

Aging of the HF-H₂SO₄ electrolyte used for the electropolishing of niobium superconducting radio frequency cavities: Origins and cure

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Electropolishing (EP) in the HF-H₂SO₄ electrolyte is the most desirable surface treatment for niobium superconducting radio frequency cavities yet demonstrated, in terms of performance and surface finish. However, the efficiency of the electrolyte declines quickly with time (decrease in removal rate, deterioration of the niobium surface, increased sulfur generation). Previous studies at CEA Saclay have highlighted the impact of the water content in EP mixtures rather than the content of dissolved niobium. Knowledge of the electrochemical system was improved thanks to studies using a rotating disk electrode (RDE). Measurements with a RDE give precious information concerning mass transport of the different ionic groups present in the solution. The performed measurements prove that EP is controlled by the diffusion of fluorine ions and the value of the related diffusion coefficient D_{F^-} was estimated for different mixtures. Electrochemical impedance spectroscopy (EIS) measurements were also performed with different EP mixtures. Both volt ampere metric and EIS measurements prove the central role of fluorine during EP and show that EP mechanisms evolve with the aging of the bath. Another major problem related to electrolytes is the formation of impurities such as sulfur. We have proved that working at a reduced voltage of 5 V does not alter cavity performance and makes it possible to reduce the undesirable particulate contamination in electrolytes and to increase their lifetime.

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I. INTRODUCTION

Electropolishing (EP) in the HF-H₂SO₄ electrolyte has been efficiently used for the polishing of niobium superconducting radio frequency (SRF) cavities for decades: the electrolyte is in contact with the cavity (anode) and with an aluminum cathode set in the symmetry axis of the cavity. When a voltage is applied, niobium is removed in a very uniform way (micropolishing). The smoothing of the surface is generally attributed to the presence of a viscous layer at the niobium surface in a diffusion regime. Recently, progress has been made in different laboratories to explain electrochemical mechanisms involved in EP and the origins of the aging of the electrolyte [1–4]. Electrochemical impedance spectroscopy (EIS) has been used and the existence of a compact film at the niobium's surface has been put forward [3]. EIS could be also used to evaluate the quality of the electrolyte [5]. In this paper, we will report improved understanding of the process thanks to the use of a rotating disk electrode (RDE). Complementary EIS measurements have been done in different mixtures. They prove that electrochemical mechanisms are likely to evolve with the aging of the bath. In particular, an increased diffusion of fluorine could make it possible to accelerate the removal rate or to counterbalance the aging

of the bath. Moreover, high operating voltage during EP is responsible for the premature aging of the electrolyte (early loss of the smoothing effect and increased particulate contamination). We successfully proved good results after EP at a reduced voltage of 5 V [process abbreviated as low-voltage EP (LV-EP)]. We have also shown after experiments on samples that EP at lower voltage reduces contamination. However, precise boundary conditions related to parameters such as fluorine concentration should be fixed precisely in the future.

II. EVALUATION OF FLUORINE DIFFUSION

EP experiments on static niobium samples have been used for a long time to understand and optimize the process. Previous experiments [2] using different [HF]/[H₂O] ratios and different concentrations of Nb in the electrolyte prompted us to anticipate fluorine diffusion as the limiting step of the electrochemical system. In addition, too high a water content was found to dramatically deteriorate the surface.

The first reported usage [6,7] of a rotating disk electrode for electrochemical measurements of niobium in concentrated HF-H₂SO₄ EP electrolytes made it possible to get precious information concerning mass transport of ionic species.

We used a system with three electrodes (see Fig. 1): (i) the rotating disk electrode (rotating at speed ω); (ii) a

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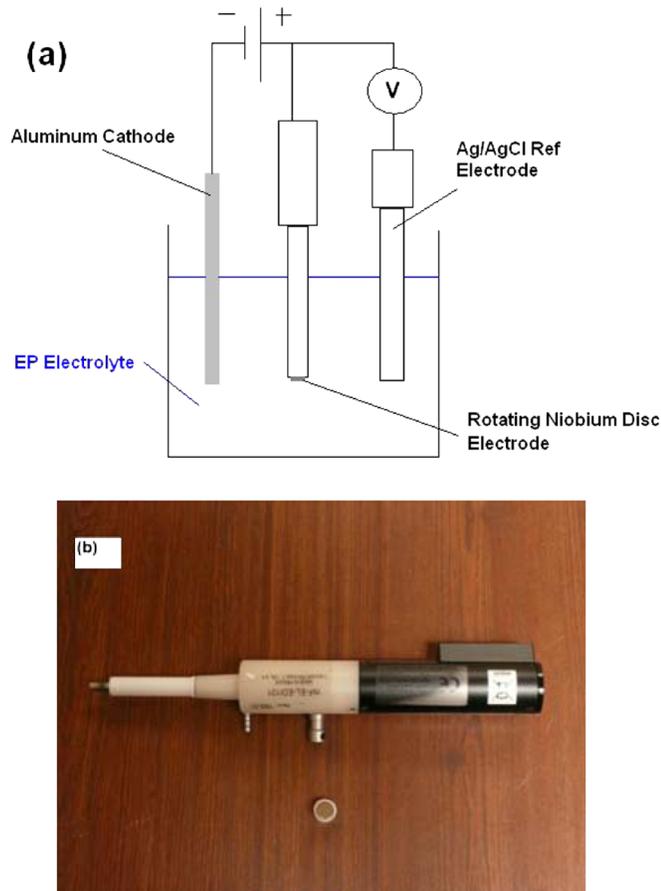


