SC Cavities; Material, Fabrication and QA

W. Singer, DESY

- Material for SC cavities
- Conventional Fabrication (deep drawing, EB welding)
- QA (quality assurance)
- Fabrication R&D (seamless, large grain LG and single crystal SC)



Material

Nb is a very valuable material



(Nb jewelry)

Nb is mostly used for SC cavities.

- high transition temperature Tc= 9,3 K
- high critical magnetic field Hc=200 mT
- chemically inert (surface covered by oxide layer)
- can be machined and deep drawn
- is available as bulk and sheet material in any size



Niobium is mostly obtained from mineral known as pyrochlore (NaCaNb₂O₆F). The pyrochlore mineral is processed by primarily physical processing technology 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.



The world's largest niobium deposit is 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.











CBMM Plant

Niobium Production at 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



Fig. 3: Production flow chart at CBMM.

Electron Beam Melting



During of the ingot melts, molten metal globules fall into a pool on the ingot which is contained in a water cooled copper cylinder (sleeve). Impurities are evaporated and pumped away. Power impact is maintained to keep the pool molten out to within a few mm of the crucible wall. During melting the ingot formed is continuously withdrawn through the sleeve. The rate of withdrawal has to be carefully coordinated with the rate of the material to insure complete melting of the feed material and proper outgassing.

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.





Fa. ALD (Germany) and VON ARDENNE (Germany) produces EB furnace for melting of refractory metals

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 cutting away a short section from the bottom are recommended for a purer final product



The melting temperature is a compromise between the maximization of purification and minimization of the material losses by evaporation. RRR=300-500 are reachable currently

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







Crucibles

Finished ingot (Wah Chang). Diameter: 250-480 mm





Handling of EB melted ingots at CBMM (Brazil)





Nb-Purification by Electrolysis in Molten Salts



Fundamental investigations of the electrodeposition of Nbfrom LiF-NaF-KF melts containing K₂NbF₇, at T = 1000 K have shown that this electrolyte was not only suited for the production of coatings, but also that the material refined in this way;

Nb become extremely pure especially concerning of Ta

Efficiency of Nb-Purification by Electrolysis in Molten Salts.

Sheet : Technical Specification for High Purity Niobium

Concentration of impurities in wt.ppm				Mechanical properties	
Ta*	≤ 500	H*	≤ 2	Yield strength**, σ _{0,2}	50<σ _{0,2} <100 N/mm ² (Mpa)
W *	≤ 70	N *	≤10	Tensile strength**	> 100 N/mm ² (Mpa)
Ti*	≤ 50	O *	≤10	Elongation at break**	30 %
Fe*	≤ 30	C *	≤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

Fabrication of Nb sheets at Tokyo Denkai



Forging



2000 ton open die forge (Wah Chang)

Rolling



700 mm wide cold 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.

Annealing

Temperature °C



The temperature and the total pressure in the chamber during annealing of Nb300.

Outgassing of hydrogen



Hydrogen partial pressure versus annealing temperature During the high-temperature annealing of niobium in vacuum, hydrogen is picked up at the surface and the rate determining process is the transport in the gas atmosphere or the diffusion in the bulk, depending on the partial pressure and the diffusion rate. It must also be taken into account that considerable quantities of H may be picked up during cooling down.

The rate of H absorption depends on the relative number of O_2 or H_20 and H_2 molecules touching the surface per time unit.

How the outgassing of hydrogen take place?

Nb hydrides decomposes mostly at T> 100-150°C. Outgassing of hydrogen is however not possible at these temperatures because the Nb₂O₅ protecting layer is stable up to 250-300°C. The most efficient annealing of high purity niobium is 750-800°C, 2 hours.

- Complete outgassing of hydrogen
- Recrystallization of Nb. That means lattice has less defects and become less vulnerable to a new hydrogen contamination



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 during annealing the point defects, decrease and change of orientation of dislocations

Recrystallization: - nucleation of new grains and growing of new crystals Grain growth: - grain size increases



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The grain structure influences the formability



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Low temperature behavior of Nb



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Material Purity: Electrical resistivity of metals at low temperatures is an excellent tool to determine impurity concentrations. The residual resistivity at T =0K is caused mainly by scattering of electrons on impurities.



