CAVITY FABRICATION

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1



500 MHz, Single-cell









350 MHz, 4-cell, Nb on Cu









1500 MHz, 5-cell











1300 MHz 9-cell









Nb = SRF

- Niobium is the material of choice to fabricate SRF cavities:
 - High critical temperature ($T_c = 9.25K$)
 - High critical field ($H_c(0K) \cong 200mT$)
 - Chemically inert (surface covered by oxide layer)
 - Easily machined and deep drawn
 - Available as bulk and sheet material in any size









Niobium, Brief History

 Named after Niobe (daughter of Tantalus, Greek mythology)

 Discovered in 1801 by Charles Hatchett in England from a columbite ore from Connecticut. He called it Columbium







Niobium, Brief History

- It was confused with tantalum until 1846, when it was re-discovered by Heinrich Rose and Jean Charles Galissard de Marignac, who called it *Niobium*
- "Niobium" was officially adopted as the name for the element 41 in 1950

 It was found to be a superconductor (zero electrical resistance) in the 1920s



Niobium Production



World Niobium Production in 2006 based on the Comodity Report of the United States Geological Survey 2006

Niobium is mostly obtained from mineral known as pyrochlore (NaCaNb₂O₆F). The pyrochlore mineral is processed to give a concentrate ranging from 55 to about 60% niobium oxide (Brazil, CBMM).

Columbite ((Fe, Mn)(Nb,Ta)₂O₆), a mineral with a ratio of Nb₂O₅:Ta₂O₅ ranging from 10:1 to 13:1, occurs in Brazil, Nigeria, and Australia, also other countries in central Africa. Niobium is recovered when the ores are processed for tantalum.







Niobium mines





The world's largest niobium deposits are located in Araxá, Brazil owned by Companhia Brasileira de Metalurgia e Mineração (CBMM). The reserves are enough to supply current world demand for about 500 years, about 460 million tons. The mining of weathered ore, running between 2.5 and 3.0% Nb₂O₅, is carried out by open pit mining without the need for drilling and explosives. Approximately 85 to 90% of the niobium industry obtains its niobium ores.

Page 11



Niobium Processing Plant





CBMM Plant

 CBMM hosted the International Workshop on Single Crystal Niobium Technology in 2006







Nb Production (CBMM)

- Niobium Ore in Araxa mine (open air pit) is pyrochlor with 2.5% Nb₂O₅
- The ore is crushed and magnetite is magnetically separated from the pyrochlor.
- By chemical processes the ore is concentrated in Nb contents (50 –60 % of Nb₂O₅)
- A mixture of Nb₂O₅ and aluminum powder is being reacted to reduce the oxide to Nb
- This Nb is the feedstock for the EBM processes

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Fig. 3: Production flow chart at CBMM.





Page 13

Electron Beam Melting



Page 14

Electron beam melting of Nb



Electron Beam Melting

• As a result of the increasing demand for refractory metals in the last few decades, the electron-beam furnace has been developed to a reliable, efficient apparatus for melting and purification.

• There are several companies, which can produce high purity refractory metals in larger quantities: WahChang (USA), Cabot (USA), W.C.Heraeus (Germany), Tokyo Denkai (Japan), OTIC (China), CBMM (Brasil), H.C. Starck (Germany, USA)

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Page 15

Electron Beam Refining Furnace



Furnace "S10" at ATI Wah Chang

- 2,250 kW
- 3 EB guns
- Crucible diameter: 25-48

cm

Ingot length: 2.9 m







Nb EB Refining



Metal-gas and gas-gas reactions during Nb EB refining

Page 17

• The heating temperature is a compromise between the maximization of purification and minimization of the material losses by evaporation.

RRR=300-500 are reachable currently.





Nb EB Refining

- One problem sometimes observed with e-beam melted ingots is the nonhomogeneous distribution of impurities.
- The **skin** of the ingot has been found to contain more impurities than the inside.
- Top to bottom inhomogeneity has also been observed. The first part of the melt which usually ends up at the bottom getters impurities in the early stages of the melt.
- Machining away the skin and cutting away a short section from the bottom are recommended for a purer final product



Niobium Ingot

Crucibles



Intermediate









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Fabrication of Fine-Grain Nb Sheets





Forging



2000 ton open die forge (Wah Chang)







Rolling



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700 mm wide cold rolling mill (Wah Chang)

800 mm wide hot rolling mill (Wah Chang)

Hot rolling, used mainly to produce sheet metal is when industrial metal is passed or deformed between a set of work rolls and the temperature of the metal is generally above its recrystallization temperature. Cold rolling takes place below recrystallization temperature.



