CONTROL OF PARTICLE CONTAMINATION WITH LIQUID AND AIR PARTICLE COUNTERS DURING PREPARATION OF THE TTF 1.3 GHZ RESONATORS

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A series of 15 superconducting niobium cavities to be used for the TESLA Test Facility (TTF) was tested at DESY. During the processing of these cavities one part of the activities was to study the feasibility of 25 MV/m acceleration gradients with low fieldemission. During the acceptance tests of the TTF cavities subsequently the standard treatment resulted in cavities with very low electron loading at field gradients above 20 MV/m, while in other resonators strong fieldemission started at low levels. During commissioning of the first TTF module, somtetimes the cavities showed a degradation of the applicable acceleration voltage due to enhanced fieldemission. To find the origin of fieldemission we controlled every step of the assembly, cleaning and preparation procedure in respect to particle contamination due to the process. A liquid particle counter is installed in the HP rinsing stand to control the water draining from the cavity beam-tube as well as the clean-water plant. The assembly procedure of the cavities is monitored with an air particle counter. Investigations in material and material combinations with respect to particles production were done with an air particle counter setup, that allows particle measurements outside and inside of a cavity torus. We report on the set up and commissioning of the liquid particle counter and results obtained from the series of tests and cavity assemblies.

Introduction

For future accelerators in the TeV range several proposals are under discussion. One of these proposals bases on the application of super conducting (s.c.) resonator structures with a minimum acceleration gradient (Eacc) of 25 MV/m. A test facility is build at DESY. Here the state of the art of s.c. cavities (9 cell 1,3 GHz) made by industrial companies is under investigation. A set of 8 cavities is combined with a s.c. quadruple to the first cryo model, which is under test since summer 97. 32 more cavities are ordered where all information of improved fabrication technology and treatment, gained during the first set of 12 cavities for the first module, will be applied.

A) Quality control and analysis:

During the commissioning of the module a reduction of the cavity performance compared to the results of the vertical and horizontal test of individual cavitiy was found. During the assembly of the string a particle control by an air particle counter type MET ONE* took place. This air particle counter was in use as a quality control instrument and for analysis of the equipment. To ensure the reproducibility of procedures the air particle counter is in use to monitore the process.

A I) Set Up

All components to be used in the class 10 area and in contact to the cavity are cleaned according to the standard cleaning procedure of TTF given in Table 1.

To reproduce the quality for parts to be connected to the cavity a MET ONE * air particle counter is placed on a fixed position inside the class 10 area. The air stream of an ionization N_2 gun, equipped with a 0,02 µm point of use filter is in use to blow of loose particles into the direction of the counter (Fig. 1). The blow off of particles from parts to be controlled, is continued until the number of counts in the 0,3 µm channel reduces down to class 10 ASTM specification.

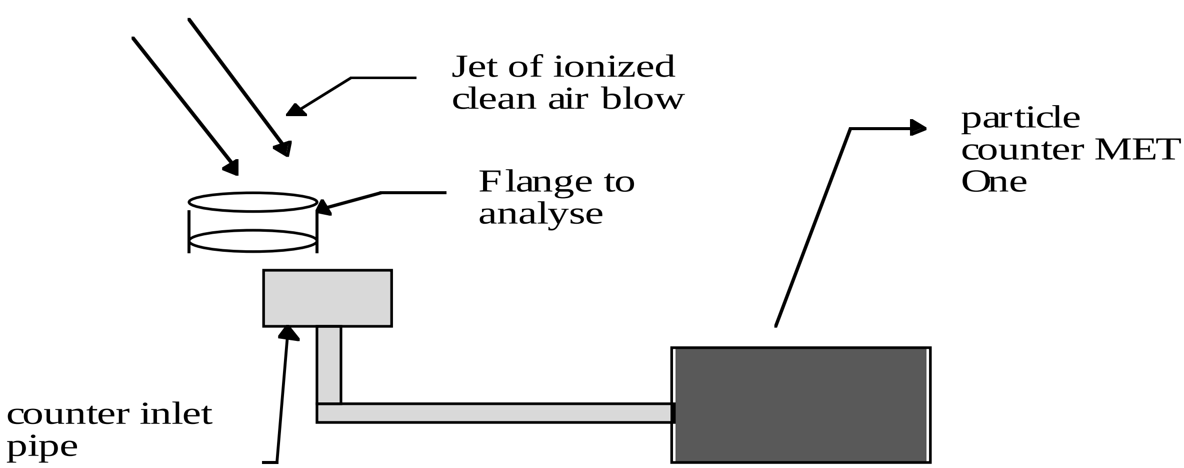


Fig. 1: Schematics of test set up to analyse particle contamination by blowing with a N_2-jet from a ionization N_2 gun

The measurements done by blow off with the N_2 jet stream towards the particle counter is not able to give absolute numbers. The direction of the jet as well as turbulences resulting from the geometry of the units under test and the limited space of the inlet of the counter influence the measurement. Results obtained with this method are relative numbers. They allow to compare different designs used for the same application and to find best designs. All units tested had to undergo the standard cleaning procedure applied for TTF (Table 1).

sequence No	treatment	cleanroom class	application of
1	rough degreasing and rinsing in a car wasch	grayroom	detergent Ticopur/ Di water 5-10 MΩcm
2	Ultrasound cleaning	10.000	detergent Ticopur circulation and filtration of detergent
3	DI water rinsing	10.000	ultra pure water 18,2 MΩ cm, point of use filter 0,2 µm
4	fine rinsing	10-100	fine rinsing with ultra pure water in class 100 area
5	drying	10	drying sluice cleanroom air class 10 quality

Table 1: Standard cleaning procedure for TTF applied for cleaning to class 10 (ASTM) application

A II) Measurements and results

AIIa) Flange systems for TTF

The vacuum flange system in use for the module 1 and 2 of TTF is designed on Helicoflex* sealing technique. The Helicoflex seal made from aluminum is compressed between a flange made from stainless steel and a Niobium lip rolled out from the beam-pipe. All cavities are postpurified with Titanium at 1400 C [Ref. 1]. The stainless steel parts have to be removed for this treatment. The dismountable flange assembly consists of a splitted ring (5), the sealing flange (2,6), a sliding ring (3) to prevent the splitted ring from tilting, bolts and nuts (1) and the sealing itself (4) (See Figure 3).

