

ALTERNATIVE APPROACHES FOR NB SUPERCONDUCTING CAVITIES SURFACE TREATMENT

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Abstract

With improvement of fabrication techniques and ultra cleanliness, the limitation of performances of SC cavities seems to be now limited by the surface state generated by the etching process. The influence of various surface treatment are now studied in several laboratories (KEK, DESY, CERN...). This papers presents the results obtained at Saclay, mainly on cavities, along with surface studies conducted on Niobium samples (morphology, surface composition).

1 CAVITIES PERFORMANCES

General results on cavities performances can be shortly summarized by the following issues :

- Cavities treated with standard BCP, (hereafter called "FNP", for hydroFluoric, Nitric, ortho-Phosphoric acids), commonly reach ± 25 -30Mv/m, but starting about 20-25 MV/m they exhibit a decrease in the Q factor. This degradation can be attributed to a general heating of the cavity of 5 to 10 mK [1]. (NB This effect can mainly be observed on heat treated or high RRR cavities since their earlier performances were not high enough to allow this slope to be observed.)
- Electropolished cavities also exhibit a slope in the $Q = f(E_{acc})$ curves, even on non heat treated cavities [2].
- Baking leads to significant improvement of the Q slope [3], whatever the former treatment, but the effect is dramatic on electropolished cavities. Even non heat treated cavities can reach accelerating gradients as high as 35-40 MV/m [2].
- We could observe that other surface treatments, like "FNS" (a chemical polishing with different reactants, described in §2), could modify the Q-slope .
- This surface effect is reversible : for instance improved electropolished cavities were again degraded by further FNP and vice versa ;and there

is a kind of "memory effect" : a minimum of 100 μ m seems to be necessary to reach a full result.

- It is not clear yet in what extent those surface treatments also influence quench field, but some differences where observed.

Looking for alternative chemistry appears to be a way to experiment different surface state on cavities. Many of them have been tested on samples as described in §2, but a few could be applied to cavities for practical reasons. That's why we also are conducting a complete surface study on sample in order to determine which parameters are modified by the different treatments, and which ones are the most preponderant. Preliminary results are detailed in §3. Three main factors can be explored at first sight :

- Influence of surface morphology (roughness at high and low scale).
- Surface chemical composition.
- Nb lattice strain induced by superficial oxide layer. This last point somehow ensues from the previous one as chemical composition of the surface can influence the structure of the oxide layer. We will see in the discussion of § 4 that this hypothesis is fairly probable.

2 ALTERNATIVE CHEMICAL TREATMENTS

Literature points out many possible "recipes" for etching niobium, see for example [4] or [5]. In all these mixture we can find the same basics :

- A niobium complexant, i.e. a chemical species that reacts with ions Nb^{5+} and forms a compound soluble into water.
- An oxidant, which reacts with metallic niobium Nb^0 and turns it into the oxidized form Nb^{5+} , which in its turn will be solubilized.
- Additional compounds like buffer or brightening agents.

Note that in the case of electropolishing, there is no need of an oxidant as a high potential (=oxidant) is already

imposed to niobium by the mean of the electrodes and the solution.

The drawback of usual BCP, is that it etches rather than polishes niobium surfaces, and after heavy etching, it tends to etch preferentially at grain boundaries, leaving some crevices which are difficult to rinse correctly and which enhance the surface roughness. Moreover, ortho-phosphoric acid is known to incorporate into the oxide layer in the form of PO_x (x~2) ions [6], and is regularly found at the metal-oxide interface by chemical analysis [7].

Even before testing it on cavities, developing alternative chemical polishing among all the possibilities was somehow difficult : what should we privilege ? Roughness, brilliance, etching speed, or safety ?

One of us (A.A.) has made a systematic chemical study of FNS (hydroFluoric, Nitric and Sulfuric acids in various proportions) for several reasons :

- It gives rise to very shiny surfaces,
- There is not grain boundaries preferential etching
- It does not contain H₃PO₄.

But it has several drawbacks, among them : it is difficult to prepare and to handle.

Nevertheless, this surface treatment is now under systematic study on cavities in order to understand which differences it brings to cavities performances.

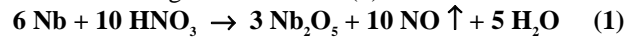
On behalf of a bibliographic study [4, 5, 8, 9, 10,...] alternative chemical baths like HF-H₂O₂, or alkaline media were also tested, but with less success. They will be also briefly described.

