

# SURFACE INVESTIGATION ON PROTOTYPE CAVITIES FOR THE EUROPEAN XFEL

X. Singer<sup>#</sup>, S. Aderhold, A. Ermakov, W. Singer, K. Twarowski, (DESY, Germany)  
M. Hoss, F. Schölz, B. Spaniol (W.C. Heraeus, Germany).

## Abstract

Accelerating gradient Eacc of XFEL prototype cavities manufactured at the industry and treated at DESY demonstrates wide-range scattering from 15 to 41 MV/m. Most cavities satisfy the XFEL specification. Few cavities with low performance (15-17 MV/m) are limited by thermal breakdown without field emission. The T-map analysis detected quench areas mainly close to the equator. Optical control by high resolution camera has been applied and allowed to monitor the defects in some cases with good correlation to T-map observation.

In order to understand the cause of reduced performance and get more detailed information of the origin of defects, some samples have been extracted from two cavities and investigated by light microscope, 3D-microscope, SEM, EDX and Auger spectroscopy.

Several surface flaws with sizes from a few  $\mu\text{m}$  to hundreds of  $\mu\text{m}$  detected by microscopy.

The defects can be separated into two categories. The first category of defects consists of foreign elements (often an increased content of carbon). Inclusions with increased content of carbon adhere on the surface and presumably have a hydrocarbon nature. Deviation from a smooth surface profile characterizes the second type of defects (holes, bumps and pits).

## INTRODUCTION

As described in [1] ca. 50 prototype cavities have been mechanically manufactured at the industry by two vendors, treated (partially at the factory and partially at DESY) and RF-tested at DESY. The treatment for all cavities was: removal by EP of a 110-140  $\mu\text{m}$  surface layer, followed by an ethanol rinse and 800°C annealing under UHV conditions. Two options of final treatment have been applied. Final EP of 40-50  $\mu\text{m}$  with subsequent ethanol rinse, ultra pure high pressure water rinsing (HPR) and 120°C bake or alternatively a final BCP of 10  $\mu\text{m}$  (called Flash BCP), HPR and 120°C bake. The XFEL requirements are fulfilled with a yield of approximately 90%.

The main task of this work was concentrated on five cavities (Z110-Z111, Z130-Z132) produced by one vendor that have an Eacc below 20 MV/m. As it can be seen in Fig. 1a the  $Q_0(\text{Eacc})$  behaviour of these cavities seems to be similar: rather small change of  $Q_0$  between 5-15 MV/m and after that a thermal break- down at 15-17 MV/m. Four cavities were treated by Final EP and only one cavity (Z111) had the Flash BCP final treatment. One of the cavities (Z110) was subjected to attempt to improve performance by post purification with titanium by

annealing at 1400°C for 4 hours and subsequent BCP of ca. 100  $\mu\text{m}$ . Unfortunately, this did not lead to improvement of accelerating gradient.

In the last 15 years of cavity production for TTF (FLASH), a case of failure by the fabrication resulted in a sequence of 6 cavities (S7-S12) with reduced performance (Fig.1b) occurred. Thorough analysis at that time together with the company allowed to find out that after local grinding the surface was not sufficiently cleaned and contamination probably penetrated into material during electron beam welding.

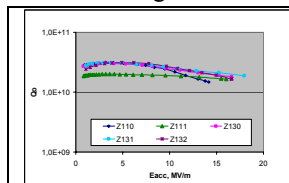


Figure 1a: Cavities of XFEL prototype manufactured with Eacc below 20 MV/m.

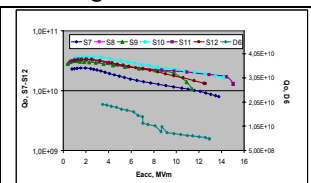


Figure 1b: Cavities S7-S12 and D6 of the 1st generation of FLASH fabrication.

It is interesting that two series of cavities with reduced performance demonstrate comparable behaviour (Fig.1a and Fig. 1b). Contaminated cavities S7-S12 exhibit a bit more steep  $Q$  reduction. In both cases Eacc is limited by a strong quench below 20 MV/m.