AIIb) Flanges:

The efficiency of cleaning (procedure see Table 1) was tested by a comparison of commercial flange systems (Conflat NW 100) and flanges designed for cavity application. Figure 2 gives average numbers found on more than 5 independent preparations and tests.

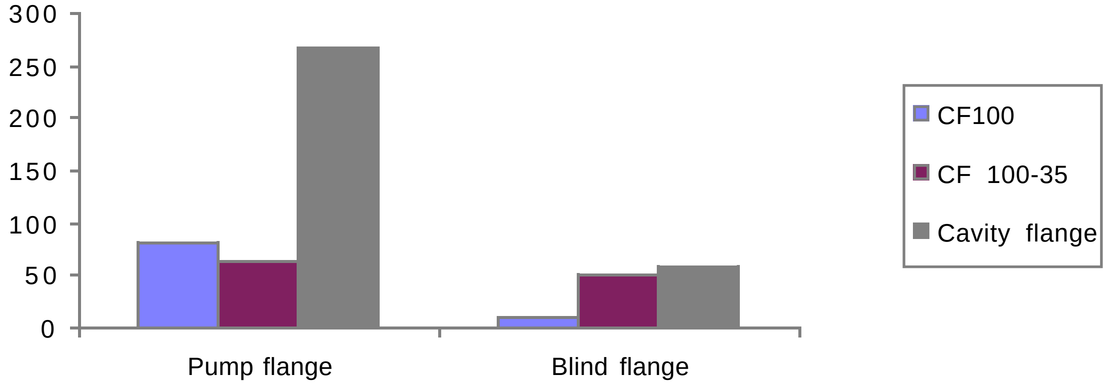


Table 2:average particle counts after standard cleaning procedure (Accumulative 0,3-10 µm size)

According to these measurements the cleanliness of blind flanges is not correlated to the design of the vacuum component. The pumping flanges show a factor of 2 higher average particle contamination. Most of the particles were blown off from the M6 threaded holes build in to connect to standard vacuum components. A 2,5 times higher particle contamination was found on TTF pump out flanges. This is related to the rough surfaces on the non-vacuum side of the flange and the groves machined in for aligning the Helicoflex seal. In general, for holes and grooves as well as on rough surfaces the interaction of the ultrasound and the simultan rinsing off of particles, lifted from the surface, is very inefficient.

Plane and smooth areas as well as grooves with sufficient opening can be cleaned to a standard of class 10 or better with the standard cleaning equipment of TTF.

