New Nb Material for Cost Saving

Akira Yamamoto (KEK-CERN)

In cooperation with
A. Kumar, T. Dohmae, T. Saeki (KEK), G. Myneni (BSCE), G. Ciovati, P. Dhakal, R. Rimmer (JLab), M. Carl, A. Fajardo, and N. Lannoy (ATI)

LCWS-2023, Acc. SR Session, at SLAC, Sept. 16, 2023
Motivations

• The best cost-effective SRF cavity production:
  • Quality:
    • Clean Nb and RF surface critically important,
  • Cost:
    • Simple production process inevitably required.

• Efforts for a solution:
  • Fine Grain (FG):
    • Mechanically best, but contamination due to forging and rolling
  • Large Grain (LG):
    • Best for cleanness and cost reduction, but mechanically not stable resulting in HPGS code not applicable
  • Medium Grain (MG):
    • Optimized for cleanness, mechanical stability, cost reduction, and applicable to high pressure gas safety (HPGS) regulations.
References


• A. Kumar et al., “Mechanical property of directly sliced me-dium grain niobium for 1.3 GHz SRF cavity”, JACOW-SRF-2021-MOPCAV004.

• T. Dohmae et al., “FABRICATION OF 1.3GHz SRF CAVITIES USING MEDIUM GRAIN NIOBİUM DISCS DIRECTLY SLICED FROM FORGED INGOT*, JACoW-SRF2021-MOPCAV012.

Production of Nb Discs/Sheets for 1.3 GHz SRF Cavities

EB melting

Ingot, as it is

Slicing by wire-saw

Large Grain (LG) Disc
Grain Size >> 1 cm

Ingot forged

Ingot to Billet (smaller dia.)
formed and annealed

Slicing by wire-saw

Medium Grain (MG) Disc
Grain Size < 1 mm

Ingot forged

Rolling

Fine-Grain (FG) Nb Sheet
Grain Size << 0.1 mm

• ATI(US) provided directly sliced disc from forged MG Nb ingot.
• RRR ~ 100.
• Grain size: ASTM 3.5–5.5.
• Disc thickness: 3.1 mm.
• Forged MG Nb disc was appropriately heat-treated.
• The MG Nb disc was press-formed at KEK.

Note: ATI(US) is the company which supplied most of NbTi material for CERN-LHC superconducting magnets.

For further information about ATI, please see the last slide (Appendix) of this presentation.

2023-5-16
LCWS-2023, SLAC
Process flow of the industrial Nb production

1. Mother Material
   - TELEDyne WAB CHANG ALBANY
     - Nb Grade 1 powder
     - -60 +200 Mesh
   - Forging
   - Test Piece
   - RRL-X-Ray, ICP
   - Base plate
   - Use for next Ingot

