EVOLUTION OF THE DEFECTS ON THE NIOBIUM SURFACE DURING BCP AND EP TREATMENTS

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Abstract

In the cavity production process some defects may appear on the niobium surface, after the chemical and/or electrochemical processes, mainly in the region close to the equatorial weld and in the HAZ (Heat Affected Zone). We have produced defects on the Nb surface, with geometrical characteristics similar to the defects that may be produced during the mechanical machining of the cavity cells. We have investigated the evolution of these defects, during the usual chemical and electrochemical surface preparation stages.

INTRODUCTION

The realization of the new large accelerator facilities, such as XFEL and ILC, forces a mass production of SC cavities with a reduced risk of presence of defects. Nevertheless some defects may appear on the cavity surface that may affect seriously the cavity performances [1,2,3,4]. Defects found on the Nb cavity surface, at the end of the production process, may be generated by the evolution during the etching process of defects already existing on the surface or produced during the mechanical fabrication of the cavity.

Therefore we produced artificial pits, bumps, scratches, cuts and holes, with different geometrical characteristics (depth, size, height, etc.), on Nb samples. After an optical inspection using a Leica DMRME metallographic microscope with IC (Interference Contrast), samples have been etched using procedures similar to the ones foreseen for the XFEL cavities production (BCP + EP + final EP /BCP). After each step of the etching procedure, the evolution of the defects has been analyzed and the results are reported in this paper [5,6].

EXPERIMENTAL SET UP

EP Experimental Set Up

A parallel plate electrolytic cell was set up (Fig. 1).

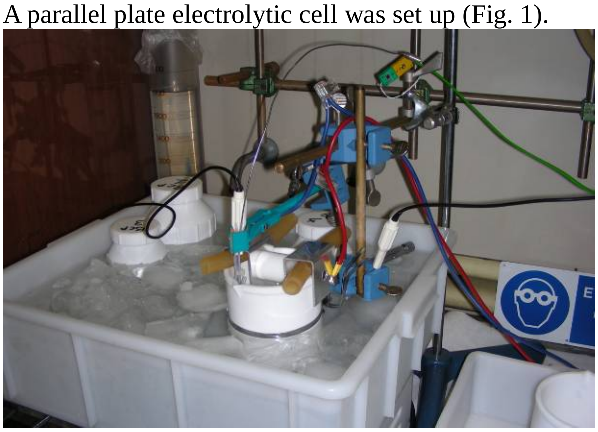

Figure 1: EP experimental set up.

Two vertical electrodes (front surface about 35 cm^2) with an electrode distance of 40 mm have been used. One electrode is pure Al (99.5 %) while the other one is the Nb plate to be treated. The EP solution (1 litre HF: H_2SO_4 1:9) is contained in a PTFE beaker kept at the desired temperature by immersion in a water basin cooled by ice. A magnetic stirrer agitates the acid during the EP (Fig. 2).

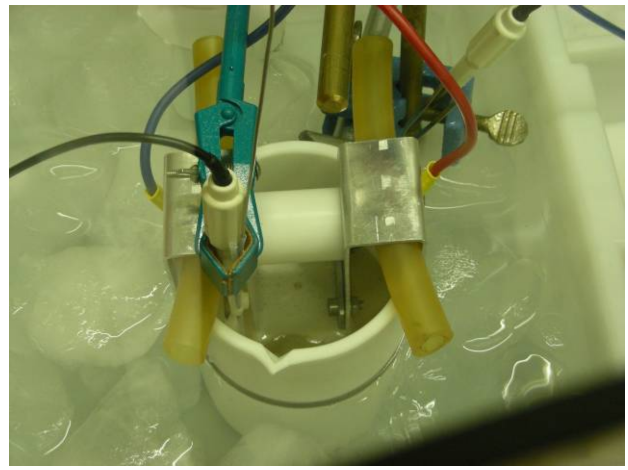

Figure 2: EP process: Electrical connections, electrodes (Nb and Al) and temperature sensors are visible.

The operative temperature is maintained between 20°C and 35°C: reaching this temperature the process is stopped waiting for acid to cool. Cell voltage and current are recorded at 1 Hz, together with the acid temperature. All measurements described in this paper have been done at constant voltage (17 V).

Typical behaviour of the EP process is shown in Fig 3.

