

A POLISHING PROCEDURE FOR HIGH SURFACE ELECTRIC FIELDS IN SUPERCONDUCTING LEAD RESONATORS*

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Low Temperature Physics, 63-37
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ABSTRACT

A chemical polishing procedure has been developed which allows fields of 20-30 MV/m to be routinely achieved at the surfaces of superconducting resonators constructed of lead-plated copper. This has made possible the construction of lead plated resonators with accelerating fields equivalent to those achieved with niobium, since the performance of these resonators is limited by electric field breakdown.^{1,2} The polish itself is based on a weak acidic solution of hydrogen peroxide and EDTA, a chelating agent. Other polishes have not proven suitable for the thin (5-10 μ) lead layer because of their very high polishing rates. The present procedure, however, gives good polishing action even at rates as slow as 5 μ /min. Details of the procedure as well as comparative electric field breakdown data are presented.

INTRODUCTION

Procedures for the treatment and preparation of superconducting Lead surfaces have been developed which now provide a sophisticated technology comparable to that which has been developed for processing Niobium by other groups. Procedures have been investigated for anodizing, cleaning, polishing and passivating very thin electroplated Lead surfaces in an attempt to go beyond the previously accepted procedure which involved simply electroplating a thin Lead layer onto a copper substrate, and then trying to preserve the surface of that layer against contamination or oxidation during drying and assembly. That is, instead of trying to create an acceptable Lead surface during the plating process, we felt it would be advantageous to make the kind of surface which we wanted from Lead which had already been electroplated. A variety of procedures have been developed, and a series of them were found which were compatible with each other and which combined to form a surface with excellent superconducting properties.

The most important of these individual procedures is a chemical polish for Lead surfaces which is effective at very low concentrations and so works at a slow enough rate to give good control over the polishing of very thin Lead layers. Furthermore, the polish is effective on rounding the sharp corners of the polycrystalline Lead which are formed by electroplating onto OFHC copper. Before polishing, such a surface appears "white" when illuminated by an intense light beam irrespective of the angle from which it is viewed (diffuse scattering), thus indicating a surface which is very rough on a microscopic scale. After polishing, the surface appears "black" when viewed more than 45° away from the specularly reflected light beam, thus indicating that the maximum angle at the surface is less than 22.5°. Since the surface angularity is directly related to electric and magnetic surface field enhancement, and since a microscopic breakdown area is sufficient to limit the maximum field obtainable in a large cavity, such a polished surface should show superior high field properties in superconducting applications. This has proven to be the case, most especially in the case of breakdown induced by electric field emission. In tests of polished cavities, we have found that field emission induced losses were dramatically reduced both

in small sample test resonators and in cavities designed for use in a heavy ion accelerator. The results compare favorably with those achieved with Niobium resonators by other laboratories.

PROCEDURE

Lead surfaces are deposited onto electropolished copper substrates using standard commercial fluoborate plating processes and chemical solutions.¹ Surfaces are plated to a thickness of 5 to 10 μ at a plating current density of .010A/in² after initially "flashing" on a thin layer (<.1 μ) at a current density of 0.2A/in². This very slow plating gives excellent uniformity without complicated electrode shapes.

After plating, the resonator is rinsed with water, dried, and the polishing and rinsing baths are prepared. Oxidation of the surface at this point is not critical since the surface layer of the Lead is removed during polishing. Speed is, however, essential to the polishing and rinsing process. The entire procedure usually takes between 30 sec and 1 minute to accomplish. The other essential characteristic of the process is that liquid flow patterns must never wash from "dirty" areas of the resonator to "clean" areas. This has been accomplished in either of two ways: parts can be immersed in baths of solutions if they have no cracks, crevasses or screw holes exposed to the liquid; or, if screw holes are required, they are arranged to be on the "back" side or "outside" of a cylindrically symmetrical part. In this second way, the part is mounted on a spinner and rotated about its axis at 180-600 rpm while a continuous flow of solution is applied to its surface. Centrifugal force then insures that the direction of flow away from the central "clean" area. However, vigorous immersion of a relatively small part in large containers of solution gives the best results and is used where possible.

Each solution is applied in turn for the indicated time. Water rinses are not used between the various stages. Drying is accomplished in every case by mounting the part in the spinner and blowing dry with a large volume flow of Nitrogen gas. In this way, surfaces can be dried in one or two seconds.

The detailed processing sequence is as follows:

1. Electropolish copper part (20 sec. at 10v potential).
2. Water rinse thoroughly.
3. Soak in dilute citric acid (1%) to keep copper bright.
4. Water rinse.
5. Electroplate lead (~ 3 hours).
6. Water rinse - mount on spinner while wet.
7. Chelate (30 sec.).
8. Ammonia (10 sec.).
9. Acetone (10 sec.).
10. Blow dry

Prepare New Polish Solution

11. Polish (7-1/2 sec., polish applied to dry surface).
12. Chelate (10 sec., strong flow to stop polish).
13. Ammonia (6 sec.).
14. Acetone (8 sec.).
15. Blow dry.

*Work supported by NSF Grant No. PHY76-20406.

If more polishing is desired, or if any streaking has appeared, steps 11 through 15 can be repeated as often as desired. If an immersion process is used, the part is agitated into and out of each bath several times.

Solutions

The solutions used in the polishing and rinsing process were made as dilute as was possible without impairing their effectiveness. If difficulties are encountered, they can be prepared with 2 or 4 times the concentrations below without changing the basic nature of what they do. Deionized water with a volume resistivity greater than $2M\Omega\text{-cm}$ was used.

