

## New Aspects of Quality Control during Preparation of TTF 1.3 GHz Cavities

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### Abstract

Though the quality of processing of the superconducting niobium cavities to be used for the TESLA Test Facility (TTF) improved continuously during the last years, some cavities still show strong field emission far below the aim of  $E_{acc} = 25$  MV/m. To our today's knowledge the main origin of field emission is particle contamination, which either is caused during the preparation process or cannot be removed in spite of various cleaning steps. This view is confirmed by an analysis of all available processing data of the nine-cell structures in 1998 and 1999. In case of irregularities during the preparation a reduced onset field for field emission is observed. The earlier efforts to measure the particle contamination of the cavity drain water in the High Pressure Rinsing (HPR) Stand were intensified using membrane filters analysed under an optical microscope. An apparatus for integral field emission measurements using a transparent phosphor screen for imaging the electrons is under commissioning inside the TTF cleanroom.

### 1 INTRODUCTION

Obviously, the goal of quality control (QC) during the cavity preparation are reproducible high gradients and high Q-values. This paper focuses on the QC investigations during the cleanroom processing (degreasing, etching, high pressure rinsing, assembly) of 1.3 GHz nine-cell cavities for the TESLA Test Facility. Compared to silicon wafers with their similar requirements of clean work during processing and handling, the complicated shape of the cavities prevents the use of well established QC-methods directly applied to the processed surface. Up to the final rf measurement only indirect methods like samples processed together with the cavities, analysis of the process media like ultrapure water, pure gases, acids, vacuum etc. or control of the clean environmental condition, e.g. air particle counting, are possible [1, 2, 3].

### 2 FIELD EMISSION ONSET ANALYSIS

The development of the average field emission onset together with the average maximum gradient during the last years is shown in Figure 1. Some closer analysis is done for 1998 and 1999. The gradient of the field emission onset of the vertical nine-cell cavity tests is compared with all available processing data. For each year

the cavities are sorted in two groups: Most of the cavities have a regular preparation without any peculiarity, but a smaller number shows irregularities during the preparation. Typical irregularities are repeated assemblies of the flanges due to vacuum leaks, exchange of vacuum flanges without following HPR or faults of the cleanroom air condition and ultrapure water system. In 1998, the time between the final chemistry incl. HPR and the assembly of the vacuum flanges was increased from one day to three days for some cavities, which seemed to harm their performance. Beside the obvious possibility of a degradation by air exposure, other explanations cannot be ruled out due to simultaneous changes of the cavity handling procedure. As no further studies have been undertaken, no final explanation of this phenomenon can be given.

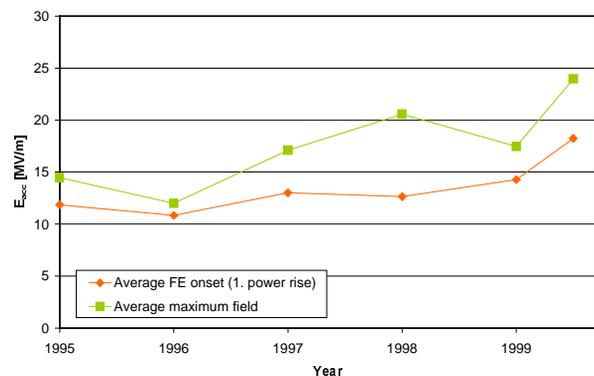


Figure 1: Historical development of average field emission onset (first  $Q_0(E_{acc})$ -measurement) and average maximum gradient

First of all the analysis is concentrated on the field emission onset of the first  $Q_0(E)$ -measurement (Figure 1) of the vertical cryotests. These data, taken before the in-situ cw processing during the high field measurements, describe best the quality of the clean room surface preparation. In case of irregularities the average of the field emission onset is reduced by 7,5 MV/m and 4 MV/m in 1999 and 1998, respectively. This emphasizes the importance of well-defined, reproducible and documented processes during the cavity preparation. In addition, the average field emission onset without irregularities increased from 13,7 MV/m in 1998 to 18,2 MV/m in 1999. To our opinion this positive effect is caused by a number of improvements of the etching and rinsing installations and processes introduced beginning of 1999.

