## Q degradation of Niobium cavities due to Hydrogen contamination

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#### Abstract

This paper presents a review on the Hydrogen contamination of Niobium by various surface treatments, and on the subsequent degradation of the properties of superconducting Niobium cavities.

#### Introduction

In the past two years, several laboratories <sup>1-9</sup> around the world have observed degradations of the Q value of superconducting Niobium cavities under certain cooldown conditions. A remedy has to be found soon, since this phenomenon affects cavities for various accelerators presently under construction. The purpose of this paper is threefold: 1) make a synthesis of the symptoms associated with this "Q disease"; 2) discuss the mechanisms which are responsible for the effect; 3) propose cures to get rid of this anecdotic, but troublesome phenomenon.

#### Cavity preparation

Q degradation has been observed on Niobium superconducting RF cavities in several laboratories, using different ways to prepare their cavities: Hydroforming (Saclay), deep drawing (Cornell, CEBAF, Wuppertal...), spinning (DESY, CERN...) and electron beam welding of high purity Niobium sheet half cells (thickness of a few millimeters, RRR 200 to 300). Most laboratories have in common a chemical polishing based on a mixture of HF,  $HNO_3$  and  $H_3PO_4$  (hereafter called "Buffered Chemical Polishing", BCP) in various proportions, followed by a rinsing in ultrapure water. Cavities from KEK are electropolished (ECP), rinsed and baked in a UHV furnace.

#### Influence of thermal cycling

Cavities that experience fast cooldown (i.e  $\geq 1 \text{K/min}$ ) exhibit commonly a residual surface resistance between 10 and 40 n $\Omega$ . It has been established  $^{1-6,10,12}$  that some large additional resistance appears reproducibly whenever a BCP or ECP treated cavity stays long enough at temperatures around 120 K. The order of magnitude of the degradation is quite variable, and can reach values as large as  $Q_{nondegr}^{res}/Q_{degr}^{res}=2000$ . In most laboratories (Cornell <sup>3</sup>, CEBAF <sup>4</sup>, Saclay <sup>2</sup>, Wuppertal <sup>12</sup>), the degradation is reversible, and disappears totally with a new fast cooldown after warmup at 300K. The sensitive temperature region is comprised <sup>4,5,10</sup> between 77 K and 170 K. The degradation depends on the time spent by the cavity in the dangerous temperature zone. A nice example was measured at Saclay <sup>2</sup> (fig. 1). For this 1.5 GHz cavity, a significant degradation was observed already after a 1 h stay at 120 K. Additional and longer cryocycles showed stronger degradation. Saturation was achieved in the third cycle, keeping the cavity at 150 K for 3 hours. At DESY <sup>6</sup> and Darmstadt <sup>25</sup>, the degradation is irreversible, ie the first cooldown gives a better Q value than the subsequent ones (figs. 2, 3).

At Saclay and Wuppertal, temperature maps indicated that the losses are distributed more or less uniformly over the whole cavity surface. (fig. 4).

#### Influence of RRR

Only high RRR cavities are affected by the disease: at Darmstadt <sup>25</sup>, 11 multicell cavities of different RRR underwent the same cryocycle (2K - 130 K - 2 K). Only some high purity structures displayed the degradation. A cavity with RRR 40 was also measured at CEBAF <sup>10</sup>, and showed no degradation.

#### Influence of baking

Quite generally, a baking  $^{5,6}$  of the cavities in a UHV furnace (T  $\geq 500^{\circ}$  C, 2 hours) cures the degradation. (fig. 5). A further BCP treatment brings again an attenuated degradation, or even no degradation at all in some cases (figs. 6.7).

#### Frequency dependence of the degradation

The general impression gathered from most laboratories is that the Q degradation is strongly frequency dependent: The "low  $\beta$ " resonators, which usually work at frequencies lower than 300 MHz, have no symptoms of Q disease; CERN cavities (350 MHz) do not display any observable degradation; at Cornell, S band cavities are affected, whereas L band cavities are not. At Saclay, systematic measurements have been made on accelerating cavities at 1.5 GHz and on two modes of a pillbox Niobium cavity at f = 4 and 5.7 GHz. It can be seen from Table 1 that the surface resistance brought about by the degradation scales roughly like  $f^2$  (with two different proportionality factors at low and high field, however). This  $f^2$  dependence is also weakly supported by data from a multimode cavity at Wuppertal  $^{26}$  (fig. 8).

#### Superconducting aspects

It can be seen on Fig. 9 that the degradation cannot be interpreted as a mere residual resistance added to the BCS resistance of Niobium. Indeed, the slope of  $R_s(1/T)$  is different for degraded and non degraded cavities, showing that the disease affects the BCS properties of the material.

In the case of low bath temperature, one always notices a particular characteristic of the Q behaviour: starting at low field from an already low quality factor, Q steps down to a still lower level for a surface magnetic field of about 6 mT at 1.6 K and 1.5 GHz, and remains then approximately constant (fig. 10). At higher bath temperature, eg 4.2 K, the steplike behaviour is no longer observed, and the Q degradation is more or less independent of the accelerating field (fig. 11).

A steplike behaviour of the function  $R_s(1/T)$  (at low field) has also been observed at Saclay (fig. 9). Many other laboratories  $^{10,26}$  confirm the existence of irregularities in the  $R_s(1/T)$  curve of degraded cavities. All these observations might be interpreted as a superconducting transition occurring either in a thin layer or in islands of an unknown phase located near the surface (figs 11, 12).

The Q of degraded cavities have been measured at Saclay at T=10 K, with no significant difference with the value obtained for non degraded cavities, indicating that the surface RRR of the material (averaged on the normal state penetration depth, ie about  $1 \mu m$ ) is not appreciably modified by the underlying phenomenon.

#### Role of Hydrogen

In view of these symptoms, it seems quite plausible that Hydrogen contamination is the cause of the degradation. No other species is expected to cause structural changes in Niobium at temperatures as low as 77 K; moreover, no other species escapes from the Niobium lattice during a baking at 500°C. In addition, it has long been recognized <sup>9</sup> that electropolished cavities are polluted by

Hydrogen, and that this contamination brings forth a Q degradation, readily cured by a baking of the cavities. The connexion between these facts and the present problem has been made only recently.

However, when can the Hydrogen enter in the case of chemically polished (BCP) cavities? The chemical treatment of Niobium is probably guilty, and much effort has been devoted to study its influence, on cavities and on samples. The following facts have been reported:

#### Sources of Hydrogen pickup

#### Role of the chemical treatment

The chemical treatment of cavities can introduce Hydrogen in the material. Bulk measurements made on samples at Heraeus, DESY <sup>6,14,17</sup>, and at Saclay <sup>13</sup> show that the bulk contamination (ie averaged over the sheet thickness) is small, of the order of 100 to 400 at. ppm, but depends significantly on the nature of the treatment. (fig. 13). This amount is too small to influence the bulk RRR of the material in a measurable way.

