# SRF CAVITY AND MATERIALS R&D AT FERMILAB \*

P. Bauer, L. Bellantoni<sup>#</sup>, T. Berenc, C. Boffo, R. Carcagno, C. Chapman, H. Edwards, L. Elementi, M. Foley, E. Hahn, D. Hicks, T. Khabiboulline, D. Mitchell, A. Rowe, N. Solvak, Y. Terechkine.

FNAL, Batavia, IL 60510, USA

M. Jewell, D. Larbalestier, P. Lee, A. Gurevich, A. Polyanskii, A. Squitieri,

Applied Superconductivity Center, University of Wisconsin-Madison, WI 53706, USA

### Abstract

Fermilab has been steadily developing its SRF cavity expertise, infrastructure and technology base. We particularly emphasize here recent developments in understanding basic material properties and developing a new chemistry treatment facility.

#### **INTRODUCTION**

Two types of 3.9 GHz superconducting RF cavities are under development at FNAL for use in the upgraded A0 photo-injector facility. A  $TM_{110}$  mode cavity will provide streak capability for bunch slice diagnostics, and a  $TM_{010}$ mode cavity will provide linearization of the accelerating gradient before compression for better emittance. Fig. 1 shows the upgraded A0 facility beam line layout including the 3.9 GHz transverse deflecting and 3<sup>rd</sup> harmonic modules. The performance of recent prototype cavities is given in [1]. The use of a  $TM_{010}$  cavity for TESLA bunch compression is discussed in [2].



Figure 1: Upgraded A0 facility beam line layout.

## **CAVITY FABRICATION**

We purchase RRR 300 Nb sheet from industry, cut disks via wire EDM, and stamp, coin, and machine them into cells. Prior to forming, the disks are inspected for imperfections and inclusions with an eddy current scanner. The eddy current scanning, which was formerly performed at DESY, is now done at FNAL with a scanner on loan from SNS. RRR measurements on select samples are performed to check if the material meets the specifications. The latest batch of material received from Wah Chang achieves a high RRR~450. Electron beam welding is performed near FNAL at Sciaky Inc. Our vacuum bake facility (1200°C) is used for annealing and hydrogen removal. 1:1:2 BCP acid etching can be done in-house for cavity parts. The fully assembled cavities, however, have been kindly etched for us by Jlab. We are in the process of commissioning a small manual acid etch

\*Supported by the U.S. Department of Energy. #bellanto@fnal.gov facility operated jointly with K. Shephard, M. Kelly and M. Kedzie at Argonne National Lab. Additionally, a new, larger, remotely controlled, semi-automatic chemistry facility for FNAL cavities is under development. After commissioning at FNAL, the facility will be transferred to ANL to become a part of the ANL-FNAL Surface Treatment Facility, located at ANL. Recent developments regarding this new chemistry facility are going to be discussed in further detail below. A high pressure rinse with 18 M $\Omega$  H<sub>2</sub>0 in a class 10 clean room can be done onsite. RF field flatness tunings are all performed on-site, as is mechanical 3-dimensional high resolution profilometry. We have contracted with Advanced Energy Systems in Medford, NY to fabricate two cavities, the first of which is currently in the welding stage.

### **CHEMISTRY FACILITY**

Chemical treatment with BCP is a well-established procedure that allows the removal of a mechanically damaged and chemically contaminated layer of Nb from the cavity surface. It is used several times during the cavity fabrication with varying thicknesses of Nb removed at each step.

Fig. 2 shows the full-scale mockup of the BCP facility at FNAL, where it is being tested with water. The facility will be installed in a ventilated leak-proof process compartment in order to provide acid containment in case of a spill. This facility is designed not only for 3.9 GHz cavities but also for treatment of the larger 9-cell, 1.3 GHz TESLA-type cavities, which require up to 250 liters of acid. Up to one TESLA-sized cavity a day can be processed. To maintain a precise control over the processing time, the cavity fill-time is required to be 1 minute or less. This requirement is met by elevating the gravity feed tank for acid and by using a large (50 mm) diameter filling pipe. The system operates in the pumpthrough mode with a minimum acid flow speed through the cavity of 1.1 liters/min. When a TESLA-type cavity is processed at a typical etching rate of 1 µm/min, the power required to keep the solution temperature constant (including heat loss) is about 1 kW. A 2 kW heat exchanger keeps the temperature of the acid at 15°C. For a 1 m<sup>2</sup> Nb surface area, about 15 liters of NO<sub>2</sub> are released each minute. Besides NO<sub>2</sub>, there is also emission of NO, HNO<sub>3</sub> and HF – fumes that are considered dangerous pollutants. The facility is designed for air exchange and subsequent scrubbing at a rate of 700  $\text{m}^3/\text{h}$  (200 l/sec).

The acid will be brought into the chemical isolation room in double-walled barrels (HDPE outside, Teflon



Figure 2: BCP chemistry facility assembly at FNAL.

inside) placed in DOT-approved over-packs (HDPE) and connected to the cooling and filling circuits. All the acid circulation circuits are confined to the interior of the chemical confinement room.

The acid mix in the barrel is cooled, and then transferred into the process tank where the BCP process starts. The inner and outer surfaces of the cavity are processed separately, by using different "protection jackets". Acid used for etching is collected and can be reused if the Nb concentration in the acid does not exceed 10 g/l. Rinse water is quite acidic and will be neutralized in an automated system. After acidity rinsing, the cavity is moved to an adjacent clean room for rinsing without the jacket, until a high resistivity is achieved in the outflow rinse water.

