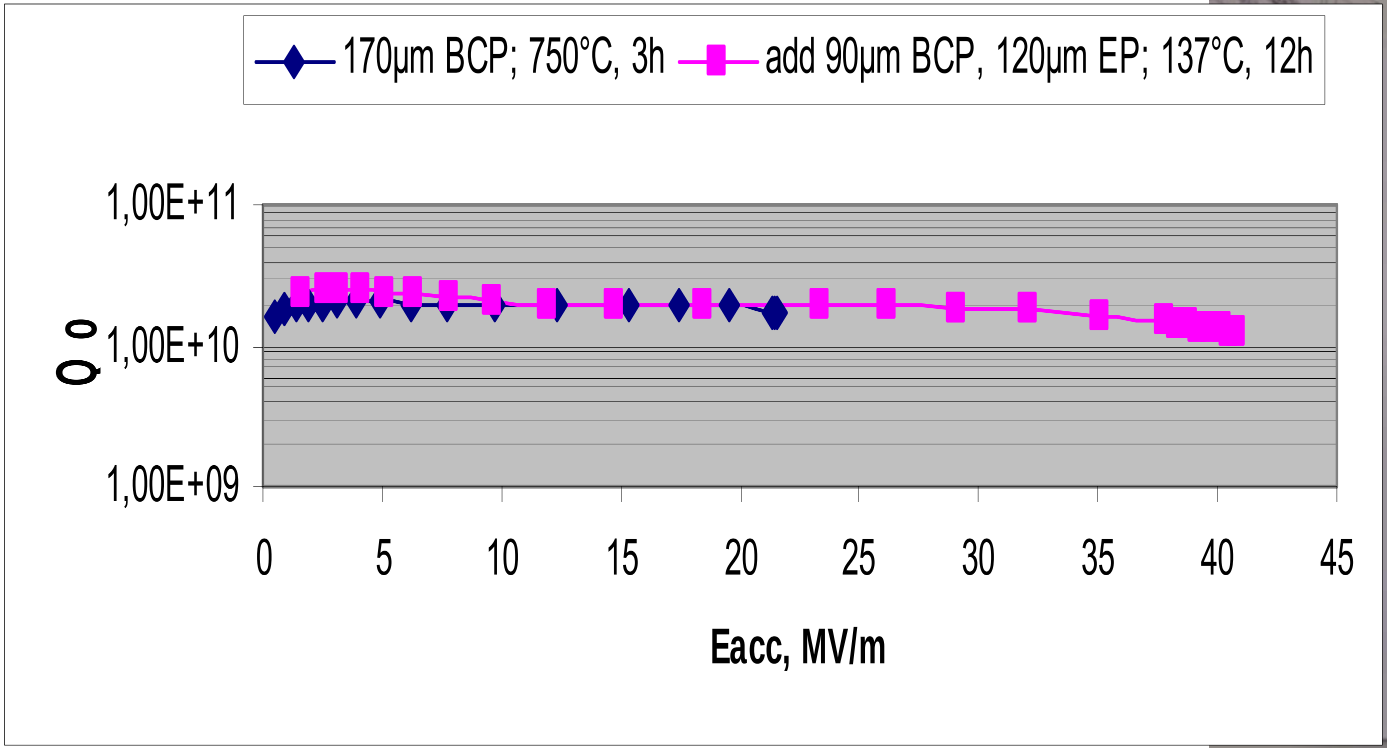
1AC8

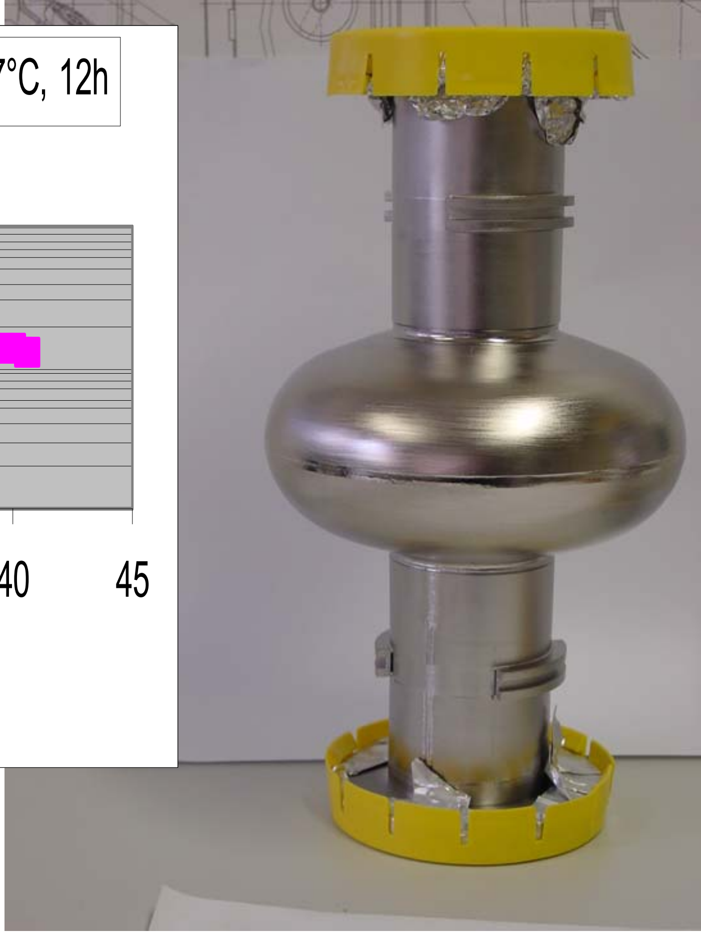
Q(Eacc) curve of 1AC8 after only 112 μm BCP and in situ baking 120°C for 6 hrs 37,5 MV/m (equivalent to 160 mT)



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

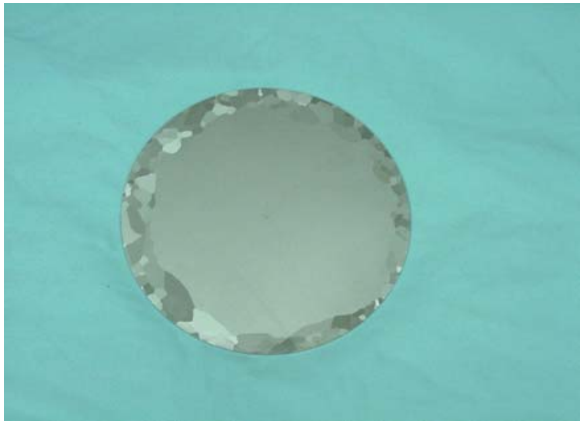
Preparation and RF tests of P.Kneisel, JLab