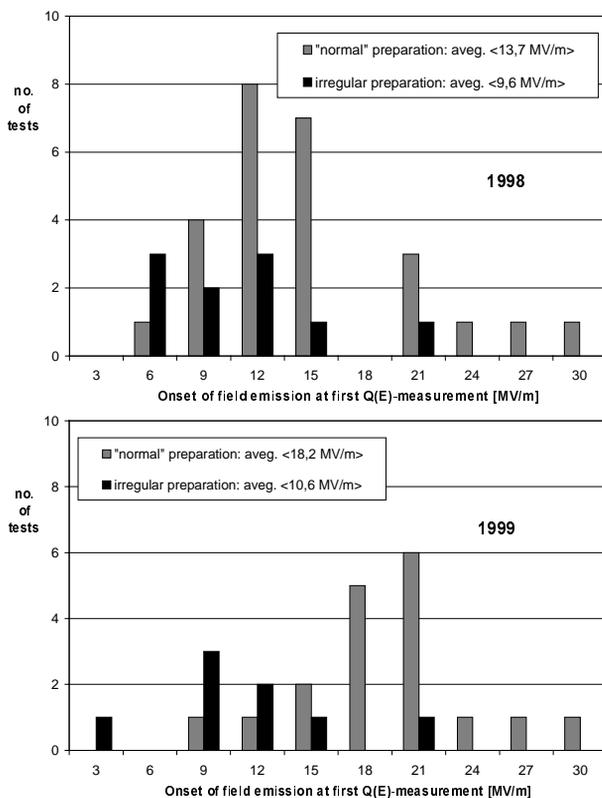


Figure 2: Comparison of field emission onset for 1998 (upper diagram) and 1999 (lower diagram) after “normal” and irregular preparations

The  $Q_0(E)$ -curves after high field operation usually show higher fields for the average of the field emission onset as expected after some cw processing up to 200 W incident power and the same trends. The average gradient achieved without field emission could be increased from 17,7 MV/m in 1998 to 20,7 MV/m in 1999. Irregular conditions during the preparation reduced this value by 3,2 MV/m and 5,5 MV/m.

### 3 PARTICLE COUNTING DURING HPR

#### 3.1 General

During the last years the idea to count and identify the particle contamination of the HPR drain water to control the cleaning effect of High Pressure Rinsing was followed continuously. First attempts were undertaken using a laser particle counter [2]. As our laser particle counter cannot distinguish between air bubbles and particles, we tried to suppress the counting of air bubbles by both using a special sampling apparatus, collecting and pressurizing 50 ml per 2 minutes, and several tubing arrangements, which should allow the air bubbles to degas before reaching the particle counter. Nevertheless, no trustable and reproducible results could be achieved and this approach was given up.

The new set-up (Figure 3) uses membrane filters to collect the particles. The advantages are up to a factor of

100 larger amount of water suck through the filter and the possibility of a later elemental analysis. Disadvantageous is the off-line character of the measurement, which does not allow any correlation between the position of the spraying cane and the particle number.

