

ECR plasma cleaning: an in-situ processing technique for RF cavities

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A condition for Electron Cyclotron Resonance (ECR) can be established inside a fully assembled RF cavity without the need for removing high-power couplers. As such, plasma generated by this process can be used as a final cleaning step, or as an alternative cleaning step in place of other techniques. Tests showed filtered dry air plasma can successfully remove sulfur particles on niobium surface while the surface oxygen content remains intact.

The present cleaning regimen for superconducting RF (SRF) cavities involves the use of dangerous concentrated acids for etching the internal surfaces. Alternative cleaning processes that avoid the use of acids while still obtaining good results would be highly desirable from a safety point of view. One such effective process is plasma cleaning, in which surface material is removed via plasma-enhanced chemical reactions and simple ion bombardment. While direct ion bombardment generally does not efficiently remove all contamination, addition of reactant gases allows surface material to form gaseous compounds that can be exhausted through the vacuum system. Glow discharge (DC discharge or RF discharge), magnetron plasma and Electron Cyclotron Resonance (ECR) plasma are among the common techniques applied widely in applications with metal surfaces, as described earlier^{1,2}. Along these lines, glow discharges of H₂, Ar or Ar/O mixtures have been extensively studied for storage ring vacuum vessel cleaning³. N₂ RF plasma was successfully used to reduce the water vapor and other contaminants in a megawatt gyrotron RF source⁴.

The main particulate contaminants in niobium SRF cavities include dust, grease, flakes introduced from screw threads and other tooling, sulfur particles left behind from electropolishing, and material extending from scrapes and scratches of the cavity surface itself. Ion bombardment can be used to ablate such particles. Chemically reactive plasmas are effective when they attack the base niobium, oxides, hydrocarbons, and sulfur. Reactive gas choices vary: oxygen can effectively break down hydrocarbon contaminants, while hydrogen breaks down oxides and sulfur. Niobium can be etched by introduction of halogens or oxygen, such as via a mixture of CF₄ and oxygen in Ar carrier gas^{5,6}. If the cavity temperature can be elevated, chlorine or bromine can be used to attack niobium also. Recent work at Thomas Jefferson

National Accelerator Facility (JLab) and Old Dominion University⁷ has explored plasma etching based on earlier niobium etching studies. Similar efforts of surface cleaning in RF systems using plasma are pursued at INFN/Laghero⁸.

A central difficulty in the above plasma cleaning techniques is the need to attach specialized fittings to introduce a plasma-generating device (e.g. a magnetron). This means that there is a risk for re-contamination during the disassembly of the hardware after plasma cleaning and during the subsequent step of attaching RF couplers to the cavity. This risk can be mitigated by generating plasmas inside cavities that are fitted with high-power input couplers and sealed against sources of re-contamination.

If magnetic field is applied to an operating cavity as shown in Figure 1, so that the ECR frequency $\omega_c = eB/m_e$ is close to the cavity resonance frequency ω_0 , plasma generation can result. The required field for 3.9 GHz cavities, for instance, is approximately 1400 Gauss. The ECR condition for plasma generating applies as long as injected electrons can stimulate electron emission from the niobium surface, which takes place over a wide range partial pressure of reactant gases. At lower end of pressure range, the mean free path of ions is large, which enhances the direct interaction between plasma ions and the cavity walls. This feature gives flexibility to optimize the ECR plasma parameters for effective cleaning of different types of surfaces. Also the electron energy gained through ECR can be adjusted by varying the magnetic field magnitude and partial pressure, allowing accommodation of different ionization cross sections.