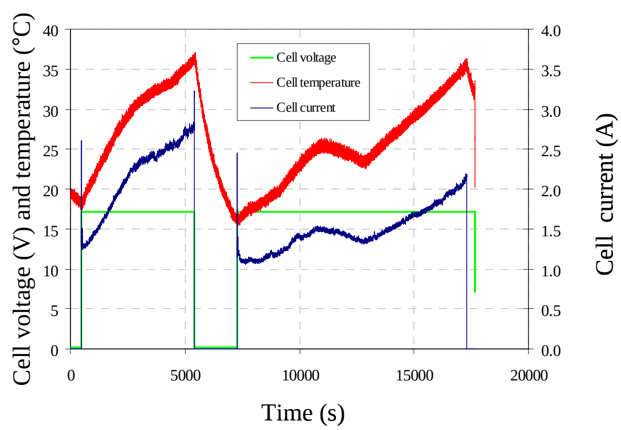

Figure 3: Typical behavior of the voltage, current and temperature during the EP process.

The ratio between the amount of Nb dissolved in the process and the charge passed through the cell gives information about the electrochemical reaction in the EP cell. Data of our processes are shown in Fig. 4. The angular coefficient of the curve indicates what kind of oxidation reaction is under way in the electrolytic cell. Data are in accordance with the formation of the Nb_2O_5 (2 electrons for each Oxygen atom, Oxygen/Nb ratio 2/5) as shown in Fig. 4.

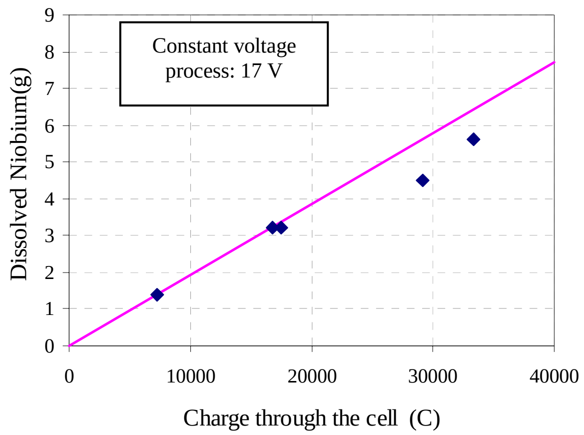

Figure 4: Behaviour of the dissolved Nb vs. the charge through the cell for 5 EPs. Blue dots are the experimental data, the pink line is the Nb_2O_5 theoretical value.

BCP Experimental Set Up

The BCP experimental set up (Fig. 5) use the same ice cooled basin and PTFE beaker as the EP one. Samples are suspended in the BCP solution (1:1:2, HF, HNO_3, H_3PO_4) while a magnetic stirrer ensures the acid agitation.

Figure 5: BCP experimental set up.

SAMPLE PREPARATION

Niobium Samples Preparation

Two kinds of Nb samples have been used: with and without e-beam weld. All samples have been etched with BCP 1:1:2 for 20 µm after the cutting and machining.

The Nb samples to be e-beam welded (RRR 300) have been firstly machined, in the welding area, to the FLASH 1.3 GHz equatorial area thickness. Later, these samples have been welded using the same parameters as for the equatorial e-beam weld of 1.3 GHz cavities. Nb samples without welds come from RRR 150 material.

Just before the artificial production of defects, all samples were etched with BCP (6 µm) to simulate the surface refreshment treatments (typ. 3 µm each) that are applied in the cavity production before welds.

Artificial Defects Production

We have produced a large number of controlled defects, with different shapes and dimensions, to observe their evolution during the BCP and EP treatments (see Fig. 6).