FIG. 1. (Color) (a) Setup with three electrodes used (top) and (b) picture of the rotating system for the disk sample (bottom).

high purity aluminum cathode; and (iii) an Ag/AgCl reference electrode (quoted hereafter as “Ref”).

The samples (10 mm diameter disks) were identically mechanically polished before electrochemical measurements: current intensity vs potential was plotted at different ω .

Note.—For measurements on samples, potentials are given with respect to the Ag/AgCl reference electrode. For cavity experiments, the voltage corresponds to the potential drop between the cavity and the cathode: no reference electrode is used.

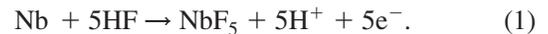
Measurements were made at 30°C ($\Delta T = \pm 1^\circ\text{C}$) with four electrolytes prepared with HF (40% or 48%) and H₂SO₄ (95%) in proportion 1–9. Mass percents are used and a mixture prepared with HF (48%) will be noted: 1–9(48%).

The studied electrolytes are: (i) A: 1–9(48%) freshly prepared; (ii) B: 1–9(40%) freshly prepared; (iii) C: 1–9(48%) + 9 g/L Nb (addition of Nb₂O₅ powder, to simulate Nb dissolution occurring during EP), freshly prepared; and (iv) D: aged mixture coming from previous EP experiments on samples (several hours) in open atmosphere.

The aim of mixture D is to evaluate the evolution of electrochemical mechanisms during EP. This mixture is characterized by the presence of dissolved fluorinated complexes and a reduced HF concentration due to: (i) chemical consumption to build Nb-F complexes; (ii) losses by evaporation (high saturated vapor pressure of HF); and (iii) dilution due to water incorporation in relation to hydrophilic properties of sulfuric acid.

Characteristics of studied mixtures are given in Table I. The concentrations in Table I are estimated considering ten atoms of F to dissolve one Nb₂O₅ molecule.

The reaction at the anode is the oxidation of niobium under its pentoxide form and its dissolution by hydrofluoric acid. The considered resulting simplified reaction is



I(V) curves were plotted at various rotation speeds as shown on Fig. 2. The graph exhibits standard features: (i) a first increase in current density with potential; (ii) a current instability area, and (iii) a “plateau” where current does not depend on voltage.

More chaotic oscillations are also expected at higher potentials, but we could not reach this area because of the limitation of the power supply.

The shift of the plateau towards higher currents at increased rotation speed is usually attributed to a diffusion-limited mechanism. The only ion which might be involved should be fluorine since: (i) water addition in EP mixture reduces the removal rate [1], (ii) addition of niobium in mixture at a constant [HF]/[H₂O] ratio does not induce a decrease in current [2], (iii) an increase in H₂SO₄ concentration is responsible for a decreased current.

We investigated the current evolution for the mixtures described in Table I as a function of ω . In the case of a system limited by diffusion, current intensity in steady state i_{ss} , measured with a rotating disk electrode, is given by the Levich equation [8]:

$$I_{ss} = 0.62 \times 10^{-3} n F S D^{2/3} \nu^{-1/6} \omega^{1/2} c \quad (2)$$

with (i) I_{ss} (A): current in steady state; (ii) n : number of

TABLE I. Formulation of the studied mixtures.

| Mixture | A 1–9(48%) | B 1–9(40%) | C 1–9(48%) + 9 g/L Nb | D 1–9(48%) Aged by use |
|------------|------------|------------|-----------------------|------------------------|
| [HF] mol/L | 2.78 | 2.26 | 2.30 ^a | <2.23 |
| Nb g/L | 0 | 0 | 9 | >10.28 |

^a*Note.*—Concentration of solution C is evaluated considering the HF consumed by the oxide dissolution.