2.1 FNS

These ternary hydrofluoric (F), nitric (N) and sulfuric (S) acidic baths have already been studied by Uzel & al. [11], first on niobium test sheets then on resonators. They used in particular a chemical polishing mixture made of HF (40%) - HNO₃ (65%) - H₂SO₄ (96-98%) in a volume ratio 1:1:2, with a 78 °C bath temperature. Good results were obtained with regard to the classical FNP-1-1-2 mixture, but major disadvantages were raised by operating under that high temperature : loss of HF by distillation, increased degradation of HNO₃ in NO, NO₂ and N₂O₄ species (causing orange fumes), short immersing times (~ 30 s) because the dissolution rate of Nb is very high : 70 µm.mn⁻¹. So as to avoid these problems, we have tried to stay at room temperature.

2.1.1- Chemical reactions between the three acids and Niobium in aqueous media:

Nitric acid is an oxidizing agent towards Nb a protons donor according to the reaction (1):



When that reactant is not introduced (i.e. FNS 1: 0: 1) or fully consumed (e. g. FNS 1: 1/2 :9 after 300 min on Fig.1), the attack reaction respectively does not start or stops since H₂SO₄ and HF are not oxidizing agents towards niobium.

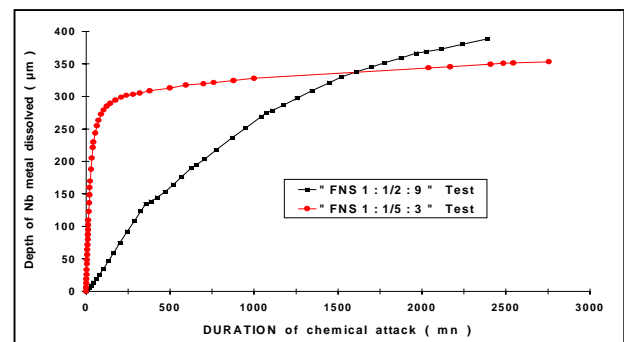
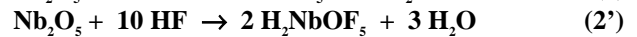
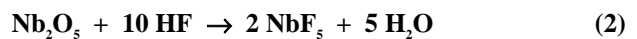
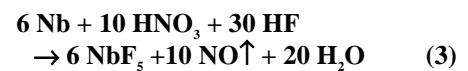


Figure 1 : Dissolution Kinetics of Nb by " FNS 1: 1/2: 9 and 1: 1/5: 3 " chemical Baths stirred at 400 trs.min⁻¹. Note that after a while, the reaction rate slows down, due to the total consummation of nitric acid.

In the presence of HF, the niobium pentoxyde Nb₂O₅ layer is rapidly transformed into soluble niobium pentafluoride NbF₅ or pentafluoxyniobic acid H₂NbOF₅ (hydrated form of NbF₅). This acid also acts as a protons in accordance with:



The resulting effects of these two acids can be so resumed by equation (3) given hereafter :



Sulfuric acid and other sulfuric compounds are often used like a brightening agent, although its exact action mechanism is not well known, and sulfur is likely to incorporate inside the oxide layer [12, 13]. Sulfuric acid is highly hygroscopic when concentrated (H₂SO₄ ≥ 10.5 mol.L⁻¹), and can reduce the dissociation of HF and HNO₃ into the ternary bath, as suggests the falls of Nb removing rates with increasing H₂SO₄ concentration. When this reactant is in a large excess towards both HF and HNO₃, the complexing of Nb^v by the fluoride anions F⁻ decreases on behalf of sulfate anions SO₄²⁻ and the synthesis of

niobium pentavalent oxysulfates [9] are favored in accordance with the general equation given below, where $n = 1, 2$ or 4 :



The effect of increasing concentration of each acids, when the two other acids are kept at a constant ratio is shown in figure 3.

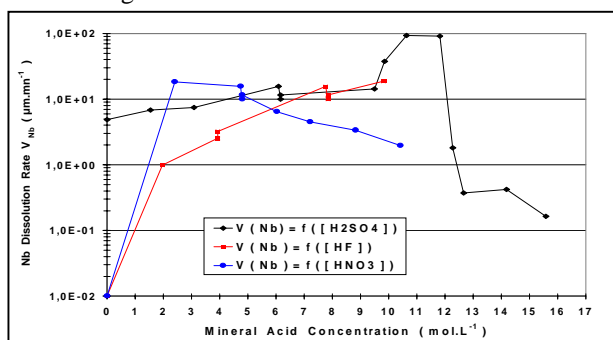


Figure 2 : Evolution of Nb Dissolution Rate versus Acid Concentrations. Note that etching rate goes through a maximum with increasing HNO_3 and H_2SO_4 concentration.

