SIGNIFICANT REDUCTION IN FIELD EMISSION ON NIOBIUM SURFACES AFTER GCIB TREATMENT*

A.T. Wu+ and D.R. Swenson[1]
Thomas Jefferson National Accelerator Facility, Newport News, U.S.A.
[1]: Passport Systems Inc, 70 Treble Cove Road, Billerica, U.S.A.

Abstract

Field emission is one of the major obstacles for achieving constantly high accelerating gradient for Nb superconducting radio frequency (SRF) cavities, although various techniques and procedures have been adopted trying to keep the inner surfaces of Nb SRF cavities clean and free from field emission for a couple of decades in the past. In this report, it is shown that significant reductions in field emission on Nb surfaces can be achieved by means of a new surface treatment technique called gas cluster ion beam (GCIB). When a relevant treatment agent is selected with optimal treating parameters, it is demonstrated that a reduction in field emission as much as 87.5% is possible through measurements using a home-made scanning field emission microscope. Possible mechanism regarding the suppression of field emission on Nb surfaces by GCIB treatments will be discussed.

INTRODUCTION

Field emission has been a serious problem challenging the SRF community for a long time. Even up to now, many Nb based SRF cavities still suffer from field emission during the initial RF tests and have to be returned for cleaning or other surface treatments again. In regular accelerating structures, field emission often limits the cavity performance starting at a surface field of 20 MV/m. Heating from field emission increases exponentially with the surface field, leading to a dramatic decrease in the quality factor Q_o of the excitation curves of Nb SRF cavities.

The key here is to produce and maintain a clean surface that does not have micron or submicron particulates, chemical residues, and scratches or other sharp surface features. Various techniques [1] such as, for instance, clean room assembly, high pressure water rinse, ultrasonic cleaning with detergent, and recently dry ice cleaning [2] has been employed to mitigate particulates on the surfaces of Nb cavities.

In this report, it will be shown that there is now another technique that can be under our disposal for battling with field emission. This technique is called GCIB. It will be demonstrated through measurements that GCIB can suppress field emission on Nb surface to as much as 87.5% under a suitable treatment condition.

SAMPLES AND EXPERIMENTAL INSTRUMENTS

The samples used for this study were fabricated from the same Nb batch. These were special samples designed particularly for doing field emission scan using the scanning field emission microscope (SFEM) built at JLab. A typical sample is showed in Fig. l. These samples were treated by the standard BCP to remove 150 μm from the surfaces. Afterwards, the samples were rinsed by DI water and cleaned by ultrasound with micro for one hour followed by DI water rinsing again. Finally the sample surfaces were blown by a dry nitrogen gun.

Our SFEM is a home-made one as described in Ref.3. An important feature of the SFEM is that it is coupled with our scanning electron microscope (SEM) and energy dispersive x-ray (EDX) systems. It uses the SEM chamber as a load-lock entrance for samples. Through appropriate marking, the coordinate of a sample can be transferred from the sample holder of SEM to that of SFEM, which allows an emitter to be checked at the same location before and after field emission.

WORKING PRINCIPALS OF GCIB TECHNIQUE

The working principal of GCIB is schematically illustrated in Fig. 2. Various types of gases can be used for GCIB treatments. The gases can be inert such as Ar, Kr, Xe etc. or chemically reactive such as O_2, N_2, CO_2, NF_3, CH_4, B_2H_6 etc. that may react with the surfaces under treatments depending on what the application one has in mind. After selecting an appropriate gas species, the gas is forced through a nozzle that has a typical pressure of $7.6X10^{3}$ Torr on one side and a vacuum of $7.6X10^{-3}$ Torr on the other side. Therefore the gas undergoes a supersonic expansion adiabatically that slows down the relative velocity between the atoms of the gas, leading to the formation of a jet of clusters. A typical cluster contains atomic numbers ranging from 500 to 10,000 that are held together by van der Waals forces. A skimmer is then used to allow only the primary jet core of the clusters to pass through an ionizer. The clusters are ionized by an ionizer via mainly electron impacts and the positively charged clusters are electrostatically