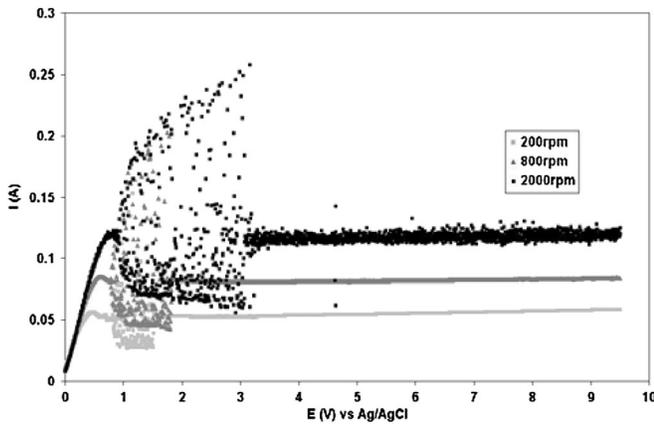


FIG. 2. $I(V)$ curves at different rotation speeds for mixture A.

electrons per diffusion unit (1 per HF) in Eq. (1); (iii) S (cm²): surface of the electrode; (iv) D (cm² s⁻¹): diffusion coefficient; (v) ν (St): viscosity; (vi) ω (rad s⁻¹): rotation speed of the electrode; (vii) c (mol l⁻¹): concentration of the active ion; and (viii) F (C mol⁻¹): Faraday constant.

Measurements were made for each mixture at 5 and 9.5 V, for different rotation speeds. $I_{ss} = f(\omega^{1/2})$ curves have been plotted for each mixture at 5 V and exhibit linear behavior (see, e.g., Fig. 3).

We have observed that: (i) in each case, the current intensity is proportional to $\omega^{1/2}$; (ii) the coefficient of proportionality is lower for the aged mixture D .

We can conclude from these results that EP of niobium in HF-H₂SO₄ electrolytes is limited by diffusion of F⁻: the corresponding mechanism is the “acceptor” model [9], i.e., the fluorine concentration tends to zero towards the niobium surface and the concentration gradient together with the diffusion constant governs the kinetic of niobium dissolution. From Eq. (2), it is possible to estimate D_{F^-} . The only missing parameter is the value of the viscosity of the electrolyte. As an approximation, we have assimilated HF in the EP mixture to H₂O and have chosen the viscosity of the corresponding concentrated H₂SO₄ mixture found in

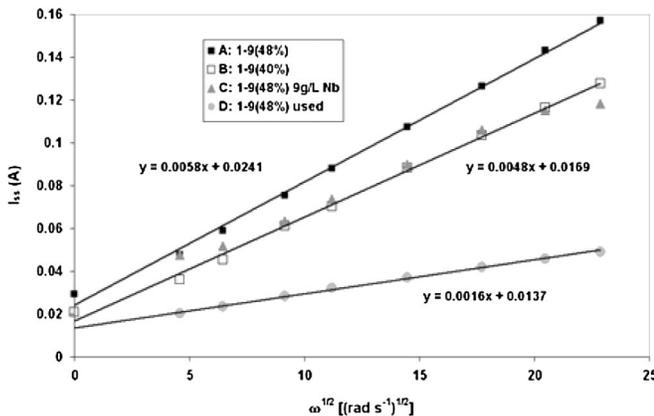


FIG. 3. Proportional relation between I_{ss} and $\omega^{1/2}$ at 5 V.

TABLE II. Values calculated for the diffusion coefficient D_{F^-} at 5 and 9.5 V.

| Mixture | Potential/ref (V) | D_{F^-} (10^{-8} cm ² s ⁻¹) |
|---------------------|-------------------|---|
| A 1-9(48%) | 5 | 13.11 |
| A 1-9(48%) | 9.5 | 14.13 |
| B 1-9(40%) | 5 | 13.80 |
| B 1-9(40%) | 9.5 | 14.23 |
| C 1-9(48%) 9 g/L Nb | 5 | 10.91 |
| C 1-9(48%) 9 g/L Nb | 9.5 | 10.65 |

[10]. Values for D_{F^-} are reported in Table II. We might conclude that this approximation is valid, since coefficients are similar to those obtained with a viscometer [11]. We do not report here values for D mixture because uncontrolled HF losses which have occurred during the aging of this mixture can not be precisely estimated. Values in Table II are in accordance with diffusion coefficients reported for different EP systems in concentrated electrolytes. For example, according to [12], the diffusion coefficient of water for Cu electropolishing in H₃PO₄, is between 10^{-9} and 10^{-7} cm² s⁻¹, depending on the solvation model considered. D_{F^-} is found lower in mixture C. Additional experiments are required to know if the decrease is due to the presence of dissolved niobium in solution or if the calculation must be improved to consider the physical modification of the mixture (change in viscosity). Because of the highly viscous electrolytes used, magnitudes of the diffusion coefficients are lower compared to those achieved in aqueous solutions ($\sim 10^{-5}$ cm² s⁻¹).

In all cases, a high rotation speed, responsible for the thinning of the diffusion layer, did not alter the surface finish after EP (see Fig. 4). For example, in the case of the mixture D , the surface of the sample deteriorates in static configuration, whereas it is smoothed at high ω . We might then by analogy anticipate that it is possible to improve the surface finish of a cavity electropolished with an aged mixture by increasing its rotation speed during treatment.