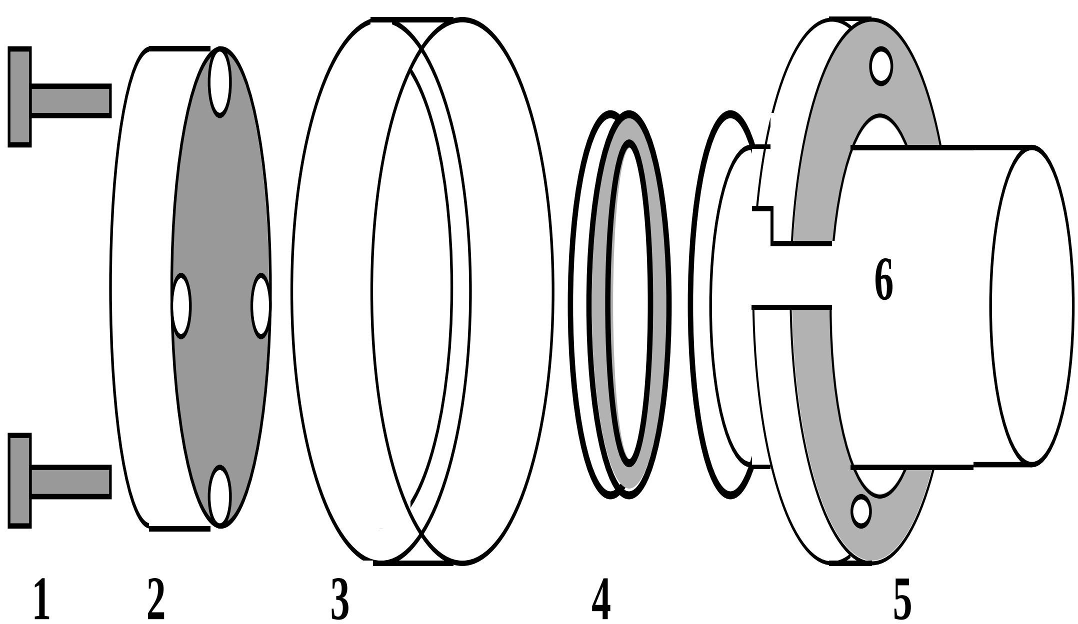


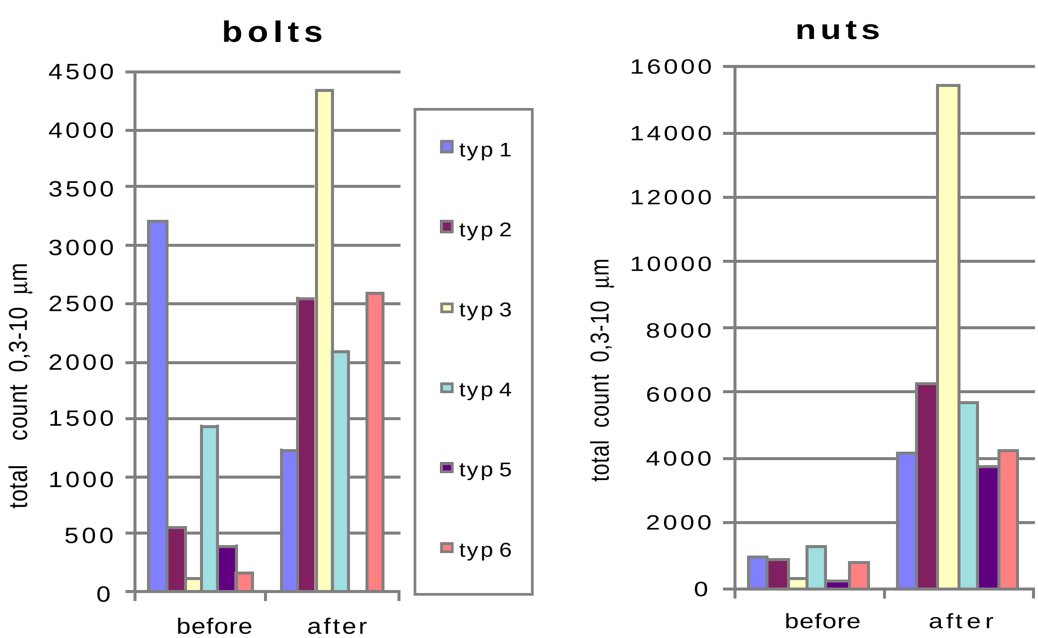
Figure 3: Schematics of the flange assembly for TTF Helicoflex sealing technique

AIIc) Bolts and nuts

The sealing technique of vacuum flanges requires bolts and nuts. The torque reacting on the sliding surface of non lubricated bolts and nuts results in the production of large quantities of particles by smoothening the surface, cold welding and skin friction.

The measurements during the assembly procedures (see Chapter A IIIa) show that bolts and nuts are the origin of the largest particle contamination found in the assembly procedure. Investigations are made on several combinations of materials and fabrication techniques to find lowest number of particle production.

Before assembly all bolts and nuts are cleaned (procedure see table 1) and checked. Rolled bolts and threaded rods show lower number of residual particles, while cutted threads show up to a factor of 1000 higher numbers. Most particles remain inside the basis area of the thread. Typical numbers found in that region gain from 300 „rolled



Typ of bolt and nut combined: typ 1: M8 rod (cutted thread) 2 sides + 2 nuts CUNISIL
typ 2: bolt SS M8 (cutted thread) + 1 nut CUNISIL
typ 3: bolt SS M8 (rolled thread) + 1 nut SS
typ 4: bolt SS M8 (cutted thread) + taped hole
typ 5: threated rod SS M8 (rolled thread) + 1 nut CUNISIL + 1 taped hole M6
typ 6: bolt SS (cutted thread) + 1 taped hole

Table 2: Typical particle contaminations for different bolts and nuts combined

surface finish“, to several 1000 of 0,3 to 10 µm particles for taped threads. All nuts tested so far show typical contaminations around 1000 particles independent from material or fabrication technique. The cleaning procedure applied is not able to remove particles from the thread area, specially small size nuts don’t show significant reduction of particles. New methods for cleaning have to be investigated.

After the assembly on a flange, the bolt and nut combination is dismounted and remeasured in the test set up. The particle contamination found here is related to the material combination, the surface roughness of bolts and nuts (Table 2). Rolled threads show a smother and harder surface than cutted once. In combination with CUNISIL nuts the lowest number of particle contamination was measured.

The largest number of particles ranging from 0,3 to 10 µm was measured on stainless steel bolts combined with non lubricated stainless nuts. Lowest numbers of contamination was measured on rolled threated rods combined with CUNISIL nuts.

AIIc) Valves

For TTF standard CF 35 all metal valves are in use. They pass the same cleaning procedure like all other components. Special care is taken for cleaning the bellow hidden in the top of the valve to give full pumping speed in vacuum. According to the test results shown in table 3 the valve have to be opened 1/10 of full range of diameter during the cleaning process to reach best cleaning results. Valves of smaller size have to be disassembled and cleaned in separate sub assemblies.

	valve open during cleaning and rinsing			valve 1/10 open during cleaning and rinsing	
	inside	outside		inside	outside
before assembly	* 206	* 1059		* 2	* 663
open+close on a cavity	188	---		10	---
after assembly	* 263	* 1954		* 17	* 4360
assembled twice	420	19775		---	---
disassembled twice	1886	17565		---	---

* Average of several measurements
All numbers are total counts in the range of 0,3-10 µm

Table 3: Measurements of particle contamination inside a cavity origin from CF35 all metal valves

Final rinsing is always done manually with a water jet of ultra pure water. Only this final rinse, concentrated on the bellow region of the valve, resulted in particle counts well below class 10 quality.

