Progress on Large Grain and Single Grain Niobium – Ingots and Sheet and Review of Progress on Large Grain and Single Grain Niobium Cavities

Peter Kneisel
Jefferson Lab, Newport News, Virginia, USA

- Status at SRF 2005

- What has happened since then?

  - Investigations on Material
  - Cavity Fabrication and Test Results

Status at SRF 2005(1)

- Four papers discussed large grain/single crystal niobium (mechanical properties, flux penetration, cavity results)
- Several cavities of different shape and frequency from CBMM material had been tested;

  among those 2 single crystal cavities at 2.3 GHz with gradients up to E_{acc} = 45 MV/m ($H_{peak} \geq 165$ mT)
- Large grain cavities from Ningxia and Wah Chang material were awaiting testing
- DESY had started with material investigations on large grain niobium and had contacts with W.C.Heraeus as another source for large grain material

SRF 2005

What are the potential advantages of large grain/single crystal niobium?

- Reduced costs
- Comparable performance
- Very smooth surfaces with BCP, no EP necessary
- Possibly elimination of “in situ” baking because of “Q-drop” onset at higher gradients
- Possibly very low residual resistances (high Q’s), favoring lower operation temperature(B.Petersen)
- Higher thermal stability because of “Phonon-Peak”
- Good or better mechanical performance than fine grain material (e.g.predictable spring back..)
- Less material QA (eddy current/squid scanning)

What has happened since 2005?

- Large grain/single crystal technology has been a topic at every conference and workshop
- Material is now available from several source (TD will start soon)
- Many more labs have started with material – and cavity studies
- A “single crystal” workshop was held in Araxa, Brazil, Oct. 30 – Nov. 1, 2006
- Many single cell, single crystal and multi-cell cavities have been fabricated and tested from as much as 10 different ingots from 4 different producers with encouraging results

Material Studies

Subject	**Comment**	**Reference**
Mech. properties	YS,TS,elongation, bulging, residual stress	1,2,3
Thermal properties	Therm. Conductivity, phonon peak, effect of strain	4,5,1
Magn.,/electr. properties	Hc1,Hc2, Hc3 for diff. crystal orientations	14
Crystal orientation/recrystall.	Grain orientation in diff. material, etch rate, dislocation density	4,5,13
Forming/welding	**Increase large grain to single crystal any size**	6,7,8
Flux penetration	Influence of grain boundaries on flux penetration	9,15
Oxidation	**Diff. crystal orientations, oxide composition**	10,11,16
Field emission	**Emitter density, cleaning, grain boundary segregation**	12,17

Material Studies

References
[1] G.R.Myneni, AIP Conference Proceedings 927, p. 41
[2] W. Singer, https://indico.desy.de/conferenceDisplay.py?confld=200
[3] T. Gnaeupel-Herold et al.; AIP Conference Proceeings 927, p. 48
[4] C. Compton, AIP Conference Proceeings 927, p. 98
[5] W. Singer, AIP Conference Proceeings 927, p. 123
[6] W. Singer et al.; AIP Conference Proceeings 927, p. 133
[7] A. Ermanoe et al.; Eucas 2007
[8] V. Levits, http://tdserver1.fnal.gov/project/workshop/RF Materials/agenda.htm
[9] P. Lee et al.; AIP Conference Proceeings 927, p. 113
[10] A.D. Batchelor et al.; AIP Conference Proceeings 927, p. 72
[11] H. Tian, http://tdserver1.fnal.gov/project/workshop/RF Materials/agenda.htm
[12] G. Mueller et al.; JRA1/CARE Meeting Sept. 17-19, 2007, Warzaw, Poland
[13] C. Compton, paper TUP05
[14] W. Singer, paper TUP 12
[15] Z.H.Sung et al.; paper WE 105
[16] A. Romanenko, paper TU 103
[17] A. Dangwal et al.; paper WEP 24

Cavity Studies

- Work on large grain/single crystal cavities is going on at
  - Cornell University:single cell re-entrant
  - DESY:single cell, single crystal, 9-cell
  - Fermi Lab ?
  - Jlab : single cell, single crystal, 9-cell
  - KEK: single cell
  - MSU: single cell (in collaboration with Jlab)
  - PKU: single cell (in collaboration with Jlab)

Single Crystal/Large Grain Niobium Workshop

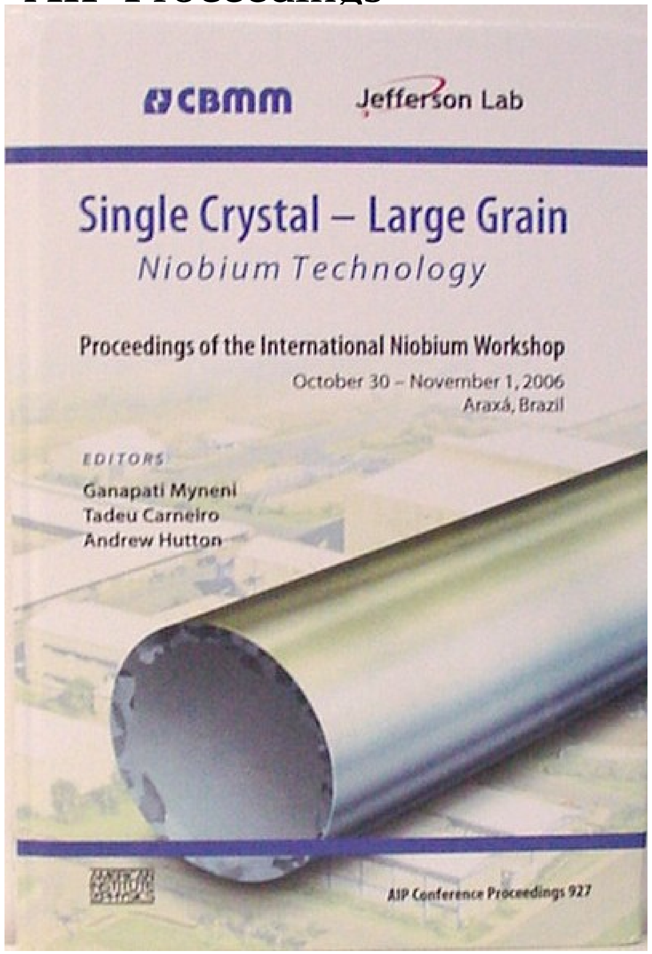
- Workshop held in Araxa, Brazil, Oct. 30 – Nov. 1, 2006, sponsored by CBMM
- 25 participants + CBMM employees
- 8 Industrial companies (AES,ACCEL,TD,Heraeus,Starck,Plansee,CBMM,Wah Chang)
- 10 Labs (FNAL,Jlab,DESY,KEK,INFN,MSU, FSU,NIST,NCU,PKU)
- Overview Talks(ILC,Nb Technology)
- Lab Reports
- Reports from Industry
- Conclusion

  It will be quite difficult to produce single crystal ingots of large diameter

  for large grain application there is no real "show-stopper"

  more experience and confidence needed for large scale application, e.g 8x9 cell cryomodule

AIP Proceedings



FIB (Focussed Ion beam)Prepared TEM Cross Sections for Jefferson Labs Nb (100), (110), (111) Oxidation

Phil Russell
Dale Batchelor
Donovan Leonard

Analytical Instrumentation Facility
North Carolina State University

Overview

- **Samples sputter coated with 60nm of Au-Pd to prevent surface damage from Ga Focused Ion Beam (FIB)**

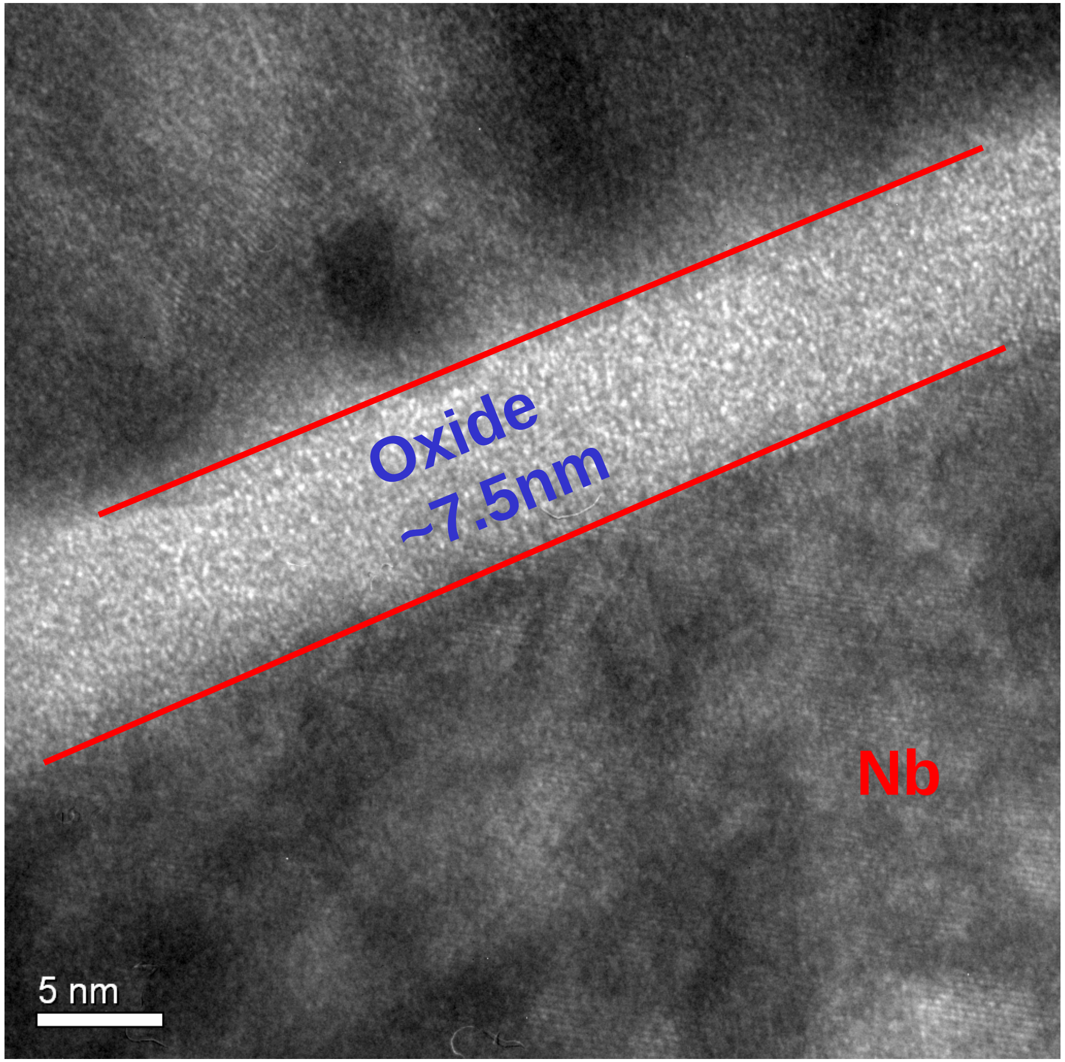
- **Samples additionally coated by in situ deposition with 2μm of W in region of analysis**

- **FIB preparation performed with an Hitachi FB-2100 Focused Ion Beam System**

- **TEM micrographs captured with a Gatan Digital Camera on a JEOL JEM2010F High Resolution TEM/STEM**

- **Oxide Nb(100) ~4.9nm, Nb(110) ~8.3nm, Nb(111) ~7.5nm**

Nb (111)



