Physics and Chemistry of Niobium Materials in the Context of Superconducting RF-Cavity Applications

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Superconducting RF cavity in high energy particle accelerators

Superconducting RF cavities excel in applications where one needs 'continuous wave or long-pulse' acceleration with gradients above a few million volts per meter (MV m⁻¹)

- X-FEL at DESY Hamburg uses SCRF cavities in its elctron Linac
- SNS, Oakridge USA uses SCRF cavities in its proton LINAC
- European Spallation Neutron Source will use SCRF cavities
- Future International Linear Collider will use SCRF technology
- Planned Indian Spallation Neutron Source will use SCRF technology

Radio Frequency (RF) Cavity

-Beam tube-



Symmetry axis Electric field Iris Magnetic field Hpk Equator

 \bigcirc

Cell

Beam tube-

Magnetic field

Epk

'An RF power source' fills the RF cavity via a 'coupler'.

EM field will impart energy to the charge particles & accelerate them.

What do we want from a good RF cavity ?

High Quality Factor: **Q** = (Stored energy)/(Dissipated power) High Accelerating Gradient : **E**_{Acc}

Dissipated power:
$$P_d = \frac{R_s}{2} \int |\vec{H}|^2 dS$$

 $R_{s \ normal} = \sqrt{\frac{\omega\mu_o}{2\sigma}} \longrightarrow \text{For copper @ 300 K &1.3 GHz } \frac{R_{s \ Copper}}{R_{BCS}} \approx \lambda_L^3 \omega^2 \ell \exp(-1.76 T_c/T) . \longrightarrow \text{For bulk Nb at 2K } \frac{R_{BCS}}{R_{BCS}} \sim 10 \text{ n}\Omega$

Two fundamental limits for Niobium SCRF cavities

1. A critical RF magnetic field above which the perfect superconducting state is destroyed - limits the Accelerating Field or Gradient.

2. Superconducting surface resistance R_s, inversely proportional to Q - limits Quality Factor Q



• For good quality Nb material $H_{c1} \simeq 1.8-1.9$ kOe at 2K.

• This will correspond to a gradient of \sim 50 MV/m, For a 1.3 GHz elliptical cavity operating at 2K,

Materials and surface problems in Niobium SCRF cavities

Extrinsic effects

- Surface roughness, grain boundaries \rightarrow Reduce Q and E_{ACC}
- Impurities → Degrades superconductivity (??)
- Surface Oxides → Suspected to degrade Superconducting response??
- Field emission and multipacting \rightarrow Quenching of the Cavity.



Most of these problems are solved with proper cavity shape, and chemical treatment and cleaning of cavity surface. **Field emission free 1.3 GHz elliptical cavities reaching up to 35-40 MV m⁻¹ are obtained regularly in various labs**.

Is 35-40 MV m⁻¹ the upper limit of achievable gradient ? Answer is No!

Puzzles and Open Issues in Niobium SCRF cavities

Puzzles:

- All cavities fabricated in the same way do not give high gradients.
- Cavity gradient seldom reaches above 45 MV/m.
- Recent report of a 9 Cell Tesla type cavity reaching 45 MV/m

Open Issues:

- (1) What is the RF critical magnetic field in Niobium? Is it
- Thermodynamical critical field-H_c or field for first flux line penetration-H_P?
- Does it depend on the processing of the SCRF cavities?
- (2) Why does the RF surface resistance of niobium increase sharply at high RF magnetic field?
- -- High-field slope in the quality factor-Q-slope



Existing method for Niobium Material qualification

- Current method mainly relies on improving the residual resistivity ratio (RRR) of the Nb.
- It is based on the belief that impurity elements degrade superconducting properties of Nb. High RRR (>300) seemingly signifies high purity level of Nb !!
- High RRR Nb + right cavity shape + chemical treatment
 ⇒ Low extrinsic (+ surface) defects leading to High Gradient and Q.
- Niobium refinement process is very expensive, especially in reducing Ta impurity level from >1000 ppm to <500 ppm.

Residual Resistivity Ratio (RRR) of SCRF Niobium material



 $\mathsf{RRR} = \rho_{300 \,\mathrm{K}} \, / \, \rho_{5\mathrm{K}} \, \sim 300$

Residual Resistivity Ratio (RRR)



 $\mathsf{RRR} = \mathsf{R}_{300\mathsf{K}}/\mathsf{R}_{10\mathsf{K}}$

- RRR gives an idea of the defects in a metal
- Defects in a metal do not mean impurity elements alone, but also encompass point defect like vacancy, line defect (dislocations), grain boundaries etc.
- High RRR, however, does not necessarily say how good (or bad) are the <u>superconducting properties of a</u> <u>material</u>
- Gives indirect information on thermal conductivity of the normal state via Wiedemann-Franz law
- Thermal conductivity in the superconducting state is non-trivial

Residual Resistivity Ratio (RRR)

Resistivity of Metals

The resistivity ρ is defined by scattering events due to the imperfections and thermal vibrations.

