Investigation of Plasma Etching for Superconducting RF Cavities surface Preparation

Proposal for LCRD/UCLC 2005

Classification (subsystem)

RF cavity for the main linac

Personnel and Institutions requesting funding

Leposava Vuskovic, Old Dominion University

Collaborators

S. Popovic, Old Dominion University
M. Raskovic, Old Dominion University
A. -M. Valente, Thomas Jefferson National Accelerator Facility
L. Phillips, Thomas Jefferson National Accelerator Facility

Project Leader

Leposava Vuskovic
vuskovic@physics.odu.edu

Department of Physics
4600 Elkhorn Avenue, Room 306
Old Dominium University
Norfolk, VA 23529
Telephone: (757) 683-4611
FAX (757) 683-3038

Old Dominion University
Research Foundation
2033 Hughes Hall
P.O. Box 6369
Norfolk, VA 23508
Telephone: (757) 683-4293
Fax: (757) 683-5290

Introduction

Of the many recipes used to prepare SRF cavity surfaces, none presently will provide repeatable performance reasonably close to fundamental limits, and none are understood at the microscopic level in terms of their effect on variability of RF performance. In this context Thomas Jefferson National Accelerator Facility (Jefferson Lab) and the Atomic Collision and Plasma Physics Group of the Physics Department, Old Dominion University (ODU) in Norfolk, Virginia have joined forces to investigate plasma etching as an alternative technique for surface preparation of high purity bulk Nb cavities. The Atomic Collision and Plasma Physics Group at ODU has proven its expertise in the field of plasma modification of surfaces. The collaboration between Jefferson Lab and the Atomic Collision and Plasma Physics Group at ODU will strongly impact the Nb surface preparation procedures as well as advance the understanding of surface chemistry of high purity niobium for superconducting RF cavities, which will be beneficial for a future linear collider.

Motivation

The standard (commonly used) surface preparation techniques for Nb superconducting RF cavities are chemical (BCP) or electrochemical (EP) polishing. Those make use of extremely corrosive acids, which present both safety and environmental concerns. With wet chemical polishing, the formation of an Nb oxide layer is unavoidable since oxidation starts as soon as the chemical process stops. More over it has been demonstrated that these techniques, due to the baths composition, introduce hydrogen in the oxide layer and the bulk material [1]. Acid residues are usually left by phosphoric acid on the surface. These chemical residues are eliminated by High Pressure Water Rinsing (HPWR) with de-ionized water for several hours. Finally, especially in the case of EP, the cavity is baked under vacuum at moderate temperature (90ºC-140ºC) in order to outgas the water adsorbed on the surface and to reduce the surface hydroxides [2].
It has long been suspected that "weak links" formed by intergranular oxidation could be responsible for the RF performance degradation or "Q droop" [3]. It is also suspected that niobium surfaces under certain circumstances will form lossy sub-oxides in addition to Nb_20_5. Testing before and after oxidation would provide extremely valuable insight into this issue.
Plasma etching is one form of dry chemistry providing a unique opportunity to explore oxide-free surfaces by directly testing a cavity surface after processing without exposure to air. This technique allows also "control" on the final oxidation phase that cannot be avoided with SRF cavities (oxidation of the fresh Nb surface after treatment by O_2/N_2 ("dry air") gas injection in the system). The relatively scarce literature describes niobium [4] and niobium oxide [5] etching in reactive discharge plasmas. In the first case [4] etching rates of the order of 0.2-0.3 mm/min were obtained. In the second case [5], significant structural changes of niobium oxides were obtained. This limited experience can be used as a starting point for a systematic study of the inner surface modification of superconductive cavities using low-pressure discharge plasmas.

Advantages of dry etching:

- Takes place under vacuum.
- Allows "control" on the final oxidation phase
- Allows the possibility to avoid final oxidation
- Lower process cost.
- Reduces the number of steps for the final surface preparation.
- Gets rid of the hazard to humans and environment induced by the chemicals used for BCP and EP.
- No acid residues on the surface.
- No hydroxides formation on surface.

The investigation of plasma etching for Nb cavity surface preparation will improve the surface quality of the SRF cavities and therefore improve their performance for a linear collider.

Current ODU Collaboration with Jefferson Lab

The Atomic Collision and Plasma Physics Group at Old Dominion University has proven its expertise in the field of Plasma Physics. In the recent years, their research in plasma processing and related field has been focused on the use of subcritical surface microwave discharges for surface modification [6], the extraction of oxygen by dissociation of carbon dioxide [7], and for generating aerodynamic effects [8].

