

## STUDY OF GAS CLUSTER ION BEAM SURFACE TREATMENTS FOR MITIGATING RF BREAKDOWN \*

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### Abstract

Surface processing with high-energy Gas Cluster Ion Beams (GCIB) is investigated for increasing the high voltage breakdown strength of RF cavities and electrodes in general. Various GCIB treatments were studied for Nb, Cu, Stainless Steel and Ti electrode materials using beams of Ar, Ar+H<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub>, Ar+CH<sub>4</sub>, or O<sub>2</sub>+NF<sub>3</sub> clusters with accelerating potentials up to 35 kV. Etching using chemically active clusters such as NF<sub>3</sub> reduces the grain structure of Nb used for SRF cavities. Smoothing effects on Stainless steel and Ti substrates were evaluated using SEM and AFM imaging and show that 200 nm wide polishing scratch marks are greatly attenuated. Using a combination of Ar and O<sub>2</sub> processing for stainless steel electrode material, the oxide thickness and surface hardness are dramatically increased. The DC field emission of the 150-mm diameter sample of GCIB processed stainless steel electrode material was a factor of 10<sup>6</sup> less than a similar untreated sample.

### INTRODUCTION

Reaching higher gradients in SRF cavities has required ever more stringent surface preparation and yet the field emission of electrodes remains orders of magnitude greater than the prediction of the Fowler-Nordheim theory. It is widely believed that field emission and cluster emission from nano-scale surface imperfections trigger RF breakdown and are a limiting factor in achieving higher gradients [1-2]. We are investigating surface processing with high-energy Gas Cluster Ion Beams (GCIB) [3], a new technology that achieves an atomic level of smoothness on planar and non-planar surfaces, to increase the RF breakdown strength of SRF cavities and electrodes. With GCIB processing a surface is bombarded by clusters of atoms, typically 10,000 atoms in size with a charge of +3 and a velocity of 6 km/s [4,5].

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At this velocity the cluster impacts create shallow craters with diameters on the order of 10 nm [6]. With typical doses every point on the surface is affected many times resulting in effective smoothing. Asperities with dimensions of 350 nm diameter and 35 nm high on OFE Cu electrode material are easily removed using GCIB [7,8], and sub-micron-scale whisker-like structures should be especially susceptible to GCIB smoothing. Clusters of chemically active atoms are easily made and the extreme temperatures and pressures induced during the surface collisions provide a unique environment for chemical reactions. GCIB has been used in industries such as optical thin films, fixed disk memory systems, EUV lithography and semiconductor fabrication, for nano-scale smoothing, cleaning, etching, doping and deposition. For smoothing electrodes GCIB has the desirable feature that it is applied under high vacuum conditions. Thus GCIB processing could be applied after the high voltage structure has been assembled, baked out, and is under vacuum, as the last surface preparation and conditioning step before cavity operation. This paper presents results of GCIB processing of Nb and Stainless steel electrode materials, and the first field emission measurement of a GCIB processed electrode.

### TECHNIQUE AND RESULTS

Figure 1 shows schematically the GCIB beamline [9]. Clusters are formed as high-pressure gas expands supersonically into high vacuum inside a nozzle. The resulting jet of clusters is ionized by electron impacts and accelerated electrostatically (using as much as 35 kV potential in these experiments). A dipole magnet removes monomers and dimers from the beam. The electron flood provides neutralization of the space charge of the beam but was not needed in these experiments. Samples were mechanically scanned through the beam to assure uniform irradiation. The Faraday cup provided dosimetry. For the experiment, coupon samples of Nb electrode material prepared using BCP polishing, and highly polished 150-

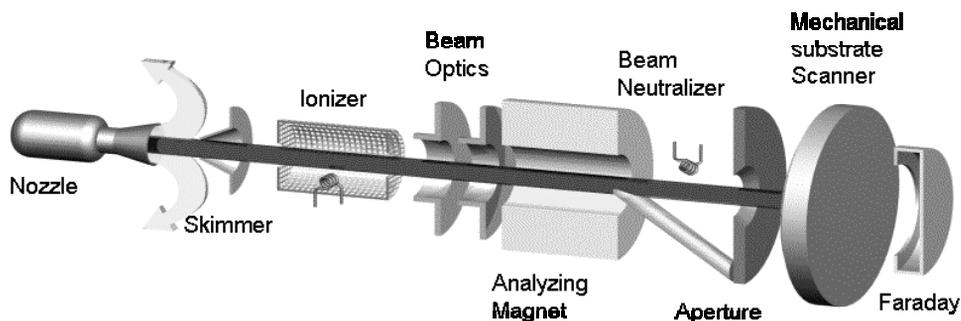


Figure 1 Schematic of GCIB beamline

mm diameter samples of stainless steel electrode material, similar to that used at Jefferson Laboratory in high-field photoemission electron guns, were provided by Cornell University.