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DC (direct current) -method: extrapolation of U(T) curve

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Influence of impurities on RRR



Contribution of different defects in the scattering mechanism (Schulze) $RRR = \frac{R(300K)}{R(10K) + \sum_{i=1}^{4} \frac{\partial R_i}{\partial C_i} C_i}$ $R(300K)=1,46.10-5 \ \Omega \ \text{cm}, R(10K)=8,7.10-9 \ \Omega \ \text{cm}, C=1$ wt. ppm

Relationship between RRR and nonmetallic impurities measured by Tokyo Denkai

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Thermal conductivity of high purity Nb at low temperatures

Normal conducting cluster triggers the quench, if the temperature exceeds Tc

Rule of thumb

$$\lambda(4,2K) = C \cdot (\frac{W}{m \cdot K}) \cdot RRR$$
$$C \approx 0,25 \div 0,14$$
$$\lambda_{T} \approx RRR \approx purity$$



Thermal conductivity of single crystal and polycrystalline niobium.

Phonon peak is clearly pronounced for single crystals.

$$\lambda(T, RRR,G) = R(y) \cdot \left[\frac{\rho_{295K}}{L \cdot RRR \cdot T} + a \cdot T^2\right]^{-1} + \left[\frac{1}{D \cdot \exp(y) \cdot T^2} + \frac{1}{B \cdot G \cdot T^3}\right]^{-1}$$



bcc lattice (body centered cubic)





The anisotropic properties of a material is the average of anisotropic properties of its crystallites



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

TEXTURE BY EBSD ELECTRON BACK-SCATTERING DIFFRACTION



Electron back scattering diffraction EBSD

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

Local defects



Iron particles, probably imbedded during rolling T17



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



Search for clusters in Nb sheets. Eddy current system.





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

Principle of eddy current measurement



Frequency 1.3 GHz

High purity niobium RRR 300

Deep drawn from sheets

Welding with electron beam

Operating temperature 2K

Experiences of ca. 20 years of industrial cavity fabrication by deep drawing and EB welding are available



Half cells are produced by deep drawing.

Dumb bells are formed by electron beam welding.







After proper cleaning eight dumb bells and two end group sections welded by electron beam together

Important: clean conditions on all steps shape accuracy, preparation and EB welding





Shape accuracy: Optical and mechanical 3D measurement of the half cell shape
Frequency and length adjustment





Equipment for RF measurement on half cells and dumb bells HAZEMEMA is developed at DESY for XFEL fabrication (SRF2009, Poster THPP0071)

Cavity welding: the general way There are differences of welding processes in industry 1 piece 1 pieces 8 pieces Degreasing and rinsing of parts Drying under clean condition 2. Chemical etching at the welding area (Equator) 3.

- 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 EBW



Microstructure of the EB welding area. The grain size $G{=}50 \div 2000 \ \mu m$

Electron Beam Welding Machine





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



Focusing possibilities (above focus; below focus)



Welding Scheme (circular raster) 1-Electorn beam (P₀-power of the beam, rspot 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).

RRR degradation

TE 2.3E-8 mbar 2E 2.0E-6 mbar 3E 2.3E-7 mbar 4E 6.5E-8 mbar - Reference 5E 1x10E-5 mbar

320

310

300

Position. mm

-5

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

-20

-15

-10



5



Oxygen distribution along the welding seam. RRR= 280 in the welding seam and RRR= 207 in the overlapping.

15

20

10

Electron beam welding of niobium



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.

Does the refining or pollution take place during EB welding? Pressure – concentration isotherms of oxygen solubility in Nb in steady state condition



Kinetic of process is of grate importance

$$\Delta C \approx \frac{1}{2} \cdot p \cdot \frac{t}{d}$$

For concentration change ΔC of 4 wt. ppm, sample thickness d=2 mm, equilibrium pressure p=10⁻⁶ mbar the degassing time is t=800 sec.

Partial pressure in the EB chamber during welding of Nb300 sample



Temporal changes of gas partial pressure in vacuum chamber during the EB welding process.

Correlation of hydrogen content and RRR at ultra high vacuum



Pressure – concentration isotherms of hydrogen in Nb in steady state condition

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)

Clean conditions are essential. Example: C (carbon) on the inside cavity surface even after EP

Where the carbon come from?

- Diffusion pump
- Dirt EB chamber (oiled parts have been welded before cavity welding)
- Not sufficient rinsing after machining (oil layer on the surface of the acid remains on the part surface)
 ???



