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 $E_{\text{acc}} = 45 \text{ MV/m}$ ($H_{\text{peak}} \geq 165 \text{ mT}$)

• 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

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<th>Comment</th>
<th>Reference</th>
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<tbody>
<tr>
<td>Mech. properties</td>
<td>YS, TS, elongation, bulging, residual stress</td>
<td>1,2,3</td>
</tr>
<tr>
<td>Thermal properties</td>
<td>Therm. Conductivity, phonon peak, effect of strain</td>
<td>4,5,1</td>
</tr>
<tr>
<td>Magn./electr. properties</td>
<td>Hc1, Hc2, Hc3 for diff. crystal orientations</td>
<td>14</td>
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<tr>
<td>Crystal orientation/recrystall.</td>
<td>Grain orientation in diff. material, etch rate, dislocation density</td>
<td>4,5,13</td>
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<tr>
<td>Forming/welding</td>
<td>Increase large grain to single crystal any size</td>
<td>6,7,8</td>
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<td>Flux penetration</td>
<td>Influence of grain boundaries on flux penetration</td>
<td>9,15</td>
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<tr>
<td>Oxidation</td>
<td>Diff. crystal orientations, oxide composition</td>
<td>10,11,16</td>
</tr>
<tr>
<td>Field emission</td>
<td>Emitter density, cleaning, grain boundary segregation</td>
<td>12,17</td>
</tr>
</tbody>
</table>
References
[9] P. Lee et al.; AIP Conference Proceedings 927, p. 113
[13] C. Compton, paper TUP05
[14] W. Singer, paper TUP 12
[16] A. Romanenko, paper TU 103
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, CBM M, 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
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)

Oxide ~7.5nm

5 nm
DC Field Emission Results on Nb Samples

A. Dangwal and G. Müller
FB C Physics Department, University of Wuppertal, Germany
gmueller@uni-wuppertal.de
18\textsuperscript{th} September 2007
JRA1/CARE Meeting, Warzaw, Poland
Optical microscope images of SC and LG Nb samples measured by X. Singer at DESY

SCNb1 – BCP 30 µm

SCNb2 – BCP 30 µm

SCNb7 – BCP 100 µm

LGNb3 – BCP 100 µm

BCP 100 µm provides the best surface

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Surface roughness of SC and LG Nb samples by AFM measured by X. Singer at DESY

SCNb2, BCP 30 µm
Rms: 17.6 nm

SCNb4, BCP 100 µm
Rms: 7 nm

LGNb1, BCP 30 µm
Rms: 62.7 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.

$E_{\text{acc}} = 40$ MV/m (ILC)

$E_{\text{acc}} = 30$ MV/m (XFEL)

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In-situ bakeout at 150 °C effects on LGNb3 and SCNb4

PID-regulated $U(x,y)$ for 2 nA
scanned area = 5×5 mm²
flat W-anode $\bar{a} = 100$ µm, $U_{\text{max}} = 5000$ V, $\Delta z = 25, 20, 16.6$ µm

After baking more emitters appear for LG but not for SC at E > 250 MV/m
$\Rightarrow$ first evidence for impurity segregation to grain boundaries

After Baking more emitters appear for LG but not for SC at E > 250 MV/m
$\Rightarrow$ first evidence for impurity segregation to grain boundaries
Intrinsic FE of SCNb in defect-free sample areas measured with W-tip anodes of $\varnothing_a = 5$-10 $\mu$m at $U = 5000$ V and $\Delta z > 1$ $\mu$m

SCNb4, (111) orientation
SCNb7, (100) orientation

Initially intrinsic field emission of Nb with slope $\beta = 1 \Rightarrow \Phi = 3.8$ eV
creation of an emitter at $\sim 1000$ MV/m by a microdischarge $\Rightarrow$ crater in Nb

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

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Conclusions

• DIC effectively removes particulates and weakens protrusions ⇒ 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 ⇒ reliable alternative for SRF cavities with less FE! (SRF 2007)

• Evidence for a correlation between onset field and emitter size ⇒ fast FE quality control on samples for XFEL! (SRF 2007) + FP7

