Nitrogen Doping and Infusion in SRF Cavities: A Review

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Abstract

Advances in SRF technology over the last 40 years allowed achieving accelerating gradients ~ 50 MV/m corresponding to peak surface magnetic field close to the theoretical limit of niobium. However, the quality factor decreases significantly with increasing accelerating gradient. This decrease is expected since increasing the rf field increases the number of quasiparticles and therefore the rf losses. Recently, a new phenomenon of increase in quality factor with the accelerating gradient has been observed when SRF cavities are doped with certain non-magnetic impurities. In particular, the diffusion of nitrogen into the niobium cavities inner surface has been successfully implemented into the commercialization of SRF technology. The quest is still ongoing towards process development to achieve high accelerating gradient SRF structures with high quality factors for future high power accelerators. Here, we present the review of the research and development via nitrogen diffusion, materials analysis and current theoretical understanding in high $Q_0$ SRF cavities.

I. Introduction

Superconducting radiofrequency (SRF) technology is being used not only for the basic fundamental nuclear physics research but also for applications that have benefited society [1]. Most of the future accelerating machines in large or compact systems completely rely on SRF technology. Few examples of the current and future projects based on accelerator technology are continuous wave (CW) free electron lasers, x-ray laser oscillators, ERL based light sources, short photon pulses in storage ring light sources, electrons and ions colliders, accelerator driven system for medical isotope production and nuclear waste transmutation. SRF technology which is based on the superconducting hollow structures (cavities) with high duty factor provides the required accelerating gradient to accelerate the charge particles close to the velocity of light.

The superiority of superconducting cavities over the normal metal cavities is its ability to store a large amount of energy with much lower dissipation. The performance of the SRF cavities is measured in terms of the quality factor $Q_0 = G/R_s$, where $G$ is the geometric factor which depends on the cavity geometry and $R_s$ is the surface resistance, as a function of accelerating gradient, $E_{acc}$. For typical cavities of resonating frequency 0.5–3 GHz operating at a temperature ~2.0 K the quality factor is in the $10^{10}$–$10^{11}$ range. In the last four decades, much research work has been

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focused improving the accelerating gradient and quality factor of these SRF cavities. The theoretical gradient limit on these cavities, limited by the superheating field, has been achieved on some occasions. Considerable effort has gone into pushing the quality factor to higher values as well. Higher $Q_0$ for the reduction of losses (lower cooling capacity) and higher $E_{acc}$ for high energy and compact accelerators are desired (Fig. 1). Currently under constructions and proposed accelerators such as FRIB, ILC, XFEL, LCLS-II, ADS and ERLs have different specifications for the gradient and quality factor. Machines such as ILC and XFEL are designed to be operated in pulse mode whereas LCLS-II, ADS and ERLs are designed to operate in CW mode [2,3]. For the CW applications, the reliable and affordable cryogenic refrigeration system currently limits the optimal gradient to ~20 MV/m for long term accelerator operation. Continuous research and development already pushed the achievable accelerating gradient towards the fundamental limits [4]. The historical landscape of the R&D of the L-band SRF cavity on single and multi-cell cavities is shown in Fig. 2.

**Figure 1.** Schematic of SRF cavity performances.

Thus current and future accelerators would highly benefit from cavities with higher quality factors and gradient. A higher quality factor can be achieved by minimizing the surface resistance of SRF cavities. Surface resistance in superconducting materials is the sum of the temperature-independent residual resistance ($R_{res}$) and temperature-dependent Bardeen-Cooper-Schrieffer (BCS) resistance ($R_{BCS}$) [5,6]. The sources of the $R_{res}$ are trapped magnetic flux during the cavity cool down, impurities, hydrides and oxides, imperfections, and surface contamination. The BCS surface resistance results from the interaction between the RF electric field within the penetration depth and thermally activated quasi-particles in the superconductor. The BCS surface resistance depends on superconducting material parameters which may vary strongly due the presence of the metallic impurities within the rf penetration depth.

In past decade, much development of the high quality factor SRF cavities via the material diffusion in the thin layer of the inner surface of the cavities has been achieved. The possibilities of higher quality factor in SRF cavities were first realized by the titanium doping during annealing ($\sim 1400 \, ^\circ C$) without any post-annealing chemistry [7,8] and later by nitrogen doping at 800 $^\circ C$, 2
followed by electropolishing [9]. The diffusion process not only showed the increase in quality factor at low field levels, but also an increase in quality factor with increasing accelerating gradient, contrary to the previously observed $Q$-slope. The nitrogen doping recipe rapidly implemented into the industrialization of the technology in processing of SRF cavities for LCLS-II cryomodules production. One of the challenge faced during the cavity processing is the degradation of the accelerating gradient after the nitrogen doping compared to the gradient limit would have been achieved with the available processing technique available to that date.