The chemical treatment of cavities does cause Q degradation. A 1-1-2 BCP removing 50  $\mu m$  caused a Q degradation after a long warmup at 100 K, on a 1.5 GHz accelerating cavity previously depolluted by a 1500 degree baking in an UHV furnace. The degradation, however, was rather weak (a factor 3, see fig. 7), thus suggesting that the 1-1-2 BCP may not be the only, or even the main cause of Hydrogen contamination.

Experiments done at Cornell <sup>3</sup> on samples also show that a BCP treatment does not necessarily contaminate much, and even, in some cases, decontaminates the Nb sheet. All this suggests that part of the contamination may occur in earlier steps of the process.

#### Influence of the bath composition

The composition of the bath evidently plays some role with respect to Hydrogen contamination. To suppress the pickup of Hydrogen during etching and to cure contaminated cavities that cannot be baked, different acid bath compositions have actively been researched. The replacement of  $H_3PO_4$  by lactic acid yielded promising results (fig. 13), but proved  $^{3,14,17}$  to be inapplicable for safety reasons. We know from cavity measurements at Saclay that 1-1 CP (ie without  $H_3PO_4$ ) does not pollute Niobium, previously purified by baking  $(750^{\circ}C, 2h, 10^{-6} \text{ Torr})$ , even if 130  $\mu m$  are removed. However, this type of mixture has too high an etching rate, and tends to give rise to dangerous thermal runaways. Besides, further tests revealed that the 1-1 CP does not cure a previously polluted cavity, even though it does not bring by itself additional contamination.

Attention then focussed on a more controllable mixture, namely 1-9 CP, which

has a reasonable etching rate and exothermicity, and which is supposed to incorporate little Hydrogen, due to its high concentration in oxidizing acid  $HNO_3$  (fig. 16). Here again, the results were negative (fig. 16): this treatment does not cure a previously polluted cavity, so that this mixture cannot be a useful remedy.

The Water chemical polishing (WCP, i.e HF,  $HNO_3$ ,  $H_2O$ ) was first tried at DESY <sup>6</sup>, with encouraging results. It was proved that this type of mixture (1-1-5) could reduce the bulk Hydrogen content to 100 at. ppm (fig. 17) if large acid volumes are used, and that the amplitude of Q degradation was also reduced. Unfortunately, this reduction was not confirmed at Saclay (fig. 16), possibly because the acid volume was too small in this case.

#### Electrochemical aspects

Clearly, more experiments are needed to find an improved chemical treatment. We propose the following guideline for future investigations: It is known  $^{18}$  that the usual BCP contains  $HNO_3$  to oxidize the Niobium, and a complexant acid, HF, which dissolves the Niobium oxide as soon as it is formed. The role of the third acid  $(H_3PO_4)$  is to slow the extremely exothermic reaction down to an acceptable rate. We assume that HF and  $H_3PO_4$  are the acids which favor incorporation of Hydrogen in Niobium, whereas  $HNO_3$ , due to its oxidizing nature, plays an antagonist role. Similarly, polarization of Nb in the bath has a polluting influence  $^{9,29}$  if it is cathodic (it is the case of electropolishing), and protective if it is anodic (it is the case of anodisation). Such a polarization may occur, even in the absence of applied voltage, if other metals are present in the bath (this is true also for a usual BCP treatment). An electrolytical cell can then be formed with the Niobium to be treated, with hardly predictable results. The influence of foreign metals on Niobium contamination by Hydrogen has never been investigated so far, but we think that it deserves some attention.

#### Are HCl and HF harmful?

The Hydrogen concentration measurements made at Saclay by ERDA (Elastic Recoil Detection Analysis, Nuclear probe) show that the HCl treatment used on the raw Nb sheets to detect surface defect incorporates large amounts of Hydrogen in the material (up to 100000 at. ppm in surface, and of the order of 1000 at. ppm in the bulk). The Wuppertal laboratory apparently confirms the same result, since a previously titanified cavity, treated with a 1-1-1 BCP + HCl displayed a considerably degraded Q value, ie  $3.10^6$  at 4.2 K and 1.5 GHz. The bulk Hydrogen content of the sample following the cavity was 1300 at ppm, about 3-10 times larger than the usual bulk concentration for a typical BCP treatment. It may be conjectured from the literature  $^{18,19,24}$  available on the HF + Nb and HCl + Nb systems that their behaviour is roughly the same, since the chemical properties of

these two acids are very akin. Indeed, HF diluted in water is known to incorporate huge quantities of Hydrogen in Niobium powder, causing precipitation at room temperature <sup>15</sup>. It is also known <sup>16</sup> that corrosion appears on Nb sheet samples soaked in a diluted HF solution. Since HF is used in all laboratories to remove the thick oxide layer after anodization of the half cells, this step might be an important source of degradation, too. In order to test the harmful role of HCl and HF, a dedicated experiment series has been run at Saclay. Three 1.5 GHz accelerating cavities have been baked in an UHV furnace (750° C, 2 hours) in order to clean them from any previous Hydrogen contamination. After baking, cavity A underwent only a 5  $\mu m$  BCP, and displayed no Q degradation, even after a long warmup at 100K. On the other hand, cavities B was stored for 24 hours in HCl (2 percent) and cavity C had HCl (2 percent, 24 h) and HF (48 percent, 15 min.). A 5  $\mu m$  BCP finished the surface preparation in both cases. Disappointingly, neither cavity B nor cavity C exhibited Q degradation.

Influence of other process parameters.

#### Open vs closed treatments

It has been shown in Cornell <sup>3</sup> and DESY <sup>6</sup> that the ratio  $\Omega = Treated\ area/Acid\ volume$ , plays an important role on Hydrogen incorporation, the Hydrogen contamination being larger for large values of  $\Omega$  (figs. 13, 15).

#### Duration of the treatment

The duration t of the chemical polishing treatment seems to influence the Hydrogen contamination: Measurements at DESY <sup>6</sup> showed that  $[H]_{bulk}$  decreases with t in the case of a 1-1-5 WCP (fig. 17). The characteristic time of this decrease is about 15 minutes, ie the same order as the value  $\tau \simeq l^2/(\pi^2 D)$  that would be expected if all the Hydrogen atoms diffusing in the bulk Niobium leave the metal when they arrive at the Nb - bath interface. (I = sheet thickness, D = diffusion coefficient). No information is available for  $[H]_{bulk}(t)$  in the case of a BCP, but the H contamination seems to increase with t for this mixture.