Q(Eacc) of DESY single crystal cavity 1AC6 at 2K ($B_{p,max}$=174 mT)

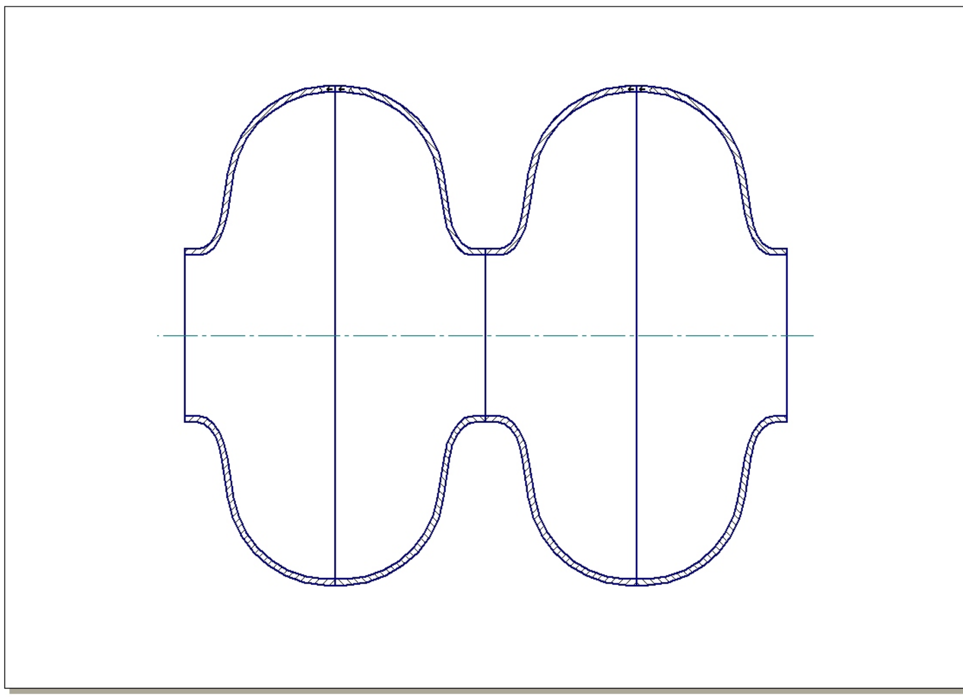
CBMM single crystal (diameter ca. 200 mm. Crystal orientation (111), 800 ppm of Ta, RRR=ca. 250)

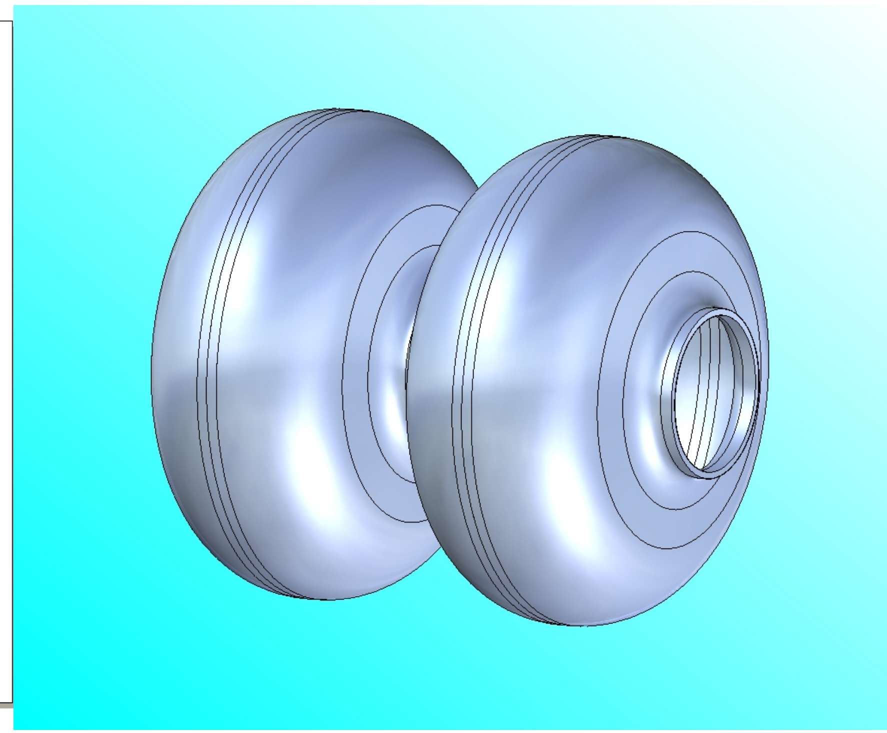


Single crystal single cell cavity 1AC6 fabricated by ACCEL (spinning and EB welding) from CBMM single crystal. Preparation and RF tests of DESY cta

SC. It works. The proposed method can be extended on fabrication of multi cell cavities.

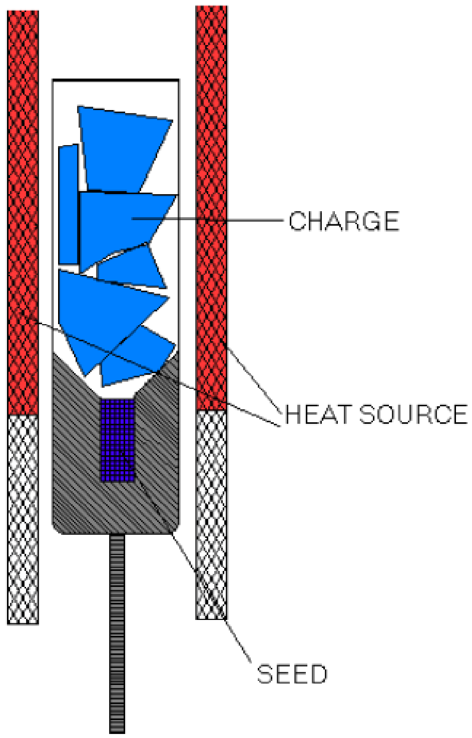
It is worthwhile to check the single crystal option for fabrication of multi cell cavities



