NIOBIUM IN SUPERCONDUCTING RF CAVITIES

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Abstract

Niobium is the favorite metal for the fabrication of superconducting accelerating cavities. While the majority of these cavities are formed from niobium sheet material a large number of copper cavities have been made with a thin niobium film on the inner surface produced by sputter coating. The resonators are operated well below the transition temperature of niobium (9.2K). A high thermal conductivity in the cavity wall is needed to guide the dissipated radio frequency (RF) power to the liquid helium coolant. In the case of bulk niobium cavities this requires niobium of exceptional purity with a gaseous contamination below 10 ppm (by weight) and a tantalum contents of less than 500 ppm. The Nb material must be free of foreign inclusions or metallurgical defects down to a scale of 20 µm. Considerable care must be applied during handling or machining the Nb parts in order to avoid any additional contamination. Most bulk niobium cavities are fabricated by deep drawing of half-cells from sheet material and electron beam welding. Hydroforming the accelerating structure from a seamless pipe is a cost-effective alternative. This technology has yielded excellent results in single-cell cavities and should be available for multicell structures in the near future. Final cleaning of the finished cavity by chemical or electrochemical methods and rinsing with ultraclean high-pressure water are essential steps to achieve a defect-free inner Nb surface such as needed in RF cavities at high fields. The production of niobium for cavities is a challenge for the fabrication process: low tantalum contents of the raw material, stringent vacuum requirements during electron-beam melting of the ingots, clean and well controlled conditions during sheet rolling, cutting and recrystallisation heating. Some Nb producers have developed adequate procedures. Open communication between these companies and the user laboratories was very helpful to reach the required specification. Up to now a total amount of about 25 tons of high purity niobium has been purchased for the fabrication of superconducting cavities. During the last years typically 2 tons per year have been ordered. There is worldwide interest in new superconducting accelerators for elementary particle physics and fourth generation synchrotron light sources, in particular Free Electron Lasers in the ultraviolet and X-ray regime. Therefore a continuously growing demand for high purity Nb is expected. The proposed linear collider project TESLA requires 500 tons of high purity niobium at a fabrication schedule of three years.

Introduction

The accelerating structures in high energy particle accelerators are usually radio frequency (RF) cavities with a frequency ranging from 50 MHz to above 10 GHz, depending on the velocity v of the particles and on geometric considerations. The high frequency fields induce surface currents in the metallic boundary which lead to power dissipation proportional to the resistance of the metal. Therefore, a high-conductivity metal is chosen to limit these losses but even for copper the loss in the metallic walls is the dominant part of the required radio-frequency power. The excessive cooling demand in normal conducting accelerating structures limits the maximum accelerating gradient to values of less than 3 million volts per meter (3 MV/m) in continuous wave operation. In pulsed operation accelerating fields in excess of 50 MV/m seem accessible but the pulse duration must be kept quite low, in the order of microseconds.

In the late 1960s the development of superconducting radio-frequency accelerating structures was started. The critical magnetic field of niobium would in principle allow for gradients up to 50 MV/m. There exist superconductors with a higher critical field, Nb₃Sn or YBa₂Cu₃O₇ for example, but in practice cavities based on these materials have shown much inferior performance as compared to Nb cavities.

The main advantage of superconducting accelerating structures is the fact that they enable high gradients under continuous wave or pulsed operation with millisecond long RF pulses. It should be noted, however, that in contrast to superconducting magnets, which are operated with a dc or a low frequency ac current without any power dissipation, the high-frequency alternating currents in microwave cavities are always associated with the generation of ohmic heat which has to be removed by a refrigerator. Nevertheless, the overall energy efficiency of a superconducting cavity system, including the cryogenic efficiency of the helium liquefier, is considerably higher than that of a normal conducting system.

At present, the maximum operating accelerating gradient of a superconducting accelerating system is 23 MV/m. Individual tests of multi-cell and single-cell cavities demonstrated values of 34 MV/m and 43 MV/m, respectively. There are two major limitations, thermal instabilities and field emission of electrons:

• normal conducting defects in the surface produce heat and may eventually drive the superconductor above the critical temperature. The resulting breakdown of superconductivity is called a quench. To avoid a quench the niobium surface must be kept as free as possible from normal conducting defects. Furthermore a high thermal conductivity of the bulk material is required to thermally stabilize a possible quench location.

• in the region of high electric field electrons may be emitted from sharp tips or from foreign particles sticking to the surface via the process of field emission. These electrons gain energy in the RF field, impact on the superconducting surface and produce excessive heating. Perfect cleaning of the cavity surface with high-pressure ultra-clean water is the best remedy against this type of breakdown.

Resonators for Acceleration

There are two different classes of accelerating structures, depending on the speed v of the particles to be accelerated:

• near to the speed of light c (0.5 < v/c < 1)

• or smaller than 50% of the speed of light (v/c < 0.5).

