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PARTICULATE SAMPLING AND ANALYSIS DURING REFURBISHMENT OF PROTOTYPE EUROPEAN XFEL CRYOMODULE*

N. Krupka†, C. Bate, D. Reschke, S. Saegebarth, M. Schalwat, P. Schilling, S. Sievers,
DESY, Hamburg, Germany

Abstract

The cryomodule PXFEL3_1 is one of three prototype cryomodules for the European XFEL. In preparation of the series module assembly it was used for the qualification of infrastructure and personnel at CEA Saclay. After transport and tests at DESY the cryomodule was stored for several years. Last year we decided to refurbish this module with new cavities for the installation in the FLASH accelerator.

During the disassembly of the cavity string in the clean room at DESY we took several particulate samples for analysis. Optical and laser optical microscopy give us an insight on the quantity and type of the particulates. We expect to get hints where the particulates come from and how they are transported through the cavity string during transport and operation.

INTRODUCTION

Inspired by the work at Thomas Jefferson National Accelerator Facility [1] we decided to probe the cavity string of PXFEL 3_1 for particulate contamination during disassembly in the clean room.

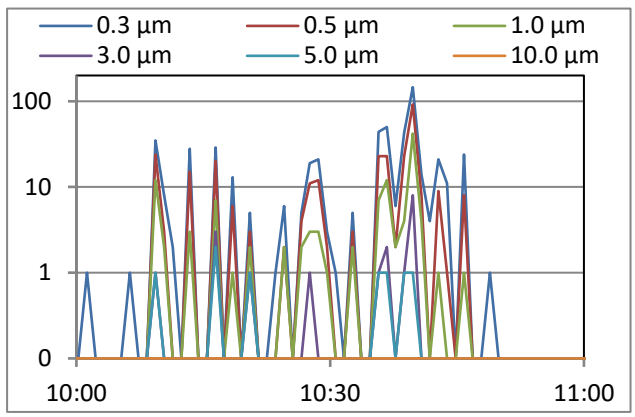

Figure 1: Typical particle count (measured in 1 minute interval) during the disassembly of a bellow between two cavities.

