MECHANICAL PROPERTIES OF HIGH RRR NIOBIUM AT CRYOGENIC TEMPERATURES *

M. G. Rao and P. Kneisel

Continuous Electron Beam Accelerator Facility
12000 Jefferson Avenue
Newport News, VA 23606-1909 USA

ABSTRACT

High RRR niobium superconducting rf cavities are fast becoming the basic accelerator structures of a new breed of particle accelerators. Mechanical properties of the high RRR niobium play a critical role in the physical integrity of these structures. The mechanical properties, yield strength, ultimate tensile strength and percent of elongation of Nb are routinely measured at CEBAF in the temperature range 300 to 4.2 K as a quality assurance measure. In this paper the mechanical properties of high purity niobium from Fansteel and Teledyne are presented as a function of temperature between 300 and 4.2 K. As can be expected the yield and tensile properties improve with a decrease in temperature. However, a dip in the elongation versus temperature curve is observed at about 100 K. This seems to be attributable to the presence of interstitial hydrogen. A detailed investigation of the Q-degradation of the Nb cavities also observes a dip in the Q_0 versus temperature curve at about 100 K.

INTRODUCTION

Rf superconductivity has become an important technology in the design and construction of new particle accelerators in nuclear and high energy physics as well as free electron laser applications. Presently large scale projects are under construction in Virginia [CEBAF] and in Switzerland [LEP/CERN] and two major projects in Japan [TRISTAN] and Germany [DESY] are in successful operation. Future applications of the technology in a linear collider are under consideration[1].

In all these applications the material of choice for the superconducting accelerating modules has been niobium because of several reasons: of the elemental superconducting materials, niobium has the highest transition temperature [T_c = 9.2 K] and the highest thermodynamical critical magnetic field [$H_c \approx$ 200 mT]. Both quantities are of importance for accelerator application: T_c determines the wall losses in a cavity made from this material at an operating temperature of 2 K $\leq T \leq$ 4.2 K, and H_c poses the ultimate fundamental limit for the achievable accelerating gradients in such a cavity. In a typical accelerating cavity as used at CEBAF at a frequency of 1497 MHz, the losses in the cavity are reduced by a factor of $\approx 10^5$ compared to a copper cavity at room temperature; these cavities are operated at gradients between 5 MV/m and 15 MV/m, corresponding to

*This work was supported by DOE contract DE-AC05-84ER40150

Advances in Cryogenic Engineering, Vol. 40,
Edited by R.P. Reed *et al.*, Plenum Press, New York, 1994

magnetic surface fields of 22.5 mT and 67.5 mT, respectively, far below the theoretical field limitation as mentioned above. There are several reasons why until now the achievable gradients in these cavities are inferior to the theoretical limits: superconducting surfaces are very sensitive to any kind of surface irregularity in the form of scratches, crevices, cracks or foreign material inclusions or contamination by chemical residue, particulates or dust. Such features cause anomalous losses ("defects"), leading to local thermal instabilities in the material resulting in "quenches" of the superconducting state and to field emission loading under the influence of the electric surface fields, limiting the achievable gradients.

In both cases of limitations significant progress in understanding the mechanisms has been made and techniques have been implemented to push the limitations towards increased fields. Thermal model calculations in conjunction with diagnostic tools like the temperature mapping technique have shown that localized defects can be stabilized by improvement of the thermal conductivity of the niobium material. The application of "clean" processing and assembly techniques such as clean room assembly, ultrapure water and solvent rinsing of the sensitive surfaces as well as high temperature heat treatments under vacuum has contributed to the recent improvements in cavity performances.

Notably the purity of the presently commercially available niobium of RRR-value $\geq$ 250 is of critical importance for reliably achieving gradients of 5 MV/m to 15 MV/m.

Further improvements have been demonstrated by increasing the RRR-values to RRR $\geq$ 600 by high temperature heat treatment in the presence of a solid state gettering material [2].

Such treatments significantly alter the mechanical properties of the material. Superconducting accelerator cavities are subject to a variety of mechanical stresses especially at temperatures below room temperature such as helium pressure excursions during cooldown, tuning stresses, or radiation pressure, and the knowledge of the mechanical properties of the material is important to maintain the physical integrity of these structures.