The results of the GCIB processing were evaluated using standard techniques. AFM and SEM imaging were used to study nano-scale structure and smoothness. For stainless steel, XPS depth profiling was used to measure the oxide thickness, and a diamond tipped nano indenter with subsequent AFM imaging, was used to determine surface hardness. The DC field emission was measured at Jefferson Laboratory using the Large Area Electrode Test Chamber.

### *Nb samples*

The Nb sample had large grains of approximately 50  $\mu\text{m}$  lateral extent and projecting to a height of 2  $\mu\text{m}$  above the surface that were easily visible with an optical microscope. The grains are typical of BCP processing and the sharp edges of the grain boundaries are sites for field emission and local quenching in SRF cavities. Four separate GCIB processes were tested and compared to an unprocessed area of the sample: 1) High energy Ar clusters, used for chemically inert processing; 2) High energy Ar clusters followed by a mixture of  $\text{CH}_4$  in Ar; this is typically used to deposit diamond like carbon, 3) High energy Ar followed by a mixture of  $\text{H}_2$  in Ar; this is used to reduce oxides on the surface. 4) A mixture of  $\text{NF}_3$  gas in  $\text{O}_2$ ; an aggressive chemical etch with approximately 100 times greater etch rate than the physical sputtering/evaporation of Ar GCIB. Blunting of the grain edges was evaluated at several randomly selected locations using AFM measurement of the edges. The AFM maps of surface elevations were fit to a model to determine the angle and radius of curvature of the edge. The angles in Table 1 are the angle of deviation from flatness. The data show significant flattening of the grains, particularly using the fluorinated chemistry, and

using SEM imaging it was apparent that some of the grains were removed. The measured radii and the scatter in the measurements are large so changes in radius were not statistically significant.

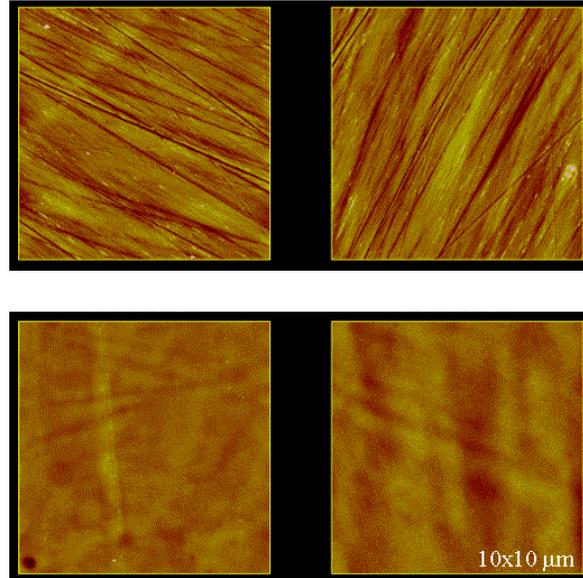


Figure 2 Before (upper row) and after (lower row) AFM images of stainless steel processed with Ar GCIB. The images are 20x20  $\mu\text{m}$  except as noted.

Table 1. Effect of GCIB treatments of Nb [10].

Treatment	Mean angle (deg)	Mean Radius ( $\mu\text{m}$ )	#
unprocessed	$12.80 \pm 1.5$	$2.71 \pm 1.0$	12
Ar	$11.97 \pm 1.8$	$2.88 \pm 0.7$	13
Ar then Ar + $\text{CH}_4$	$5.65 \pm 0.9$	$1.43 \pm 0.3$	8
Ar then Ar + $\text{H}_2$	$11.75 \pm 1.9$	$5.05 \pm 2.0$	6
$\text{NF}_3 + \text{O}_2$	$4.82 \pm 1.6$	$3.38 \pm 1.1$	8

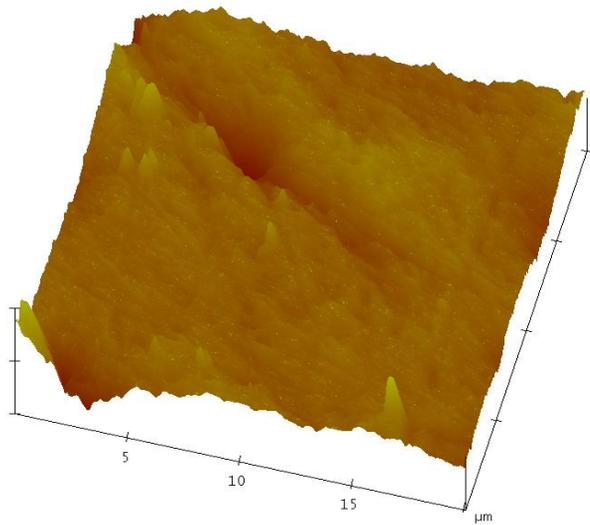


Figure 3 AFM image of a 20x20  $\mu\text{m}$  area on unprocessed stainless steel electrode. The vertical scale is 80 nm/division