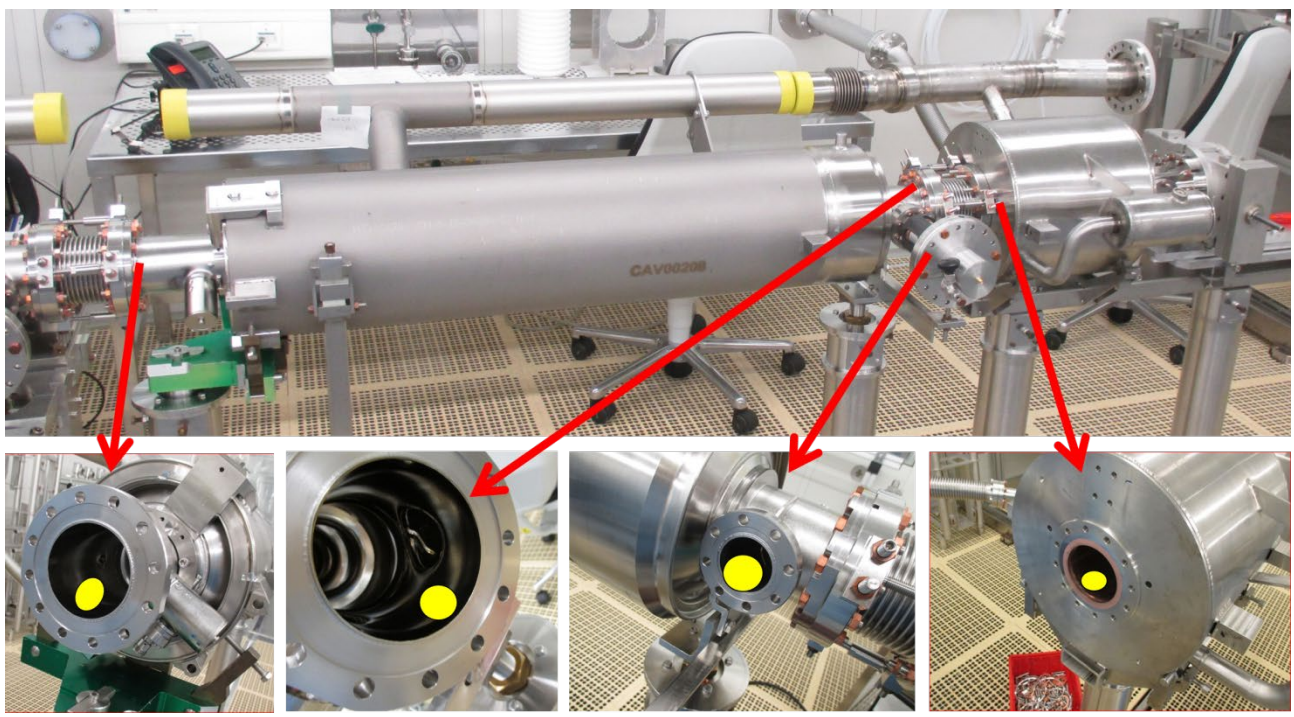

Figure 2: Cavity string in the DESY SRF clean room. The yellow spots represent the positions where the samples have been taken.

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† nicolay.krupka@desy.de

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The assembly and disassembly of cavity strings takes place in the ISO 4 part of the DESY SRF clean room, which has an area of 53 m² integrated in the almost 120 m² ISO 5 area [2]. The velocity of the laminar air flow on working height is about 0.55 m/s. During the disassembly we monitor the working area by particle counter. Figure 1 shows a typical particle count over one hour during the disassembly of a bellow between two cavities.

To prevent particulates from entering the open connections the cavity string is flushed from both ends with filtered N_2 with a rate of about 10 l/min.

During disassembly three flanges on each cavity are opened and can be probed. Both beam tube flanges (long side (L) and short side (S)) and the main coupler flange (MC). The HOM and pick-up couplers remain untouched.

In sum we collected 25 samples, 8 x 3 from the cavities plus one from the quadrupole of the BPM quadrupole unit (BQU). The yellow spots in Fig. 2 represent the positions where the samples have been taken.

SAMPLE TAKING, PREPARATION AND ANALYSIS

The probing of the cavity string should be quick and as clean as possible. The only retreatment step foreseen for the cavities from PXFEL3_1 before their further use for another accelerator module was a high pressure water rinsing (HPR) process [3]. Additionally we wanted to use mainly tools, materials and facilities which were already available.

For the daily particulate count to monitor the quality of the HPR processes we use an optical microscope inside the clean room [4]. This microscope was also chosen for the analysis of the samples from the cavity string.

As the analysis of the samples taken with Millipore Isopore filters from the HPR is automated, it was obvious to use these filters also for the probing of the cavity string. The filters are made of polycarbonate and have a pore size of 2 µm. They have a diameter of 47 mm and a thickness of 23 µm. The computer analysis scans a 25x25 mm² area of the filter in bright field and in dark field with a magnification of 50.

First we tried to swab the contamination with “cleanfoam swabs” from the cavity surface onto the filter discs (Fig. 3). With this method we could not detect any particulates on the filters. As we expected to find at least some particulates, we tried another method. We placed the filter discs with plastic tweezers inside the cavities and pressed them directly onto the surface. This way we were able to transfer particulates from the surfaces to the filter discs for analysis (Fig. 4). Unfortunately we have no assumption of what fraction of particulates can be removed from the surface of the cavity with this method.

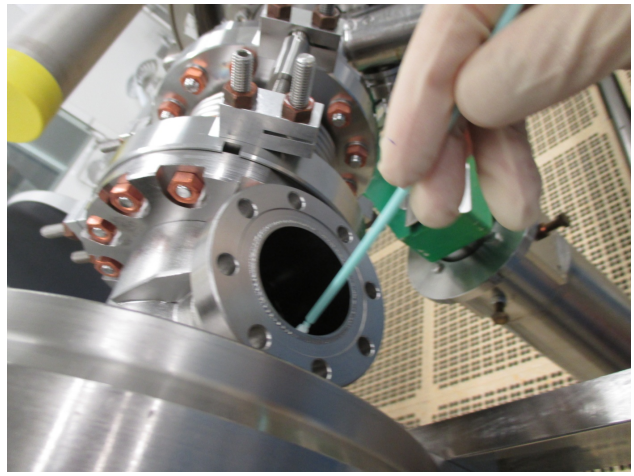

Figure 3: Swabbing contaminations from cavity main coupler port.

