OPTIMIZATION OF BCP PROCESSING OF ELLIPTICAL Nb SRF CAVITIES

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Abstract

At present, electropolishing (EP) is considered a key technology in fabricating Nb SRF cavities performing at or above 35 MV/m. Nevertheless buffer chemical polishing (BCP) is still a cheaper, simpler and effective processing technique for single grain high gradient and polycrystalline lower gradient cavities. BCP has also been adopted to chemically process the third harmonic 3.9 GHz cavities, operating at or above 14 MV/m, being fabricated at Fermilab [1]. The dimensions and the shape of these cavities pose the problem of uneven material removal between iris and equator of the cells. This paper describes the thermal-fluid finite element model adopted to simulate the process, the experimental flow visualization tests performed to verify the simulation and a novel device fabricated to solve the problem.

INTRODUCTION

Surface polishing is a necessary step of SRF cavity production, which removes the superficial layer of damaged material though either chemical reaction or mechanical friction. The damaged layer is created primarily during the rolling of the niobium sheets used to make the cavity. The damage layer is typically from 80-120 μm and the total amount of Niobium removed from the cavity surface is typically 200 μm. Three surface polishing techniques are commonly used for material removal: buffered chemical polishing (BCP) [2], electropolishing (EP) [3] and centrifugal barrel polishing (CBP) [4]. The first two processes involve chemical reaction, while the third one is based on mechanical friction. BCP, adopted 15 years ago, is recognized as the most reliable and stable process, but its preferential action at the grain boundaries increases surface roughness, thus limiting cavity performance to below 30 MV/m. EP generates a relitvely smoother surface which allows for cavity accelerating gradients above 40 MV/m. But EP is still in the R&D phase resulting in a large spread in cavity performances. CBP substitutes chemical etching with mechanical polishing. It has the potential to improve the surface finish, reduce personnel exposure to hazardous chemicals, and decrease the environmental impact of cavity processing. Currently, after the CBP process is complete chemistry must still be used to remove the 40 micron damage layer created by CBP. Fermilab has established BCP and EP capabilities in collaboration with ANL. Fermilab is also establishing a single cell cavity processing program capable of EP, BCP and CBP R&D.

In terms of cavity production, Fermilab is fabricating and commissioning a 3.9 GHz SRF cryomodule to be delivered to DESY. The cavities for this unit are considerably smaller than standard ILC/TESLA cavities, posing a number of critical issues both for fabrication and conditioning. In particular the shape and the dimensions of these cavities increase the effect of differential etching between iris and equator of the cells. The present simulation work investigates the basic mechanisms of BCP and of the physical phenomena governing the process. This modeling has increased the understanding of the differential etching between the iris and equator and has helped in finding a proper solution. The simulations were performed using a commercial finite element software, COMSOL-MULTIPHYSICS, which is commonly used for the investigation of "multiphysics" problems. A powerful feature of this software is that it gives the operator full control over the system of partial differential equations used to model the physical system. The software allows for the simultaneous solution of multiple partial differential equations while taking into account the cross couplings.

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BUFFERED CHEMICAL POLISHING BCP

Buffered chemical polishing of niobium, performed using the standard mixture of acids, is an exothermic reaction. BCP commonly uses a 1:1:2 part mix of the three following acids respectively: Hydrofluoric HF (49% wt), Nitric HNO_3 (69.5% wt), and Orthophosphoric H_3PO_4 (85% wt). This acid mixture has a room temperature viscosity of 0.022 Pa*s and a density of 1545 Kg/m3 [5]. Nitric acid reacts with Nb to form di-Niobium pent-oxide (Nb_2O_5). Hydrofluoric acid reacts with this pent-oxide to form hydrosoluble Niobium Fluoride NbF_5. Orthophosphoric acid serves as a buffer that helps maintain a constant reaction rate (approximately 1μm/min at 12°C, Figure 1). The reaction rate is strongly dependent on temperature, concentration of the Nb in solution and acid flow rate.

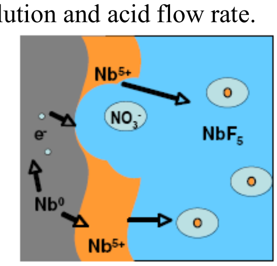

Fig.1: Chemistry of the BCP Etching.

$$Nb_2O_5+10HF\rightarrow 2NbF_5+5H_2O$$
$$2Nb+10HNO_3\rightarrow 10NO_2+5H+Nb_2O_5$$
$$H_3PO_4\leftrightarrow H^++H_2PO^-_4$$

The material removal rate, and thus the reaction heat generation, depend on the fluid temperature and on the acid velocity. However in order to simplify the modeling; the etching rate was assumed constant for the whole surface of the cavity with a value of 1 μm/min. Under this hypothesis, the measured reaction enthalpy (H_{rxn}) is 700 kJ/mol and consequently the reaction heat generation is 1200 W/m^2. Based on experience, the rule of thumb suggests keeping the acid temperature below 20°C and concentration of Nb in the BCP bath below 15 g/l. Therefore, a minimum of about one liter of BCP is needed to remove 1 μm of Nb for each 1 m^2 of treated surface area.

