DRY ICE CLEANING FOR SRF APPLICATIONS

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Abstract

Dry ice cleaning using the sublimation-impulse method removes particulate and film contaminations without any residues. The gases involved in this process, i.e. CO_2 and N_2, are chemically inert, thus no negative impact on materials like niobium, copper, alumina etc. used in a superconducting (s.c.) accelerator is expected. As high gradients in s.c. cavities require surfaces free of enhanced field emission, the dry-ice cleaning process was applied to a series of intentionally and defined contaminated niobium samples. The cleaning effect was investigated by means of a dc field emission scanning microscope and an optical microscope, comparing the onset fields and number of emitters as well as the number of particles before and after the cleaning process. A drastic reduction of field emission up to fields of 100 MV/m and of residual particles was achieved without any kind of surface damage by the dry ice jet. These first results encourage us to improve and develop the dry ice cleaning technique for s.c. cavities, for water-sensitive components like rf coupler ceramics and for the final treatment of s.c. accelerating structures.

1 INTRODUCTION

At present enhanced field emission (EFE) imposes the major high gradient limitation of superconducting accelerator structures, e.g. at about 25 MV/m for the TESLA nine-cell niobium cavities [1]. In order to push their performance to gradients of 35 MV/m required for TESLA 800 [2], electric surface fields of at least 70 MV/m should be achieved reliably without EFE. Therefore, advanced final cleaning procedures must be developed to avoid surface contamination with particles without introducing any new surface damage [1,3,4]. Though high pressure rinsing with ultrapure water has proven to be a powerful technique to reduce the EFE of cavities [4,6] and Nb samples [5], dry-ice cleaning might have additional cleaning potential. Moreover it avoids a wet cavity surface with its enhanced sensibility against recontamination and should be applicable to ceramics (coupler windows) without loosing the gain of an earlier conditioning. Due to these properties dry ice cleaning is considered as very attractive for the final treatment of the horizontally assembled cavity with its power coupler.

2 DRY ICE CLEANING

A jet of pure carbon dioxide snow loosens and removes different types of surface contaminations by its unique combination of mechanical, thermal and chemical effects. The cleaning process acts local, mild, dry, without residues requiring no additional cleaning agent. The spontaneous relaxation of liquid carbon dioxid leaving the nozzle results in a snow/gas mixture with 45 % snow and a temperature of 194.3 K (-78.9°C). This jet is surrounded by supersonic nitrogen, which firstly gives an acceleration and focussing of the jet and secondly prevents the condensation of humidity at the cleaned object. The cleaning effect is based on thermomechanical and chemomechanical forces. The former are created by three effects: brittling the contamination as a result of rapid cooling (shock-freezing), the tough pressure and shearing forces due to the high momentum of the snow crystals hitting the surface and the powerful rinsing due to the 500 times increased volume after sublimation. Particles down to 100 nm can be removed. Chemomechanical forces occur, when high momentum snow particles hitting the surface partially are melting at the point of impact. In its liquid phase carbon dioxid is a good solvent for non-polar chemicals, especially for hydrocarbons and silicons. The thermal effect of shock-freezing is thereby directly correlated with the snow intensity, while the mechanical effect however depends on the velocity and angle of the jet and the chemical effect depends on the momentum of the crystals. An optimal cleaning impact is achieved, if the thermal gradient between contamination and substrate is high. Therefore pulsing the jet instead of continuous operation may be useful. To avoid recontamination an effective and well-defined exhaust system is necessary. In summary the advantages of the carbon dioxid dry ice cleaning are:

- dry cleaning process,
- no cleaning agents,
- removal of particulate and film contaminations,
- no polluting residues.

3 EXPERIMENTAL

Flat samples with a diameter of 28 mm were machined from high purity niobium (RRR = 300) and etched 80 µm with standard BCP ($HF:HNO_3:H_3PO_4$ volume ratio 1:1:2).