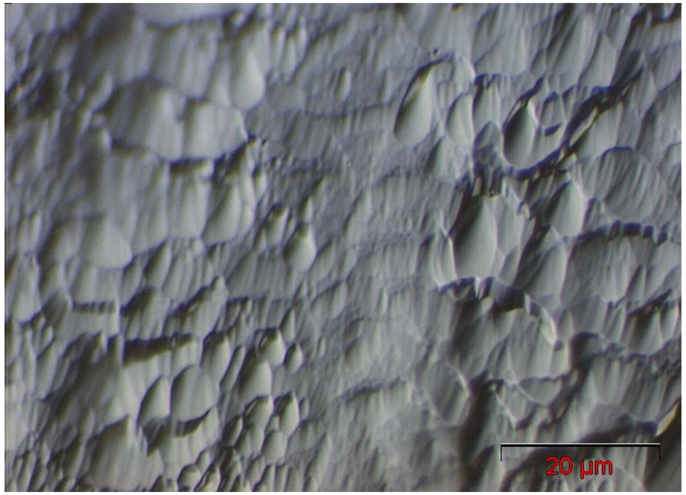
DC Field Emission Results on Nb Samples

A. Dangwal and G. Müller
FB C Physics Department, University of Wuppertal, Germany
gmueller@uni-wuppertal.de

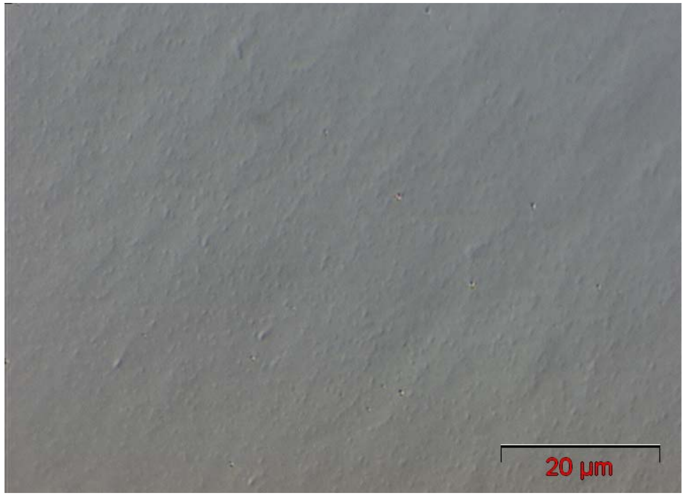
18th September 2007

JRA1/CARE Meeting, Warzaw, Poland

Optical microscope images of SC and LG Nb samples



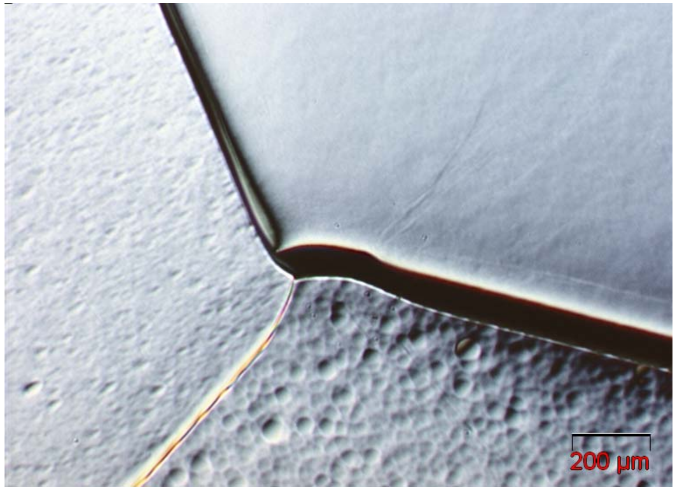
SCNb1 – BCP 30 µm



SCNb2 – BCP 30 µm

measured by
X. Singer
at DESY

SCNb7 – BCP 100 µm

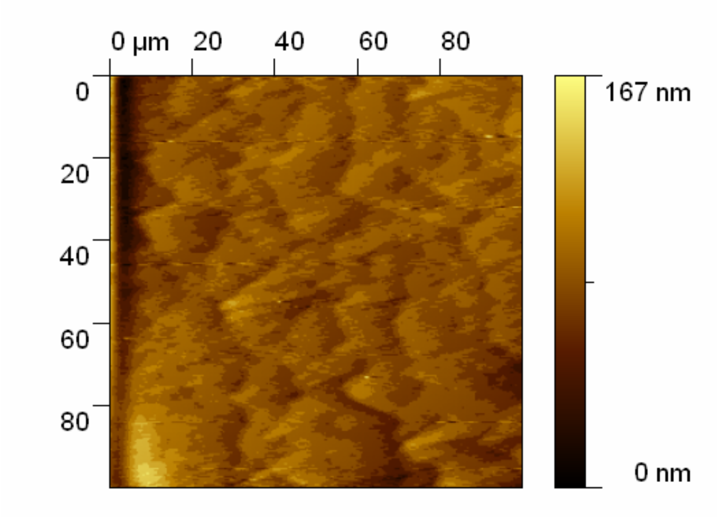


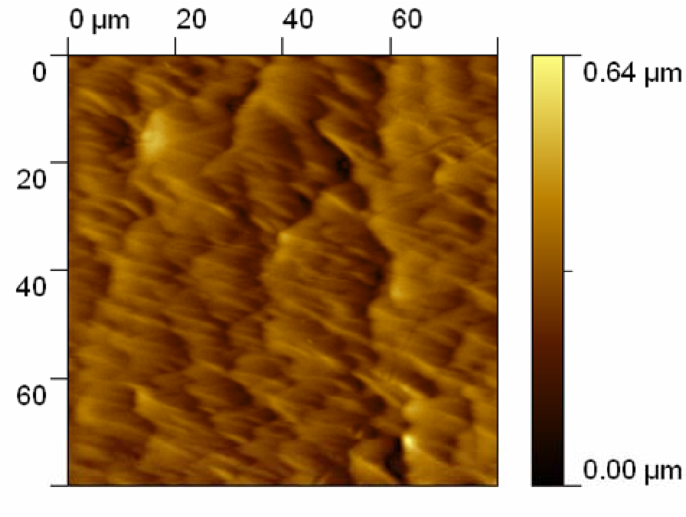
LGNb3 – BCP 100 µm

BCP 100 µm provides the best surface

Surface roughness of SC and LG Nb samples by AFM

measured by X. Singer at DESY



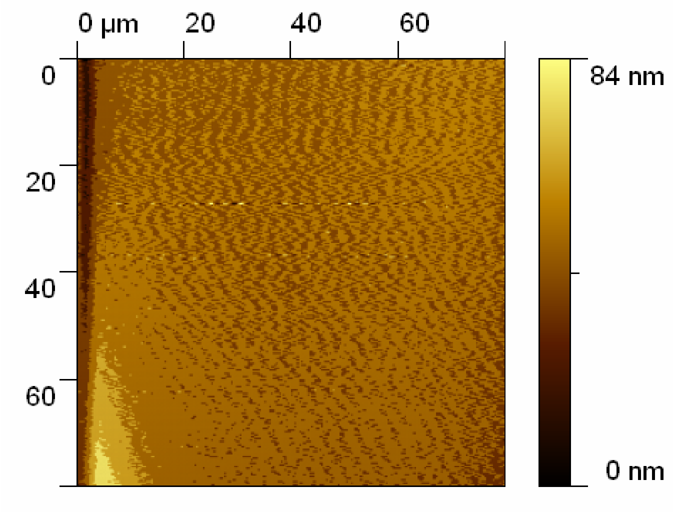


SCNb2, BCP 30 µm

Rms: 17.6 nm

LGNb1, BCP 30 µm

Rms: 62.7 nm



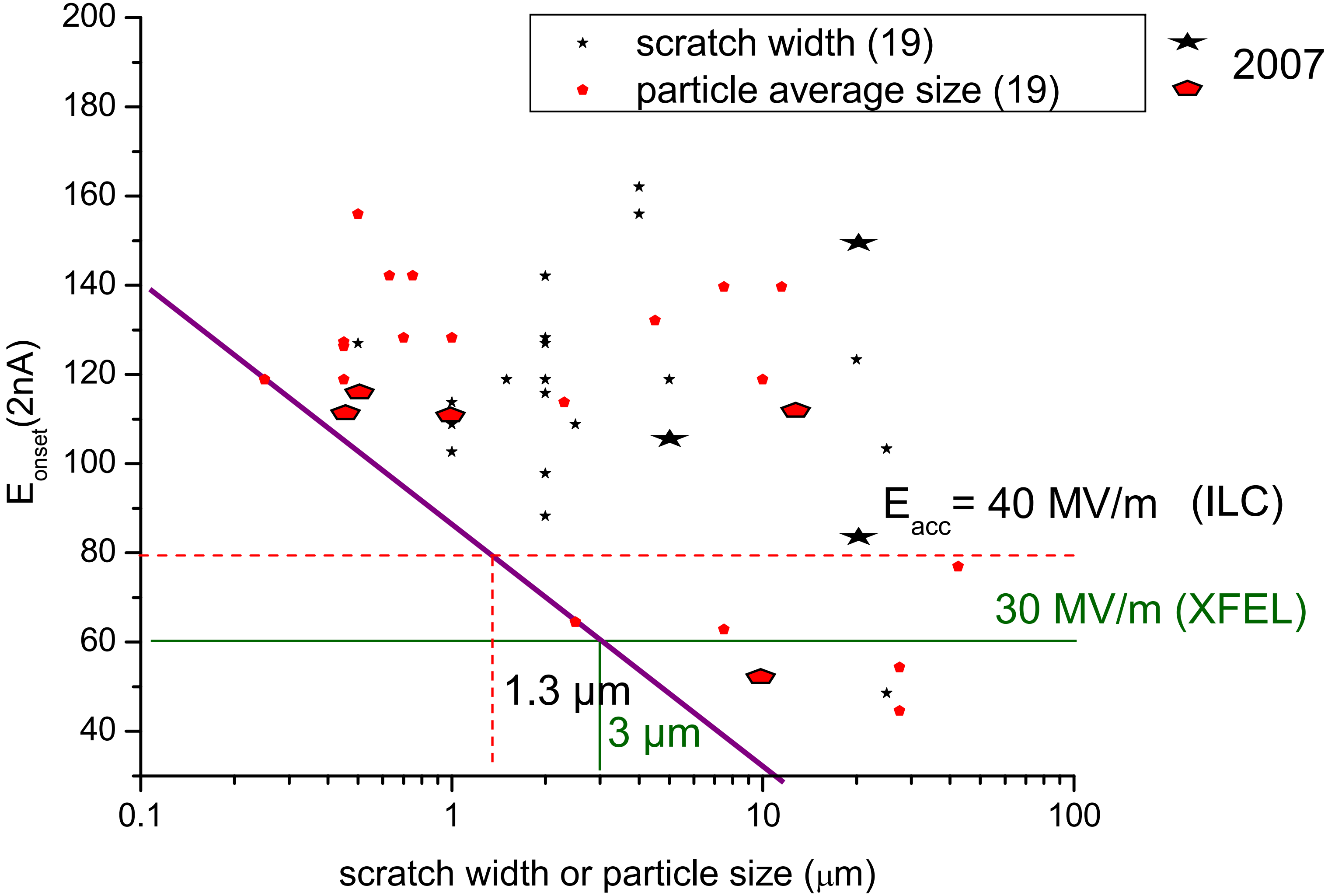
SCNb4, BCP 100 µm

Rms: 7 nm

Mirror-like surfaces

Correlation between FE onset field and defect size ?

based on FE measurements and SEM analysis of 38 field emitters



Evidence for correlation $\Rightarrow$ fast FE quality control by defect size

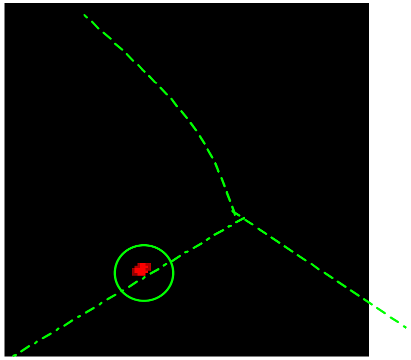
In-situ bakeout at 150 ºC effects on LGNb3 and SCNb4