A semi-automated operating mode is used to control the different etch process steps. An interlock system prevents accidental or unauthorized access to the manual mode. Sensors provide all the relevant information about the status of the system at each stage of the process to a control system [3]. Temperature gauges measure both acid and coolant temperature, and a differential pressure gauge is used to measure the status of the acid filter. Resistivity meters are used as acid indicators to warn of spills due to equipment failure.

The process compartment is lined from the inside with PVC sheets and equipped with sealed doors to form a watertight container. Most of the critical components are provided with double containment vessels. Pieces of piping with joints and valves where acid is circulating for a long time are equipped with gutters to confine acid spills. In the case of a spill, acid is collected in a sump area with 250 liters capacity. It can be sensed by an appropriate gauge and pumped out before the room is rinsed with water and treated to neutralize remains of the spill.

## MATERIAL STUDIES

Material studies are an important part of FNAL's SRF effort. The purpose of these studies is both to check the cavity manufacturing process and to understand the observed surface resistance and field-emission effects in the first prototypes of the transverse deflecting and 3<sup>rd</sup>

harmonic cavities. In-house measurement capabilities now available include scanning electron microscopy, energy dispersion X-ray analysis, RRR measurements and eddy-current scanning. In addition, FNAL has a collaboration with the Applied Superconductivity Center at the University of Wisconsin-Madison, which has access to a wide variety of electron microscopes and surface analysis systems and where there is an extensive facility for testing superconducting materials. These are, just to mention the most important, SOUID magneto-meters, magneto-optics. and high precision transport measurements.

# Microscopy and Grain Size

Microscopy studies are conducted on small samples subjected to the various steps of cavity fabrication. Fig. 3 shows high resolution images obtained on a sample prepared with our baseline processing. The major preparation steps consisted of a 100 µm BCP followed by an 800°C, 5 hour heating (in vacuum) and a final 20 µm etch. The surface appearance obtained is more or less consistent with that obtained with BCP in other labs [4]. Grain size measurements were performed on samples before and after heat-treatment. The (linear) grain size is typically 50  $\mu$ m, changing only by a few percent during the heat treatment. Grains are much larger, on the scale of 1 mm, in the electron-beam welds at the iris and equator, with one micron deep V-grooves at the grain-boundaries (measured from SEM stereo pairs). It is well known that



Figure 3: a) 3D red-blue anaglyph FESEM image and b) Laser Confocal Microscope (Olympus LEXT courtesy Erich Zeiss of Leeds Precision Instruments). Images at similar magnification of BCP etched surface representative of the FNAL cavities.

even smoother surfaces can be obtained with electropolishing. FNAL is currently undertaking first steps in exploring the electro-polishing technology.

### **RRR** Measurements

RRR studies are done to measure the impact of the cavity fabrication steps on the niobium purity. RRR measurement equipment has been developed over the last few years at FNAL [5]. Fig. 4 shows that the RRR of the Wah-Chang material used for the latest FNAL cavity prototypes is ~450 and remains unchanged throughout the etchings and heat treatments applied in the cavity production stages. The "after treatment" samples were etched ~100 µm with BCP, heat treated in vacuum (800°C, 5 hrs) and then lightly etched with BCP (~20 µm). The RRR is defined as the ratio of the sample resistance at room temperature to that at ~10 K (just above the transition point). There is a particular interest in the thermal properties of Nb in weld seams, because the magnetic field in TM<sub>110</sub> mode cavities is sharply concentrated in the weld region. The RRR in welds was measured and, to within the 5% measurement accuracy, no particular change of RRR was found to occur.



Figure 4: RRR for material used in the fabrication of prototype3<sup>rd</sup> harmonic and transverse deflecting cavities.

### Magnetization Measurements

The measurement of DC magnetization provides, information about the annealing state of the material used for the cavities. Both FNAL and the University of Wisconsin have performed such measurements. Fig. 5 shows the magnetization measured at FNAL on a sample (made from a sheet bent into a cylinder), before and after an 800°C, 5 hrs heat treatment and BCP etching. The plot clearly shows a narrowing of the magnetization hysteresis, which is interpreted as the result of the removal of flux pinning sites due to the annealing. Note that in order to obtain the samples for the magnetization measurements the sheet was heavily cold-worked. Of course, the deep-drawing process used to form the 1/2 cell shapes for cavity manufacture is also a work-hardening process. Comparison to magnetization measurements



Figure 5: Magnetization of an "open" niobium cylinder with height 40 mm and diameter 16 mm at 4.2 K.

performed on similar material and following comparable treatments at the University of Hamburg show reasonable agreement [6]. The University of Wisconsin's magnetization measurements at 1.8 K revealed flux jumps.

### Grain Boundary Experiments

**FNAL** in collaboration with the Applied Superconductivity Center at the University of Wisconsin is also involved in basic research on superconducting RF We are investigating the effect of grain materials. boundaries on the RF surface resistance, using a transport combination of magneto-optics and measurements Preliminary magneto-optical [7]. measurements on mm-size grain samples indicated preferential flux penetration through the grain-boundaries. This is an indication of depressed superconductivity in the grain boundaries. New experiments are on the way to measure the inter-grain critical current.

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