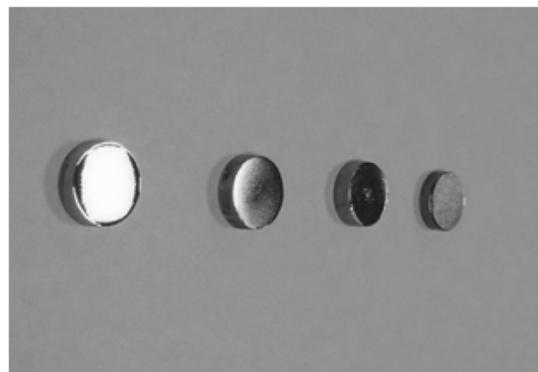


FIG. 4. Bright samples after experiments at high rotation speeds.

III. EIS EXPERIMENTS WITH DIFFERENT MIXTURES

In addition, complementary EIS measurements have been done on the mixtures described in the previous paragraph (with different HF concentrations and different amounts of dissolved niobium). The samples used were parallelepipeds, set vertically. Nyquist diagrams were plotted at 4.5 V vs Ref for frequencies between 100 kHz and 0.1 Hz. We notice that the features at high frequencies are similar to previous results obtained by Tian *et al.* [3]. The characteristics of these features R0 and R1 [see Fig. 5(a)] evolve as a function of the composition of the mixture [see Fig. 5(b)]: (i) R0: limit of the impedance at high frequency; (ii) R1: diameter of the semicircle.

In particular, we observe that R1 is much higher in the aged mixture D. A reduced $[HF]/[H_2O]$ has been foreseen to provoke the increase in the polarization resistance R1 [3]. However, this explanation is not sufficient since the addition of water in 1–9–2 mixture (two additional volumes of water) does not increase the R1 value: the niobium dissolved in solution does also influence the polarization resistance. We can assess that a higher water content in the EP mixture presents two antagonist effects: (i) a light addition [in 1–9(40%) compared to 1–9(48%)] provokes a reduction of R0 which should be attributed to a better dissociation of the acids; (ii) a more decreased $[HF]/[H_2O]$

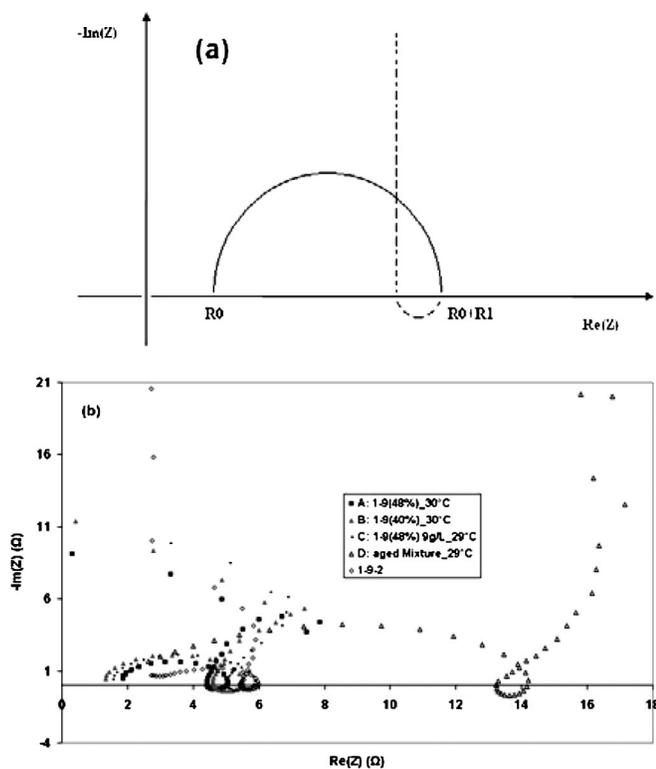


FIG. 5. (a) General shape at high frequencies of Nyquist diagrams (top) and (b) spectra obtained at 4.5 V vs Ref range 100 kHz and 0.1 Hz (bottom).

ratio in 1–9–2 or aged mixtures provokes a shift of R0 towards higher impedances. This might be due to a modification of the niobium interface.

Therefore we investigated if the compact structure of the interface film [3] could evolve with bath composition or aging of the electrolyte. In that way, we studied the evolution of R0 in A, B, C, and D mixtures as a function of the potential. An increase in R0 with potential would be the signature of a porous structure of the film which thickens with an increasing voltage [13]. As in [3], R0 is found independent of the voltage in case A. However, for mixtures B, C, and D, we notice such an increase in R0 with potential (see Fig. 6). This could be attributed to the thickening of the external surface of the film through a porous structure [13]. The oxide layer would evolve towards a “duplex structure” with the aging of the bath. It is in accordance with other publications [14,15].

