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A SIMPLE TECHNIQUE FOR FABRICATION OF TUNNEL JUNCTIONS USING Nb WIRE

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Résumé. — Nous décrivons une technique simple pour faire des jonctions tunnel à base de niobium. Cette base est constituée par l'extrémité polie d'un fil de niobium. L'utilisation d'un bombardement ionique après le polissage mécanique permet d'obtenir des caractéristiques $I(V)$ reproductibles. La qualité des jonctions, déterminée par le courant de fuite à basse tension, est comparable à celle des jonctions obtenues avec des films de niobium déposés par pulvérisation RF.

Abstract. — A simple technique is described for making niobium based superconductive tunnel junctions using, instead of a deposited niobium film, the polished end of a niobium wire. By use of ion bombardment after mechanical polishing volt-ampere characteristics of reproducible shape are produced. Junction quality, as determined by excess current for low voltage, approaches that of junctions made with sputtered Nb films. Josephson junctions made with this technique have large critical currents although the magnetic field dependence of these currents suggest somewhat nonuniform tunneling barriers.

1. Introduction. — Because of its high transition temperature, good mechanical properties, and its relatively non-porous and stable oxide, niobium is a very desirable base material for superconductive tunneling devices. There are, however, some well known problems with the material which have hindered its application in such structures. First of all it is difficult to produce niobium thin films with high values of T_c . Successful evaporation is normally accomplished only with a high power electron beam gun in very good vacuum and with a heated substrate. Sputtering of Nb is also not trivial, requiring various combinations of substrate heating, substrate bias, and the gettering action of the surrounding deposited material [1]. Even after a successful deposition technique is attained small variations in procedure or deterioration of equipment can drastically reduce T_c . This reduction is presumably due to disorder in the film [2] or due to absorbed gases [3].

The second problem with niobium is that mere production of high T_c film is not sufficient to insure that junctions with good repeatable tunneling characteristics can be obtained. Excess currents in S-I-S junctions using Nb as the base material are very commonly seen and in fact one never obtains the near perfect tunneling volt-ampere curves exhibited by materials such as Sn or Al. Fairly repeatable results are possible with DC getter sputtered films [4] and a further stabilization of the characteristics can be

obtained on RF sputtered films by sputter etching the Nb before oxidation.

Although tunneling to bulk niobium has been done successfully for materials study purposes [5], [6], there appear to have been no attempts to use bulk niobium for devices except for point contact devices. To produce tunnel junctions, very careful surface preparation has been necessary. Usually this involves a very high temperature outgassing in high vacuum.

The present paper describes a simple technique for making Nb based junctions by using the polished end of a Nb wire instead of a Nb film. Repeatable characteristics are obtained, not by thermal outgassing, but by the much less difficult sputter etching steps previously used on films. The process allows rapid fabrication of many devices with the use of a minimum of specialized equipment. It is necessary to use an RF sputtering system or ion gun, a simple vacuum evaporator for Pb and some mechanical polishing equipment.

2. Fabrication technique. — The structure described here was designed for insertion into a microwave cavity in such a way that only the junction surface would be within the cavity, all connecting leads leaving the back of the structure. This particular device shape is not of crucial importance to the fabrication technique but it is potentially useful for other application as well.

The basic structure is shown in figure 1. It consists of a niobium wire supported by insulating material inside a small copper or brass cylinder. A tunnel junction is formed between the end of that wire and a Pb film deposited on the top surface. To insure good 4 terminal measurements of tunneling characteristics an outer copper cylinder was added to provide two separate connections to the Pb film. Variations on this structure include the use of any number of Nb wires to allow making more than one device. Also copper wire can be used to make connection to the Pb film rather than using two copper cylinders.