The proposed experiment is devoted to the use of metal-to-discharge plasma interfaces to generate adequate environment for transformation of Niobium oxides from the surface of SRF cavity into removable volatile compounds. When developed, the oxide removal process could serve as a complementary surface modification method to those currently used in preparing the SRF cavities for operation. The collaboration between the Jefferson Laboratory, with the expertise in SRF cavity material development and testing and the ODU team, with the experience in plasma processing will bring the needed synergy for developing the process.

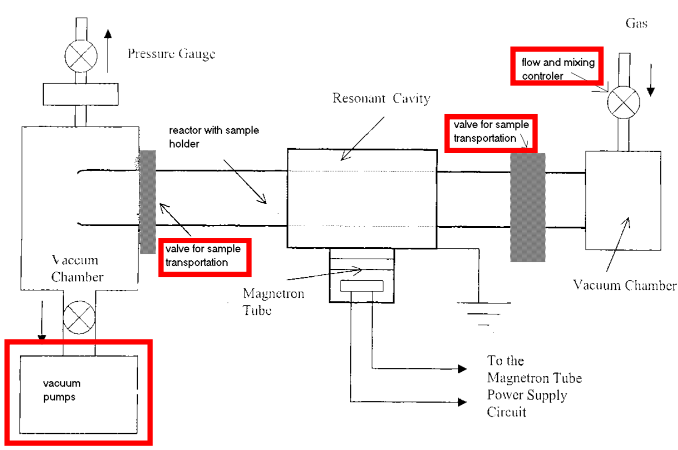
Project Overview

ODU and Jefferson Lab have agreed to collaborate on the investigation of plasma etching, using the experimental systems developed for plasma studies at ODU. According to the agreement, Jefferson Lab will be responsible to provide Nb material and surface analyses for the plasma etching studies on Nb conducted at ODU. Both parties will share the efforts related to analysis and publication.

The following approach has been defined:

1) Microwave discharge in a cylindrical cavity at various pressures of
   a. Ar plasma with a biased sample
   b. Ar/Cl mixtures
   c. CCl_2 and CF_4 flow
   d. Other etchant gases as C_2F_6, SF_6, NF_3.

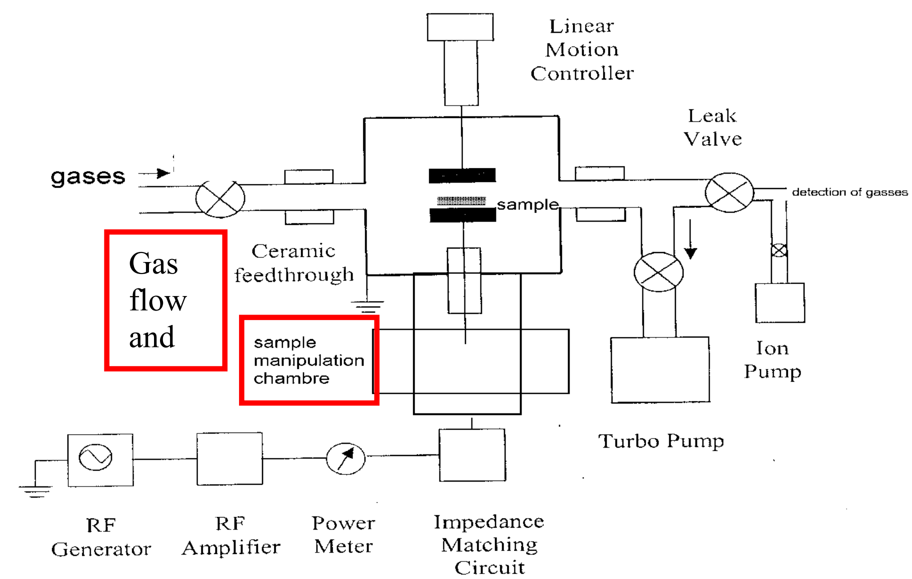
Microwave discharge
Scheme of experimental set-up



2) Capacitively-coupled RF discharge with Nb sample as one electrode, at various pressures of

a.Ar plasma with a biased sample
b.Ar/Cl mixtures

<u>RF discharge</u>
Scheme of experimental set-up



During the development of the techniques, the plasma process will be characterized with plasma diagnostics (temperature, electron density, different species density) and process controls (emission and absorption spectroscopy). The characterization of the samples produced will involve surface morphology (SEM) and composition (XPS) analyses and surface resistance measured by calorimetric method in a TE011 cavity.