2. Pressing
   - Size: 65x80x380 [mm]
   - Weight: 12.5 Kg / block

3. EB Melting (1st)
   - The material is supplied from Side bar feeder.

4. Forging
   - Cold Forging
   - This process is done at another company.

5. Cutting
   - 720°C x 120min < 1E-5 Torr

6. Annealing
   - HF:HNO₃:H₃PO₄
     - 1:1:1

7. Mechanical grinding
   - Remove a scale
   - 35 x 195 x L [mm]

8. Rolling
   - 4.0 x W x L [mm]

9. Annealing
   - 720°C x 120 min < 1E-5 Torr

10. Rolling
    - 4.0 x W x L [mm]

11. Cutting
    - 4.0 x W x L [mm]

12. Polishing
    - Emery Paper

13. Chemical Polishing
    - HF:HNO₃:H₃PO₄
     - 1:1:1

14. Annealing
    - 720°C x 120 min < 1E-5 Torr

15. Testing
    - Hardness
    - Gas content
    - RRR
    - Grain size
    - Tensile test

16. Packing

 Courtesy: Tokyo Denkai
**Process flow of the industrial Nb production**

<table>
<thead>
<tr>
<th>Step</th>
<th>Process</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Mother Material</td>
</tr>
<tr>
<td>2.</td>
<td>Pressing</td>
</tr>
<tr>
<td>3.</td>
<td>EB Melting (1st)</td>
</tr>
<tr>
<td>4.</td>
<td>EB Melting (2nd, 3rd)</td>
</tr>
<tr>
<td>5.</td>
<td>Rolling</td>
</tr>
<tr>
<td>6.</td>
<td>Forging</td>
</tr>
<tr>
<td>7.</td>
<td>Mechanical grinding</td>
</tr>
<tr>
<td>8.</td>
<td>Rolling</td>
</tr>
<tr>
<td>9.</td>
<td>Annealing</td>
</tr>
<tr>
<td>10.</td>
<td>Rolling</td>
</tr>
<tr>
<td>11.</td>
<td>Cutting</td>
</tr>
<tr>
<td>12.</td>
<td>Polishing</td>
</tr>
<tr>
<td>13.</td>
<td>Chemical Polishing</td>
</tr>
<tr>
<td>14.</td>
<td>Annealing</td>
</tr>
<tr>
<td>15.</td>
<td>Testing</td>
</tr>
<tr>
<td>16.</td>
<td>Packing</td>
</tr>
</tbody>
</table>

**LG-Nb** realized very good cost performance. But LG-Nb material causes broad mechanical strength distribution (less uniformity).  
(\(\rightarrow\) Minimum strength becomes low)

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2023-5-16

LCWS-2023, SLAC
Uniformity of Nb material is achieved by Medium-Grain (MG) structure.
# Summary of Nb Sheet/Disc Fabrication Technologies for SRF

<table>
<thead>
<tr>
<th></th>
<th>Fine Grain (FG) Rolled Nb sheets</th>
<th>Medium Grain (MG) Forged Ingot Nb discs</th>
<th>Large Grain (LG) Ingot Nb discs</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fabrication process</td>
<td>Up to fourteen manufacturing steps</td>
<td>EB melted ingot w/ larger dia. Forging required dia to be sliced</td>
<td>EB melted ingot of required diam. to be sliced</td>
</tr>
<tr>
<td>ASTM Grade Grain size</td>
<td>~ 5 ~ &lt; 50 µm</td>
<td>0 – 3, (1~2 av.) &lt; 1 mm</td>
<td>n/a &gt;1 cm</td>
</tr>
<tr>
<td>Cleanness</td>
<td>Possible prone to contamination due to rolling</td>
<td>clean surface with direct slicing</td>
<td>Proven clean surface with no-forging and direct slicing</td>
</tr>
<tr>
<td>Mechanical properties</td>
<td>Very Uniform</td>
<td>Reasonably uniform</td>
<td>Non uniform, widely distributed</td>
</tr>
<tr>
<td>Cost</td>
<td>Expensive</td>
<td>Cost advantage</td>
<td>Best cost advantage</td>
</tr>
</tbody>
</table>
Properties of MG Nb disc realized in cooperation with ATI

Table 1. Material properties of MG Nb material produced by ATI.

<table>
<thead>
<tr>
<th>Chemical Composition (%)</th>
<th>Other properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ta : &lt; 0.01%</td>
<td>Dimensions: 260 m (dia.), 2.8 mm (t)</td>
</tr>
<tr>
<td>W : &lt; 0.003 %</td>
<td>RRR : &gt; 450</td>
</tr>
<tr>
<td>Ti, Si, Mo, Fe : &lt; 0.003%</td>
<td>Recrystallization : 100 %</td>
</tr>
<tr>
<td>Ni : &lt; 0.002 %</td>
<td>Grain size : 0.2 ~ 0.3 mm (ASTM: 1 ~ 2)</td>
</tr>
<tr>
<td></td>
<td>Hardness (HV0.1) : 40 ~ 44</td>
</tr>
<tr>
<td>H2 : &lt; 0.003%</td>
<td>Mechanical Strength :</td>
</tr>
<tr>
<td>C : &lt; 0.002 %</td>
<td>Ultimate strength (RT) &gt; 141-146 Nmm^{-2}</td>
</tr>
<tr>
<td>N2 : &lt; 0.002 %</td>
<td>Yield strength (RT) &gt; 56-61 Nmm^{-2}</td>
</tr>
<tr>
<td>O2 : &lt; 0.004 %</td>
<td></td>
</tr>
</tbody>
</table>

Special Thanks for the Cooperation by ATI!
# A Reference: Comparison of NbGr1 & RRR Nb