Most recently, efforts have been made to preserve high accelerating gradients while also increasing the quality factor of SRF cavities. In these new nitrogen “infusion” cavity processing recipes, cavities were heat-treated at high temperature (>800 °C), then the furnace temperature is reduced to 120–200 °C and nitrogen was introduced into the furnace at a partial pressure of ~25 mTorr for several hours. This process has shown an improvement in $Q_0$ over the baseline measurements, without the need for post-annealing chemical removal of material from the inner surface [10,11,12]. Even though the diffusion of the nitrogen into the bulk of the SRF cavity is limited in-depth at these low temperature, the introduction of nitrogen is sufficient to modify the cavity surface within the rf penetration depth as seen from rf results, which are similar to those previously reported for high-temperature nitrogen doped cavities. Furthermore, while post-doping electropolishing is required to remove coarse nitrides from the surfaces of high-temperature nitrogen doped cavities, no further processing is required for the low-temperature infusion recipe showing a clear benefit in reducing processing steps as well as keeping higher gradient with high $Q_0$ values.

**Figure 2.** Historical landscape of the R&D of L-band SRF cavity on single and multi-cell cavities. Also shown is the accelerating gradient specification for several operational, under constructions and proposed future SRF based accelerators [13].
The historical development of SRF technology can be found on several conference and workshop proceedings [14]. This paper is organized as follows. In section II, we present the invention of nitrogen doping recipe. In section III, we present the recipe developments related to furnace requirements, nitrogen diffusion time and electropolishing. In section IV, we comment on the industrialization efforts related to LCLS-II. In section V, we present the recent development on recipe modification towards the high gradient, high quality factor SRF cavities. In section VI, we present the sample studies in order to understand mechanism behind the improvement in SRF cavity performances. In section VII, we present the available theoretical models that explains the high $Q$ and high gradient cavity performance. Section VIII presents the summary and future outlooks.

II. Invention of Nitrogen Doping

Over the last several decades, several attempts were made to increase the quality factor of SRF cavities. Initially, with the recognition of high concentration of surface hydrogen leading to formation of Nb-H phases which in turn leads to poor cavity performance, there were attempts to reduce surface H concentrations by passivating the Nb surface with the growth of protective oxides [15] and nitrides [16,17]. The idea behind these surface passivation is to avoid the reabsorption of hydrogen on the bulk of niobium such that the surface is transparent to rf field without dissipating any extra rf power. The surface oxidation experiments were carried out at high temperature showed the enhanced $Q$ [15]. Furthermore, additional titanium impurities diffused into the Nb surface during the high temperature heat treatment. The Ti source was Nb-Ti flanges of the SRF cavities at high temperature. The diffusion of Ti into the Nb surface layers showed that the performance of the corresponding Nb cavities improved with increase in quality factor with the accelerating gradient as shown in Fig. 3(a) [7,8].

The similar experimental exploration was carried out at Fermi Lab in 2013 in an attempt to form niobium nitride on the inner surface of SRF cavity by reacting bulk niobium cavities with nitrogen in a high temperature UHV furnace [9]. Although Nb-N proved to be a non-ideal surface, it was discovered by careful controlled electropolishing and cavity measurements of subsequent

![Figure 3. First demonstration of increase in quality factor with rf field after (a) after Ti-doping on 1.5 GHz single cell cavity at 2.0 K [7] and (b) with nitrogen doping followed by electropolishing on 1.3 GHz cavities at 2.0K [9].](image-url)
layers that layers below the Nb nitride surface that were revealed by electropolishing showed increase in quality factor with accelerating gradient when inner surface of the cavity was removed by electropolishing as shown in Fig. 3(b). The $Q_0(E_{acc})$ curve showed the anti-Q-slope similar to that was observed when the cavity was heat treated at high temperature in titanium environment [7,8]. It was concluded that the interstitial diffusion of the non-magnetic impurities (Ti and N) indeed enhance the quality factor by decrease in BCS surface resistance as the accelerating gradient increases. The advantage of nitrogen doping over the Ti doping is that nitrogen being a gaseous, it is easier to control the partial pressure in high temperature environment and nitrogen diffusion takes place at lower temperature compared to titanium, which required temperature higher than 1250 °C [8].