#### Influence of the temperature

The temperature of the acid bath is also an important parameter (cf fig. 18). It has been shown that the Q degradation (Cornell  $^3$ ) and the Hydrogen content (DESY  $^6$ ) increase with  $T_{acid\ bath}$ . This observation is especially important, because measurements made with an infrared camera  $^{17}$  suggest that the surface temperature of the Nb sheet during a standard BCP treatment fluctuates strongly, and can reach locally values as high as  $120^0$  C, even when the main bath temperature is stabilized at  $25^0C$ . The heating may be even higher when the sample is removed from the bath, leaving only a thin acid film on the Niobium surface. Thus, an effective stirring of the acid bath and a fast rinsing after the etching seem

to be very important to minimize Hydrogen contamination.

Unfortunately, all these parameters (Bath composition,  $\Omega$ , t,  $T_{acid\ bath}$ ) are closely interconnected: for example, a modified bath composition results in a modified etching rate, and therefore changes t; in addition, the exothermicity of the reaction is also generally modified, thus changing  $T_{acid\ bath}$ . Consequently, it is very difficult to determine which parameters are really important for our Hydrogen contamination problem. We only know that a cool and open treatment should be preferred in order to minimize Q degradation.

#### Remedies

Among sure facts, we know that a short BCP (1-1-2,  $5 \mu m$ , room temperature) does not pollute a cavity previously purified by baking (750 deg, 2 hrs, vacuum of  $10^{-6}$  Torr). This recipe is now qualified, and is a sure remedy to the Q degradation phenomenon.

Anodization is also potentially promising, because polarization of the cavity to positive voltages may impede the entry of  $H^+$  in Niobium. Indeed, it was proved at CEBAF <sup>4</sup> and Cornell <sup>3</sup> that anodized cavities did exhibit a reduced (or even suppressed) Q degradation. We may have here a useful remedy, but it remains to be ascertained that a thick oxide layer has no other drawback for accelerating cavities (eg, sensitivity to radiation damage..).

#### Other sources of H contamination

From the experiments discussed above, it is proved that the chemical treatment does affect the Hydrogen concentration in Niobium significantly. However, we have seen that the chemical treatment might not be the only, or even the main source of Hydrogen contamination. It is known from the literature  $^{9,11}$  that the Nb lattice can incorporate large amounts of Hydrogen from its surroundings (eg the atmosphere...) if the oxide layer which acts as a barrier is destroyed. For instance, the partial pressure of Hydrogen in the atmospheric air is  $4.10^{-4}$  Torr, and according to Sievert's law  $[H]_{eq} \sim p_{H_2}^{1/2}$ , this corresponds to an equilibrium concentration  $[H]_{eq} = 5.10^5$  at. ppm at room temperature (fig. 14). Of course, this huge equilibrium concentration is never reached because of the protective role of the oxide layer. But one may wonder whether a crack in the film can let in significant amounts of Hydrogen. The stamping (or spinning or hydroforming) of half cells can crack momentarily the oxide layer, and may turn out to be a dangerous step for this reason.

Also, the electron beam welding of half cells is usually done in a poor vacuum, and brings the whole cavity to high temperatures, certainly high enough to remove the oxide barrier. The Hydrogen equilibrium concentration between the sheet and the residual vacuum can then be reached, corresponding to high H concentrations,

of the order of a few hundreds at. ppm if the vacuum is  $10^{-4}$  torr (fig. 14). These are only speculations, but we feel that they should be investigated further.

Hydrogen is concentrated near the surface!

Measurements of the Hydrogen concentration near the surface have also been performed in various laboratories. All methods (Nuclear probe, SIMS, Glow discharge spectroscopy,...) converge towards this surprising result: Hydrogen is concentrated near the surface  $^{13,14,19,20}$ . The concentration peak is at least a factor of 10 larger than  $[H]_{bulk}$  (fig. 19). The spatial resolution of the probes used does permit to say that the width of the concentration peak is about 40 nm, ie the same order of magnitude as the penetration depth in Niobium. This fact might explain why Hydrogen contamination is so effective in reducing the superconducting capabilities of Niobium. The measured surface concentrations are of the order 10000 - 50000 at. ppm, ie close to the precipitation threshold at room temperature. These values are averages, so that locally, the concentrations might be even higher. Indeed, an experiment done with a nuclear microprobe at Saclay  $^{20}$  on a polluted sample, with a transverse resolution of 30  $\mu m$  did show 30 percent [H] fluctuations on this scale.

Hydrogen is known to be very mobile in Niobium. Given the very high value of the diffusion coefficient of H in Nb, any concentration gradient should be equalized in very short times. How then can we reconcile this with the observation of a surface peak for [H]? Hydrogen in bulk Niobium is submitted to no net force, so that the usual Fick's law is obeyed:

$$j = -D.grad(c),$$

where D is the diffusion coefficient, j the Hydrogen current, and c the Hydrogen concentration. In the neighbourhood of a defect (impurity, dislocation, interface...), the H atom feels a force f = -grad(W), and the Fick's law must be modified and writes:

$$j = -D(grad(c) + c.grad(W)/kT).$$

The equilibrium solution of this equation generally displays concentration gradients in the vicinity of the defect. The surface is a major defect of the material, and this may explain the Hydrogen accumulation near the surface if the Hydrogen - surface interaction is attractive.

The same kind of argument also stresses the influence of impurities, constraints and metallurgical defects on Hydrogen trapping. Regarding impurities, it should be remembered that substitutional or interstitial impurities may retard the Hydrogen precipitation by stabilizing the  $\alpha$  phase <sup>27,28</sup>, a fact which might explain why low RRR cavities do not exhibit Q degradation <sup>10,25</sup>. We also know <sup>23</sup> that the surface of the Niobium is deeply serrated because of the volume difference between the oxide and the Niobium from which it originates. The constraints in the lattice due to this serration might be the cause of the observed Hydrogen accumulation near the surface. Moreover, dislocations are known <sup>22</sup> to attract Hydrogen (Cottrell clouds). This suggests that the different magnitude of the Q degradation observed in different laboratories might (also) be due to differences in the metallurgical preparation of the cavities.

