Contents lists available at ScienceDirect



Nuclear Instruments and Methods in Physics Research A

journal homepage: www.elsevier.com/locate/nima

Extreme diffusion limited electropolishing of niobium radiofrequency cavities



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ARTICLE INFO

Keywords: SRF Niobium Cavity Electropolish Residual resistance

ABSTRACT

A deeply modulated, regular, continuous, oscillating current waveform is reliably and repeatably achieved during electropolishing of niobium single-cell elliptical radiofrequency cavities. Details of the technique and cavity test results are reported here. The method is applicable for cavity frequencies in the range 500 MHz to 3.9 GHz and can be extended to multicell structures.

1. Introduction

Intermittent, diffusion-limited electropolishing of niobium for superconducting radiofrequency (SRF) cavities was first described by researchers at the Siemens Corporation [1]. Continuous electropolishing of rotating niobium cavities, using principles developed at Siemens, was introduced by researchers at KEK and Nomura Plating [2]. Ref. [2] is a particularly valuable source for the history of the development of SRF cavity electropolishing.

An important aspect of the Siemens research is that best results for electropolishing of niobium are obtained by operating in a state of large current oscillations. A graph of current verses time is reprinted from [1] in Fig. 1. A brief explanation of the cause of the current instability is as follows: The electrolyte mixture contains sulfuric acid (H₂SO₄) and hydrogen fluoride (HF). Sulfuric acid acts as an oxidizing agent, forming niobium pentoxide (Nb2O5), an electrical insulator. HF dissociates niobium pentoxide. Under the influence of the electrical potential, both processes occur simultaneously, and at a reasonably high rate. As electropolishing proceeds, a dielectric layer of niobium salts develops at the niobium (anode) surface. It takes time for HF from the electrolyte to diffuse through the dielectric layer and produce a concentration high enough to break down the Nb₂O₅ layer, thus allowing current to reach a maximum. The HF concentration is then locally depleted, and current is minimized until enough time has passed to allow diffusion to build up the HF concentration at the niobium pentoxide surface. The electrochemical cycle then repeats indefinitely, provided the bulk electrolyte solution can supply the required amount of HF.

effect that a significantly large amount of the voltage drop from the cathode to the anode surface is across the layer. This is what makes it possible to have a reasonably large amount of material removal at the large diameter equators of elliptical cavities at the same time as at the smaller diameter irises without the necessity of a conformal cathode that maintains a uniform spacing to the anode.

Since the introduction of the current oscillation concept, it has been a goal to achieve continuous large amplitude oscillations for elliptical cavity electropolish procedures. The best results observed to date in this regard have been with polishing of open half cells [3]. This was due in large part to the use of a conformal cathode and because of the upward facing orientation of the niobium surface. A high density dielectric layer forms easily on such a surface due to the effect of gravity. A downward facing surface will not build up a layer that provides an optimal voltage drop with the type of electrolyte mixture used here.

A technique for achieving a regular, periodic, deeply modulated current waveform during electropolishing of fully assembled cavities has been developed at Fermilab and will be referred to in this report as the "cold EP" method. The buildup of a high quality dielectric layer and control of the rate of diffusion of HF through this layer are of prime importance in achieving the goal. The starting point for the work is an electropolishing tool similar to the KEK horizontal electropolishing system as per Saito [2]. In the next section the critical process parameters will be identified and specified.

The presence of the dielectric layer has the additional beneficial

http://dx.doi.org/10.1016/j.nima.2017.01.006 Received 14 November 2016; Received in revised form 27 December 2016; Accepted 3 January 2017 Available online 04 January 2017 0168-9002/ © 2017 The Author. Published by Elsevier B.V.

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¹ Fermilab is operated by Fermi Research Alliance, LLC under Contract No. De-AC02-07CH11359 with the United States Department of Energy.

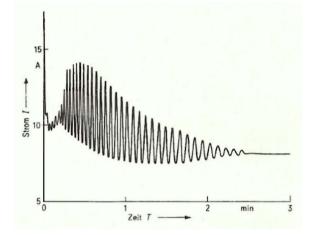


Fig. 1. Current oscillations. The horizontal axis is time and the vertical axis is current.