In the following sections this paper reports about the measurements of mechanical properties such as yield strength, ultimate tensile strength and percentage of elongation in the temperature range of 4.2 K $\leq T \leq$ 300 K carried out on niobium samples of different suppliers and purity levels.

EXPERIMENTAL PROCEDURES

Sample Preparation

For this investigation we used high purity niobium with residual resistivity rations RRR $\geq$ 250 of two suppliers (Teledyne Wah Chang and Fansteel Corp.). Superconducting cavities for accelerators are nowadays manufactured from such material. The material had specified impurity levels of interstitial impurities like H, N, C and O, which to a large extent determine the RRR-value and which are achieved during the manufacturing process by multiple electron beam melting of the ingots in a good vacuum[3]. Further reductions of the interstitial impurities can be made by solid state gettering. During this process the niobium is heated up to temperatures $T \geq 1200^{o}$C in an ultrahigh vacuum in the presence of a material with a higher affinity for these impurities than niobium and kept at this temperature for several hours. Titanium has been found to be an excellent getter material and significant improvements in the thermal conductivity have been realized. Material with such improved thermal properties is highly desirable for rf-cavity application because it significantly reduces the sensitivity of the superconducting surface to thermal instabilities; on the other hand the mechanical stability of the material suffers from both increased purity and grain size, and for the application it is important to know changed mechanical properties.

Several of the samples, which had been machined to a geometry in accordance with ASTM standards (gauge length 3.81 cm, cross section ~ 20.2 mm^2), were post-purified with titanium as a solid state getter material at a temperature of 1400^{o}C for 6 hours in the case of the Teledyne material and at 900^{o}C for 4 hours in the case of the Fansteel niobium. Thermal conductivity measurements on samples prepared under the same conditions indicated improvements up to a thermal conductivity of $\approx$ 240 W/mK for the high-temperature-treated samples.

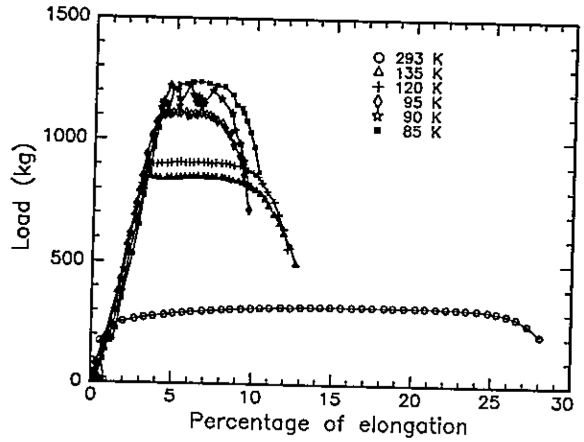

Figure 1(a). **Load-percentage of elongation for high RR Nb**—as received Teledyne Nb.

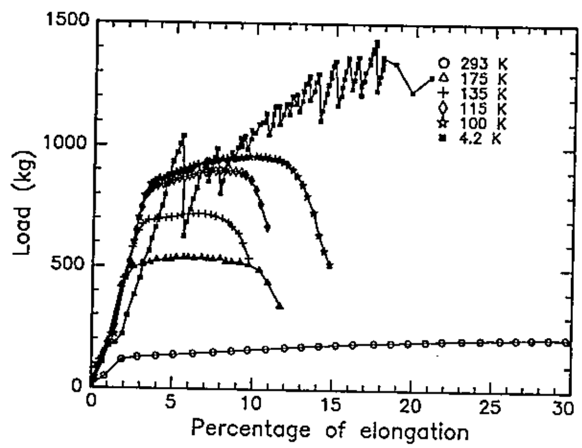

Figure 1(b). **Load-percentage of elongation for high RR Nb**—teledyne Nb heat treated at 1675 K.