A III) Monitoring

AIII a) Measurements inside a cavity

To study an optimized assembly and disassembly procedure, the particle counter inlet was placed inside the cavity beam-tube and monitored the assembly procedure continuously. The diameter of the inlet tube was build out conical so that 70 % of the beam-tube area (ID = 78 mm) is covered by the counter inlet. The opposite beam-pipe of the resonator remained open to ensure the air flow of 1 qft/min. The sequence of the flange assembly was done with the same set of flanges, that was always recleaned and checked using the air particle counter (see Chapter A II).

assembly of	split ring	seal+ blind flange	fix seal	bold down flange	loosen bolts	loose seal	dismount flange and seal	dismount split ring
Type K	38	9	21	131	216	19	150	20
Type O	46	18	56	109	47	13	31	87

Table 4: Total particle counts (0,3-10 µm) measured inside a cavity during assembly of a blind flange perpendicular to the laminar airflow of class 10 cleanroom

In a first test sequence the dependence of particle contamination and assembly position was tested. It was found, that the assembly parallel to the laminar flow resulted in 30 to 50 % lower particles inside the cavity. A position perpendicular to the laminar flow resulted in the highest particle contamination inside the cavity. These results are in a wide range independent on the crew and can be related to turbulences around the beam pipe. Table 4 shows typical results obtained during the assembly of a blind flange perpendicular to the laminar flow. For this position a clear reduction of 50% of particle contamination inside the cavity is reached by changing the design. The assembly of a cavity string is done on a rail with the laminar flow perpendicular to the beam-pipes. During this assembly procedure a permanent clean gas flow is passing the cavity towards the assembly position. The influence of this clean gas flow could not be tested in this set up.

AIIIb) Assembly of module

The first TTF module was assembled in the class 10 cleanroom. Most of these cavities are tested in horizontal position [Ref. 2] before installation in the string. After module assembly the cavities show a degradation of the acceleration voltage Eacc due to enhanced field emission [Ref. 2]. This degradation can be related to the fact that the completion of the module is more complicated than the assembly for vertical test-deware. In addition, no HP rinse can be applied on cavities after they are equipped with a trained power-coupler [Ref. 3]. The reduction in field, typical for string assemblies [Ref. 4], can be correlated to the complexity of the assembly.

For monitoring the installation of a module, the air particle counter is set to a continuos mode and is placed on defined position right under the assembly area. To guide the assembly procedure the process is interrupted at critical assembly steps (Table 5).

assembly step	Cavity	Action	particle counter	interrupt for cleaning until area is
1	n / n+1	blow cavity and beam-pipe	control	**< class 10**
2	n+1	venting and continuos gas flow	-	no
3	n / n+1	install tooling for flange / clean area	monitor/ control	**< class 10**
4	n / n+1	remove bolts / clean area	monitor/ control	**< class 10**
5	n / n+1	remove split ring / clean area	monitor/ control	**< class 10**
6	n+1 first / n last	remove flange and control continuous gas flow	monitor	counts are stable for 5 Minutes
7	n+1 first / n last	assemble new flange and gasket	monitor	no
8	n/ n+1	close flanges leak tight	monitor	no

Table 5: Principle flow scheme for string assembly procedure with interrupts for quality control

During the interrupt, the jet of the N_2 gun is used to blow off loose particles into the direction of the counter. A fixture, developed for the assembly procedure, presses the flange onto the sealing gasket and keeps the cavity closed even when all bolts of the connection are removed. The assembly procedure continues only when a minimum of particle contamination blown off by the N_2 jet is reached.

Typical results measured during the assembly of module 1 as well as measurements found during the assembly of injector No 2 cavity are shown in Table 6.

Action	Assembly step No	total particle counts of size 0,3-10 μm
remove bolts from beampipe flange	4	5000 -> 8000
Blow area after removal of bolts	4	8000 -> 20000
remove split ring and clean area	5	10000 -> 23000
assemble new flange	7	1000 -> 4000
tighten flange	8	6000 -> 12000

Table 6: Typical particle contamination measured during module assembly outside of the cavity on a fixed position below assembly area

B) Water particle counting

B I) Ultra pure water plant and test set-up

The final cleaning and rinsing of cavities and components for the TTF Linac is done in a class 10.000 (partly cl. 100) cleanroom using ultra pure (‘superpurity’) water. The water plant processes town water using a reverse osmosis system, ion exchangers, ultraviolett radiation and a stepwise particle filtration down to 0,04 µm for the final rinsing. The resistivity is > 18 MΩcm at a flow rate of > 30 l/min. After etching and assembly of the cavities, they are water rinsed with a pressure of 100 bar and the same supply water quality.

The quality is controlled by routinely monitoring the resistivity and the number of particles of the supply water. Particle counting is done behind the Point-of-Use (PoU) filters of the closed chemistry and rinsing system and the final high pressure rinsing (HPR). Particle counting of the drain water during the HPR is under commissioning. Fig.4 shows a flow schema of the set-up.