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Nb Gr1 (MG) Measured</th>
<th>RRR Nb (MG) Measured</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>RRR</strong></td>
<td>~ 100</td>
<td>450, 523</td>
</tr>
<tr>
<td><strong>Re-crystallization</strong></td>
<td>100 %</td>
<td>100 %</td>
</tr>
<tr>
<td><strong>Grain size (ASTM)</strong></td>
<td>0.5 - 5</td>
<td>2, 1, 1.5</td>
</tr>
<tr>
<td><strong>Edge, Mid., Center</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Grain size (mm)</strong></td>
<td>0.05 - 0.3</td>
<td>0.2, 0.25, 0.21</td>
</tr>
<tr>
<td><strong>Edge, Mid., Center</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Y.S.-0.2% (RT)</strong></td>
<td>~ 67 MPa</td>
<td>61 MPa</td>
</tr>
<tr>
<td><strong>T.S. (RT)</strong></td>
<td>&gt; 147 MPa</td>
<td>141 MPa</td>
</tr>
</tbody>
</table>

Courtesy: ATI and G. Myneni,
Room temperature property Comparison

Mechanical strength of MG-Nb achieved the criteria of HPGS regulation for KEK/STF-Cavity

- MG-Nb closer to FG-Nb than LG-Nb
- Elongation is lower than FG Nb but unnecessary for HPGS
- High elongation necessary for press forming of half cells.

- MG Nb data: [https://jacow.org/srf2021/papers/mopcav004.pdf](https://jacow.org/srf2021/papers/mopcav004.pdf)
- FG Nb and LG Nb data is for Mid-RRR annealed material (M.Yamanaka et al., SRF’21 WEPFDV005).
Room temperature property Comparison

Mechanical strength of MG-Nb achieved the criteria of HPGS regulation for KEK/STF-Cavity

- MG-Nb closer to FG-Nb than LG-Nb
- Elongation is lower than FG Nb but unnecessary for HPGS
- High elongation necessary for press forming of half cells.

HPGS issues to be more discussed by K. Umemori, tomorrow, Wednesday!
**Low Temperature Property Comparison**

- Tensile Strength of MG-Nb at LHe-T is better than LG-Nb.
- Brittleness and low elongation of MG-Nb are not observed at LHe-T after annealing.
## Summarized Results

### ATI MG Nb Specimens

<table>
<thead>
<tr>
<th>Temperature [K]</th>
<th>Sample Processing</th>
<th>Young’s Modulus [GPa]</th>
<th>0.2% Proof Strength [MPa]</th>
<th>Tensile Strength [MPa]</th>
<th>Elongation [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>300</td>
<td>Annealed</td>
<td>88.7±9*</td>
<td>39±2</td>
<td>123±5</td>
<td>25.3 ± 3</td>
</tr>
<tr>
<td>300</td>
<td>ASR</td>
<td>89.7±6</td>
<td>43±4</td>
<td>145±7</td>
<td>23.9 ± 4</td>
</tr>
<tr>
<td>4.2</td>
<td>Annealed</td>
<td>114.0±11</td>
<td>283±34</td>
<td>651±60</td>
<td>7.5±2</td>
</tr>
<tr>
<td>4.2</td>
<td>ASR</td>
<td>115.4±14</td>
<td>284±22</td>
<td>351±28</td>
<td>1.8±1</td>
</tr>
</tbody>
</table>

* Error is standard deviation.
Summary of Mechanical Test Results

• ATI MG Nb’s mechanical properties does **clear the mechanical strength criteris required for HPGS code** for KEK-STF SRF Cavity.

• However, elongation of the material needs to be improved for better yield with press forming.

• MG Nb room **temperature** properties are **closer to FG Nb**.

• Low **temperature** behavior of MG Nb is **closer to LG Nb**

• Elongation is same as FG and LG Nb at low temperature; however, some specimens did show lower elongation of **5-6%** too.

• **First Serration** usually starts at **250 to 300 MPa** stress.
MG Cavity Fabrication:
MG-Nb Disc successfully press formed to half-cells,

- ATI(US) provided directly sliced disc from forged MG Nb ingot.
- RRR ~ 100.
- Grain size: ASTM 3.5–5.5.
- Disc thickness: 3.1 mm.
- Forged MG Nb disc was appropriately heat-treated.
- The MG Nb disc was press-formed at KEK.

Directly sliced Nb disc from forged MG Nb ingot recently demonstrated in cooperation with ATI.


Note: ATI(US) is the company which supplied most of NbTi material for CERN-LHC superconducting magnets.

For further information about ATI, please see the last slide (Appendix) of this presentation.