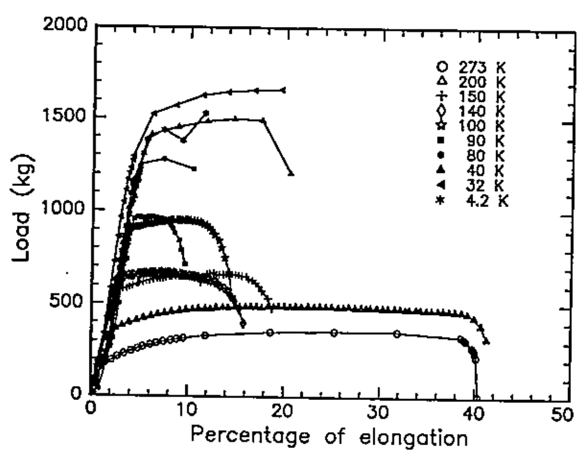

Figure 1(c). **Load-percentage of elongation for high RR Nb**—as received Fansteel Nb.

Sample Testing

Prior to mounting the samples in the test fixture, strain gauges (WK-06-125BB-350 from Micro-Measurements Group Inc.) were attached to each sample with M-Bond 610 adhesive. This additional instrumentation provided independent strain measurements from the displacement module of the specially built tensile test machine (Applied Test Systems, Pa model ATS 900). In addition, the strain gauges gave high resolution measurements. Matched Si diode thermometers were attached to each of the sample grips for measuring the sample temperature. The sample could be maintained at any temperature between 300 to 4.2 K with the help of liquid nitrogen or liquid helium. The average temperature fluctuation of the sample during the test is within 2 K. The cross head speed is maintained at 2.12×10^{-6} m s^{-1}(5.56×10^{-6} m m^{-1} s^{-1}) during these tests.

Results and Discussions

The results of this investigation are summarized in Fig. 1 to 4. In Fig. 1 the load-elongation curves for as-received and heat-treated niobium supplied by Teledyne Wah Chang and for as-received Fansteel material are plotted; Fig. 2 shows the total elongation at break versus temperature for both materials in the as-received and heat-treated conditions. In Fig. 3 the stress-strain curves are plotted and in Fig. 4 the temperature dependence of the yield and ultimate strengths are shown. From these data one can extract the following information:

a) The as-received Fansteel and Teledyne Nb has exhibited serrated yielding at 32 K and 90 K, respectively. After the post-purification at $T = 1675$ K, the Teledyne niobium showed serrated yielding also at 4.2 K with a large number of load drops (see Fig. 1). Serrated yielding has been previously observed in high purity single crystals[4]. The load drops are not due to twinning, and slip traces were observed on the sample surfaces. The observed phenomenon has been explained by thermal instabilities occurring during the plastic deformation process.

b) As can be seen in Fig. 1a, b and c, the percentage of elongation for each set of similar samples goes through a minumum as a function of temperature. This can clearly be seen in the total elongation at break versus temperature plots shown in Fig. 2 for both Fansteel and Teledyne high purity niobium in the as-received and heat-treated conditions. As-received niobium has shown sharp drops in the total elongation at 90 K for Fansteel and 95 K for Teledyne niobium. After heat treatment this minimum in the elongation vs. temperature is broadened and seems to shift to a higher temperature.

This type of behavior has been observed in the past[5] and was attributed to the precipitation of interstitially dissolved hydrogen during the deformation process. A severe effect of hydrogen precipitation on the mechanical properties of niobium has been observed in internal friction measurements between 100 K–200 K[6,7].

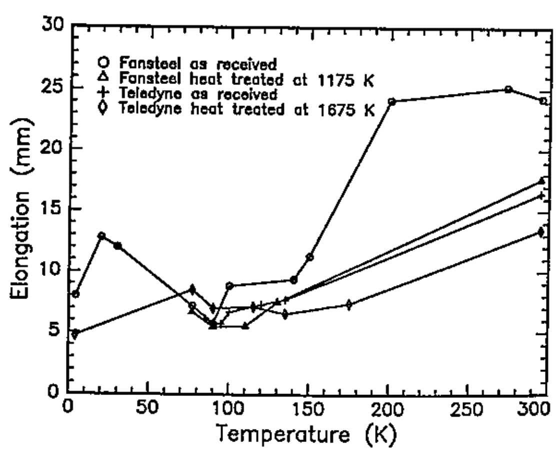

Figure 2. Elongation at break vs. temperature.

