



Characterization of Niobium Films and a Bulk Niobium Sample with RRR, SIMS and a SQUID Magnetometer

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If niobium thin film cavities could be shown to have properties similar to bulk niobium cavities, the cost of constructing facilities like the Stanford Linear Accelerator (SLAC) pictured above could be dramatically reduced.

Scientists have discovered that everything in our Universe is comprised of a small number of basic building blocks called elementary particles. Some of these particles are stable and form normal matter, while others are unstable and live for only fractions of a second before decaying. For a few instants after the Big Bang, all of these particles existed together. The extremely high energies that can be achieved in Superconducting Radio Frequency (SRF) particle accelerators have allowed physicists to recreate the environment present at the origin of the Universe. Turning back the clock to the Big Bang provides the theoretical framework to understand the formation of stars, earth, oceans, trees, and most importantly, ourselves!

Currently, all high-gradient accelerators utilize large voltages per meter to speed up particles and have cavities made of ultra-pure bulk niobium (Nb). Niobium is used in cavities because it becomes a superconductor at the highest temperature of any element, thus making it the most inexpensive to cool. The extremely high magnetic fields obtained from superconducting Nb are used to accelerate particles to close to the speed of light and smash them together. Physicists use detectors to monitor these high-energy collisions and can identify particle components or discover new ones, revealing the nature of the sub-atomic interactions between them. The cost of producing accelerator cavities has a major impact on the decision of whether or not to build a new facility. Although bulk cavities can obtain higher energies than thin-films cavities, they are much more expensive, at an approximate cost of fifty million dollars per kilometer (km). Huge sums of money could be saved in a thirty to fifty km long accelerator if thin-films were proven to have qualities comparable to bulk Nb. Accordingly, my research for this summer involved experimentally analyzing Nb thin-films and a bulk Nb sample in an effort to find evidence about the performance of thin-films in a high-gradient accelerator.

Our samples included a bulk Nb sample with a Residual Resistivity Ratio (RRR) of 282 from the Deutsches Elektronen-Synchrotron (DESY) TeV-Energy Superconducting Linear Accelerator (TESLA) group in Germany. The value of RRR is an indication of the purity and the low-temperature thermal resistivity of the Nb. In general, a more pure sample would have a higher value of RRR and would perform better in an SRF cavity. We were also supplied a Nb film on a copper substrate produced at the European Organization for Nuclear Research (CERN) in Switzerland. Our other samples were epitaxial Nb films on single crystal sapphire substrates produced at Cornell by DC magnetron sputtering. Sputtering is the process by which gas ions from plasma are accelerated toward a target of the element (Nb in our case) desired for the film. Material is then detached, or sputtered, from the target and deposited on the substrate.

Our experiment included finding the critical fields of magnetization versus applied magnetic field in the bulk Nb and thin-films. We used a Quantum Design Material Property Measurement System (MPMS) Superconducting Quantum Interference Device (SQUID) magnetometer. Critical fields of magnetization are a good indication of what kind of energies a specimen in an accelerator cavity could withstand. After placing our sample in the instrument, we cooled it down to a superconducting temperature, either 1.90 or 4.20 K, and then applied an external magnetic field. At this time, our sample excluded the external field by producing currents that flow on the surface of a superconductor, called Meissner Currents. These surface currents produce their own magnetic field, which oppose the external change in field (Faraday's Law) and prevent flux from entering the niobium. When the SQUID moved our sample up and down through its coils, the magnetometer was able to detect and measure the area void of field. By continually increasing the external field, we could find the first critical magnetic field, H_{C1} , at which point the Meissner Currents were maximized

and flux started to enter the sample. With the addition of even more field, the sample eventually became completely saturated with flux and ceased being a superconductor. This upper critical field is called H_{C2} . H_{C1} and H_{C2} are shown in Figure 1.

H_{C1} is more directly related to cavity performance than H_{C2} . It is believed that any flux entry into the superconducting cavity wall will lead to excessive heat dissipation resulting in thermal breakdown, thus setting the effective maximum attainable accelerating gradient of the cavity. Therefore, by knowing the magnetic field (Oe) value of H_{C1} , we can find the proportional value for the maximum accelerating gradient in MeV/m: the higher H_{C1} , the larger the gradient, and consequently the higher achievable energy in the accelerator. Please refer to Figure 4 for all our measured values. The value of H_{C1} for pure Nb is published, but the value for the DESY sample was measured on site. Such close agreement indicates that our techniques are correct. Our relatively high value of H_{C1} for Film 30 could be from a surface barrier effect, which causes the lines of magnetic flux to pile up and prevents them from entering the film. Although highly unlikely, discovering how to alter a thin-film in such a way as to increase the lower critical field, H_{C1} , would be a significant step in allowing Nb thin-films to replace expensive bulk Nb in future accelerators.