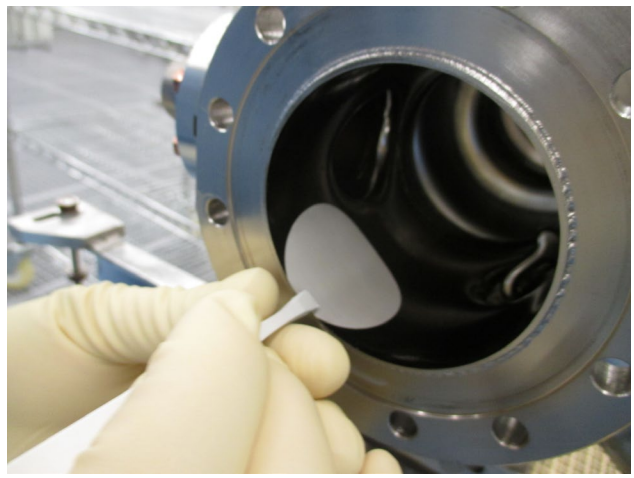

Figure 4: Insertion of a filter disc with plastic tweezers.

Table 1: Particulate Count of the Different Spots

Cavity Position	Sample from		
	Beam tube long side	Beam tube short side	Main Coupler
1 (AC124)	1	0	9
2 (Z138)	2	4	13
3 (Z135)	1	4	3
4 (Z134)	2	1	2
5 (Z104)	1	0	1
6 (Z101)	0	2	44
7 (Z97)	5	0	15
8 (Z140)	0	1	4
Sum	12	12	91
BQU	16	-	-

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Table 2: Material and Number of Gathered Particulates from PXFEL3_1

Cavity Position	Number of particulates							
	Copper	Aluminium	Organic	Niobium	NbTi	CuNiSi	undefined	Sum
1 (AC124)	0	0	1	1	0	1	7	10
2 (Z138)	1	0	9	1	0	0	8	19
3 (Z135)	0	0	4	0	0	2	2	8
4 (Z134)	0	0	1	1	1	1	1	5
5 (Z104)	0	0	0	0	1	0	1	2
6 (Z101)	15	0	14	5	6	4	2	46
7 (Z97)	2	0	2	8	1	8	2	20
8 (Z140)	0	0	1	1	1	2	0	5
Sum	18	0	32	17	10	15	23	
Origin	gaskets, coupler	gaskets	personnel	cavity	flanges	nuts		

Table 3: Size Distribution of Gathered Particulates

Particulate size in µm	Sample from		
	AC124 MC	Z97 MC	6 hours of final HPR
Less than 6	2	1	5
6 to 12	4	4	10
12 to 18	2	2	3
18 to 24	0	4	4
24 to 30	0	2	2
30 to 36	1	0	2
36 to 42	0	1	1
42 to 48	0	0	1
48 to 54	0	1	0
54 to 60	0	0	0

The automated scan of the filters gave us the particulate count only. The identification of the material of single particulates has to be done manually. For this process we use well-known reference samples and profit from the experience gained by the daily quality control of meanwhile more than 2800 filters from the HPR cycles during the last 18 years.

Tables 1 and 2 give an overview of the particulates we found. The first table shows the particulate count on the 25x25 mm² area of each sample. In the second table the particulate count is summed up for the single cavities but shows the distribution of different materials. Table 3 shows the size distribution of the particulates for the sample taken at the main couplers of AC124 and Z97. For comparison the result of the count of an HPR filter of the last 6 hours of the final 12 hour rinse is added.

As the analysis with the optical microscope only gives us two dimensions of the found particulates, we investigated one filter further with a 3D laser scanning microscope (LSM). The LSM is routinely used to scan the 3D surface geometry of replica and has a lateral resolution of 1 µm [5]. We used the LSM to determine the size of the particulates in all three dimensions. An example average organic and non-organic particulate is shown in Fig. 5.

RESULTS

Most samples had a very low amount of particulates in the range of up to 15, with one outlier of 44. There was no difference to be seen between the samples from the both beam tubes of the cavities. The samples from the main coupler areas showed much more particulates, including the outlier.

Most materials are of known origin. The copper particulates can be explained with the couplers and the CF-gaskets used at the angle valves. Niobium and NbTi originate from the cavity itself and the nuts we use are made in CuNiSi. The organic particulates are probably introduced by the personnel, but could also be residues in the ultra-pure water. Most interesting of course are the "undefined" particulates which will be further analysed.

The particulate sizes found during disassembly are in accordance to the particulates typically removed with the HPR process of the cavities surface. We used the LSM to determine the size of the particulates in all three dimensions, found on the filter.

The copper particulate from Fig. 5(a) has a diameter of approximately 50 µm and a height of about 5 µm and thus has a relatively flat appearance as can be seen from the profile measurement. The organic particulate in Fig 5(b) measures more than 60 µm in height and therefore exceeds the laser focus capability to get the exact full height. It seems to pile up on the filter as can be seen from the profile.