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+ andywu@jlab.org

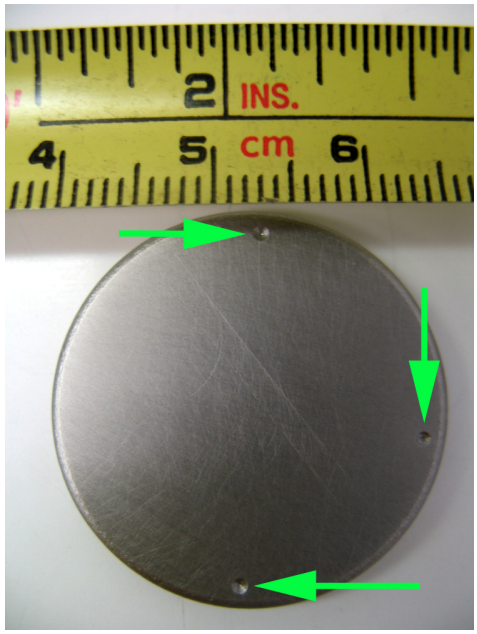

Figure 1: Standard Nb flat coupon used for the study described in this chapter. The arrows indicate the markings for coordinate identification.

accelerated via a typical voltage ranging from 2 kV to 35 kV and focused by a beam optics. Monomers and dimers are removed from the beam by a dipole magnet before the beam is neutralized with an electron flood. The aperture in Fig. 2 after the neutralizor is used to collect the the monometers and dimers. Surface GCIB treatments are done through mechanically scanning an object. Typically, the impact speed of the clusters to the surface of an object under GCIB treatements is 6.5 km/s, and the current of a gas cluster beam can be as high as 1 mA.

The selection of an appropriate gas species for doing GCIB treatment is very important. When an inert gas is chosen, the major effects on the treated surfaces are smoothing and asperity removal due to lateral sputtering. Chemical gases, on the other hand, can produce some additional effects such as, for instance, doping, etching, and depositing, etc. depending on the properties of the treated object and the gas species selected. Implantation is only limited to the top several atomic layers during GCIB treatments due to the low individual atomic energy. One can also combine the use of different gas species in a specific order for a particular application, although less work has been done in this research direction so far.

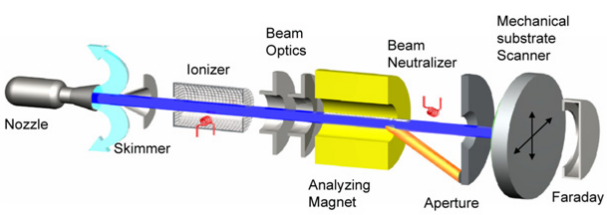

Figure 2: Schematic of working principal of GCIB.

For the study reported in this chapter, only Ar, O_2, N_2, and NF_3 were used in the GCIB treatments on Nb. Ar was selected because of its smoothing effect. O_2 GCIB is interesting due to the possible chemical reactions between O_2 and Nb and so is true also for N_2, although in case of using N_2 we were hoping that NbN could be formed on the treated surface since the superconducting transition temperature (T_c) is 16.2 K that is much higher than 9.2 K for Nb. NF_3 is expected to have a relatively higher etching and removal rates on Nb than those from other chemically reactive gas species.

EXPERIMENTAL RESULTS AND DISCUSSION

To study the effect of GCIB treatments on Nb, following experiment was performed: First standard Nb coupons as the one shown in Fig. 1 were fabricated from the same Nb sheet followed by the standard BCP 112 removal of 150 μm. The samples were covered partially via a 25 μm thick stainless steel for GCIB treatments employing O_2, Ar, and NF_3. After appropriate GCIB treatments, samples are transferred into SFEM measurement chamber via a load-lock entrance purged with flow nitrogen to prevent contamination on the surfaces of the samples.