We used Secondary Ion Mass Spectrometry (SIMS) to obtain the element versus depth profiles for our films. The SIMS process involved an ion microprobe shooting a beam of positive cesium ions into our film, essentially boring a hole. Secondary anions from the hole were monitored and recorded. By analyzing the data from SIMS, we were able to obtain vital information about the ratios of oxygen to niobium and carbon to niobium in our films. In addition, SIMS enabled us to determine the level of impurity on the surface and at the film-substrate boundary. A SIMS plot for the CERN film is shown in Figure 2.

The amount of oxygen and carbon in our films dramatically affected the observed value of RRR, which ranges from 2080 for pure single crystals to 11 for polycrystalline specimens. For example, a significant amount of oxygen and carbon were introduced into Film 29 by annealing in a furnace with a poor vacuum. This resulted in a RRR value of 4. Film 30, on the other hand, with very little oxygen and purity close to the DESY sample, had a RRR of 65. In general, any film made with a RRR value greater than 60 could be considered "good."

We used the Desert Cryogenics Full Wafer Probe Station to find RRR and the critical temperature, T_C , of our films. T_C is the temperature at which a material transitions to a superconductor. A lock-in amplifier sent a current through our film while we measured the voltage. Gold contacts were sputtered at the corners of the films prior to the measurement. For example, to find T_C we simply watched for the voltage reading to drop to zero. At this point the resistance must also be zero, since $V = IR$, and the specimen starts superconducting. Our temperature scan for Film 30 is shown in Figure 3. The fact that this film shifts from normal to superconducting in less than 0.2 K indicates its quality.

Magnetic Moment vs. Magnetic Field in DESY Sample with Temperature Constant @ 4.20 K

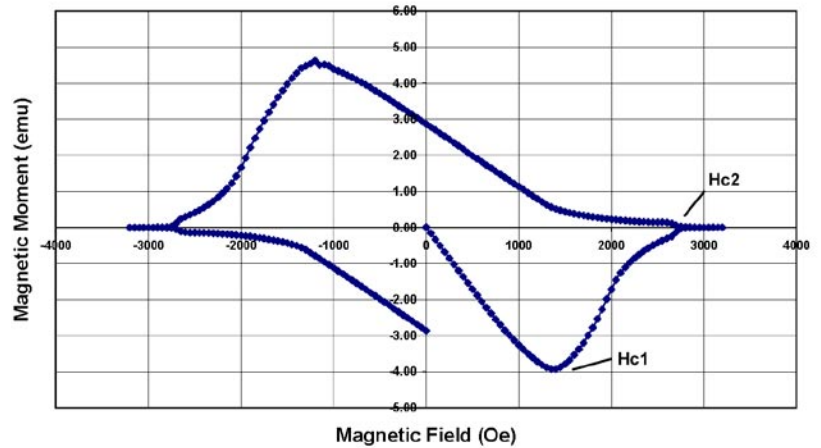


Figure 1.

CERN (200 nA) Intensity vs. Depth

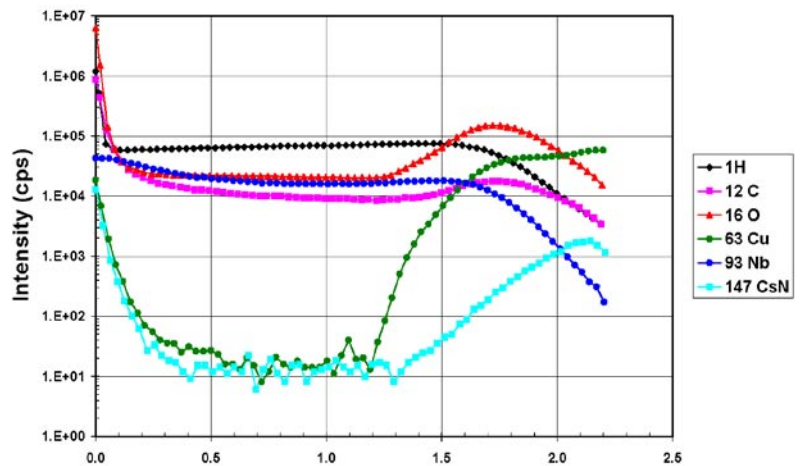


Figure 2.