We might also add that typical features are observed with an optical microscope [Fig. 7(b)] for samples deteriorating after EP in aged electrolyte. These features might be considered as the signature of the incriminated surface film.

Similar patterns are also observed for different electrochemical systems and occur, for example, during electro-polishing of aluminum in acidic electrolytes [16].

We can draw a sketch for the evolution of electrochemical mechanisms during EP: (i) In a fresh EP mixture, high HF concentration is responsible for important fluorine diffusion and efficient removal rate of niobium; a compact film is present at the surface of niobium. (ii) With the aging of the bath (consumption and evaporation of HF, presence of dissolved niobium), the removal rate of the oxide decreases. (iii) The oxide layer becomes thicker with a “duplex-type” structure: the outer surface is porous. (iv) This duplex structure generates typical patterns at the niobium’s surface which might cause the deterioration of the surface aspect.

A high voltage should in that way speed up the deterioration of the surface by thickening the porous layer. It is in accordance with results observed on flat samples: lifetime

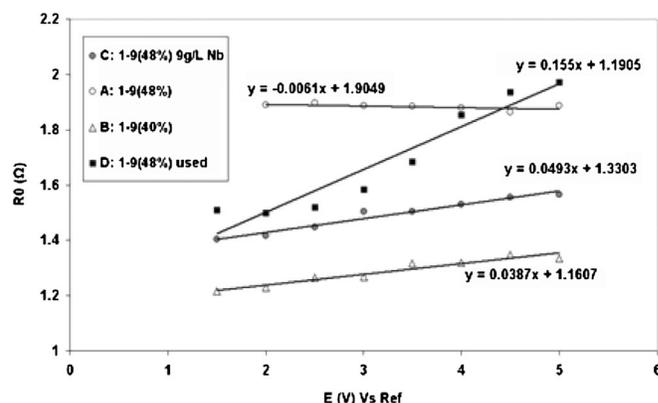


FIG. 6. R0 vs potential for A, B, C, and D mixtures.

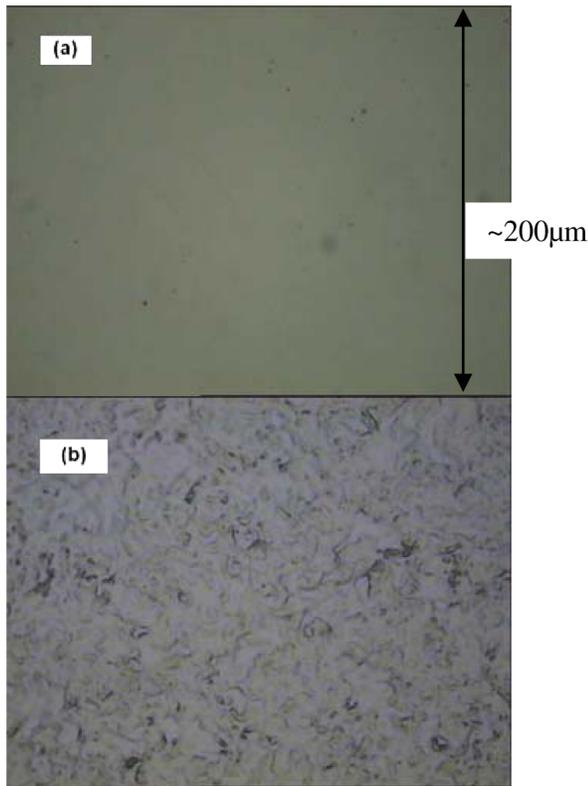


FIG. 7. (a) Smooth surface obtained after EP in fresh EP mixture (top) and (b) typical features observed on samples after EP in an aged mixture, with limited fluorine diffusion (bottom). The same scale is used for both pictures.

of the electrolyte in terms of surface finish is increased by using a lower voltage [2].

It is then necessary to boost the flux of F⁻ ions towards the niobium interface to increase the lifetime of the EP electrolyte (in terms of simultaneous sufficient removal rate and smoothening of the surface). It might be achieved by: (i) increasing the flow rate of the acid; (ii) increasing the HF concentration.

Nevertheless, an upper limit for HF concentration has to be determined, because previous experiments show that a too high HF concentration leads to the loss of the diffusion plateau [1]. Such a mixture has been tested on single-cell cavity. A pristine untreated “1AC03” cavity was electropolished by standard EP (~100 μm removed at 17 V and 30–35°C) and then electropolished with a mixture prepared with HF(40%), H₂SO₄ (95%), and additional water in proportions 3–6.72–0.28. The voltage was between 3 and 4 V and the removal was approximately 50 μm (in this paper, the average removed thickness on cavity is estimated from mass losses). The cavity was tested at 1.4 K (Fig. 8). A low residual surface resistance ($R_s = 2.86 \text{ n}\Omega$) and an impressive quality factor Q_0 (10^{11}) were achieved at low gradient (2.45 MV/m) but a strong Q slope is found with an onset at 5 MV/m without any field emission. This Q slope should not be attributed to hydrogen contamina-

