## Low RRR Materials for ILC

## Ganapati Myneni

Jefferson Lab ISOHIM & VNECA

LCWS13, The University of Tokyo

Nov 11-15, '13 – Tokyo

## Outline

## **Early History**

### **Present Status**

## **Future Prospects**

## Summary

# **Historical Example of Ingot Niobium 1**



FIG. 1. An electron-beam welded  $TM_{010}$  mode Nb cavity. The cavity is resonant at 8.6 GHz and is 3.6 cm in over-all length.

H<sub>pk</sub>~ 108 mT with BCP

#### **Stanford solid niobium cavity 1970**

## **Historical Example of Ingot Niobium 2**

#### Siemens solid niobium cavity 1973



 $H_{pk}$ ~ 109 mT with BCP  $H_{pk}$ ~ 130 mT with EP

Fig. 1. Single piece  $TM_{010}$ -niobium cavity with a resonant frequency of 9.5 GHz.

EP' d reactor grade fine grain niobium cavity set a record Hpk of 159 mT



# **Highlights of Early SRF Technology**

- Cavities were mostly made from ingot niobium
  - Process and procedures were similar and as varied as today
- Reactor grade Niobium material in ingot, bar, plate sheet and tube form was available
- Achievable gradient limited by multipacting and/or field emission
- Residual surface resistance (~ 1nΩ) was not well understood
  Still the case
- At highest frequencies (Electropolished fine grain, X-band) Hpk ~ 159 mT Q0 ~5x10<sup>9</sup>
- (BCP'd ingot Nb, 1970's) Hpk ~ 108 mT & Q0 ~1 × 10<sup>11</sup> @ 1.2 K CW
- For comparison (CEBAF upgrade spec.) Hpk ~ 76 mT Q0 ~ 7 × 10<sup>9</sup> @ 2 K CW (2008)

# Extrinsic and intrinsic contamination of Nb determines the performance of the cavities

#### Extrinsic

- Surface contamination
  - Molecular and particulate

#### Intrinsic

- Niobium is a prolific hydrogen absorber in the absence of the natural surface oxide
  - Hydride formation
  - Dislocations

## **Niobium Specifications – Past & Present @JLab**

- Polycrystalline Niobium with ASTM #5 Grain Size or finer ~ 50 micro meters & 90% recrystallized
- Percentage of elongation > 25
- Yield Strength > 10.7 KSI (~75 MPa) (7 KSI for SNS)
- RRR > 250
- Tantalum < 1000 wt ppm

Note:

Recrystallization and high yield strength (YS) are mutually exclusive, the "kiss pass" used for increasin the YS introduces significant surface damage

Note: These specifications are wrt the physical structure only & do not include SC properties

# **Niobium For SRF Cavities**

- At present, Niobium for SRF cavities comes from Columbite/Tantalite ore
  - Niobium is present as "impurity"
  - Niobium is produced as a by-product
- Primary reason the Tantalum content is lower
  - Tantalum is generally believed to negatively impact
    SRF properties of Niobium
  - JLab data shows reducing Tantalum content below 1000 ppm has no advantage for Superconducting RF cavities
  - Low Tantalum niobium is relatively expensive

#### **Process steps - fine grain Niobium**





## **Multi cell cavity fabrication**



~90% of CEBAF cavities were made with CBMM Pyrochlore ore based niobium In comparison to present day use of Tantalite/Columbite ore based niobium

# Niobium cavity – performance (CW)





In nearly 40 years  $E_{acc}$  improved by a factor of 5, DOE NP and Jlab improved  $Q_0$  by a factor of ~4 Ti doping (2011) FNAL ~4 N doping(2012)



Purity specification determines no of E-beam melting cycles and cost

# **Cavity ILC processing steps**

- Buffer chemical polishing (BCP) ~ 150 micro meters
- Electro polishing (EP) ~ 50 micro meters
- High pressure ultra pure water rinse
- ~ 600 900 °C heat treatment
- Light EP/Flash BCP
- High pressure ultra pure water rinse
- Vacuum bake ~120 °C for up to 48 hours
- RF test

**Present state of the art** 

Lower construction cost



Ingot niobium Rs is low and phonon peak improves thermal stability

Our goal is to improve both Q<sub>0</sub> and E<sub>acc</sub>

# Uniform distribution of Ta has no effect on SC Parameters, BCP has



Sample	Ta-Content	$T_C$ at 100 Oe	$H_P$	$H_{C2}$	$H'_{C2}$
	(ppm)	(K)	(Oe)	(Oe)	(Oe)
Technical-Niobium-1P	$1339{\pm}36$	9.18	1700	4150	7500
Technical-Niobium-2P	$800 \pm 80$	9.18	1600	4125	7500
Technical-Niobium-3P	$243{\pm}10$	9.2	1600	4090	7500
Technical-Niobium-1CT	$1285 \pm 35$	9.05	1150	3930	-
Technical-Niobium-2CT	$684 \pm 54$	9.05	1290	3735	-
Technical-Niobium-3CT	$149 \pm 11$	9.06	1350	3820	-