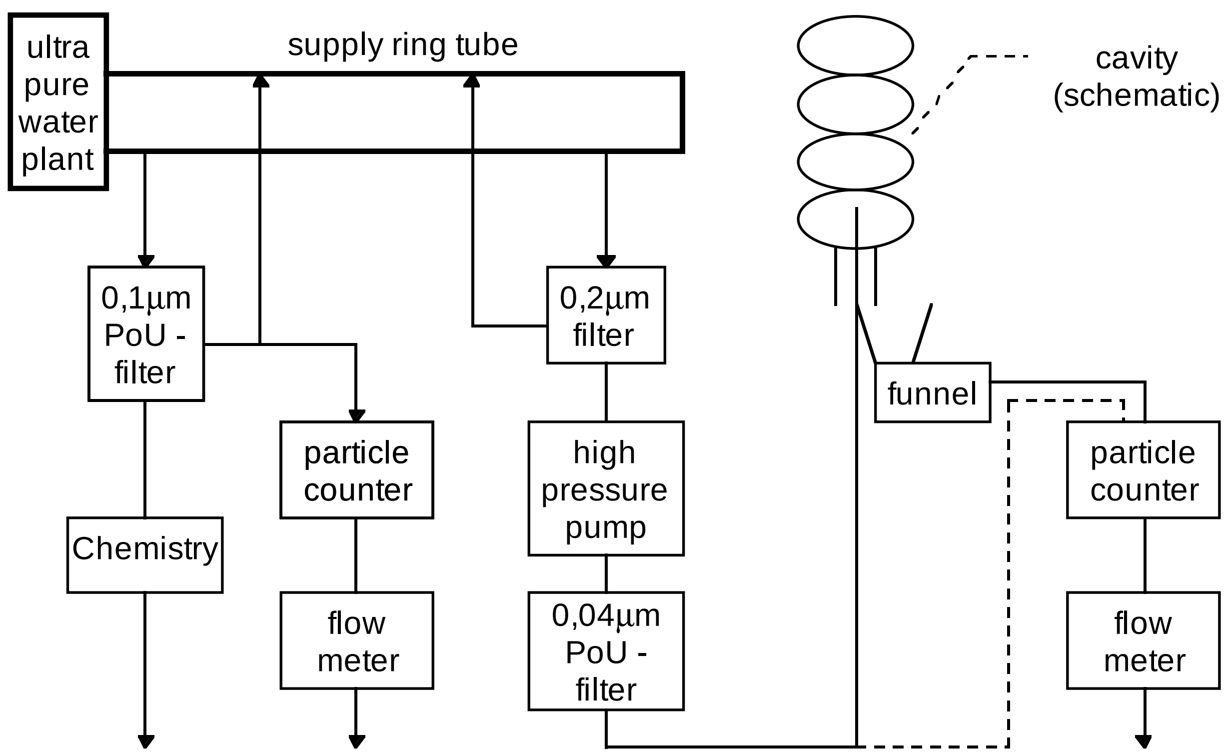


Fig.4: Schematic flow scheme of the set-up for water particle counting

B II) Results at the PoU Chemistry System

All PoU-filters at the 4 bar supply ring are rinsed with 150 l/min continously. This allows clean and stable conditions for particle counting. A continous flow of (35 - 100) ml/min controlled by a flow meter runs through the particle counter and is a representative measure of the water quality used for rinsing the cavities after etching (Fig.5). Typically, less than 2 particles per 100 ml with as size > 0,3 µm are counted. The peaks in Fig.5 are due to a change of the ion exchangers and filters. The time for recovery of the system is about two hours.

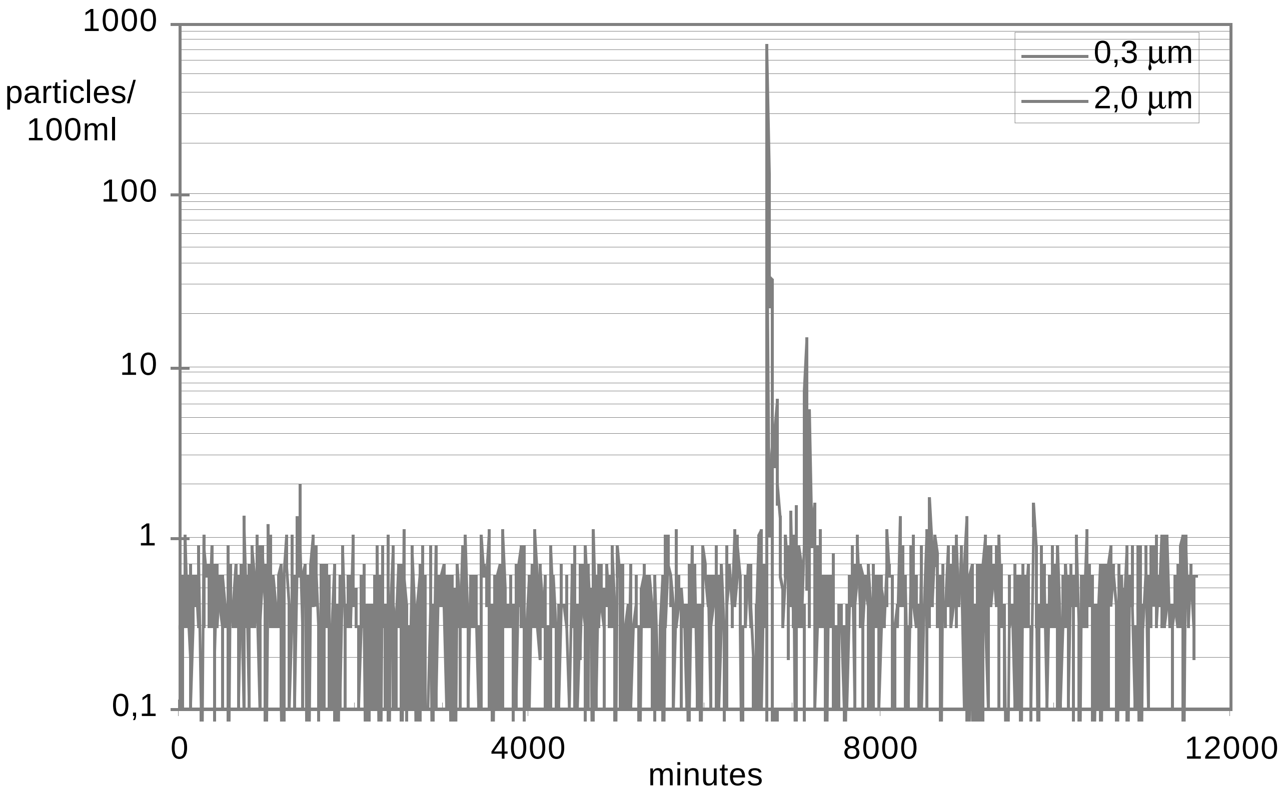


Fig.5: Particles per 100ml measured at the PoU 'Chemistry system'
(The peak is caused by the change of the ion exchangers)