PID-regulated U(x,y) for 2 nA scanned area = 5×5 mm²

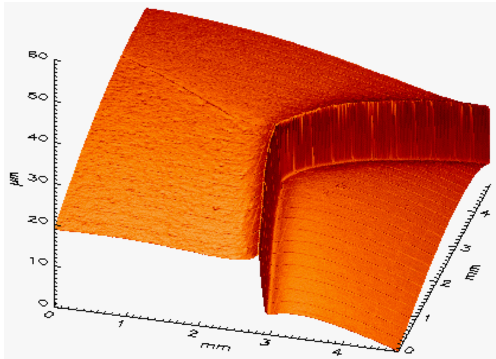
flat W-anode $Ø_a$ = 100 µm, U_{max} = 5000 V, Δz = 25, 20, 16.6 µm

LGNb3

before baking



250 MV/m

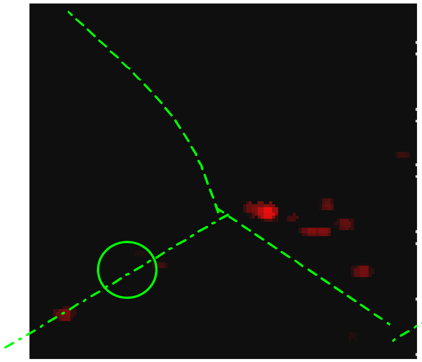


SCNb4

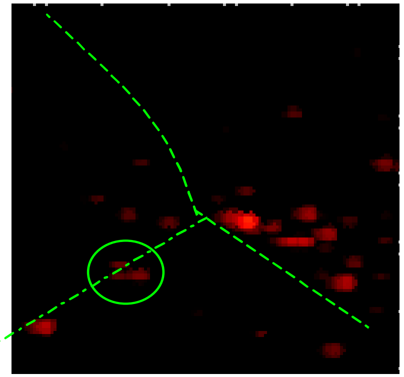
before baking

250 MV/m

after 14 h baking at 150 ºC

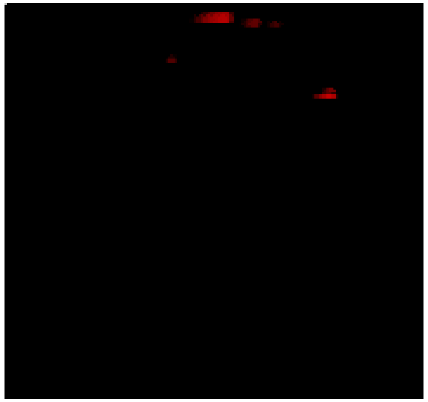


250 MV/m



300 MV/m

after 8 h baking at 150ºC



250 MV/m

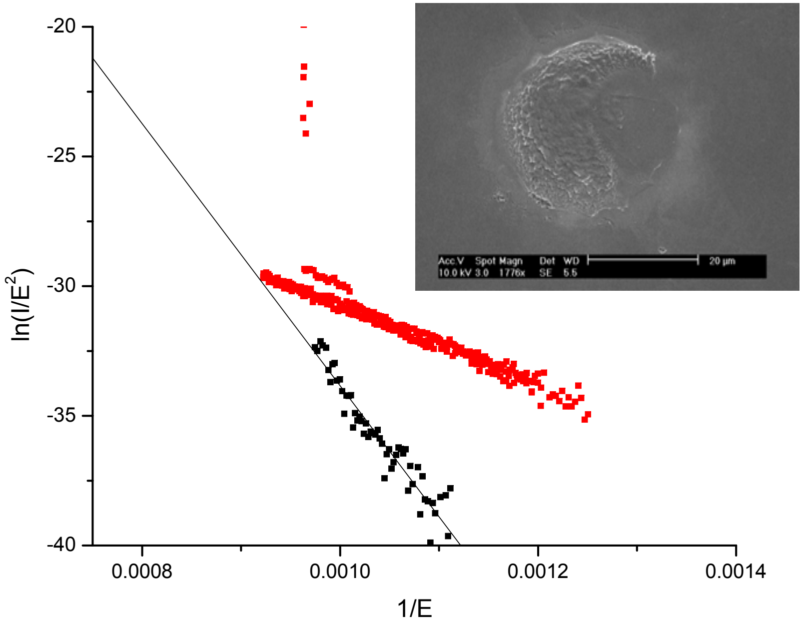
After Baking more emitters appear for LG but not for SC at E > 250MV/m

⇒ first evidence for impurity segregation to grain boundaries

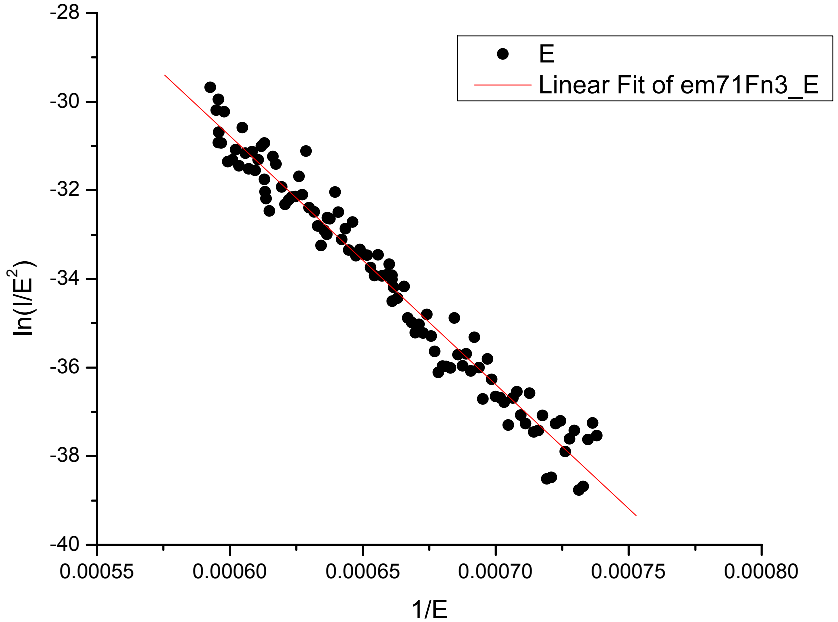
Intrinsic FE of SCNb in defect-free sample areas

measured with W-tip anodes of $Ø_a$ = 5-10 μm at U = 5000 V and Δz > 1 μm

SCNb4, (111) orientation



SCNb7, (100) orientation



Initially intrinsic field emission of Nb with slope β = 1 $\Rightarrow$ Φ = 3.8 eV

creation of an emitter at ~ 1000 MV/m by a microdischarge $\Rightarrow$ crater in Nb

Intrinsic field emission of Nb

slope β = 1 $\Rightarrow$ Φ = 4 eV

$\Rightarrow$ SCNb samples reveal anisotropy of work function Φ

Conclusions

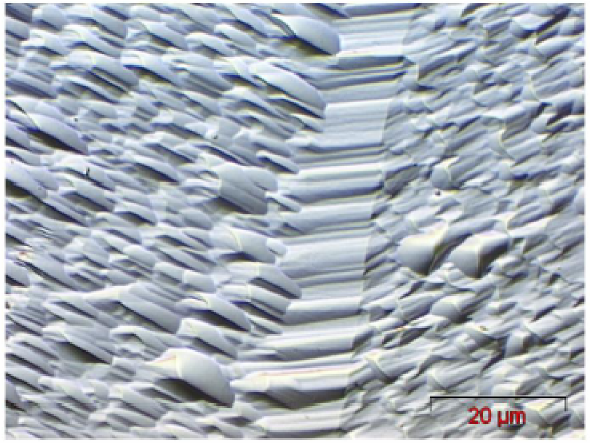
- DIC effectively removes particulates and weakens protrusions
  $\Rightarrow$ in situ repair cleaning of FE cavities in module ! (JAP102, 2007)
- Large/single crystal Nb samples show after BCP30/100-HPR better FE results than EP-HPR samples of various kinds
  $\Rightarrow$ reliable alternative for SRF cavities with less FE ! (SRF 2007)
- Evidence for a correlation between onset field and emitter size
  $\Rightarrow$ fast FE quality control on samples for XFEL ! (SRF 2007) + FP7
- Evidence for impurity segregation to grain boundaries in LGNb after bakeout at 150ºC $\Rightarrow$ reduced FE in SCNb ! (SRF 2007) + FP7
- Intrinsic FE on SCNb with β = 1 and Φ = 4 eV partially obtained
  $\Rightarrow$ surface roughness enhances FE of particulates ! (SRF 2007)

PhD thesis of A.Dangwal will be presumably available in November 2007

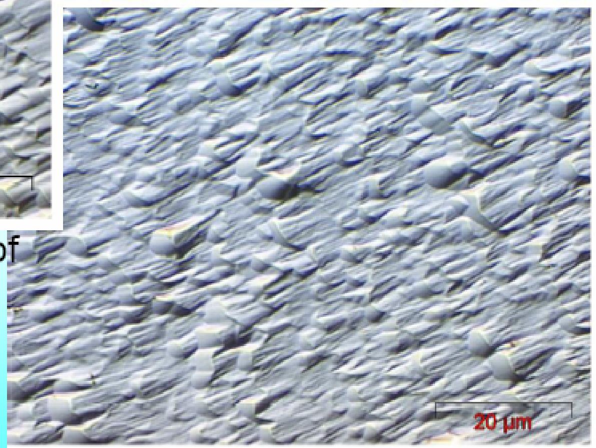
Single Crystal Cavity Fabrication

Single Crystal Cavity Fabrication

- Single crystals will remain single crystals after up to 60% of mechanical deformation and appropriate annealing
- Single Crystals maintain the crystallographic structure and orientations after deep drawing and 800 C annealing
- Single crystals can grow together under appropriate EBW conditions



Left: Electron beam welding connection of two single crystals without regarding of crystals orientation (the grain boundary is pronounced)



Right: EB welding connection of two single crystals after assembling considering the crystal orientation (the grain boundary is absent)

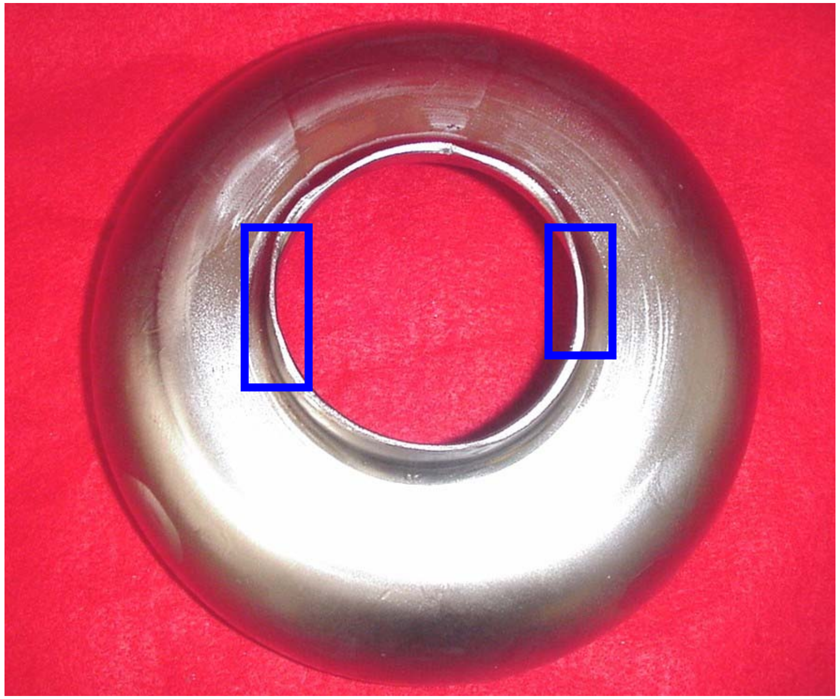
W. Singer, Single Crystal Nb Technology Workshop, CBMM, Brazil, Oct. 30-Nov. 1, 2006

