Evolution of the Defects on the Niobium Surface During BCP and EP Treatments

European XFEL

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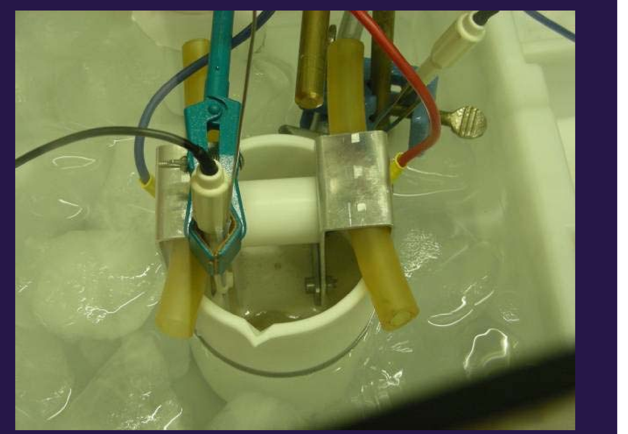
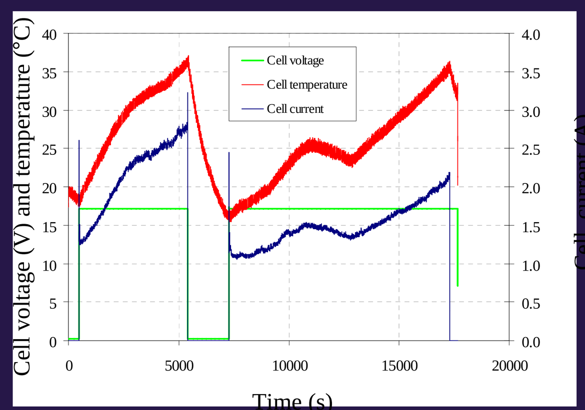
In the cavity production process some defects may appear on the niobium surface, after the chemical and/or electrochemical processes, mainly in the region close to the equatorial weld and in the HAZ (Heat Affected Zone). We have produced defects on the Nb surface, with geometrical characteristics similar 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.

Experimental Set Up

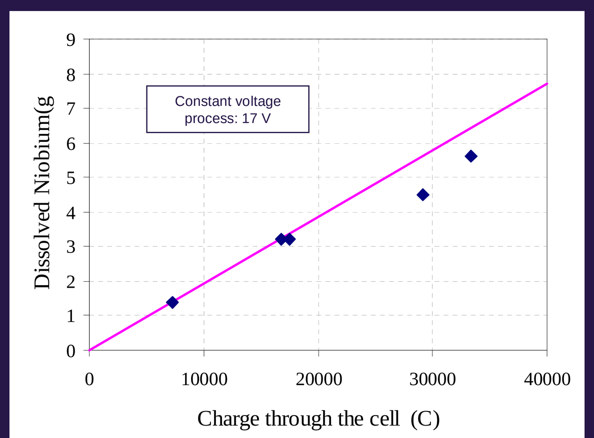
ElectroPolishing set up:

- EP solution 1 litre HF: H_2SO_4 1:9
- a parallel plate electrolytic cell
- two vertical electrodes
- front surface 35 cm^2
- electrode distance of 40 mm
- one electrode is pure Al (99.5 %)
- temperature: 20° C - 35° C
- constant voltage process: 17 V





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.
The angular coefficient of the curve indicates what kind of oxidation reaction is under way in the electrolytic cell. Our data are in accordance with the formation of the Nb_2O_5 (2 electrons for each Oxygen atom, Oxygen/Nb ratio 2/5) .

Buffered Chemical Polishing set up:

- the BCP experimental set up 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)
- a magnetic stirrer ensures the acid agitation.