The first case is typical for electrons above a few MeV of kinetic energy and protons above 100 MeV. In principle the accelerating structure consists of a series of pill box cavities which are joined by short beam pipe sections. In real structures one prefers a smooth elliptical cross section which has the advantage to suppress another undesirable performance limitation called multipacting (1), the resonant multiplication of electrons via the acceleration in the RF field and secondary electron emission upon impact on the surface. The TESLA cavity (2), shown in Figure 1, is typical for superconducting multi-cell structures.



Figure1: Side view of the TESLA 9-cell cavity as example for an accelerating structure for electrons. The cavity is made from 2.5 mm thick niobium and cooled by superfluid helium at 2 Kelvin.

The second case is typical for the acceleration of ions. Here a variety of different geometries (e.g. coils, capacitive cones) are placed inside a metal housing. The accelerating gap is short enough that the "slow" particles can cross it within one half RF cycle.

The yearly average demand of niobium for such accelerating structures is less than 10% of that required for electron and proton accelerators. Therefore the further discussion in this paper is concentrated on the first type of structures.

Past and Present Operating Systems

TRISTAN Storage Ring at KEK

KEK is a national Japanese laboratory for high-energy physics. In the early 1980s the energy of the TRISTAN (3) electron-positron storage ring was upgraded by installing 32 superconducting cavities in addition to the existing 104 normal conducting resonators. The production, assembly and installation of the superconducting cavities were carried out mainly by industrial firms. The cavities were fabricated from niobium sheets by spinning, electron beam welding, electro-polishing and heating at 800 °C. The heat treatment was needed to clean the Nb from hydrogen which was picked up during electrolytic polishing. Two five-cell cavities were housed in one cryostat. The superconducting cavities were operated at a gradient between 3 and 4.7 MV/m. The performance (maximum accelerating field and radio frequency losses) did not deteriorate during seven years of operation.

LEP Storage Ring at CERN

CERN is a European center for high-energy physics, situated at Geneva, Switzerland. In the first stage the accelerating system of the LEP electron-positron storage ring consisted of normal conducting cavities (352 MHz). To upgrade the beam energy from 45 GeV to 104 GeV, 288 superconducting four-cell cavities (352 MHz) were installed in addition to the 84 normal conducting resonators (4). With the exception of the first 20 cavities, the superconducting resonators were not made from solid Nb material but by sputtering Nb onto Cu. Two major arguments are quoted in favour of this technology: cost savings by the reduced amount of Nb material (a 5 μ m layer instead of a 4 mm thick sheet) and stabilization of thermal instabilities by the high thermal conductivity of Cu.

The technology of sputter-coating large surface areas was developed at CERN during the 1980s and then transferred to industry (5). After a learning process the cavity fabrication reached a high standard. The superconducting accelerating system was operated from 1989 to 2000. The operation was ended for the installation of the Large Hadron Collider LHC in the LEP tunnel.

HERA Electron-Proton Storage Ring at DESY

DESY is a national German laboratory for high-energy physics, situated at Hamburg, Germany. The HERA storage ring facility (6) collides 920 GeV protons with 27 GeV electrons or positrons. The radio frequency system of the electron ring consists of 82 normal conducting and 16 superconducting 500 MHz cavities. The superconducting cavities were produced by industry (spinning of half-cells from Nb sheets, electron beam welding, surface preparation by tumbling and chemical cleaning) whereas DESY staff carried out the assembly and installation. The average gradient of the installed cavities was 5 MV/m. The superconducting accelerating system has been in operation since 1992.



Figure 2: Superconducting accelerating system for the HERA storage ring. Nb cavity with (right) and without (left) the vessel for liquid helium. The cryostat with flanges for the helium pipes (top) and the input couplers (middle) is seen in the background.

CEBAF at Jefferson Laboratory

Jlab (Jefferson Laboratory) is a laboratory for nuclear physics research. It is situated at Norfolk, Virginia, USA. In the recirculating linac CEBAF (7) electrons are accelerated to energy of up to 6 GeV. Key performance characteristics are the energy resolution of 10^{-4} and the continuous wave operation.

The accelerator consists of two superconducting linacs with four magnetic bends for recirculation of the beam. The superconducting cavities were fabricated by industry from Nb sheets. The final cleaning, testing and installation were carried out by CEBAF staff. The accelerator was commissioned in 1995 and physics runs started in 1996. The CEBAF machine is at present the largest superconducting cavity installation. In total 330 five-cell 1500 MHz cavities are operated at 2K.

The TESLA Test Facility TTF

Within the framework of an international collaboration, a development project was launched to explore the feasibility of the superconducting linear collider TESLA (2) (TeV Energy Superconducting Linear Accelerator). The TESLA Test Facility (TTF) at DESY incorporates the facilities for chemical etching, high-pressure water rinsing, high-temperature heat treatment, clean-room assembly and RF testing of superconducting multi-cell cavities as well as the installation and operation of an experimental superconducting accelerator with 500 MeV beam energy. The nine-cell cavities (see Figure 1) are made from Nb sheet material and resonate at 1.3 GHz. For a detailed description see (8). They are treated by an automated chemical system under clean room conditions and are processed by high-pressure water rinsing and, if necessary, by conditioning with RF pulses of high instantaneous power. Eight cavities are grouped in one cryostat, four of these modules are needed for the first test accelerator. The main goal is to operate the experimental linac at a gradient of 20 MV/m and to upgrade the cavity performance to the TESLA design value of 23.5 MV/m. A second development goal is to simplify the cavity design and fabrication techniques in order to reduce the investment costs of a future linear collider.