Fabrication Issues

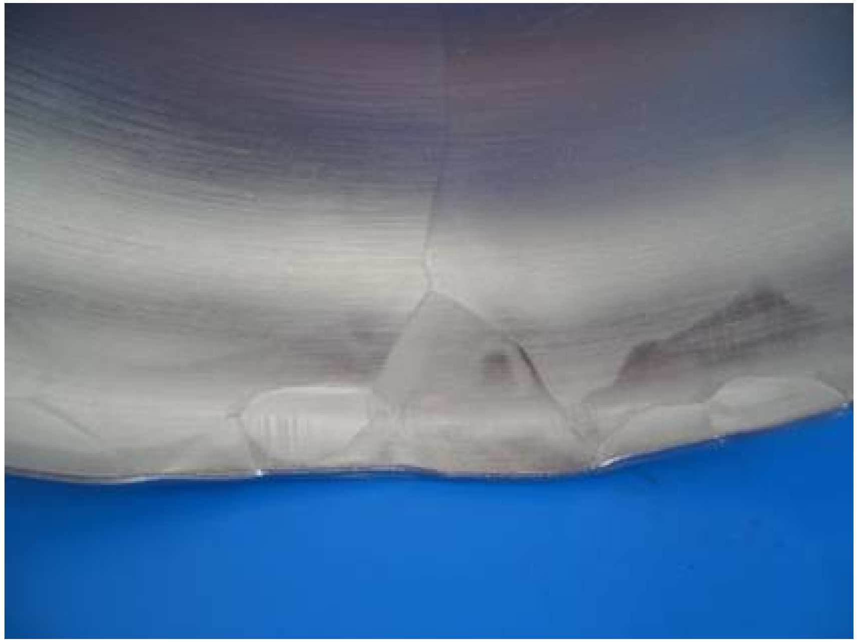
- Large grain sheets deep draw with ragged edges
- Sometimes the material is thinning or ripping at the irises, if the grains “meet” in these areas
- There is some spring back after the deep drawing, making the half cells “oval”
- The same happens after the trimming for EBW
- Assembly for EBW sometimes more difficult than with fine grain material. However, no problem with single crystals
- On a few occasions, holes occurred during welding

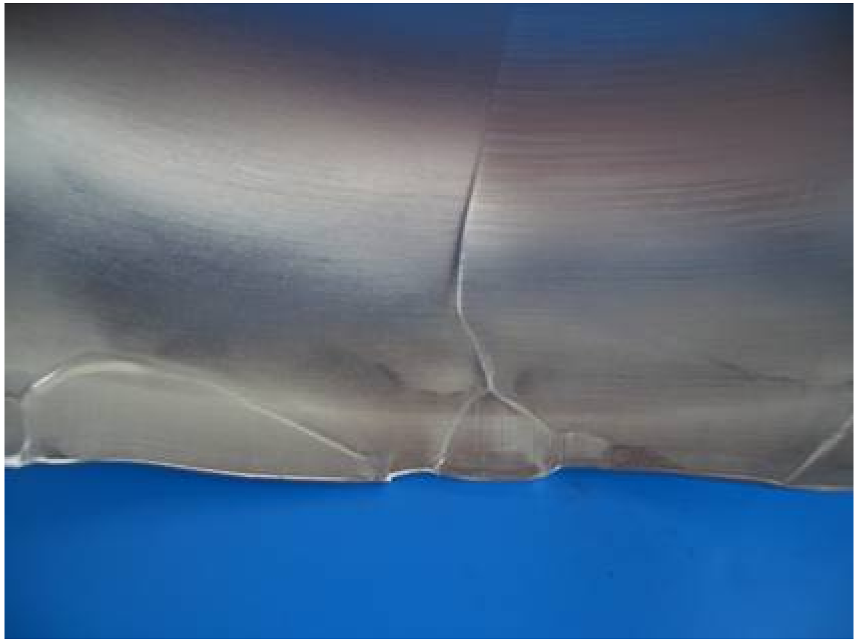
Fabrication Issues (2)

Half cell from CBMM Ingot “B”



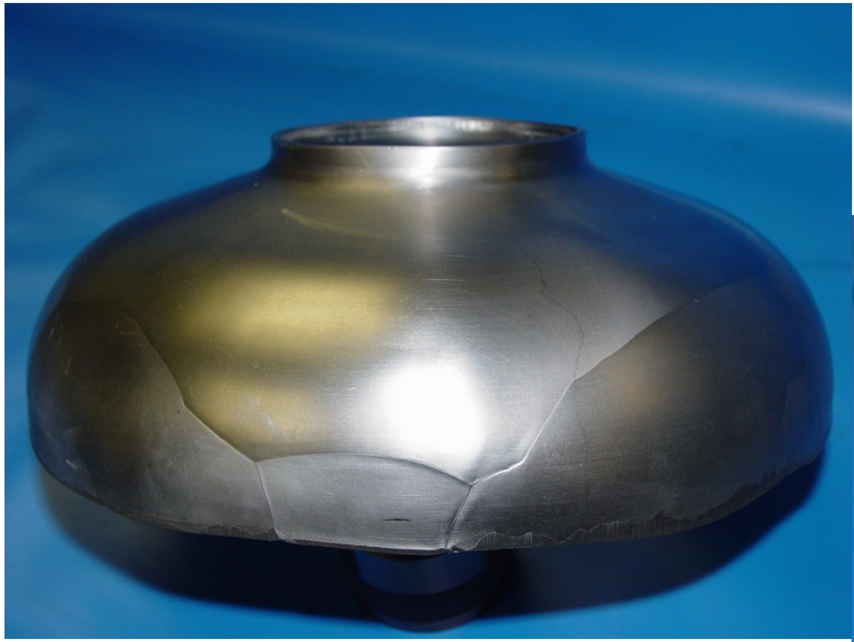
Grain steps from forming process


Almost no grain steps


Large grain steps

- possibly room for improvement by variation of deep drawing parameters

Single crystal option is more exciting [X.+W. Singer]



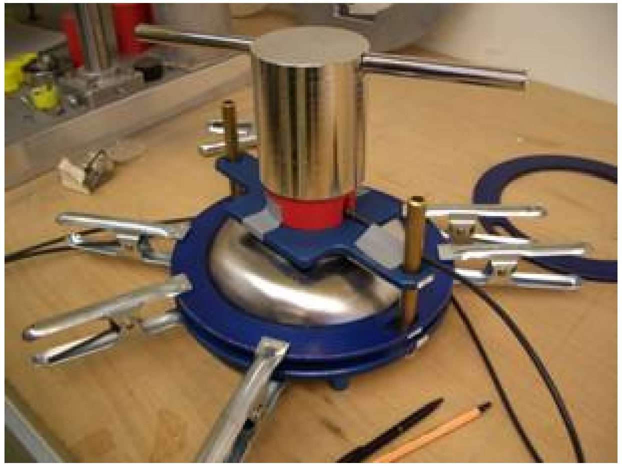
Deep drawn half cell of large grain niobium; grain boundaries pronounced, anisotropy of properties (earing)

Deep drawn half cell of single crystal niobium

Predictable properties

Large grain 9-cell cavity production [M.Pekeler, ACCEL, Single Crystal Nb workshop

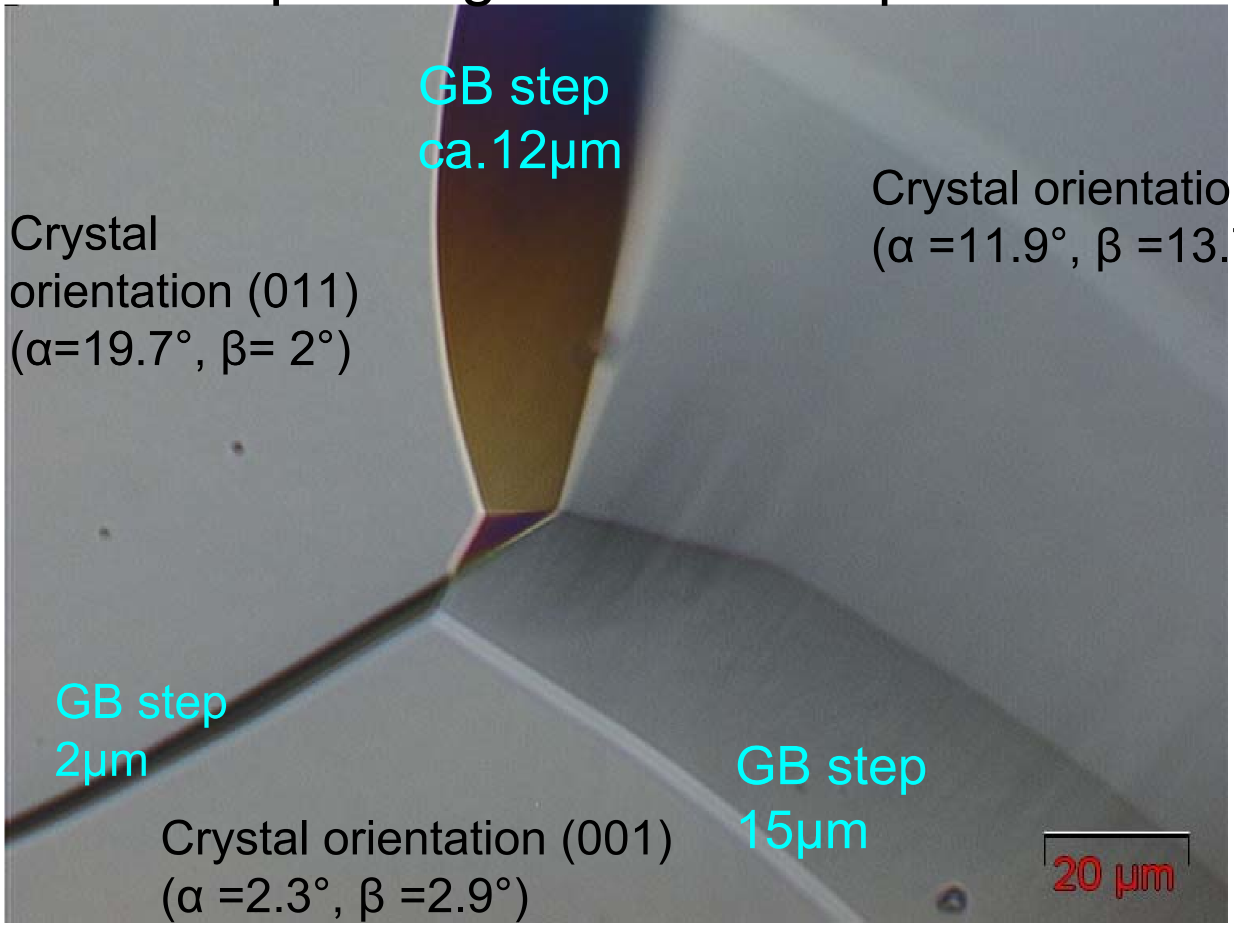
No problems observed during manufacturing, assembly for equator welds a bit more complicated as cells are not as round as fine grain cells, but basically no difference in production time and cost for large grain and fine grain 9-cell cavity



Frequency control

LG: Grain boundaries [X. Singer]

Light microscope image of LGs sample after 100 µm BCP



Single crystal has no grain boundaries, not such disadvantages as LG

Cavity Results

Test Results (Jlab)

- In nearly all tests/all cavity types the cavity performances were limited by "Quench"
- Therefore, for comparing the results, the magnetic quench field is the right property to look at
- For "calibration" the table below lists the required performance in terms of H_{max} for ILC and XFEL cavities

Project	E_{acc} [MV/m]	H_{max} [mT]
XFEL	28	119
ILC(BCD) ILC (ACD)	35 45	149 162

What had been done at the end of 2006 at Jlab?