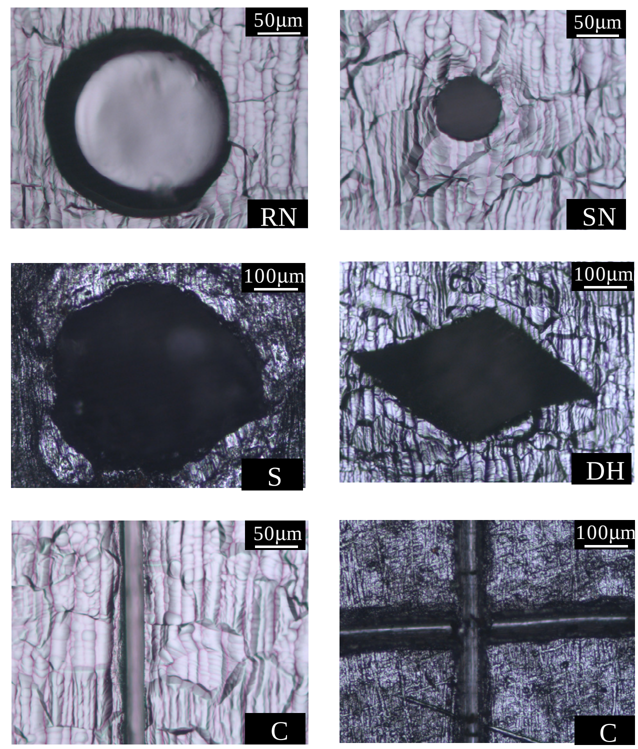

Figure 6: Typical defects produced on Nb samples. On the bottom, defects obtained with cutter used in compression (left) and scratching the surface (right).

The defect shapes have been chosen similar to the ones found during optical inspection of cavities [7,8,9]. Three different families of defect have been identified: pits, bumps, scratches (linear marks). Table 1 summarizes the main characteristics of the produced defects.

Table 1: Artificially Produced Defects

tool	shape	size (µm)	depth (µm)
RN	round	Φ = 130÷300	10÷50
SN	round	Φ = 30÷180	20÷240
S	round	Φ = 500÷700	280÷400
DH	rhomboid	d1 = 330÷630 d2 = 270÷380	40÷150
C	linear	width = 20÷60	30÷20

RN: Round Needle SN: Sharp Needle S: Screw
DH: Drill Head C: Cutter

Our work has been focalized mainly on pits and scratches. These defects can be due to different processes: mechanical machining or half-cell deep drawing. These defects have been produced with different tools (round and sharp head needles, cutter), changing either the pressure exerted on the Nb surface by the tool or the way they are used (i.e. the cutter has been used both scratching and cutting).

EP and BCP Treatments of Samples after the Artificial Defects Production

After the defects production and before any etching, images of samples have been taken using a flat scanner (2400 dpi), in order to have a full sample image with good resolution. These scans are used as a reference map for the distribution of the defects on the sample surface.

Tab 2 summarizes the different etching treatments on the four Nb samples.

Table 2: List of Treatments on the Different Nb Samples. Samples D2, D3 and P1 are e-beam welded.

Sample	Step 1	Step 2	Step 3	Step 4
D2	BCP 6.5 µm	EP 50.8 µm	EP 71.4 µm	--
S3	BCP 6.2 µm	EP 21.9 µm	EP 50.1 µm	BCP 13.2 µm
D3	BCP 6.4 µm	BCP 6.0 µm	--	--
P1	BCP 2.4 µm	BCP 50.0 µm	--	--

After inspection with the optical microscope, samples have been etched with EP and/or BCP.

The removal values reported in Table 2 for the EP treatment are the mean values of the EP process all over the sample surface: during the EP process, samples were partially electropolished also on the back side.

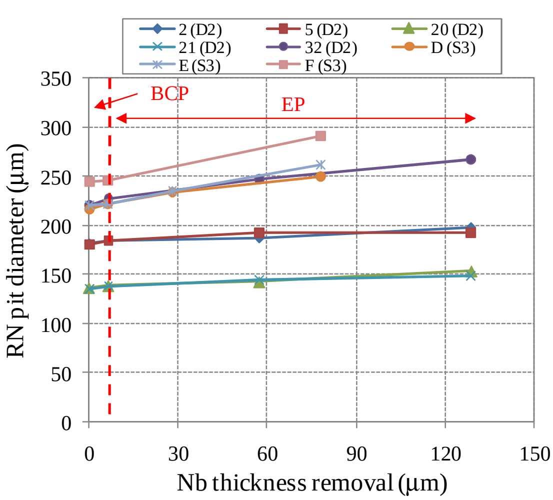

Figure 7: Diameter evolution vs. Nb thickness removal of RN pits.

After desired thickness removal, the treatment was stopped and samples were weighted. Images of the defects have then been acquired and used to study the defects evolution during the etching process.

ANALYSIS AND EVOLUTION OF THE ARTIFICIAL PRODUCED DEFECTS

Pits Evolution

We have firstly analyzed the evolution of Round Needle (RN) and Sharp Needle (SN) pits, produced on sample D2 and S3. We have observed the shape changes and measured the diameters and depths of the pits analyzing images acquired by the microscope. The diameter and depth changes measured for the RN pits, after each steps of the treatments, are shown respectively in Fig. 7 and 8.