Selected Voltage vs. Temperature Around T_C in Film 30

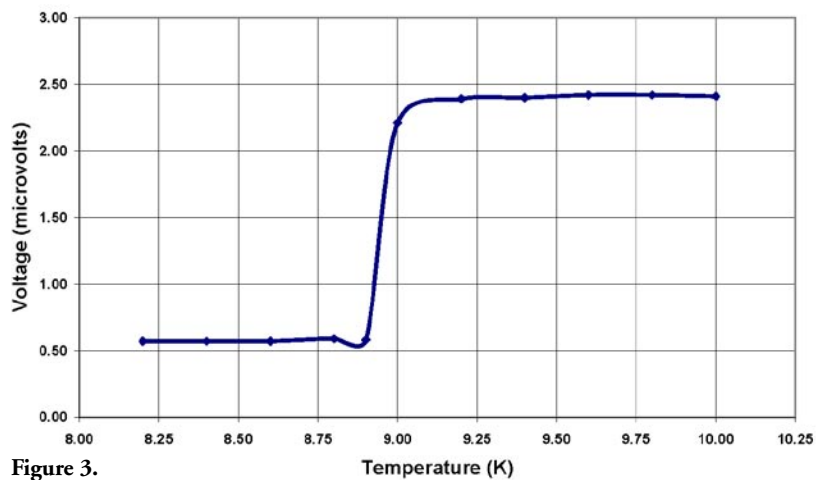


Figure 3.

Sample	Ultra Pure Nb	DESY	CERN	Film 19	Film 29	Film 30
Area (cm ²)	-	0.309	0.224	0.336	0.36	0.513
Thickness (cm)						
RRR	-	0.114	1.50x10 ⁻⁴ (nom.)*	-	1.22x10 ⁻⁴	3.29x10 ⁻⁴
SIMS	-	0.114	3.357x10 ⁻⁵	1.75x10 ⁻⁴	9.50x10 ⁻⁵	2.35x10 ⁻⁴
Volume (cm ³)	-	0.0353	3.357x10 ⁻⁵	5.887x10 ⁻⁵	3.910x10 ⁻⁵	1.688x10 ⁻⁴
-dm/dH (emu/Oe)						
1.90 K	-	3.388x10 ⁻³	3.194x10 ⁻⁵	1.118x10 ⁻⁵	-	1.569x10 ⁻⁵
4.20 K	-	3.405x10 ⁻³	3.093x10 ⁻⁵	1.474x10 ⁻⁵	-	-
{-4 /V} x {dm/dH}	-					
1.90 K	-	1.206	1.257	2.386	-	1.168
4.20 K	-	1.212	1.158	3.1476	-	-
RRR	1600±400	282 [†]	11.5±0.1*	-	4.10	65.3
T _c (K)	9.26	9.26	9.50±0.02*	9.10±0.10	8.40±0.08	9.20±0.05
H _{C1} @ 1.90 K (Oe)	1,676	1653±70	1471±60	1454±80	-	1946±50
H _C @ 1.90 K (Oe)	1,909	1621±300	1926±200	2146±260	-	-
H _{C2} @ 1.90 K (Oe)	3,677	3940±100	10615±230**	8805±120	10740±500	7170±100

* These values are published values from CERN- C. Benvenuti et al, Physica C 351 (2001) 429-437.

** This value is scaled to 1.90 K from the published value at 0 K, our measurement at 1.90 K is 9700 ± 800 Oe.

ψ All values in this column are published in D. K. Finnemore et al, Phys. Rev. 149 (1966) 231-243.

† Measured at DESY.

Figure 4. Cornell Center for Materials Research (CCMR) Data Collected on Niobium, summer 2003.

Our conclusions included that sputtered niobium films do not have the same superconducting properties as bulk niobium of similar purity. Please see the Figure 4 for our data. Although some superconducting properties, such as H_{C1} and T_c, are similar, others are not. We believe the higher density of dislocations (defects) in our films is a prime suspect for creating the higher H_{C2} values seen in the table. We hypothesize that high temperature annealing is necessary to make films with the same properties as the bulk niobium. This summer's research supports our belief that films can be made to perform under high-gradient conditions. Considering the huge amount of money that would be saved with film cavities, continued research in niobium films is worthwhile.

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1. Finnemore, D.K., et al. 1966. Superconducting Properties of High-Purity Niobium. Physical Review, 149: 231-243.

2. Benvenuti, C., et al. 2001. Study of the residual surface resistance of niobium films at 1.5 GHz. Physica C, 351: 429-43.

Jason Thompson is currently a senior pursuing a B.S. in Mechanical Engineering. He plans to attend graduate school for the same discipline. Jason is chair of the University of Rochester American Society of Mechanical Engineers (ASME) student section, and likes following the New York Yankees and weight training in his spare time.