Analysis of the manufacturing steps of Z cavities at the company did not contribute to an understanding of the reason for failures. It was decided to cut the cavities and investigate the surface in the hot spot areas that have been indicated by the DESY T-map system for 9-cell cavities. Cavity destruction for investigation is a rare action. Only one of those intended for FLASH cavity (D6) with affected performance (see Fig.1b) was destroyed in the past [2] with success. A material flaw was detected clearly at the location identified by the T-map. A weak superconducting local defect:- cluster of the area of ca. 0,5  $\text{mm}^2$  with increased tantalum content was responsible for the local heating, that caused the quench and specific  $Q(\text{Eacc})$  behaviour (Fig. 1b), in particular, the abrupt  $Q_0$  drop at ca. 7 MV/m (Q-switch).

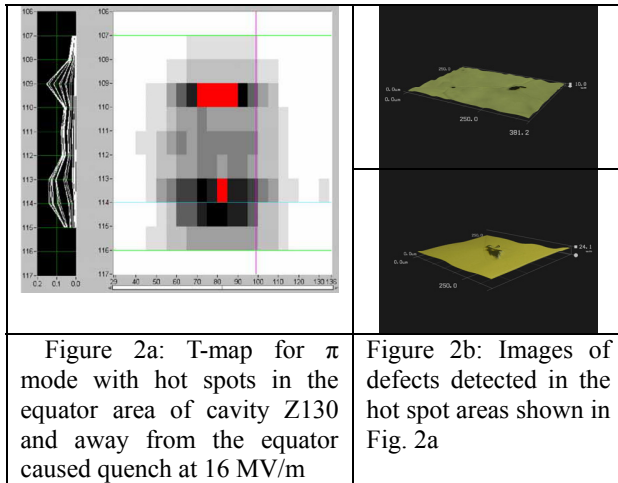
Several samples were taken from cavities Z111 (BCP-Flash treated) and Z130 (Final EP treated). Cavities additionally were previously high pressure water rinsed. The samples were taken from cavity quench areas or areas with increased heating that were localized by T-mapping during vertical RF-Test. Light microscopy, 3D-microscopy, SEM, EDX and Auger spectroscopy were used for investigation. Several surface flaws were detected. It has to be mentioned that the suspicious area localized by T-mapping has a dimensions in square centimetres, while the size of suspicious flaws lays in the

<sup>#</sup>xenia.singer@desy.de

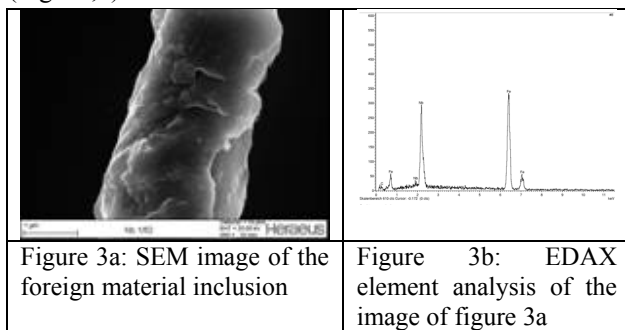
$\mu\text{m}$  order of magnitude. Therefore the absolutely unambiguous conclusion about which of the detected defects is responsible for the main heating in the local area is currently not possible. The detected defects can be separated in two categories. The first category of defects indicates foreign elements. Deviation from a smooth surface profile characterizes the second type of defects (holes, bumps and pits).

## DEFECTS WITH FOREIGN MATERIAL

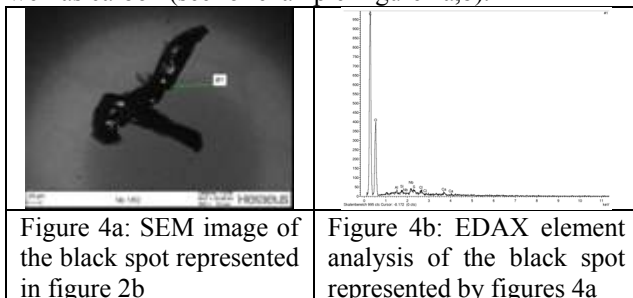
Foreign material inclusions in the hot spot areas were detected in both cavities.