III. Nitrogen Doping Recipe Development

After the demonstration of exceptionally high quality factor on nitrogen doped SRF single cell cavities, the focus quickly shifted to the application of nitrogen doped cavities in multi-cell cavities and eventually in superconducting linear accelerator. Around the same time, the Linac Coherent Light Source-II (LCLS-II) project quickly adopted the nitrogen doped SRF cavities to be installed in cryomodules [18]. The LCLS-II baseline design required 1.3 GHz 9-cell TESLA shaped cavities [19] with an average intrinsic quality factor $Q_0 \sim 2.7 \times 10^{10}$ with accelerating gradient of 16 MV/m at 2.0 K [20]. Joint LCLS-II R&D efforts among different laboratories towards achieving the high quality factor were started with aiming towards the development of the cavity processing technique that can be commercially produced in large numbers [21]. Three partner labs to LCLS-II projects: Fermi Lab, Jefferson Lab and Cornell University rapidly developed separate recipes with nitrogen doping on single cell cavities and were successfully applied to 9-cell cavities [22,23,24,25].

A. Furnace Requirement

Generally most of the SRF facilities are equipped with the furnace capable of reaching temperature of 1250 °C with resistive heaters. The SRF facilities at Fermi Lab [26], Jefferson Lab [27] and Cornell University [28] used furnace manufactured by TM vacuum [29]. These furnace are equipped with dry roughing pump, cryopumps, residual gas analyzers, vacuum gauges and gas injection system. The high purity nitrogen (> 99.9999%) was used during the doping process with the flow controlled by mass flow controller. Typically, the temperature of the furnace is ramped up to desired temperature (800-1000 °C) at the rate of 3-5 °C/min and hold for few hours (~3 hours), mainly for the hydrogen degassing purpose. At the end of degassing purpose, high purity nitrogen gas was injected for a period of time (2-30 min), also called soaking time, with specified partial pressure of nitrogen. During the early stage of doing recipe development, the gas was turned off, the furnace was evacuated and the furnace was naturally cooldown to the room temperature. Later the recipe was modified in such a way that after the soaking time, the furnace was evacuated and the cavity was further annealed before cooling down to room temperature. For example, a research group at KEK initially weren’t able to reproduce the doping results [30] and with the improvement on the furnace pumping system, they successfully reproduced the doping results [31]. The diffusion of other elements such as hydrocarbons and other residual metal particles in furnaces are found to be detrimental to cavity performance.
B. Nitrogen Diffusion

Thermal diffusion of nitrogen into Nb is a method which had been used in the past to produce NbN films [32,33,34], with higher superconducting transition temperature than Nb. Earlier attempts to produce the nitride phase for surface passivation in SRF cavities didn’t result in the Q-rise phenomenon, however the overall increase in quality factor was observed in all accelerating gradient when the SRF cavities were heat treated in nitrogen environment at temperature ~400 °C with partial pressure of ~ $10^{-5}$ mTorr [17]. The study suggested that the temperature and partial pressure of nitrogen was insufficient to diffuse in to the bulk of niobium or formation of any nitride phase in the surface of niobium.

Historically, Nb-N system is interesting because of the superconducting behavior of the $\delta$-NbN$_{1-x}$ phase [35]. Several multi-phase regions exist in the equilibrium phase diagram of Nb-N depending on N: Nb ratio as shown in Fig. 4. In the low N concentration regime below 10 at % of N, the main phase is that of nitrogen as an interstitial in Nb, and as concentration increases forms a two phase mixture of $\alpha$-Nb-(N), where N is an interstitial and a stoichiometric $\beta$-Nb$_2$N phase [36]. From the phase diagram, the phase of the nitride on niobium is determined by the concentration of the nitrogen and temperature of niobium substrate [37,38]. The apparent activation energy during the nitrogen diffusion decreases with increasing temperature up to about 650 °C. Then it stays constant up to 1300 °C, above which it increases to a new value of 50 kcal/mol [39]. Most of the diffusion data available in literature are done at higher temperature with most on atmospheric nitrogen pressure on sample coupons as shown in Fig. 5.