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

  CBMM (4 different ingots)

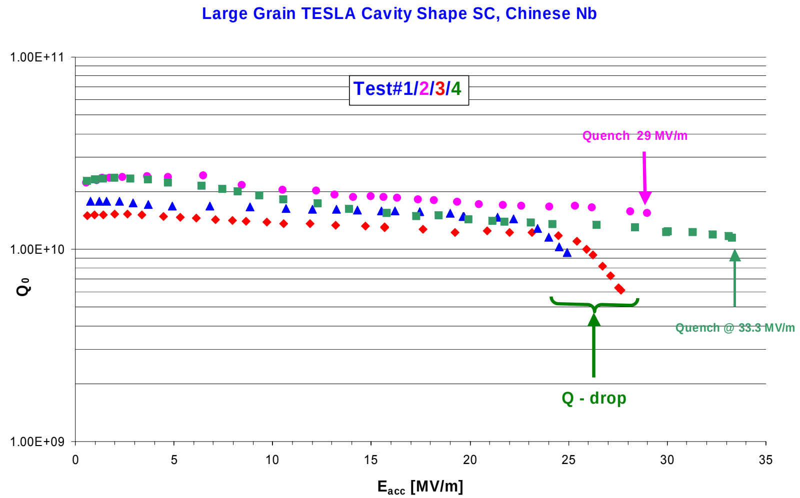
  Ningxia (3 different ingots)

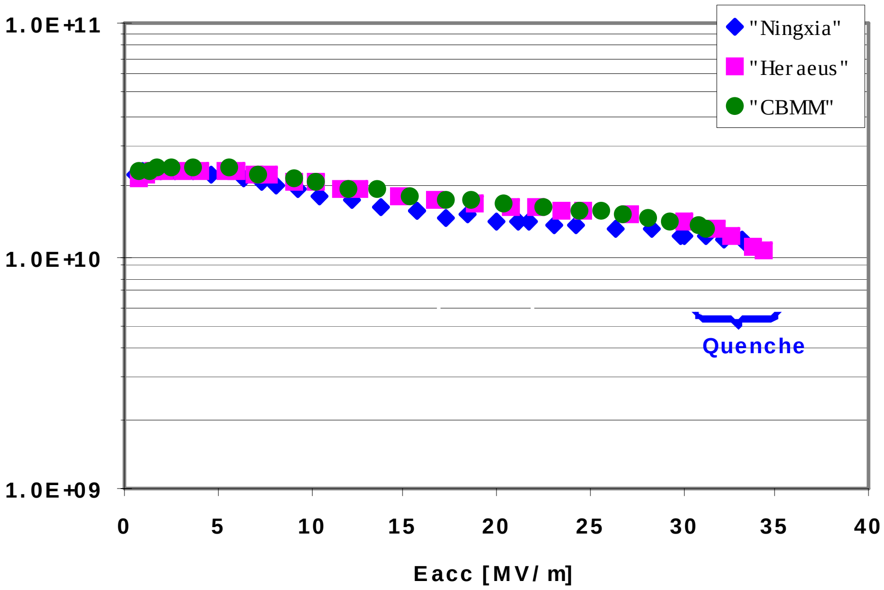
  W.C. Heraeus (2 different ingots, 1 used for single crystal-DESY)

  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 we have fabricated and tested 17 single cell cavities and 3 multi-cell cavities and carried out close to 100 tests

Other Developments:large grain/single crystal niobium(2)[P.Kneisel,EPAC 2006]

Test results from recent tests at Jlab



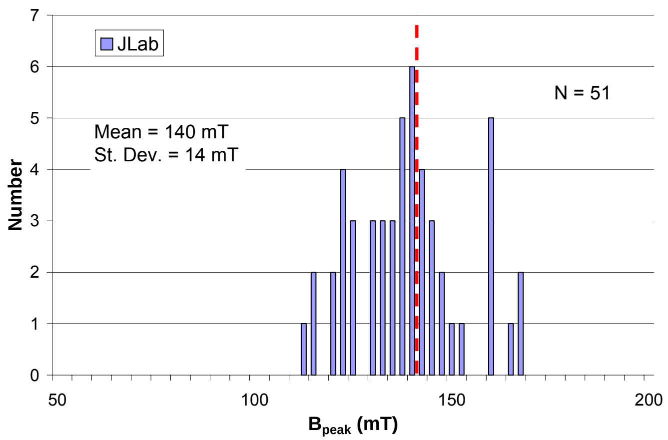


Potential benefits:

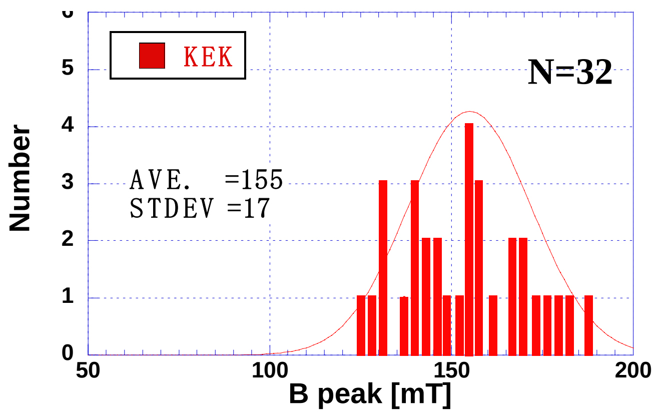
- lower costs at comparable performance
- very smooth surfaces with bcp, no EP
- streamlining of procedures/QA
- less spread in data?

Summary of large grain/single crystal single cell tests(2)

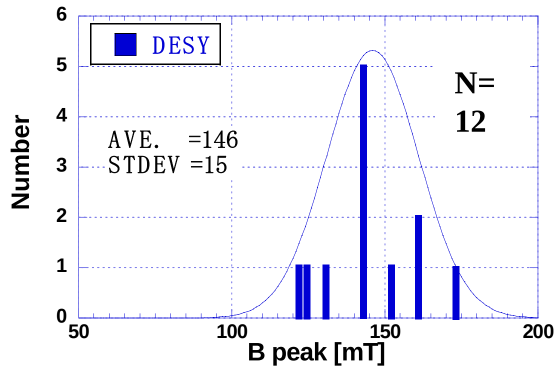
Jlab



KEK



DESY



Material Comparison

Supplier	Cavity Type	Ta - contents	RRR	Test ½ H_{max} [mT]	Test ½ Q_0 at H_{max}	Test ¾ H_{max} [mT]	Test ¾ Q_0 at H_{max}
W.C.Heraueus	TESLA	<500 ppm	500	**140**	1.15×10^{10}	**146**	1.05×10^{10}
Ningxia	TESLA	~ 100 ppm	330	**123**	1.5×10^{10}	**142**	1.14×10^{10}
CBMM	TESLA	~ 800 ppm	280	**133**	1.3×10^{10}	**131**	1.04×10^{10}
CBMM	Proton Driver	~ 800 ppm	280	**139**	7.5×10^{9}	**148**	6.9×10^{9}
CBMM	Proton Driver	~ 800 ppm	280	**133**	4.4×10^{9}	**135**	1×10^{10}

KEK [Z.G.Zong et al, PAC 2007, paperWEPMN047]

- Three Ichiro-type single cell cavities were fabricated from Ningxia large grain niobium
- Different amounts of material were removed by CBP(only cavity #1 and #2),CP and EP
- Cavity # 1 and #2 reached E_{acc}=47.9 MV/m (H_{peak}~ 170 mT) and E_{acc}= 43 MV/m (H_{peak}~ 155 mT), respectively, after 90 μm CBP,10 μm and 80 μm EP
- 48 hrs of baking after EP needed
- Low residual resistances measured

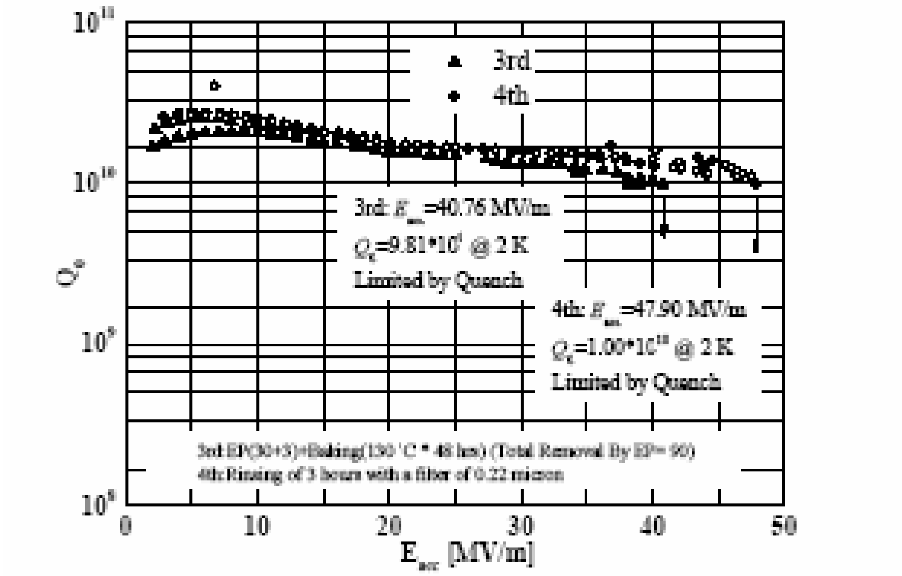
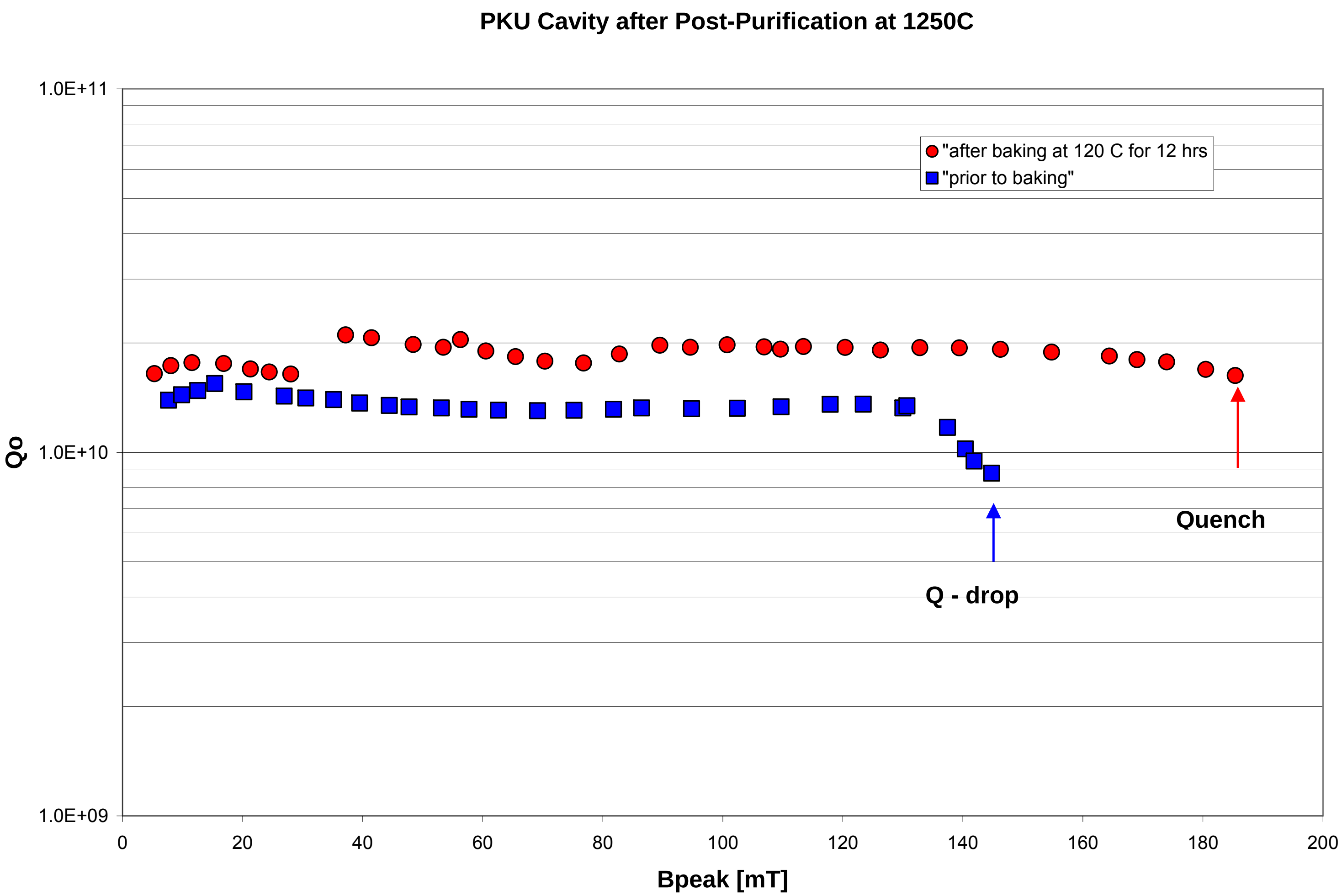