For a typical cavity processing cycle, the required amount of polishing mix can exceed 100 liters. This translates into the need for significant attention to issues of personal and environmental safety and suggests processes should ultimately be controlled remotely. An immediate (and harmful) byproduct of the process is Nitrogen Dioxide NO_2. Five moles of NO_2 are generated for each mole of reacted Nb. With a 1 μm/min etching etching rate, about 25g (or ~12 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 also considered dangerous pollutants. The fumes of HNO_3 and HF are released by evaporation. If the fumes are not captured, concentrations of theses gases in the etching room can easily exceed the TWA acceptable limit of 2 ppm for HNO_3 and 0.5 ppm for HF. Obviously, the situation can become even worse if an acid spill occurs and the acid evaporation surface increases. The type and amount of gases released during the process necessitate the use of proper ventilation and exhaust fume scrubbing. Proper air handling is needed to guarantee minimal exposure of personnel to harmful gases and to avoid environmental contamination. The 3.9 GHz cavities are etched on the inside and outside. The most critical step in terms of RF performances is the etching of the inside of the cavity to remove the damage layer. The etching of the outside of the cavity is performed to avoid contamination of the inner surface during the heat treatments and to increase heat transfer capability.

At the beginning of the etching procedure the cavity, assembled in a cooling jacket, is held in vertical position and filled with acid by means of a gravity-feed system. Later, the acid is slowly circulated to provide cooling and mixing while the outside of the Nb walls are cooled with water circulating in the jacket. The acid velocity is a fundamental parameter used to assure a uniform niobium removal rate. The acid velocity has been optimized, for the 3rd harmonic cavity, at 1.5 gpm. At an acid flow rate of 1.5 gpm, the removal rate ratio between the iris and equator is 1.9. Most cavities are currently processed in the ANL facility (figure 2) since the FermiLab BCP system is in the process of safety review.

Process simulations were performed using system parameters from both Fermilab's and ANL's facilities.

Fig.2: ANL Facility

The two facilities are very different in terms of layout and of automation of the process, but only a few facility layout details need to be taken into account during the modeling. In the Fermilab facility the cooling jacket is completely immerged in the acid tank, while in the Argonne facility the cooling jacket is a simple plastic container directly in contact with ambient air. Table 1 summarizes the main differences between the two facilities relevant for the computational study of the BCP process.

Tab. 1 Facilities parameters relevant for the simulations

		FNAL Facility	ANL Facility
Acid Flow Rate	GPM	1.5	1.5
Water Flow rate	GPM	5	10
Acid Inlet Temperature	K	288	288
Water Inlet Temperature	K	288	279
Average Reaction Heat Generation	W/m^2	1200	1200

DIFFERENTIAL ETCHING BETWEEN IRIS AND EQUATOR

In both EP and BCP the overall removal of material in the cells is not uniform. The local material thickness is measured with an ultrasonic gauge in several spots of the cell both before and after the material removal process. As a result one discovers that the removal rate at the iris is much higher than at the equator by a factor varying between 1.3 and 2. This phenomenon is related to the high viscosity of the acid mix and to the geometry of the cavity. The combination of these two factors favors the stagnation of the acid within the cells reducing the etching rate. Flow velocities near the iris are higher, increasing the etching rate. In the case of low frequency cavities it has already been proposed to introduce a system of rigid diverters to force acid flow into the cells. In the case of 3.9 GHz cavities, due to their small dimensions, this solution presents a high risk of scratching the surface at the end of the process. As an alternative it was proposed to insert a system of nine PET balloons inside the nine cell cavity (figure 3).

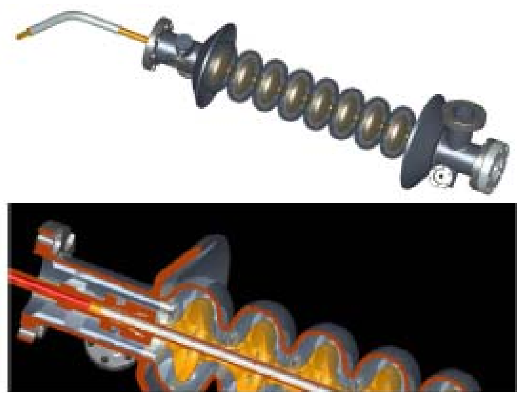
Fig. 3 Flow diverter into the cavity

PET samples tested in an acid environment comparable to the BCP process, showed good acid resistance after four hours exposure, which is more than twice the duration of a typical etching procedure. For fabrication purposes the balloon is molded in three separate segments, which are later glued together. For this purpose several samples of PET bonded with cyanate and UV-cured adhesives were tested. As a result the UV-cured glue was chosen as more reliable. The balloon is fixed to a PVDF tube that serves as centering device and a deflating/inflating system. The device, after insertion, is inflated in the cavity and expands within the cell volume. When the process is finished, the balloon is collapsed by releasing the air pressure and then covered by a Teflon sleeve for easy extraction.