An accelerating gradient of about 130 V/m is required to maintain emission of electrons from the niobium surface and sustain the plasma. The power requirement to reach this threshold is considerably less than that required for in-situ cleaning via high power pulsed processing. With a variable RF input coupler, ECR cleaning only requires a CW amplifier of a few hundred watts power, which are commonly available in vertical testing setup. In a more practical case, a dressed RF cavity has a fixed coupler that is designed for high-power connection to the cold cavity. At room temperature, it is very weakly coupled to the cavity. Moreover, the excitement of plasma introduces losses that reduce the cavity quality factor and the accelerating field. Still, the power available from many klystrons is sufficient to support enough field strength inside a dressed RF cavity. Figure 2 shows an estimation of the margin at room temperature for different coupling factors. Different RF modes such as TE mode may have stronger external Q for certain input couplers. Such modes could reduce the RF power requirement and allow the use of a convenient solenoid magnet. To satisfy the ECR condition, the magnetic field strength increases in proportion to the RF frequency of a given mode. For now, we focus on the TM₀₁₀ mode, which has strong electric field at the iris and should be effective to mitigate the field emission problem.

Our first attempt to achieve ECR plasmas cleaning used a setup shown in Figure 3. The cavity was connected to RF power through a beam pipe coupler commonly used during cavity vertical testing. The coupler has external Q of about 1000. This reduced the power requirement for RF amplifier for initial test as described in Figure. 2. Directional coupler was inserted into the transmission line and a power meter measured incident and reflected power. The RF power was provided by a 120 W traveling wave tube amplifier. The pickup probe for the cavity was optional. The cavity was connected to inlet gas line at the RF input side. A needle valve was installed in the inlet gas line to provide fine adjustment of cavity gas pressure between 10^{-3} Torr and 10^{-6} Torr. A pump station was connected at the other side of the cavity. Two fixed valves were attached to the cavity for future cold test. Initially, cavity was evacuated to better than 10^{-7} torr. A dipole magnet provides the relatively uniform external magnetic field perpendicular to the axis of the cavity. Center field of the magnet was calibrated as a function of current. For a 3.9 GHz cavity, the required magnetic field is 1393 Gauss.

At first, argon was introduced to understand the relationship between plasma power absorption, gas pressure, magnetic field strength and RF frequencies. Plasma light can be seen through view port. The argon plasma could be initiated at various conditions, verifying the flexibility that was hoped for. Once the plasma was excited, it becomes self sustaining at a broad range of conditions. The 130 V/m mentioned earlier of ionization threshold was experimentally verified.

As plasma absorbed the RF power, the loaded Q reduced to below 1000. This made the bandwidth of RF resonance relatively large. Still, plasma could be sustained within a frequency range as wide as 40 MHz. This allowed us to insert a sample further to the iris of the cavity and still have sustained plasma. The pressure range for stable plasma was also very large, which could provide future opportunity to tune the plasma ion density and ion energy.

A purposely contaminated niobium sample was prepared using sulfur powder and its initial condition was characterized by scanning electron microscopy (SEM). Some other contaminants such as carbon are also present. The sample was inserted into the beam pipe near the iris of the cavity as illustrated in Figure 2 and plasma was initiated. Low density argon plasma was introduced into the cavity to promote gas adsorption. Duration of this stage was 20 minutes. Then a filtered air flow was introduced into the cavity. A needle valve controlled the air flow so the actual pressure near the cavity was maintained at $\sim 2.5 \times 10^{-4}$ Torr. RF power was increased to around 50 W. ECR condition was supported for 30 minutes. After this, the sample was taken out to the SEM protected in a clean container. Dramatic result of the surface cleaning can be seen from Figure 4. Several particulates were not completely removed as the processing time was rather short, yet their sizes were evidently smaller. The cavity temperature did not exceed 48°C during the entire plasma treatment period.

The oxygen ions within the air plasma may alter the surface oxide composition which played very important role in terms of cavity performance⁹. The same sample in Figure 4 was characterized by secondary ion mass spectrometer (SIMS). Another reference sample which underwent the same surface preparation except plasma treatment was also characterized by SIMS. The result was almost identical as shown in Figure 5. This proved that the oxygen ion within the plasma did not penetrate into the niobium surface layer to cause extra niobium oxidation.