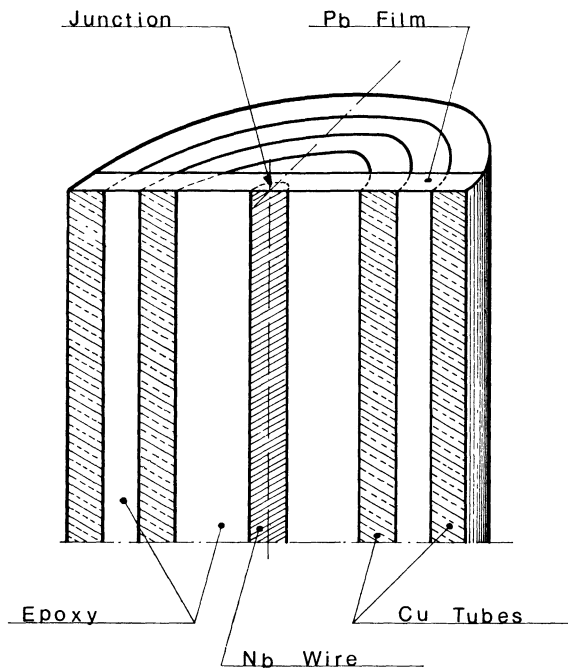


FIG. 1. — Niobium wire junction structure.

The details of fabrication are as follows :

- 1) Copper wires are soldered to the bottoms of the two cylinders.
- 2) The smaller cylinder is placed inside the larger cylinder and the space between is filled with epoxy [7].
- 3) After the epoxy has cured, nylon insulated Nb wires [8] are inserted through the inner cylinder. They are then cemented in place with epoxy.
- 4) The wires are cut flush with the cylinder surface and the structure is mechanically polished. The final polishing grit should be less than a few microns in diameter.
- 5) The polished structure is attached to the cathode of a RF sputtering system [9]. It is then sputter-etched with a power density of about two watts per square centimeter for a considerable period of time (at least 20 min). Even with a water cooled cathode there may be some problem with overheating the epoxy or nylon and it may be necessary to sputter for only 2 or 3 min at a time letting the device cool periodically.

6) The sample is oxidized in wet oxygen for times between 2 and 5 min and at temperatures between 120 °C and 150 °C. This step can be done outside of the vacuum system.

7) A top layer of Pb is deposited ($\sim 5\,000\text{ Å}$) on the surface. The Pb evaporation is not a critical step but is acceptable if performed in a vacuum of 5×10^{-7} torr or less.

8) A protective layer of SiO or photoresist is desirable to prevent eventual deterioration of the Pb film.

3. Result, volt-ampere characteristics. — It was found that the most critical fabrication step is the sputter-etch step. Without sputter etching the resulting volt-ampere curves are completely unpredictable. An inadequate sputter etch can produce a fairly repeatable curve which exhibits, however, superconducting shorts, probably microshorts. Figure 2 shows volt-ampere curves for 5 junctions made together in one copper tube. This sample had been sputter etched for 5 min.

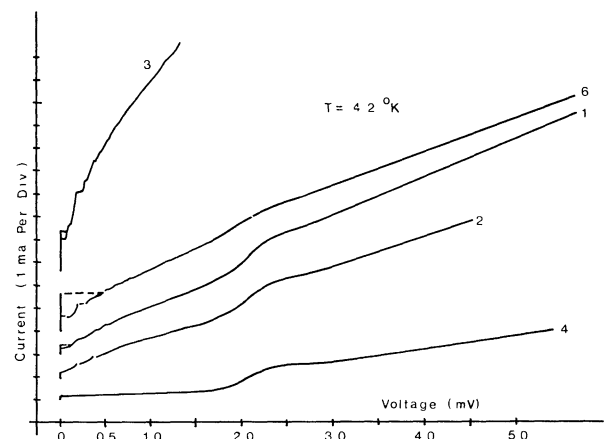


FIG. 2. — Current-voltage characteristics for 5 Nb wire junctions on the same substrate, measured after first etch (5 min, 100 W).

Figure 3 shows volt-ampere curves for the same devices after the original Pb layer was removed by sputter etching (5 min), the etch was continued for another 2.5 min, the sample was reoxidized, and a new Pb film was deposited. In this second case the current at zero voltage is at least 90 to 98 % Josephson current and not due to shorting. This was determined by observing that magnetic fields of a few gauss reduce the critical current by at least that percentage. The volt-ampere characteristics deviate from theoretical S-I-S characteristics in two ways. First of all there is an excess current for voltages less than the sum of the energy gaps $\Delta\text{Nb} + \Delta\text{Pb}$. This produces a rounding of the curve. Secondly there is a negative resistance region in the vicinity of $\Delta\text{Nb} + \Delta\text{Pb}$. Both anomalies are commonly seen in Nb junctions qualitative explanations involving proximity effect and gaplessness having been offered [10], [11].