In Fig. 7, we clearly observe an increase of the pit diameters with the material removal, more enhanced for defects with initial larger diameter.

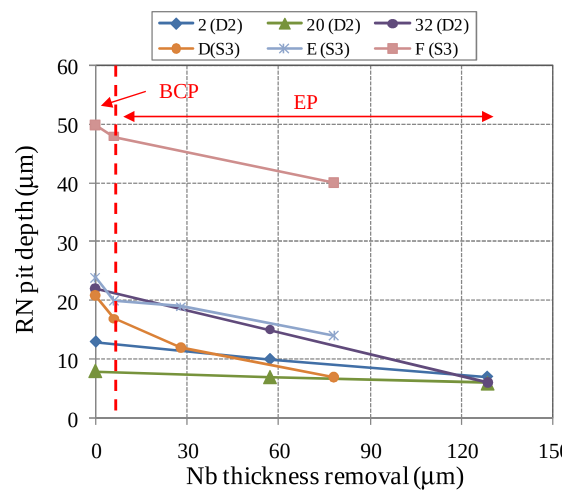

Figure 8: Depth evolution vs. Nb thickness removal of RN pits.

Fig. 8 reports the depth evolution of the pits. The pit depth decreases, as expected, but initial holes do not disappear even when the thickness of the removed material is larger, more than one order of magnitude, than their depth.

This is better highlighted in Fig. 9 where the evolution of two specific pits is reported. The schematic evolution of the two RN pit (#20 and #32) shapes, after 128.7 µm of Nb removal, is shown. Both pits change during the decrease of the surface thickness induced by the treatment. At the end of the etching process, the two pits have nearly the same depth, despite different initial values, and larger diameter than at the beginning. Fig. 9 also reports the microscope images of the RN pits under investigation before any treatment and after the last EP. These images clearly show that after the EP process, the diameter of the two pits are larger than at the beginning and the depth of the deeper pit is largely reduced.

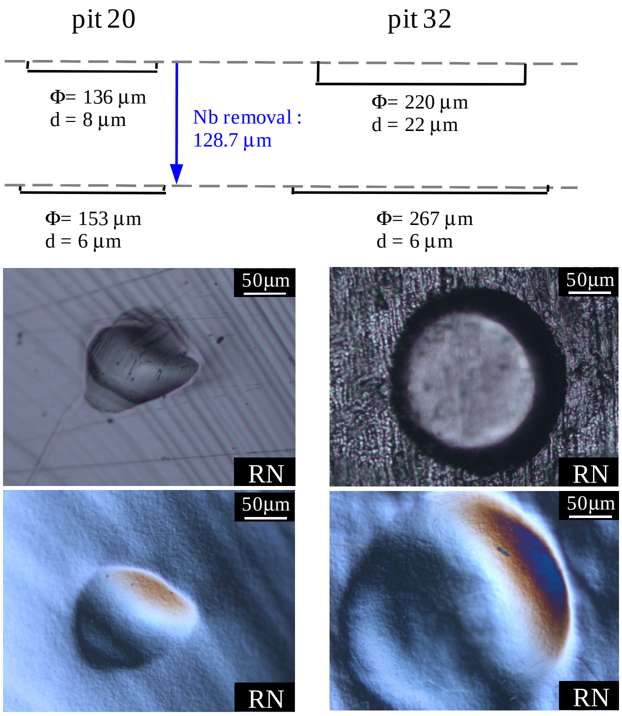

Figure 9: RN pits geometry changes during etching.

The same analysis of the evolution of depth and diameter has been done on SN pits. Fig. 10 and 11 report respectively the two summary plots of the behavior of the SN pit diameters and depths vs. the Nb thickness removal.

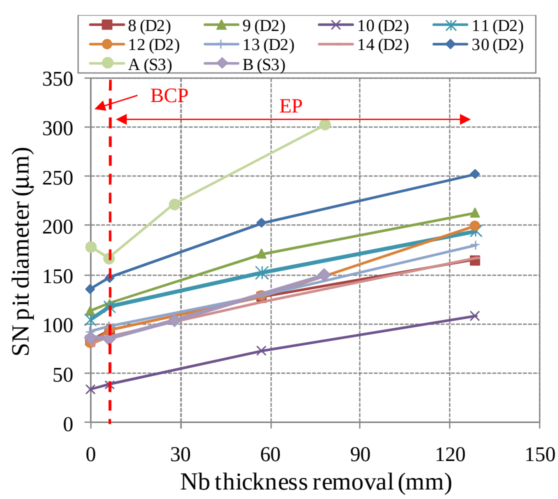

Figure 10: Diameter evolution vs. Nb thickness removal for SN pits.