Except for very high etching rates ($10.6 \text{ mol.L}^{-1} \leq [\text{H}_2\text{SO}_4] \leq 11.8 \text{ mol.L}^{-1}$), the obtained surface states were equivalent, as long as all the reactants are present in solution. It is therefore possible to master the etching rate in order to get more controlled chemical polishing.

The important differences observed on the surface states during the experiments done with FNS baths (brilliant and smooth samples to visual inspection, see also figure 9) suggest that the species created with H_3PO_4 from FNP 112 into the interfacial film are more penalizing than those generated from H_2SO_4 .

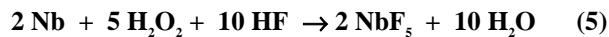
More details about FNS based chemical polishing will be detailed in reference [14]

2.1.2 Other chemical polishing

Other baths were also developed for in order to change the chemical environment where the oxide layer starts to grow. Indeed as anions presents in the polishing bath are likely to incorporate in the oxide layer, and thus to modify its crystalline structure, change in the chemical media could give indication on its effect on the cavities behavior. For instance, H_2O_2 is known to be oxidant and a niobium complexant. We are then starting to develop chemical bath where it replaces HNO_3 .

The reaction mechanisms which occur in these kinds of baths are expressed by the equation (5) where hydrogen

peroxide is the oxidizing agent which leads Nb to its oxidation state +5 and hydrofluoric acid is the complexing one which produces niobium pentafluoride :



Various mixtures of HF ($5\text{-}15 \text{ mol.L}^{-1}$) and H_2O_2 ($2.5\text{-}5 \text{ mol.L}^{-1}$) were applied on Nb test samples ; but in general, the surface was always degraded : severe grain-boundaries etchings (Nb grains taken off), strong roughness (mean peak-to-valleys $> 50 \mu\text{m}$) observed by visual and microscope inspections.

HF and HNO_3 1-9 in proportion was also tested (FN 1-9, etching rate $\sim 1.2 \mu\text{M}/\text{min}$) : it leads to mid rough surfaces, without apparent grain boundary etching, but did not produce any change when it was applied to a cavity (quench field $\sim 21\text{MV}/\text{m}$).

Some other media are now under study and will be detailed in [16]

2.1.3 “memory effect”

Apparently, more than $100 \mu\text{m}$ need to be etched before getting rid of the previous treatment effects when we change from one treatment to another. Is that related to grain boundaries pollution, or more complicated phenomena ?

Evidence of remaining of the oxide built during previous chemical polishing has already been observed on niobium, even after noticeable dissolution of the metal.[12, p. 217]. By the way, this effect seems to be general among all metals [15]. Indeed, in certain potential conditions, depending on the electrolytes presents in solution, and/or on an applied external potential (electropolishing), the dissolution of metal in the solution is a field assisted migration of the metallic cations through its oxide layer. The oxide layer itself becomes an ionic conductor due to the presence of a depassivating agent (F^- for instance), it allows the metal to diffuse toward the solution, but is dissolved in its turn much more slowly, as same as for the impurities that might be incorporated in it. So even after a noticeable dissolution of the metal, the oxide layer present at the beginning of the chemical attack is still there in some extent ! Some indications already exist in literature to overcome this effect, but of course, we need some additional experimental work on this problem.

3 MORPHOLOGY

More details about the experimental techniques can be found elsewhere in this workshop proceedings [16], but we can shortly summarize it by the following points :

3.1 Available experimental techniques and samples preparation.

It is important to well characterize the surface morphology at different scale : at the micron scale , roughness is not expected to play any role in superconductivity, but might play a role by perturbing the electromagnetic field repartition or by enhancing effective surface. At lower scale (~10-100 nm) surfaces are more difficult to observe ; but it is worthwhile to explore if there are important differences between the different treatments. Moreover it also plays a role in the case of Kapitza resistance where the mechanism of heat transfer is partly due to phonons with ~100 nm wavelength [17].

Arithmetic (R_a) or quadratic (R_q or σ_{ms}) roughness can easily be measured by profilometry, but one has to keep in mind that this parameter does not characterize well a surface : first, different types of morphology can give the same mean roughness , second profilometry is generally not able to measure accurately small scale roughness. In theory, Scanning Tunneling Microscopy (STM) can reach atomic resolution and appears to be an idealistic tool, but due to practical reasons (limitation on the range of heights for the tip) we need fairly plane surfaces.

