Summary of the Single Crystal Nb Workshop

Ganapati Rao Myneni
Outline

- Single Crystal Niobium Technology Workshop summary
- Where do we go from here
- A brief synopsis of historical developments (if time permits)
Success Stories with Polycrystalline Nb

- CEBAF – presently the world's largest SRF accelerator
- Heavy Ion Accelerators around the world – ATLAS, ALPI, New Delhi ……
- CESR and other light sources
- TTF
- SNS switched to SRF due to TTF successes
The Birth of Large Grain/Single Crystal Nb

Comparison of Single and Poly Crystal RRR niobium

- Load (Pounds)
- Percentage of elongation
Micro-yielding Issue PXTAL Vs XTAL
SC/LG Nb Technology

NCSU

RMS: 1274 nm fine grain bcp
251 nm fine grain ep
27 nm single crystal bcp

Hc ~ 185 mT
Large grain performance

CBMM Ingot E Eacc ~ 36 MV/m
Hc1 ~ 153 mT

BCP reduces the Hc1

RRCAT/JLab Dec. 2006
Large grain Nb
(Ta content 490 wt. ppm)
Pristine sample

Hc1 ~ 240 mT

RRCAT/JLab

Same Nb sample but with BCP

Hc1 ~ 140 mT

RRCAT/JLab

BCP, post-purification at 1250°C, 120°C 12h bake

T=2 K

T=2 K
World Wide Interest in SC/LG SRF Technology

- Almost all of the world’s SRF groups became interested in this exciting & promising path
- A Single Crystal Niobium Technology Workshop was jointly organized by CBMM – JLab and hosted at the mine by CBMM with the Motivation: to bring the Industrial Partners and SRF Scientists/Engineers/Technologists to a forum for exploring the manufacture of single crystal/large grain niobium, a potentially cost-saving direction for SRF cavity technology
- 30 Experts from the World Wide SRF Community represented at this successful workshop
SC Nb Technology Workshop

- The workshop program – 6 sections
  1. Plant Tour and Environ./Community
  2. Introductory by Carlo Pagani
  3. SC Tech. – Schoelz and Graham
  4. Lab Reports
  5. Industrial Capabilities and
  6. Discussions

- Workshop Presentations:
  http://srf.jlab.org/SingleCrystalNb/index.htm

- The AIP Proceedings of the workshop are expected to be out in July 2007
Araxá Mine & RRR Niobium

From ore to oxide to large grain ingots

The CBMM open cast mine

Conveyor belt bringing the ore to concentration plant

Electron beam furnace for the refinement of Niobium metal, producing 210 tonnes per annum

Finished RRR Nb ingot from the Pyrochlore ore
Status of SC/LG Nb Development

- Graham and Schoelz outlined:

1. The difficulties in developing reproducible single crystal (~265 mm) technology with the only possible e-beam drip melting process after briefly reviewing various technologies

2. Process control challenges include optimum melting conditions during the melting process, thermal and mechanical turbulences due to the dripping of the molten niobium droplets into the molten pool etc.

3. The nucleation and solidification of the grains start at various inhomogeneities in the molten pool that become dendrites. Supercooling at the periphery of the molten pools leads to additional nucleation sites, and a rim of equiaxed grains

4. No computer analytical programs are available to simulate/develop single crystal growing processes

5. Internal funding is not available to develop this technology and external funding is required to provide the incentive for further developments
Various Single Crystal Technologies

- Czochralski-process
- Bridgman-processes
- Zone melting
- E-Beam Melting
1. JLab (GM, PK) and collaborators from NIST (RR, TG-H) and NCSU (PR)

2. DESY (DP, W&XS) and collaborators ACCEL (MP)

3. FNAL (CA, HE) and collaborators from MSU (CC) and X-Wisconsin (PL of FSU)

4. KEK (KS)

5. Industries; CBMM (AO), Heraus (BS, FS), H. C. Starck (PJ), Tokyo Denkai (HU) and last but not least from the oldest and largest RRR Nb processing capable Wah Chang (RG)
Large Grain/Single Crystal Niobium

- CBMM
  - Ingot “A”, 800 ppm Ta
  - Ingot “B”, 800 ppm Ta
  - Ingot “C”, 1500 ppm Ta

- Ninxia
  - Ingot “D”, 800 ppm Ta

- Wah Chang
  - Ingot “E”, 490 ppm Ta

- Heraeus

Thomas Jefferson National Accelerator Facility
Operated by the Jefferson Science Associates for the U.S. Department Of Energy
Minor Fab Issues are being successfully addressed

Half cell from CBMM Ingot “B”