The diameter evolution, as reported in Fig. 10, of SN pits show a larger increase with respect to the RN pits.

Concerning the depth change, reported in Fig. 11, the evolution is similar to the RN pits.

In Fig. 12 we show the evolution of two selected pits during the surface treatment (pit #10 and pit #30).

Also in this case we observe that, beside a large Nb thickness removal, the two SN pits are not removed. Also for SN pits a "hole", with a depth seven times smaller than the Nb thickness removed, still remains visible on the sample surface (pit #10).

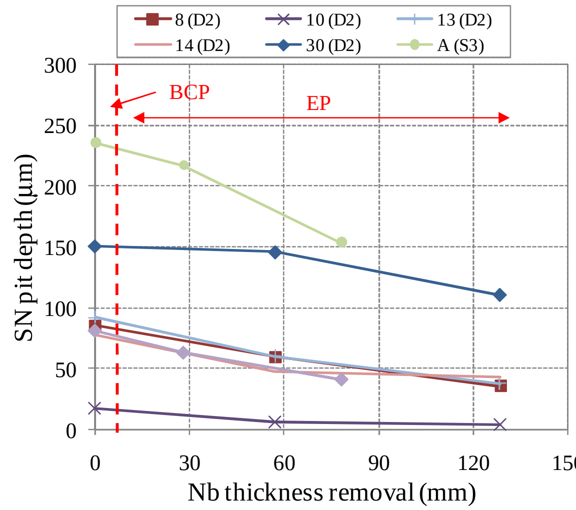

Figure 11: Depth evolution vs. Nb thickness removal for SN pits.

Also SN pits with depth larger than the thickness removal (pit #30) show a depth reduction lower than the total removed thickness. Images in Fig.12 show the increase in diameter due to the etching process.

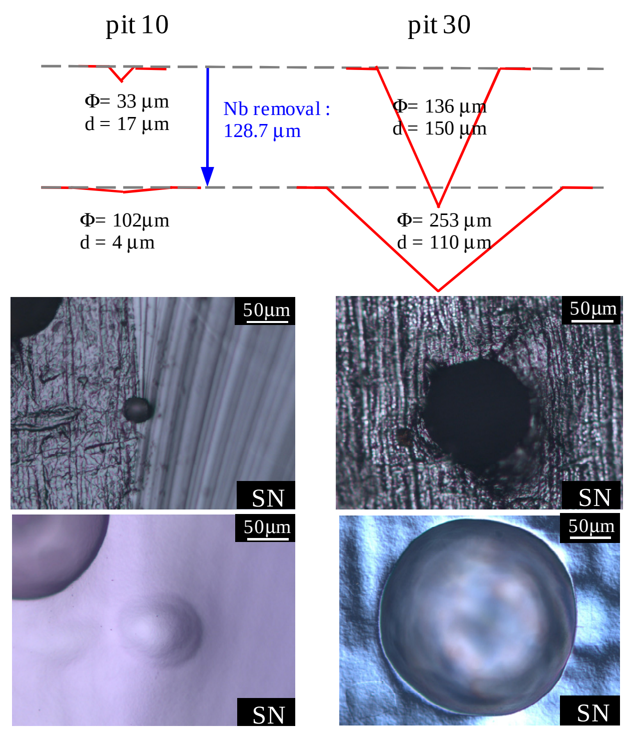

Figure 12: SN pits changes during etching for two different geometries.

Other Artificial Defects Evolution

In Fig. 13 the rhomboid defect (DH) and the linear defect (C) evolution during the various steps of the surface etching are shown.

DH defects have been produced compressing the surface with a small drill head (ϕ = 1.2mm). The main effects on DH defect are the edge rounding while the enlargement is less pronounced.

The C defects have been produced punching a cutter blade on the sample surface. The effect of the etching is an enlargement of the width more than a factor two.