Sample Preparation

Niobium samples preparation

- two kinds of Nb samples used:
  - with e-beam weld as for the FLASH 1.3 GHz equator (RRR 300)
  - without e-beam weld (RRR 150)
- all samples etched with BCP 1:1:2 for 20 µm
- before the artificial production of defects, samples etched with BCP (6 µm) to simulate the surface refreshment treatments

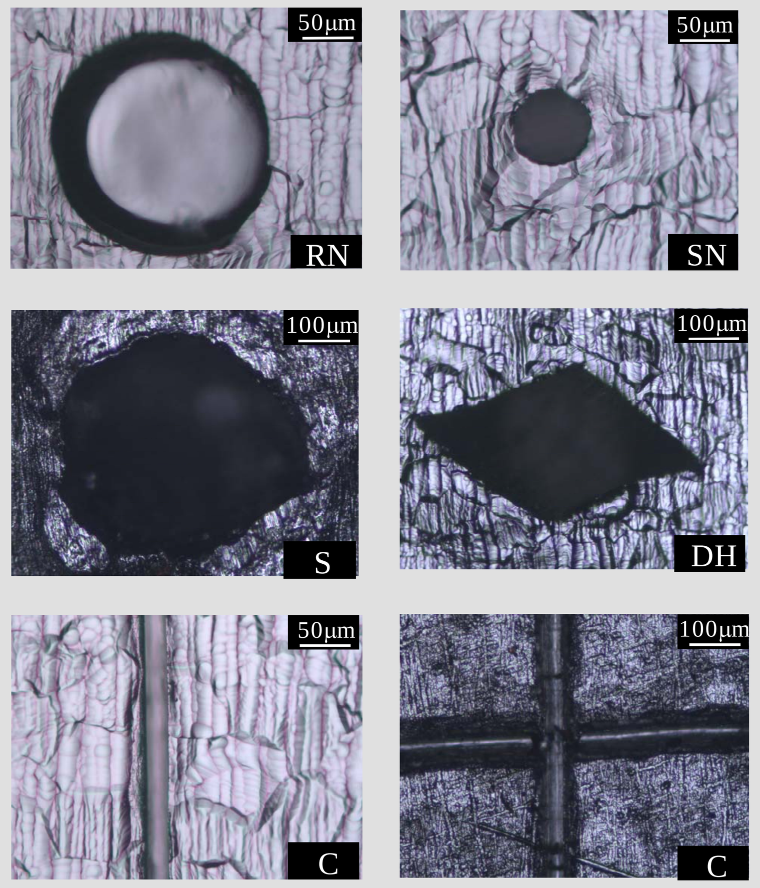
Sample and etching treatments

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	--	--

Artificial defects production

- produced a large number of controlled defects
- different shapes and dimensions of defects to observe their evolution during the BCP and EP treatments

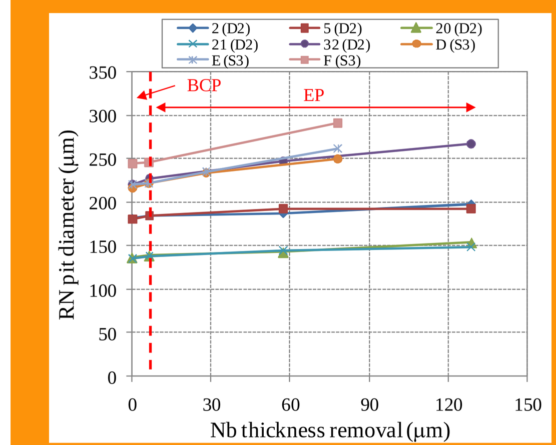
tool	shape	size (µm)	depth (µm)
RN	round	Φ = 130÷300	10÷50
SN	round	Φ = 30÷180	20÷240
S	round	Φ = 500÷700	280÷400
DH	rhomboid	d1 = 330÷630 d2 = 270÷380	40÷150
C	linear	width = 20÷60	30÷20

