

## FIRST EXPERIENCE WITH DRY-ICE CLEANING ON SRF CAVITIES

D. Reschke<sup>#</sup>, A. Brinkmann, DESY, D-22603 Hamburg, Germany

D. Werner, Fraunhofer IPA, D-70569 Stuttgart, Germany

G. Müller, FB C, University of Wuppertal, D-42097 Wuppertal, Germany

### Abstract

The surface of superconducting (s.c.) accelerator cavities must be cleaned from any kind of contamination, like particles or chemical residues. Contaminations might act as centers for field emission, thus limiting the maximum gradient. Today's final cleaning is based on high pressure rinsing with ultra pure water. Application of dry-ice cleaning might result in additional cleaning potential. Dry-ice cleaning relies on the sublimation-impulse method and removes particulate and film contaminations without residues. As first qualifying step intentionally contaminated niobium samples were treated by dry-ice cleaning. It resulted in a drastic reduction of the particle numbers and of DC field emission up to fields of 100 MV/m. The dry-ice jet caused no observable surface damage. First cleaning tests on single-cell cavities showed Q-values at low fields up to  $4 \times 10^{10}$  at 1.8K. Gradients up to 33 MV/m were achieved, but field emission still is the limiting effect. Further tests are planned to optimise the dry-ice cleaning technique.

### INTRODUCTION

Despite the substantial improvement of the preparation procedures, enhanced field emission still imposes the major high gradient limitation of superconducting accelerator structures, e.g. for the 1.3 GHz nine-cell structures used in TTF at DESY [1]. In order to achieve a gradient of 23 MV/m required for the XFEL [2] or to push the performance to 35 MV/m required for TESLA [3], electric surface fields of at least 46 MV/m and 70 MV/m, respectively, should be achieved reliably without field emission loading of the Q-value. Therefore, advanced final cleaning and handling procedures must be applied to avoid surface contaminations like particles, hydrocarbons, etc. and mechanical damages like scratches. The above mentioned have been shown to cause enhanced field emission (EFE) at the envisaged field levels. Though high pressure rinsing with ultrapure water has been proven to be a powerful technique to reduce the enhanced field emission of cavities [1, 4, 5], dry-ice cleaning might have additional cleaning potential. Moreover it avoids a wet cavity surface with its enhanced sensitivity against recontamination. It should be applicable to ceramics (coupler windows) without losing the gain of an earlier conditioning. Due to these properties dry-ice cleaning is considered as very attractive for the final treatment of horizontally assembled cavities with its power coupler. According to the stimulating first results on flat Nb samples [6], the adoption of this technique to srf cavities will be reported in this paper.

<sup>#</sup>detlef.reschke@desy.de

### 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 locally, mildly, dryly, without residues and requires no additional cleaning agent. The spontaneous relaxation of liquid carbon dioxide leaving the nozzle results in a snow/gas mixture with app. 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 partially the condensation of humidity at the cleaned object. The cleaning effect is based on thermo mechanical and chemo mechanical 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. Chemo mechanical forces occur, when high momentum snow particles hitting the surface partially are melting at the point of impact. In its liquid phase carbon dioxide is a good solvent for non-polar chemicals, especially for hydrocarbons and silicones. The thermal effect of shock-freezing is thereby directly correlated with the snow intensity. 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. To avoid recontamination an effective and well-defined exhaust system is necessary. In summary the advantages of the carbon dioxide dry-ice cleaning are:

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

### NB SAMPLE EXPERIMENTS

Flat samples with a diameter of 28 mm were machined from high purity niobium (RRR = 300) and etched 80  $\mu\text{m}$  with standard BCP (HF:HNO<sub>3</sub>:H<sub>3</sub>PO<sub>4</sub> volume ratio 1:1:2). In order to get typical EFE, each test sequence started with a new surface treatment, i.e. etching and rinsing inside a cavity [7] or intentional contamination with particles [6]. The chosen particle materials are typically ambient during the assembly of accelerating structures like latex (gloves), metal oxides, copper, iron and stainless steel. At first, these samples were inspected with