A comparative study of three surface treatments was done on niobium RRR~135 monocrystals. All the sample were mechanically polished (mirror like), prior the chemical treatments. In this way it was hoped to be less influenced by roughness due to grain boundaries etching or machining, and to be able to explore low scale roughness. The surface treatments were the following :

- Electropolishing (EP) : we have use the process developed Wuppertal and now used at KEK, namely in a $\text{HF-H}_2\text{SO}_4$ 15%-85% in volume, with 8-10 Volts applied on the sample (as an anode). 40-50 μm removed.
- Chemical polishing FNP : standard BCP with HF, HNO_3 and H_3PO_4 1-1-2 in volume. ~100 μm removed.
- Chemical polishing FNS : CP with HF, HNO_3 , H_2SO_4 1-1-1 in volume. ~100 μm removed.

3.2 Profilometry

Profilometry is a mechanical measurement : a diamond tip with a final radius of ~5 nm skims over the sample surface and corresponding displacements are registered.. It is important to note that explored length play an important role on the results (because of the superimposition of different roughness scale), and it should be mentioned for each comparison.

Figure 2 give examples of the registered profilometry at two different scales. A minimum of 10 measures were taken at random on each sample and the mean roughness was calculated.

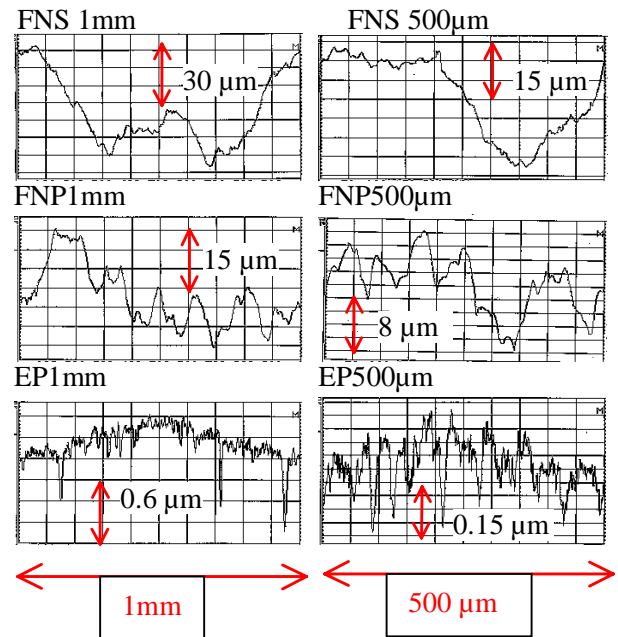


Figure 3 : Examples of profilometry of Nb monocrystal surfaces treated with Chemical polishing with hydrofluoric, Nitric and Sulfuric acids (FNS), Chemical polishing with hydrofluoric, Nitric and orthophosphoric acids (FNP), and Electropolishing (EP).

The obtained results are summarized in figure 5 along with the results of the other techniques. Note that the scattering in the measures results from the non uniformity of the niobium surfaces, and is not due to measure uncertainty.

Differences in mean peak-to-valley distances as well as peak frequencies is obvious on these monocrystalline samples. The same tendency can be observed on polycrystalline samples (not shown here), with measured mean roughness slightly higher, due to the apparition of steps or crevices at grain boundaries.

3.3 Scanning Tunneling Microscope

Several images were taken at random, at three different scale. Only a very small portion of the surface could be observed for practical reason (acquisition time), but we have tried to show the most characteristic surface aspects. The images were then statistically treated in order to get the height distribution. The average value could be compared to R_a and the mid height width was assimilated to the dispersion.