![Figure 4. Nb-N Phase diagram, taken from Ref. [36]](image)

With the first demonstration of N-doping in SRF cavities, more attentions towards diffusion mechanism of nitrogen into niobium was given. The diffusion of the nitrogen in niobium usually starts with the formation of stoichiometric nitride phase on the surface and diffusion of the nitrogen in the bulk takes place. Depending on the temperature and amount of nitrogen being introduced on the surface of niobium, the diffusion of nitrogen can be few to hundred micrometers [40, 41, 42, 43]. The nitride features are clearly visible on the surface of niobium and those are identified as Nb$_2$N with TEM analysis [44] as well as XPS measurements [45, 46] consistent with
the earlier observations of sub nitride having a composition of hexagonal Nb$_2$N [47]. Tetragonal, a face-centered cubic, and two hexagonal nitrides having compositions of NbN$_{0.75}$ to NbN were also observed in earlier studies when nitrogen diffusion was done at atmospheric pressure and at higher temperature [48]. At the temperature of interest for SRF cavities, very small amount of nitrogen will diffuse to the bulk of the Nb and most forms nitride layers on the surface starting at temperature as low as 400 °C [49].

![Image](image.jpg)

**Figure 5.** (a) Reaction rate of nitrogen in atmospheric pressure [48]. (b) Nitrogen diffusion as a function from temperature [38].

There is very limited work been done on the crystal orientation dependence on the diffusion on nitrogen on SRF niobium [40,50]. Since niobium is a body centered cubic structure, which should not exhibit diffusion profile based on the crystal orientation. The doping study done cavities made from large grain niobium also showed the improve $Q$ as good as cavity made from fine grain niobium, if not better [9, 43,51,52]. In SRF cavities with large surface areas, the diffusion of nitrogen along the grain boundaries and weak dislocations networks is possible. No performance enhancement was observed on cavity made from low purity niobium, probably due to the higher concentration of impurities preventing the optimal diffusion of nitrogen in the bulk [43]. However, more systematic studies are still needed on the diffusion of nitrogen in niobium with respect to the temperature, partial pressure of nitrogen, duration of diffusion and the metallurgical state of niobium.

C. Post Doping Electropolishing

One of the required step after the nitrogen doping in order to achieve high quality factor is controlled electropolishing on inner surface of cavity. A recent study on single cell cavity also showed an encouraging result when the post doping material removal by buffer chemical polishing (BCP) [53]. Historically, the EP process is applied to achieve a smoother cavity surface and found to be superior to the BCP process and accelerating gradient can be enhanced with low temperature baking in UHV environment [54]. The standard EP acid mixture contains nine volume parts of sulphuric acid H$_2$SO$_4$ (96%) and 1 part of hydrofluoric acid HF (48%). EP is a surface finishing process whereby the anodization of Nb by H$_2$SO$_4$ forces the growth of Nb$_2$O$_5$ and F$^-$ dissolves Nb$_2$Os. A lot of progress was made in optimizing the electropolishing technique over the years.
The typical horizontal electropolishing systems consists of the cathode-anode set up with cavity being anode and aluminum cathode inserted along the cavity axis as shown in Fig. 6. The I-V characteristics, temperature of the EP mixtures, temperature of cavity surface plays the significant role in etching or polishing the niobium surface. The details on process development can be found in Ref. 59.

Figure 6. Schematic flow chart for a cavity electropolishing system [54].

The effect of amount of material removal on the performance of nitrogen doped cavities were studied in details [21-28]. It was found that the accelerating gradient increases while decreasing the overall quality factors as more material was removed from the cavity’s inner surface, especially in heavy doped SRF cavities (20N30 recipe) as shown in Fig. 7. This confirms the fact that some optimal nitrogen concentration is needed for high $Q_0$, however the gradient is limited to the lower values. Questions persist on the relationship between material removal, $Q_0$ and $E_{acc}$. How much material removal is necessary for optimal performance? It is believed that the early cavity quenches are due to the large segregation of nitrogen on localized site, driving the region into normal conducting state. Further, removal of material may reduce the localized site but the overall concentration of nitrogen is lower and superconducting properties approach the clean limit.
The effect of successive EP on single cell cavity \((B_p/E_{acc} = 4.23 \text{ mT}/(\text{MV/m}))\) with nitrogen doped with 20N30 recipe [51]. The accelerating gradient increase with increasing the EP removal, while decreasing the overall quality factor.