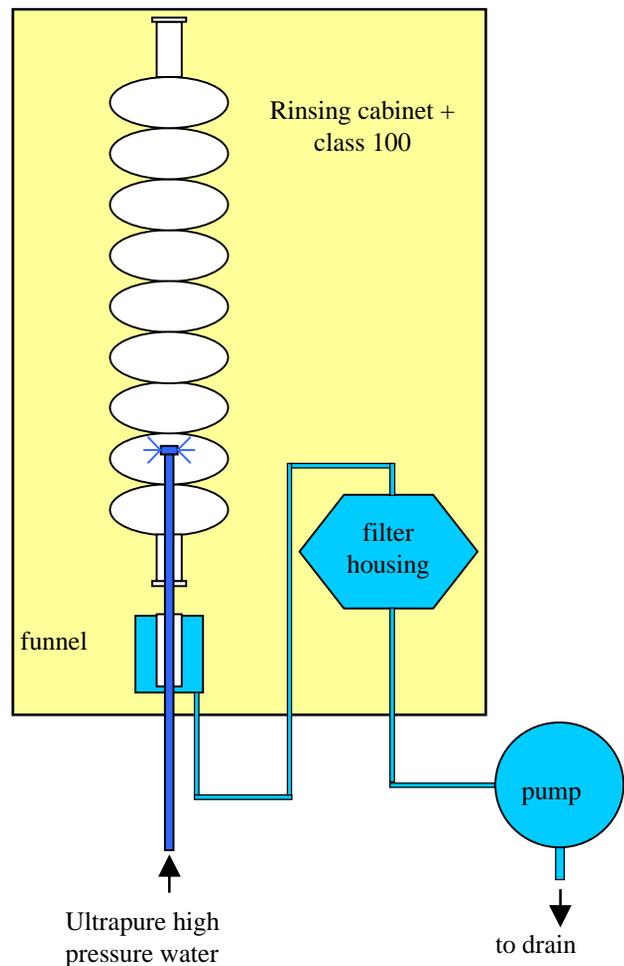


Figure 3: Schematic of particle sampling during HPR

#### 3.2 Sampling and Analysis

Starting with a simple funnel and gravitational water flow, the sampling arrangement is optimized to maximum water collection simultaneous with high cleanliness. A cylindrical funnel together with a diaphragm pump enables a water flow of (1-2) l/min of the total  $\approx 10$  l/min drained water. Funnel, piping and filter housing are made of PVDF and PP. An additional by-pass line to the funnel, not shown in figure 3, allows the rinsing of the system with ultrapure water.

Usually filters with a pore size of  $2 \mu\text{m}$  are used. After use they are stored and inspected using slide frames. For counting an optical microscope is used located inside the cleanroom, which allows with its 90x magnification a resolution of  $\approx 5 \mu\text{m}$ . Due to the duration of counting only  $4 \text{ cm}^2$  of the  $14 \text{ cm}^2$  total surface area are analyzed.

Elemental analysis using EDX is possible, but unfortunately the available SEM is not located inside a cleanroom resulting in the danger of foreign contamination. The large time needed for one filter (1-2 filters/day) and the very difficult re-identification of particles found with the optical microscope make the EDX-analysis unsuited for routine operation.

### 3.3 Results and Future

A typical picture of particles lying on the membrane filter is shown in Figure 4. Surprisingly, for all cavities the filters show many large particles up to a size of 500  $\mu\text{m}$ . As all media (pure gases, water, acid) used during the preparation process are filtered to  $\leq 0,2 \mu\text{m}$ , this observation is really astonishing, but in agreement with similar measurements at the Jefferson Lab [2] and CERN [4]. Particle counting on unused filters and subsequent elemental analysis of these particles (see below) clearly prove, that the origin of the particles is the cavity. However, it is still under investigation to which amount the bottom flange with its bolted connection to the vacuum valve, which is opened and closed before the final rinsings, has a part in the contamination.



Figure 4: Particles on a filter with 2  $\mu\text{m}$  pore size (magnification 100x)

The standard cavity procedure includes a first HPR after the final chemistry and before the cleanroom assembly of the vacuum flanges. The second and third HPR take place as the ultimate cleaning step after the cleanroom assembly. As expected for an improving cleanliness, the particle numbers decrease during the threefold rinsing. Typical particle numbers with the actual funnel design are:

- 1. HPR:  $> 150 \text{ part/cm}^2$
- 2. HPR:  $(50 - 150) \text{ part/cm}^2$
- 3. HPR:  $(20 - 40) \text{ part/cm}^2$

It should be mentioned again, that only particles larger than  $\approx 5 \mu\text{m}$  can be detected.

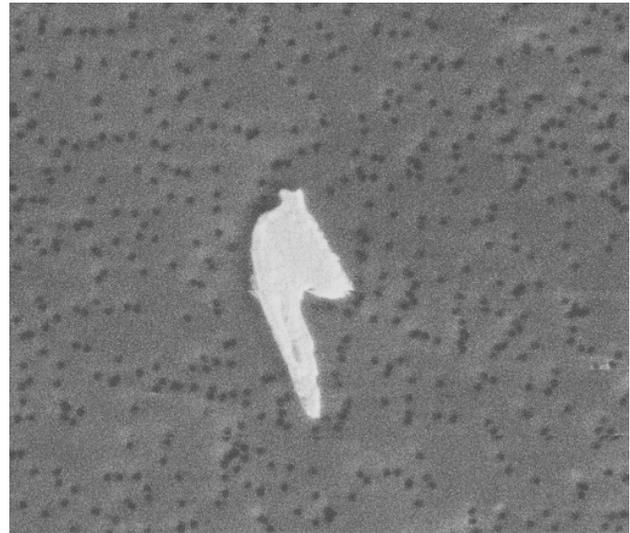


Figure 5: SEM-picture of a stainless steel particle of 60  $\mu\text{m}$

The EDX analysis of 5 filters of different cavities mirrors all used materials during the preparation process. Without any attempt of a statistical analysis, it can be said, that many analysed particles consist of stainless steel (Figure 5), niobium or the copper alloy of the bolts. Especially, after the first rinse viton particles are found, most probable caused by the o-rings used during etching. In addition, the elemental composition of many particles is very complex and no obvious source can be determined. At least, some of those particles may be due to the “dirty” handling at the SEM.