Figure 3: Measured performance of the latest 9-cell cavity production for TTF. Plotted is the quality factor as a function of the accelerating field.

Up to the summer of 2001 a total of 79 nine-cell cavities were produced and measured. Fig 3 shows the measured performance of the latest cavity production.

In 1999 a proposal was approved to upgrade the TTF linac energy to 1 GeV and to add an undulator section for production of Free Electron Laser radiation with a tunable wavelength from soft X-rays (6 nm) up to the ultraviolet regime (9). The user facility for FEL radiation will be commissioned in 2004.

Projects Under Development/Installation

Spallation Neutron Source (SNS)

Neutrons are an important tool to probe the structure and properties of matter. A continuous flux of slow neutrons is available for this purpose at various research reactors. New applications in basic and applied research (chemistry, biotechnology, geo-science and other fields) require a pulsed and high intensity neutron flux. These neutrons can be produced by spallation: an intense proton beam hits a high-density target (e.g. mercury) so that the spallation process creates neutrons. The Spallation Neutron Source SNS (10) is under construction at Oak Ridge, Tennessee. The protons will be accelerated by copper cavities to 186 MeV and then by superconducting resonators up to a final energy of 1 GeV. The superconducting linac will consist of 93 resonators of 5 cells each. The operating frequency is 805 MHz. In early 2001 the prototype cavities were successfully tested. The commissioning of the SNS laboratory is scheduled for 2006.

Superconducting Accelerating Cavities for High Beam Currents

At Cornell University (Ithaca, NY, USA) the storage ring CESR (11) for electrons and positrons has been operated for particle physics since many years. The latest upgrade of the beam current could be achieved by replacing the normal conducting with superconducting resonators. The shape of superconducting resonators is much more suited to accelerate high beam current without excitation of beam instabilities. Such an accelerating structure is typically a single-cell cavity with a high power RF input coupler. One resonator is housed in one cryostat, the damping element for beam-induced higher frequency power is attached to the room temperature section of the beam pipe. Three of these resonators have been operated in the storage ring until late 2000, two more will be added in the near future.

Two resonators of the Cornell type are under production by the ACCEL Company for the Taiwan synchrotron light source. Also in this case the beam current will be upgraded (from 50 mA to 500 mA) by replacing the old normal conducting resonators.

A similar system as in CESR is operated at the KEKB (12) storage ring (KEK laboratory, Tsukuba, Japan). Six single cell resonators with a resonant frequency of 501 MHz are in use since 2000.



Figure 4: Superconducting accelerating unit for high electron current (Cornell). This design is characterised by the use of a single cell cavity with an enlarged beam pipe (as compared to the multicell design in figure 1), a large input coupler and an absorber of higher-order mode power.

| Laboratory | Operational (planned) | Frequency (MHz) | Active length (m) | Comme | |
|--------------------------|--------------------------|--------------------|----------------------|-----------------------|--|
| KEK, TRISTAN | 1988-94 | 508 | 48 | decommissioned | |
| Darmstadt. S- DALINAC | 1990 | 2997 | 10.25 | operational | |
| DESY, HERA | 1991 | 500 | 19.2 | operational | |
| CEBAF | 1996 | 1497 | 169 | operational | |
| CERN, LEP | 1997 | 352 | 462 | decommissioned | |
| SNS, Oak Ridge | (2006) | 805 | 76 | under construction | |
| TTF, DESY | 1999 | 1300 | 16 | operational | |
| CESAR III, Cornell | 1997 | 500 | 1.2 (+0.6) | operational | |
| KEKB | 1998 | 508 | 2.4 | operational | |
| TTF-FEL | (2003) | 1300 | 48 | under construction | |
| TESLA | ? | 1300 | 20400 | Technical Proposal | |
| RIA | ? | Several | (250?) | under discussion | |

Table I Larger superconducting accelerator installations (SNS: protons, RIA: ions, else: electrons)

<u>TESLA</u>

TESLA is a proposed linear collider for electrons and positrons. The two beams will be accelerated to an energy of 250 GeV each. The major aim is to investigate the most urgent problems in particle physics, the Higgs mechanism and supersymmetry. An integral part of the facility are tunable X ray Free Electron Lasers with a wavelength down to 1 Å which will driven by a 25 - 50 GeV electron beam derived from the main linac. The superconducting accelerator will consist of twenty thousand 9 cell cavities. In March 2001 the Technical Design Report (2) was published. A decision on the project is expected in 2004.