#### Hydride islands or Hydride film?

Hydrogen is concentrated close to the surface: it is natural to think that it is this surface Hydrogen which plays the dominant role and causes the Q degradation, by forming a Hydride film after a long stay at 100 K. From the superconducting point of view, the system is then simply bulk Niobium coated with a normal or superconducting uniform layer of thickness e (fig. 20.a). Calculations done at Saclay <sup>2</sup> show that this model can account for the observed surface resistance, and even for the steplike behaviour of  $R_s(T, E_{acc})$  if one admits that the thin polluted layer is weakly superconducting, with  $T_c \simeq 2.8K$ ,  $H_c \simeq 60$  Gauss, and has a thickness  $e \simeq 28$  nm and a normal state conductivity  $\sigma \simeq 1.6 \cdot 10^8 \, \Omega^{-1} .m^{-1}$ . The frequency dependence of the degradation is predicted to be  $R_{deg} \sim f^2$ , in agreement with the experimental results. One drawback of this model lies in the fact that the Nb H phase diagram suggests only rather far fetched phase transitions around 100 K for the Hydrogen concentration values measured at the surface.

Another possible mechanism for the Q degradation is the "Hydride island" hypothesis. Here, it is assumed that the surface Hydrogen does not play the dominant role, because it is trapped and cannot undergo phase transition. The bulk Hydrogen atoms are in the  $\alpha$  phase (solid solution) at room temperature. During cooldown, they start to precipitate and form Hydride islands randomly distributed within the volume of the material, as soon as the solubility limit (fig. 20.b) becomes lower than the Hydrogen bulk concentration. This model has the merit of explaining nicely why 77 - 170 K is the dangerous temperature region, but it is hard to obtain large Q degradation with this type of explanation, since only the rare hydride islands located close enough to the surface can cause RF dissipation.

The two models presented above are only suggestions. Quite possibly, a third one might be right!

#### Conclusion

Finally, where are we in our fight against the Q degradation phenomenon? For sure, the symptoms are now well described. Cures are also known: fast cooling is very effective, but not always possible, especially for cavities in horizontal cryostats, with a large thermal inertia. To date, baking is the only sure way to get rid of the problem. If a chemical treatment is needed, it should be cool and open in order to minimize the Hydrogen incorporation. Anodization has given promising results; certainly, new (electro) chemical treatments should be tried in the future.

Between symptoms and cures, the mechanisms of Hydrogen incorporation and subsequent Q degradation turn out to be rather elusive, and are very poorly known. The Niobium lattice is very open, and its affinity for Hydrogen is famous. For this reason, virtually all steps of cavity preparation (forming and welding of half cells, chemical treatments, etc) are liable to bring Hydrogen contamination. To date, very little is known on this subject: only the final chemical treatment has been investigated in some details, and further experiments on samples and on cavities are clearly needed to identify the most dangerous step(s) in the cavity preparation process.

Incomplete as it is, the present study already makes available an improved reproducibility in superconducting cavity performance. We hope that the collaboration between the laboratories involved in this especially teasing topic will continue with success!

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### Table 1

Typical surface resistances measured at Saclay <sup>2</sup>

R0: Non degraded cavities.

R1: Degraded cavities, low field (≤ 60 G)

R2: Degraded cavities, high field (≥ 60 G)

F GHz	R <sub>O</sub> nΩ	R <sub>1</sub> nΩ	R <sub>2</sub> nΩ
1.5	20	350	1000
4	75	7100	13800
5.6	130	16600	24000

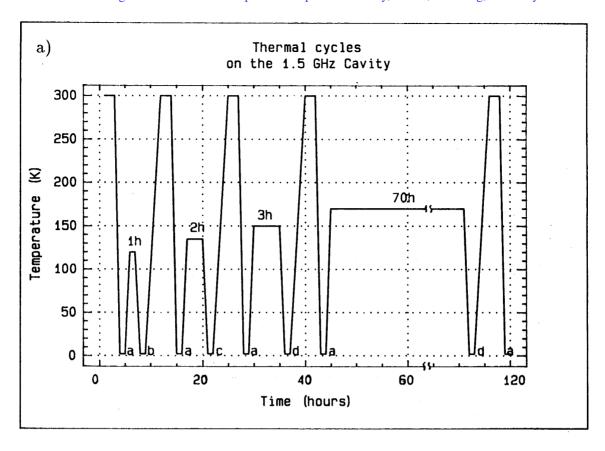
#### Figure captions

- 1. a) Thermal cycles on a 1.5 GHz cavity at Saclay.
- 1. b) Q(E) after thermal cycles. Labels refer to fig. 1.a.
- 2. a) Influence of cooling speed on the Q value of a DESY  $^{6,14}$  cavity (500 MHz, 4.2 K).
  - 2. b) Irreverible behaviour of the Q degradation observed at DESY.
  - 3. Q degradation observed at Darmstadt <sup>25</sup> (3 GHz, 1.8 K).
  - 4. Temperature map of a degraded cavity, measured at Wuppertal.
- 5. A KEK electropolished cavity (500 MHz, 4.2 K) shows no Q degradation after annealing  $(750^{\circ}C, 2hrs)$ .
- 6. A Saclay cavity (1.5 GHz, 1.8 K) shows no Q degradation after an annealing (750°C, 2hrs) and a short BCP (1-1-2, 3  $\mu m$ ).
- 7. A Saclay cavity (1.5 GHz, 1.8 K) shows a slight Q degradation after a firing (1500°C, at Cornell) and a long BCP (1-1-2, 53  $\mu m$ ).
- 8. Frequency dependence of the degradation. Results from multimode cavities at Saclay and Wuppertal.
- 9. Temperature dependence of the surface resistance for a polluted cavity at Saclay (TE011 mode, 4 GHz) a) fast cooldown; b) thermal cycle at 120 K.
  - 10. Field dependence of the Q value for degraded cavities. (1.5 GHz, 1.8 K).
  - a) Saclay; b) Wuppertal; c) CEBAF.

Note the steplike behaviour of the  $Q(E_{acc})$  curve at  $E_{acc}=1$  MV/m for degraded cavities.

- 11. Temperature and field dependence of the surface resistance of a degraded cavity (1.5 GHz).
- 12. The steplike behaviour of the temperature and field dependence of the surface resistance of degraded cavities could be explained by a superconducting transition.
- 13. The bulk Hydrogen concentration in Nb after various BCP treatments (from ref. 6).
- 14. The relationship between the equilibrium content of Hydrogen in Niobium and the partial pressure of molecular Hydrogen in its surroundings (Sievert law).

- 15. Open versus closed treatments. a) Bulk Hydrogen content (from ref. 6); b) Surface resistance (from ref. 3).
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  - 17. Reduction of Hydrogen content in Niobium by WCP. (from ref. 6).
- 18. The Hydrogen contamination depends on bath temperature. (from ref. 6).
- 19. ERDA measurements of the surface concentration of Hydrogen for variously treated Niobium sheet samples (RRR 200). (from refs. 13, 19)
  - V : HCl
  - C : HCl + 1-1-1 BCP +  $H_2O_2$  rinsing
  - $S: HCl + 1-1-2 BCP + H_2O rinsing$
  - $F: HCl + Furnace (750^{\circ}C, 2 hrs)$
- 20. Two possible mechanisms explaining the Q degradation: a) Hydride film; b) Hydride islands.



