BCP PROCESSING FACILITY[*]

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Abstract

FNAL is engaged in the fabrication of two types of superconducting RF cavities: 3.9 GHz deflecting mode cavities for the RF separator of the CKM experiment and 3.9 GHz 3rd harmonic accelerating mode cavities for the FNAL-NICADD photo-injector upgrade. Thorough surface treatment at several fabrication stages is required for both cavity types to achieve acceptable performance. Buffered chemical polishing (BCP) was chosen as a baseline treatment process. This paper describes the details of the BCP process implementation at the facility under development at FNAL.

INTRODUCTION

Significant progress in the field of RF superconductivity during the last decade has resulted in the increased motivation to use superconducting RF devices in nuclear and high-energy physics. The CKM experiment at Fermilab is planning to use several 3.9 GHz, deflecting mode, superconducting cavities made of Niobium (Nb) for high energy particle separation [1]. A plan to upgrade the FNAL-NICADD photoinjector towards higher energy and brightness implements superconducting 1.3 GHz and new 3.9 GHz accelerating mode cavities [2]. Since the design parameters of both 3.9 GHz cavity types are quite challenging, developing appropriate infrastructure and procedures for cavity surface treatment are imperative.

Chemical treatment (etching) is accepted worldwide as a method to remove mechanically damaged and contaminated surface layers of SCRF cavities [3]. There are two major approaches to chemical etching used in SCRF cavity development – Buffered Chemical Polishing (BCP) and Electropolishing (EP). Although recently EP has proved to show better results [4,5,6], FNAL will implement BCP treatment as the primary procedure due to its widely understood process techniques [4, 7, 8].

BCP uses a mix of three acids: Hydrofluoric HF (49%), Nitric HNO_3 (69.5%), and Orthophosphoric H_3PO_4 (85%). Nitric acid reacts with Nb to form Niobium pentoxide Nb_2O_5. Hydrofluoric acid reacts with this pentoxide to form Niobium Fluoride NbF_5, which is hydrosoluble. Orthophosphoric acid serves as a buffer that helps to keep the reaction rate constant. This rate depends also on the concentration of Nb in the solution. It has been found [4] that the acceptable surface quality can be reached if the concentration of Nb does not exceed 15 g/l. A maximum of 10 g/l of Nb in the mix is acceptable for FNAL processing. Therefore, a minimum of one liter of BCP is needed to remove 1 μm of Nb for each 1 m^2 of surface area treated. As a result, for a typical cavity processing cycle, the required amount of the polish mix can reach 100 liters or more. The large volume of acid requires safe handling and remote process control [7].

Although different laboratories use slightly different proportions of the mix, it is quite typical to use a mixture in the volumetric proportions of 1:1:2 (HF : HNO_3 : H_3PO_4). The thorough description of the chemical reactions can be found elsewhere [8].

A very important and potentially harmful byproduct of the reaction is Nitrogen Dioxide NO_2. Five moles NO_2 evolve for each mole of reacted Nb. Assuming the typical etching rate of 1 μm/min, about 30 g (or ~15 liters) of NO_2 are released each minute from 1 m^2 of the cavity surface. Besides NO_2, there is also emission of NO, HNO_3, and HF, which are considered dangerous pollutants. Although HNO_3 and HF are released only by evaporation, even during normal operation, the evaporation rate and etching time can easily raise respective concentration in the etching room above the acceptable limit of 2 ppm. Assuming an air flow near the surface of the mix of 1 m/s and the acid temperature of 15°C, the evaporation rate is 7 g/(h·m^2) for HF and 12 g/(h·m^2) for HNO_3. Given the room volume of 60 m^3, it takes only few minutes to reach a dangerous level of concentration for the two gases during normal operation. In case of an acid spill, the situation is clearly more severe.

The type and amount of gases released during the procedure necessitate the use of an exhaust fume scrubbing system and proper ventilation to guarantee a safe work environment.