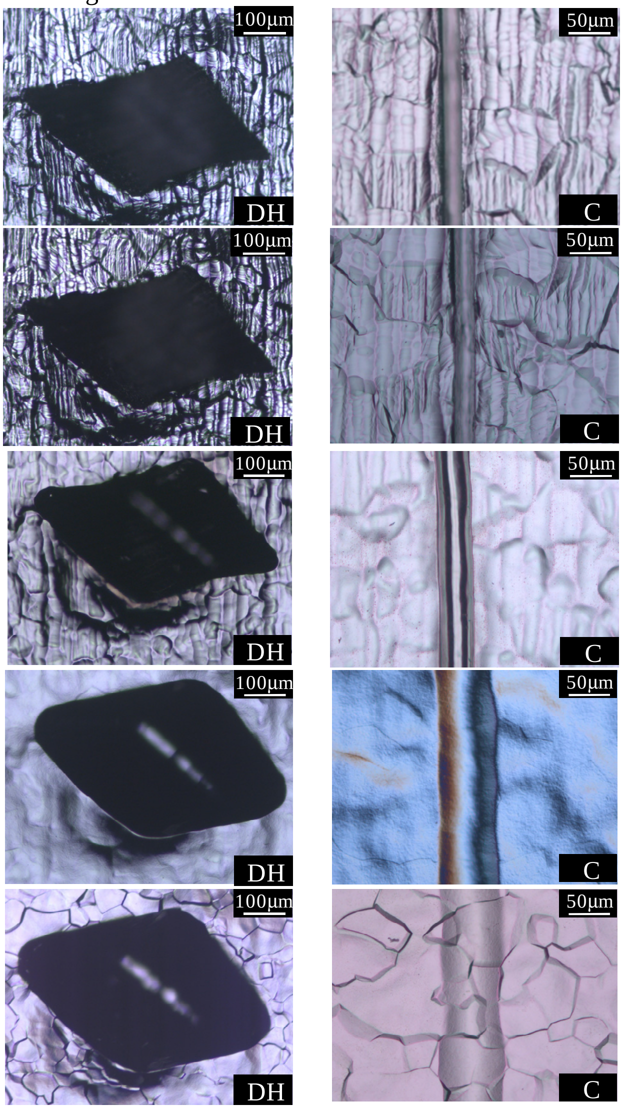

Figure 13: DH and C defects (sample S3) evolution during the surface etching. From the top: Before any treatment, BCP 6.2 μm, EP 21.9 μm, EP 50.1 μm, and final BCP 13.2 μm.

Not Artificially Created Defects Produced During the Various Etching Steps

Other defects appeared on the samples surfaces during the various etching processes but the ones that we have artificially produced.

In particular we have observed the formation of many circular defects during short BCP treatments (few microns) on a fresh Nb surface. They are well visible in the weld area and quite visible also all over the sample. They can be due to the presence of localized area with higher reactivity or to some gas bubbles attached to the Nb surface. These defects mostly disappear after long EP or BCP treatments. Fig. 14 shows the typical sample surface after short BCP and after 50 μm BCP.

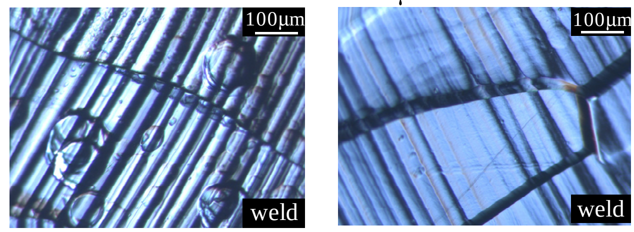

Figure 14: Typical image of circular defects on the weld after BCP treatments (left BCP 6 μm, right BCP 50 μm).

Anyhow an extremely light trace of these defects is sometimes still visible using the IC microscope also after long EP treatments (> 100 μm).

The same short time BCP process applied on an electropolished surface, for simulating the “flash BCP” etching after a long EP treatment, does not produce such defects.

CONCLUSIONS

The full etching procedures foreseen for the Nb cavity production do not completely remove defects from the surface. Also defects with dimensions less than one order of magnitude with respect to the etched material are still visible.

The typical behaviour of defects is an increase of their diameter, more evident for sharp needle pits.

No different evolution has been observed on defects produced on the welds, on the HAZ and in the bulk.

For these reasons extremely care should be used during the Nb sheet quality control, the sheet handling, the deep drawing and the mechanical machining, as in the handling of partially assembled parts (dumbbells, end groups).

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