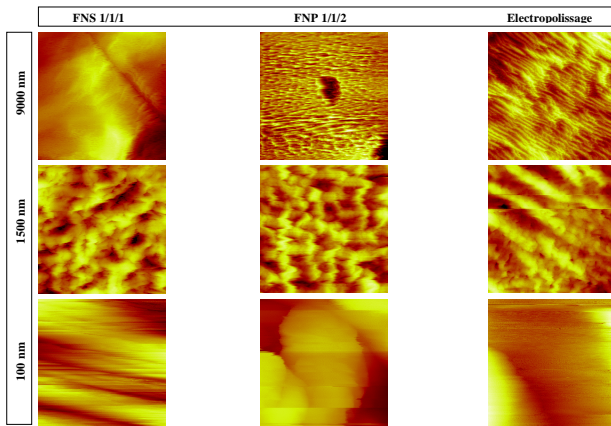


Figure 4 : Examples of Scanning Tunneling Microscopy vues of Nb monocrystal surfaces treated with Chemical polishing with hydroFluoric, Nitric and Sulfuric acids (FNS), Chemical polishing with hydroFluoric, Nitric and orthoPhosphoric acids (FNP), and Electropolishing (EP). At higher observation scale (9 μm sided) the differently treated samples present marked difference in aspect. The differences appear to be less marked when one observe the samples at lower scale (1.5 μm and 0.1 μm sided). At this last scale, the roughness is still so high compared to the tunneling microscope dynamic, that it was impossible to reach the atomic resolution. Moreover, as we get on the picture only a small portion of the surface, and then a small portion of, let's say a "valley" or a "peak", the results are obviously out of range. At least no visible substructure appear at this scale. (For a more detailed discussion on these results see [16].

3.4 Comparison of Ra at different scale

As pointed out before, the roughness measurements are strongly affected by the observation scale : at lower scale, we are less sensitive to regular micron-sized defects. We can conclude from the figure 5 that the three surface treatment that we have tested have a strong effect at high scale, but at lower scale the surfaces states are very alike. We can model the three surface states like on the scheme figure 6.

In summary, Electropolishing smooths the surface at a micron scale, but does not affect the microroughness of the niobium. Other chemical polishing give rise to more or less pronounced roughness at the micron scale, with a same substructure.

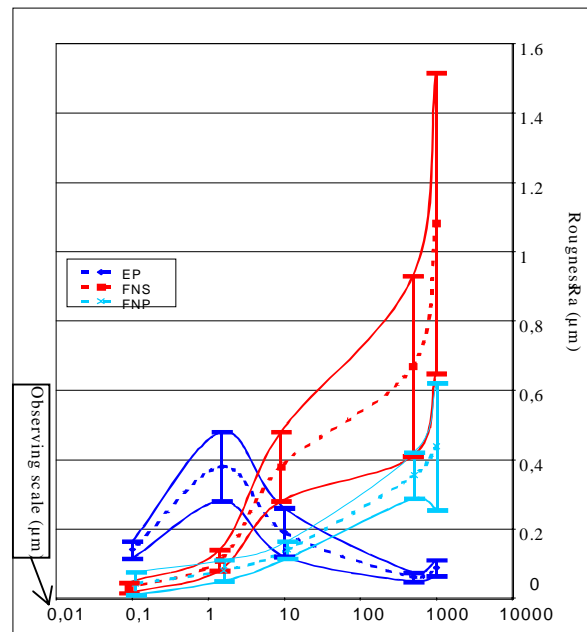


Figure 5 : Comparison of Ra at different scale of Nb monocrystal surfaces treated with chemical polishing with hydroFluoric, Nitric and Sulfuric acids (FNS), chemical polishing with hydroFluoric, Nitric and orthoPhosphoric acids (FNP), and Electropolishing (EP) : the roughness measurements are strongly affected by the observation scale, and discrepancy of the results ensues from non uniformity of the Nb surface itself.

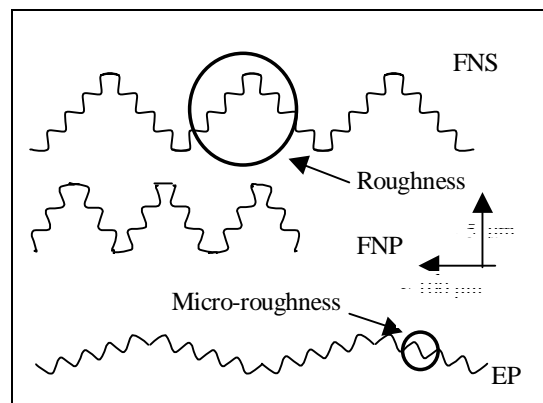


Figure 6 : Schematic comparison of Ra for Nb surfaces treated FNS, FNP, and EP.

. One must be aware that in the whole, surfaces are fairly smooth, with "peaks" not higher than a few microns for some hundreds of microns large. This type of roughness nearly does not affect the effective surface.

3.5 Microscopy, effect of purification annealing on the morphology

Optical microscopy is a convenient approach to evaluate surface morphology. But one can notice very important differences between monocrystals and polycrystals, and among polycrystals, between normal samples and annealed ones, which undergo heavy recrystallization.