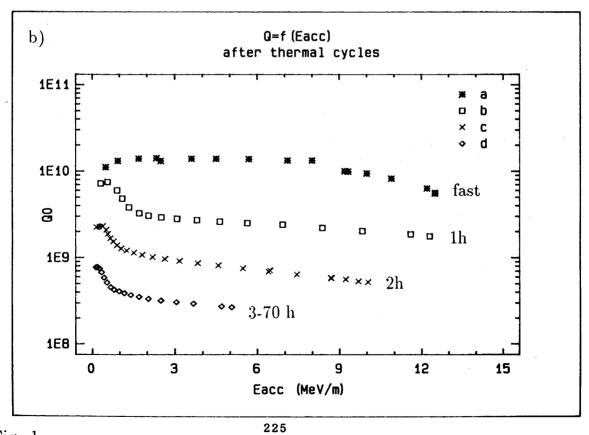
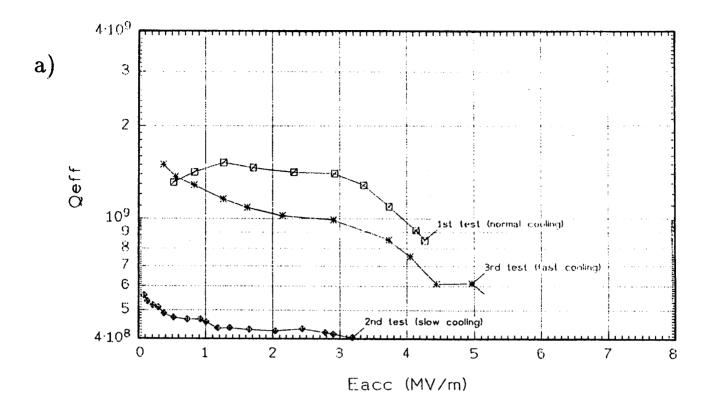
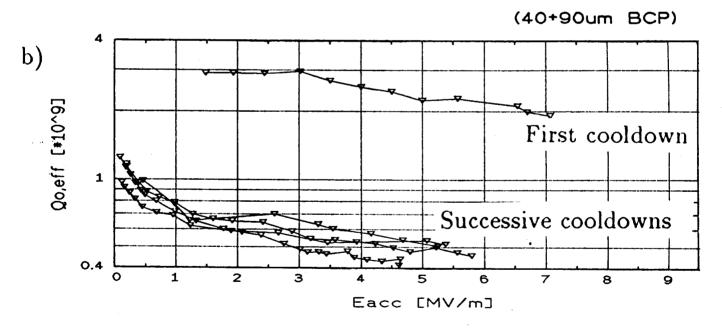