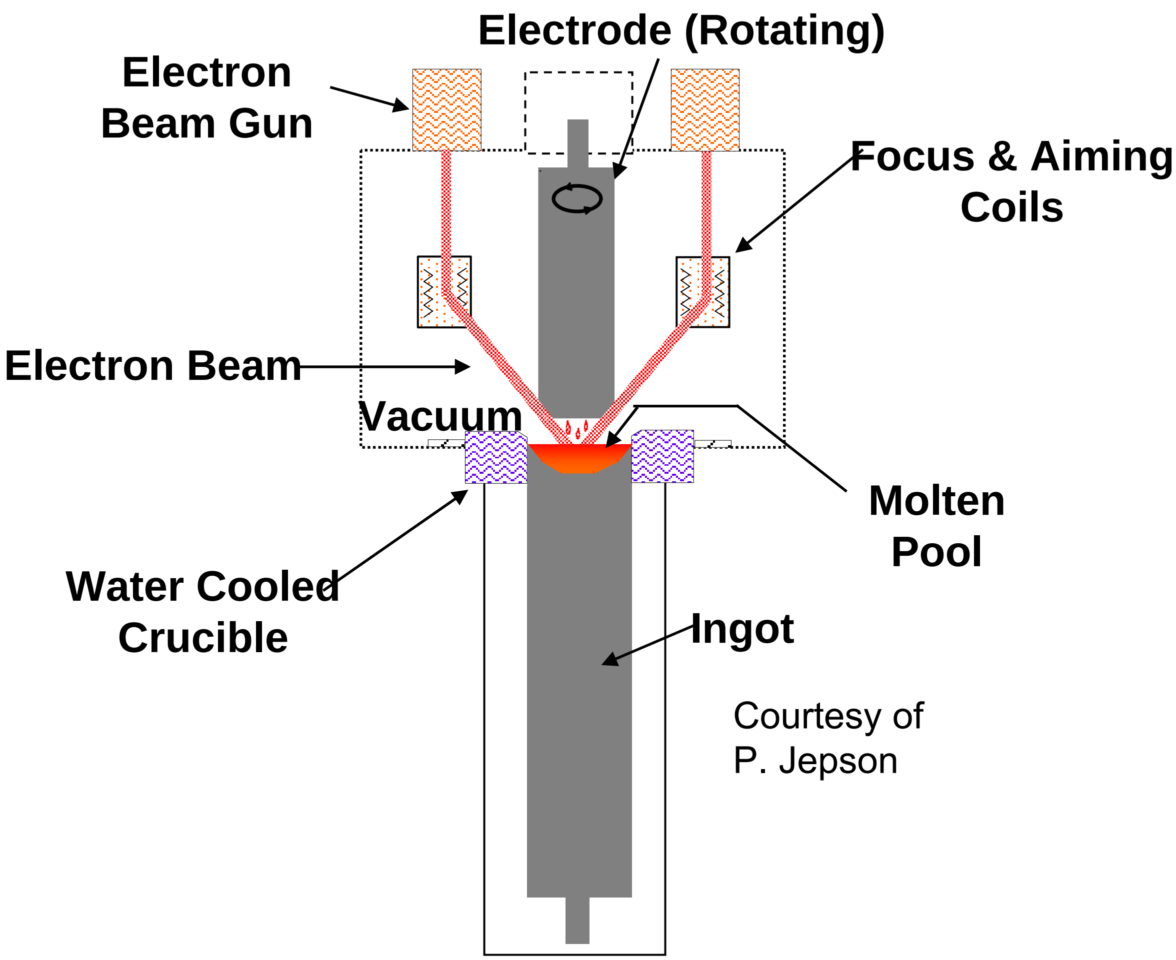


Is it realistic produce single crystal cavities of dimensions required for ILC?

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

Last HERAEUS Ingot of RRR300 with a central single crystal

W. Singer, Cavities-COM, September 19-21, 2007

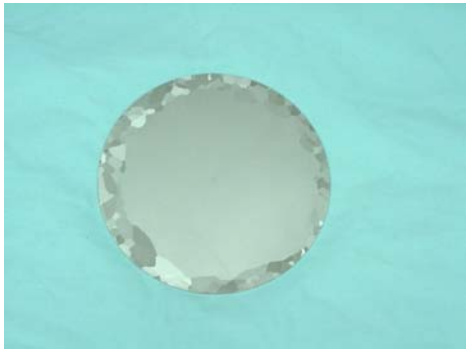
Question for SC

Does the crystal orientation play a role? What orientation will be preferable?

Experiences are not sufficient:

HERAEUS SC: (100) like orientation

Hquench 166 mT after BCP



CBMM SC: (111) like orientation

Hquench 174 mT after EP

Using the same crystal orientation will definitely provide reduction of performance scattering

SC: Conclusions and outlook

- **SC is the most exciting option**

- The first results on single crystal cavities produced by enlargement of the single crystal Nb discs are very promising. It seems that performance of BCP treated and EP treated SC cavities are comparable (in opposite to LG)
- Higher onset of field emission for SCs compare to EP fine grain is observed
- Simplification of the treatment procedure (BCP only) and reduction of performance scattering can be expected. No statistic is available. More work in this direction is very desirable.
- It is worthwhile to check the single crystal option for fabrication of multi cell cavities.
- It seems not to be realistic that single crystal discs of required dimensions (diameter ca. 265 mm) will be produced in short term. An essential investment is necessary.