Experimental results are shown in Fig. 3, Fig. 4, and Fig. 5. The sample used in Fig. 3 was masked into quadrants as shown in the figure. No GCIB treatment was done on the region marked "Unprocessed". "P1" region was treated by Ar. "P1+P2" region was treated by Ar first followed by O_2. O_2 GCIB treatment was done on "P2"

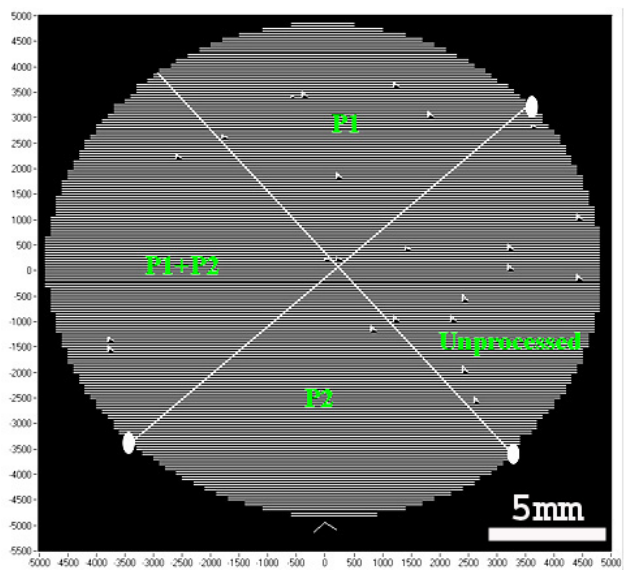

Figure 3: SFEM plot of field emitters on the surface of a BCP treated Nb coupon. The sample was masked into equal quadrants for treatments with Ar and/or O_2 GCIB or not treated as designated in the figure (see text for more details).

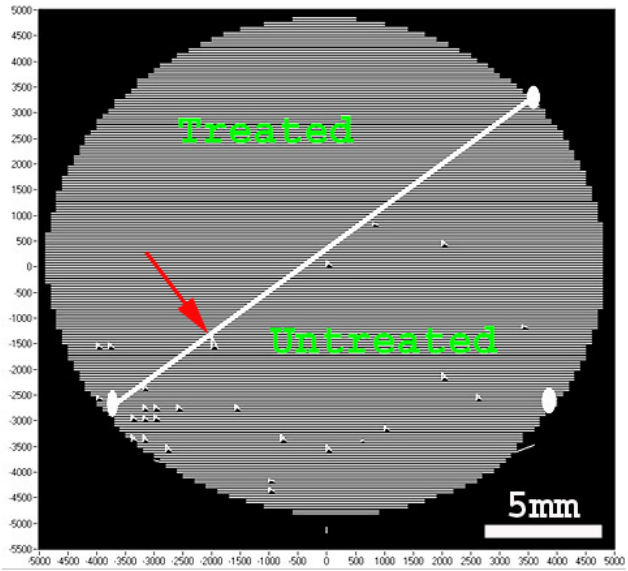

Figure 4: SFEM plot of field emitters on the surface of a BCP treated Nb coupon. Half of the coupon was treated with O_2 GCIB whereas the other half was not.

region. The locations of the triangles in these figures show where the emitters are and the height of a triangle indicates how strong the emitter is. The taller a triangle is the lower on-set field gradient the emitter has. All treated regions showed fewer emitters than the unprocessed quadrant. The number of emitters in each egion shows the following trend: P2<P1+P2<P1<Unprocessed. Comparing these results to a binomial distribution shows less than a 1 in 70 chance that this is a random