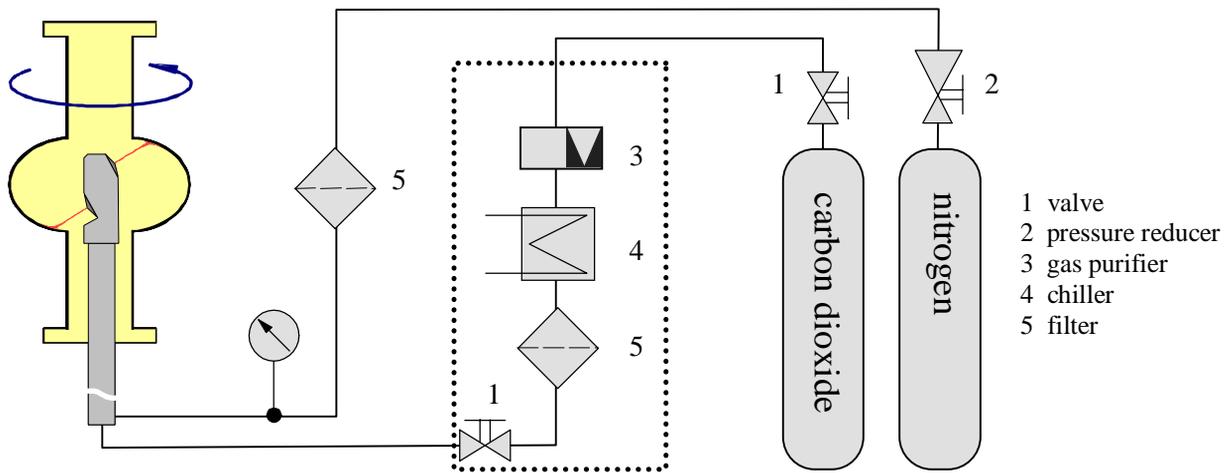


Figure 1: Schematic of the prototype set up for dry-ice cleaning of monocell cavities.

an optical microscope under cleanroom conditions (class 10000). The particle number densities of the intentionally contaminated samples varied between 400/mm<sup>2</sup> and 1100/mm<sup>2</sup>. Then the EFE properties were determined with a field emission scanning microscope [8] (FESM). The dry-ice cleaning was performed in a class 10 cleanroom with a one-nozzle system perpendicular to the surface [6]. 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 [7] was used and opened under cleanroom or UHV conditions only.

The results achieved on 9 samples can be summarized as follows:

- a) Two samples etched and ultrapure water rinsed inside a nine-cell cavity showed few emission spots before but none after dry-ice cleaning up to 100 MV/m.
- b) An intentional contamination with metal particles of 5 samples created a significant number of field emission spots up to 101 at 80 MV/m before dry ice cleaning. After dry-ice cleaning, both the number of microscopically visible particles as well as the number of field emission sites decreased dramatically. On two samples no field emission was found up to 100 MV/m (typ. scanning area 6 mm x 6 mm).
- c) Two samples contaminated with latex and metal-oxide particles showed nearly no dc EFE up to 100MV/m before dry-ice cleaning and were not investigated further.
- d) No kind of mechanical damage was found on the dry-ice cleaned niobium samples.

## CAVITY EXPERIMENTS

### *Dry-Ice Cleaning Apparatus and Procedures*

An overview of the prototype set-up for cleaning of monocell cavities is given in figure 1. A major component is the chiller/purifier system for purifying and particle filtering of the technical-grade CO<sub>2</sub>. The temperature of

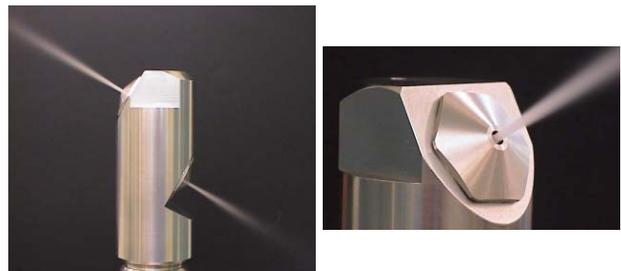


Figure 2: Nozzle system for cavity cleaning.