B III) Results at the PoU High Pressure Rinsing

At the PoU High Pressure Rinsing there are two substantially different modes of measurement:

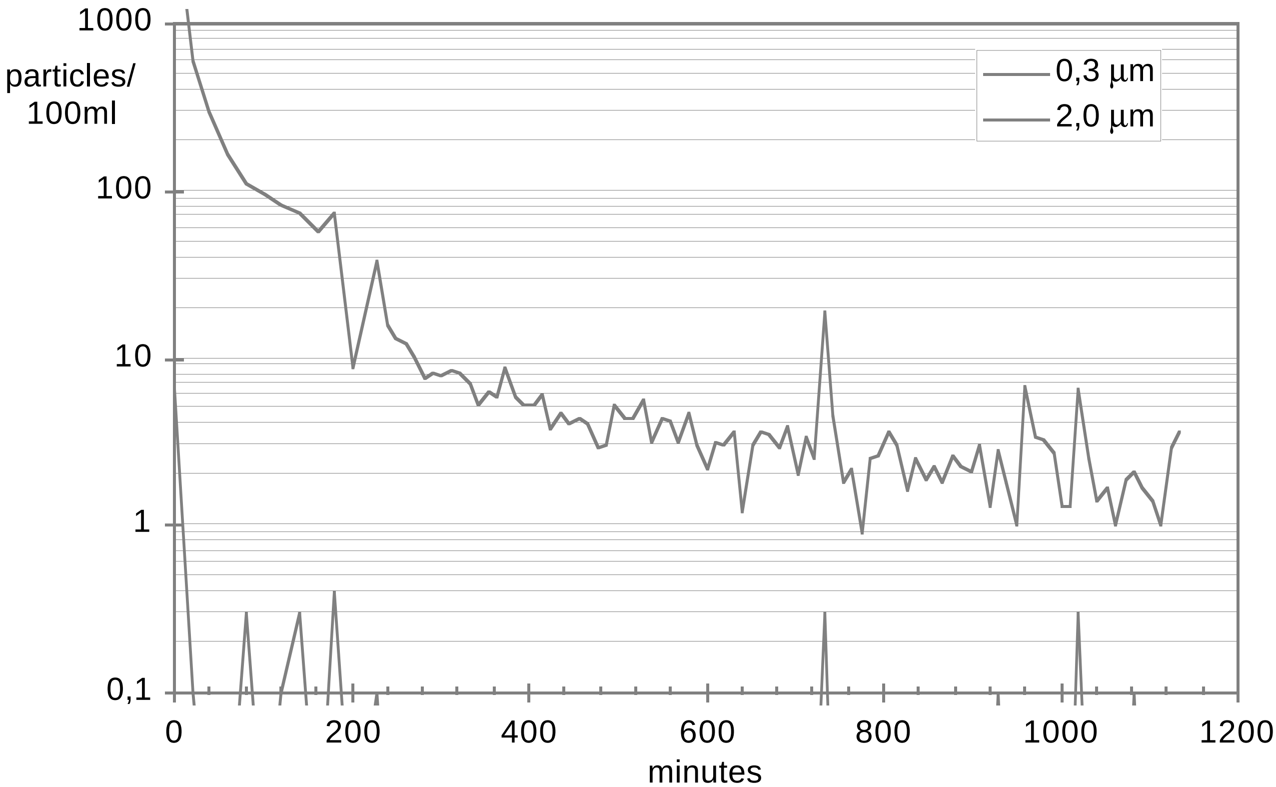


Fig.6: In-line counting at the PoU 'HPR' using a PVDF-"nozzle"
(particles per 100ml ; 10.06.97)

An in-line measurement behind the 0,04 µm PoU-filter with a continous water flow is possible, if the pump is not working, but requires the change of the spraying nozzle to an adapter piece for particle counting. No high pressure rinsing is possible with this adapter and, as a consequence, no data during rinsing a cavity can be taken. After changing the nozzle, counts per 100 ml decrease to less than 10 particles > 0,3 µm within one hour (Fig.6). So, the water at the spraying nozzles has the same quality as the water used for the final in-line rinsing after etching the cavity and should not be a source of particle contaminations. Of course, abrasion of the nozzle is not checked by this measurement

Second, supply water from the PoU Chemistry System is continously collected using a funnel and runs through the in-line particle counter by gravity. Though it allows no continous control of the supply line of the HPR system, measurements during high pressure rinsing a cavity can be done without any changes of the test set-up. In this case, the drain water out of the cavity is collected in the funnel together with the above mentioned supply water. Without high pressure rinsing less than 10 particles per 100 ml with a size > 0,3 µm are counted (Fig.7). First measurements during HPR show a high sensitivity for air bubbles created in the funnel and counted as particles. The system allows no decision between particles and bubbles, but the overall distribution of the particles counted, is characteristically changed in the presence of bubbles. Fig.8 shows the particle counts per 100 ml during a high pressure rinsing of cavity C22. The peak at the beginning occurs before the nozzle moves into the cavity and is most probable due to air bubbles. A cleaning effect of all tubings, high pressure rinsing system and funnel, cannot be excluded. It is remarkable, that during rinsing the number of particles larger than 2 µm is very low.