Damage Layer by Rolling



Finite element simulation of 2% reduction of 3.5 mm sheet with 1 cm diameter rolls. Strain is concentrated in the near-surface region



Nb 2% Reduction Rolling Pass



As-received RRR Nb Sheet, 20 μm below surface (ion milled thin foil)

High dislocation density.

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12.0

10.0

Equivalent Plastic Strain (%)

Transmission electron microscopy image (BFTEM)



Page 23

Annealing





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Annealing: recrystallization



Choosing the proper annealing conditions is important to produce the correct grain size near 100% recrystallization and keep the highest possible purity (RRR).

Recovery: removing point defects, decrease and change direction of dislocations

Recrystallization: nucleation of new grains and growing of new crystals **Grain growth:** increase in grain size







Recrystallization





Recrystallization

The grain structure influences the formability





Nb Mechanical Properties at Low T









Nb Sheets: Technical Specifications

Concentration of impurities in wt.ppm				Mechanical properties	
Ta*	≤ 500	H*	≤ 2	Yield strength**, $\sigma_{0,2}$	50<σ _{0,2} <100 N/mm² (Mpa)
W*	≤ 70	N*	≤ 10	Tensile strength**	> 100 N/mm² (Mpa)
Ti*	≤ 50	O *	≤ 10	Elongation at break**	30 %
Fe*	≤ 30	С*	≤ 10	Vickers hardness** HV 10	≤ 60
Mo*	≤ 50	RRR*	≥ 300	Absence of foreign material inclusions*	Proven by scanning
Ni*	≤ 30	Recrystal. degree. Grain size* ,** ?	≈ 50 µm	Texture *, ** ?	

* - relevant for performance

****** - relevant for successful fabrication





Nb Sheets Quality Control

東京電解(株) RRR Tensile test measurement Tokyo Denkay (Japan) 東京電解(株) 東京電解(株) ・窒素分析装置 なる Gas analysis N,の場合 酸化プロセス Sample Flow ガス熱伝導度測定 試料溶融 CO₂除去 He

Gas analysis (Hydrogen, Oxygen, Nitrogen) : HORIBA



Nb Purity: RRR

Electrical resistivity of metals at low temperatures is related to the impurity concentrations. The residual resistivity at T=0K is caused mainly by scattering of electrons by impurities. Residual Resistivity Ratio:

$$RRR = \frac{\rho(295K)}{\rho(4.2K)}$$









RRR Measurement



DC method: extrapolation of U(T) curve







Influence of Impurities on RRR



mechanism:

nonmetallic impurities measured by Tokyo Denkai

O

N

300

$$RRR = \frac{\rho(300K)}{\rho_{ideal} (4.2K) + \sum_{i} \frac{\partial \rho_{i}}{\partial C_{i}} C_{i}}$$

ρ(300K) = 14.6 μΩ cm $RRR_{ideal} \cong 35,000$

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Expected RRR contribution for Nb for 1 wt ppm of impurities

inent	RRR	Element	RRR
Н	2640	Zr	102 000-239 000
N	4230	Hf	200 000
С	4380	W	262 000-721 000
0	5580	Mo	717 000
Ti	53 700	Ta	$1\ 140\ 000$



Nb Thermal Conductivity





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Nb Thermal Conductivity





Texture



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Microstructure of the three grains of Nb after proper etching (optical microscope). The crystal lattice in neighboring grains is the same (bcc) but has a different orientation

Electron back scattering diffraction EBSD

Niobium sheet after annealing. Colors refers to crystal orientations (orientation image)


- High RRR Nb (RRR ~ 300): ~ \$ 530/kg
- Reactor grade Nb (RRR ~ 30): ~ \$ 130/kg







Quality Control of Nb Sheets



DESY eddy current scanning apparatus for niobium discs. 100% Nb sheets for TTF scanned and sorted out. Feedback to manufacturer was very important



Principle of eddy current measurement





Defects in Nb Sheets



Iron particles, probably imbedded during rolling T17

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Iron signal distribution in one of the locations of the Nb sheet T17 measured Synchrotron Radiation Fluorescence Analysis (SURFA) and defects image.