the liquid CO<sub>2</sub> is reduced below -5°C to increase the fraction of snow. As the cooling capacity of the available chiller was insufficient, the temperature could not be kept constant (see below). Pure nitrogen is supplied up to a pressure of 18 bar and particle filtered to < 0,05 µm. A dedicated nozzle system for cavity cleaning was developed using two opposite nozzles spraying with an angle of 30 degrees upward and downward with respect to the horizontal plane for optimised cleaning of the cavity iris area (Figs. 2 + 3). The used temporary motion unit caused severe restrictions of the experiments due to a missing gas extraction system, an insufficient heating possibility of the cavity and bearings generating particles.

Each cavity was rotated and cleaned at least one time up and down. The cleaning duration was varied between 10 to 50 minutes. Due to the inadequate chiller the cleaning process had to be interrupted and restarted several times causing an enhanced danger of particle contamination. After the dry-ice process, the final assembly, evacuating and leak-checking was performed in a class 10 cleanroom.

Table 1: Dry ice cleaning parameters

CO <sub>2</sub> -pressure	~ 50 bar
N <sub>2</sub> -pressure	12 - 18 bar
Particle filtration	< 0,05 µm
Temperature of liquid CO <sub>2</sub>	-5° - -40° C
Environment of cleaning	Laminar flow class 10



Figure 3: Nozzle test in a cut NbCu cavity (top) and in the beam tube of a Nb monocell (bottom).

### RF Results

In total 11 niobium monocell cavities were rf tested after dry-ice cleaning. All tests show high Q-values above  $10^{10}$  at 2 K typical for superconducting niobium cavities at 1.3 GHz. The highest Q-value of  $4 \cdot 10^{10}$  at 1.8 K proves that no surface contamination is caused by dry-ice cleaning. Though gradients up to  $E_{acc} = 33$  MV/m are achieved (fig. 4), still field emission is the limiting effect in most tests indicating that both, installations and cleaning process, need further optimisation.

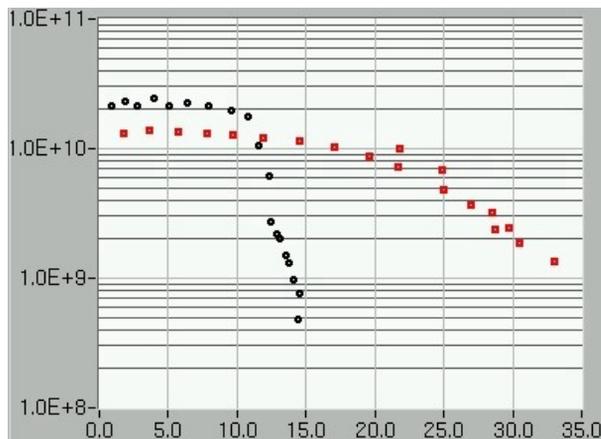


Figure 4:  $Q(E_{acc})$ -performance of a monocell cavity at 2 K before (black) and after (red) dry-ice cleaning.

## SUMMARY AND OUTLOOK

Based on the evident experiences in semi-conductor industry and the successful cleaning of several Nb samples, dry-ice cleaning was applied for the first time to superconducting accelerator cavities. Resulting in typical high Q-values and gradients up to 33 MV/m the proof-of-principle was successful. Nevertheless significant improvements of the cleaning apparatus and optimisation of the parameters are necessary.

A more powerful chiller/purifier unit for  $CO_2$  is ordered and an improved motion unit, capable for horizontal cleaning of one- to three-cell cavities, is under construction. An effective gas extraction system and a controlled heater system to avoid humidity condensation on the cavity are under development. During the next tests the cleaning speed as well as the dry-ice jet properties defined by nozzle diameter,  $N_2$  pressure,  $CO_2$  temperature will be topics of detailed investigations.

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