FACILITY DESIGN APPROACH

Personnel and environmental safety and reliability were the key elements of the facility design approach. The basic concept of the design is to have the ability to remotely operate the chemical process from outside the process room. Remote operation prevents facility operators from contacting the acids and resultant fumes. A semi-automated control system with the operator's decisive input between fully automated stages of the process was chosen as the main mode for process control. The control system is described in the accompanying paper [9]. Acid and process byproduct exposure to personnel was minimized in the facility design. To prevent any exposure during operation, all the acid circuits are confined in the room. The acid is transported into and out of the room in double contained barrels (HDPE outside, Teflon inside) placed in DOT-approved over-packs (HDPE). The room ventilation is provided by a scrubbing system with the efficiency of 95% for NO_2 and 99% for the rest of the gases. This fume scrubbing

level exceeds local environmental standards. The requirements for the scrubbing system are based on the conservative operating scenario of treating one TESLA-type cavity per day. The capacity of the scrubber allows using acid-filled tanks with an open surface of up to 1 m^2. Using local ventilation hoods and an air pollution monitoring system permits properly dressed personnel in the room when some small-scale etching is required, e.g. e-beam welding preparation.

PROCESS DESCRIPTION

The hydraulic scheme of the facility is shown in Fig. 1. There are three major parts of the process:
- acid cooling and gravity tank filling,
- etching,
- rinsing.

System preparation and testing as well as post-process clean-up are equally important to successful operation.

Acid cooling

The etching process is exothermal - for each mole of etched Nb, 312 kJ of heat is released into the solution. Since the rate of the process increases with temperature and also depends on the proportions of the acids in the solution, the acids must be mixed thoroughly to avoid stratification and the process temperature must be kept below 20°C to avoid temperature run-out [4]. Cooling the acid in the barrel is the first stage of the active etching process. The barrel pump Pmp1 (Fig. 1) pumps acid from the barrel B1 through the filter F1 and the heat exchanger

HE, returning it back to the same barrel. We use a GF CALORPLAST immersion style acid heat exchanger made of PVDF, with a surface area of 1 m^2 and with heat dissipation capacity up to 2 kW (acid flow 25 l/min, acid input temperature 15°C, and cooling bath water temperature 10°C). The water in the heat exchanger tank is cooled by the primary heat exchanger (4 kW capacity) with circulating propylene glycol. A circulating refrigerator (water chiller WC) with a 4.3 kW capacity at 5°C provides the cooling power. The hydraulic scheme used to test the cooling circuit is shown in Fig. 2.
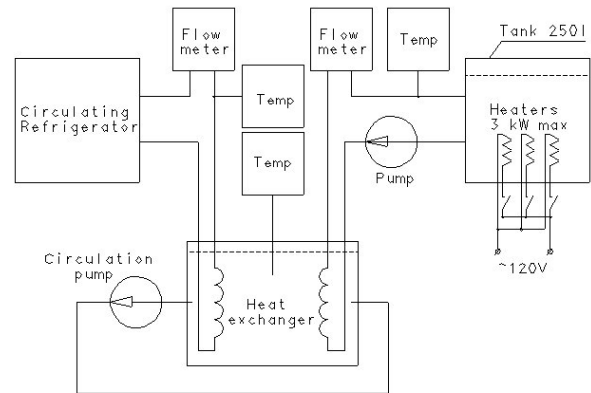
Figure 2: Heat exchanger test scheme