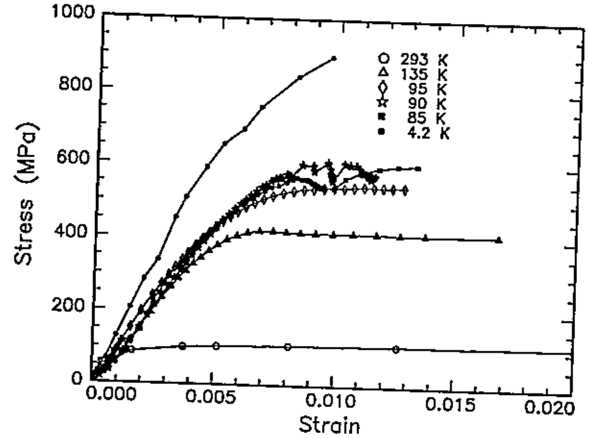

Figure 3(a). **Stress-stain curves at different temperatures**—as received Teledyne Nb.

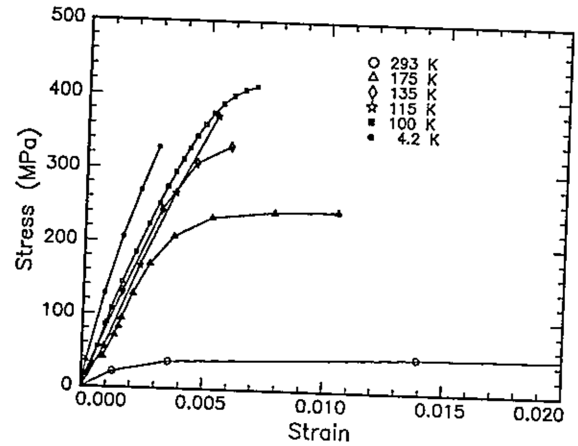

Figure 3(b). **Stress-stain curves at different temperatures**—Teledyne Nb heat treated at 1675 K.

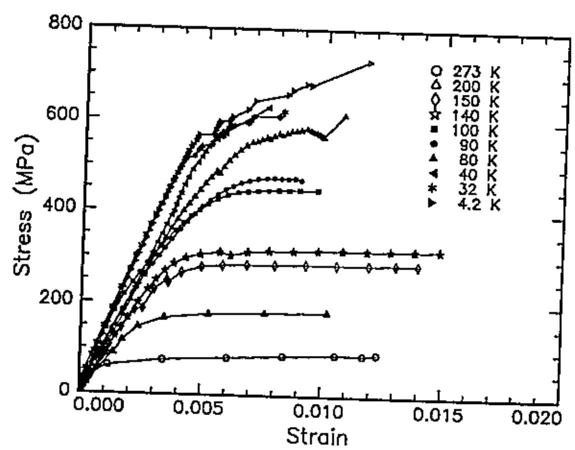

Figure 3(c). **Stress-stain curves at different temperatures**—as received Fansteel Nb.

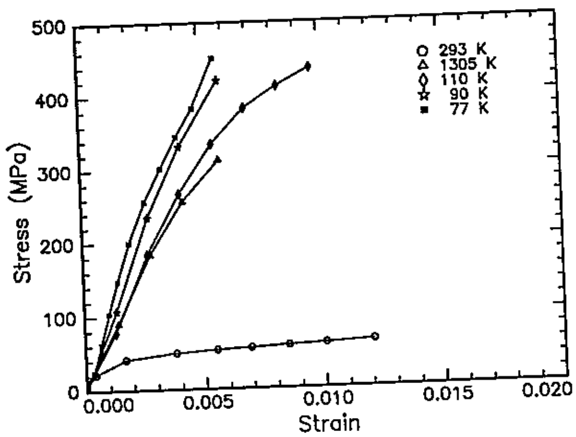

Figure 3(d). Stress-stain curves at different temperatures—Fansteel Nb heat treated at 1175 K.