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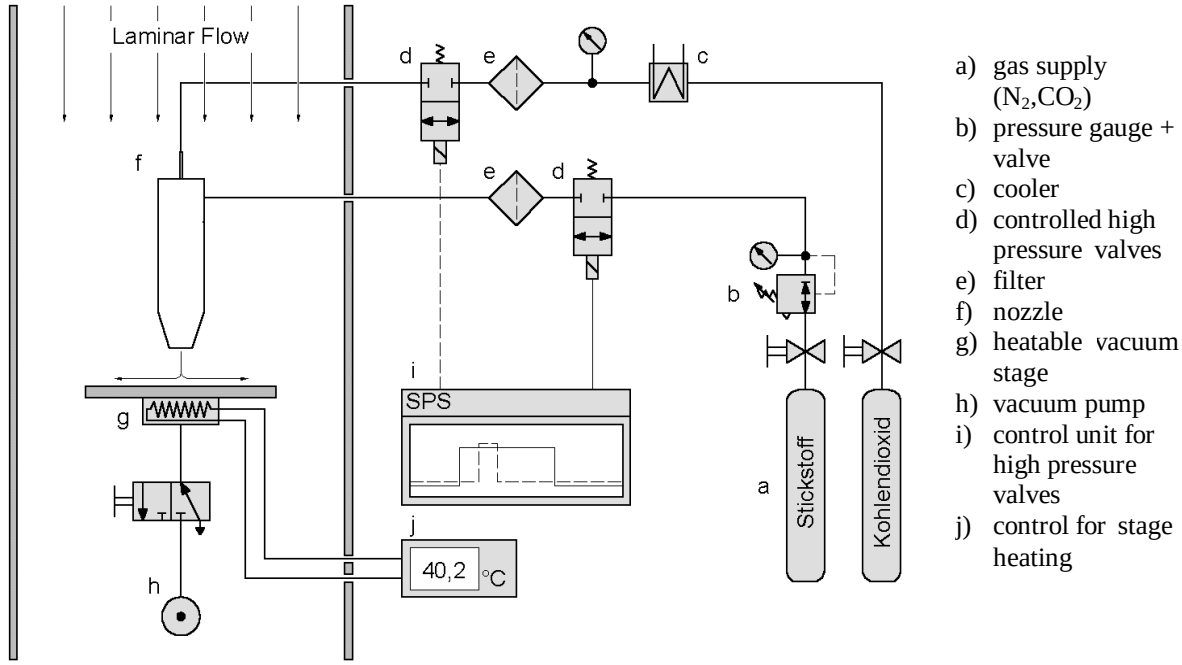

Figure 1: Schematic of the prototype set up for the dry ice cleaning of flat samples.

In order to get typical EFE, each test sequence started with a surface treatment (see 3.1), i.e. etching and rinsing inside a cavity or intentional contamination with particles, which are typically ambient during the assembly of accelerating structures. At first, these samples were inspected with an optical microscope under cleanroom conditions (class 10000). Then their EFE properties were determined with the field emission scanning microscope (FESM)(see 3.3). After dry ice cleaning in a class 10 cleanroom (see 3.2), the cleaning effect was investigated with the FESM and an optical microscope (class 10) again. For the sample transfer between the laboratories, an approved clamped cap system [5] was used and opened under cleanroom or UHV conditions only.

3.1 Sample preparation

Up to now the samples went through two preparation sequences. For the first sequence six samples were in use. Two samples (#1 and #2) were additionally etched 20 µm (BCP 1:1:2) inside a TTF nine-cell cavity [3] including a thorough rinse with ultrapure water, but no high pressure rinsing. The samples #4 and #5 were artificially contaminated with latex spheres of 3.2 µm mean size and metal-oxide particles of 1-80 µm size, which might be represenative for gloves and ceramics respectively. For comparison, the samples #7 and #0 were polluted with stainless steel particles of 10-65 µm size, which were caused by abrasion of nuts and bolts. After finishing the measurements of the first sequence, three of the six samples were etched 10 µm (BCP 1:1:2) again for the second sequence. This batch was contaminated with both iron (< 50 µm) and copper particles (mean size 6-8 µm), which occur near Con-Flat flanges. The resulting particle number density was determined with an optical microscope by counting an arbitrarily chosen surface area of about 20 mm^2 on each sample. It varied between 400/mm^2 and 1100/mm^2 for particle sizes above 2 µm.

3.2 Dry ice cleaning apparatus

The dry ice cleaning of the samples was performed with a prototype apparatus as shown in figure 1, which has been developed for non-destructive cleaning of electronic chips. The process parameters used for the cleaning of the samples are listed in table 1.