The cooling circuit was tested using water instead of acid. It took about 3 hours to cool 250 liters of water from room temperature down to 12°C.
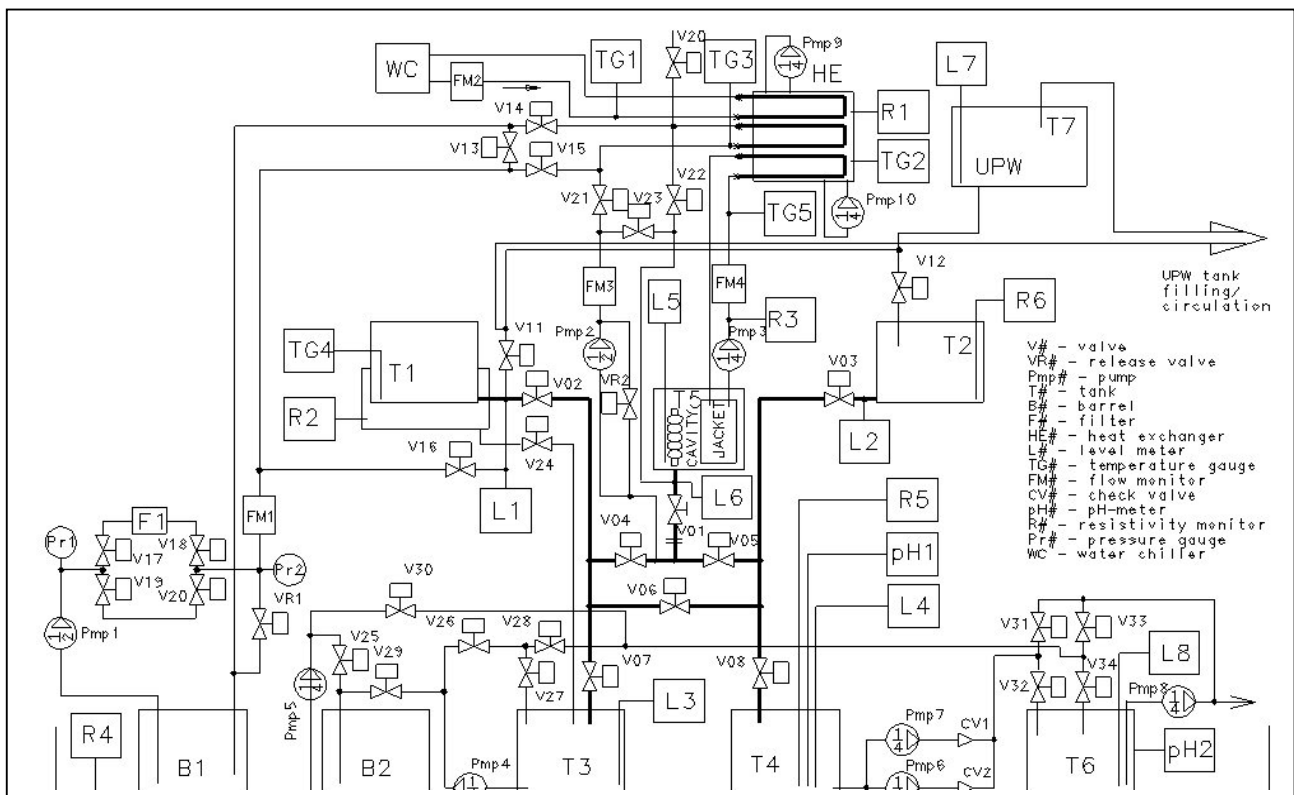
Figure 1: Hydraulic scheme of the BCP facility

As soon as the goal temperature in the barrel B1 is reached, the acid can be transferred into the acid gravity feed tank in preparation to filling the process tank. The filling time must be below 1 minute to reach uniform etching and reduce exposure of the cavity surface to air. To make this possible, 50-mm ID piping and elevated gravity feed tank are employed.

Etching

When a Nb cavity with surface of 1 m^2 is processed, the power required to keep the solution temperature constant (including heat loss) is about 1 kW. The same heat exchanger HE (Fig. 1) is used to cool the acid during the etching stage of the process; pump Pmp2 is used to circulate the acid.

The etching cycle was modeled using the hydraulic scheme in Fig. 2 with water replacing acid as in the acid cooling loop test. The chart in Fig. 3 shows the dependence of the temperatures in the heat exchanger tank and in the process tank with respect to the heating power in the process tank for two different temperature settings of the water chiller. During processing, the temperature can be kept below 20°C even if the power released in the process tank is 2 kW.
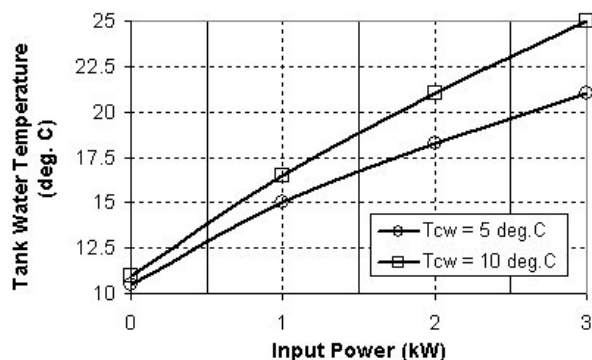

Figure 3: Process tank temperature at different settings of coolant temperature and heating power

Rinsing

After the etching process is completed, the acid is dumped from the etching tank into the used acid tank T3 and then pumped out into the barrel B2. The process tank must be immediately filled with ultra-pure water, which is circulated by the pump Pmp2 for better rinsing.