Figure 4: Excitation curves of the third and fourth tests of China LG #1.

PKU:TESLA Shape, Ningxia Nb



Cornell University

G. Eremeev, H. Padamsee,EPAC 2006, paper MOPCH176

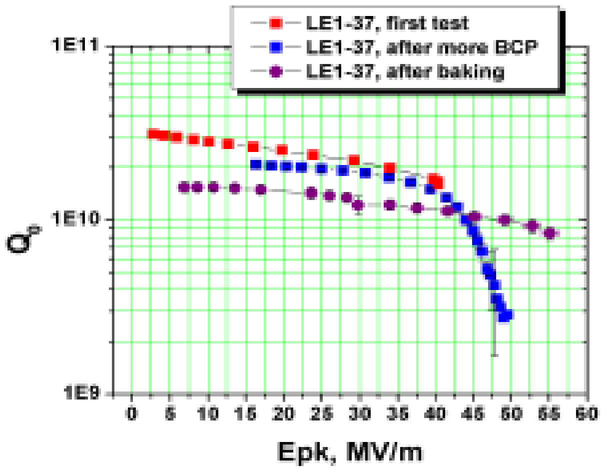


Figure 1: Results for the large grain cavity.

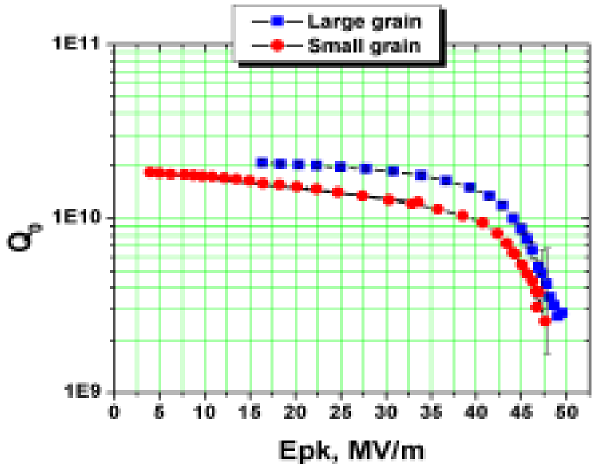


Figure 4: Results for the large grain cavity and small grain cavity before baking.

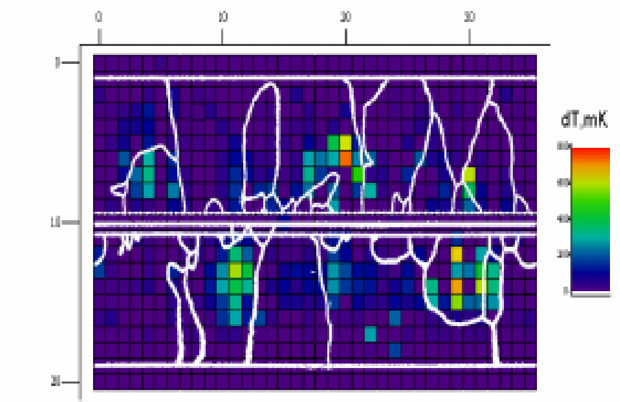


Figure 3: Temperature map for second test at Epk = 49 MV/m.

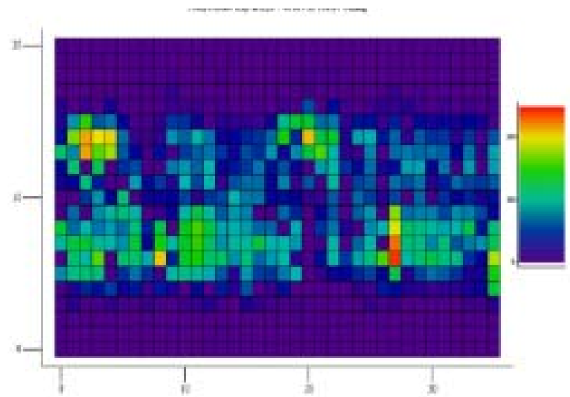
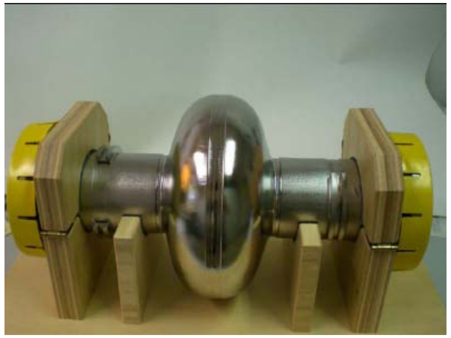
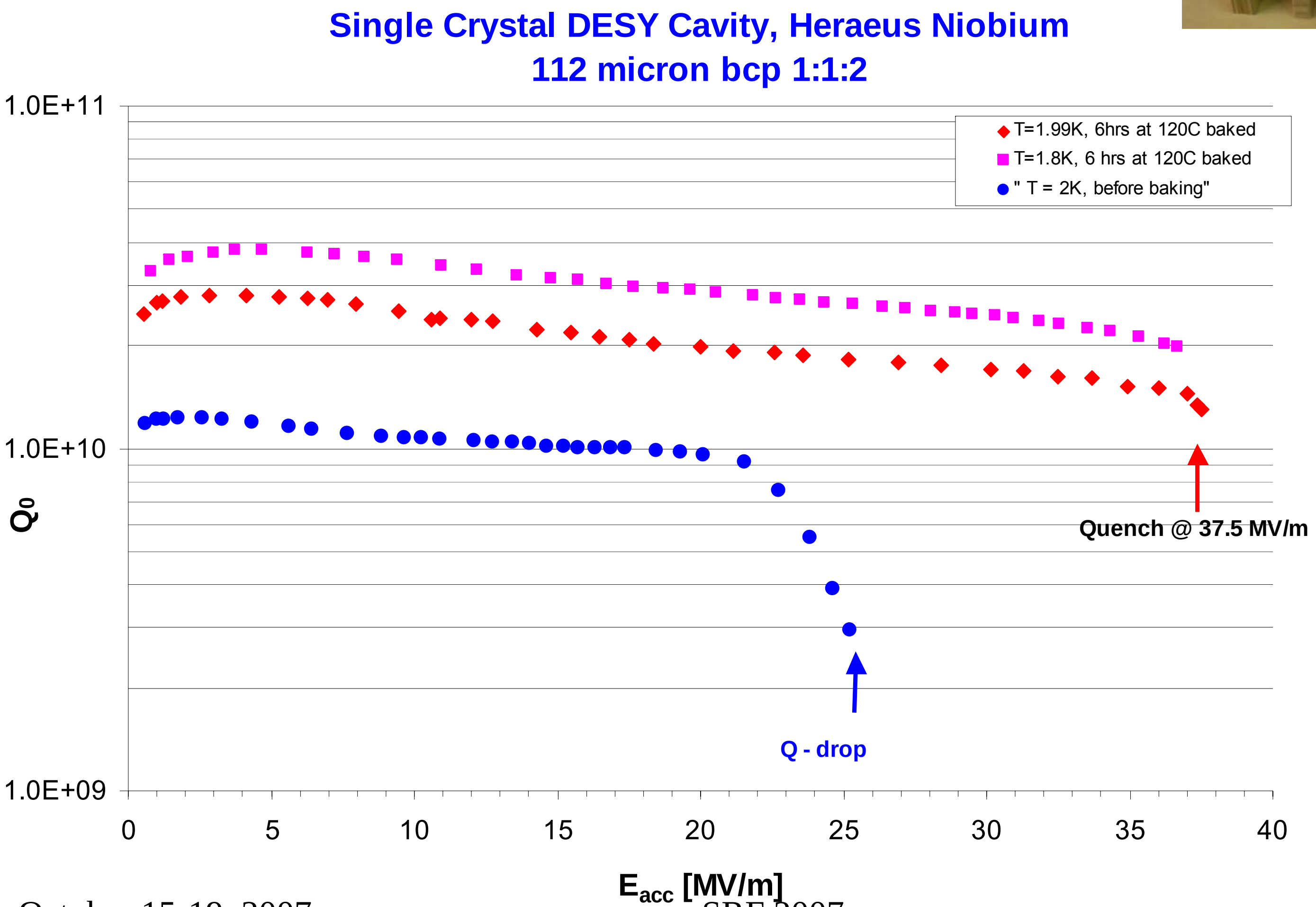


Figure 5: Temperature map for small grain cavity.