Synchrotron Light Sources with Superconducting Resonators

Conventional synchrotron light sources consists of an electron storage ring with various undulator and wiggler magnet insertions for the production of radiation ranging from the optical into the X ray regime. Superconducting cavities are well suited to accelerate high beam currents without distortion of the beam quality. The Cornell resonator (see Fig. 4) is a typical example of such a design. Only a few resonators are needed at one facility, but there are proposals in many countries to build such radiation laboratories.

Free Electron Lasers with Superconducting Linacs

Free electron lasers (FELs) offer the possibility to generate radiation (infrared, visible, UV, Xray) of unprecedented quality: brilliance, spectral purity and short pulse length. X ray FELs will be many orders of magnitude more powerful than any other X-ray source. A large advantage in comparison with conventional lasers is the tunability of the wavelength, which is simply achieved by varying the electron energy. A superconducting linac is the ideal drive source of an FEL as it provides a beam of small transverse emittance and high peak current. As mentioned above the TESLA facility will incorporate X-ray FELs.

Proposals for stand-alone FEL facilities with superconducting acceleration systems are under preparation at several laboratories. Energy recovery is possible by de-celeration of the spent electron beam and has been demonstrated at Jlab (14).

Proton Accelerators for Nuclear Waste Transmutation and Neutron Production

There is a growing demand for superconducting proton accelerators in the energy range of several GeV. The SNS project presently under construction is an example for the production of high flux of pulsed neutrons.

Burning of nuclear waste could be done with the use of a proton accelerator. Long living radioactive isotopes (typical lifetime of 20000 years) will be converted into isotopes with shorter lifetime (about 500 years). It is expected that the conversion process actually will produce more energy than needed to operate the nuclear waste burner. Key development goals are the superconducting high current linac and the high power target station. The development time for a demonstrator and an operational facility is expected to take 20 and 30 years, respectively.

Rare Isotope Accelerator

The Rare Isotope Accelerator (RIA) is a next generation radioactive beam facility in preparation in the USA. In this machine, intense heavy ion beams of typically 400 MeV per nucleon would be directed at a thin target. The exotic fragments would be re-accelerated for nuclear structure and astrophysics studies. The accelerator could also produce protons with energies up to about 700 MeV. This beam could be used to irradiate thick target in an "Isotope Separation On-Line" (ISOL) mode. In RIA the beams would essentially be continuous. The design of both the primary and post accelerator relies on superconducting cavity technology. The RIA project is under detailed technical discussion (15), the time schedule for R&D and realization is in the order of 10 years.

Physics of RF Superconductivity

Choice of Superconductor

The existing large scale applications for superconductors are magnets and accelerating cavities. A common requirement is a high critical temperature¹, but there are distinct differences concerning the critical magnetic field. In magnets operated with a dc or a low-frequency ac current, `hard' (type II) superconductors are required, with high upper critical fields (15 - 20 T) and strong flux pinning in order to achieve high current density; such properties are only offered by alloys like niobium-titanium or niobium-tin. In microwave applications the limit is essentially set by the thermodynamic critical field, which is well below 1 T for all known superconductors. Strong flux pinning is undesirable as it is coupled with losses due to hysteresis. Hence a 'soft' superconductor must be used. Pure niobium is the best candidate, although its critical temperature T_c is only 9.2 K, and the thermodynamic critical field about 200 mT. Niobium-tin (Nb₃Sn) with a critical temperature of 18 K looks more favourable at first sight, however the gradients achieved in Nb₃Sn coated niobium cavities were always below 15 MV/m, probably due to grain boundary effects in the Nb₃Sn layer. For this reason niobium is the preferred superconducting material.

Microwave Surface Resistance

Superconductors suffer from energy dissipation in microwave fields since the radio frequency (RF) magnetic field penetrates a thin surface layer and induces oscillations of the unpaired electrons. According to the Bardeen-Cooper-Schrieffer (BCS) (16) theory of superconductivity, the surface resistance is given by the expression

$$R_{BCS} \propto \frac{f^2}{T} \exp\left(-1.76T_c/T\right),\tag{1}$$

where f is the microwave frequency. For example the niobium BCS surface resistance at 1.3 GHz is about 800 nO at 4.2 K, and drops to 15 n at 2 K (see Figure 5). Because of the exponential temperature dependence, operation at 1.8-2 K is essential at this frequency for achieving high accelerating gradients in combination with very high quality (Q) factors.

¹ The High-T_c ceramic superconductors have not yet found widespread application in magnets, mainly due to technical diffculties in cable production and coil winding. Cavities with High-T_c sputter coatings on copper have shown much inferior performance in comparison to niobium cavities.

Superfluid helium is an excellent coolant owing to its high heat conductivity. In addition to the BCS term there is a residual resistance R_{res} caused by impurities, frozen-in magnetic flux, or lattice distortions. This term is temperature independent and amounts to a few nO for very pure niobium, but increases dramatically if the surface is contaminated.



Figure 5: Measured surface resistance of a 9-cell TESLA cavity. The residual resistance of 3 nO corresponds to a quality factor of 10¹¹.