Figure 5: Histogram of measured roundness.
An Issue in Press-Forming Process
- settled with enlarging the disc, inner-hole diameter prior to press-forming

<table>
<thead>
<tr>
<th>Disc #</th>
<th>Hole Diameter [mm]</th>
<th>Annealing</th>
<th>Forming Results</th>
<th>Purpose</th>
</tr>
</thead>
<tbody>
<tr>
<td>#6</td>
<td>56</td>
<td>No</td>
<td>Light crack</td>
<td>R18</td>
</tr>
<tr>
<td>#7</td>
<td>56</td>
<td>No</td>
<td>Light crack</td>
<td>R18</td>
</tr>
<tr>
<td>#8</td>
<td>56</td>
<td>Yes</td>
<td>Deep crack</td>
<td>Test</td>
</tr>
<tr>
<td>#9</td>
<td>59</td>
<td>Yes</td>
<td>No crack</td>
<td>Test</td>
</tr>
<tr>
<td>#10</td>
<td>58</td>
<td>Yes</td>
<td>No crack</td>
<td>Test</td>
</tr>
<tr>
<td>#57</td>
<td>60</td>
<td>Yes</td>
<td>No crack</td>
<td>R18b</td>
</tr>
<tr>
<td>#58</td>
<td>58</td>
<td>No</td>
<td>Light crack</td>
<td>R18b</td>
</tr>
<tr>
<td>#59</td>
<td>56</td>
<td>No</td>
<td>Deep crack</td>
<td>Test</td>
</tr>
<tr>
<td>#60</td>
<td>58</td>
<td>No</td>
<td>Deep crack</td>
<td>Test</td>
</tr>
</tbody>
</table>
Evaluation of MG Surface Roughness Issue

**Surface roughness**

<table>
<thead>
<tr>
<th></th>
<th>Ra [μm]</th>
<th>Rz [μm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Disc</td>
<td>0.7</td>
<td>-</td>
</tr>
<tr>
<td>Formed</td>
<td>2.6 ~ 5.7</td>
<td>11 ~ 28</td>
</tr>
<tr>
<td>Polished</td>
<td>2.6 ~ 3.7</td>
<td>2.6 ~ 11</td>
</tr>
<tr>
<td>EP (100μm)</td>
<td>1.1 ~ 1.5</td>
<td>3.4 ~ 6.7</td>
</tr>
</tbody>
</table>

**Surface**

Became rough after forming.
(especially at curvature regions)

R18: Mechanically polished (equator)
MG 1-Cell Cavity RF Performance compared with FG 1-cell Cavity

**RF Testing**

**Surface treatment**
1. Initial electropolishing of 100 μm
2. Annealing at 800 °C × 3 hours in a vacuum furnace
3. Second electropolishing of 20 μm
4. High pressure rinsing with ultra-pure water
5. Baking at 120 °C × 48 hours

**Note:** 9-cell MG Cavity fabrication **in progress**
Summary of Features for the FG, MG, LG Nb

### Refining

<table>
<thead>
<tr>
<th>Process</th>
<th>FG</th>
<th>MG</th>
<th>LG</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Primary material</td>
<td>✔</td>
<td>✔</td>
<td>✔</td>
</tr>
<tr>
<td>2. Pressing</td>
<td>✔</td>
<td>✔</td>
<td>✔</td>
</tr>
<tr>
<td>3, 4. EB Melting (multiple)</td>
<td>✔</td>
<td>✔</td>
<td>✔</td>
</tr>
<tr>
<td>5. Separating Ingot</td>
<td>✔</td>
<td>✔</td>
<td>✔</td>
</tr>
<tr>
<td>6. Forging to reduce</td>
<td>✔</td>
<td>✔</td>
<td>-</td>
</tr>
<tr>
<td>(thickn. or dia.)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7. Milling</td>
<td>✔</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>8. Rolling</td>
<td>✔</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>9. Polishing</td>
<td>✔</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>* Annealing</td>
<td>✔</td>
<td>✔</td>
<td>-</td>
</tr>
<tr>
<td>10. Rolling</td>
<td>✔</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>11. Cutting (sheet)</td>
<td>✔</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>* Direct Slicing (disc)</td>
<td>-</td>
<td>✔</td>
<td>✔</td>
</tr>
<tr>
<td>12. Annealing</td>
<td>✔</td>
<td>✔</td>
<td>✔</td>
</tr>
</tbody>
</table>