We concluded the in-situ ECR plasma can be very flexible compared to the other plasma techniques. The sample experienced short processing time showed the processing will likely be effective for sulfur contaminant. Oxygen depth profile by SIMS showed there were no sign of oxygen deep penetration into the material, let alone the alteration of the surface oxide composition. However, cavities will experience more varieties of contaminants. The final cavity performance will hinge on the successful removal of all contaminants while not compromising surface quality. Future studies will be focused on finding an optimized procedure and gas mixtures to effectively remove as many contaminants as possible and their effects on surface composition. RF test using single cell cavities will be employed to finally qualify the best procedure.

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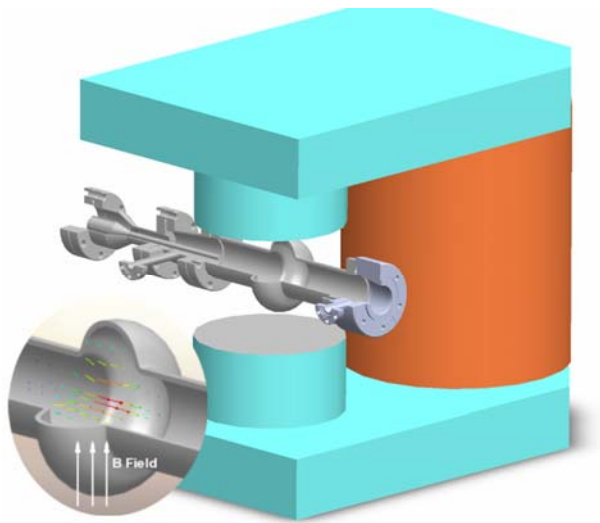


FIG 1: A single cell cavity with input antenna, gas inlet and pumping port under the dipole magnet. Lower left zoomed view shows the orientation of magnetic field and cavity electric field.

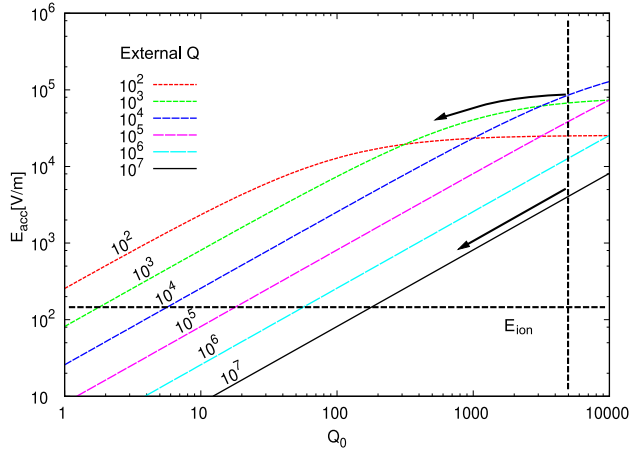


FIG 2: Curves represent the cavity electric field (E_{acc}) for applied RF input power of 50 W for different values for the input coupling. For the tested cavity, $Q_0 = 5000$ at 300 K. This is enough to establish ionization of electrons and surface cleaning (denoted by the threshold $E_{ion} \sim 130$ V/m) when directly connected to the klystron (input coupling $\sim 10^3$). Since Q decays when plasma is established in the cavity, E_{acc} also decays as indicated by the arrows. Still, E_{acc} remains above the electron ionization potential of ~ 130 V/m even when high power couplers are used (input coupling $\sim 10^7$).