Feedback Example: Final Rolling



Example of the feedback to companies: Tokyo Denkai improved the cleanness around of the rolling equipment







Overview of Cavity Components (ILC)





Dumb-bell Fabrication

- 1. Deep drawing
 - 2. Mechanical measurement
 - 3. Cleaning (by ultra sonic cleaning + rinsing)
 - 4. Trimming of iris region and reshaping of cups if needed
- 5. Cleaning
- 6. RF measurement of cups
- 7. Buffered chemical polishing + Rinsing (for welding of Iris)
- 8. Welding of Iris
- 9. Welding of stiffening rings
- 10. Mechanical measurement of dumb-bells
- 11. Reshaping of dumb-bell if needed
- 12. Cleaning
- 13. RF measurement of dumb-bell
- 14. Trimming of dumb-bells (Equator regions)
- 15. Cleaning
- 16. Intermediate chemical etching (BCP, 20- 40 μm) + Rinsing
- 17. Visual Inspection of the inner surface of the dumb-bell, local grinding if needed + (second chemical treatment + inspection)

Dumb-bell ready for cavity





Dumb- bell



Deep Drawing



- Deep drawing with hydraulic press
 - 100 tons + 25 tons for iris coining for 10.25" discs (ILC)
 - 400 tons + 50 tons for iris coining for 15.5-16.5" discs (SNS)







Deep Drawing







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Shape Measurement



Shape accuracy: optical and mechanical 3D measurement of the half cell shape



Mechanical Grinding



 Mechanical grinding of visible local defects with aluminum oxide grinding discs

Page 46





RF Measurements of Dumb-bells and Middle-Cups

Elongation ALe in the magnetic field region

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Frequency and length deviation of middle cups 2 2 Frequency deviation [MHz] 0 -1 -1 -3 -4 -0.5 -0.4 -0.3 -0.2 -0.1 -0.0 0.1 0.2 03 0.4 0.5 M3 deviation [mm]

Wah Chang (EDMS-DB)



Measure frequency and length to determine how much to trim at the equator to obtain target frequency and length of the cavity fully welded





Page 47

Cavity Parts



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- Welding sequence:
 - Two cups form a dumb bell
 - Stiffening ring at iris
 - Welding together two dumb-bells
 - Add next dumb bell
 - Add end group
 - For mass production: weld all dumb bells at once





Page 48

Cavity Welding Preparation Steps



- 1. Degreasing and rinsing of parts
- 2. Drying under clean condition
- 3. Chemical etching at the welding area (Equator)
- 4. Careful and intensive rinsing with ultra pure water
- 5. Dry under clean conditions
- 6. Install parts to fixture under clean conditions
- 7. Install parts into electron beam (eb) welding chamber
 - (no contamination on the weld area allowed)
- 8. Pump down to vacuum in the EBW chamber E^{-5} mbar
- 9. Welding and cool down of Nb to $T < 150^{\circ}$ C, venting
- 10. Leak check of weld



Electron Beam Welding

- Welding under good vacuum, 10⁻⁵ mbar range
- Broad welding seam
 - Operate with defocussed beam
 - Smooth underbead
- Overlap at end of welding to avoid accumulation of impurities
- Wait to cool down before opening chamber





Electron Beam Welding



Rhombic raster pattern for the beam during EBW. This rastering produces a well-defined and reproducibly defocused beam

Welding Scheme (circular raster) 1-Electron beam (Po-power of the beam, r-spot radius on the surface, L-scanning amplitude, V-velocity of the beam movement)

2-Nb sheet

3-melting zone (z-depth of the melting zone).



2,8 mm

1,7 mm





Microstructure of Nb EBW Joint



Microstructure of the EB welding area. The grain size is 50 $\div 2000 \ \mu m$







EBW Machine (JLab)



Tack- Welding:	4 tacks, focused beam
Voltage :	50 kV
Current:	15 mA
Rotational Speed :	20 inches/min
Distance of gun to work : 6 "	
Final weld Current:	33 mA
Rotational speed:	18"/min
Focussing:	elliptical pattern











EBW Machine (DESY)



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Specification of DESY Electron Beam Welding Machine Voltage: 70 - 150 kV Beam power: max. 15 kW Beam current: 0 bis 100 mA Chamber size: 3300mm x 1400mm x 1600mm (ca. 7,4 m³) Vacuum: > 5x10⁻⁶ mbar (ca. 2x10⁻⁸ mbar) Pumping time: ca. 20 min =3x10⁻⁶ mbar 2 Cryogenic - Pumps: ca. 2 × 10.000 l/s Displacement along the X-Axes ca. 1400 mm



Page 54

Nb RRR After EBW



RRR in the welding seam versus pressure in the welding chamber The RRR degradation at welding seam started since pressure of ca. 10⁻⁵ mbar.