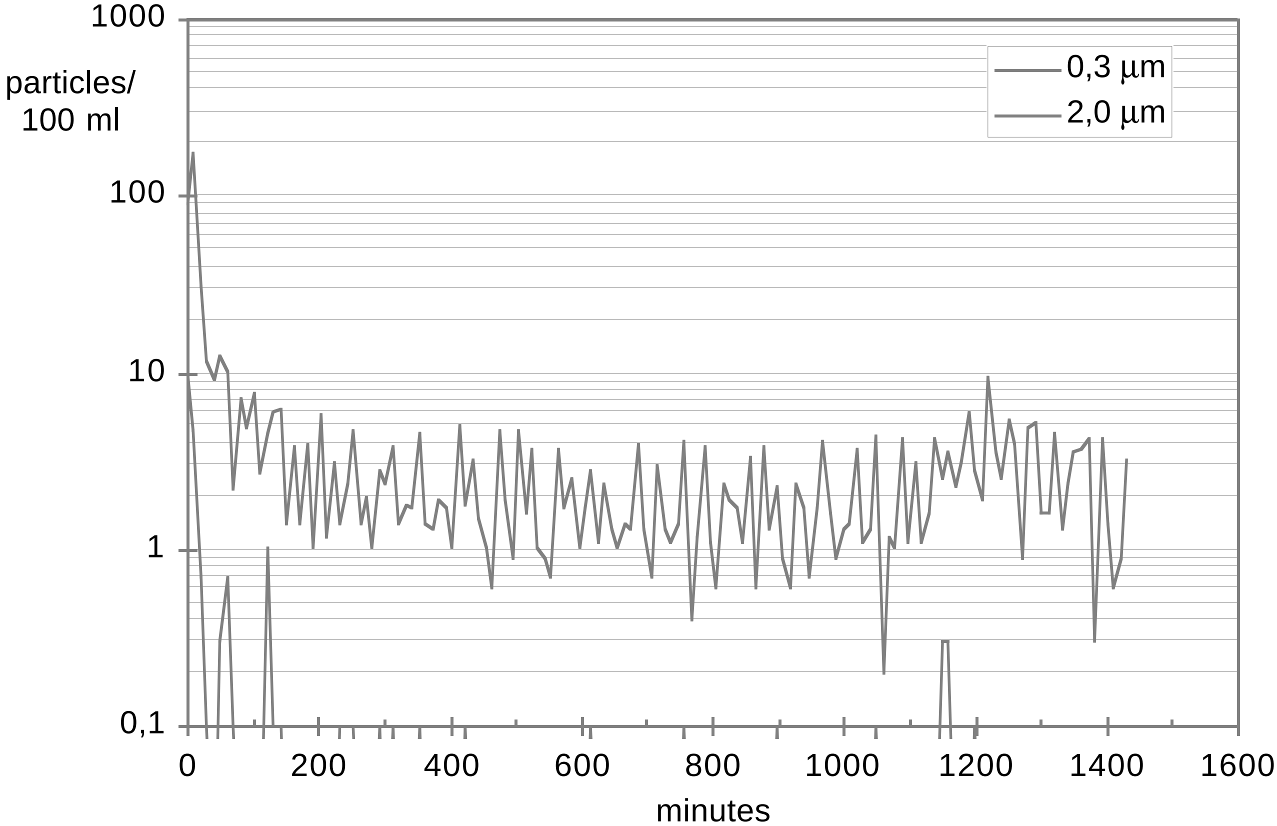


Fig.7: Particles per 100ml measured at the PoU 'HPR' (22./23.7.97)

As an additional mode of measurement the in-line particle counter is changed to a sampling apparatus, collecting 50 ml per 2 minutes and analyzing it. The idea of the sampler is to avoid air bubbles counted as particles by pressurizing the collected sample. Fig.9 shows the insufficient results of this device, resulting in high and instable counting rates. Furthermore the measurements are sensitive to air bubbles.