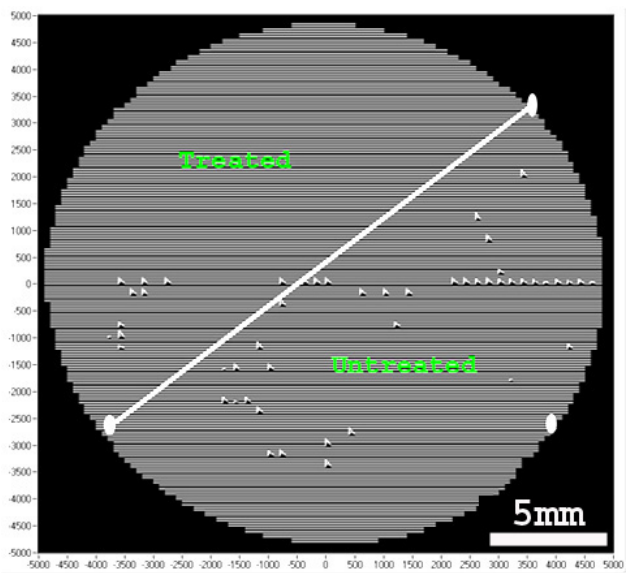

Figure 5: SFEM plot of field emitters on the surface of a BCP treated Nb coupon. Half of the coupon was treated with NF_3+O_2 GCIB whereas the other half was not.

distribution. It is remarkable to see that in the O_2 treated quadrant there is only one emitter that is located close to the unprocessed region. The measurement also suggests that O_2 treatment is more effective in reduce the number of field emitters. Encouraged by the first test, another coupon was treated by O_2 GCIB. The result of SFEM scans is showed in Fig. 4. In this case, half of the coupon surface was covered. Again a dramatic reduction in the number of field emitters was found on the treated region. By assuming a non-preferential distribution of the emitters on the Nb surface before the treatment, the reduction rate for O_2 is 87.5%. The most important difference between Ar and O_2 is that O_2 is reactive with Nb whereas Ar is not. This inspired us to use a more reactive gas species for treating Nb surface. Ref. 4 demonstrated that NF_3+O_2 can significantly etch Nb and blunt the angles of the grains that protrude from the surface. Therefore NF_3+O_2 was adopted for the next treatment. Fig. 5 shows the result of SFEM scans on the Nb coupon where half of the surface was covered. Reduction in field emitter number is again seen for the treated half. The reduction rate is 75.0% that is less than 87.5% for the O_2 treated region.

These results seem to imply that the smoothing effect is not the main reason responsible for the reduction as evidenced from Ar GCIB treatment. Chemical reaction is clearly important. But this does not mean that the more chemical reaction the better since the reduction in field emission is more for O_2 treated region than that in NF_3+O_2 treated region. We tentatively attribute the effectiveness of O_2 treatment to the modifications of the surface oxide layer structure on Nb surface as reported in another paper [5]. We believe that the following three effects from GCIB treatments contribute to the reduction in field emission. First effect is the smoothing effect of GCIB treatment. GCIB treatments can remove sharp tips

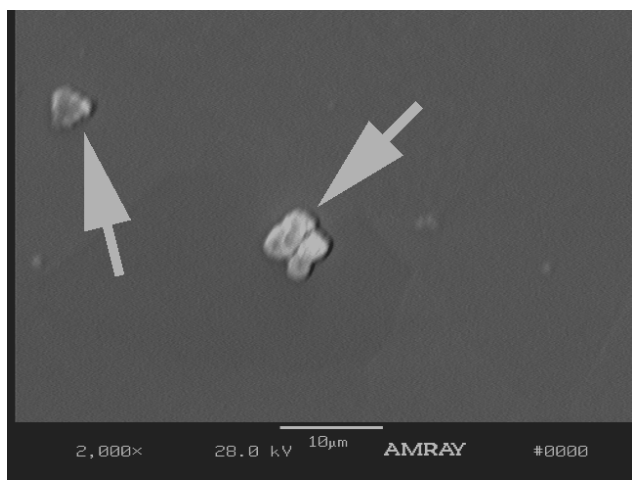

Figure 6: SEM image taken on an O_2 GCIB treated Nb surface. Two potential emitters as indicated by the arrows were suppressed by the treatment via removing sharp edges and tips.