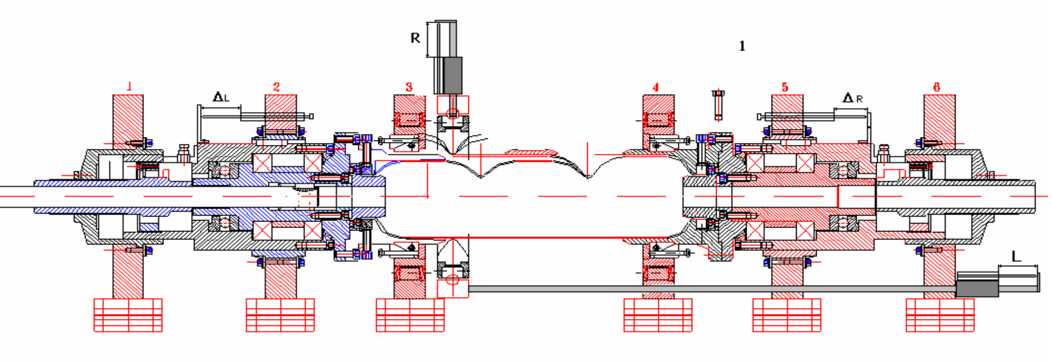
NbCu clad cavities

Fabrication of cavity from bimetallic bonded NbCu tube by seamless technique (spinning or hydroforming).

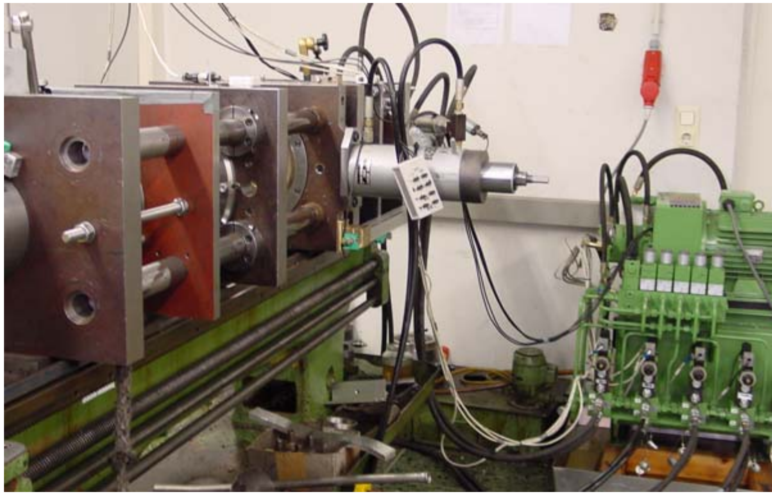
Advantages of NbCu clad option

- cost effective: allows saving a lot of Nb (ca. 4 mm cavity wall has only ca. 1 mm of Nb and 3 mm Cu). Especially significant for large projects like ILC. **NbCu clad cavity cost /Nb cavity cost = 70.5 % (W.S. estimation)**
- bulk Nb microstructure and properties (the competing sputtering technique does not have such advantages)
- the treatment of the bulk Nb BCP, EP, annealing at 800°C, bake out at 150°C, HPR, HPP can be applied (excluding only post purification at 1400°C).
- high thermal conductivity of Cu helps for thermal stabilization
- stiffening against Lorentz - force detuning and microphonics can be easily done by increasing of the thickness of Cu layer.
- fabrication by seamless technique allows elimination of the critical for the performance welds especially on equator

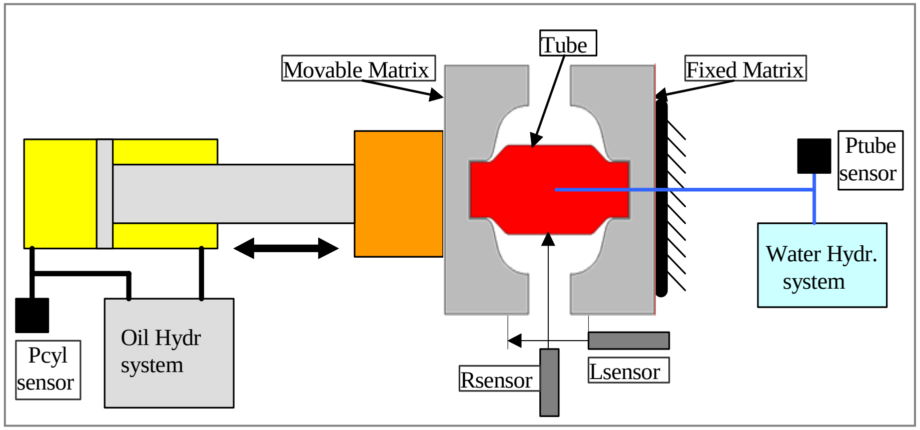
Hydroforming technique



Principle of tube diameter reduction in the iris area (necking)



Necking equipment



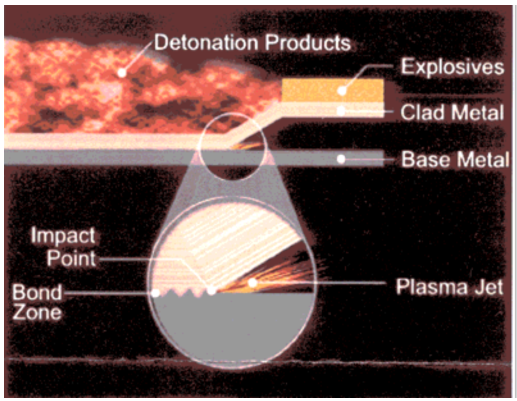
Principle of hydroforming

Hydroforming machine HYDROFORMA

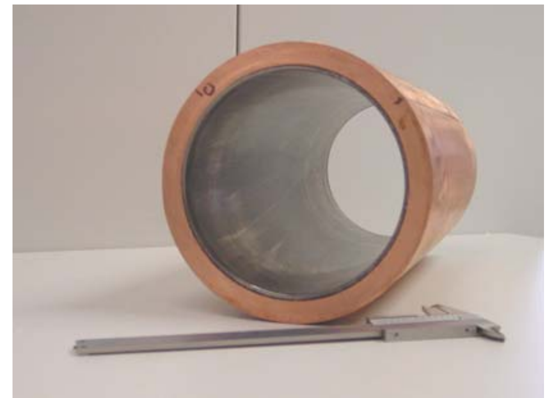
Explosively bonded NbCu tubes:

-Explosive bonding of seamless Nb tube ca. 4 mm wall thickness (RRR=250) with Cu tube of wall thickness 12 mm