#### Tantalum and RRR have minimal influence on phonon peak



Specimen	Estimated RRR	Tantalum content (ppm) [3]	Heat Treatment	Titanium getter
1	191	1275	600 °C, 6 hrs	No
2	131	668	600 °C, 6 hrs	No
3	190	756	750 °C, 2 hrs	Yes
4	196	756	750 °C, 2 hrs	Yes
5	104	1322	800 °C, 2 hrs	No
6	143	523	800 °C, 2 hrs	No

PhD thesis at MSU

#### **Economic path for future SRF systems**



#### Araxá Mine in Brazil & Ingot Niobium

#### The CBMM open cast mine





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

## From ore to oxide to large grain ingots

Conveyor belt bringing the ore to concentration plant





Finished RRR Nb ingot from the Pyrochlore ore



Residual stress is largest near the equator and Iris Peak surface magnetic filed is highest near the equator, where quenches originate, etch pits seen and is the bed for hydrogen-dislocation interactions

## **Hydrogen-dislocation interactions**



M. G. Rao and P. Kneisel, Mechanical properties of high RRR niobium at cryogenic temperatures, Adv. in Cryogenic Engineering Vol. 40 1383-1390, 1994

We would like to further investigate this phenomena

Intrinsic contamination of Nb & proton-dislocation interaction appear to determine the performance of the cavities

- Niobium is a prolific hydrogen absorber in the absence of the natural surface oxide<sup>\*</sup>
  - Hydride formation
  - Dislocations-proton interaction

\* R.E. Ricker, G. R. Myneni, J. Res. Natl. Inst. Stand. Technol. 115, 353-371 (2010)

# Our goal is to Simplify process steps

- Minimize the process steps
  - H-free mechanical polishing ~ 100 micro meters  $^*$
  - High pressure ultra pure water rinse
  - High temperature heat treatment
  - Megasonic cleaning
  - RF test
- These simple steps reduce proton-dislocation interactions and hence expected unparalleled high performance delivery

\* T. Higuchi and K. Saito, "Hydrogen Absorption in Electropolishing of Niobium", CP 671 Hydrogen in Materials and Vacuum Systems pp 203-219 2002 Edited by G. R. Myneni and S. Chattopadhyay

#### Improved mechanical properties with 1400 C HT



R.P Walsh and D. M. McRae NHMFL 2012

# **Future Outlook**

- Ingot niobium technology (low RRR, high tantalum content) has proven to be ideal for CW SRF applications
- We expect that this technology will be the preferred choice for future superconducting CW linacs worldwide
- Several Labs from the three continents are discussing a joint program to optimize the ingot niobium multi cell cavity processes for high efficiency & high intensity CW linac applications

#### JLab's worldwide network of collaborators

Tadeu Carneiro, Marcos Stuart – CBMM

F. Stevie, P. Maheswari, D. Griffis – NCSU

R. Ricker – NIST

J. Wallace – Casting Analysis Corporation

Björgvin Hjörvarsson – Uppsala University B. Lanford – UNY, Albany R. Pike and summer student interns – W&M Hani Elsayed-Ali, Ashraf Hassan Farha – ODU Asavari Dhavale & J. Mondal – BARC/HBNI Sindhunil Roy – RRCAT Saravan Chandrasekaran – MSU

ingot niobium technology niobium surface science hydrogen-niobium system co-PI DOE ONP ARRA Q<sub>0</sub> improvement program hydrogen-niobium system nuclear reaction analysis **XRD** analysis of niobium niobium nitride ingot niobium properties SC properties of niobium ingot niobium properties

International Symposium On Hydrogen In Matter (ISOHIM) non profit organization for education/training

Acknowledgements to all colleagues at JLab

# International Symposium On Hydrogen In Matter (ISOHIM) Publications

Hydrogen in Materials and Vacuum Systems AIP CP 671

http://www.virtualjournals.org/dbt/dbt.jsp?KEY=APCPCS&Volume=671&Issue=1

Hydrogen in Matter AIP CP 837

http://www.virtualjournals.org/dbt/dbt.jsp?KEY=APCPCS&Volume=837&Issue=1

Single Crystal Large Grain Niobium AIP CP 927 http://www.virtualjournals.org/dbt/dbt.jsp?KEY=APCPCS&Volume=927&Issue=1

Superconducting Science and Technology of Ingot Niobium AIP CP 1352 http://scitation.aip.org/dbt/dbt.jsp?KEY=APCPCS&Volume=1352&Issue=1