BCP PROCESS MODELING

A comprehensive model of the BCP process is very difficult to solve numerically. Such simulation involves the solution of the Navier-Stokes equations for a complex geometry. This involves simultaneously solving equations that describe the conductive and convective thermal systems and the chemical reaction equilibrium, which itself is strongly coupled to the thermal-fluid variables. The solution of the simulations for the Fermilab 3.9 GHz cavity with cooling jacket are reported in [6]. Since the chemical aspect of the process is deeply related with the thermal-fluid dynamics of the system, an exhaustive thermal-fluid dynamic study was performed. In particular the thermal simulation was coupled to the fluid model in order to evaluate the temperature distribution in the cavity during the process. The results, seen in Figure 4, show how in the ANL facility the cold acid flows only in the center of the cavity while a lack of circulation is observed within the cell. In this case, however, the temperature profile is really uniform from cell to cell due to the strong water-cooling.

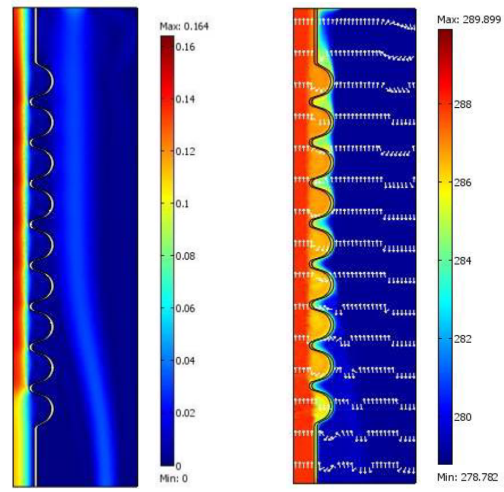

Fig. 4 ANL Facility flow pattern (left) and temperature map (right)

The FNAL facility studies show that in absence of strong water-cooling and due to the acid inlet at the bottom cell of the cavity, the cell temperature (and consequently the etching rate) rises through the cavity, even in the case of water in counter-flow with respect to the acid (figure 5). This simulation, limited in accuracy by not taking into consideration the acid speed and temperature dependence of the etching rate, is consistent with experimental observations of the BCP process.

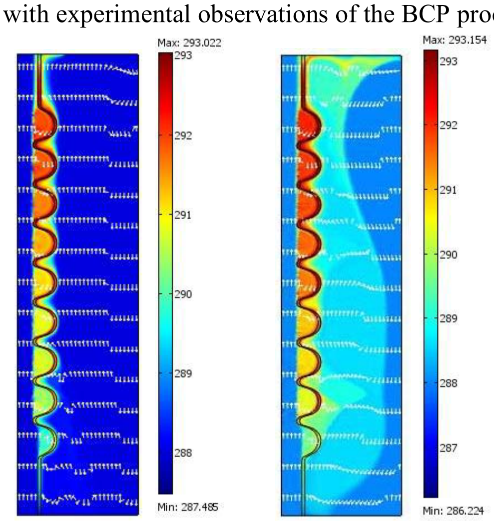

Fig. 5 FNAL Facility. Temperature maps. Acid and water in parallel flow (left) and counter flow (right)

In particular it confirms that limited cell circulation is the main factor influencing the differential etching between iris and equator. In addition it demonstrates that increasing the water-cooling system capacity helps optimizing the cell to cell temperature distribution. These results are also consistent with benchtop experiments performed at Fermilab [7] demonstrating that for this type of cavity, given their acid volume to Niobium surface ratio, the reaction rate slows down and eventually stops after 30 minutes if the acid is not properly circulated. In order to fully simulate the BCP process in all its aspects, one should also take into consideration the chemical reactions and the diffusion of the species in solution. In all previous models the heat generation was considered constant through the process and uniform on the whole surface. In reality the speed of the reaction depends on both the temperature and the concentration gradient of species in solution. In fact, the stagnation of acid in the cells, which is mostly due to its viscosity and to the geometry of the cavity, leads to the generation of high niobium concentration areas in which the chemical reaction is much slower with respect to fresh acid areas. In order to take into account this effect, a third physical phenomenon was added to the simulation by introducing the equation system regulating the mass transport due to diffusion and convection (figure 6). In order to improve the process, several simulations were performed with the goal of evaluating the impact on flow and temperature of a flow diverter inserted into the cavity.

