Fermilab

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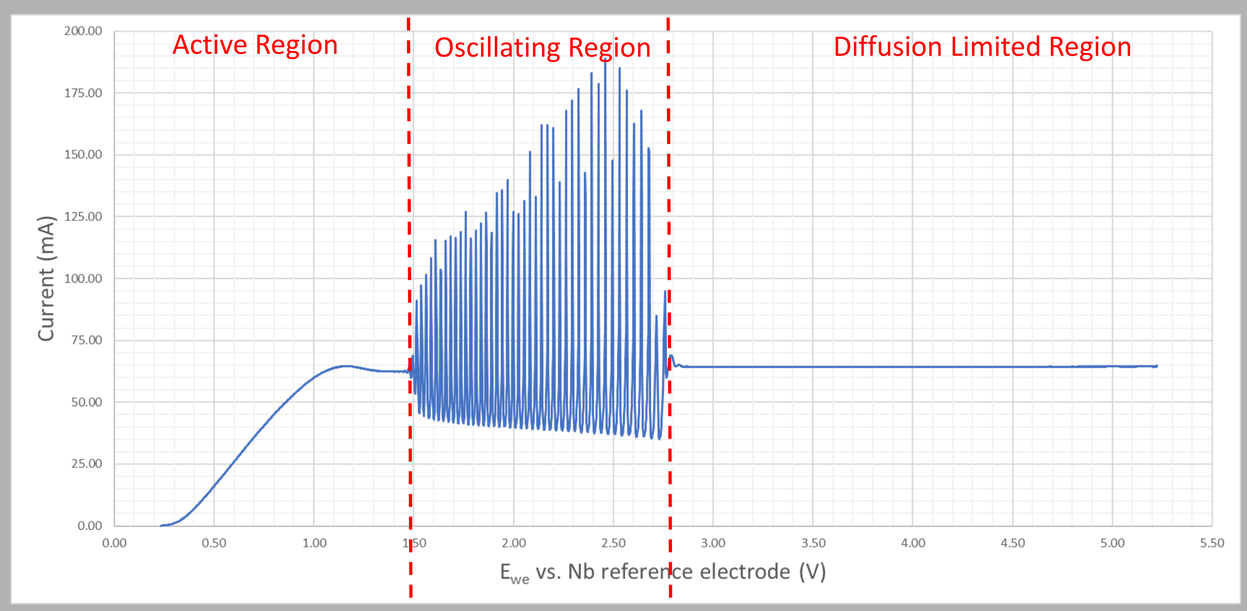
Studies on the Fundamental Mechanisms of Niobium Electropolishing

Introduction

Electropolishing is a well-established process for smoothing the inside surface of niobium SRF cavities. Despite this, the scientific knowledge regarding this process is lacking in many areas. The chemistry of the electropolishing reaction is highly complex due to the interactions between diffusion mechanisms, surface oxide structure, and multiple chemical species. Electropolished cavities sometimes end up with a rough surface finish or surface defects on the surface without any explanation. To improve the quality and repeatability of the electropolishing process we polish niobium samples at different potentials and temperatures. The samples surfaces are analyzed using optical and scanning electron microscopy. We find that electropolishing niobium samples at lower potentials leads to rough surface features such as pitting and grain etching. Some of the surface features seen show similarities to surface features found in niobium cavities.

Physical background and motivation

Niobium is a passivating metal, meaning that when an electric potential is applied, a protective oxide layer is formed on the surface preventing the metal from being dissolved. In HF containing solutions, the oxide is chemically dissolved by the HF. The mechanism for how this reaction occurs is dependent on many variables such as the etching potential, the temperature, and the amount of electrolyte mixing. The dissolution mechanisms can be broken down into two main types: active dissolution and diffusion limited dissolution. In the active region, the reaction rate is limited by the kinetics of the dissolution of the oxide film. In the diffusion limited region, the current is limited by the diffusion of HF to the surface of the Niobium. Separating these two regions is a region of instability where the current spontaneously oscillates. By studying the surface of samples polished in each of these regions we hope to gain a better understanding of what polishing parameters lead to a smooth niobium surface.