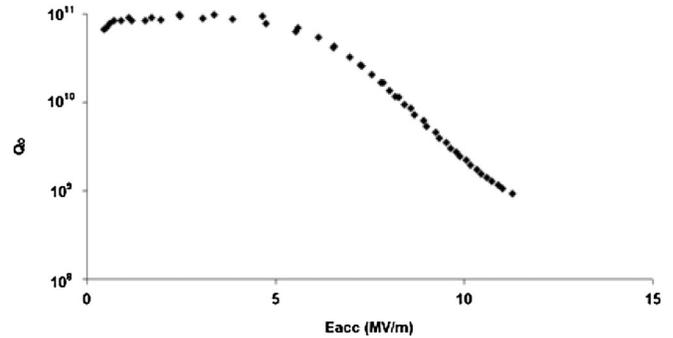


FIG. 8. The rf test of the cavity 1AC03 after 100 μm removed with 1–9(40%) and 50 μm removed with 3–6.72–0.28 mixture. Q_0 deterioration at low gradient.

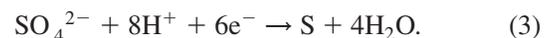
tion since EP experiments on samples with this mixture show that hydrogen uptake is not increased (1 ppm after 6 hours electropolishing at 5 V) compared to standard EP. However, insufficient material removal on this cavity should not be excluded and further experiments are required to confirm and explain this result.

IV. VOLTAGE AND PARTICULATE CONTAMINATION

HF-H₂SO₄ electrolytes have been known for a long time to generate impurities, and more precisely sulfur [17]. Field emission observed on cavities could be attributed to the presence of such particles, remaining on the cavity surface because of insufficient rinsing procedures. Systematic rinsing with different chemicals (detergents, solvents) has made it possible to improve the yield of cavity performance [18]. However, it would be more reliable to prevent sulfur generation at its root. Chemical origins of contaminants were described in a previous study [19]: (i) S and H₂S generated by chemical attack of Al by H₂SO₄ and by cathodic reduction of SO₄²⁻, and (ii) aluminum salts, due to light oxidation of Al in concentrated HF-H₂SO₄ electrolytes.

Investigations demonstrated that a low [HF]/[H₂SO₄] content and voltage were favorable to high sulfur generation [19]. In this present paper, we intend to focus on the impact of the voltage parameter in highly concentrated HF-H₂SO₄ electrolyte. In fact, two main electrochemical reductions might take place at the aluminum cathode during EP: (i) reduction of H⁺ into H₂, and (ii) reduction of SO₄²⁻ ion into H₂S and S.

Simplified electrochemical reaction for SO₄²⁻ reduction was proposed [7]:



The main reaction is the reduction of H⁺ into H₂. Nevertheless, a cathodic overpotential might modify this balance, as suggested by previous results in [19,20]. With this purpose in mind, we used an electrolyte with reduced HF concentration to generate significant and measurable

TABLE III. Parameters for experiments E and F.

| Experiment | E | F |
|------------------|----------|----------|
| Voltage | 20 V | 5 V |
| Duration | 4041 min | 6907 min |
| Temperature °C | 25–35°C | 25–30°C |
| Mass removed | 9.18 g | 9.11 g |
| Spots at the end | Yes | No |

impurities forming: long term EP experiments were carried out on flat samples in a mixture prepared with HF (40%), H₂SO₄ (95%), and deionized (DI) water, respectively, in proportions 1–9–1. Two baths E and F of 900 ml were prepared for experiments at 20 and 5 V. Two similar niobium square samples (30 × 30 × 3 mm³) were used for experiments in E and F mixtures. Table III summarizes data concerning experiments E and F.

Note.—“E” or “F” will refer to the corresponding electrolyte as well as the sample which is electropolished inside.

After the EP experiments, samples were extracted from EP mixtures and rinsed with DI water. (i) In case E, the whole surface of the sample is covered with sticky yellow spots (see Fig. 9). (ii) In case F, the surface is bright.

The yellow stains can be removed neither by additional ethanol rinsing nor chloroform rinsing. They are so sticky that it is necessary to scratch the surface with a sharp metallic tool to remove them. Preliminary x-ray photoelectron spectroscopy (XPS) results on the stains show the presence of sulfates, as well as the presence on the surface of impurities such as Zn (one of the major impurities contained in the acids) or Si (probably originating from dust). It is then clear that the use of high voltage combined with HF depleted electrolytes favors parasitic reactions responsible for the surface deterioration of niobium.

To confirm that point, we went on with the analysis of electrolytes E and F.

