Contribution of Oxide Layers to the Residual Resistance of Niobium

F. Palmer
Laboratory of Nuclear Studies
Cornell University
Ithaca N.Y. 14853

The oxide layer has been thought to contribute to the residual resistance of Niobium for a long time.[1] In 1963,De Sorbo[2] observed that dissolved oxygen decreased the critical temperature of Niobium by about 1° per atomic percent. In 1975, the Stanford group[3] studied reentrant cavities whice had been fired at 1800°C and then tested without exposure to air. They observed that the quality factors, Q_o, of these cavities degraded from about 10^{10} to 10^9 if the cavity vacuum was allowed to degrade. In 1981, a group at Bell Labs[4] improved the loss characteristics of Nb tunnel junctions by reducing the Niobium Oxide layer with Aluminum.

There is a vast literature on the behavior of Oxygen in or on Niobium. Only a few details can be mentioned here. Surface oxides are always absorbed into the bulk at temperatures of 1600°C or more; however, if the dissolved Oxygen in the bulk exceeds 1% atomic, oxides will precipitate to the surface as the sample cools. In 1972, the Brookhaven group[5], using Auger spectroscopy, observed that less than one monolayer of Oxygen precipitated to the surface of a sample containing 0.12 atomic% Oxygen. (This concentration corresponds to a residual resistance ratio, RRR, of 27.) We have done a similar Auger study on 0.5mm thick samples with RRR of 150 and 1000. These samples precipitate about 1/3 and 1/10 of a monolayer respectively when cooled down from firing at 1050°C. (The sample with RRR=150 developed a few monolayers of sulfur upon heating, but this was permanently removed by heating to 1400°C.)

Niobium with low bulk Oxygen concentration has usually been obtained by UHV firing at temperatures greater than 1900°C, but the availability of large amounts of very pure Niobium seems to have made this step unnecessary.

In the present syudy, x-band elliptical cavities are drawn from 0.5mm Nb. and fired at 1100°C for 1 hour by direct resistance heating in a furnace with separate vacuum systems for the interior and exterior of the cavity. A single RF probe with external Q of about 10^{10} is attached to the cavity before firing. After firing, the cavity is sealed off from its vacuum pump with an all-metal valve, and tested in a dewar. The RF system pulses the cavity with about 1 watt for 1 msec at a frequency near the cavity resonance. The decay curve is then observed with a sensitive amplifier which was protected during the pulse. This technique allows measurement of Q_o values less than 10^7. Higher Q_o values are measured in the conventional manner.

Thus far, one cavity (RRR = 100) has been fired and tested 3 times. The second 2 firings produced some useful data. Both of these firings yielded Q_o's of at least 10^{10} (surface resistance = 25 n ohm). Preparation with chem-polish and solvent rinses yielded no more than $4X10^9$. After the third test, the cavity was exposed to 0.1 torr of O_2 for 1 hour. This degraded the Q_o to $5X10^9$. These data are sufficient to prejudice the author to believe that oxide layers can contribute on the order of 10^{-8}ohms to residual resistance, but this study is not complete.

