# 

# Niobium Electropolishing in an Aqueous, Non-Viscous HF-Free Electrolyte: A New Polishing Mechanism



M. Inman, T. Hall, E.J. Taylor, Faraday Technology, Inc., Clayton, OH 45315, U.S.A. C.E. Reece, O. Trofimova, JLAB, Newport News, VA 23606, U.S.A.

#### Need

- Superconducting radio frequency cavities are fabricated from pure niobium
- Processing of the cavities requires polishing of the interior surface to a mirror finish
- State-of-the-art polishing technology for the Nb cavities uses either buffered chemical polishing or conventional electropolishing
- However, these process employ hydrofluoric acid, which is an "environmental insult" and hazardous to workers
- Ideally, a polishing process for superconducting RF Nb cavities will have attributes that include the following:



# **Objectives, Approach, and Key Advantages**

#### **Overall Objective:**

- Development of the FARADAYIC HF-Free Electropolishing Process for polishing alloys used in the manufacturing of cavities
- Understand the interactions between polishing parameters, surface finish, microstructure, oxide formation and 2K RF test performance

#### Approach:

Reduce the oxide film by an electrochemical process instead of relying solely on aggressive chemicals (e.g HF)

- Electrolyte free of hydrofluoric acid
- Control of surface roughness to a microscale finish, Ra < 0.1  $\mu$ m
- Surface free from contamination after polishing
- Current distribution control that enables uniform polishing across the entire cavity surface
- Minimization of the absorption of hydrogen into the bulk material
- Controlled removal of at least 100  $\mu m$  of Nb during polishing

Development of Chemical-Mechanical Polishing for Superconducting Cavities, S. Mishra, M.J. Oreglia C. Spiro, ANL-FNAL-UofC Collaboration Meeting

#### **Sample Preparation**

#### 99.9% Nb foil

- Waterjet cut into coupons 25.4 mm x 25.4 mm x 3 mm
- SiC grinding paper used to prepare a consistent finish

#### **Effects of Flow Velocity on Ra**

There did not appear to be a significant effect of electrolyte velocity when electropolishing Nb

#### **Polarization Curves**

- 2-electrode polarization curves of Nb in various electrolytes
- Breakdown of the Nb was not observed
- Current measured assumed to be associated with water electrolysis and Nb anodization (Nb<sub>x</sub>O<sub>y</sub>)
- This data demonstrated the tenacity of the Nb oxide film under DC conditions



#### Key Advantages:

- Eliminate need for aggressive chemical baths, and the high cost associated with waste disposal
- Improve process control (constant driving force)
- A cost-effective and environmentally benign manufacturing process



### **Patents Filed**

Filed a utility patent (U.S. and International) on the Eco-Friendly

However, results at an electrolyte velocity of 0 cm/s suggested that there must be some degree of agitation in order to achieve uniform, smooth polishing of the Nb surface



# **Electropolishing Cell**

- Controlled flow
- Anode-cathode gap adjustable
- Flow meter device
- Filtration system included
- 30% wt  $H_2SO_4$  selected as electrolyte





#### **Surface Finish/Polishing Rates**

- Polishing rates range from 0.8 to 6  $\mu$ m/min
- Ra as low as 0.05 µm assessed over 12 mm length
- JLab assessed surface finish on coupon using AFM
  - Ra of < 1 nm over 10 x 10  $\mu$ m area
  - Extremely clean surface noted

# Surface Finish



polishing technology:

- Title: Electrochemical System and Method for Machining Strongly Passivating Metals
- U.S. Patent Application No. 10240426
- Foreign (PCT) Application No. PCT/US11/39354

Scan Size µm	Scan Number	R <sub>max</sub> nm	R <sub>a</sub> nm	RMS nm
50x50	1	35.00	2.71	3.34
	2	37.30	3.54	4.73
	3	69.66	3.74	4.69
10x10	1	22.59	2.25	2.87
	2	16.16	0.41	0.54
2x2	1	9.42	0.36	0.46

# **Time Study**





#### Summary

The FARADAYIC HF-free electropolishing process has shown feasibility to uniformly electropolish Nb to a microscale finish in an HF-free electrolyte.

The process is anticipated to be cost effective compared to conventional polishing methods due to the robust control mechanisms and minimal associated waste.

Acknowledgments: Work for this poster has been supported by a DOE Phase I SBIR program (DE-FG02-08ER85053) & Faraday Technology, Inc. research funding