• Evidence for impurity segregation to grain boundaries in LGNb after bakeout at 150ºC ⇒ reduced FE in SCNb! (SRF 2007) + FP7

• Intrinsic FE on SCNb with $\beta = 1$ and $\Phi = 4$ eV partially obtained ⇒ surface roughness enhances FE of particulates! (SRF 2007)

PhD thesis of A.Dangwal will be presumably available in November 2007
Single Crystal Cavity Fabrication

1. Take out central single crystal of definite thickness
2-3. Cutting through the disc and increasing of diameter by special rolling
4. Deep drawing
5. EB welding considering the crystal orientation

Single crystals after deep drawing at ACCEL

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

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

- possibly room for improvement by variation of deep drawing parameters
Deep drawn half cell of large grain niobium; grain boundaries pronounced, anisotropy of properties (earing)

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

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

Crystal orientation (001)
($\alpha = 2.3^\circ$, $\beta = 2.9^\circ$)

Crystal orientation (011)
($\alpha = 19.7^\circ$, $\beta = 2^\circ$)

Crystal orientation (111)
($\alpha = 11.9^\circ$, $\beta = 13.7^\circ$)

GB step ca. 12µm

GB step 2µm

GB step 15µm

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 $H_{\text{max}}$ for ILC and XFEL cavities

<table>
<thead>
<tr>
<th>Project</th>
<th>$E_{\text{acc}}$ [MV/m]</th>
<th>$H_{\text{max}}$ [mT]</th>
</tr>
</thead>
<tbody>
<tr>
<td>XFEL</td>
<td>28</td>
<td>119</td>
</tr>
<tr>
<td>ILC (BCD)</td>
<td>35</td>
<td>149</td>
</tr>
<tr>
<td>ILC (ACD)</td>
<td>45</td>
<td>162</td>
</tr>
</tbody>
</table>
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

Mean = 140 mT
St. Dev. = 14 mT

KEK

AVE. = 155
STDEV = 17

DESY

N = 12

N = 32

ABA. = 146
STDEV = 15

November 15-19, 2007

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# Material Comparison

<table>
<thead>
<tr>
<th>Supplier</th>
<th>Cavity Type</th>
<th>Ta - contents</th>
<th>RRR</th>
<th>Test $\frac{1}{2}$ $H_{\text{max}}$ [mT]</th>
<th>Test $\frac{1}{2}$ $Q_0$ at $H_{\text{max}}$</th>
<th>Test $\frac{3}{4}$ $H_{\text{max}}$ [mT]</th>
<th>Test $\frac{3}{4}$ $Q_0$ at $H_{\text{max}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>W.C.Heraeus</td>
<td>TESLA</td>
<td>&lt;500 ppm</td>
<td>500</td>
<td>140</td>
<td>1.15 x $10^{10}$</td>
<td>146</td>
<td>1.05 x $10^{10}$</td>
</tr>
<tr>
<td>Ningxia</td>
<td>TESLA</td>
<td>~ 100 ppm</td>
<td>330</td>
<td>123</td>
<td>1.5 x $10^{10}$</td>
<td>142</td>
<td>1.14 x $10^{10}$</td>
</tr>
<tr>
<td>CBMM</td>
<td>TESLA</td>
<td>~ 800 ppm</td>
<td>280</td>
<td>133</td>
<td>1.3 x $10^{10}$</td>
<td>131</td>
<td>1.04 x $10^{10}$</td>
</tr>
<tr>
<td>CBMM</td>
<td>Proton Driver</td>
<td>~ 800 ppm</td>
<td>280</td>
<td>139</td>
<td>7.5 x $10^{9}$</td>
<td>148</td>
<td>6.9 x $10^{9}$</td>
</tr>
<tr>
<td>CBMM</td>
<td>Proton Driver</td>
<td>~ 800 ppm</td>
<td>280</td>
<td>133</td>
<td>4.4 x $10^{9}$</td>
<td>135</td>
<td>1 x $10^{9}$</td>
</tr>
</tbody>
</table>
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 $E_{\text{acc}} = 47.9 \text{ MV/m}$ ($H_{\text{peak}} \sim 170 \text{ mT}$) and $E_{\text{acc}} = 43 \text{ MV/m}$ ($H_{\text{peak}} \sim 155 \text{ mT}$), respectively, after 90 µm CBP, 10 µm and 80 µm EP
- 48 hrs of baking after EP needed
- Low residual resistances measured
PKU: TESLA Shape, Ningxia Nb

PKU Cavity after Post-Purification at 1250°C

- Red dots: "after baking at 120°C for 12 hrs"
- Blue squares: "prior to baking"

- Quench
- Q-drop

Q vs. Bpeak [mT]
Figure 1: Results for the large grain cavity.