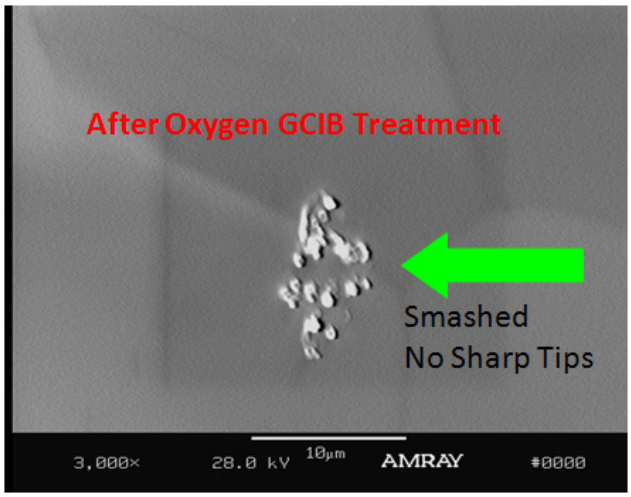

Figure 7: SEM image taken on an O_2 GCIB treated Nb surface. The arrow indicates a potential emitter being "smashed" into pieces as if it were stepped on by a heavy Japanese sumo wrestler.

or edges so as to suppress field emission. A typical example is shown in Fig. 6. Chemically reactive smoothing effect seems to be more effective in reducing the number of emitters than pure mechanical one does as in the case of Ar. The second effect is the so-called "smashing effect" as shown in Fig. 7 where a potential emitter in the oxide treated region was found to be suppressed by the bombardment of O_2 clusters and broke into pieces as if it were stepped on by a heavy sumo wrestler. The third effect is the modification of the surface chemical composition, especially the increase of the thickness of the surface insulating layer of Nb such as in the case of O_2 (see Ref. 5 for more details).

SEM and EDX examinations were done on the emitters inside the treated and untreated regions of Figs. 4 and

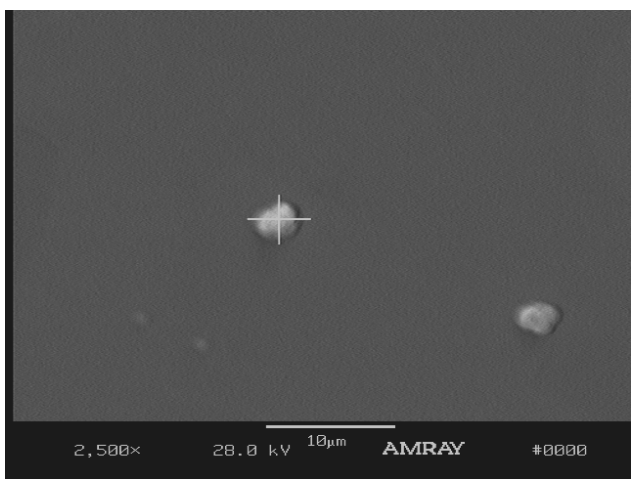

Figure 8: Close-up SEM image of two niobium oxide particles on the Nb surface treated by high energy NF_3+O_2 GCIB. The two niobium oxide particles appeared to have smooth surfaces and to be embedded into the surface of the Nb coupon.