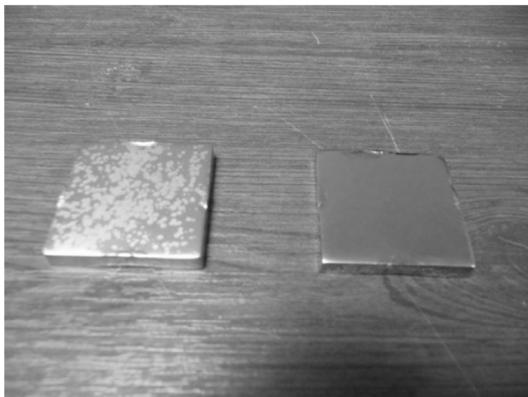


FIG. 9. E (left) and F samples after long EP experiments at 20 and 5 V. Sample E is covered with yellow spots.

TABLE IV. Mass of impurities found after chloroform extraction.

| Sample | E (20 V) | F (5 V) |
|-----------|------------|------------|
| 1st rinse | 4.5 mg | 2.6–2.7 mg |
| 2nd rinse | 1.2–1.3 mg | 0.9–1.2 mg |

The described protocol was carried out to extract impurities in both electrolytes: (i) one sample of 200 ml is taken from each mixture; (ii) the 200 ml of electrolyte are mixed with 100 ml of chloroform in a pear shape separating funnel; (iii) distinct acid and organic phases are separated; (iv) the acid phase is rinsed a second time with additional 100 ml of chloroform; (v) distinct acid and organic phases are separated for the second rinsing step; (vi) the two organic phases obtained are rinsed several times with DI water in order to remove residual acidity; and (vii) once the pH of the rinsing water is neutral, the two organic phases are evaporated.

As shown below, impurities were found in the four solution samples. Masses measured are related in Table IV.

The mentioned impurities are yellow colored and exhibit the characteristic smell of sulfur compounds and were attributed to sulfur contamination as observed in [21]. They were analyzed with energy dispersive x-ray (EDX) and infrared (IR) analysis. The EDX analysis exhibits a large carbon signal but no large quantity of S, whereas IR analysis indicates clearly the presence of an organic acid. Therefore we think that sulfur contamination appears along some form of organic contamination, which renders it particularly sticky to the surface and prevents its removal through high pressure rinsing. We thus obtain precious information concerning the nature of the contaminants found in aged electrolytes. The amount observed increases with voltage.

In both E and F experiments, the temperature in the bulk electrolyte was controlled at 30°C. We did not have access to the local temperature at the niobium sample surface. In those conditions, we cannot distinguish between a contamination due to chemical reactions occurring at higher local temperature or/and due to additional undesirable electrochemical reactions favored by higher voltage. Additional experiments at high voltage and lower temperature are required to better distinguish between the two possible origins. Whatever the contamination origin is, a direct consequence or a derivative one, high voltage is not recommended for niobium electropolishing.

V. LOW-VOLTAGE ELECTROPOLISHING OF SRF CAVITIES

EP at a lower voltage of 5 V (LV-EP) has been experienced on single-cell cavities at CEA Saclay with a horizontal setup to tackle the generation of contaminants [5,7,20,22]. In fact, their production will be reduced (see previous paragraph). However, parasite cathodic reactions

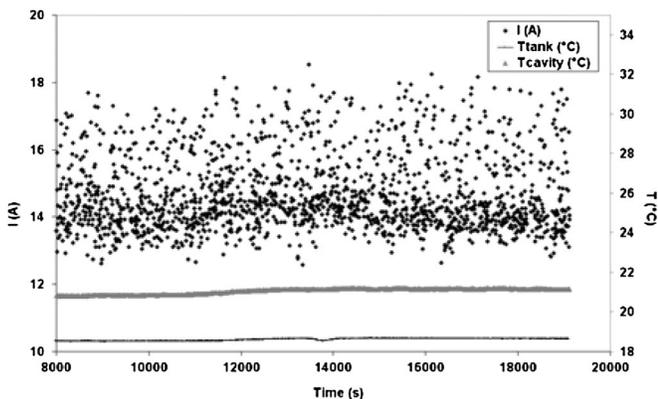


FIG. 10. Current intensity and temperature vs time for the C1-21 cavity during LV-EP (5.4 V). A characteristic current instability is observed. The electrolyte temperature is very stable.

still occur since H₂S is smelled after EP treatments. The potential has been chosen in order to operate in a current instability area (see Fig. 10). EP experiments based on current-oscillation control have been made in different laboratories but at higher potentials (9–15 V [23,24]).

Because of this lower voltage, the heat produced by the Joule effect during EP is decreased. A beneficial consequence is the easier control of the electrolyte temperature. It is even possible to work without a heat exchanger by operating at 20 °C (temperature of the acid in the storage tank).