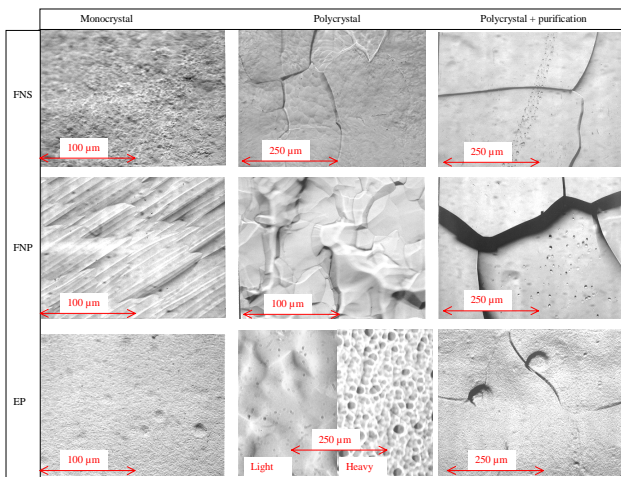


Figure 7 : Comparison of visual aspect of Nb surfaces treated FNS, FNP, and EP, for monocrystals, polycrystals, and heat treated polycrystals (purification annealing as in [1]).

Note that recrystallisation does not only modify the visual aspect of the samples, it also change the roughness and the microroughness (see figure 8). But on the contrary, purification annealing even if it enhances the Q_0 factor and improves the quench limit of cavities, apparently does not modify the Q slope.

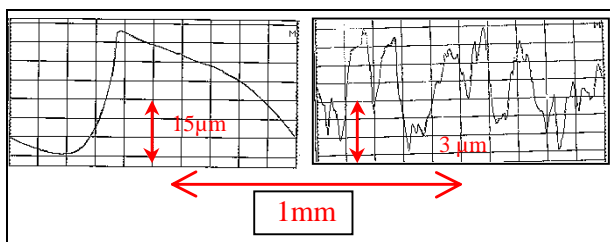


Figure 8 : Roughness measurement of a Nb sample, after and before the purification heat treatment.

As most of the cavities are nowadays annealed, it is obvious that the experiments described at §3.2 and 3.3 should be completed with work on annealed samples.

4 SURFACE CHEMICAL ANALYSIS

4.1 Available experimental techniques and samples preparation.

It is quite difficult to find surface analysis techniques sensitive enough to explore the very surface of niobium, without being perturbed or screened by the surface contamination and the oxide layer. Two techniques were retained : Time-Of-Flight SIMS and ESCA ; they are shortly described hereafter.

TOF-SIMS is a profiling method. A primary ion beam etches the samples, with a controlled etching rate, but during analysis static conditions (very low beam intensity) are applied. In this way only ONE monolayer is analyzed at the time. This very good depth resolution allows to observe the metal-oxide interface. As it is a very sensitive method (in certain conditions ppm and even ppb can be reached), one can observe also the impurities at the interface or incorporated inside the oxide layer.

These analysis were conducted on samples prepared in a way as close as possible as for cavities : samples were RRR 200 niobium which underwent a further purification annealing, then surface treatment (EP 40-50 μm, or 100 μm FNS or FNP according to the case), then HPR, and then analyses after a determined period in the air.

We have been trying , in a first step, discover whatever differences there was in surface chemical composition between FNP, FNS and EP, and also the influence of air exposure.

ESCA : samples are submitted to a monoenergetic X-rays beam. Electrons from the core orbitals of the atoms are ejected by photoelectric effect and their kinetic energy is correlated to their binding energy, which is characteristic of one orbital of a element. Moreover, if this element is linked to a more or less electronegative specie, its binding energy will be slightly displaced, allowing to know the chemical environment of the considered atom. In our case, it is very easy to distinguish metallic niobium (Nb^0) from its oxide(s), we can also get some information about the presence of an hydroxide and their relative thickness. Indeed the photoelectrons don't travel much across the material, and this technique explores only 30 to 80 nm on the surface, depending on the mean free path of the electrons inside the material and the detection angle. Unfortunately it is not a very sensitive method and one cannot analyze traces ($\sim < 0.1\%$). Meanwhile, by varying the detection angle it is possible to determine approximately the oxide layer thickness [18, 19] with a relative error.

The two following paragraphs describe shortly our results. Once again, the reader is invited to consult ref.[16] for discussion of the experimental conditions and complementary results.