The rinsing process can be divided in two parts.

Once the acid is removed from the tank T3, the water used to rinse the process tank T5 can be dumped into T3. This water is quite acidic, and must be neutralized in the tank T6 before it can be dumped into a sewage system - several rinsing cycles are needed in order to reach pH of about 4 as regulations mandate.

When pH rises above 4, tank T4 is used to dump the rinsing water from T5. The water conductivity and pH are monitored at this stage. The operator can stop the rinsing process when the goal water resistivity is reached. At this point the process tank is filled with UPW and can be removed from the process room for further rinsing in a clean room environment. After the acid is pumped out remotely into the barrel B2, the room is rinsed with water and treated to neutralize the acid residues

Clean-up

A system of auxiliary valves is used to direct Ultra-Pure water (UPW) to rinse the piping and the tanks from acid so that it is safe to disconnect the process tank and the barrels, which requires personnel to work inside the process compartment. Proper clothing and precautionary measures are required at this stage. First, the process tank is disconnected and moved into a clean room environment. Then the barrels are disconnected and removed from the room. After this, a room inspection must show if any additional cleaning is required before undressed personnel are allowed to enter the process room.

FACILITY LAYOUT

The layout of the facility has been developed based on the TESLA-type cavity specifications. The size of the cavity defines the capacities of all tanks (250 liters), the height of the room (4.5 m), and the compartment and associated equipment footprint. The process compartment is lined on the inside with PVC sheets for watertight containment and equipped with splash-proof doors. Since most of the critical components in the room, like barrels and tanks, are provided with secondary containment vessels and critical piping circuits are equipped with gutters, the room serves as a tertiary container. In the case of a major spill, the acid is collected in a sump area at one side of the processing room with the capacity exceeding 250 liters.

The topology of the facility was developed to minimize the length of piping. Parts of the system where acid can't be removed (e.g. filter and heat exchanger) were separated from other components. The parts that personnel must contact can be rinsed. The overall room dimensions are 5 m in length, 3.5 m in width, and 4.5 m in height.

The vessels in which cavities are etched and the type of etch performed on a given cavity also affect the facility layout. Inside and outside etching typically require different etch depths and physical setups. When etching on the inside, the exterior of the cavity must be protected and vise versa. The flanges of the cavities, made of NbTi, also need protection since this material shows a higher etching rate with respect to Nb, and the surfaces of the flanges used for high vacuum seal can go out of tolerance after etching. Each process may need a different etching vessel, thus affecting how flexible the processing room design must be to accommodate the varying processes.

To allow different cavity types and etching procedures, a system of protective jackets was developed to separate inside and outside etching as well as allow for different cavity types and lengths. An example of the jacket design is shown in Fig. 4.
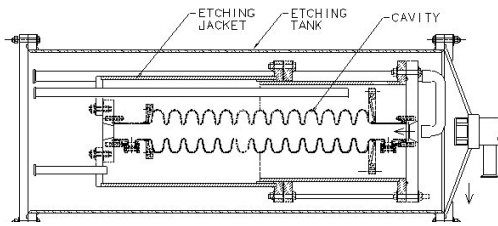
Figure 4. 13-cell CKM cavity etching assembly

The jacket is assembled using two cylindrical vessels that allow telescopic connection with adjustable length.

CONCLUSION

A Buffered Chemical Polishing (BCP) facility is under development at FNAL. This facility is to be a part of the ANL-FNAL SC cavity surface treatment facility located at ANL. Since both the RIA cavity development efforts at ANL and the CKM and 3rd harmonic cavity fabrication at FNAL require a production level surface treatment facility, we are optimistic about the possibility of commissioning the facility in 2004.

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