Influence of Magnetic Fields

<u>Superheating Field.</u> Superconductivity breaks down when the RF magnetic field exceeds the critical field of the superconductor. In the high frequency case it is believed that the so-called "superheating field" is relevant which for niobium is about 20% higher than the thermodynamical critical field of 200mT.

<u>Trapped Magnetic Flux</u>. Niobium is in principle a soft type II superconductor without flux pinning. In practice, however, weak magnetic dc fields are not expelled upon cooldown but remain trapped in the niobium. Each flux line contains a normal-conducting core whose area is roughly px_0^2 where x_0 is the coherence length which amounts to 40 nm in Nb. Trapped magnetic flux results in a surface resistance

$$R_{mag} = \left(B_{ext} / 2B_{c2}\right)R_n \tag{2}$$

where B_{ext} is the externally applied field, B_{c2} the upper critical field and R_n the surface resistance in the normal state. At 1.3 GHz the surface resistance caused by trapped flux amounts to 3.5 nO/µT for niobium. Cavities, which are not shielded from the Earth's magnetic field, are therefore limited to Q_0 values below 10^9 .

Thermal Instability and Field Emission

One basic limitation of the maximum field in a superconducting cavity is thermal instability. Temperature mapping at the outer cavity wall usually reveals that the heating by RF losses is not uniform over the whole surface but that certain spots exhibit larger temperature rises, often beyond the critical temperature of the superconductor. Hence the cavity becomes partially normal conducting, associated with strongly enhanced power dissipation. Because of the exponential increase of surface resistance with temperature this may result in a run-away effect and eventually a quench of the entire cavity. Analytical models as well as numerical simulations are available to describe such an avalanche effect (17). Input parameters are the thermal conductivity of the superconductor, the size and resistance of the normal conducting spot and the Kapitza resistance (thermal resistance between the outer niobium wall and the liquid Helium). The tolerable defect size depends on the residual resistivity ratio² RRR of the material and the desired field level. As a typical number, the diameter of a normal-conducting spot must exceed 50 μ m to be able to initiate a thermal instability at 25 MV/m for RRR>200.

There have been many attempts to identify defects, which were localized by temperature mapping. Examples of defects are drying spots, fibers from tissues, foreign material inclusions, weld splatter and cracks in the welds. There are two obvious and successful methods for reducing the danger of thermal instability:

- avoid defects by preparing and cleaning the cavity surface with extreme care;
- increase the thermal conductivity of the superconductor.

Considerable progress has been achieved in both aspects over the last ten years.

Heat Conduction in Niobium

The heat produced at the inner cavity surface has to be guided through the cavity wall to the superfluid helium bath. The thermal conductivity of niobium exhibits a strong temperature dependence in the cryogenic regime and scales approximately with the RRR, which can be calculated in terms of the impurity contents:

$$\mathbf{I}(4.2K) \approx 0.25 \cdot RRR \quad \left[W / (m \cdot K) \right] \tag{3}$$

$$RRR = \left(\sum_{i} f_{i} / r_{i}\right)^{-1}$$
(4)

where the f_i denote the fractional contents of impurity i (measured in weight ppm) and the r_i the corresponding resistivity coefficients which are listed in the following table.

| Table II weight factor r_i of some impurities (see equation (4)) | | | | | | | | |
|--|------|------|------|------|-----|--|--|--|
| Impurity atom <i>i</i> | Ν | 0 | С | Н | Та | | | |
| r_i in 10 ⁴ wt. ppm | 0.44 | 0.58 | 0.47 | 0.36 | 111 | | | |

Table II Weight factor r_i of some impurities (see equation (4))

² RRR is defined as the ratio of the resistivities at room temperature and at liquid helium temperature. The low temperature resistivity is usually measured at 4.2 K, applying a magnetic field to assure the normal state.



Figure 6: Measured heat conductivity of samples from the niobium sheets used in the TESLA cavities: before and after the 1400 °C heat treatment (RRR = 280 and RRR = 500 respectively).

Diagnostic Methods and Quality Control

In the first series production of TESLA cavities deficiencies have been found which could be traced back to material effects and inadequate cleaning procedures during electron-beam welding. As a consequence new diagnostic methods and quality control procedures were introduced.

Eddy-Current Scanning

A practical device for the quality control of all niobium sheets going into cavity production is a high-resolution eddy-current system (18)developed by the Bundesanstalt fuer Materialforschung (BAM) in Berlin and DESY. In the xy scanning system the frequency used is 100 kHz corresponding to a penetration depth of 0.5 mm in niobium at room temperature. The maximum scanning speed is 1 m/s. The scanning probe containing the inducing and receiving coils floats on an air pillow to avoid friction. The machined base plate contains holes for evacuating the space between this plate and the Nb sheet. The atmospheric pressure is sufficient to flatten the $265 \times 265 \text{ mm}^2$ niobium sheets to within 0.1 mm which is important for a high sensitivity scan. The performance of the apparatus was tested with a Nb test sheet containing implanted tantalum deposits of 0.2 to 1 mm diameter. The scanned picture demonstrates that Ta clusters are clearly visible. Using this eddy-current apparatus the tantalum inclusion in a defective cavity was easily detectable.