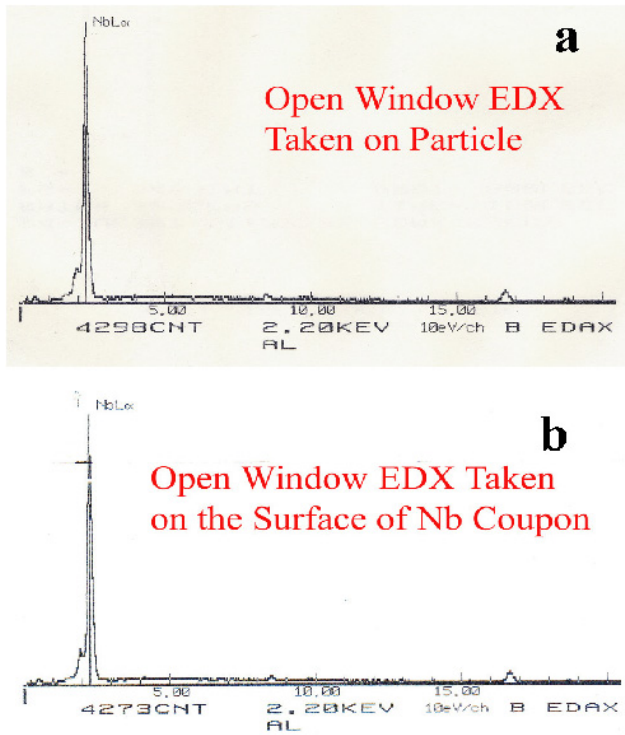

Figure 9: EDX spectra taken on a) one of the particle shown in Fig. 8, b) on the surface of the Nb sample.

5 hoping to find more clues about the characteristics of the emitters, possible origins, and why some emitters were still active after the GCIB treatments. For the untreated region in Fig. 4, 14 different elements were detected from the total 24 emitters in this region. The most frequent found elements are S, Fe, Cl, Al, Mg, and Si. It is interesting to notice that S, Fe, Al, and Si are also the most frequent found elements [6] in the particulates collected in a filter form high pressure water rinse line for Nb SRF cavities at Jlab. S, Si, Al, and Cl are the most frequent found elements in the particulates [7] collected from the rear side of a vacuum pump line for Nb SRF cavities at JLab. Therefore, it is plausible that the emitters are mostly air-born particles or dusts, and/or residuals from BCP treatment, and/or deposits from rinse water. Five different elements found from the emitters in the treated region. It appears that there is not a correlation between the elements detected in the untreated and treated regions. The sizes of the emitters in the untreated region range from several tens micron to submicron. Among the 24 emitters detected, the emitters with larger sizes tend to have a lower emission onset field. For instance, the emitter indicated by the red arrow in Fig.4 has field emission onset at 17 MV/m whereas other emitters that have onset field at least 89 MV/m have a typical length scale less than 10 µm.

For the untreated region in Fig. 5, 15 different elements were found from the 40 emitters of this region. Most of the 15 elements were also seen in the untreated region in Fig. 4 except Cu, Ag, and Ni, implying therefore that the emitters might originate from the same sources as those

for Fig. 4. Almost all the elements detected in the untreated region appeared in the treated region except Ag. This is in agreement with the hypothesis that the field emitters are randomly distributed over the surface of an Nb coupon. Unlike the oxygen GCIB treated sample where only 3 emitters were found in the treated region, here there were 10 emitters. Therefore the chance for all the elements detected in the untreated region to appear in the NF_3 treated region increases substantially. One undesirable feature found in the treated area in Fig. 5 was a lot of small niobium oxide particles. Those particles were presumably a result of NF_3 bombardment and were not active emitters. Close examine revealed that those niobium particles had very smooth surfaces (see a typical example in Fig.8). They seemed to be embedded in the surface the Nb coupon. The particles are niobium pent-oxides since the oxygen peak intensity in the open window EDX spectrum (Fig. 9a) taken at the particle is the same as that (Fig. 9b) taken on the surface of the Nb coupon. It is plausible that the O_2 GCIB treatment after NF_3 turns these particles from Nb or Nb suboxides into pent-oxides and smoothen their surfaces. This also explains why NF_3+O_2 GCIB treatment has a relatively larger etching rate [8].

It is also interesting to note that most of the active emitters are particulates consisting of more than one metallic or semiconductor elements and Nb itself can be an emitter if it exists as a particle.

SUMMARY

It was demonstrated in this paper that GCIB could be used to treat Nb surfaces for the purpose of reducing field emission. Experimental results obtained from an SFEM revealed that under a suitable condition a reduction in field emission as much as 87.5% could be obtained. The mechanism responsible for the reduction in field emission was discussed.

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