- Flow forming into NbCu tube, wall thickness ca. 1mm Nb, 3 mm Cu

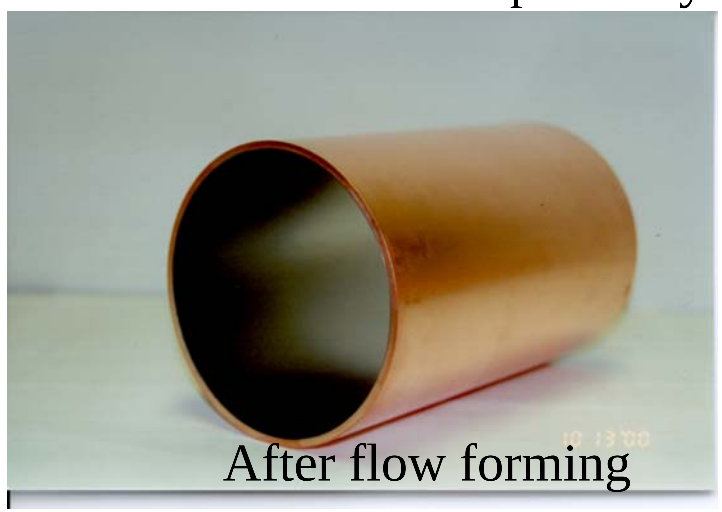


Bonding takes place by an explosively driven, high-velocity angular impact of two metal surfaces.

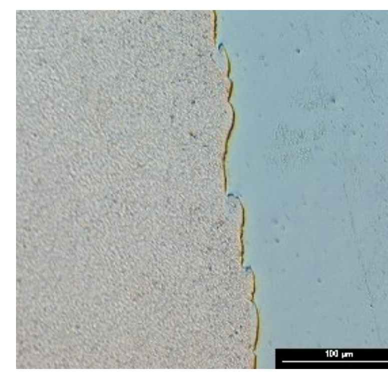


Explosively bonded NbCu tube

Flow forming of NbCu tube

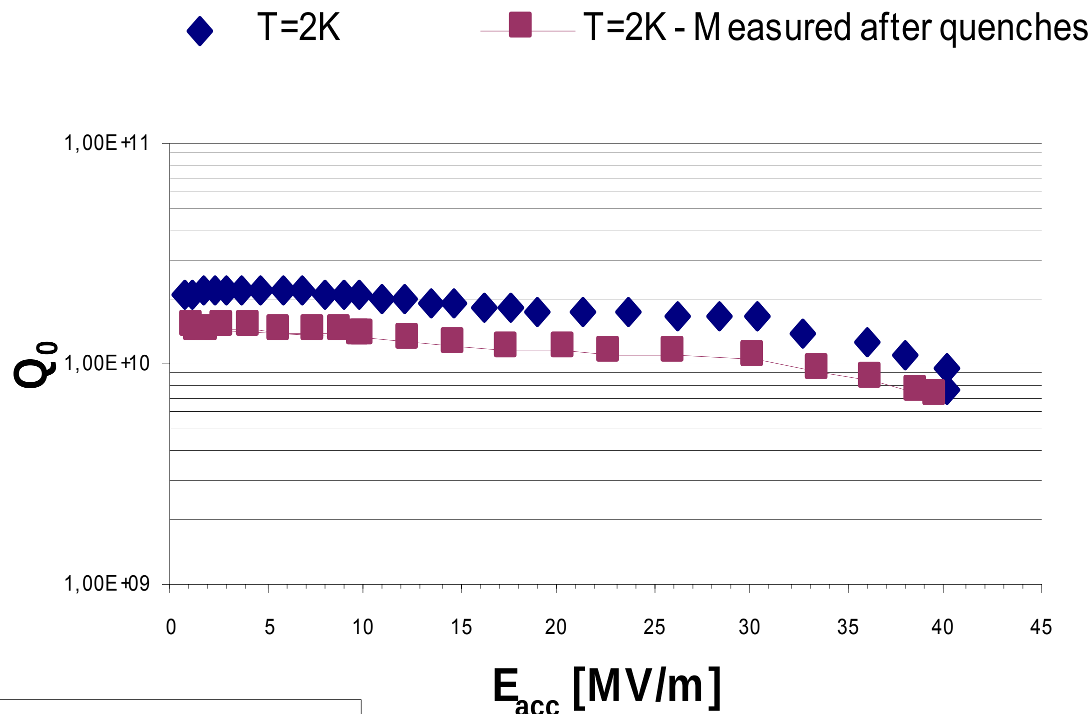


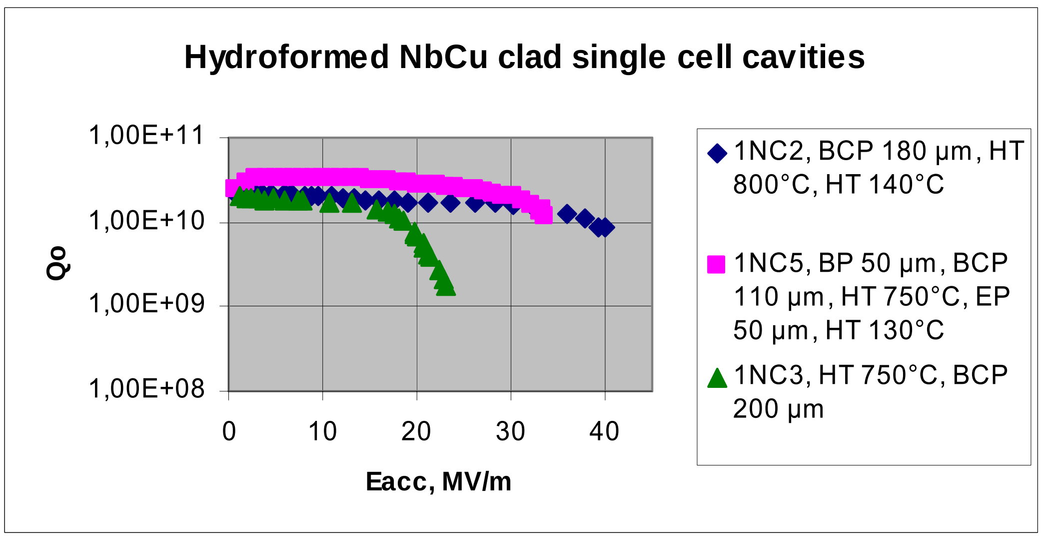
After flow forming



Structure of Nb/Cu interface

NbCu single cell cavity 1NC2 produced at DESY by hydroforming from explosively bonded tube. Preparation and HF tests at Jeff. Lab: 180 µm BCP, annealing at 800°C, baking at 140°C for 30 hours, HPR.