Fig. 1





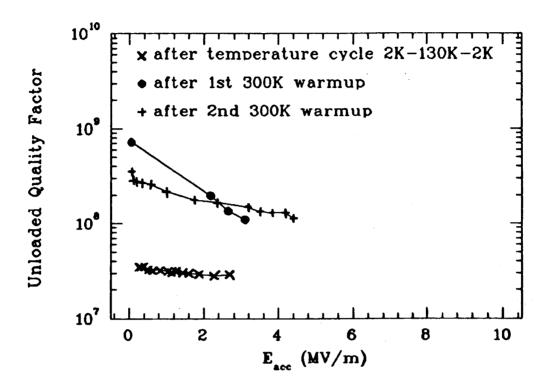


Fig. 3

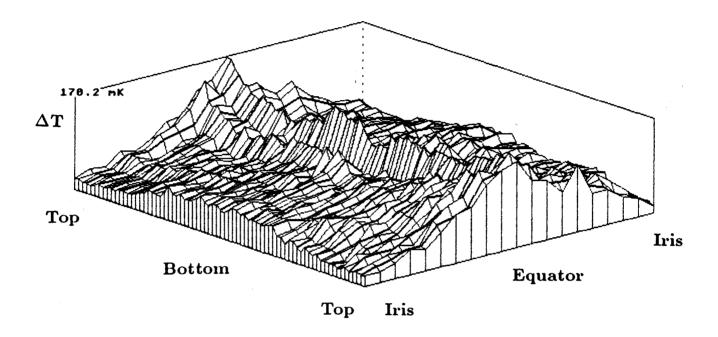


Fig. 4

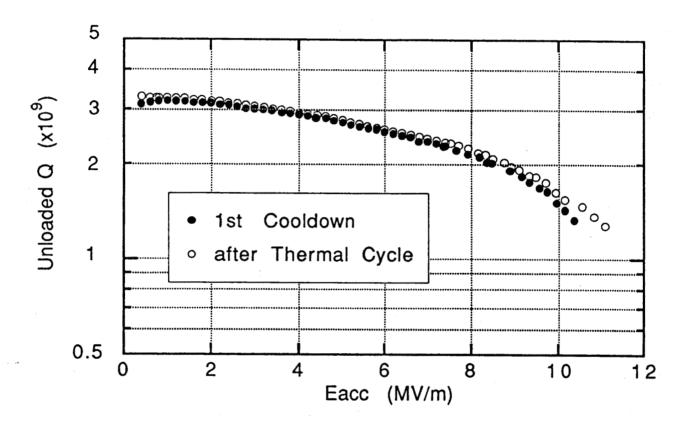
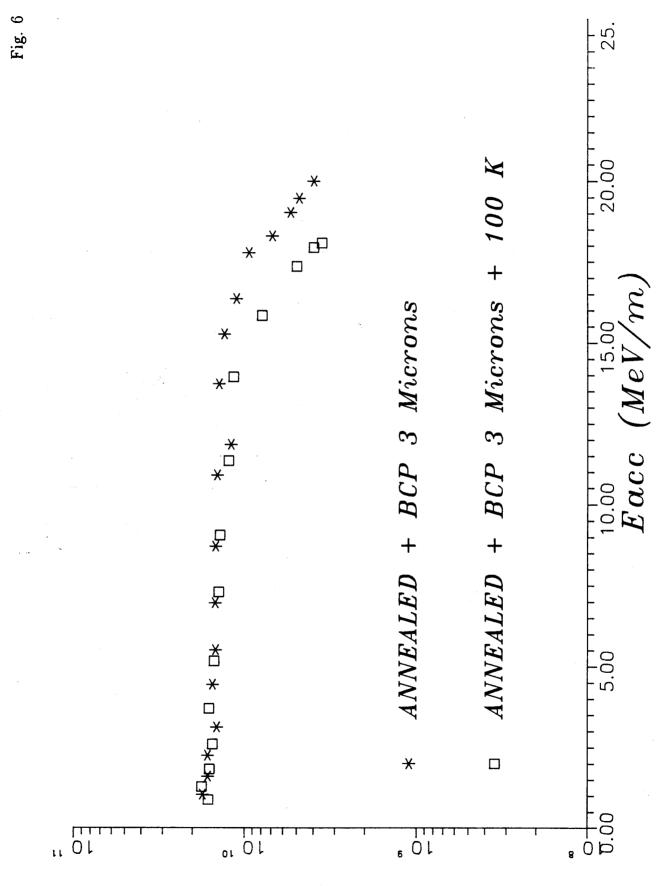
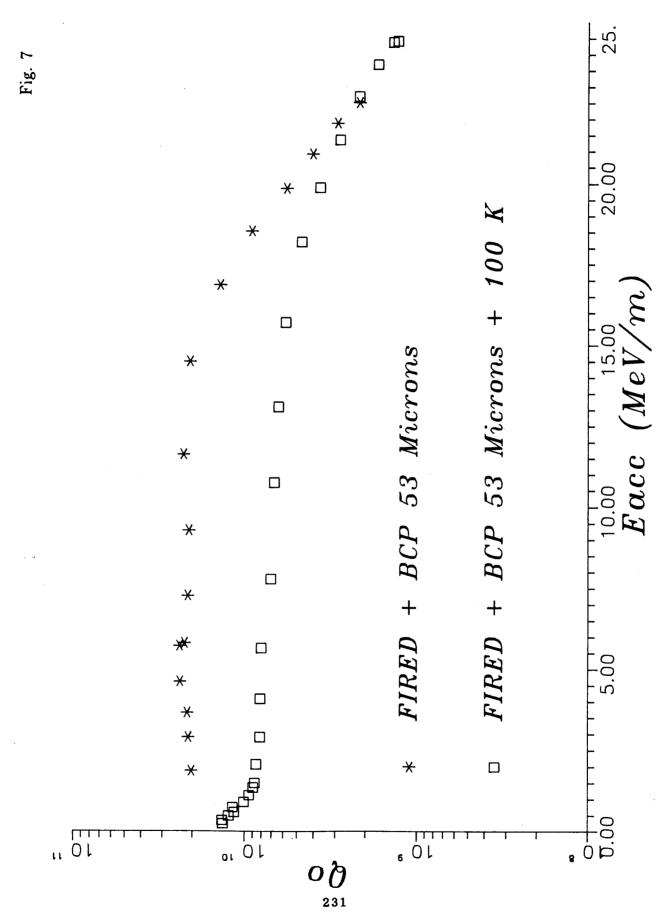
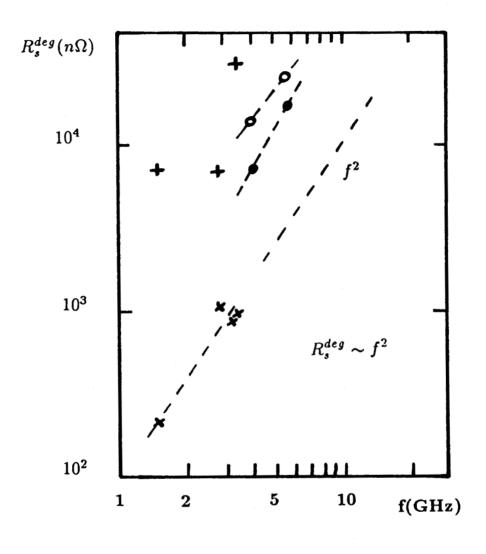


Fig. 5

**229** SRF91D01







- **X** Wuppertal WS1
- + Wuppertal WW2
- o Saclay high field
- Saclay low field

Fig. 8

SRF91D01 232

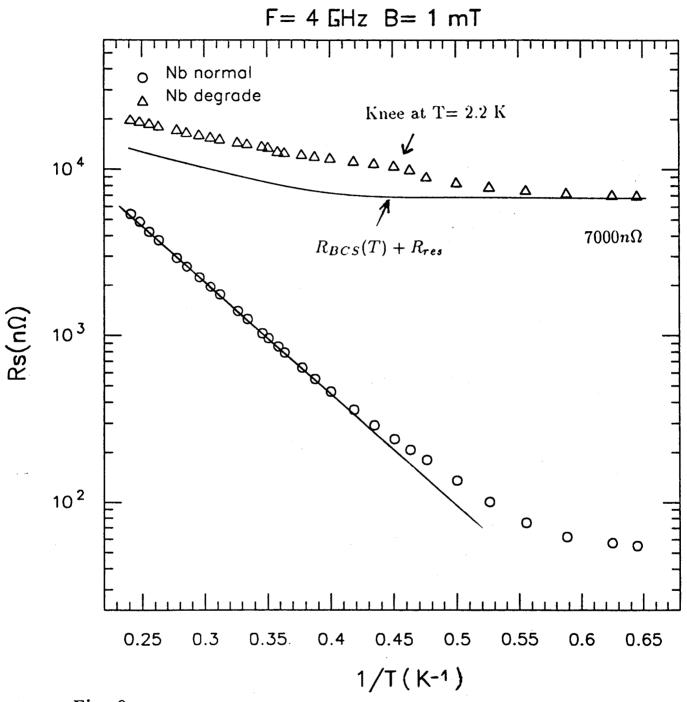
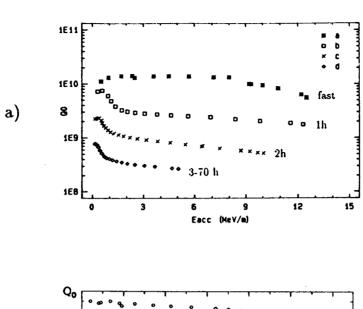
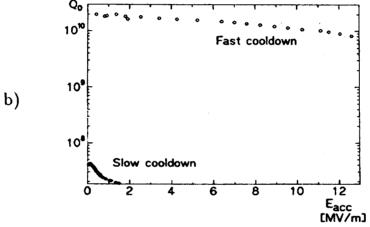