Precisely, most of the cavity measurements presented in this paper result from LV-EP at 20–25 °C. As a consequence, the removal rate was low (around 10–15 mA cm⁻²) compared to standard EP at 17 V and 30 °C. As the working point is located at the beginning of the plateau on I(V) curves, and because of the exponential evolution of current intensity with temperature [3], it is logical to expect a decreased removal rate difference for LV-EP at 30 °C. Different strategies might be considered to compensate the decrease in removal rate: boosting the diffusion of fluorine during the process (see Sec. II), and the use of a vertical configuration during EP, characterized by a larger niobium surface in contact with acid.

The rf performance of two single-cell tesla-shaped cavities has been tested after LV-EP: (i) CERCA C1-21 has been dedicated to LV-EP experiments. After the first sequences, the cavity reached 32 MV/m. Additional material was removed to evaluate the performance of the cavity. It still improved (see Fig. 11) and reached 35 MV/m (total ~150 μm removal) and 39 MV/m (total ~175 μm removal). (ii) In addition to this, the DESY IDE1 single-cell cavity has been used for complementary experiments. This cavity was previously electropolished several times at 17 V [25]. The last test (35 MV/m, limited by quench) has been taken as a reference. Additional 50 μm were then removed by LV-EP. The obtained gradient is comparable to the previous one achieved at 17 V (Fig. 12).

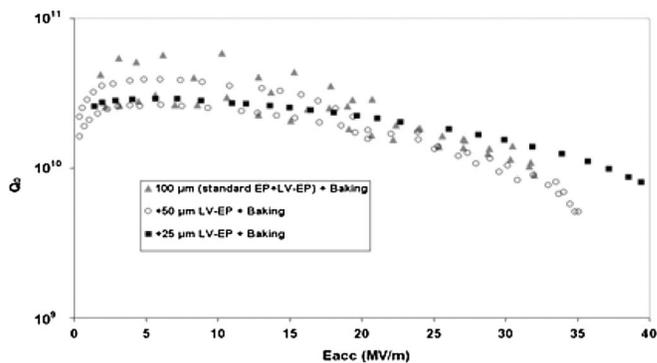


FIG. 11. The rf tests at 1.4 K. Improvement of the gradient for C1-21 cavity after successive LV-EP sequences.

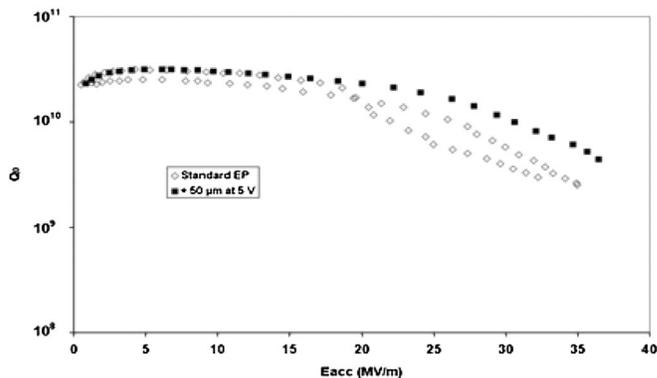


FIG. 12. Successive rf results at 1.4 K on the single-cell IDE1 cavity after standard EP at 17 V and LV-EP at 5 V. No decrease in performance is observed after LV-EP.

These results confirm that LV-EP provides gradients similar to those obtained with standard EP. An operating voltage between 5 and 17 V should not be considered to have an impact on cavity performance. The observed inner surface of the C1-21 cavity is very bright with a different aspect compared to cavities electropolished at 17 V: the surface appears to be rougher. This rougher surface could result from the absence (or the thinning) of the viscous film at the niobium surface during EP. Replicas of the surface have been made in order to compare qualitatively surfaces achieved at 5 and 17 V. If a rougher surface is confirmed at 5 V, rf performance should not be correlated to that physical aspect.

VI. CONCLUSION

Voltamperometric and EIS measurements highlight the origins of the decreased current and deterioration of the surface occurring with aging EP mixtures: studies show that electropolishing of niobium is limited by diffusion of fluorine towards the niobium surface. This limitation is effective for fresh and aged mixtures which suffer from uncontrolled losses of HF due to vaporization and chemical consumption. In this latter case, the flux of fluorine is

decreased and is responsible for the apparent deterioration of the surface, which can be attributed to the growth of a porous oxide layer.

The decrease in HF concentration also favors the apparition of solid impurities deposited onto the surface of niobium, according to a complex mechanism that needs further exploration.

The results on low-voltage EP offer new perspectives and procedures refinement for the treatment of elliptical niobium cavities: (i) It makes it feasible to use electropolishing of cavities with high inner surface without any limitation due to overheating of the electrolyte. (ii) The standard cavity treatment for the European x-ray free electron laser (XFEL) application combines a bulk EP (heavy removal of 150 μm), followed by a final EP with lower removal which aims at obtaining a clean and smooth surface. Low-voltage EP is particularly adapted to this final step because lower removal rate is less prejudicial for a light surface removal.

With low-voltage EP, fewer impurities should be generated and the process would be improved toward obtaining particle-free cavities to be assembled in the clean-room.

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