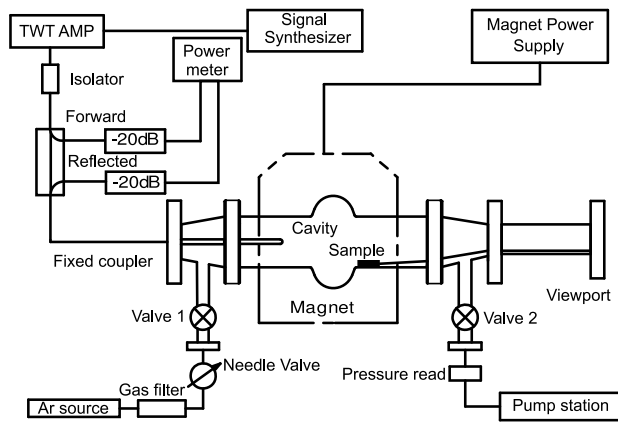
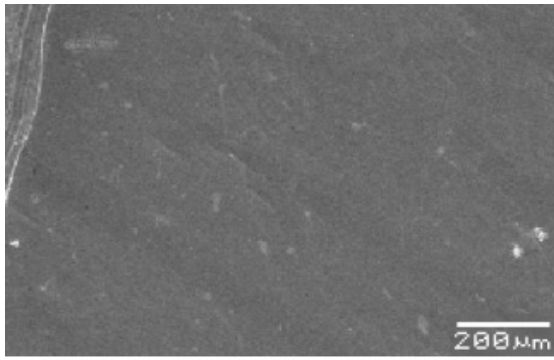
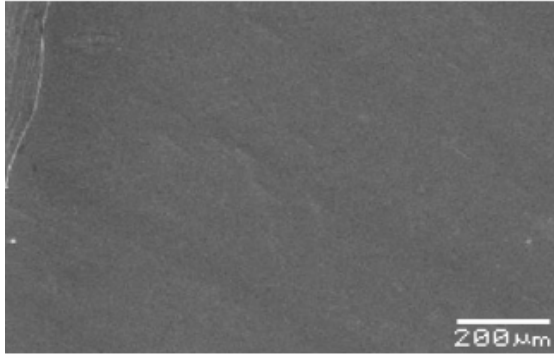


FIG 3: Schematics of plasma excitation in a cavity and sample placement.



(a)



(b)

FIG 4: SEM surface images for a niobium sample contaminated by sulfur; a) before plasma treatment; b) after plasma treatment

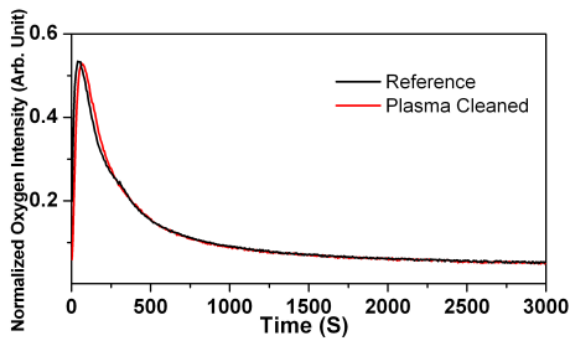


FIG 5: Oxygen depth profile of plasma treated and reference samples.

- ¹S.M. Rosnagel, J.J. Cuomo, and W.D. Westwood, eds. *Handbook of Plasma Processing Technology: Fundamentals, Etching, Deposition, and Surface Interactions (Materials Science and Process Technology)* 1990, Noyes Publications. 523.
- ²R.J. Shul and S.J. Pearton, eds. *Handbook of Advanced Plasma Processing Techniques*. 1 ed. 2000, Springer. 653.
- ³H. F. Dylla, *J.Vac.Sci. Technol. A*, **6**, 3 (1987).
- ⁴William E. Cohen, R.M.G., Reginald L. Jaynes, Christopher W. Peters, and Y.Y.L. Mike R. Lopez, Scott A. Anderson, and Mary L. Brake, Thomas A. Spencer, *Appl. Phys. Lett.* **77**, 23, 3725 (2000).
- ⁵M. Chen and R. H. Wang, *J.Vac.Sci. Technol. A*, **1**, 2 (1983).
- ⁶Jay N. Sasserath and John Vivalda, *J.Vac.Sci. Technol. A*, **8**, 6 (1990).
- ⁷L. Phillips, private communication
- ⁸N. Patron, R.B., L. Phillips, M. Rea, C. Roncolato, D. Tonini, and V. Palmieri., in *Proc. of Thin Films and new ideas for pushing the limits of RF superconductivity*. 2006. Padua, Italy.
- ⁹G. Ciovati, *Appl. Phys. Lett.* **89**, 022507 (2006).