4.1 TOF-SIMS results

A first series of experiment showed us that high pressure rinsing and air exposure could modify the surface ; and there was clear indications that different surface treatments could induce change in the oxide thickness. Moreover, we could confirm the incorporation of several solution anions inside the material (for instance Pox in the case of FNP, S in the case of electropolishing) ; Fluor was found in any case, but its concentration is greatly reduced after HPR.

For the second set of sample, we put apart one electropolished sample in dry ultrapure Ar, less than one hour after the HPR. It was introduced in the experimental area the day after, being exposed to the air less than 5 min.

Even after high pressure rinsing, the oxide layer on electropolished sample is extremely thin as can be observed on figure 7, even though former results (measured on air exposed samples) had given indication of the contrary.

After 48 hours in air, the oxide thickness is comparable to the one obtained on FNP samples. This behavior is to be compared with results obtained at KEK on air exposed cavities [2]. There is a strong indication that the structure of the surface oxide plays a major role in the high field cavities behavior.

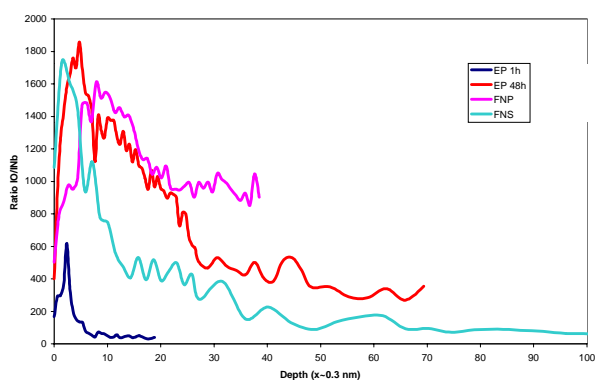


Figure 7 : TOF-SIMS profiling Nb samples treated with various treatment, showing the variation of the oxygen/niobium signal.

Impurities like P (in the form of PO and/or PO₂) were found in the oxide layer of FNP treated samples while Sulfur is also observable in electropolished and FNS treated samples. This incorporation of anions from

electrolyte is well known, especially for phosphates and sulfates [12] and has already been evidenced for niobium [6]. Incorporation of impurities can modify the behavior of the oxide layer (structure , growth rate, internal strain, ...) which in turn can influence the superconducting properties of the metallic lattice in the vicinity of the surface.

4.2 ESCA results

As ESCA can give us some information about the chemical neighborhood of a considered element, it was worthwhile to attempt to determine chemical differences between EP and the classical FNP.

Influence of high pressure rinsing and stay in the air on a classical FNP treated sample.

As can be observed on figure 9, even after a thorough normal rinsing, the oxide layer has not reached its maximum thickness : indeed the Nb⁰ doublet signal (right side of the figure) is clearly apparent, while the oxide signal (on the left is more feeble). HPR allows the oxide layer to grow further, It goes on growing within the 4 next hours of exposition to the air (increase of the oxide doublet peaks while the Nb₀ peaks decrease). But further exposition to the air does not bring more changes (even after one week, not shown here). Meanwhile, on the grazing angle spectrum for oxygen (not shown here) one can observe that the part of the oxygen signal attributed to the hydroxide form increases after a long stay in the air.

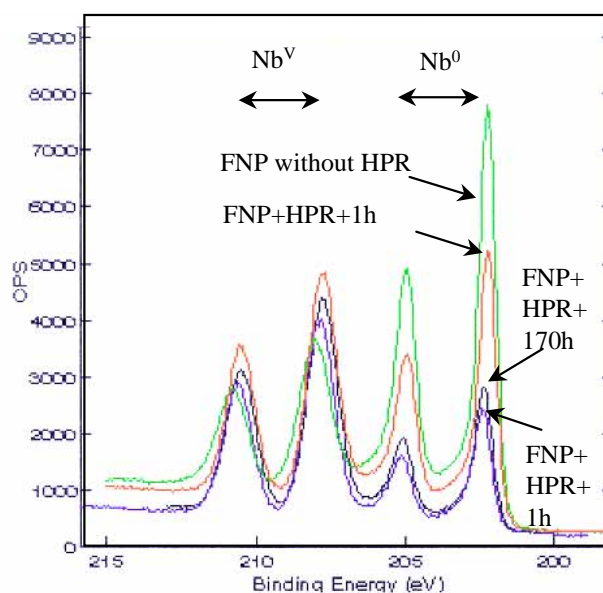


Figure 9 : Influence of high pressure rinsing and stay in the air on a classical FNP.