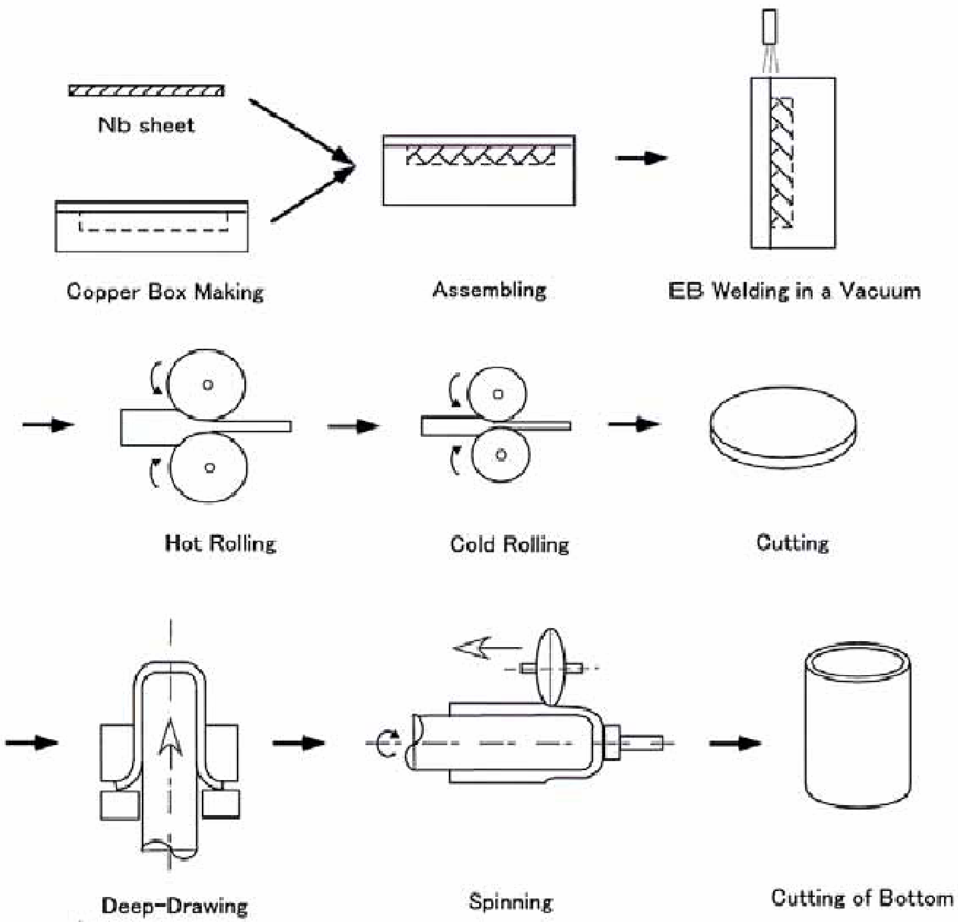


Big scattering in performance. Difficult to get reproducibly high bonding quality (one cavity failed). Difficulties by fabrication of multi cell cavities

Hot bonding fabrication procedure of NbCu tubes seems to be more promising

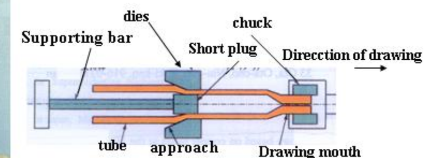
Preparation and RF tests at JLab, KEK, DESY

Hot bonded NbCu tubes

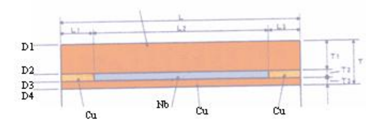


Fabrication principle of sandwiched hot rolled Cu-Nb-Cu tube (KEK and Nippon Steel Co.)

Hot roll bonded Cu-Nb-Cu tube produced at Nippon Steel Co.



Principle of the tube drawing technology



Cu-Nb-Cu Sandwiched Tubes (KEK)

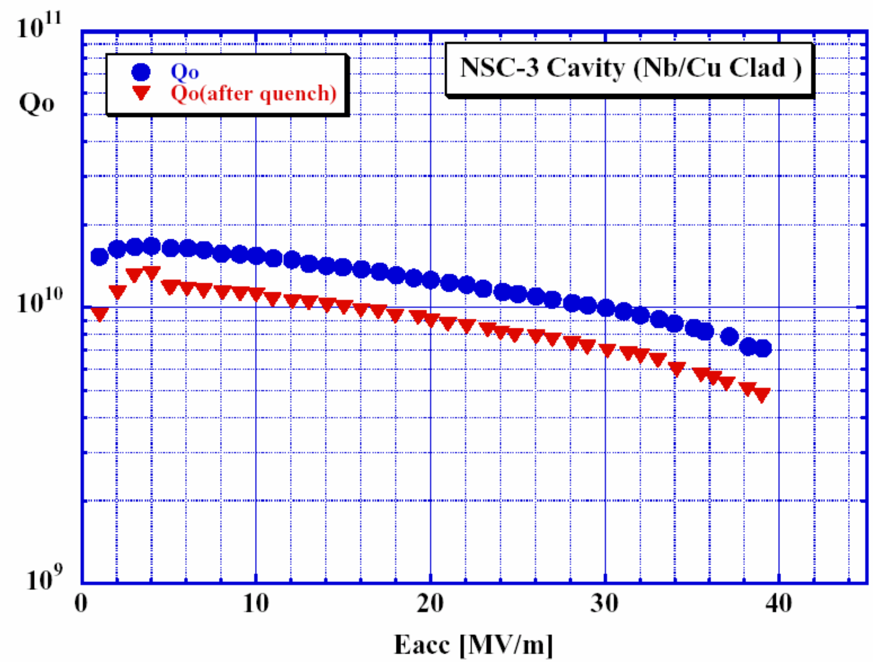
Fabrication principle of sandwiched coextruded Cu-Nb-Cu tube (KEK)

Best RF tests result

Single cell and multicell NbCu cavities produced at DESY by hydroforming from KEK sandwiched tube.