Total resistivity ρ_{tot} can be described by the **Matthiessen rule:**

 $\rho_{\text{total}} = \rho_{\text{thermal}} + \rho_{\text{impurity}} + \rho_{\text{deformation,}}$

Where,

- $\rho_{thermal}$ from thermal vibrations,
- $\rho_{impurity}$ from impurities,
- $\rho_{deformation}$ from deformation-induced defects



RRR = ρ_{total} / ρ_0 where, $\rho_0 = \rho_{impurity} + \rho_{deformation}$

A pure but deformed metal will have relatively low RRR

Residual Resistivity Ratio (RRR)



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Superconducting TESLA cavities

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Deppe,4 B. Dwersteg,4 D. A. Edwards,4,5 H. T. Edwards,4,5 M. Ferrario,6 M. Fouaidy,9 P.-D. Gall,4 A.
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.....A good thermal conductivity is the main motivation for using high purity niobium with RRR ~ 300 as the material for cavity production.....

Thermal design studies in superconducting rf cavities: Phonon peak and Kapitza conductance

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RRR of Nb material in the formed SCRF cavity will be significantly different from the RRR of starting Nb-sheet metal. So will be the thermal conductivity !



Residual Resistance in a Nb SCRF Cavity

- Improvements in the surface preparation of bulk Nb SCRF cavities over the years have reduced the typical residual resistance (R_{res}) value from 100 nΩ to 1-10 nΩ.
- R_{res} becomes the dominant term in the surface resistance at low frequency (<750 MHz) and low temperatures (< ~2 K), where R_{BCS} becomes exponentially small.
- There are several possibilities contributing to the R_{res}.
 - (1) losses due to trapped magnetic field,
 - (2) losses due to normal-conducting precipitates near the surface,
 - (3) grain boundary losses,
 - (4) metal/oxide interface losses, and
 - (5) losses due to normal-conducting electrons in subgap states.
- RRR measurement of starting Nb material will not necessarily give the information regarding these issues!

G Ciovati, CondMat ArXiv:1501.07398v1, 2015

SCRF Materials R&D: Approach of a Condensed Matter Physicist

Superconducting transition temperature, Superconducting Critical Fields, Surface Resistance and thermal diffusivity are the most important parameters for SCRF materials

Question we are asking:

What is the tolerable level of elemental impurities in sustaining the superconducting and other relevant materials properties of Niobium, which are required for obtaining best performance in a Superconducting Radio Frequency (SCRF) cavity ?

Characterization of Nb materials for SCRF cavity applications should be done in terms of

- **1.** Superconducting transition temperature,
- 2. Lower critical magnetic field
- 3. Superconducting surface resistance
- 4. Thermal conductivity and specific heat in the superconducting state : thermal diffusivity

Critical Fields in a Superconductor



- External magnetic field is totally expelled below a lower critical field limit H_{C1}.
- In a type-I superconductor above H_{c1} normal state is reached.
- In a type-II superconductor magnetic field penetrates the materials above H_{C1} in the form of quantized flux lines; the material remains superconductor until a upper critical field H_{C2}
- H_{C1} <H< H_{C2} \rightarrow Abrikosov lattice or Vortex state \rightarrow important for high critical current (J_c) applications e.g. SC magnets.
- H < H_{C1} \rightarrow Meissner state.
- H_{C1} determines the limit of gradient in a SCRF cavity

Effect of Ta and Fe impurities in Nb materials

Determined average concentrations of impurities in Nb samples with Indus-2 synchrotron source

Sample	Ta-Content	Fe-Content	Cu-Content	Zn-Content
	(ppm)	(ppm)	(ppm)	(ppm)
Technical-Niobium-1P	1339 ± 36	330 ± 50	5 ± 3	51 ± 15
Technical-Niobium-2P	802 ± 80	290 ± 60	19±7	43 ± 10
Technical-Niobium-3P	243 ± 10	260 ± 60	12±8	13 ± 7
Technical-Niobium-1CT	1285 ± 35	40 ± 15	8±5	14±4
Technical-Niobium-2CT	684 ± 54	43 ± 10	14±7	32 ± 8
Technical-Niobium-3CT	$149{\pm}11$	18 ± 5	8±5	$14{\pm}4$

Supercon. Sci. Tech. 25 115020 (2012)

Effect of Ta Impurities on the SC Properties of Nb

Study of Magnetization versus Temperature of superconducting Nb samples. Allows accurate determination superconducting transition temperature (T_c) critical fields.