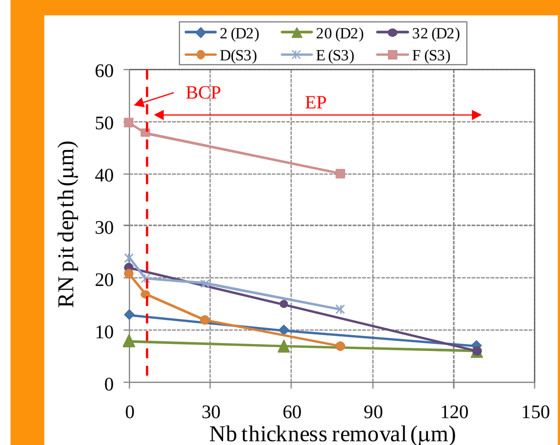


Analysis and Evolution of Artificial Produced Defects: RN and SN pits

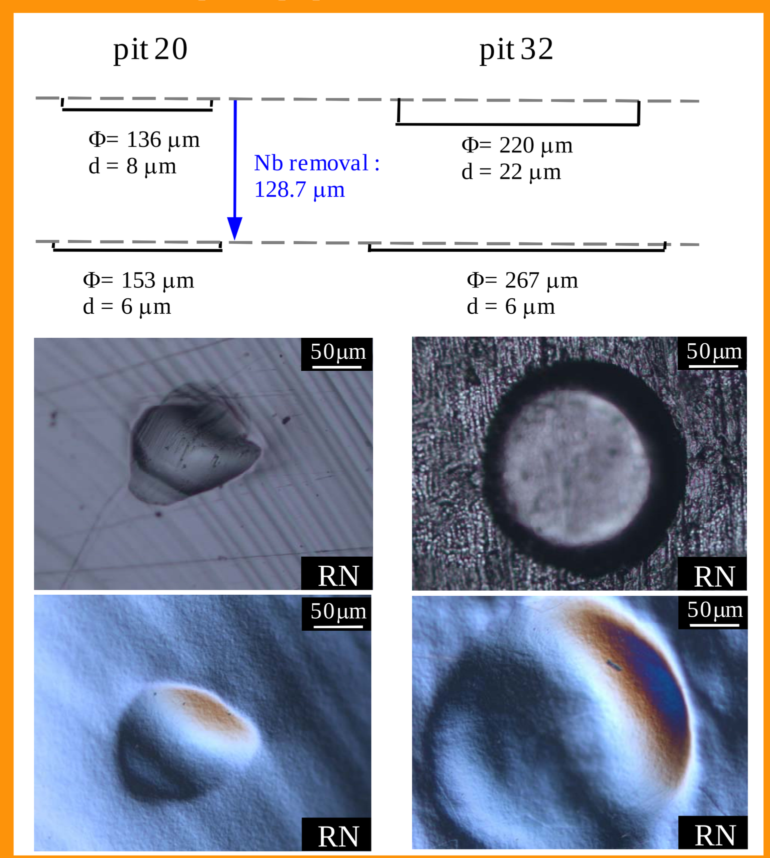
We have firstly analyzed the evolution of Round Needle (RN) and Sharp Needle (SN) pits, produced on sample D2 and S3.
We have observed the shape changes and measured the diameters and depths of the pits analyzing images acquired by the microscope.