10

kev Germany



Skalenbereich 995 cts Cursor: -0.172 (0 cts)



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Quality Assurance – Main Strategy

- The Manufacturer has to create and maintain a Quality Management System according EN ISO 9000 and
- According to requested by Orderer quality checks incl. documentation
 - The most of these checks are to be done by the contractor anyway but documentation must be transferred to Orderer "just in time" (QA action)
 - Special attention
 - Cell area of the Cavity
 - Welding connections

Quality Assurance – Manufacturers Qualification

 The manufacturer and all its subcontractors have to demonstrate with their certification according ISO 9001:

the ability consistently provide product that meets Orderer requirements and applicable statutory and regulatory requirements, and

 aims to enhance Orderer satisfaction through the continual improvement of the system and the assurance of conformity to customer requirements and applicable statutory and regulatory requirements.

Quality Assurance – Documentation

The fixing of the Nomenclature of all existing objects will be the basis for a structured documentation

- The Nomenclature is to be used for the whole contract by Manufacturer and Orderer
- Abbreviations are to be used to generate serial numbers.

Examples:

- □ Serializing of Nb-sheets
 - The serial number is made up of
 - "code letter" of Nb supplier
 - individual 5-digit number
 - Example: P00001 (Nb-sheet number one from Fa. Plansee)
- Serializing of dumb-bells
 - The serial number of a dumb-bell is made up of
- the abbreviation for dumb-bell (DB),
- an underscore <u>"</u> and the SN of both used half-cells,
- Example: DB_H00123_H32100

Traceability

- For each completed cavity, a traceability report is to be prepared, in which the serial numbers of all incorporated semi-finished products and manufacturing groups are stated in the form of a parts list, including position information.
- It must be possible to reconstruct the development history of all products
- A conclusion concerning the raw material used should be possible.

Quality Assurance – QC lists

- Overview list
 - Lists all documents (certificates, test reports, ...) with information of
 - Prescribed or recommended
 - hand over information
 - Storage information
- Detailed inspection list
 - Lists detailed the contents of the inspection sheets
- Orderer Inspection sheets
 - data of inspection sheets should be transferred to Orderer data base using Ordered templates

×

Orderer inspection sheets



Eccentricity measurement





Optical control of the inside surface

Quality Assurance – special document

- Conformity Report (Confirmation of Conformity)
 - The manufacturer will prepare a conformity certificate for each completed cavity
 - It confirms that the cavity has been produced according to specification, and that all requirements have been checked and complied with the orderer.
 - Deviations are to be mentioned.

Fabrication R&D Large grain LG

Possible advantages (hope):

- Cost effective
- Higher purity. RRR=600 of ingot is achievable



- No danger that during many steps from ingot to sheet the material will be polluted.
- Simplified quality control (reduced number of measurements: grain size, eddy current scanning etc.)
- Higher thermal conductivity at low temperatures (phonon peak)
- Seems to be less susceptible to field emission (Univ. Wuppertal)
- Seems that the baking at 120°C works better after BCP (compare to fine grain BCP)

Discs Slicing:

1. Before all else the multi- slicing of discs was developed at W.C. HERAEUS using diamond saw (B.Spaniol, LINAC 2006, TUP024)

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



Japanese Industry (more details in presentation of K.Saito, SRF2009)

Multi cell fabrication: For example 11 LG 9-cell cavities at RI from HERAEUS material fabricated DESY (AC112-AC114, AC151-AC158)

Fabrication:

- Discs scanned only for two cavities.
- Deep drawing
- Machining
- EB welding

 Grinding of grain boundaries on 4 cavities for comparison





In discs for AC151-AC154, the main orientation of the central crystal is (100), for discs of AC155-AC158 mainly (211) or (221). For the disc with (100) orientation, a more pronounced anisotropy and quadrangular shape after deep drawing was observed. The difficulties for assembling of half cells and dumb-bells for welding have been overcome by using a special tool

Reasonable performance and reproducibility have been demonstrated on single cell LG cavities with BCP by JLAB, DESY, and KEK/IHEP



Reproducibility Tests of LG Niobium Cavities (P. Kneisel, JLab)

40

Encouraging results on multi cell LG cavities after BCP:

Q(Eacc) curve of the LG nine cell cavities AC112- AC114 at 2K after 100 µm BCP, 800°C, 20 µm BCP, HPR





Second test Q(Eacc) curve of the LG nine cell cavities AC113-AC114 at 2K after additional 20-30 µm BCP and 125°C, 50 h baking