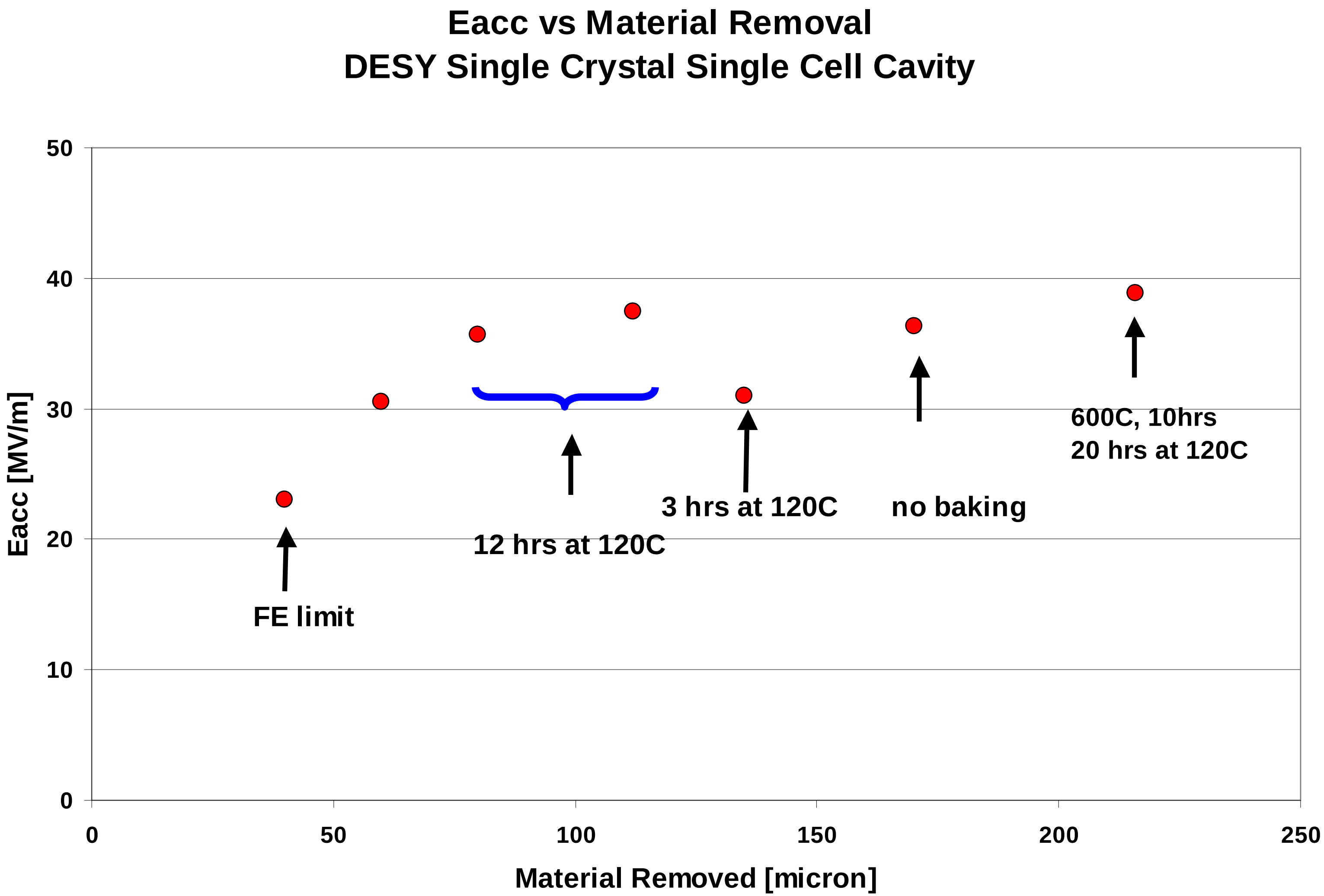
Test #6, cont'd

- The cavity was baked at 120C for 6 hrs





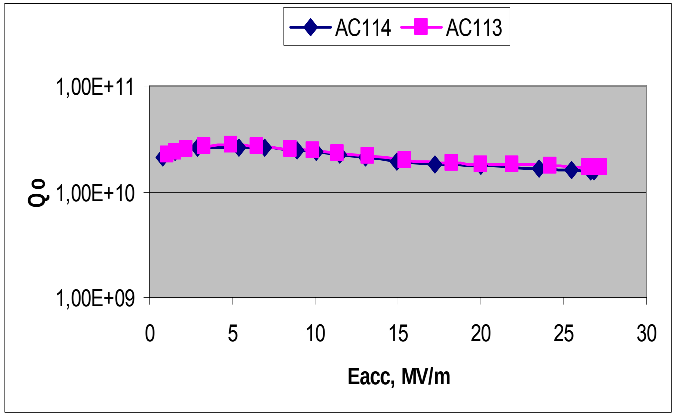
Summary of Performance



9-cell Cavity performance (DESY)

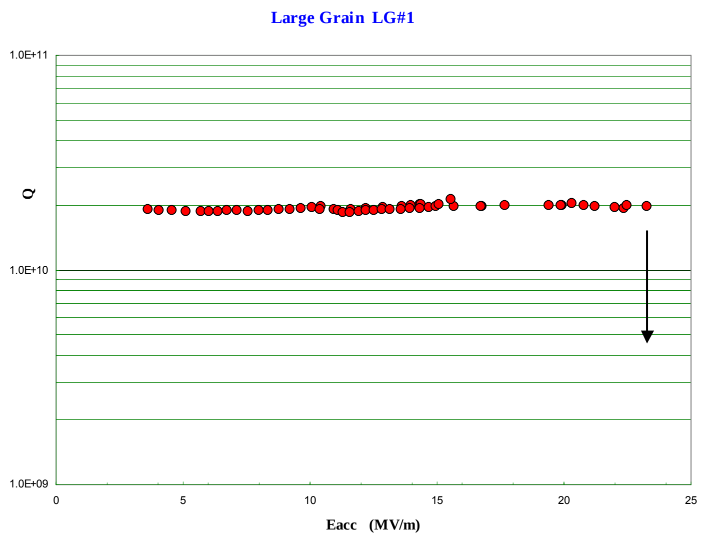
- Three 9-cell TESLA shape cavities were fabricated from Heraeus large grain Niobium by ACCEL for DESY
- These cavities performed very well after bcp only(W.Singer et al, PAC 2007, paper THOAKI01)
- In addition, several single cell cavities performed very well, better after EP (~ 40 MV/m)

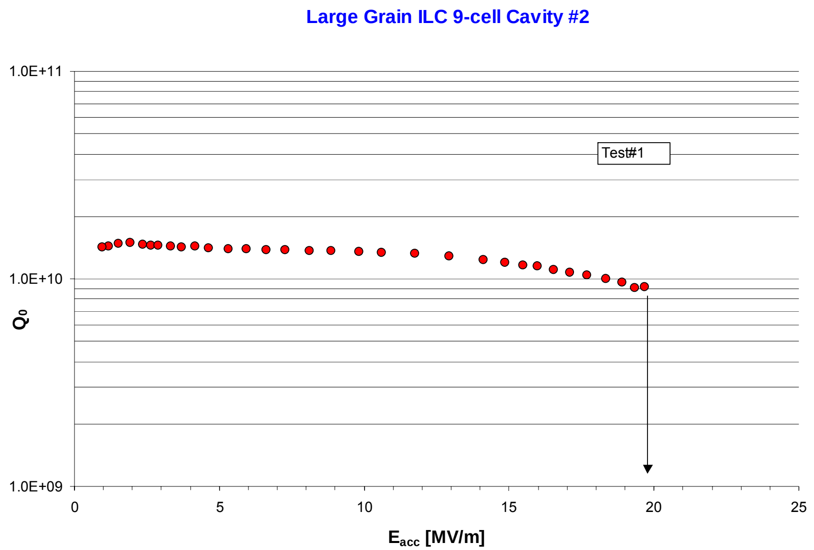
Manufacturer	Name	Treatment	E_{acc}[MV/m]	Q @ Eacc	Limit
Heraeus LG	AC 112/9-cell	130 μm bcp,800C,HPR	30.5	2 x10^{10}	FE
Heraeus LG	AC 113/9-cell	130 μm bcp,800C,HPR	27.4	2 x10^{10}	Quench
Heraeus LG	AC 114/9-cell	130 μm bcp,800C,HPR	28.7	2.1 x10^{10}	Quench,FE ind?



9-cell Cavity performance(Jlab)

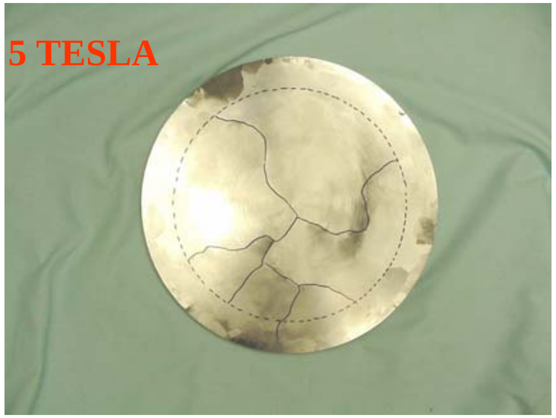
- Two 9-cell cavities (LG#1,LG#2) were fabricated at Jlab from large grain CBMM niobium (ingot”D”); several holes during EBW in both cavities
- Standard processing:pre-tuning, 100 micron bcp,hydrogen degassing at 600C for 10 hrs,final tuning, final bcp
- LG #1 received only ~ 40 micron, LG#2 ~ 57 micron bcp in final bcp
- LG#1: quench at Eacc = 23 MV/m,
- LG#2: quench at Eacc = 20 MV/m

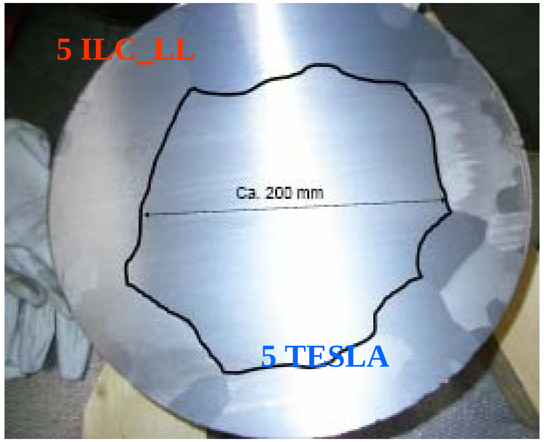




Reproducibility Tests(1)

CBMM "D" Heraeus Ningxia





Manufacturer	Ta Contents	RRR	Sheet Cutting Method
CBMM	~800 ppm	~ 280	Wire EDM
W.C.Heraeus	< 500 ppm	500	Wire Saw
Ningxia	< 100 ppm	330	Saw + machining

Reproducibility Tests(2)

Ningxia Niobium (TESLA shape)

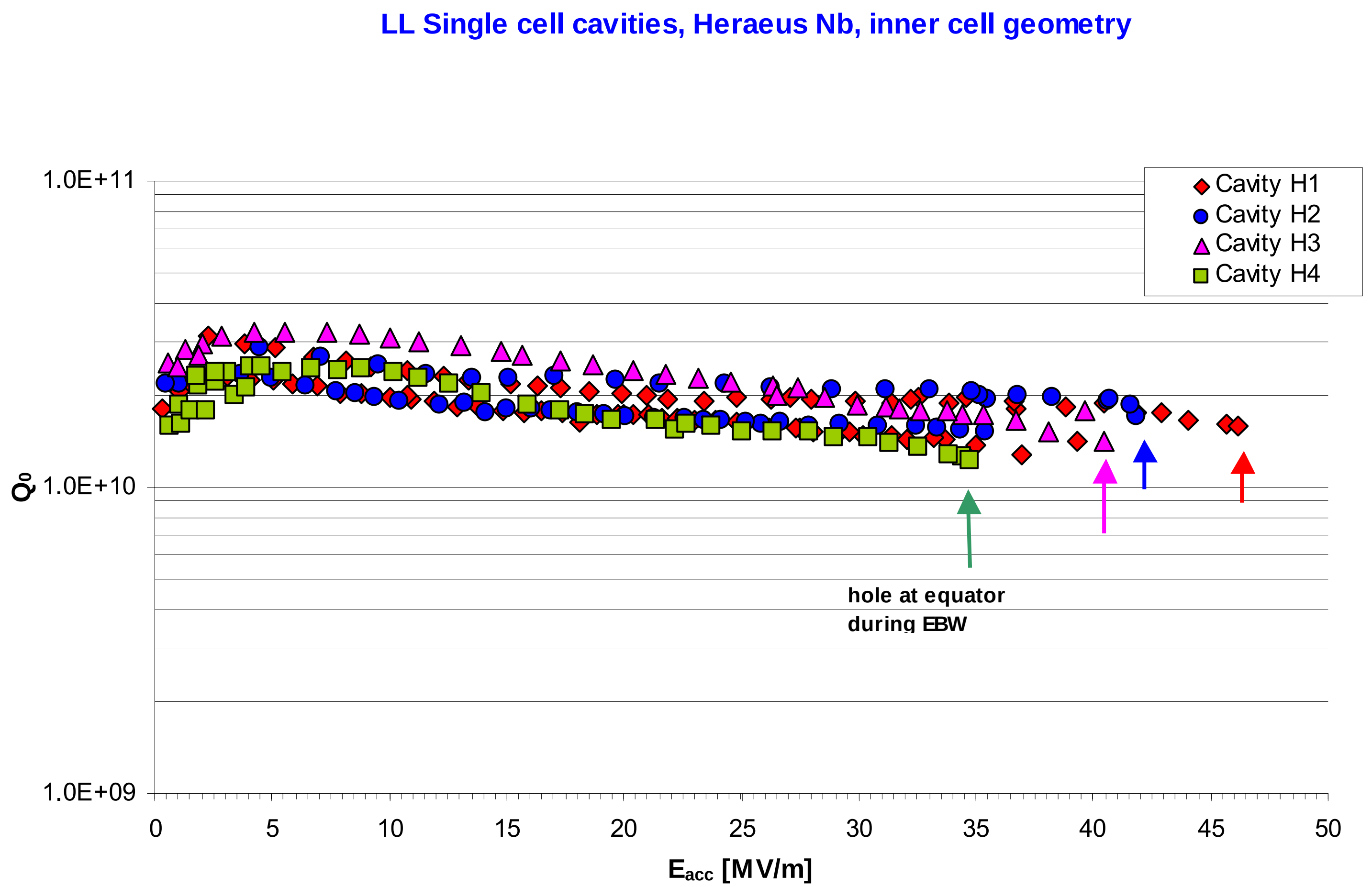
- Saw cutting of sheets (as received)
- Deep drawing
- Machining of half cells
- EBW of beam pipes
- Mechanical grinding of half cells
- Equator weld
- 25 micron bcp
- Hydrogen degassing at 600C for 10 hrs
- App. 80-90 micron bcp (1:1:1)
- HPR for 2 hrs
- In situ baking at 120C for 12 hrs