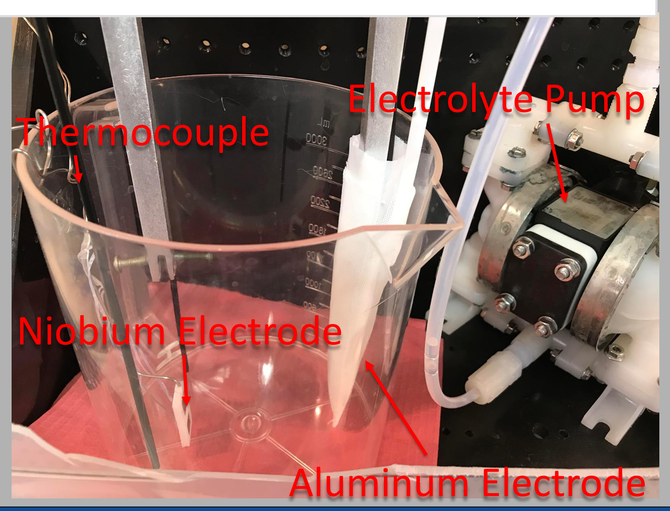


Experimental set-up

Samples were polished two at a time in a 1:9 mixture of 48% hydrofluoric acid and 99% sulfuric acid. The electropolishing potential is applied between the niobium working electrode and an aluminum counter electrode. The electrolyte is circulated by a pump and cooled by a heat exchanger to control sample temperature. The surface temperature of the niobium samples is monitored by a thermocouple attached directly to the surface and controlled by changing the acid temperature using a chiller.

An initial 18V electropolish removing 100 microns of material is performed on all samples to ensure an equal starting point. An additional five microns of material is removed during the experimental polishing conditions.

The resulting sample surface roughness is measured using an interferometric white light optical microscope and imaged using scanning electron microscopy.



Conclusion

Comparing the surface quality of niobium samples electropolished at different potentials and temperatures, we find that the surface roughness is strongly dependent on the potential and less dependent on the temperature. The surface of the samples becomes drastically different at lower potentials due to pitting and grain etching. The grain etching features have been seen in electropolished cavities as well, which may indicate that the potential was insufficient during the cavity EP.

References

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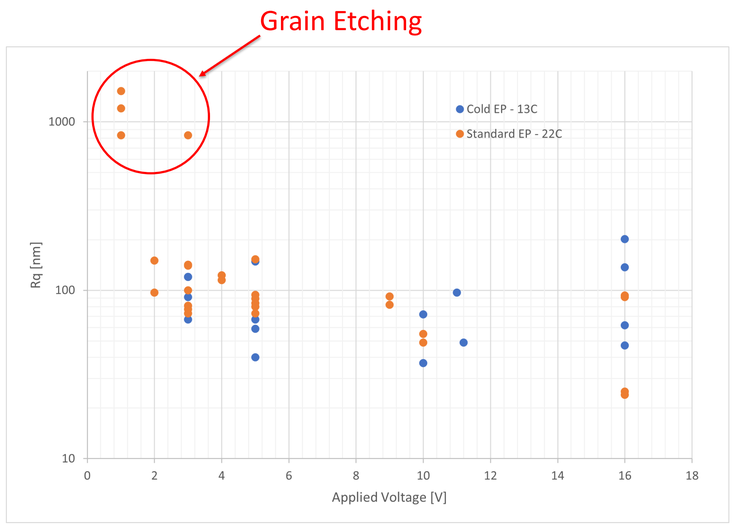
[2] A. Cano (2020, Feb). New insights on the SRF Nb cavities performance from spectroscopic data. An XPS study. Talk presented at TESLA Technology Collaboration TTC meeting, CERN, Geneva