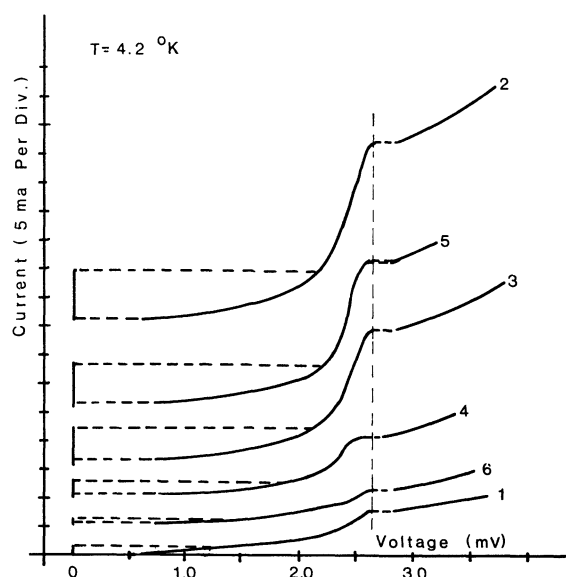


FIG. 3. — Current-voltage characteristics for 6 Nb wire junctions. Wires are identical to those of figure 2 but new junctions were formed after second etch (7.5 min, 50 W).

In order to have some quantitative comparison between devices the current and voltage values I_1 , V_1 , I_2 and V_2 , as defined by figure 4, were used. In good

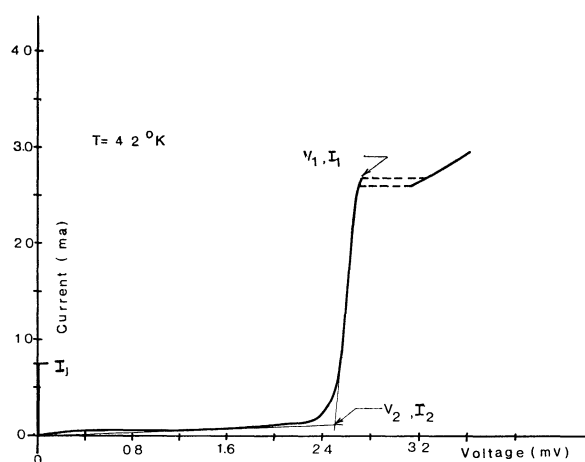


FIG. 4. — Josephson junction of Nb wire-Pb film. The best characteristic (24 min, 50 W etch).

Nb-Pb tunnel junctions made with sputtered and sputter etched Nb *films*, the value for V_1 is normally between 2.58 and 2.65 mV. The value for V_1 is near 2.2 mV and the ratio I_1/I_2 is about 15 at 4.2 K. The best Nb wire junction is shown in figure 4 and, as can be seen it has a higher value of V_2 and a leakage at least as small as the best film devices. The ratio of maximum Josephson current to I_1 is 0.33. This is also very comparable to thin film junctions.

Table I is a comparison of wire junctions which have been tested. Four observations appear to be important.

1) A ratio of I_1/I_2 between 5 and 8 is easily obtained with sufficient sputter etching time and power.

2) A higher ratio than 8 was obtained using reduced power and a long sputtering time. However repeatability would appear to be a problem with low power levels.

3) The values of V_1 and V_2 both depend upon sputtering conditions. Again the higher values are obtained with the lower sputtering powers.

4) There is a definite ageing or cycling effect on device resistance. The resistance is usually greater when tested the second time. There is much less effect on junction quality.

Observations 1 and 2 point to the need for additional experimentation with sputtering time and power. A certain balance must be obtained between the cleaning action of the discharge and its tendency to disorder the surface layer of the niobium as well as to causing embedding of argon atom. Observation 3 might indicate that indeed the surface is superconducting only via the proximity effect. However an explanation of the variation of V_1 would not seem possible with existing theories such as that of Mc Millan [12]. Observation 4 is consistent with similar behavior of Nb-Pb thin film junctions [13].