Polish:

100 cc acid mixture (see below)
150 cc saturated disodium EDTA solution
100 cc 30% H_2O_2 solution
dilute to 8ℓ with water

Acid Mixture:

100 cc glacial acetic acid
200 cc nitric acid, 70% concentration
dilute to 600 cc with water

Chelate:

100 cc saturated disodium EDTA solution
1500 cc water

Ammonia:

reagent grade ammonia diluted to 7.5% concentration

Acetone:

reagent grade

The polish solution itself has a shelf life of only one or two hours, other solutions can be mixed at any time. The EDTA solution is prepared by stirring disodium EDTA crystals² in the water for several hours, then removing excess crystals and adding enough additional water to give a clear solution.

EXPERIMENTAL RESULTS

Field emission measurements were done in the post-loaded resonator shown in Fig. 1. Electric fields are greatly enhanced in the small gap between the post and the top plate. Since the gap is very small ($\sim 0.02\text{cm}$) and since there was some variability in the thickness of the indium seals from run to run, the actual value of the gap spacing, and thus the field enhancement, was determined for each run from the measured eigenfrequency of the cavity. This, together with the measured energy in the cavity allowed an absolute calibration of the electric field in the gap.

Results obtained on polished and unpolished lead, as well as a selection of results obtained by other laboratories, are shown in Fig. 2. The Fowler-Nordheim plot gives straight lines for conventional field emission losses. As a criterion of utility in superconducting accelerator applications, a curve corresponding to resonator losses of 0.1Watt/cm^3 has also been plotted. Curve 2 was obtained for lead by the group at Karlsruhe;³ Curve 3 represents losses measured in an actual accelerating structure by Argonne;⁴ Curves 4 and 5 were similarly obtained at Stanford.⁵ In all cases, the lowest losses reported have been shown. Finally, a single point showing results from an accelerating structure using polished lead surfaces⁶ is also shown.

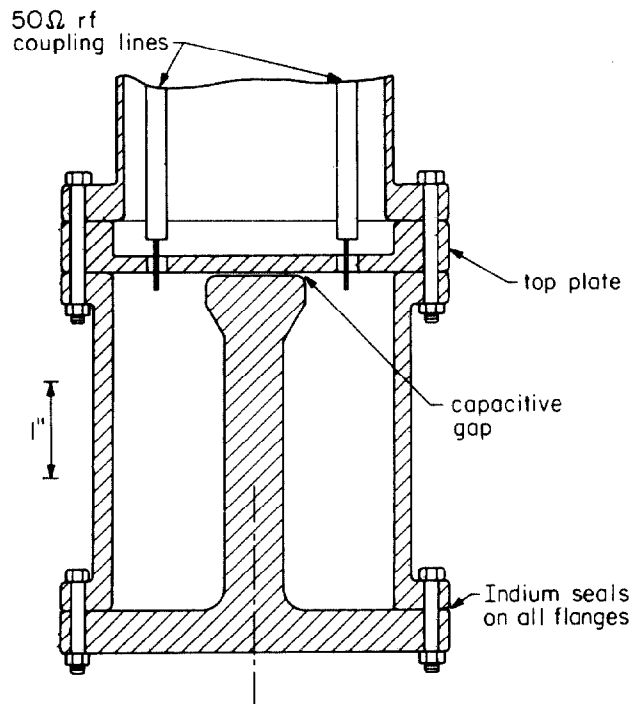


Fig. 1. 200 MHz post resonator used for field emission measurements.

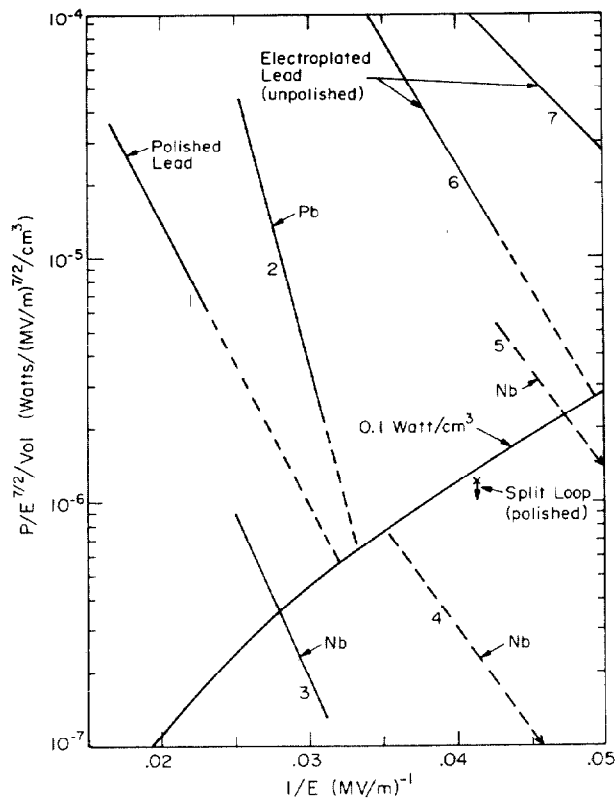


Fig. 2. Fowler-Nordheim plot of field emission losses in superconducting resonator with lead and Niobium surfaces. Curve 1, 6, and 7 are from the present work (see text).

DISCUSSION

It can be seen that the best results obtained with Lead compare favorably with those obtained with Niobium, and that the best of either material would allow useful surface fields in excess of 25 MV/m to be achieved in an accelerating structure. In general, however, the best results cannot be obtained reproducibly, and practical capability falls far below this value. Thus the choice of superconducting material for high electric field applications must depend also on considerations of reliability, low losses, and ease of handling.

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