Measurements and results

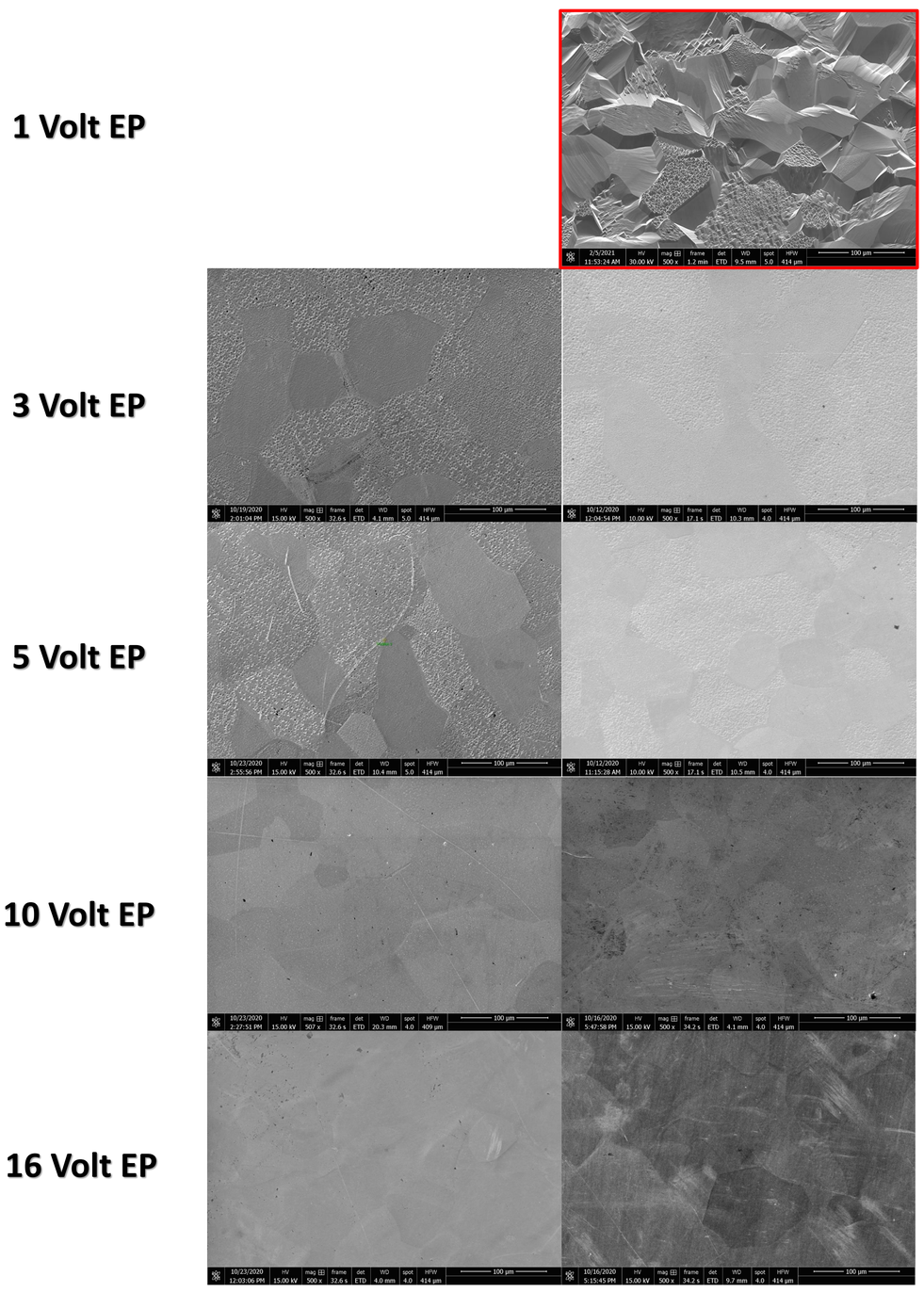
Sample EP Analysis



Samples electropolished in the low voltage range, 0-5V, end up with a rough surface finish due to pitting and grain etching.

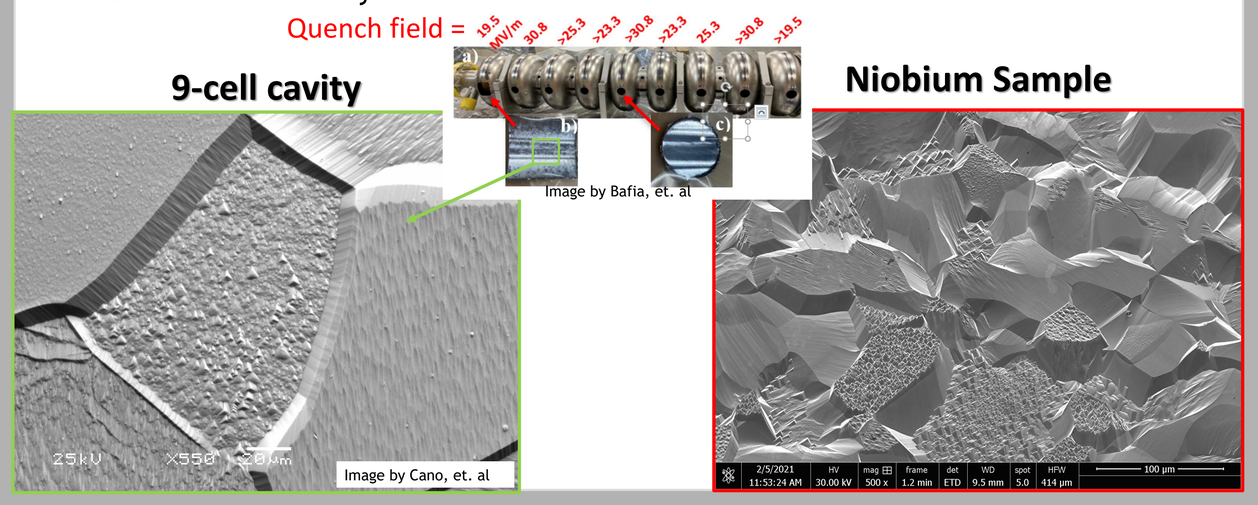
The smoothest sample was polished at 16V at a temperature of 22˚C. The cold temperature EP seems to match or outperform standard temperature EP at low to mid voltages, but samples become rougher at high voltage due to pitting.

Cold EP - 13C **Standard EP - 22C**



Grain Etching and Faceting After Low Voltage EP

Samples polished at very low voltages show a large variance in the etching rate between different crystallographic grains on the surface of the Niobium. Some grains also show faceting or rough surface morphology, presumably due to the high surface energy of these grains. This type of surface morphology has also been seen on certain areas in 9-cell cavities polished at higher voltages, suggesting that the potential distribution in the cavity is non-uniform and insufficient in certain areas.



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