### Sheet/Disc forming

<table>
<thead>
<tr>
<th>Process</th>
<th>FG</th>
<th>MG</th>
<th>LG</th>
</tr>
</thead>
<tbody>
<tr>
<td>13. Leveling</td>
<td>✔</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>15. Inspection</td>
<td>✔</td>
<td>✔</td>
<td>✔</td>
</tr>
</tbody>
</table>

### Finishing

<table>
<thead>
<tr>
<th>Feature</th>
<th>FG</th>
<th>MG</th>
<th>LG</th>
</tr>
</thead>
<tbody>
<tr>
<td>- Production cost</td>
<td>High (1)</td>
<td>Low (~2/3)</td>
<td>Lowest (&lt;2/3)</td>
</tr>
<tr>
<td>- Cleanness</td>
<td>Modest</td>
<td>Good</td>
<td>Good</td>
</tr>
<tr>
<td>- Mech. forming</td>
<td>Best</td>
<td>Good</td>
<td>Modest</td>
</tr>
</tbody>
</table>
**ITN WPP-1 Plan:** FG + MG Cavities, Indust. Product. Readiness

- **Pre-production** with 1-cell cavities to establish the **best process** including:
  - Nb sheet and SRF cavity production method
  - Inner surface treatment recipe

- **Production of > 20 (~3 x 8) x 9-cell cavities** for industrial-production readiness:
  - Globally common design with compatible High Pressure Gas Safety (HPGS) regulation
  - Best production process to be optimized in each region
  - Cavity performance expected: \( E_{\text{acc}} = <35 \text{ MV/m}> (+/- 20\%), Q_0 = 1.0 \times 10^{10}, \text{ Yield} = \geq 90\% \)

- RF performance yield to be re-confirmed (including 2\textsuperscript{nd} pass and further (3\textsuperscript{rd} …)

### Material/Sub-component

- QA of Material/Sub-C

### Cavity Production

- Surface Process

- Vertical Test = Cavity RF Test

### Production process

#### # of cavities to be produced

<table>
<thead>
<tr>
<th>Region</th>
<th>Americas</th>
<th>Europe</th>
<th>JP/Asia</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type</td>
<td>FG + MG</td>
<td>FG + MG</td>
<td>FG + MG</td>
</tr>
<tr>
<td>1-cell</td>
<td>1 + 1</td>
<td>1 + 1</td>
<td>1 + 1, (+4)</td>
</tr>
<tr>
<td>9-cell</td>
<td>4 + 4</td>
<td>4 + 4</td>
<td>4 + 4, (+4)</td>
</tr>
</tbody>
</table>

*Courtesy: S. Michizono, Y. Yamamoto*
WP-prime 3: Crab Cavity Development

- Pre-down-selection review hosted by KEK chose two primary candidates on Apr/2023
  - RFD (1st), QMiR (2nd), Elliptical (3rd)
- Development and evaluation of two prototype cavities
  - KEK will provide for necessary Nb material to produce them
- RF property simulation to optimize cavity design
- Demonstration of synchronized operation with two prototypes
- Down-selection to choose final cavity design
- Cryomodule design based on final cavity design

Both two candidates are from US!

two beamline distance
14.049m x 0.014rad = 197mm

Possible MG-Nb for Prototype
Summary

• **MG Nb** material has been **successfully developed** to qualify RF performance, qualified for 1.3 GHz SRF cavity application.

• **MG Nb** disc may save the production cost to be ~ 2/3 of the FG Nb sheet production cost.

• The RF performance has been demonstrated with a 1-cell 1.3 GHz SRF cavity satisfying the ILC SRF cavity specification. **9-cell in progress.**

• The **industrial production readiness** for 1.3 GHz 9-cell cavities is to be demonstrated in the **ILC-ITN global cooperation**.

• **For the future ...**
A Special Topic!, toward the Future Cost Saving!
1.3 GHz, 1-cell Cu full-seamless cavity with new hydroforming to be combined with thin Nb/Nb3Sn coating
-- KEK-CERN Cooperation --

Seamless Cavity Hydroforming Process, proposed by M.Y.

2023-5-16

Simulated by CERN and Realized by KEK
• Simulation and material characterization, by M. Garlashce et al. at FCC-week and at SRF2023,
• Hydroforming details will be presented by M. Yamanaka et al., at SRF2023

Courtesy:
- M. Yamamaka
- M. Carlasche

 hüg

Succeeded, last week (in 2 days)