Round Needle (RN) pits

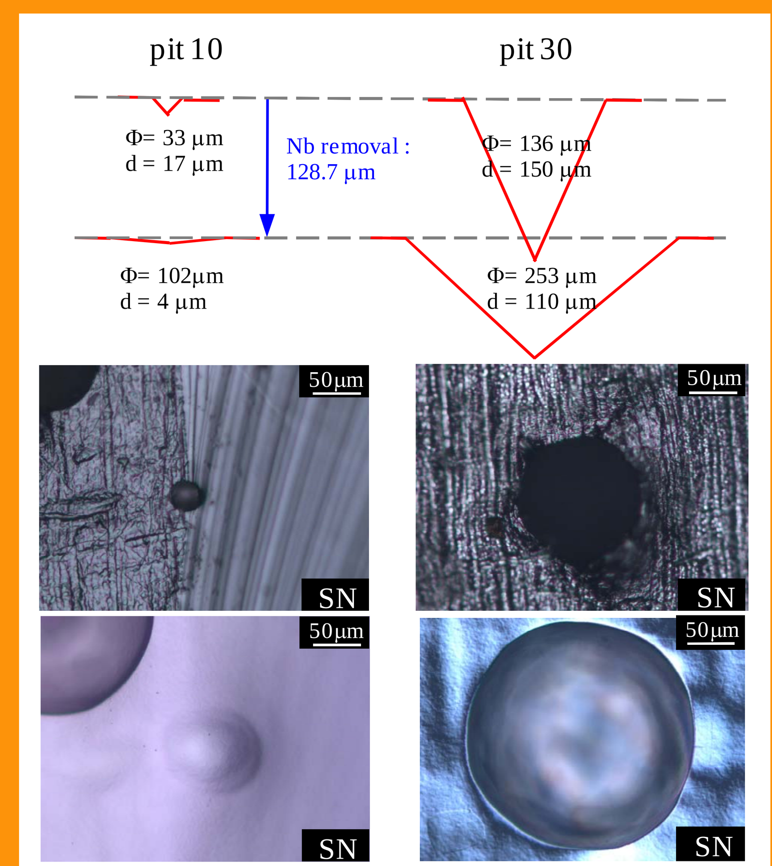




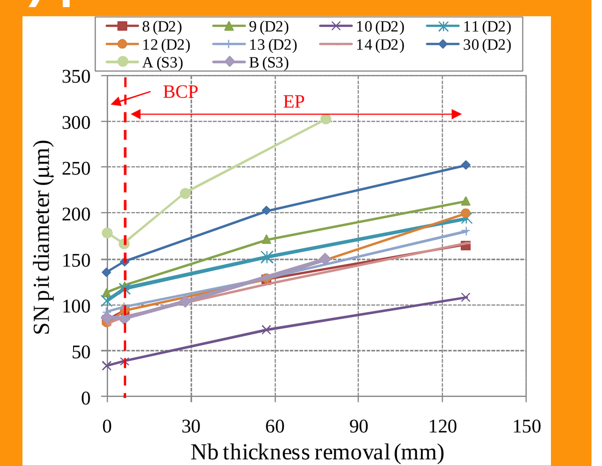
- increase of the pit diameters with the material removal, more enhanced for defects with initial larger diameter
- pit depth decreases but initial holes do not disappear even when the thickness of the removed material is larger, more than one order of magnitude, than their depth