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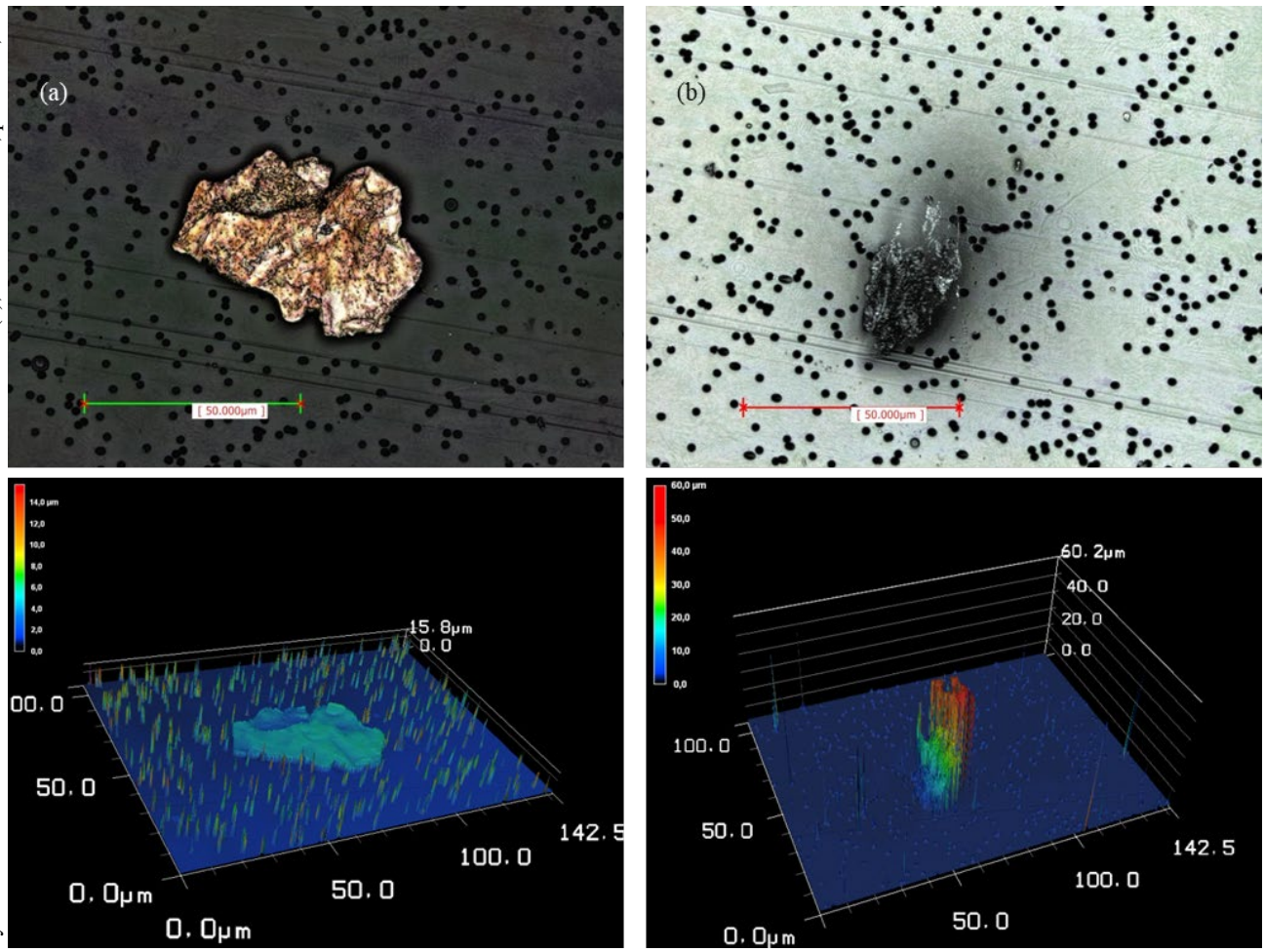

Figure 5: LSM analysis of (a) a Copper and (b) an organic particulate.

CONCLUSION

During the disassembly of PXFEL3_1 we probed a cavity string for the first time for particulate contamination inside the cavities. In total we took samples at 25 spots in the string and analysed the samples. As we expected, the count of particulates is quite low and most of the particulates are of known materials. The 23 particulates of undefined material will be further analysed. A scanning electron microscope (SEM) analysis will be applied and used to determine metallic materials. As the SEM we use has no automated particulate recognition, this method will only be used for selected single particulates, which could not be identified by the optical microscope.

For the next disassembly process we aim for an improved probing method. Mainly we want to make sure that we transfer nearly every particulate on the surface to the sample.

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