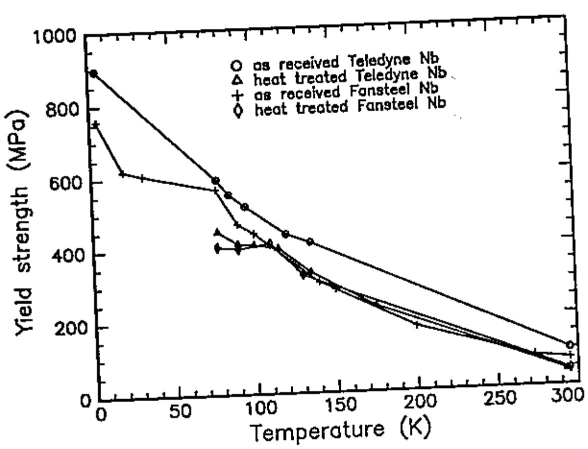

Figure 4(a). Yield and tensile strengths as a function of temperature—Yield strength.

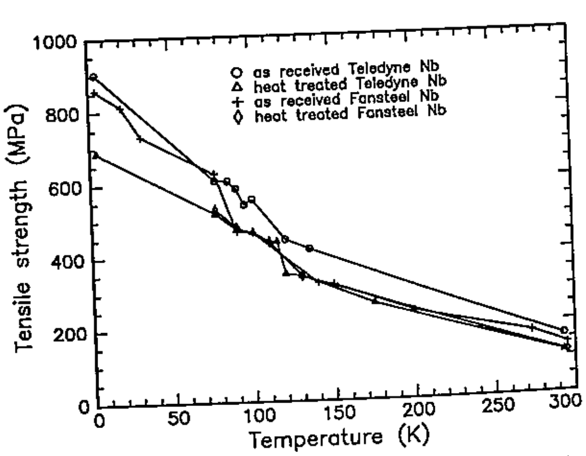

Figure 4(b). Yield and tensile strengths as a function of temperature—tensile strength.

High purity ni
kept for some time
operating helium tem
precipitation of hydr
cavities through the
The measurements of
to indicate that high
from the material. It
treated at different te

c) In some c
observed and the niol

d) The yield
various conditions in
to a factor of 6-7. Th
on the yield and tens
whereas the heat trea
an "athermal" yiel
measurements are n
niobium.

CONCLUSIONS

Thermal stab
purification of high
treatments reduce t
required mechanic
operation, it might t
external stiffening s

Acknowledgement

The authors
to this investigatio
machined. The hel
these measurement

REFERENCES

1. A summary of r
Workshop on
Synchrotron, I
2. P. Kneisel, Use o
superconducti
3. R. A. Bosch, Pro
Beam Meltin
Englewood (1
4. Y. Ano, E. Kura
K and 77 K,
5. S. Gahr, M. L.
behavior at lo
6. G. Cannelli and
niobium, *La*
7. G. Ferron, M. Q
Metallurgica
8. K. Saito and P. K
RRR values,
Jean-Genaz,
9. J. W. Christian
and flow stre
10. P. Kneisel, J. N
conductivity
State of the

High purity niobium rf cavities have shown a significant decrease in Q-value, when kept for some time at temperatures between $75 \text{ K} \leq T \leq 130 \text{ K}$ prior to cooldown to operating helium temperatures[8]. This degradation peaked at ≈ 100 K and was explained by precipitation of hydrogen in the material. It could be avoided by fast cooldown of the cavities through the dangerous temperature region or by heat treatment of the cavities. The measurements of the mechanical properties of the niobium reported in this paper seem to indicate that high temperature heat treatment does not totally eliminate the hydrogen from the material. It is planned to continue with this type of measurements with samples treated at different temperatures for varying durations.

c) In some cases as can be seen in Fig. 3 no well-defined yield point has been observed and the niobium started to plastically deform immediately under stress.

d) The yield (0.2% offset) and ultimate tensile strengths of all materials in the various conditions increased—as previously reported[9,10]—with decreasing temperature up to a factor of 6-7. The heat treatment at 900°C of the Fansteel samples had very little effect on the yield and tensile strengths over the whole temperature range of $4.2 \text{ K} \leq T \leq 300 \text{ K}$, whereas the heat treatment at 1400°C lowered both properties by $\approx 30\%$. In both materials an "athermal" yield behavior[11] seems to occur after the heat treatments; further measurements are needed for confirmation of this effect in polycrystalline high purity niobium.

