Work supported by

Office of Science
U.S. DEPARTMENT OF ENERGY

as part of R&D effort for ILC

# Plasma Treatment of Bulk Niobium Surface for SRF Cavities - optimization of experimental conditions on flat samples -

M. Rašković, J. Upadhyay, L. Vušković, and S. Popović
*Old Dominion University, Department of Physics, Norfolk, VA 23529, USA*

L. Phillips and A.-M. Valente-Feliciano
*Thomas Jefferson National Accelerator Facility, Newport News, VA 23606, USA*

## Motivation

Accelerator performance, in particular the average accelerating field and the cavity quality factor, depends on the physical and chemical characteristics of the superconducting radio-frequency (SRF) cavity surface [1]. The preparation of the cavity walls has been one of the major challenges in SRF accelerator technology. Therefore, constant R&D effort is devoted to develop surface preparation processes that will improve roughness, form surfaces with smaller grain boundaries, and lower the level of impurities, embedded in bulk niobium. Plasma based surface modification provides an excellent opportunity to eliminate non-superconductive pollutants in the penetration depth region and remove mechanically damaged surface layer improving surface roughness [2]. Here we show that plasma treatment of bulk Nb presents surface preparation method alternative to the commonly used buffered chemical polishing (BCP) and electropolishing (EP) method. Optimization of the experimental conditions in the plasma discharges and their influence on the Nb etching rate and surface properties was performed on a disk shaped Nb samples. Upon determining optimal experimental conditions on disk shaped samples, the procedures will be applied to single cell cavities, pursuing improvement of their RF performance.

## Comparison of **wet** and **dry** etching

BCP (1:1:2 $HF:HNO_3:H_3PO_4$)

$2Nb(s) + 5NO_3^- \rightarrow Nb_2O_5 + 5NO_2 + 5e^-$

$Nb_2O_5 + 6HF \rightarrow H_2NbOF_5(aq) + NbO_2Fx0.5H_2O + 1.5H_2O$

$NbO_2Fx0.5H_2O + 4HF \rightarrow H_2NbOF_5(aq) + 1.5H_2O$

Etching rate 1:1:2 mixture at 20°C is 1 μm/min [3]
Impurities: H, N and P
Oxidation inevitable

EP (1:9 $HF:H_2SO_4$)

$2Nb(s) + 5SO_4^{2-} + 5H_2O \rightarrow Nb_2O_5 + 10H^+ + 5SO_4^{2-} + 10e^-$

$Nb_2O_5 + 6HF \rightarrow H_2NbOF_5(aq) + NbO_2Fx0.5H_2O + 1.5H_2O$

$NbO_2Fx0.5H_2O + 4HF \rightarrow H_2NbOF_5(aq) + 1.5H_2O$

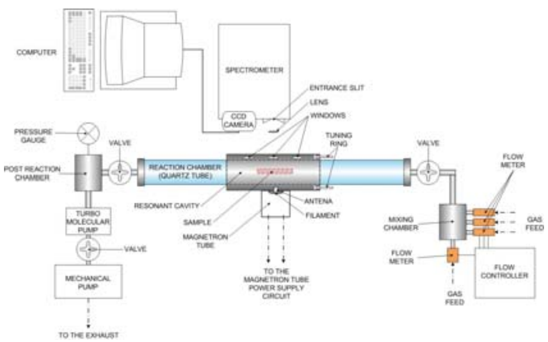
Etching rate 1:9 mixture at 20°C and 17V is 0.3 – 0.5 μm/min
Impurities: H and S
Oxidation inevitable

PE (Plasma etching)

$2Nb(s) + 5Cl_2(g) \rightarrow 2NbCl_5(g)$

No oxidizing agent needed
Etching rate 3Vol% $Cl_2$ in Ar glow discharge 1.7 μm/min
Impurities: none
Oxidation can be prevented

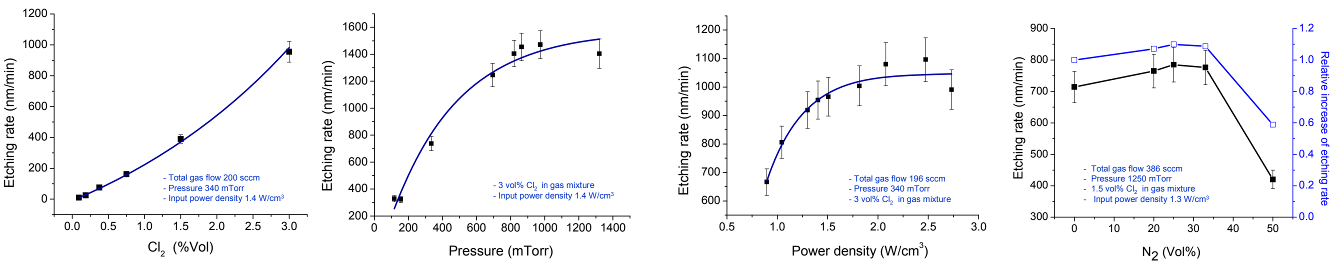
## Microwave glow discharge system set-up [4]




## Research objectives

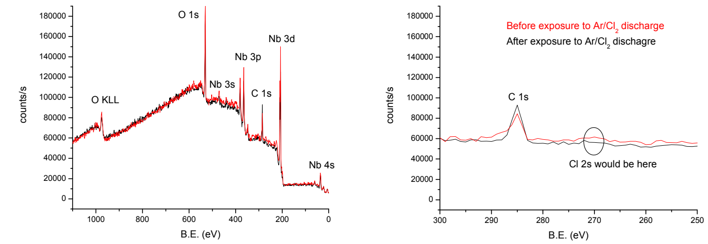
- ✓ ✓ Achieve **ETCHING RATES** comparable with etching rates during wet etching processes.
- ✓✓ Lower amount of **OXIDES** and other **IMPURITIES** in top surface layer.
- ✓ Obtain **SMOOTHER SURFACE** than obtained in wet etching processes.
- ✓ Understand $Nb/Cl_2$ plasma etching **MECHANISM** for the control of plasma based surface modification process.
- • Improve **RF PERFORMANCE** of accelerator cavities.