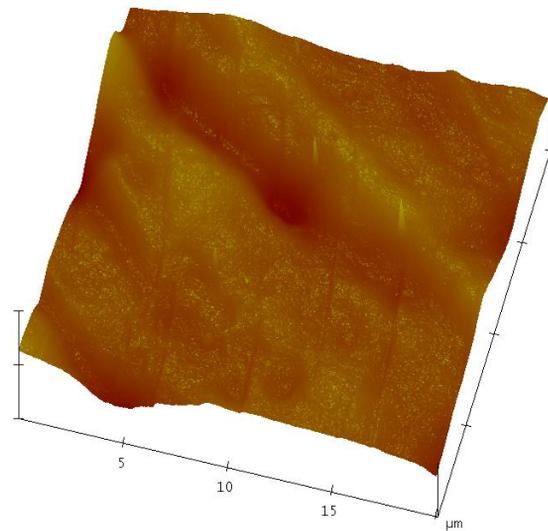


Figure 4 The same location after processing with high energy and low energy Ar GCIB.

### Stainless Steel electrode Material

The electrode samples were hand polished with diamond paste to approximately 1  $\mu\text{m}$  average surface finish. AFM scans revealed 200 nm wide scratch marks (Fig. 2) from the polishing compound (very similar results were seen on Ti electrode samples). On one sample a large feature was used as a marker to relocate the processed area allowing before (Fig. 3) and after (Fig. 4) images of GCIB processing at that location. The effect of the GCIB processing on submicron roughness is evident.

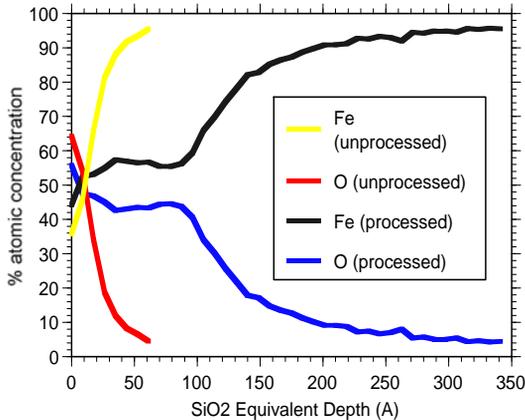


Figure 5 Elemental abundances of Fe and O as a function of depth for unprocessed and GCIB processed stainless steel. [11]

GCIB with O<sub>2</sub> clusters accelerated with 30 kV was used to increase the thickness of the surface oxide layer from 1.5 nm to more than 10 nm. The measurements (plotted in Fig. 5) were made using XPS with depth profiling. The O<sub>2</sub> treated surface was more than twice as hard as the unprocessed surface.

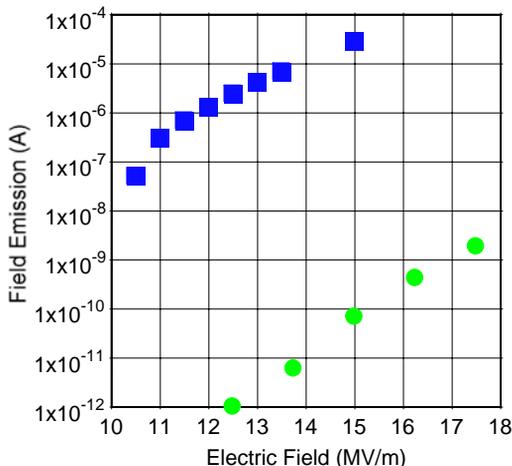


Figure 6 Field emission measurement for unprocessed (squares) and GCIB processed (circles) stainless steel electrodes[12].

Due to time constraints only one quick measurement of field emission was performed with a GCIB processed photocathode. This electrode was treated using a

sequence of high and then low energy Ar, for smoothing followed by high and then low energy O<sub>2</sub> to improve the oxide characteristics. Figure 6 shows a comparison of this electrode to a best-case non-processed electrode. In spite of the fact that the initial mechanical polish was inferior on the GCIB processed electrode, the processing caused a reduction of 6 orders of magnitude of the field emission. At this point the relative contributions of the surface smoothing and the thicker, harder oxide to this very encouraging result cannot be determined.

## CONCLUSIONS

From these and other experiments it is evident that GCIB is very effective at smoothing submicron level roughness on the electrodes and increasing oxide thickness. Further field emission and RF breakdown tests are planned to evaluate the efficacy and desirability of GCIB processed SRF cavities. In the case of high-field photoemission electron guns the first result is very encouraging. There are several other possible GCIB treatments for chemically altering surfaces yet to be tested. It remains to be seen if an electrode can be manufactured and tested that approaches the limits of the Fowler-Nordheim theory.

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