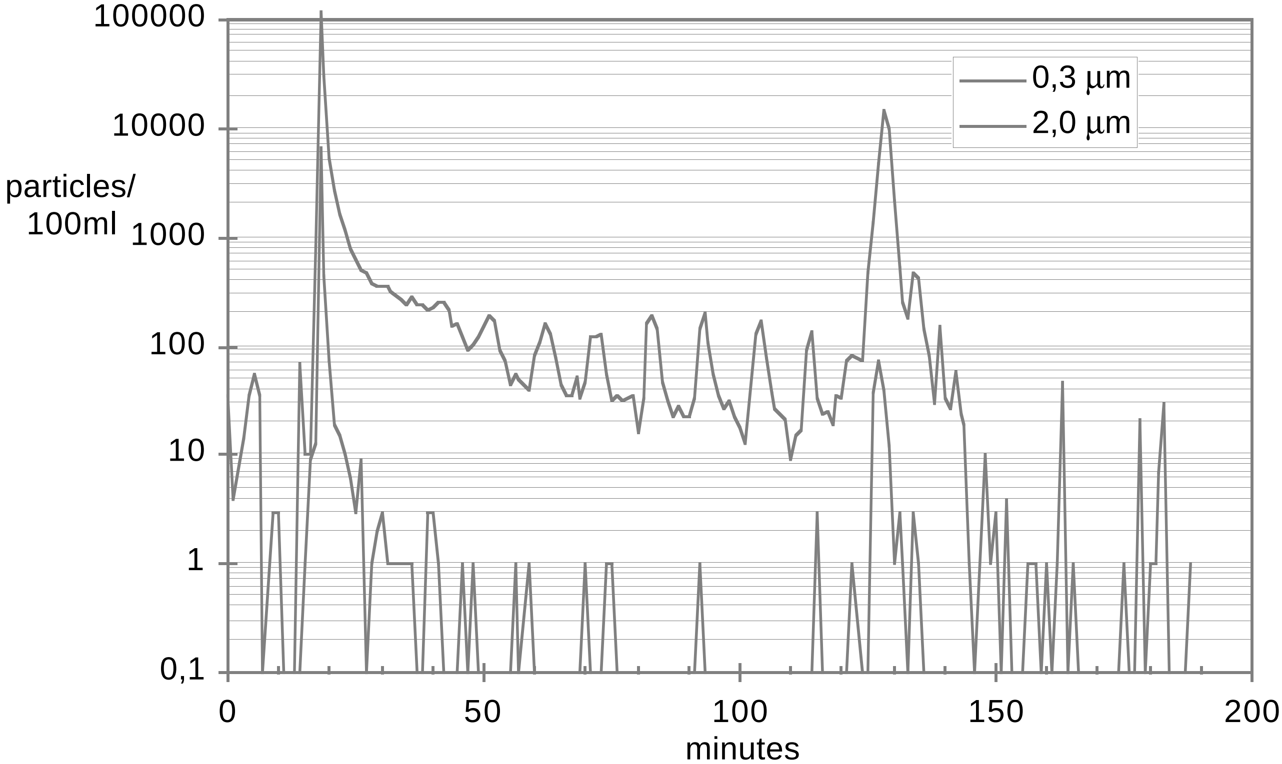


Fig.8: High pressure rinsing of cavity C22 (30.07.97)
(Attention to the changed scale)

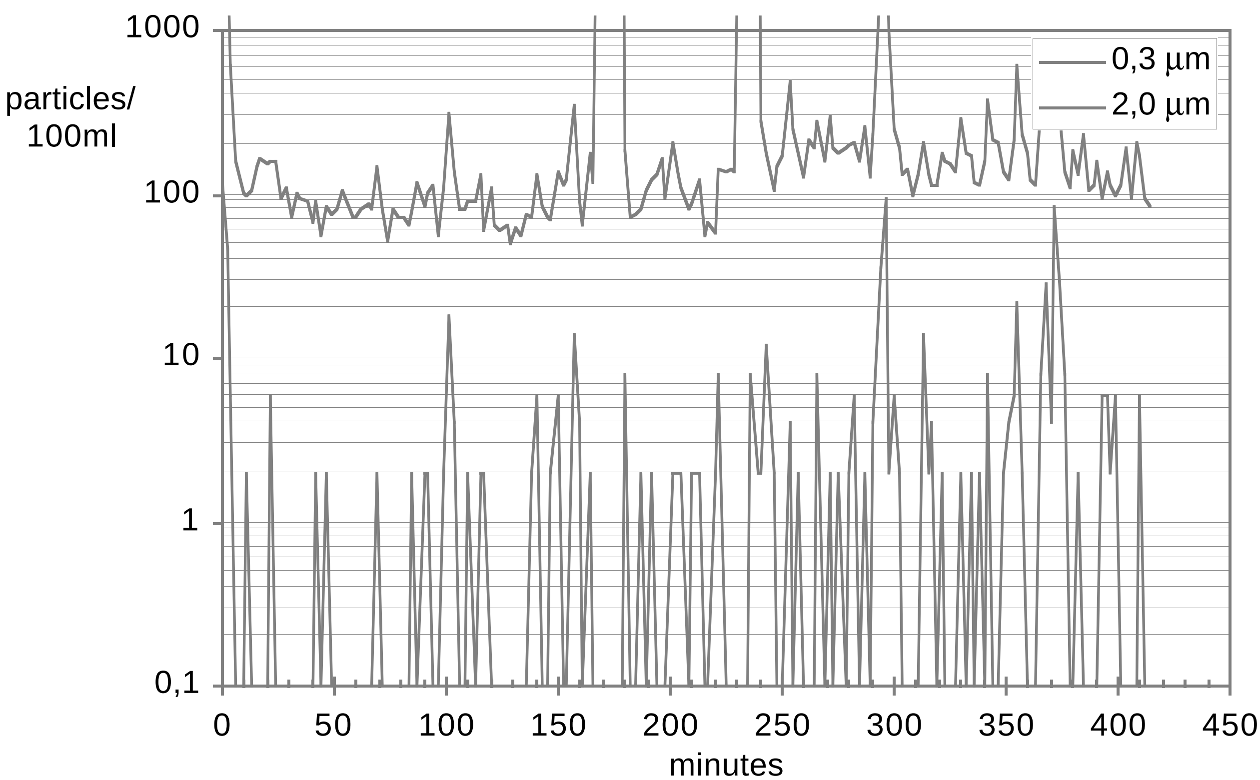


Fig.9: Sampler operation at the PoU 'HPR'
(particles per 100ml ; 15.04.97)

In addition, further analysis of the water collected in the funnel, like resistivity measurement, membrane filters for microscopic read-out, etc., is under comissioning. A TOC sensor is installed and the TOC is less than 5 ppb during routine operation of system. During HPR bubbles disturb the measurement. The future work will be concentrated on avoiding air bubbles

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