Quality of hot bonding is better compare to explosive bonding

Single cell cavity. Hot roll bonded tube fabrication at Nippon Steel Co., hydroforming at DESY, Preparation and RF tests at KEK



NSC-3: Barrel polishing, CP(10 mμ), Annealing 750°C x 3h, EP(70 μm) K.Saito

Difficulties in NbCu technology

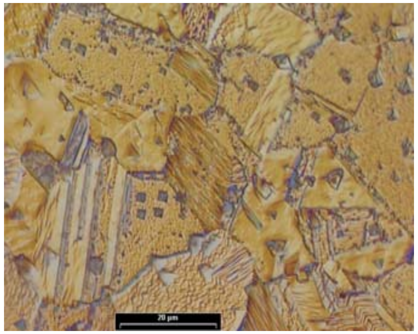
Dangerous of cracks appearance in iris area during fabrication (because of big difference in recrystallization temperature of Nb and Cu)

Microstructure of Cu and Nb after annealing at 560°C for 2 hours. Nb is not recrystallysed (hard).

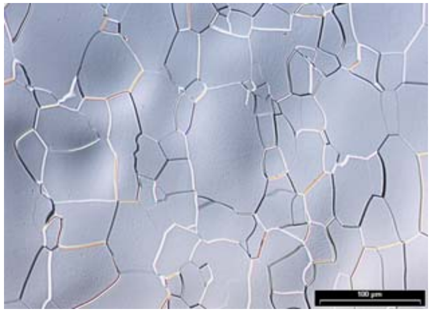
Two ways to defeat the cracks

a) Sandwiched tube (Nb is between two Cu layers. Cu layer on both sides prevent creating of cracks in Nb); removing of inside Cu layer on the cavity after forming (K.Saito). The option was checked, it works

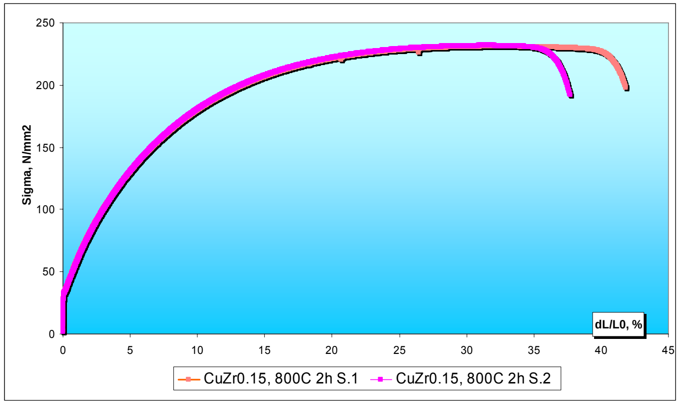
b) Using special Cu with high recrystallization temperature



Microstructure of Cu0.15%Zr (left) and Nb (right) after annealing at 800°C for 2 hours.



Stress –strain behavior and thermal conductivity of Cu0,15%Zr after annealing at 800°C for 2 hours compared with Cu and Nb.



The Cu0.15%Zr shows a high elongation after annealing at 800°C, small and rather uniform grain and can be a good candidate for replacing of pure Cu in NbCu clad tubes

Thermal conductivity can be recovered by aging at ca. 400°C/one hour. Zr leave the solid solution and creates precipitates Cu_5Zr finely distributed in Cu matrix

Cu only outside: Cu0.15%Zr special Cu with high recrystallization temperature

Up to now hydroforming only of the sandwiched tube (Nb is between two Cu layers. Cu layer on both sides prevent creating of cracks in Nb); removing of inside Cu layer on the cavity after forming chemically (costly)

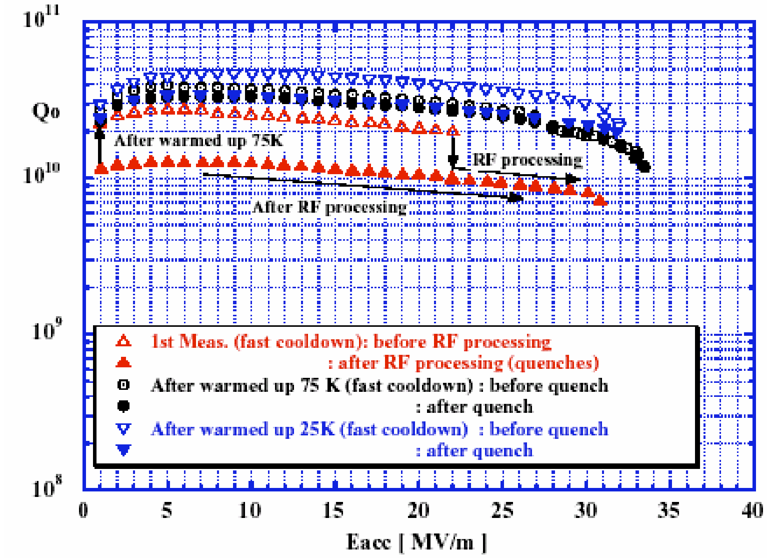
NbCu0.15%Zr tube

Single cell cavities produced from Nb/ Cu0.15%Zr clad tube

Drawback: Q degradation during fast cool down, RF processing or quench (trapping of magnetic flax caused by thermo - coupling effect).

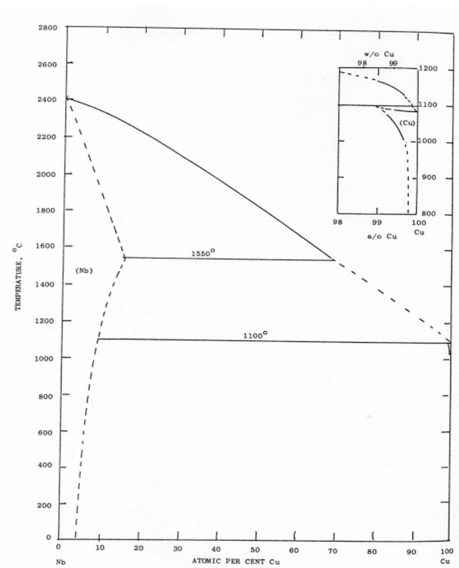
- Is not completely understood, more work is necessary. Similar effect was observed on Nb_3Sn cavities (M.Peiniger). Similar effect is to expect in sputtered NbCu cavities.

- The Q degradation can be cured by warm up over Tc and slow cool down.