In the meantime an improved eddy-current scanning device has been designed and built at BAM which operates similar to a turn table and allows for much higher scanning speeds and better sensitivity since the accelerations of the probe head occurring in xy scans are avoided. A two frequency principle is applied in the new system. Scanning with high frequency (about 1 MHz) allows detection of surface irregularities while the low frequency test (about 150 kHz) is sensitive to bulk inclusions. The high and low frequency signals are picked up simultaneously. Very high sensitivity is achieved by signal subtraction.



Figure 7: Eddy current scanning apparatus for niobium discs. This technology was developed by DESY and BAM (Bundesanstalt für Materialprüfung, Berlin). The above photo shows an equipment built for Jlab by the FER-PA GmbH (Forschung-Entwicklung-Rationalisierung), Magdeburg, Germany.

Squid Scanning

Some Nb sheets of TESLA dimensions with material inclusions, found by eddy current scanning test, were tested with a SQUID gradiometer system. The SQUID system features higher sensitivity. An iron particle with a dimension less then 50µm, not detectable with the eddy current method, was easily found inside of the Nb sheet. Detection of rather deep inclusions in the material or investigation of the inside surface of closed parts (cavities, tubes) is possible because of the high penetration depth of the SQUID method. These investigations were conducted in collaboration between the FIT Messtechnik GmbH and DESY. Recently a new research program was started in collaboration between WKS Messtechnik GmbH, W.C. Heraeus GmbH, IAP University of Giessen and DESY to develop a SQUID scanning apparatus for industrial use.

Electron Microscopy

Scanning electron microscopy with energy-dispersive X-ray analysis (EDX) is used to identify foreign elements on the surface. Only a depth of about 1 μ m can be penetrated, so one has to remove layer by layer to determine the diffusion depth of titanium or other elements.

Alternatively one can cut the material and scan the cut region. The titanium layer applied in the high temperature treatment has been found to extend to a depth of about 10 μ m in the bulk niobium. The sensitivity of the EDX method is rather limited; a Ti fraction below 0.5% is undetectable. Auger electron spectroscopy offers higher sensitivity and using this method titanium migration at grain boundaries has been found to a depth of 50-100 μ m. Hence this large thickness must be removed from the RF surface by chemical etching after heat treatment with Ti getter.

X-ray Radiography

X ray radiography is a method based on the differential absorption of X-ray radiation by the piece being inspected. Variations in density and thickness or differences in absorption characteristics lead to a varying transmission of the X-rays, which can be on film or by a LC detector. The advantage of this method is that large samples or complex geometries can be analysed. Figure 8b gives an example of X-ray radiography. The dark spot corresponds to the zone of enhanced heating seen in figure 8a. This area has been identified as the quench location of a niobium cavity.



Figure 8: (a) Temperature map of one cell of a low quench field cavity. Excessive heating was detected at a localised spot. (b) Positive print of an X-ray radiograph showing the 'hot spot' as a dark point.

X-ray Fluorescence

The narrow-band X-ray beams at HASYLAB and other synchrotron radiation facilities permit element identification via fluorescence analysis. In principle the existing apparatus allows the scanning of a whole niobium sheet such as used for producing a half-cell, however the procedure would be far too time-consuming. But in the case of a well localized quench spot it can be worthwhile, to cut the cavity and analyse the quench region. This was done for one of the early TTF cavities. A quench location as detected by temperature mapping and X ray radiography (see fig. 8) has been further analysed by X-ray fluorescence. The first spectrum (solid curve in figure 9) was recorded far away from the bad spot while the second one (dotted curve) was measured in the middle of the spot. Both spectra display the characteristic X-ray lines of niobium and tantalum. (Ta- $K_{\alpha 1}$ =57.532 keV, Ta- $K_{\alpha 2}$ =56.277 keV, Ta- $K_{\beta 1}$ =65.223 keV). The contents of uniformly dissolved Ta in the Nb sample is 200 - 300 ppm according to a chemical analysis. This Ta is responsible for the Ta- K_{α} and K_{β} signal increase by a factor of 10 which implies a Ta contents of about 3000 ppm, which is far above the tolerable limit.



Figure 9: Spectrum of K-lines of synchrotron radiation fluorescence in the Ta spot area (dotted line) and far away of the spot (full line).

Neutron Activation Analysis

The eddy-current scan allows the detection of foreign materials in the niobium but is not Neutron activation analysis permits a non-destructive determination suitable for identification. of the contaminants provided they have radioactive isotopes with a sufficiently long half life. Experiments were carried out at the research reactor BER II of the Hahn Meitner Institut in The niobium sheets are exposed to a thermal neutron flux of 10^9 cm⁻²s⁻¹ for some 5 Berlin. hours. The radioactive isotope ⁹⁴Nb has a half life of 6.2 min while ¹⁸²Ta has a much longer half life of 115 days. Two weeks after the irradiation the ⁹⁴Nb activity has dropped to such a low level that tantalum fractions in the ppm range can be identified. Figure 10 shows the implanted tantalum clusters in the specially prepared Nb plate with great clarity. Also the uniformly dissolved Ta is visible and the inferred concentration of 200 ppm is in agreement with the chemical analysis. The activation analysis is far too time consuming for series checks but can be quite useful in identifying special contaminations found with the eddy-current Ten Nb sheets from the regular production were investigated without showing any system. evidence for tantalum clusters.