Figure 6 shows the total number of particles of 12 nine-cell cavities after the final HPR. Though the particle numbers for the cavities no.16 and no.44 (with He-tank) are higher than usual, the rf results, especially the onsets of field emission, are very good, leading to the question of a possible correlation between particle numbers and rf results. Within the limited statistics we have up to now, no correlation of the particle contamination to the field emission behaviour of the cavities is found.

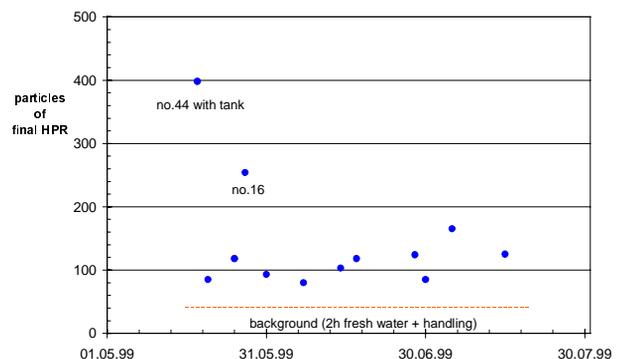


Figure 6: Particle contamination of final HPR vs. date (for actual funnel design since May 99)

With the installation of an automated rinsing of the funnel system, the period of modifications of the sampling arrangement in order to avoid cross contamination and to ensure reproducible, clean measurement conditions ended recently. A new microscope system for a full scan of the filter surface, a better resolution and a better documentation is ordered. Starting in the near future, it will help answering the obvious questions of the High Pressure Rinsing procedure:

- Where is the source of the contamination located at the cavity?
- Do we need more inside and/or outside rinsing steps or special cleaning of the flange areas ?
- Is there a correlation to the rf results?

Furthermore, the combination of the filter analysis and the integral measurement of the field emission properties, described in the next chapter, is planned to obtain more information about the nature of the emitters.

#### 4 APPARATUS FOR INTEGRAL DC FIELD EMISSION MEASUREMENT

The principle and various constructions of a DC field emission measurement apparatus using a planar diode configuration is described in a number of papers e.g. [5, 6, 7]. Figure 7 shows a schematic sketch of our configuration housed in a UHV vacuum vessel. The niobium sample (cathode) with a diameter of 25 mm and its mushroom shape allows a chemical preparation and HPR inside our standard nine-cell cavities. A transparent ITO screen (anode) visualizes the location of the emitter. The distance between the ITO screen and the sample is adjusted by PTFE spacer material. With the available voltage of 10 kV and a spacer thickness of 100  $\mu\text{m}$  a surface field of  $E_p = 100 \text{ MV/m}$  is possible. The I - U characteristic curve gives the onset of field emission and the field enhancement at the emitter. Since the system will be used for the control of the preparation and handling processes, it is located inside our cleanroom (Figure 8).

The system is under commissioning and the first emitters have been detected, recently.

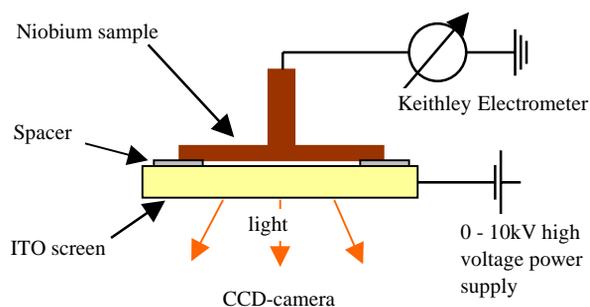


Figure 7: Schematic sketch of the apparatus for integral field emission measurement. The system is housed inside a vacuum chamber.



Figure 8: The apparatus for integral field emission measurement located inside the cleanroom.

#### 5 SUMMARY

Though during the last two years the onset of field emission increased and results became more reproducible, field emission keeps still one of the major limitation mechanisms. Two promising approaches for a better control of the cleanroom preparation processes are developed and under commissioning. The effort to improve the cleaning procedures will be continued in the future.

#### 6 ACKNOWLEDGEMENT

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