3) Application of the process to Nb cavities

Once the principal of the technique will be demonstrated and the best approach being determined, the technique will be implemented in a cavity system at Jefferson Lab. The surface of elliptical RF cavities will be prepared by plasma etching and their RF performance will be tested at Jefferson Lab.

<u>Broader Impact</u>

Successful development and implementation of plasma etching as a surface preparation technique for high purity Nb cavities will allow a cost reduction and safer procedures in cavity surface preparation. A better control on the Nb surface composition and morphology will lead to a more reliable cavity performance. This will be beneficial for a Linear Collider as well as for any accelerator project based on Nb RF Superconductivity.

One member of the team, Marija Raskovic, is currently pursuing her doctoral thesis work at Old Dominion University, working on the characterization of the Niobium processing microwave and RF-excited plasmas. In addition, parts of these experiments will be used for training the undergraduate students as their senior summer research projects. The students involved will learn the plasma physics involved in RF and microwave discharges, various surface science methods and SRF technology.

Project Activities and Deliverables

During the first year, the two experimental systems described in the previous section will be developed. A sample study of plasma etched surfaces of high purity Nb will be conducted. Reports on the technique developments and sample surface and RF analyses will be produced.
During the second and third year, the sample study will be completed and the technique will be transposed for cavity surface preparation. Nb cavities will be prepared using plasma etching and
During these three years the technique developments and surface results obtained on samples and cavities will be published in referee journals and a PhD thesis will be presented.

References:

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[3] A.M. Portis, D.W. Cooke, H. Piel, " Microwave Surface impedance of Granular Superconductors", Physica C. , 162-164 (part 2), 1547-1548 (1989)
[4] Jay. N. Sasserath, "Dry etching of niobium using CCl_2F_2 and CF_4: A comparison", J. Appl. Phys., 68 (1990) 5324-5328.
[5] H. Turcicova et al., "Plasma processing of $LiNbO_3$ in a hydrogen/oxygen radio-frequency discharge", J. Phys D: Appl. Phys. 31 (1998) 1052-1059.
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Budget

The activities outlined above will involve ODU and Jefferson Lab staff members whose salaries are not included in the present budget request.

Table 2: Budgetary estimate for the ODU-Jefferson Lab collaboration

Item	FY 2005	FY 2006	FY 2007
Faculty	$ 24, 300	$ 24, 300	$ 24, 300
Other professionals	$ -		
Graduate Student	$ -		
Total Salaries (incl. fringe benefits)	$ 24, 300	$ 24, 300	$ 24, 300
Equipment	$ 30,000	-	-
Travel	$ -	-	-
Materials and Supplies	$ 14,000	$ 7, 000	$ 7, 000
ODU Tech. Consultants	$ 12,400	$ 7, 200	$ 7, 200
Total direct costs	$ 80,700	$ 38,500	$ 38,500
Total indirect costs (42% of direct cost minus equip.)	$ 21, 294	$ 16,170	$ 16,170
Total direct and indirect costs	**$ 101, 994**	**$ 54, 670**	**$ 54, 670**

Budget Justification for FY 2005

Faculty	Vuskovic (Faculty)– ($9,300/mo) – 0 mo. Atomic collisions and Electric discharge physics Popovic (Faculty)– ($9,000/mo) – 2 mo. Electric discharge physics
Other Professionals	-
Graduate Students	1 PhD Student ($1,800/mo) - 0 mo.
Fringe Benefits & Salary	**ODU Fringe Benefits Breakdown** Research Professionals - 35% of salary Graduate Students – 35% of salary
Materials, Supplies, Software and Training	***Total ($14,000)*** Quartz tubes **($2,000)** Gas **($5,000) –** high purity gases for discharge Materials **($2,000)** – high purity materials Test equipment fees **($3,000)** Sample manipulation supplies **($2,000)**
Permanent Equipment	***Total ($30,000)*** Corrosion-resistant vacuum components **($10,000)** Corrosion-resistant gas flow components **($10,000)** MW/RF power components **($ 10,000)**
Travel	-
Consultant Services	***Total ($12,400)*** Rey Gregory (MW/RF specialist)– ($4,000/mo) – 1.5 mo. Miroslav Cingel (Metal parts design and construction specialist) – ($3,200/mo) – 2 mo.
Indirect Costs	The ODU indirect cost rate is 42% of all direct costs excluding equipment over $2,000