- No significant Variation of T_C as a function of Ta impurity contents
- In fact 1-2% Ta in Nb increases the T_C (literature report)
- \bullet Perceptible change in $T_{\rm C}$ in the chemically polished sample

Supercon. Sci. Tech. 25 115020 (2012)

Effect of Ta Impurities on the SC Properties of Nb

- Isothermal field dependence of Magnetization in Nb samples.
- Allows accurate determination superconducting critical fields (H_{C1}, H_{C2})





- Higher Ta impurity only marginally affects H_{C1} and H_{C2}
- Note there is a significant effect of chemical polishing on both the samples.

$$H_{peak}/E_{acc} = 42.6 \text{ Oe}/(MVm^{-1})$$

Supercon. Sci. Tech. Vol. 25 115020 (2012)

Some more comments on Ta impurities in Nb

What happens if Ta impurities forms cluster of micron size and reside as inclusions on the surface of SCRF cavity? Thus create hot spots?

• Ta being chemically very similar to Nb, leads to the difficulty of separating it, and Ta and Nb readily forms solid solution. So statistical probability of forming Ta cluster is low, and the probability of such Ta clusters residing on the surface is even lower.

• If such Ta clusters actually form, then they are expected to be chemically quite pure and free from structural defects. Thus at the operating temperature of SCRF cavities the electrical resistivity is expected to be quite small.

• Ta is actually a superconductor of Tc 4.3K. Thus for a Nb SCRF cavity operating at 2K, normally the Ta clusters are not supposed to become hot spots.

• Even if at 2K, at high RF fields such Ta clusters tend to become normal, being surrounded by superconducting Nb they are still likely to remain as superconductors through proximity effect !

Surface Resistance in a Superconductor

Quality factor of a SCRF cavity is inversely proportional to surface resistance

Response of a superconductor in ac field is described by two fluid model:

- Cooper pairs form superfluid.
- Unpaired electrons form normal fluid \rightarrow source of power dissipation in ac field.



Influence of impurity on Surface Resistance

In a real material like Nb $\lambda = \lambda_L \sqrt{(\xi_0/\xi)}$, where, ξ_0 and ξ are coherence lengths in the pure and real material respectively,

and $\xi^{-1} = \xi_0^{-1} + l^{-1}$

$$R_{BCS} \approx \left(\lambda_L \sqrt{1 + \frac{\xi}{l}}\right)^3 w^2 l e^{-1.76 \frac{T_C}{T}}$$

For $l >> \xi \rightarrow R_{BCS}^{Clean} \propto l$

For $l < \xi \rightarrow R_{BCS}^{Dirty} \propto l^{-1/2}$



BCS Surface rsistance vs e mean free path

Magneto thermal conductivity of SCRF cavity Nb-materials

- Thermal conductivity as function of T and H is being studied for the first time for Nb with intermediate impurity level
- Interesting field dependence near the lower critical field



Chemistry of SCRF cavity Nb-materials

- Defects in the inner surface of the cavity: dissolved foreign, abrasive particles from grinding, imprints from the deep drawing process, niobium protrusions from scratches or dirt particles sticking to the surface.
- Welding joints are source of mechanical defects.
- Cleaning the niobium with chemical methods is the most practical way to achieve a high-quality superconducting surface.
- Niobium metal has a natural Nb₂O₅ layer with a thickness of about 5 nm: Below this layer other oxides and sub-oxides can be found.
- While Nb_2O_5 is an insulator, NbO is a conductor (Tc = 1.6 K). $Nb_2O_{5-\delta}$ shows interesting magnetic properties.
- Nb₂O₅ is chemically rather inert in general, but can be dissolved with hydrofluoric acid (HF).

Buffered chemical polishing of Nb-materials



$$6 \text{ Nb} + 10 \text{ HNO}_3 \rightarrow 3 \text{ Nb}_2\text{O}_5 + 10 \text{ NO}^{\uparrow} + 5 \text{ H}_2\text{O}_5$$

 $Nb_2O_5 + 10 HF \Leftrightarrow 2 NbF_5 + 5 H_2O$

C Z Antonie: CondMat Archive

- This consists of two basic steps: (1) dissolution of the Nb₂O₅ layer by HF, (2) re-oxidation of the niobium by a strongly oxidizing acid HNO₃. Then, the new oxide layer will be dissolved by the HF again.
- The reactions are strongly exothermic; additionally, large quantities of hydrogen gases are produced.
- To obtain a better process control a buffer substance like phosphoric acid H_3PO_4 (concentration of 85%) is added
- Typically, BCP solution, the 1:1:1 or 1:1:2 (volume) mixture of HNO_3 (69%), HF (49%) and H_3PO_4 (85%).
- All crystalline defects are preferentially attacked; grains with various orientations are not etched at the same rate, which induces roughness!