Fig. 9





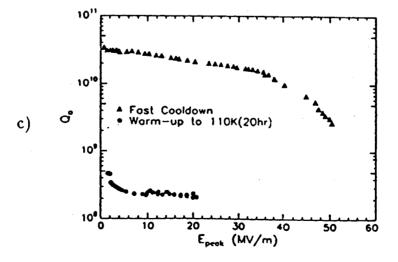


Fig. 10

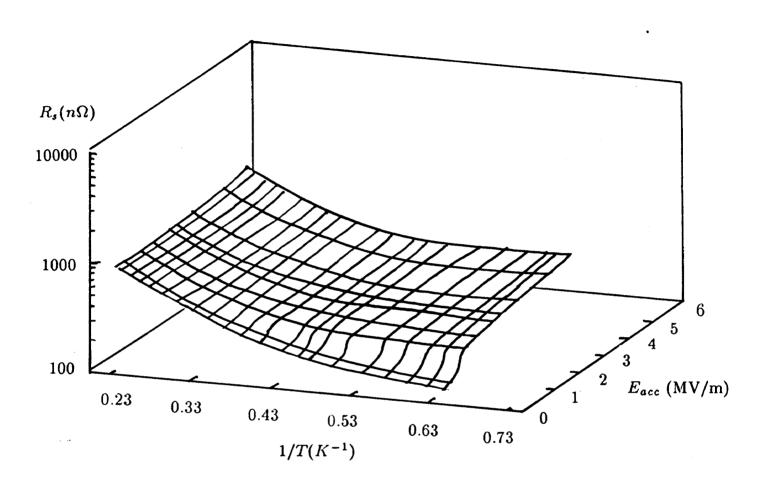


Fig. 11

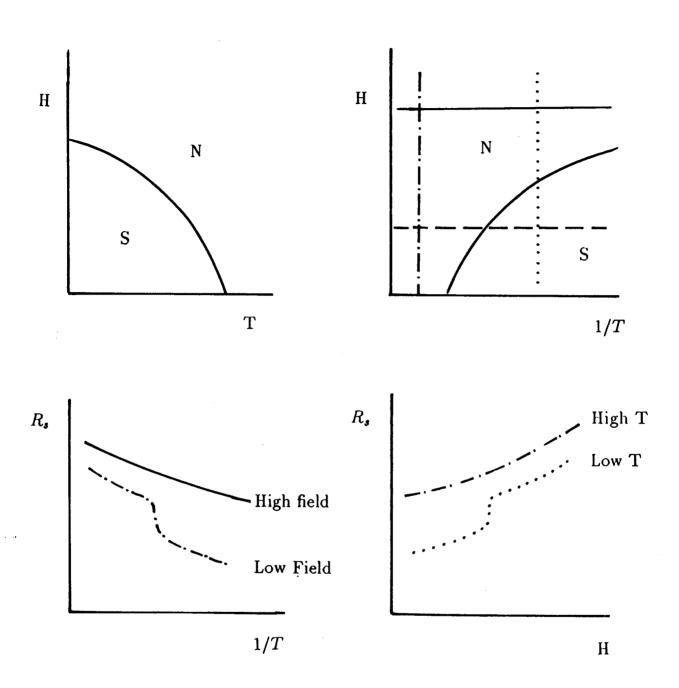


Fig. 12

# $[H]_{bulk}$ (wppm)

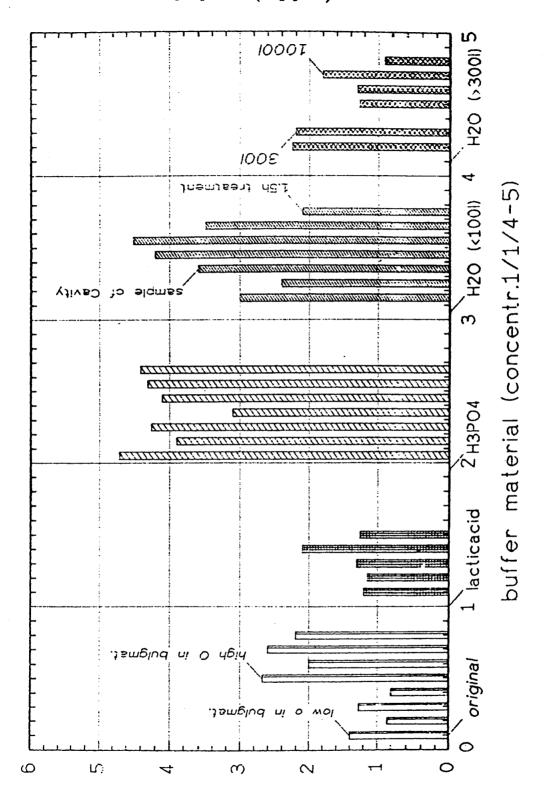


Fig. 13

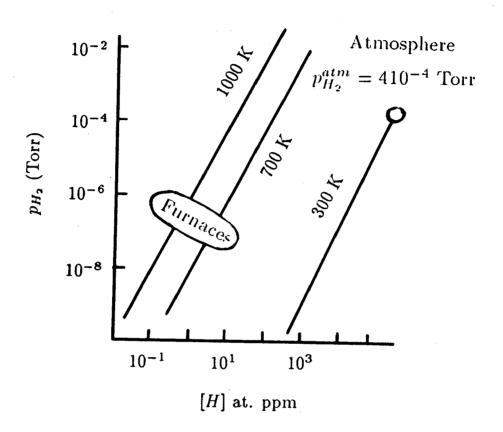
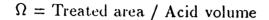
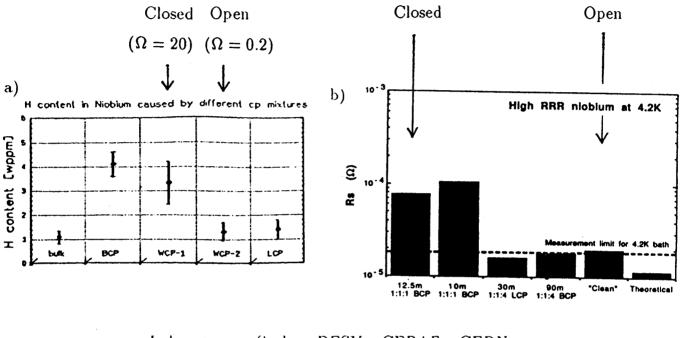


Fig. 14





Laboratory Saclay DESY CEBAF CERN  $\Omega$  5\* 12 9 9\*

Fig. 15

<sup>\*</sup> Same acid for several cavities.