Some examples of the foreign material inclusions can be seen below. Many surface inclusions with sizes from a few  $\mu\text{m}$  to hundreds of  $\mu\text{m}$  were detected by SEM on two samples separated from the cell 1 of the cavity Z130 (Fig. 2a,b). One inclusion indicates increased content of iron (Fig. 3a,b)

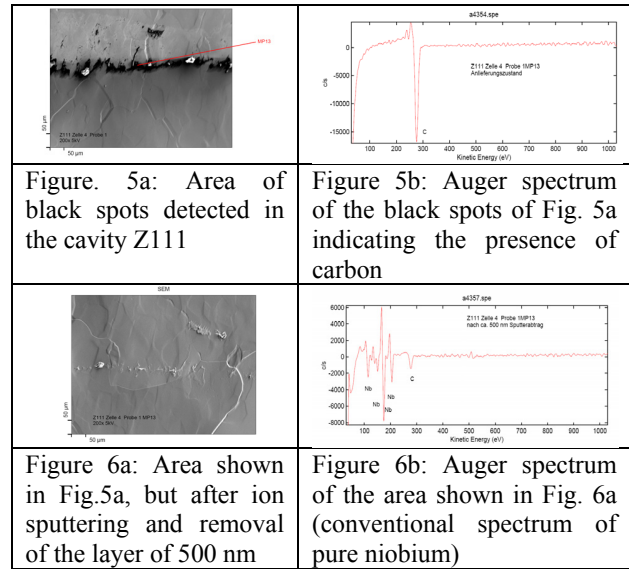


Most inclusions indicate increased content of carbon. Some inclusions indicate increased content of oxygen as well as carbon (see for example Figure 4a,b).



Similar black spots of different shapes have been observed in the cavity Z111 too (see for example figure 5a). At first glance it seems that the black spots lie on the surface (Fig. 2b upper image for example) and do not stick in, but more precise analysis shows that the material of the black spots adheres to the surface (Fig. 2b beneath).

One of the black areas (see image in the Fig. 5a) with pronounced carbon signal in the Auger spectrum (Fig. 5b) was removed by ion sputtering. The black spot and the carbon signal disappeared only after many cycles of sputtering and removal of a layer of 500 nm.



## TOPOGRAPHICAL DEFECTS

There is a lot of discussion in the community, whether or not and, if yes, how much the surface topography influences the cavity performance. At least two found topographical defects are doubtless correlated with cell performance. In both cases the defects were in the equator weld area.

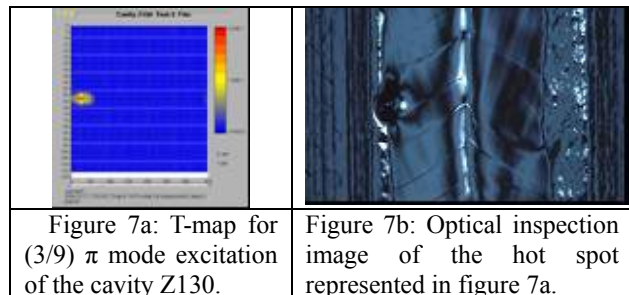
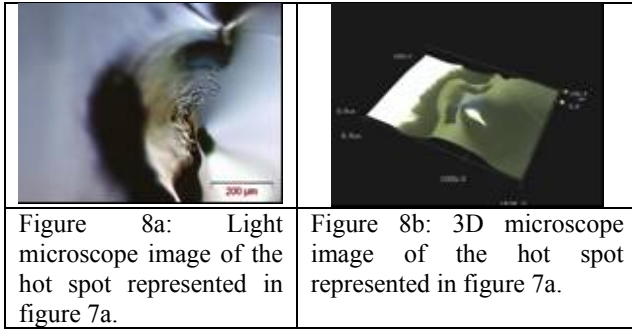
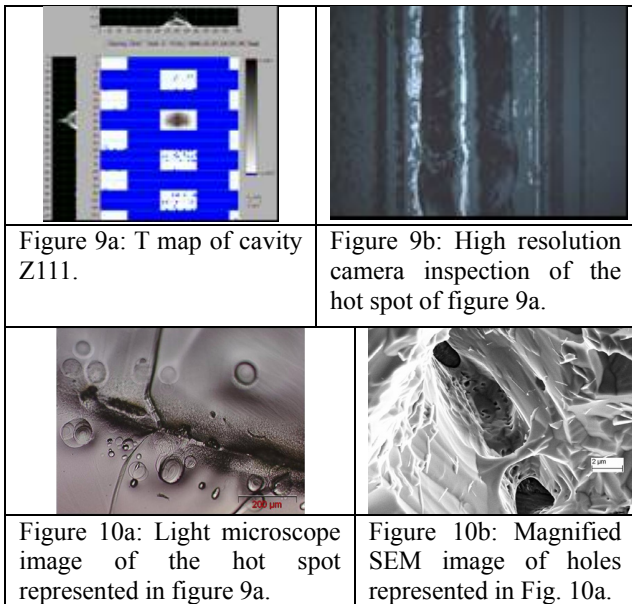