Refrences

1. Arguments pro and con are reviewed by: Giordano, Hahn, Halama, & Varmazis; J. Appl. Phys. 44, p 4185, 1973.

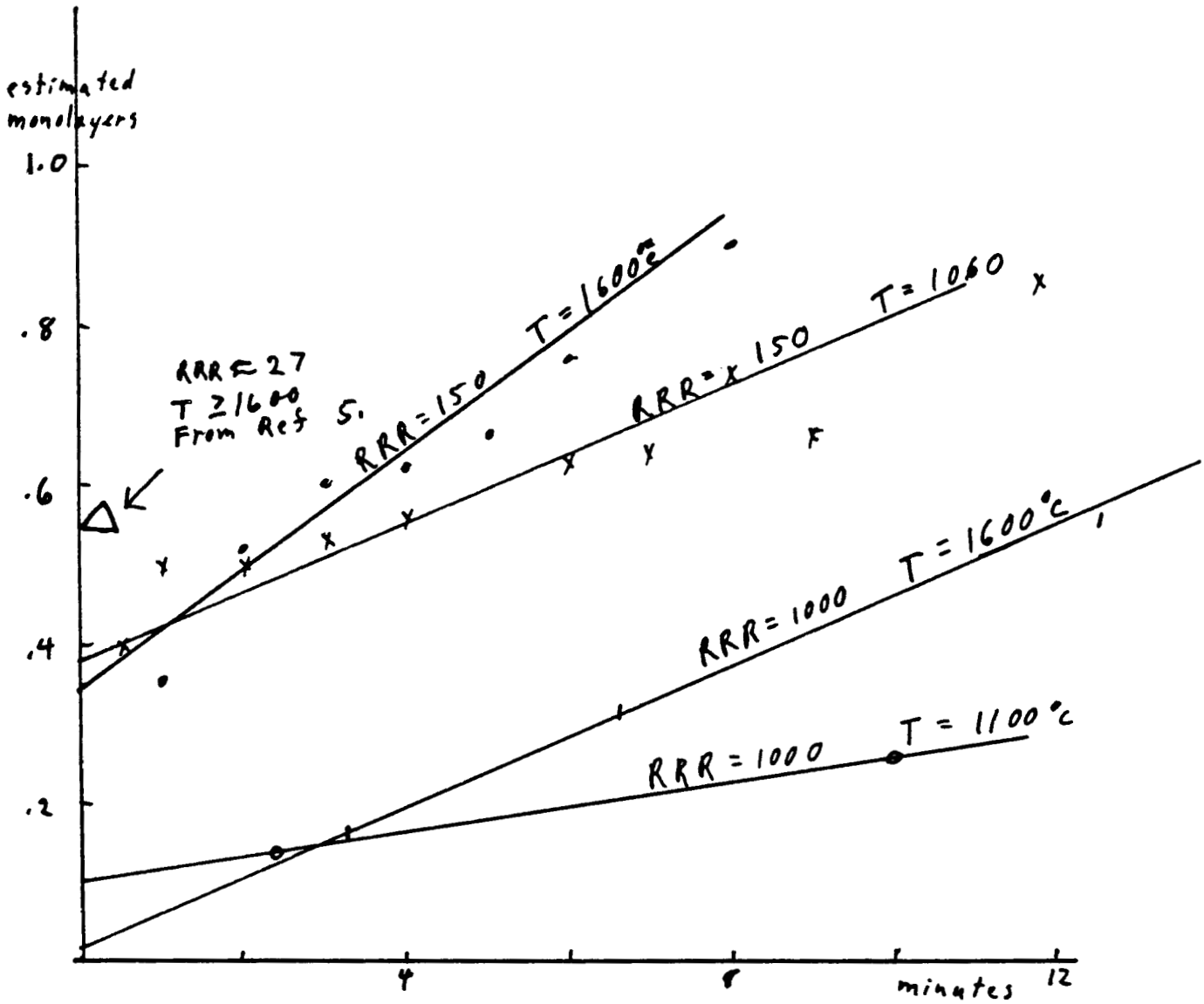
2. De Sorbo; Phys. Rev. 132, p 107, 1963.

3. Cepperley, Ben Zvi, Glavish, Hanna; IEEE Trans Nuc Sci NS-22, p 1153, 1975.

4. Rowell, Gurvitch, and Geerk; Phys. Rev. B 24, p2278, 1981.

5. Farrell, Isaacs, and Strongin; Surface Science 38, p 31, 1973.

fig 1



Oxygen coverage on the surface of Niobium as a function of time for materials of various purity after cooldown from various temperatures. Gradual increase is probably due to ambient CO. Monolayers were calibrated by exposing a clean sample to 10^{-8} torr of O_2