2. Electropolishing technique

2.1. Electrolyte

The electrolyte used at Fermilab is a 13.5 to 1 (by volume) mixture of concentrated sulfuric acid ($H_2SO_4 > 96\%$ by weight) and hydro-fluoric acid (HF ~70% by weight). The resulting mixture differs from that used in most niobium cavity electropolish operations in that the concentration of water is lower. The ratio of HF to H_2SO_4 is similar to traditional mixtures. Water interferes with the formation of the dielectric layer at the niobium surface. The lower the concentration of water, the better will be the quality of the insulating layer. As water concentration increases, current increases and the depth of the current oscillations decreases, until the dielectric layer and diffusion limited operation are lost.

Hydrofluoric acid with 70% HF concentration, by weight, is considered to have a significantly larger risk for inhalation than hydrofluoric acid with 48% HF concentration. Fermilab safety policy prohibits the use of 70% HF hydrofluoric acid at Fermilab. In order to obtain electrolyte made from 70% hydrofluoric acid, Fermilab purchases pre-mixed electrolyte in containers of 120–2001 from Acid Products Corporation of Chicago, Illinois. Thus the HF is in a dilute form when it reaches Fermilab, having been mixed with a large quantity of sulfuric acid.

Adding water to concentrated sulfuric acid is a strongly exothermic process. Significantly less heat is generated when mixing 70% hydrofluoric acid with sulfuric acid than with 48% hydrofluoric acid because of the lower water content. Lower heat generation during mixing results in improved thermodynamic control of the mixing process and aids in minimizing HF lost due to evaporation.

2.2. Fluid dynamics of the electrolyte

The geometry of the single-cell electropolish assembly is shown in Fig. 2. The cavity is axisymmetric. The cathode is made of aluminum with a minimum of 99.5% purity. The goal for electrolyte flow is to provide adequate HF concentration at the interface between the electrolyte and the dielectric layer, while causing minimal disturbance to the layer due to turbulence. Electrolyte introduction holes are located only on the top half of the hollow cylindrical cathode. This is so that flow is not directed towards the niobium surface. Multiple holes are used in the cathode in order to minimize electrolyte injection velocity while maintaining cathode structural strength and rigidity. The flow of electrolyte is adjusted to approximately one liter per minute for a 1.3 GHz 1-cell cavity.

Fig. 3 shows a photograph of the central section of the cathode. The masking material is polytetrafluoroethylene (PTFE) tape. The need for

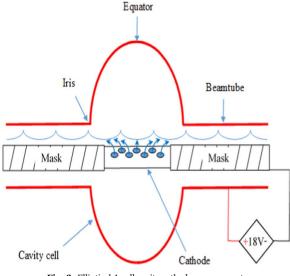


Fig. 2. Elliptical 1-cell cavity cathode arrangement.

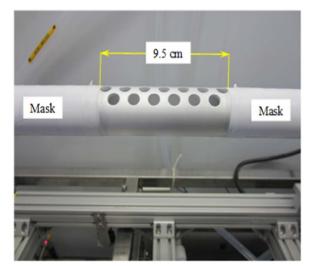


Fig. 3. The cathode, showing holes for electrolyte flow.

the use of masking is questionable, especially at low cathode temperature. It is likely that the results reported here would be obtained if no masking were in use. (The reader should note that for the case of cavities equipped with high order mode couplers, masking of the cathode at the coupler location is beneficial due to the very small gap between the coupler and the cathode. Electrical breakdown of the electrolyte can occur, especially at high electrolyte temperature.) No screen is used to contain hydrogen gas bubbles produced at the cathode. Hydrogen gas is continuously flushed from the cavity with a one liter per minute flow of nitrogen. The level of electrolyte is maintained at approximately 60% of the volume of the cavity. This provides enough liquid inside the cavity to cover the top of the cathode with approximately 5 mm of electrolyte. The cavity rotates slowly about its axis at an angular velocity of one revolution per minute for a 1.3 GHz cavity. This is equivalent to a surface speed at the equator of 1.1 cm/s.

2.3. Temperature control

The electrolyte serves two functions: (1) to provide a fresh supply of HF and H_2SO_4 for the electrochemical reaction, and (2) as a coolant to transport heat away from the niobium surface. In the first generation of cavity electropolish tools, the electrolyte was responsible for all of the heat removal. For the system described in this report, the functions

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