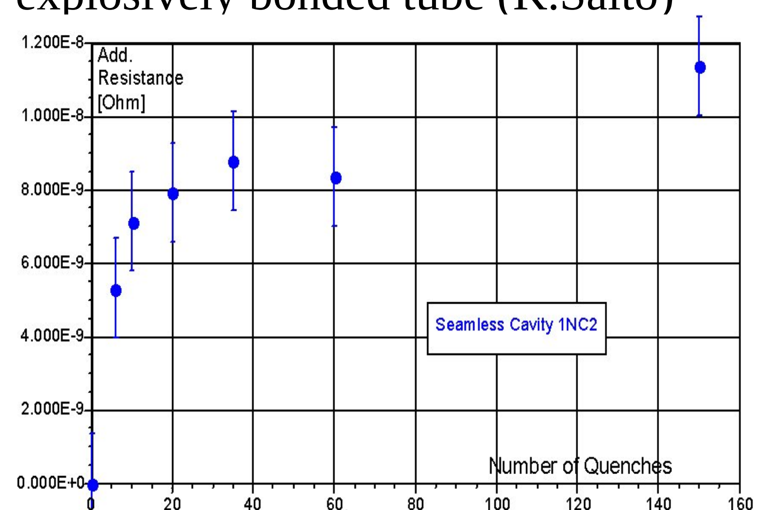


Q degradation in a seam less single cell cavity from NbCu explosively bonded tube (K.Saito)

- It seems that annealing and hot bonding (due to diffusion) should contribute to reduction of the thermal coupling effect

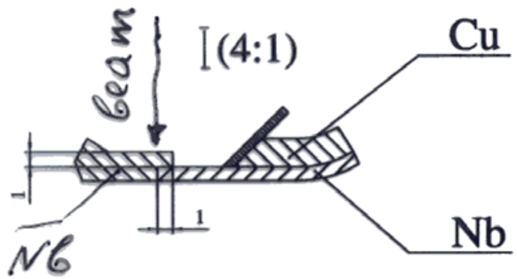


NbCu phase diagram



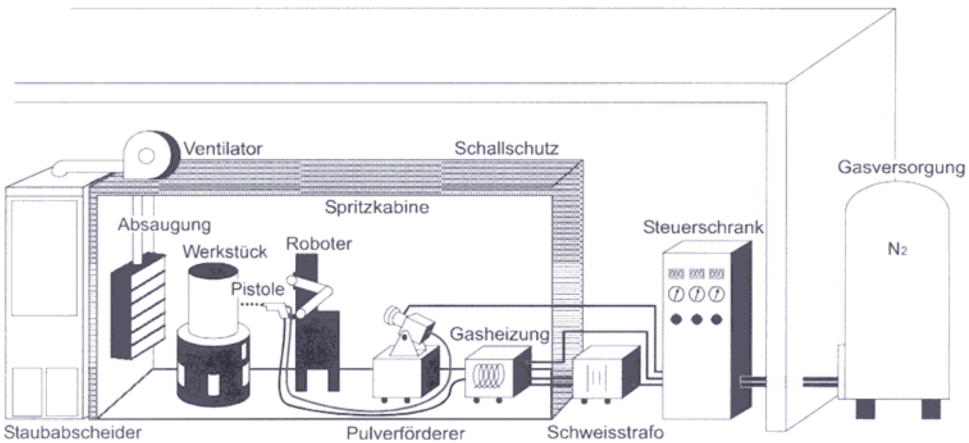
Additional surface resistance after quenches at ca 40 MV/m (P.Kneisel)

Connection of end tubes to NbCu cells

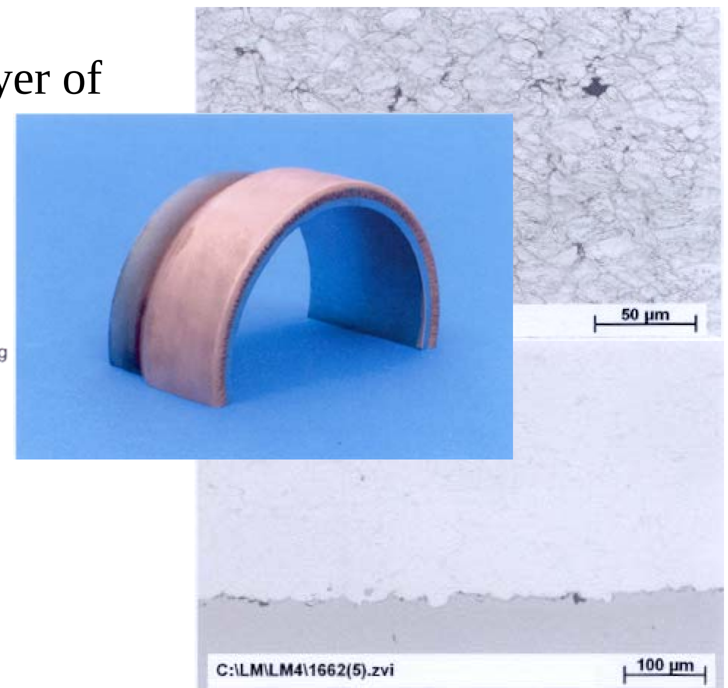


Principle of the welding of 0,7-1 mm thick Nb layer of the cavity with 2 mm thick wall of Nb end tube.

Cu layer on Nb tube. Small purity degradation of Nb during spraying: from RRR=300 to RRR=250



New Cold Gas Dynamic Spray System. Warms up the particles by only ca. 300°C and accelerate to velocities of 600-1500m/s (H.Kreye, University of the Federal Armed Forces in Hamburg)



Microstructure of CGDS Cu coating on Nb, small porosity ca. 1%, small oxidation. electr. conduct. ca.80% of bulk Cu (top: spraying by Nitrogen, bottom- by Helium)

Conclusions and outlook to NbCu

- Potentially a cost effective option. NbCu clad cavity cost /Nb cavity cost is ca. 70 %

- Fabrication of single cell and multi cell cavities as proof of principle is done

- Eacc of up to 40 MV/m can be reached on NbCu clad cavities

Drawbacks:

-Q degradation during fast cool down, RF processing or quench (trapping of magnetic flax caused by thermo - coupling effect). Nobody is working on this phenomena

- Development of the connection of end groups to NbCu cells is not finished