Electro polishing of Nb-materials





Fig. 4. Niobium surfaces after etching (a) and EP (b). SEM micro-graphs are courtesy of G. Arnau, CERN.

L. Lilje et al. NIM. A 516 (2004) 213–227

- The material is removed in an acid mixture under the flow of an electric current.
- The most widely used electrolyte is a mixture of concentrated HF and concentrated H₂SO₄ in volume ratio of 1:9
- Sharp edges are smoothed out and a very glossy surface can be obtained.
- The electric field is high at protrusions so these will be dissolved first. On the other hand, the field is low in the grain boundaries and little material will be removed here.
- The produced gases (mainly hydrogen) are rapidly removed from the wetted niobium surface

Hydrogen and oxygen in SCRF-Cavity Nb materials

- The addition of interstitial solute atoms (below the solubility limit) in Nb material degrades the superconducting properties.
- with higher hydrogen concentration interstitial H in Nb forms different stoichiometric and non-stoichiometric hydride phases during cool-down at low temperatures. Such hydrides may cause the degradation of the surface resistance and hence the quality factor Q - Q disease.
- The defects in Nb in the form of vacancies, dislocation networks and impurity atoms may trap some fraction of interstitial H, thus lowering the number of H atoms available for hydride precipitation. This will lead to smaller hydride sizes translating into higher field onset of strong cavity losses. Hence, a relatively impure Nb material may be really good for SCRF cavity performance!





Some Results on the possible effects of hydrogen and oxygen on the superconducting properties of Nb materials

Effect of Buffered Chemical Polishing (BCP) treatment on the T_C of Nb samples

Samples from Jlab, USA.



Conclusions : BCP degrades Tc considerably.

Supercon. Sci. Tech. Vol. 21 065002 (2008); Vol. 22 105014 (2009)

Some Results on the possible effects of hydrogen and oxygen on the superconducting properties of Nb materials





BCP treatment reduces H_{C1} as compared to that in pristine Nb.

SCRF cavity prepared with such BCP Nb would reach maximum 30-35 MV/m

Supercon. Sci. Tech. Vol. 21 065002 (2008); Vol. 22 105014 (2009)

Anomalous flux-pinning properties of chemically polished Nb materials



Supercon. Sci. Tech. Vol. 21 065002 (2008)

- Magnetization hysteresis, hence fluxpinning is less in chemically polished Nb samples.
- This is observed in fine grain, large grain and single crystal samples of Nb.
- Chemically polished samples is supposed to have Bean-Livingston surface barrier.
- The surface of the pristine samples is strained and have more impurity atoms. So it can have enhanced surface pinning.
- Absence of flux-jumps in BCP Nb indicates that bulk pinning is affected.

Superconducting properties of SCRF cavity Nb materials: EP versus BCP



- H_{C1} of EP treated sample is higher
- EP treated Nb SCRF-cavities show higher gradient E_{acc}

Temperature and magnetic field dependence of thermal conductivity of superconducting large grain Niobium

- Normal state κ (T) of BCP treated Nb is lower (by 10%) than that of pristine Nb.
- Both Tc and H_{C1} of BCP treated Nb is lower than the pristine Nb.
- A small but distinct dip in κ (T) is observed at H_{C1}



Temperature and magnetic field dependence of heat capacity of superconducting large grain Niobium



S B Roy et al (unpublished)

Performance of a Nb Quarter Wave Resonator



Q vs Accelerating Gradient plot

- QWR fabricated with the Nb materials at IUAC, New Delhi and installed in the superconducting LINAC.
- The electro-polished coupon of the same Nb materials has been studied in details for their superconducting properties.
- Limiting accelerating gradient is close to the intrinsic limit of accelerating gradient as predicted from the measured value of H_{C1} of the Nb coupons.

Phys. Rev. ST Accel. Beams Vol 14 12201 (2011)

Passivation with Nitrogen and annealing in Ti-gettered atmosphere of a Nb SCRF-cavity

- Both the procedures will increase the T_c of Nb material at least in the surface region.
- R_{BCS} at 2 K will be reduced in comparison to pure Nb, hence Q in SCRF cavity will go up.
- However, H_{C1} will decrease and as result E_{Acc} will also decrease.