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

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

Figure 5: Temperature map for small grain cavity.
Test #6, cont’d

- The cavity was baked at 120C for 6 hrs
Summary of Performance

Eacc vs Material Removal
DESY Single Crystal Single Cell Cavity

Material Removed [micron]

Eacc [MV/m]

0 10 20 30 40 50

0 50 100 150 200 250

Material Removed [micron]

0 50 100 150 200 250

0 10 20 30 40

3 hrs at 120C

12 hrs at 120C

600C, 10hrs

20 hrs at 120C

no baking

FE limit

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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 THOAK101)

- In addition, several single cell cavities performed very well, better after EP (~ 40 MV/m)

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Name</th>
<th>Treatment</th>
<th>$E_{\text{max}}$ [MV/m]</th>
<th>$Q @ E_{\text{acc}}$</th>
<th>Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heraeus LG</td>
<td>AC 112/9-cell</td>
<td>130 μm bcp, 800C, HPR</td>
<td>30.5</td>
<td>2 x 10^{10}</td>
<td>FE</td>
</tr>
<tr>
<td>Heraeus LG</td>
<td>AC 113/9-cell</td>
<td>130 μm bcp, 800C, HPR</td>
<td>27.4</td>
<td>2 x 10^{10}</td>
<td>Quench</td>
</tr>
<tr>
<td>Heraeus LG</td>
<td>AC 114/9-cell</td>
<td>130 μm bcp, 800C, HPR</td>
<td>28.7</td>
<td>2.1 x 10^{10}</td>
<td>Quench, FE</td>
</tr>
</tbody>
</table>
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 600°C 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 $E_{acc} = 23$ MV/m,
- LG#2: quench at $E_{acc} = 20$ MV/m
Reproducibility Tests (1)

CBMM “D”           Heraeus           Ningxia

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Ta Contents</th>
<th>RRR</th>
<th>Sheet Cutting Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>CBMM</td>
<td>~800 ppm</td>
<td>~280</td>
<td>Wire EDM</td>
</tr>
<tr>
<td>W.C. Heraeus</td>
<td>&lt; 500 ppm</td>
<td>500</td>
<td>Wire Saw</td>
</tr>
<tr>
<td>Ningxia</td>
<td>&lt; 100 ppm</td>
<td>330</td>
<td>Saw + machining</td>
</tr>
</tbody>
</table>
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 600°C for 10 hrs
- App. 80-90 micron bcp (1:1:1)
- HPR for 2 hrs
- In situ baking at 120°C 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 600°C for 10 hrs
- App. 50 micron bcp (1:1:1)
- HPR for 2 hrs
- In situ baking at 120°C for 12 hrs
Reproducibility Tests (3)
Heraeus Nb, LL shape

LL Single cell cavities, Heraeus Nb, inner cell geometry

![Graph showing reproducibility tests for Heraeus Nb, LL shape](image)
Reproducibility Tests (4)

Large Grain TESLA Cavity Shape SC, Ningxia Niobium

- N5, after baking
- N5, before baking
- N4, before baking
- N4, after baking
- N3, before baking
- N3, after baking
- N2, before baking
- N1, before baking

$E_{acc} [\text{MV/m}]$

Quench
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 120°C for 12 hrs eliminates the Q-drop and
  • Increases \( \Delta/kT_c \) by 8-10% (\( \sim 1.75 \) to \( \sim 1.9 \))
  • Residual resistance often increases, \( R_{\text{BCS}} \) is lowered by \( \sim 50\% \)
  • 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

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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)