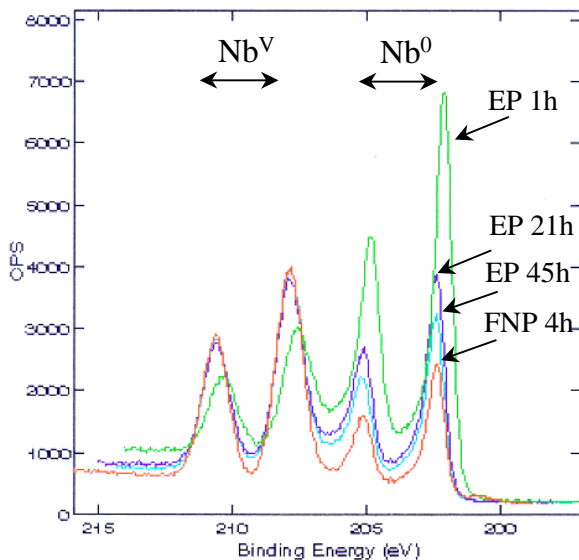


Figure 10 : Influence of air exposure on EP treated samples for the Nb signal.

Influence of air exposure on EP treated samples. One can observe that the Nb^0 signal on EP samples is still very intense after only one hour in the air, although they were HP rinsed, i. e. that the oxide layer is still very thin. It increases after air exposure, rapidly during the first 21 hours, then slower. But even after 45 h in the air, the oxide layer is still thinner compared to FNP treated samples. Once again these results are very consistent with the behavior observed on cavities at KEK. [2]

One can summarize this by saying that the oxide grown after FNP is thicker and more stable than the one grown after EP. The oxide formed on EP treated samples is thinner and more sensitive to air.

After ~ one week in the air, the two samples are very alike from the XPS point of view.

Table 1 shows the estimated thickness of niobium oxide, calculated like in reference [19], by meaning the results obtained with two different angle of detection.

Table 1

Surf. Treatment/ Air exposure	Nb_2O_5 thickness nm
FNP 4h	5 ± 2
EP 1h	$2,5 \pm 0,1$
EP 21h	$3,9 \pm 0,8$
EP 45h	$4,6 \pm 0,8$
EP 220 h	$5,0 \pm 1,5$

FNP + 48h 110°C, 10^{-9} bar	
Nb^V	$4,0 \pm 1$
Nb^{IV} (?)	2.84 ± 0.08
FNP + 170 h in air	$5,0 \pm 1,5$

ESCA is a very attractive way to study niobium surface oxides. A more thorough study should now complete these first results, in particular to precise the role of the rinsing, the sensitivity to air, the influence of purification annealing (it seems that the oxide grows a little slower on purified samples), etc..

5 DISCUSSION AND CONCLUSION.

There are (and there will be) a lot of discussion before to accept a definitive explanation about the effect of the different surface treatments applied to cavities. The results presented in this paper, although they still need to be completed, give us some indications about what we are looking for. Roughness seems a second order factor as the recrystallization brings a lot of change to the niobium surface while it does not change the Q-slope (even if the quench field is improved). Cavities with very different surface aspect due to their previous “history”, exhibit the same type of Q variation, providing indication that the overall surface roughness does not play a noticeable role. The oxide thickness and its sensitivity to air exposure was the only noticeable difference that could be observed on samples. We don’t know yet if it is the oxide thickness itself that play a role or only the fact that it traduces impurities presence in and near the oxide layer. The presence of impurities inside the first layers of the metal itself has been suggested several times but need further exploration. Another explanation can be proposed, that issues from metallurgy and corrosion science : anion incorporation in oxide layers is a well known phenomena and is known to influence oxide structures. Different structures exhibit different internal strain. Strain in thin films is also known to influence their substrate (for us the metal lattice). It could then be very interesting to try to quantify this strain in function of the different surface treatment, and to estimate its range of action. A more complete study of the influence of air exposure is also highly necessary, because it would be very interesting if we could determine a convenient surface treatment that leaves the niobium surface insensitive to air. Dry oxidation at high temperature gives us some nice indications, but is hardly applicable to industrial production of cavities..

Another aspect should also be explored : the apparent “memory” effect. Indeed some evidences can be found in literature that, depending on the electrochemical media, impurities are likely to dissolve much more slowly than the metal, because of ionic field assisted diffusion mechanism of the latter. As cavities are somehow a very sensitive tool to inform us about surface perfection, it is

evident that the “real” surfaces need far more further exploration before we could be able to master their RF properties.

6 REFERENCES

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