## Q degradation: experiments at Saclay

$$BCP \longrightarrow D \ (influence \ of \ cooldown \ conditions \ )$$

$$BCP + F + BCP \ (50\mu m) \longrightarrow D \ (weak \ degradation)$$

$$BCP + F + BCP \ (5\mu m) \longrightarrow G$$

$$BCP + F + CP \ (1-1, \ 130\mu m, \ 10 \ min) \longrightarrow G$$

$$BCP + CP \ (1-1, \ 130\mu m, \ 10 \ min) \longrightarrow D$$

$$BCP + CP \ (1-1) + CP \ (1-9) \longrightarrow D$$

$$BCP + WCP \ (1-1-5, \ 40\mu m, \ 1h) \longrightarrow D$$

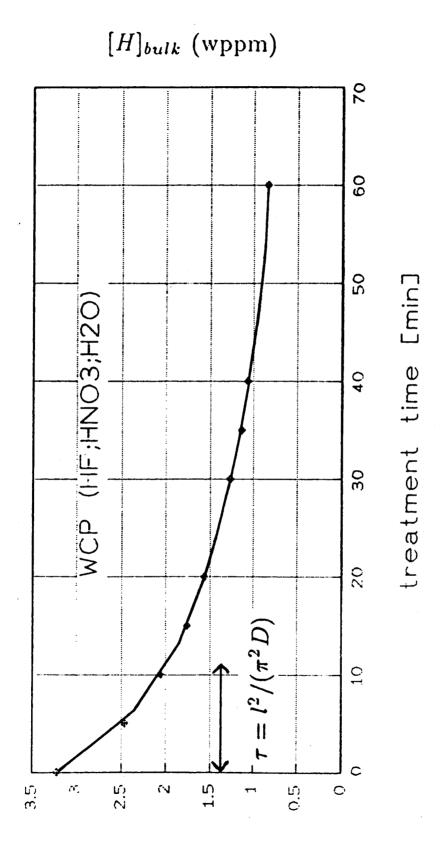
$$BCP + F + HCl + BCP \ (5\mu m) \longrightarrow G$$

$$BCP + F + HCl + BCP \ (5\mu m) \rightarrow G$$

 $D \equiv Degraded\ Q\ value\ after\ a\ long\ time\ at\ 100\ K$ 

 $G \equiv Good$  Q value after a long time at 100 K

 $F \equiv Furnace \ (750^{\circ}C \ 2 \ hrs)$ 



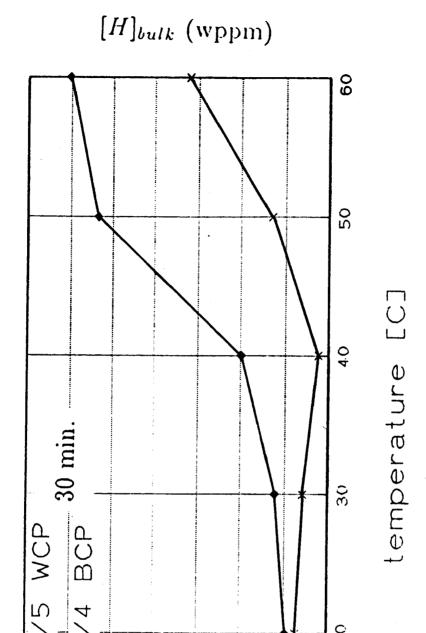


Fig. 18

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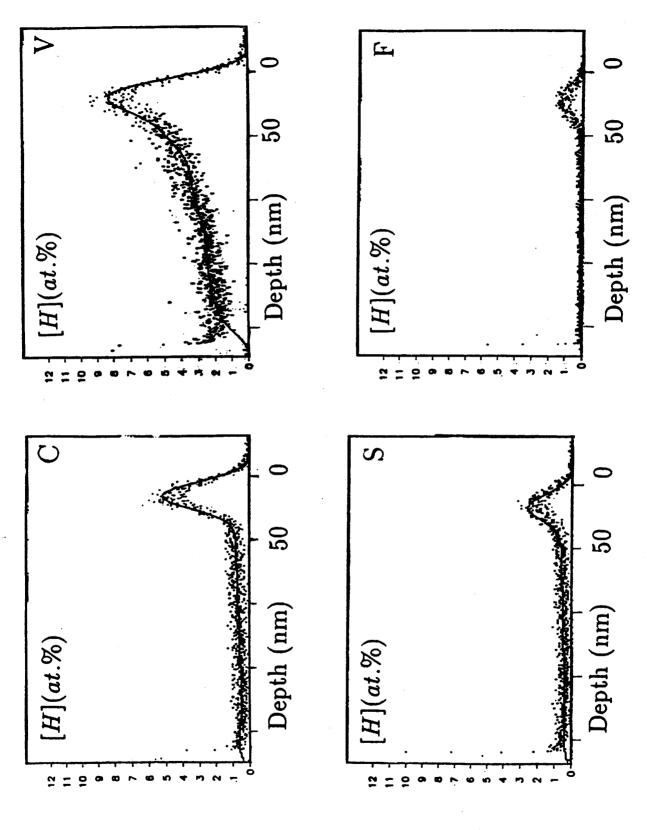
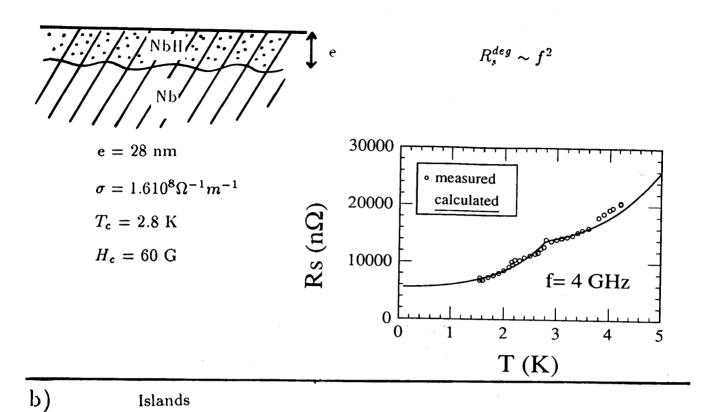
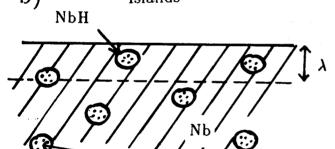


Fig. 1







 $R_s = x R_{Hydride}$   $x = a^3/d^3 = [H]_{bulk}/[H]_{Hydride} \simeq 10^{-4}$  $R_s \simeq 100n\Omega(1.5GHz);300n\Omega(3GHz)$ 

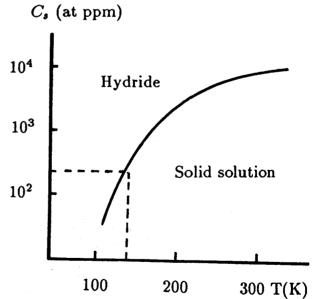


Fig. 20