Figure 10 Ta inclusions as detected by neutron activation method. The Nb test sheet (265 mm x 265 mm x 2.8 mm) contains implanted tantalum deposits of 0.2 to 1 mm diameter and was used to check the sensitivity of the neutron activation method.

Production of Nb Cavities

Niobium Specification

The niobium specification for the TESLA cavities is listed in Table III. The most important metallic impurity in niobium is tantalum, with a typical concentration of 500 ppm. The interstitially dissolved gases (mainly oxygen) act as scattering centers for the unpaired electrons and reduce the thermal conductivity. The niobium ingot is out-gassed by several melting cycles in a high vacuum electron beam furnace. The interstitial oxygen, nitrogen and carbon contamination is reduced to a few ppm. The Nb ingots are forged and rolled into sheets of 2.8 mm thickness. After rolling the Nb sheets are first degreased and cleaned by chemical etching. The sheets are then annealed for 1-2 hours at 700-800 °C in a vacuum oven at 10^{-5} - 10^{-6} mbar to achieve full recrystallization and a uniform grain size of about 50 µm. The finished Nb sheets are eddy-current checked for defects like cracks or foreign inclusions which might impair the superconducting properties.

| Impurity content in ppm (wt) | | in ppm (wt) | Mechanical Properties | | |
|------------------------------|-------|-------------|-----------------------|--------------------------------|----------|
| Та | = 500 | Н | = 2 | Residual resistivity ratio RRR | = 300 |
| W | = 70 | Ν | = 10 | grain size | ~ 50 µm |
| Ti | = 50 | 0 | = 10 | yield strength | > 50 MPa |
| Fe | = 30 | С | = 10 | tensile strength | >100 MPa |
| Mo | = 50 | | | elongation at break | 30 % |
| Ni | = 30 | | | Vickers hardness HV 10 | = 50 |

Table III Technical specification for niobium used in TESLA cavities

Cavity Fabrication

Cavity fabrication by electron-beam welding of deep-drawn half-cells is a delicate procedure, requiring intermediate cleaning steps and a careful choice of the weld parameters to achieve full penetration of the joints. First, two half cells are connected at the iris; the stiffening rings are welded in next. At this point weld shrinkage may lead to a slight distortion of the cell shape which needs to be corrected. Particularly critical are the equator welds, which are made from the outside, and a reliable method for obtaining a smooth weld seam at the inner cavity surface was required. For the TESLA cavity production a two-pass procedure was developed, where 50% of the beam power is applied to the first weld pass, and 100% on the second. In both cases, a slightly defocused electron beam rastered in an elliptic pattern is used. The electron-beam welding technique of niobium cavities has been perfected in industry to such an extent that the weld seams do not limit cavity performance below ~30 MV/m.

A challenge for a welded construction is the tight mechanical and electrical tolerances. These can be maintained by a combination of mechanical and radio frequency measurements on half cells and by careful tracking of weld shrinkage. The procedures established during the TTF cavity fabrication are suitable for large series production, requiring quality assurance measurements only on a small sample of cavities.

The TESLA cavities are equipped with niobium-titanium flanges at the beam pipes and the coupler ports. NbTi can be electron-beam welded to niobium and possesses a surface hardness equivalent to that of standard UHV flange material (stainless steel 316 LN/DIN 1.4429). Contrary to pure niobium, the alloy NbTi (ratio 45/55 by weight) shows no softening after the heat treatment at 1400 °C. O-ring type aluminum gaskets provide reliable seals in super fluid helium.

Cavity Treatment

A layer of 100-200 µm is typically removed in several steps from the inner cavity surface to obtain good RF performance in the superconducting state. The standard method applied for niobium cavities is called Buffered Chemical Polishing (BCP), and uses an acid mixture of HF (48 %), HNO₃ (65 %) and H₃PO₄ (85 %) in the ratio 1:1:2. to 1:1:4. The acid is cooled (typically to 5 °C) and pumped through the cavity in a closed loop. At some laboratories the cavity is only dipped into the acid. After the chemical etching the cavities are rinsed with ultrapure water and dried in a class 100 clean room. The TESLA procedure foresees a subsequent anneal at 800 ℃ in an Ultra High Vacuum (UHV) oven to out-gas dissolved hydrogen and relieve mechanical stress in the deep drawn niobium. In a second UHV oven the cavities are heated to 1350-1400 °C at which temperature the other dissolved gases diffuse out of the material, and the residual resistivity ratio RRR increases by about a factor of two to around To absorb the oxygen diffusing out of the niobium and to prevent oxidation by the 500. residual gas in the oven (pressure $< 10^{-7}$ mbar), a thin titanium layer is evaporated on the inner and outer cavity surface (Ti being a stronger getter than Nb). The titanium layer is later removed by 80 µm and 20 µm BCP of the inner and outer cavity surface respectively. This high-temperature treatment with Ti getter is referred to as post-purification. A severe drawback of post-purification is the considerable grain growth of the niobium: post-purified cavities are vulnerable to plastic deformation and have to be handled with great care.