W.C.Heraeus Niobium (ILC_LL_shape)

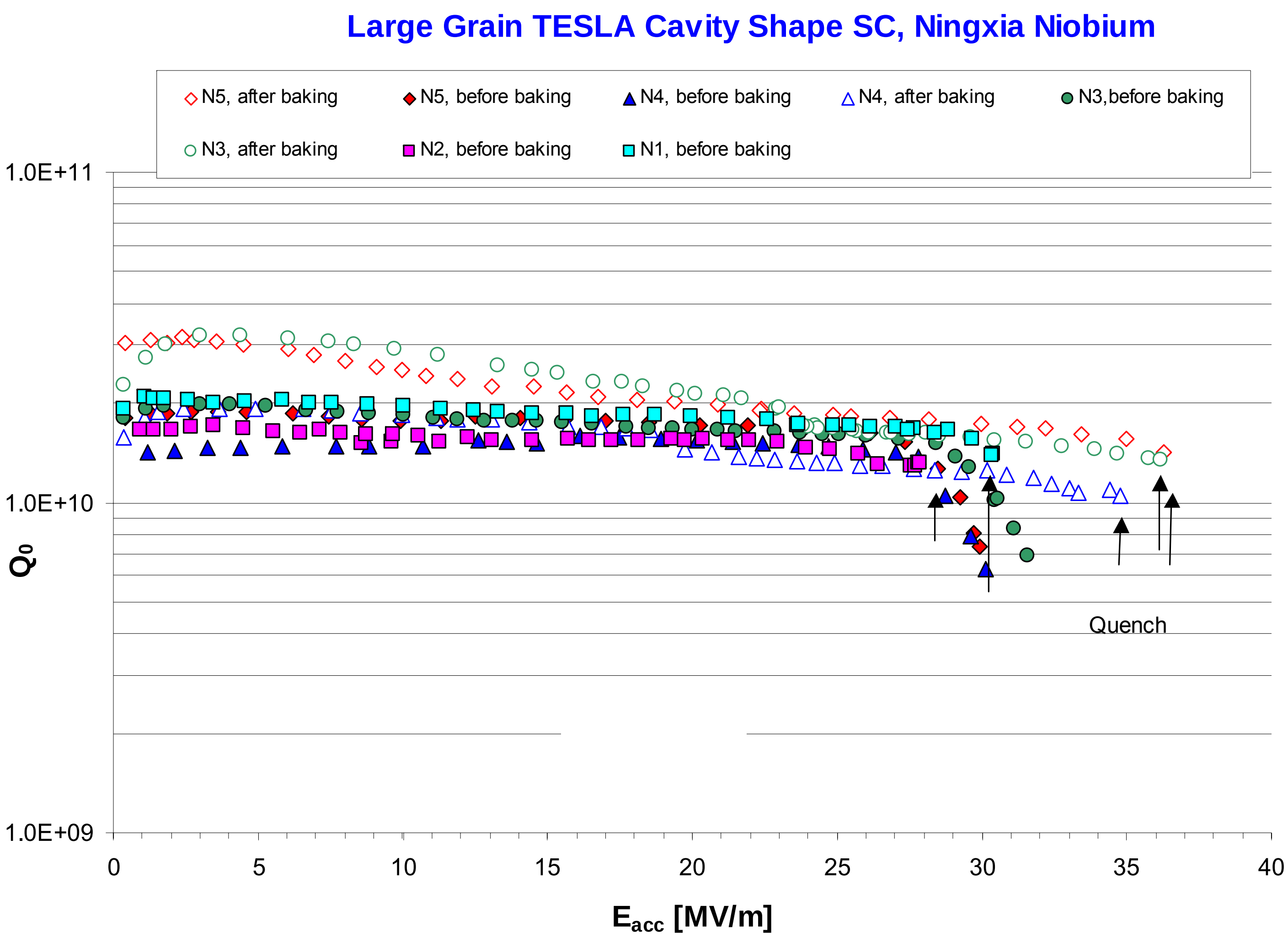
- Wire saw cutting of sheets (as received)
- Deep drawing
- Machining, EBW of beam pipes
- Grinding
- Equator weld
- 50 micron bcp
- Hydrogen degassing at 600C for 10 hrs
- App. 50 micron bcp (1:1:1)
- HPR for 2 hrs
- In situ baking at 120C for 12 hrs

Reproducibility Tests(3)

Heraeus Nb, LL shape



Reproducibility Tests (4)



Reproducibility Tests: Summary

- Ningxia

$$119\ \text{mT} \leq H_q \leq 155\text{mT}$$
$$< 141\text{mT} >$$

- Heraeus

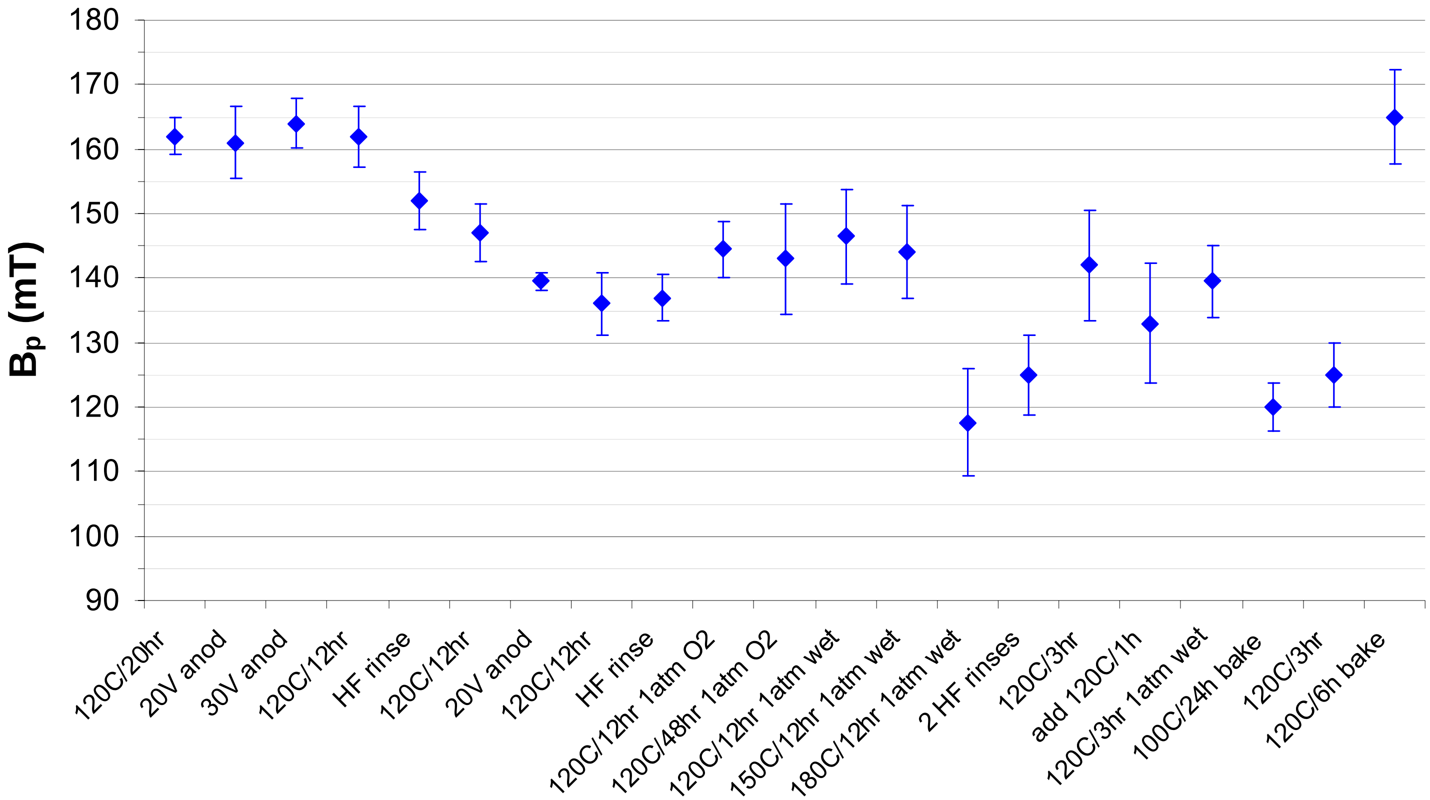
$$125\ \text{mT} \leq H_q \leq 166\text{mT}$$
$$< 147\ \text{mT} >$$

- In – situ baking at 120C for 12 hrs eliminates the Q-drop and
  - Increases Δ/kT_c by 8- 10 % (~ 1.75 to ~1.9)
  - Residual resistance often increases, R_{BCS} is lowered by ~ 50%
  - Mean free path is shortened

A “tortured” Cavity[Ningxia Nb]

G.Ciovati et al, PHYSICAL REVIEW SPECIAL TOPICS - ACCELERATORS AND BEAMS 10, 062002 (2007)

Fields are after baking, following bcp



Summary

- Large grain material provides some challenges in fabrication of cavities, but is no “show stopper”
- Single crystal sheets would be desirable, but no significant performance improvements over large grain niobium
- Performance is comparable with fine grain niobium
- But does not need electro-polishing, BCP is fine and very smooth surfaces can be achieved
- For projects such as the XFEL or cw applications cavities from large grain niobium offer “streamlined” procedures:
  - Bcp, shorter “in situ” baking times, high Q-values at high fields
- Reproducibility of performance after bcp treatment seems to be quite good – to be further “ hardened”
- Cost advantage over poly-crystalline niobium needs to be realized , effective cutting method presently only pursued by W.C. Heraeus
- Further confidence will be “built up” with add. 9-cell cavities (cryomodule)