RRR Variation After EBW



RRR in the EB welding area versus distance from the welding seam at different pressures of DESY EBW facility

The RRR degradation can take place in the welding seam itself, but also in the thermally affected area and overlapping region







RRR Variation After EBW



Partial pressure in the EB chamber during welding of Nb300 sample







RRR Variation After EBW



Absorption of hydrogen can take place at the area with moderate temperatures



Comparison of RRR and hydrogen content in welding area (pressure 2.3x10⁻⁸ mbar)





Nb Contamination After EBW

Where the carbon come from?

Diffusion pump

- Dirty EB chamber
- Not sufficient cleaning prior to weld...







Nb Contamination After EBW







Welded Cavity Parts









Welded Cavity Parts





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Page 62

Cavity Inspection



Eccentricity measurement Check of all mechanical tolerances

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- Take care with sealing surfaces
- Inspection of inner cavity surface
- Measure and adjust frequency and electrical field profile

Dimensional check









Cavity Tuning

Set-up for field profile measurements: a metallic needle is perturbing the RF fields while it is pulled through the cavity along its axis; the stored energy in each cell is recorded.









Cavity Tuning

 Small mechanical adjustments to the cavity's cells to obtain flat field profile and desired frequency







Cavity Tuning

- Computerized tuning machine at DESY
 - Equalizing stored energy in each cell

by squeezing or pulling

Straightening of cavity





External Chemistry







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Industrialization of Cavity Fabrication







Low-β **Cavity Fabrication**

The ANL 345 MHz Triple-spoke cavities



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Cavity components prior to EBW (AES)







Low-β **Cavity Fabrication**

- Work out frequency tuning sequence to arrive at final frequency – build into parts initial sizing
- Plastic deformation can be used after manufacture to achieve the final frequency
- Somewhat risky in jacketed cavity since pressure is put on the welds
- Flexible tuning plate can be deformed safely over some limited range without risk to cavity







Cavity Fabrication R&D

- Large-grain/Single-crystal cavities
- Hydroforming
- Spinning







Large-Grain Nb




Large-Grain/Single-Crystal Nb Advantages

Cost effective

- Reduced risk of contamination during sheet fabrication
- Simplified quality control
- Higher thermal conductivity at low temperatures (phonon peak)
- Smooth surfaces, comparable to EP, can be achieved by standard BCP
- Baking at 120°C works to reach high-fields





Large-Grain Nb Sheets







Material Suppliers

- Fine-Grain:
 - Tokyo Denkay (Japan)
 - ATI Wah Chang (USA)
- Large-Grain:
 - Tokyo Denkay (Japan)
 - ATI Wah Chang (USA)
 - OTIC (China)
 - CBMM (Brazil)
 - WC Heraeus (Germany)







Ingot Slicing

Successfully Multi-sliced 59 sheets (3.2t) from 201 mm long Nb Ingot

- Wire EDM (slow)
- Diamond saw
- Multi-wire



Large-grain Nb sheets are available up to 18" diameter







Deep Drawing Large-Grain Nb





- Non-uniformity in the equator area
- Visible steps at grain boundaries
- Thinning at iris region if there is no large single crystal at the center







Large-Grain Nb Cavity Fabrication



- Same procedures as fine-grain Nb
- Assembly for equator welds a bit more complicated as cells are not as round as fine grain cells











Large-Grain Single-Cell Studies





Large-Grain 9-Cell Results





Single-Crystal Nb Cavity Fabrication

Single crystal cavity fabrication (DESY- JLab)







2. Cutting through the disc



3. Increasing of diameter by special rolling with an intermediate annealing



4. Deep drawing

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5. EB welding by matching the crystal orientation



Single Crystal Cavities with three different crystal orientation





Single-Crystal Nb Cavity Results

Cavity #	E _{acc,max} (MV/m)	B _{peak,max} (mT)	Q ₀ (B _{peak,max})	Treatment
1	38	162	4×10 ⁹	200μm BCP, 800°C 3h, HPR, 120°C 48h
2	45	160	7×10 ⁹	200μm BCP, 800°C 3h, HPR, 120°C 24h
3 (1AC6)	41	177	1.2×10 ¹⁰	250μm BCP, 750°C 2h, 120μm EP, HPR, 135°C 12h
4 (1AC8)	38.9	168	1.8×10 ¹⁰	216µm BCP, 600°C 10h, HPR, 120°C 12h
5	38.5	166	7.6×10 ⁹	170μm BCP, HPR, 120°C 12h



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Single-Crystal Nb Cavities





Seamless Cavities









Hydroforming



- Tube of intermediate diameter between iris and equator
- Necking to form iris area
- Hydroforming (hydraulic expansion)







Tube Forming: Fine-Grain Nb



Pot with thick wall by spinning (or deep drawing)



Flow forming

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Flow forming over a cylindrical mandrel with three work rollers allows to produce long and very precise tubes from thick walled cylindrical part.