4. The Josephson effect. — Although the quasi-particle tunneling characteristics of Nb based tunnel junctions are not ideal, Schwidtal [10] has shown that nearly ideal Josephson tunneling behavior is possible. As is evident from figure 2, our wire junctions exhibit

TABLE I

Sample	I_1 (mA)	I_1/I_2	V_1 (mV)	V_2 (mV)	Sputter power and time	Comments
A (8 junctions)	0.35- 7.0	1.2- 4.0	2.6		10 min, 50 W	
B 1 (6 junctions)	3 -15	1.2- 4.1	2.6	1.8	5 min, 100 W	
B 2 (3 junctions)	44 -57	6.8- 8.1	2.6	2.22-2.26	7.5 min, 50 W	5 min to remove Pb retest after 8 days
B 2 (6 junctions)	7 -32	4.7- 6.4	2.55-2.67	2.17-2.27		
B 3 (4 junctions)	20 -50	5.5- 7	2.6 -2.42	2.15-2.18	8 min, 100 W	
B 3 (3 junctions)	1.25- 7	5 - 6.7	2.6 -2.68	2.1 -2.2		retest after 1 day
B 4 (5 junctions)	0.49- 0.95	5 - 8	2.72-2.75	2.55-2.6	4 min, 100 W	to remove Pb
					20 min, 50 W	additional
C 1 (3 junctions)					9 min, 50 W	Shorting behavior
C 2 (3 junctions)	2.2 - 4.4	1.6-18	2.71-2.73	2.5 -2.55	20 min, 50 W	5 min to remove Pb

large critical currents which are quite repeatable from junction to junction. The ratio of critical current I_j to I_1 is typically larger than 0.3 for junctions with one ohm or less resistance. This compares to values of about 0.5 or less obtained by Schwidtal for 2 thin film Nb-Pb junctions.

The dependence of I_j on magnetic field is however far from perfect in these wire junctions. The theoretical dependence for cylindrical junctions is given by [14]

$$I_j = 2 I_0 \frac{J_1(\pi\phi/\phi_0)}{(\pi\phi/\phi_0)}$$

where J_1 is a Bessel function of the first kind, ϕ is the total magnetic flux in the plane of the junction and ϕ_0 is the flux quantum. A comparison between this equation and one of the better experimental curves is shown in figure 5. An accurate measure of the magnetic

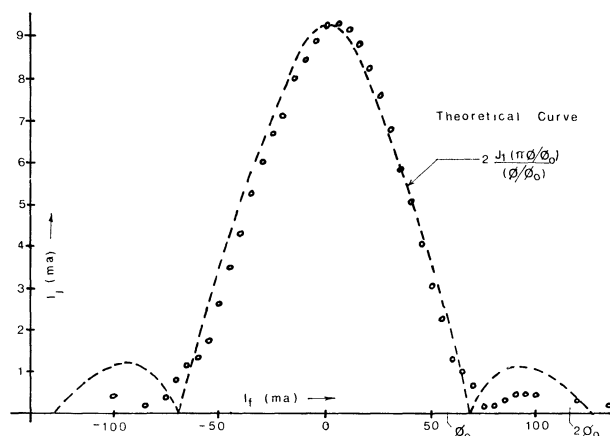


FIG. 5. — Maximum Josephson current versus current in solenoid. Field is parallel to junction surface. Sample B 2-2.

field inside this junction could not be made because it was discovered after the measurement that the device was enclosed in a superconducting loop. The applied field was about three gauss for the value ϕ_0 shown on the figure.

All devices were sensitive to magnetic fields of the order of one gauss but most exhibited unrepeatable behavior presumably associated with flux trapping. This is not surprising because of the obviously non-planar junction surface.

5. Conclusion. — A technique is described for making superconducting tunnel junctions using the polished ends of niobium wire. Reproducible characteristics can be obtained by making use of RF sputter etching of the wire before oxidation and deposition of a counterelectrode. Although the characteristics are quite comparable to many published volt-ampere curves of Nb based junctions the etch parameters are still to be optimized to produce very low leakage junctions in a consistent manner.

Because of the ease of fabrication of one or many junctions in one structure the technique may be useful for practical devices. Certainly it is a convenient structure to study the Nb surface itself and the negative resistance region near Δ Nb + Δ Pb. Josephson devices can be fabricated, however a smooth critical current dependence on magnetic field probably requires a more refined polishing technique than presently used. Coupling to high frequency fields and switching applications both require small area junctions. The present 0.125 mm diameter circular junction could be reduced by masking or perhaps more simply by electrolytically etching the wire.

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