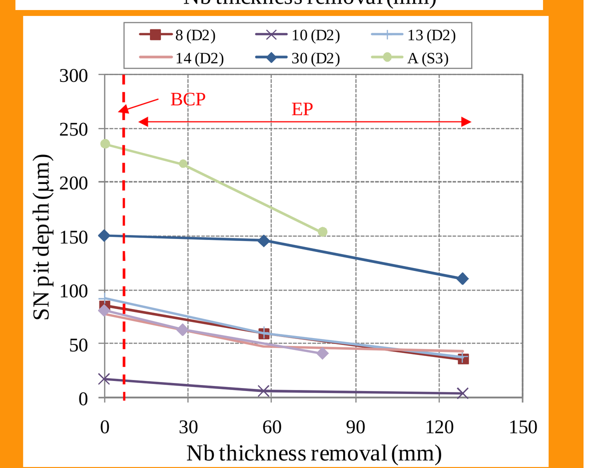


Sharp Needle (SN) pits



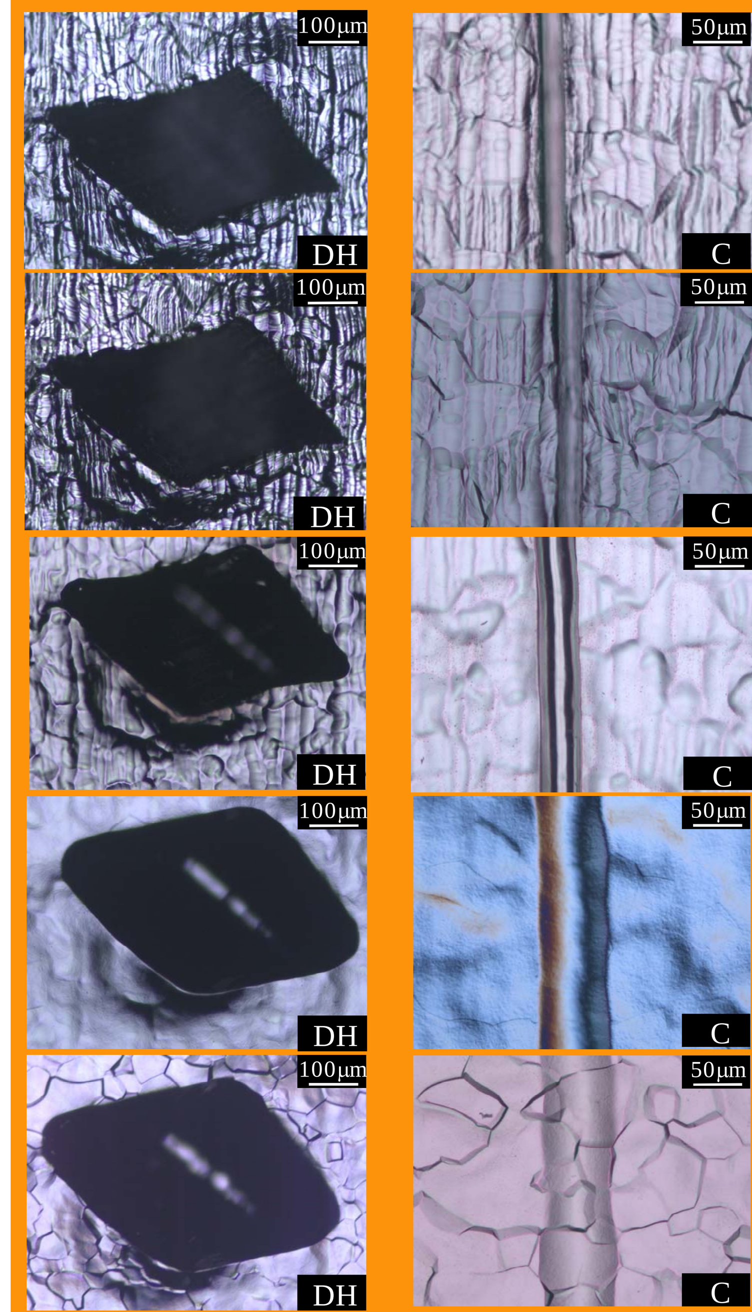
- schematic evolution of RN pits (#20 and #32) and SN pits (pit #10 and pit #30) shapes, after 128.7 µm of Nb removal
- all pits change during the decrease of the surface thickness induced by the treatment
- After the etching process:
  - RN pits have nearly the same depth, despite different initial values
  - RN pits have larger diameter than at the beginning
  - SN pits with depth larger than the thickness removal (pit #30) show a depth reduction lower than the total removed thickness
  - SN pits show a the diameter enlargement greater with respect to the RN pits





- 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).

Other Artificial Defects



before any treatment

BCP 6.2 µm

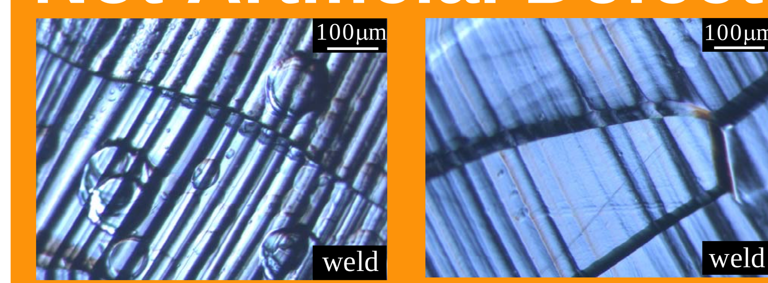
EP 21.9 µm

EP 50.1 µm

BCP 13.2 µm

- rhomboid defect (DH) and linear defect (C) evolution during the various steps of the surface etching:
  - DH defects produced compressing the Nb with a small drill head (ϕ = 1.2mm)
  - DH defects show edge rounding while the enlargement is less pronounced
  - C defects produced by punching a cutter blade on the Nb
  - C defects show an enlargement of the width more than a factor two

Not Artificial Defects



left: BCP 6 µm

right: BCP 50 µm

- other defects appeared on the samples surfaces during the various etching processes
- formation of many circular defects during short BCP treatments (few microns) on a fresh Nb surface, well visible in the weld area
- can be due to the presence of localized area with higher reactivity or to some gas bubbles attached to the Nb surface
- defects mostly disappear after long EP or BCP

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