## Etching rates dependence on the discharge parameters

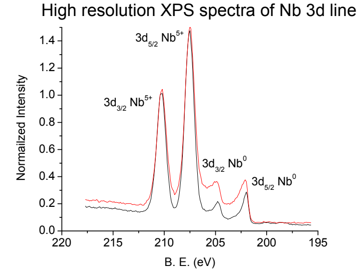



## Surface composition before and after exposure to discharge

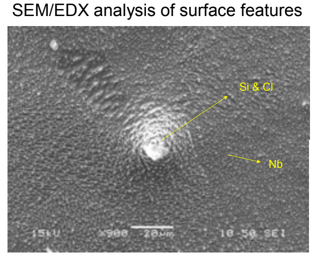
X-ray photoelectron spectroscopy (XPS) spectra of Nb sample surface




High resolution XPS spectra of Nb 3d line



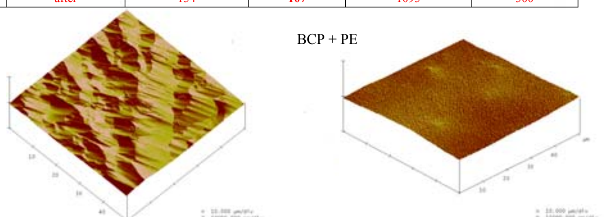

SEM/EDX analysis of surface features




## Surface roughness dependence on sample history

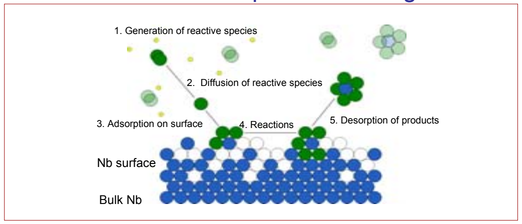
| Surface pre-processing | Plasma etching | RMS (nm) | $R_a$ (nm) | $R_{max}$ (nm) | ΔS (μm²) |
|---|---|---|---|---|---|
| Unprocessed scan size 50 μm x 50 μm | before | 254 | 210 | 1306 | 55 |
| | after | **231** | **174** | 1922 | 167 |
| Mechanical polishing scan size 20 μm x 20 μm | before | 758 | 591 | 4269 | 217 |
| | after | **637** | **509** | **3771** | 265 |
| Buffered chemical polishing scan size 50 μm x 50 μm | before | 286 | 215 | 1511 | 12 |
| | after | **215** | **169** | 1520 | 302 |
| Electropolishing scan size 50 μm x 50 μm | before | 133 | 124 | 641 | 37 |
| | after | 134 | **107** | 1093 | 300 |

BCP BCP + PE

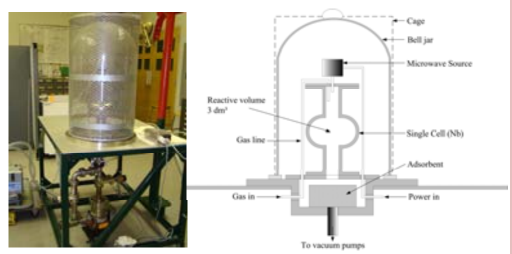


1. Pure Ar ⇒ removes physisorbed gasses and organic residues from Nb surface without damaging surface
   Time 30 min Total flow 150 sccm Pressure 500 mTorr
   Input power density 2.08 W/cm³ Etching rate 0 nm/min
2. 3 Vol% $Cl_2$ in Ar ⇒ removes surface necessary for cavity production
   Time 120 min Total flow 198.6 sccm Pressure 550 mTorr
   Input power density 2.08 W/cm³ Etching rate 1 μm/min
   removes ~120 μm of surface in 2 h
3. 1.5 Vol% $Cl_2$ in Ar ⇒ removes surface under conditions more favorable for surface smoothening
   Time 240 min Total flow 348.6 sccm Pressure 1250 mTorr
   Input power density 1.4 W/cm³ Etching rate 0.5 μm/min

## Mechanism of plasma etching




## Single cell cavity reactor




## Concluding remarks

- We have shown that the etching rates of bulk Nb as high as 1.7 ± 0.2 μm/min can be achieved in a microwave glow discharge using $Cl_2$ as the reactive gas.
- Nb etching rate depends on $Cl_2$ reactive gas concentration and discharge parameters: input power density and pressure in reaction chamber.
- Surface composition analyses show that no impurities have been introduced into Nb during microwave discharge treatment.
- Developed three step process produces surface of satisfying roughness.
- Emission spectroscopy result combined with measured etching rates, suggests that the Nb etching mechanism in $Ar/Cl_2$ MW glow discharge is more a chemical etching than a physical sputtering process.
- A modified experimental set-up that includes single cell cavity has been built. It will be used to determine the influence of the plasma treatment on RF performance of the cavities.

References:
[1] H. Padamsee, J. Knobloch, and T. Hays, *RF superconductivity for accelerators* (Wiley-VCH, Weinheim, 2008).
[2] M. Rašković, *et al.*, Nuclear Instruments and Methods in Physics Research A **569**, 663 (2006).
[3] A. Matheisen,Turorial 3b, 13th SRF workshop, Beijing 2007.
[4] M. Rašković, *et al.*, Journal of Vacuum Science and Technology A **27** (2), 301 (2009).

OLD DOMINION UNIVERSITY

Jefferson Lab

JSA