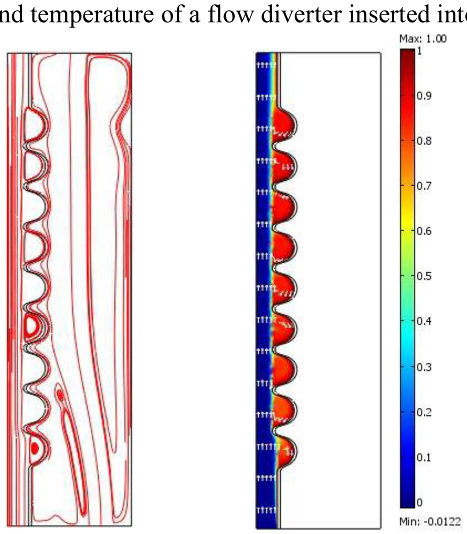

Fig. 6 Acid stagnation inside the cell. Steam line flow pattern (left) and Nb concentration map (right)

A flow diverter with optimized shape and dimensions will reduce temperature and flow gradients in the cavity during the process, thus equalizing material removal between iris and equator. The three levels of simulations, fluid dynamic, thermal-fluid, and thermal-fluid with diffusion, were repeated in the case of the flow diverter inserted in the cavity (figure 7). In all cases the deformations of the balloons, due to the acid flow, have been neglected. The analysis of the temperature and the velocity and the concentration maps demonstrated that the use of a flow diverter results in more uniform distributions of the three parameters. The acid stagnation in the cells is avoided, significantly reducing the differential etching between iris and equators

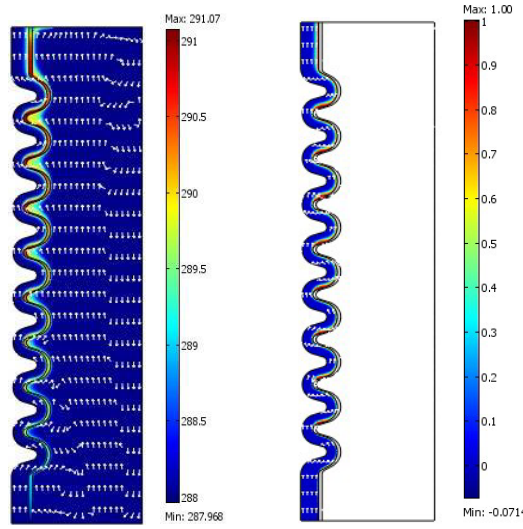

Fig. 7 Flow diverter evaluation. Temperature map (left) and concentration of Nb map (right)

As an additional step the model was used to optimize the acid velocity in the cavity. In fact by keeping the same flow rate as for the case without the diverter, several vortices could be generated, perturbing local flow. This issue was solved by simply reducing the overall acid flow rate until a full laminar flow was observed in the simulations.

EXPERIMENTAL VERIFICATION

A flow visualization experiment was performed in order to verify the numerical simulation and later the effectiveness of the flow diverter. A transparent polyurethane 9-cell cavity was produced and a basic device to introduce small amounts of colored dye in the main stream of water was designed (fig.8). The tests were performed using water and, in order to maintain the same Reynolds number, the velocity was proportionally reduced to mimic the acid flow of the BCP process. The test demonstrated that even if the flow at the center of the cavity is fully developed, the acid remains trapped in the cell volume leading to differential etching between iris and equator.

Fig.8 Flow visualization test

CONCLUSIONS

The initial goal of this work was to solve differential etching rates between the cavity equator and iris that appears in the BCP of 3.9GHz 9-cell niobium cavities which are a key component of the future DESY XFEL and possibly of the ILC injection system. The BCP process of elliptical SRF cavities was simulated, including its thermal-fluid aspects. In order to improve the process, several simulations were performed with the goal of evaluating the impact on flow and temperature of a flow diverter inserted in the cavity. Installing a simple baffle with dimensions smaller than the iris is not very effective due to the acid high viscosity. Simulations show that a flow diverter with optimized shape and dimensions will equalize the material removal between the iris and equator. A collapsible, formed PET balloon inserted inside the cavity is proposed to avoid acid stagnation inside the cells and guarantee uniform material removal. The simulations have demonstrated that the balloon option is very effective and should be pursued further. The device will be soon tested with water using the transparent cavity and later its effectiveness will be proved during an etching process. The present work can be considered a starting point for futures studies and investigations.

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