After the final heat treatment, the cavities are mechanically tuned to adjust the resonance frequency to the design value and to obtain equal field amplitudes in all 9 cells. This is followed by a light BCP, three steps of high-pressure water rinsing (100 bar), and drying in a class 10 clean room. The final acceptance step is a RF test in a superfluid helium bath cryostat.

Electrolytic Polishing (Electropolishing)

The Buffered Chemical Polishing (BCP) used at TTF to remove a 100-200 μ m thick damage layer produces a rough niobium surface with strong grain boundary etching. An alternative method is `electropolishing' (EP) in which the material is removed in an acid mixture under current flow. Sharp edges and burrs are smoothed out and a very glossy surface can be obtained. The electropolishing for niobium has been known for many years, it was applied e.g. for the cleaning of the KEK-TRISTAN resonators. But only recently very remarkable results with electropolished single cell resonators pushed further investigations with multi-cell cavities. There is hope, that electropolishing might offer the chance to eliminate the 1400° heat treatment and eventually relax the specification for the heat conductivity (RRR value).

Hydro-Forming of Seamless Cavities

The hydro-forming of a seamless bulk niobium cavity of the TESLA shape, with a ratio of equator to iris diameter of about three, is a challenging task. Starting from a seamless tube of intermediate diameter the forming procedure consists of two stages: reduction of the tube diameter in the iris area and expansion in the equator area.

Before a hydro-forming experiment takes place the strain-stress properties of the niobium tube material are determined and the forming process is studied in a computer simulation. A hydraulic two-dimensional bulging of a Nb disk into a spherical form is used to derive the strain-stress diagram. The numerical simulation of the hydraulic expansion of the tube is made with the finite element code ANSYS. The calculations have been carried out on the basis of the experimentally determined strain-stress characteristic of the niobium tubes and resulted in a

relation between applied internal pressure against axial displacement and radius for the hydroforming process (19).



Figure 11: Excitation curve of a TESLA 9-cell cavity after buffered chemical polishing (BCP) and electropolishing (EP).

A machine for hydro-forming experiments was built at the INR institute (Russia) and equipped with computer control at DESY. The hydraulic expansion is possible in stepwise as well as in continuous mode. It was found from tensile tests that the elongation before necking is almost 30% higher by application of a pulse technique instead of monotonously increasing the stress. An additional 10% of strain before necking can be gained by keeping the strain rate below 10^{-3} s⁻¹. During the actual forming process an internal pressure is applied to the tube simultaneously with an axial compression, deforming the tube into an external mould. The deformation is controlled by computer, following the theoretically determined relation between internal pressure and axial displacement. In the final step the cavities are calibrated in the mould by increasing the pressure to 1000 bar to obtain high dimensional accuracy.

Various methods for the production of seamless Nb tubes have been and are being developed in cooperation with scientific institutes and industrial companies: spinning, back extrusion, flow forming and deep drawing. A uniform, small grain and homogeneous texture of the niobium is required to provide a high degree of plastic deformability.

Several single cell cavities have been manufactured so far. The developed technique is also suitable for multicell cavities; the first double cell cavity has recently been made at DESY. Figure 12 shows two single cell resonators and the results of a RF test at Jefferson Lab (20). After electropolishing the excellent gradient of 43 MV/m at $Q_0 = 1.5 \cdot 10^{10}$ was achieved which demonstrates the high capability of the hydro-forming process.



Figure 12: Example of two hydroformed cavities and test results after chemical etching (closed circles) and after electropolishing (open circles).

Conclusion and Outlook

As result of pioneering work at Cornell and Wuppertal and intensive R&D at several high energy physics and nuclear physics laboratories superconducting cavities for accelerators have become a mature technology. The recent effort of the international TESLA collaboration further pushed the performance of superconducting cavities towards gradients above 25 million volts per meter. Methods were investigated to considerably reduce fabrication costs. There exists a fruitful discussion with the niobium producing industry to assure the fabrication of high quality niobium needed for superior cavity performance.

Many laboratories plan their future accelerators on the basis of superconducting cavities, like light sources or free electron lasers, spallation neutron sources or rare isotope accelerators. Large future installations for fundamental physics research like linear colliders or neutrino factories profit considerably from this technology. It is therefore important to cooperate with the niobium companies and prepare long term planning on the needs of high purity niobium. In particular the required 500 tons of Nb for TESLA or the estimated 30 tons for RIA will be a challenge for the production.

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