AIP Conf. Proc. Vol 1686 02006 (2011)

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(75)	Inventors:	Sindhunil Barman Roy, Indore (IN); Vinod Chandra Sahni, Indore (IN)				
(73)	Assignee: Department of Atomic Energy, Mumbai (IN)		Aizaz et al, "Thermal Limitations in Superconducting RF Cavities: Improved Heat Transfer at Niobium-Helium Interface," C2-0-06 pre- sented at 2005 CEC/ICMC Key Stone, Colarado, USA, Advances in Crocenspingering np. 1-8.*			
(*)	Notice:	Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 1590 days.	* cited by examiner			
(21)	Appl. No.:	11/978,163	Primary Examiner — Stanley S	Silverman		
(22)	Filed:	Oct. 26, 2007	Assistant Examiner — Kallamb (74) Attorney, Agent, or Firm -	ella Vijayakumar — Ladas & Parry LLP		
(65)	US 2011/0	Prior Publication Data 183854 A1 Jul. 28, 2011	(57) ABSTR	ACT		

Ongoing and future works & newer SCRF materials

- Which one is most influential: H_{C1} or field for first flux-line penetration H_P ?
- Does upper critical field H_{C2} (or H_{C3}) play any role in the SCRF cavity ?
- Thermal conductivity in the superconducting state of Nb.
- Specific heat and thermal diffusivity.
- Detailed study of the surface resistance of superconductors R_{BCS} in applied magnetic fields.
- Nb thin films \rightarrow Nb-coated Copper cavities.
- Newer materials : **Nb-Zr**, Nb-Al, TiV, Mo-Re alloys, MgB₂ etc.

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applied surface science

www.elsevier.com/locate/apsusc

Surface studies of niobium chemically polished under conditions for superconducting radio frequency (SRF) cavity production

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Study of the chemical behavior of hydrofluoric, nitric and sulfuric acids mixtures applied to niobium polishing

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Response of a superconductor in ac-field Two fluid model: An ac cutstent in a superconductor is Calified ly O cooper pains - superfluid componed & Unpained electrons - normal componen Super current density : Js = it Eoe-int Where of = 202e2. Notional cuppert density : Jn = on Eoe-int Where on = mell Total J = Jo + Js = JE E P-int Where, J= J, + i Js

RE magnetic field penetrates a
superconductor only to a depth
$$\lambda_L$$
:
 $R_{surface} = Re\left(\frac{1}{\Lambda_L} \frac{1}{\sigma_n + i\sigma_s}\right)$
 $= \frac{1}{\Lambda_L} \frac{\sigma_n}{\sigma_s^2 + \sigma_s^2}$
In microwave Asequencies, $\sigma_n \ll \sigma_s$
 $\therefore \left[R_{surface} \approx \frac{1}{\Lambda_L} \frac{\sigma_n}{\sigma_s^2} \right]$
Sudstituting σ_n and σ_s
 $\left[R_{surface} \approx \frac{\mu_o^2 e^2}{m_e v_F} \Lambda_L^3 w^2 L n_n \right]$
NOW $\eta_n \ll e^{-\frac{Eg}{2k_BT}}$
 $\left[R_{surface} \approx \Lambda_L^3 w^2 L e^{-\left(\frac{1.76}{T}\right)} \right]$
For Nio/Limp

Electrical and mechanical properties			Content of the main impurities in wt. ppm ($\mu g g^{-1}$)		
RRR	>300	Та	≤500	Н	≤2
Grain size	≈50 µm	W	≤50	0	≤10
Yield strength, $R_p 0.2$	$50 < R_p 0.2 < 100 \text{ N mm}^{-2}$	Mo	≤50	N	≤10
Tensile strength	>140 N mm ⁻²	Ti	≤50	С	≤10
Elongation at fracture	>30%	Fe	≤30		
Vickers hardness HV10	≤60	Ni	≤30		

Table 1. Technical specifications of niobium applied to the fabrication of 1.3 GHz superconducting cavities for the EXFEL.

Electrical resistivity and thermal conductivity of large grain Niobium



Resistivity vs Temperature plot

NORMAL AND SUPERCONDUCTING PROPERTIES OF NIOBIUM RICH NIOBIUM-TANTALUM ALLOYS

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- 2 at% Ta suppresses T_C of Nb from 9.23 K to 9.02 K.
- Residual resistivity increases linearly with Ta concentration indicating homogenous distribution of Ta.
- Electronic coeff. of specific heat as a function of Ta shows anomalous feature