Fig. 7a shows the T-map for (3/9)  $\pi$  mode excitation. Quench at 23,3 MV/m was found in cell 5 of the cavity Z130. Optical inspection done on the cavity by high resolution camera discovered a topographical defect of ca. 1mm size on the interface between the welding seam and heat affected zone (Fig. 7b). The defect area was studied more carefully on the separated sample. No foreign material was monitored. The topography of the defect with a big distance between the peak and the valley (more

than 150  $\mu\text{m}$ ) can be seen on images of light- and 3D microscopes (Fig. 8a,b).



Another topographical defect that limited the accelerating gradient by thermal breakdown at 16 MV/m were detected in cell 6 of cavity Z111 on the equator weld. A group of beads 1.5mm wide were observed in the heating location (Fig. 9a) by optical inspection (Fig. 9b).



More details have been observed on the sample: several holes positioned mainly along a grain boundary were clearly visible in the light microscope image (Fig. 10a). Smaller holes inside of the bigger hole were detected by high SEM resolution. The impression gained was that of local corrosion taking place in this area. Several etching pits were positioned around corrosion holes.

A lot of etching pits of different shapes and sizes were observed on the inside surface of cavities. It seemed that the nature of the pits in the heat-affected zone of the welding seam and in the other cell areas were different. Analysis of the pits in particular from the point of view of grain orientations is in progress and will be discussed separately.

## SUMMARY AND OUTLOOK

Both foreign material inclusions as well as topographical flaws were found and can be classified as sources of the thermal breakdown in investigated cavities.

If the reason for the quenches is understood, the explanation of the reason that such defects appear on the cavity surface is more difficult. Only more or less plausible speculations are possible here.

For example, it could be highly probably supposed that imbedding of iron particles happened during deep drawing or handling of subassembly parts at the factory. Particles were already present during EP treatment; the surface of the particles is smooth as usual after EP. On the other hand they were not present in the niobium sheet. The sheets were provided to the vendor after eddy current scanning [2]. Iron, as a magnetic material, contributed significantly to eddy current signal and definitely would be detected.

The topological defect presented in Fig. 7-8 seemingly could be caused at the factory presumably as a result of not sufficiently getting rid of the flake after machining; or imprecise clamping, or possibly a welding error in this location.

More difficult to explain is the cause of the black spots as shown in Fig. 2 and Fig. 4-6. The Auger spectrums indicate attendants of hydrocarbons. On one hand a lot of hydrocarbons surround the electron beam welding EBW procedure (not sufficient part degreasing, oil fractions from the diffusion pump, rest of oils on the wall of the EBW chamber etc.). At the same time it is difficult to imagine, even if the surface were contaminated, how the contaminants could survive the subsequent treatment including removal of the surface layer of ca. 150  $\mu\text{m}$  thick. On the other hand a lot of hydrocarbon-based material is used into treatment procedure too (polymers, alcohols etc.) which (if remains on surface) can adhere during 800°C annealing. Final EP removes these remainders possibly less efficiently compared to BCP Flash. Suspicion of this could perhaps explain why the bad cavities were mainly EP treated in the end. From this point of view the corrosion holes in Z111 can be considered as the residues remaining after etching away of the black spots by BCP.

The action of cavity destruction allowed us to get new information and some new knowledge. At the same time many questions remain: for example, why post purification followed by BCP did not improve the performance of the cavity Z110.

## ACKNOWLEDGEMENTS

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