CONCLUSIONS

Thermal stability of the high performance SRF cavities can be achieved by the post-purification of high RRR Niobium at temperatures above 1475 K. However, such heat treatments reduce the mechanical properties of the material. In order to achieve the required mechanical stability of the cavities under varying load conditions during operation, it might therefore be necessary either to increase the material thickness or to use external stiffening schemes for the structure[12].

Acknowledgements

The authors would like to acknowledge the help of all colleagues who contributed to this investigation. Our special thanks go to L. Turlington for getting the samples machined. The help of Mark Iacobucci in developing the Macintosh-based software for these measurements is highly appreciated.

REFERENCES

1. A summary of rf superconductivity issues and activities is given in the "Proceedings of the 5th Workshop on Rf-Superconductivity," Hamburg 1991, Report DESY M 92-01, Deutsches Elektronen Synchrotron, Hamburg.
2. P. Kneisel, Use of the titanium solid state gettering process for the improvement of the performance of superconducting rf cavities, *Journ. Less-Common Metals*, 139:179 (1988).
3. R. A. Bosch, Production of high RRR niobium discs for SURA/CEBAF, *in:* "Proc. of the Conf. Electron Beam Melting and Refining: State of the Art 1990," R. Bakish ed., Bakish Materials Corporation, Englewood (1990).
4. Y. Ano, E. Kuramoto and K. Kitajima, Orientation dependence of slip in niobium single crystals at 4.2 K and 77 K, *Scripta Met.* 18:201 (1984).
5. S. Gahr, M. L. Grossbeck and H. K. Birnbaum, Hydrogen embrittlememt of Nb I: macroscopic behavior at low temperatures, *Acta Metallurgica* 25:125 (1977).
6. G. Cannelli and L. Verdini, Relaxation effect due to diffusion of interstitial hydrogen in tantalum and niobium, *La Ricerca Sci.* 36:98 (1966).
7. G. Ferron, M. Quintard and P. Mazot, The peak in cold-worked and hydrogen-loaded niobium, *Scripta Metallurgica* 12:6223 (1978).
8. K. Saito and P. Kneisel, Q–degradation in high purity niobium cavities: dependence on temperature and RRR values, in: "Proc of the Third European Accel. Conf." H. Henkle, H. Homeyer and Ch. Petit-Jean-Genaz, eds., Editions Frontiers Cedex (1992).
9. J. W. Christian and B.C. Masters, Low-temperature deformation of body-centered cubic metals I: yield and flow stress measurements, *Proc. Roy. Soc.* A281:223 (1964).
10. P. Kneisel, J. Mammosser, M. G. Rao and K Saito, Superconducting cavities from high thermal conductivity niobium for CEBAF, *in:* "Proc. of the Conf. Electron Beam Melting and Refining-State of the Art 1990," R. Bakish ed; Bakish Materials Corporation, Englewood (1990).

11. M. I. Wood and G. Taylor, Niobium: An athermal plateau in the low-temperature yield stress, *Philosophical Magazine A* 56:329 (1987).
12. A. Matheisen, H. B. Peters and A. Mosnier, Mechanical properties of the 1.3 GHZ TESLA cavity, in: "Proc. of the High Energy Accelerator Conference," J. Rossbach ed., World Scientific, New Jersey (1993).

CRITERION C

OF PLASTIC I

V

In
A
KI

ABSTRACT

The problen
formulation for b
samples at a con
cross-section alo
tional local stres
geometric instabi
compression, and
The results obtai
deformation of v

INTRODUCTIO

During activ
stant rate, the pr
derivative appear
Unstable deforma

1. Serrated defor
and elevated te
2. Transformatio
3. Loss of stabili

Numerous ex
bility and the cha
shape, size, and s
rate, deforming r
loading); the inte
specimens from th
diversity of forms

Advances in Cryogenic E
Edited by R.P. Reed *et a*