Rugged Edges

Thinning
Heraeus and Ninxia Large Grain Half Cells

Deep drawn half cell of HERAEUS large grain niobium: Large single crystal

Deep drawn half cells of Ninxia L.G.
A Selection of the Results

Single Xtal Stress-strain curves

TEM image of the Single Xtal Nb oxide

LG Nb from 3 vendors perform equally well
Hc1 140 – 160 mT

TESLA LG Nb 9 cell cavities from ACCEL

Operated by the Jefferson Science Associates for the U.S. Department Of Energy
Best Single Crystal 1.3 GHz Cavity Result

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

- T = 1.99K, 6 hrs at 120°C baked
- T = 1.8K, 6 hrs at 120°C baked
- "T = 2K, before baking"

Quench @ 37.5 MV/m
Q - drop

E_{acc} [MV/m]
Conclusions from Lab Reports

- Single Crystal Nb is Best, yet to be available in large quantities
- Attention is mainly on the large grain Nb cavities:
  - No show stoppers, lowest Rs, no additional QA, EP is not required, in-situ baking time can be reduced. Procedures can be streamlined & may have to be slightly different from polycrystalline Nb cavities.
  - No micro-yielding, higher yield strength, orientation dependent deformation was considered to be a minor issue (ACCEL demonstrated by producing 3, 9 cell TESLA cavities with no hiccups)
Future work topics

During the course of our work in the last 2 years, we have identified several R&D topics

- Effective cutting: wire EDM too slow?
- Forming: how uniform, grain slippage
- Welding/Vacuum: do grain boundaries cause problems?
- Surface roughness: appropriate acid agitation during bcp, uniformity of material removal
- Mechanical properties depending on grain orientation?
- Internal stresses and strains due to deep drawing using neutron diffraction techniques
- Dependence of oxidation on grain orientation
SC Nb Workshop Summary

- CBMM and Heraeus are currently producing & supplying LG Niobium
- Tokyo Denkai is planning to produce LG Nb from July 2007
- If/When 200 RRR Nb is accepted for SRF cavities, production capacity can be increased and the cost of RRR Nb will be reduced considerably
Path to economic SC/LG Niobium & ILC
What is in the Future

Discussion Topics

- What industry needs to develop the Technology
  — Funding needs, facility upgrades, time frame
- What is required to demonstrate SC/LG Technology is ready to be applied to ILC?
- What would be the benefits of using LG/SC technology and how would you demonstrate the benefits
  — costs, reproducibility, performance and streamlined procedures ..... 
- I propose a special LG Nb session at the SRF workshop in Beijing later this year and also special sessions at the TTC meetings
# Many Thanks to Collaborators

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<th>Name</th>
<th>Institution</th>
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<tbody>
<tr>
<td>Tadeu Carneiro</td>
<td>CBMM/Reference Metals, Brazil</td>
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<tr>
<td>Peter Kneisel, Gigi Ciovati</td>
<td>JLAB</td>
</tr>
<tr>
<td>Richard Ricker, T. G-Herold</td>
<td>NIST</td>
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<tr>
<td>Fred Stevie, Phillip Russell</td>
<td>NCSU</td>
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<tr>
<td>Xenia &amp; Waldemar Singer</td>
<td>DESY, Germany</td>
</tr>
<tr>
<td>Bill Lanford</td>
<td>UNY Albany</td>
</tr>
<tr>
<td>Björgvin Hjörvarsson</td>
<td>Uppsala University, Sweden</td>
</tr>
<tr>
<td>Sean Agnew, B. Shivaram</td>
<td>UVa</td>
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<tr>
<td>Sindhunil Roy</td>
<td>RRCAT India</td>
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What do I plan to do next!

- Optimize the cavity processing parameters in order to maintain the highest $Hc1$ of the pristine Niobium
- Eliminate the surface oxide layer, remove the dissolved hydrogen and deposit several mono layers of NbN to passivate the surface and enhance the performance from the proximity effect
- Revisit the Nb specs with an aim to reduce the costs while maintaining the performance even with high Ta content and or reduced RRR
JLab’s CEBAF History

- Professor M. Tigner’s 1979 vision of selecting an application for improving the SRF Technology came through in 1987 – LE 5 Cornell – CEBAF
- CEBAF is presently the world’s largest solid Nb SRF Accelerator – 220 meters – operated up to 6 GeV prior to hurricane-related degradation
- Is built with polycrystalline RRR niobium originating from Pyrochlore ore in Brazil - CBMM
- Most of the prior research on SRF cavities was with Columbite/Tantalite based niobium
- Recontamination with organic/particulates from vacuum systems minimized – a success story
POST CEBAF

- DESY Initiated TTF – improved SRF Technology with EP (KEK), high pressure high purity water rinsing – minimizing particulate trapping, use of oil-free vacuum systems & QA of RRR Nb
- SNS took SRF path – most of the niobium was derived from Columbite/Tantalite ore
- Micro-yielding was a major issue with low tantalum RRR niobium and continues to be so
- Segregation of Ta could also be a problem
- JLAB/CBMM invents SC/LG Nb – ILC goes SRF