After optimization of several parameters shiny Nb surface and small wall thickness variations (less then +/-0,1 mm) have been achieved.



Tube Forming: Fine-Grain Nb



 Small and uniform grains • Elongation at break > 30%





Tube Forming: Single-Crystal Nb



Seamless single-crystal tube by back extrusion



- Welded single-crystal tube:
 - Rolling of single-crystal with intermediate annealing
 - EBW with matching of the orientation







Necking



Principle of diameter reduction in the tube end and in the tube middle







Hydroforming



Relies on the correct relationship between applied internal pressure and axial displacement (strain rate) to remain below the plastic limit









Hydroforming





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DESY hydroforming machine





Page 91

Hydroforming: Cavity Results





Spinning

- Starting from discs or seamless tubes
- Collapsible mandrels
- Very rough surface after fabrication







Spinning









Spinning: Cavity Test Results





1.3 GHz 9-cell cavity. Damaged after CBP, no test







Spinning: Cheap Sample Cavities









Nb/Cu Clad Material

Advantages

- cost effective: allows saving a lot of Nb (ca. 4 mm cavity wall has only ca. 1 mm of Nb and 3 mm Cu). Especially significant for large projects like ILC
- bulk Nb microstructure and properties (the competing sputtering technique does not have such advantages)
- the treatment of the bulk Nb BCP, EP, annealing at 800°C, bake out at 150°C, HPR, HPP can be applied (excluding only post purification at 1400°C).
- high thermal conductivity of Cu helps for thermal stabilization
- stiffening against Lorentz force detuning and microphonics can be easily done by increasing of the thickness of Cu layer.
- fabrication by seamless technique allows elimination of the critical for the performance welds especially on equator



W. Singer SRF 2005





Nb/Cu Clad Cavity Fabrication

- Nb/Cu laminated material is formed into a tube by:
 - Explosion bonding
 - Back extrusion
 - Hot bonding
- Nb/Cu clad cavity if formed from the tube by hydroforming
- Beam tubes/End groups are welded to the thin Nb layer by EBW. The Cu backing must be removed and cleaned at the weld joints







Nb/Cu Tubes: Explosion Bonding

- Explosion bonding of seamless Nb tube 4 mm thick with Cu tube 12 mm thick
- Flow forming into Nb/Cu tube, wall thickness 1 mm Nb, 3 mm Cu



The bonding takes place by an explosively driven, high-velocity angular impact of two metal surfaces.

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Explosively bonded NbCu tube





Structure of Nb/Cu interface



Nb/Cu Clad Cavities



NbCu single cell cavity 1NC2 produced at DESY by hydroforming from explosively bonded tube. Preparation and HF tests at Jeff. Lab: 180 µm BCP, annealing at 800°C, baking at 140°C for 30 hours, HPR (P. Kneisel).

40 MV/m without EP

W. Singer SRF 2005



NbCu cavities hydroformed from explosively bonded tubes at DESY.

Difficult to get reproducibly high bonding quality. Hot bonding fabrication procedure of NbCu tubes seems to be more promising







Nb/Cu Tubes: Hot Bonding



Fabrication principle of sandwiched hot rolled Cu-Nb-Cu tube (KEK and Nippon Steel Co.)

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Hot roll bonded Cu-Nb-Cu tube produced at Nippon Steel Co.





Nb/Cu Clad Cavities



Single-cell Nb/Cu cavities from "sandwiched" tube



Four double cell NbCu clad cavities produced at DESY from KEK tubes (no cracks on the inside surface) Hot roll bonded tube fabrication at Nippon Steel Co., hydroforming at DESY, Preparation and RF tests at KEK



NSC-3: Barrel polishing, CP(10 mµ), Annealing 750°C x 3h, EP(70 µm) K.Saito





Nb/Cu Clad Cavities Issues

- Possibility of leaky welds because of Cu contamination
- Nb/Cu cavities still quench, resulting in Q-degradations
- Cooldown needs to be very uniform because of thermocurrents
- Cooldown of cryomodules would need modification
- Cracks sometimes appear in iris region during fabrication