Table 1: Dry ice cleaning parameters

CO_2-pressure	55 bar
N_2-pressure	8/12 bar
Jet diameter	3 mm
Distance between nozzle and sample	20 mm
Direction of jet	Perpendicular to surface
Velocity sample to jet	5 mm/s

3.3. Field emission scanning microscope

The EFE characteristics of all samples were measured with a field emission scanning microscope (FESM) inside an UHV-chamber at a pressure of $p < 10^{-9}$ mbar, which also contains a SEM and AES for emitter identification. The FE uniformity was investigated by scanning of the extraction voltage U < 10 kV at constant current I = 1 nA over the central part of the samples, i.e. 8x8 mm^2 or 12x12 mm^2 depending on the number of large particles and the residual surface unflatness. These U(x,y) scans were performed with a ∅100 µm tungsten anode at a

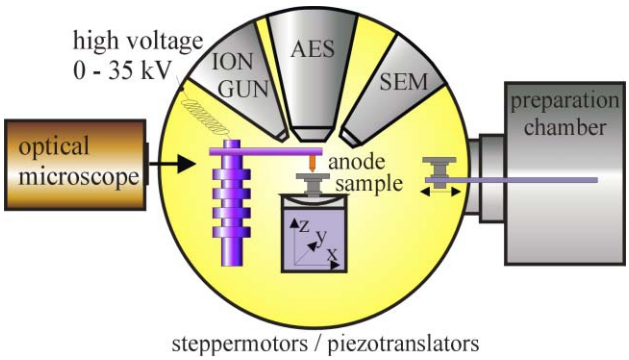

Figure 2: Schematic of the FESM in the focus of an UHV surface analysis chamber with scanning electron microscope (SEM), Auger electron spectroscopy (AES) and ion gun. The FESM consists of a set of W- needles opposite to a 3D sample positioning system driven by stepper motors and piezotranslators. The long distance optical microscope serves for sample control, and the preparation chamber as load-lock for the sample transfer.

nominal distance of 100 µm to the cathode surface. A fast high voltage regulation prevents discharges and excessive currents, thus keeping most of the emitters undestroyed. In order to obtain a significant number of emitters on the samples, the applied electric field was stepwisely increased up to 100 MV/m. Selected emitters were then located with a ∅4 µm W-needle to determine their local FE onset field and to look for their morphology by means of the in-situ SEM.

4 RESULTS AND DISCUSSION

The results of the first preparation sequence can be divided in two groups. Sample #1, prepared inside a cavity, and the samples #7 and #0, contaminated with steel particles, showed a significant number of field emission spots before the dry ice cleaning. This can be clearly seen in the voltage maps of figure 3. At least the strongest emitters are correlated with particles visible with the in-situ SEM. In confirmation of earlier results [7] by far not all particles emit. Surprisingly, sample #2 showed much less stable emitters than #1, but some more sparks, probably due to a reduced sticking of particles on the sample surface.

Table 2: Normalized number density of field emitters

Sample	**Field emitters per cm^2 before dry ice at E = 100 MV/m**	**Field emitters per cm^2 after dry ice at E = 100 MV/m**
# 1	12	0
# 7	70	7
# 0	6	4
# 2	1	Not measured
# 4	0	Not measured
# 5	2	Not measured

In comparison, samples #4 and #5, contaminated with latex and metal-oxide particles, show nearly no dc EFE in the voltage maps up to 100 MV/m before the dry ice cleaning. This result is expected for electrically insulating particles because they hardly provide field enhancement. As consequence, only the strongly emitting samples were scanned after the dry ice cleaning. The resulting normalized emitter number density of all maps is summarized in table 2. Obviously, it is drastically reduced by the dry ice cleaning procedure. It should be mentioned that local onset fields cannot be derived from the maps due to switch-on and particle size effects. However, they were partially measured for the dry ice cleaned samples, and found to be above 80 MV/m.