It seems that EP works better

Q(Eacc) curve of the single cell cavity 1AC4 after EP and BCP treatment



EP on 3 DESY LG Cavities:

Additional EP of 50-100 µm, combined with additional "in situ" baking. Enhancing the acceleration gradient by approx. 10 MV/m can be seen on two cavities (AC112 and AC113). Surprisingly, significant degradation of Eacc from 28 to 14 MV/m for the cavity AC114

Multi cell LG activities: Several Labs are working on LG multi cell cavities



JLab: Two Jlab upgrade 7-cell cavities are completed from Ningxia/ CBMM large grain Nb Jlab:Two LL/Ichiro 9-cell ILC cavities are in fabrication from Tokyo-Denkai and CBMM material



Pekin Univ: LG two cell cavity

IHEP works on two 9-cell LG 1,3 GHz CVs



JLab: 2-Cell large Grain CRAB Cavities



AFM roughness measurement (X. Singer, A. Dangwal-Pandey). Roughness of fine grain Nb after EP is 251 nm (A. Wu).

Single Crystal Option

Better not to have the grain boundaries at all Fabrication of TESLA shape single crystal single cell cavities was proposed at DESY.

Following aspects have been investigated and taken into consideration during cavity fabrication

- Definite enlargement of the discs diameter is possible without destroying the single crystal structure in an existing state.
- Appropriate heat treatment will not destroy the deformed single crystal
- The single crystals keep the crystallographic structure and the orientations after deep drawing and annealing at 800°C

• Two single crystals will grow together by EB welding, if the crystal orientations is taken into account.



Single crystal cavity fabrication (DESY-JLab)





2. Cutting through the disc



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

1. Take out central single crystal of definite thickness

4. Deep

drawing



5. EB welding by matching

the crystal orientation



Single Crystal Cavities with three different crystal orientation

One large grain (single crystal) is a promising option that allows stably reach very high gradient by simple BCP treatment

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

Is it realistic produce single crystal cavities of sizes required for ILC?



3. Interface between solid and liquid phase is shifted by movement of container or temperature gradient

2.

Electron beam melting principle Challenge for the industry
Fabrication R&D: Seamless fabrication



Example of welding connection of EP treated cavity. Up to 75 µm steps

Fabrication R&D: Seamless fabrication

Hydroforming, DESY, KEK, MSU











DESY hydroforming machine HYDROFORMA

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Spinning (V.Palmieri, INFN Legnaro)

Hydroforming, Spinning

Proof of principle is done on single cell cavities



Fabrication of 9 cell cavities became a reality

INFN Spun cavity

NEN

DESY Hydroformed cavity completed from 3x3 cell units

DESY cavity Z145: 9-cell cavity RF test results



Surface treatment at DESY: 40 mm BCP, 800 °C heat treatment, tuning; 170 mm electropolishing (EP), ethanol rinsing, 800 °C heat treatment; 48 mm EP, HPR, assembly and evacuation. After "in-situ" baking at 120 °C for 48 h. the Q-drop almost disappeared, but the Eacc was not increased. The cavity is welded into the He vessel, will be installed in one of next acc. cryomodules at DESY.

Mode limits of cavity Z145 measured on 23-Jul-2008 13:0	a 23-Jul-2008 13:08
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	Pi	8/9 Pi	7/9 Pi	6/9 Pi	5/9 Pi	4/9 Pi	3/9 Pi	2/9 Pi	1/9 Pi	Max Field	Limit.
Cell 1&9	30.29	27.34	29.67	28.39	26.26	25.39	17.62	8.21	5.95	30.29	bd
Cell 2&8	30.29	24.05	15.79	.00	17.14	34.21	35.24	20.79	17.15	35.24	bd
Cell 3&7	30.29	17.85	5.48	28.39	32.21	13.51	17.62	23.64	26.27	32.21	pwr
Cell 4&6	30.29	9.50	24.19	28.39	5.95	38.91	17.62	15.43	32.22	38.91	pwr
Cell 5	30.29	.00	31.57	.00	34.28	.00	35.24	.00	34.29	35.24	bd
Limit	bd	bd	bd	bd	pwr	pwr	bd	pwr	bd		

DESY, KEK, FNAL, MSU, Black laboratories et al continue, or started, or planning to start seamless activities



New DESY multi cell units produced by hydroforming

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