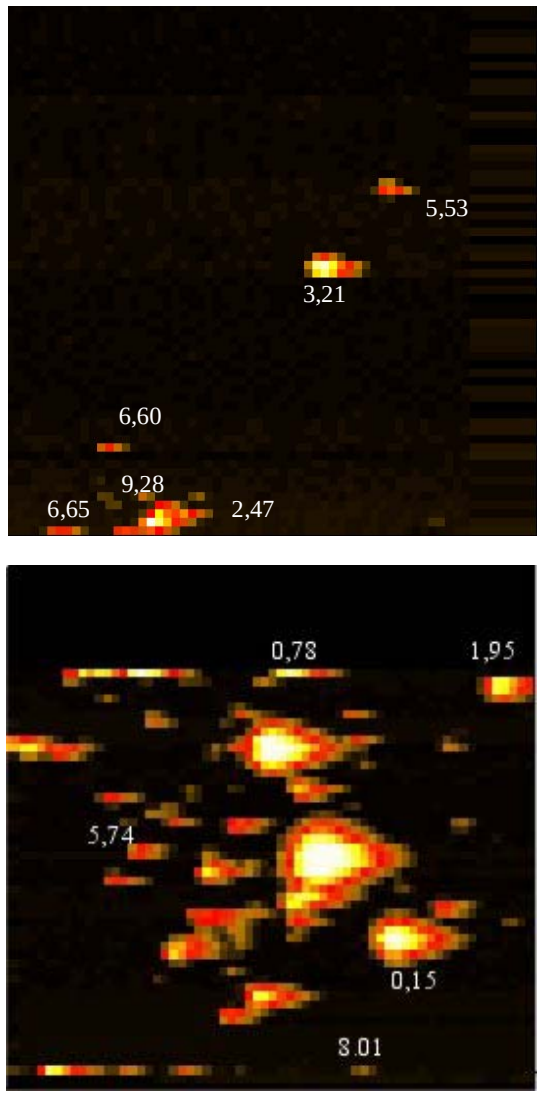

Figure 3: U(x,y) maps over 8x8 mm^2 of sample #1 (top) and #7 (bottom) at E = 100 MV/m before the dry ice cleaning. The numbers give the voltages in kV required for 1 nA current at 100 µm nominal electrode spacing.

As the second preparation sequence started recently, up to now no sample is gone through the complete test sequence and no FESM results are available yet. One sample of this sequence, however, has been optically inspected already as well as all six samples of the first sequence before and after dry ice cleaning. The resulting images confirm and substantiate the cleaning effect clearly, as demonstrated in figure 4. Only very few and small particles survive after the dry ice cleaning. Moreover, no kind of mechanical damage has been found on the niobium surface after this treatment.

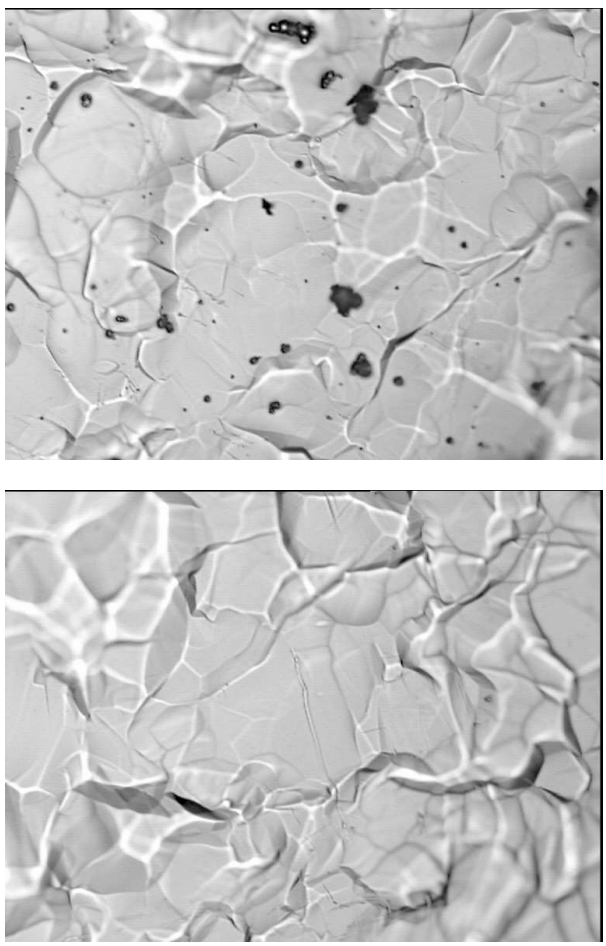

Figure 4: Optical microscope images (magnification 500x) of sample #5 contaminated with Fe and Cu particles before (top) and after (bottom) dry ice cleaning.

5 CONCLUSION

The first results obtained with the dry ice cleaning of niobium surfaces are very promising within the limited number of sample investigations. The number of particles as well as the number of field emitters have been reduced significantly. No surface damage by dry ice cleaning has been observed. In order to confirm this achievement, more measurements on other kinds of particulate contaminations are in progress. A comparison with the well-proven high pressure water rinsing for particle contaminated, strongly emitting surfaces is in preparation. The feasibility of a simple cleaning set-up for single- and 3-cell cavities will be checked in the near future. The application of dry ice cleaning for water-sensitive components like coupler ceramics as well as its use during the final cleanroom assembly is under investigation.

6 REFERENCES

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