IV. Industrialization of Nitrogen Doping

The initial research and development on nitrogen doping was limited in Fermi Lab, Jefferson Lab and Cornell University. The research group working closely started the work in single cell cavity on recipe development changing the nitrogen diffusion time. Some details of the recipe development is already mentioned in section III, A. Several trials were done on the exposure and annealing time in order to identify the best recipe that met the LCLS-II cavity performance specifications. A recipe 2N6 (2 minutes nitrogen exposure followed by 6 minutes of annealing in UHV environment) was chosen to be used in the project and technology was transferred to the cavity manufacturers Research Instruments and Zanon. The electropolishing technology was transferred from Jefferson lab to the vendor sites.

The niobium for cavity fabrications was obtained from Tokyo Denkai and Ningxia with similar material specification as XFEL [60]. Total 373 cavities were ordered and tested at Jefferson Lab and at Fermi Lab showed an excellent performance with average accelerating gradient \(22.0 \pm 3.6 \text{ MV/m}\) with quality factor \((3.1\pm0.5)\times10^{10}\) as shown in Fig. 8 [61,62]. The successful production of several hundred cavities with nitrogen doping proved the reproducibility of the technological development within short period of time. Even though, the nitrogen doping was successfully applied to several multi cell cavities in mass production, several recipe modifications were made during the production [62]. Among them are; issue with electropolishing and annealing process of cavities prior to the nitrogen doping. The electropolishing issue were mitigated by the improved cathode and parameters with the help of Jefferson lab researchers [61].

† In literature, the recipe is usually defined as xNy, where x (1-30 mins) represents the nitrogen diffusion time and y (0-60 mins) represent the annealing time in minutes in UHV environment.
Figure 8. Summary of performance of LCLS-II cavities (~373 cavities) in vertical test on cavities commercially produced in two different vendor sites, from raw Nb materials from two different vendors. The cavities were doped with 2N6 recipe followed by 5-7 μm EP at vendor sites [61].

In order to achieve the high quality factor in SRF cavities, one has to minimize the trapped residual magnetic field during the cooldown of SRF cavities. It was found that high temperature gradient along the cavity is needed during the cooldown when the cavity surface temperature transition from normal conducting to the superconducting state (~9.2 K). This is needed in order to expel magnetic field from the SRF cavities due to Meissner effect, leading to the lower residual resistance due to trapped magnetic field [63,64,65,66]. Furthermore, these doped cavities are more vulnerable to flux pinning, extra care is needed to minimize any intrinsic source of pinning center. During the production of LCLS-II cavities, several cavities’ quality factor was limited by the poor flux expulsion. Even though, the material specification from different vendors were the same, the performance of the cavities made from the two vendors showed different flux expulsion. In the middle of the production, additional R&D was done in order to optimize the maximum flux expulsion with increasing annealing temperature prior to the doping process. The cavities annealed at higher temperature (900-975 °C) showed much better flux expulsion compared to 800 °C annealed cavities [62, 67]. Study also showed that the grain size may also plays the significant role in flux expulsion [67].

The rapid progress on development of SRF cavities with nitrogen doping was mainly due to the requirement for LCLS-II project. As a parts of R&D and possible use in future accelerator projects, nitrogen doping work were carried out in other part of the world, mainly in KEK Japan [31, 68], IHEP [69] and PKU in China [70,71]. Currently, the nitrogen doping technology is considered to be used in Shanghai Coherent Light Facility (SHINE) similar to LCLS-II project.

V. Towards High $Q_0$ and High Gradient

The nitrogen doping provided an avenue for the future accelerators operations due to the high quality factors, specifically, the CW accelerators which are operated with accelerating gradient of $\leq 20$ MV/m. Coincidently, most of the SRF cavities are limited at lower accelerating gradient $\leq 20$ MV/m much lower than what would have been achieved without doping. For example, the average accelerating gradient of 743 cavities used in XFEL projects was $31.4 \pm 6.8$ MV/m [72,73] with conventional (pre doping era) cavity processing recipe. During the LCLS-II cavity production, the average accelerating gradient of ~375 cavities was $22.0\pm3.6$ MV/m [62].
This shows ~ 30% reduction on achievable maximum accelerating gradient. However, the higher $Q_0$ at accelerating gradient $E_{\text{acc}} \sim 20$ MV/m, clearly shows the benefit of using doped cavities in CW machine.

Future accelerators such as International Linear Collider (ILC) will require high gradient and the project will benefit with high $Q_0$ SRF cavities [74]. Moreover the proposed upgrade of LCLS-HE program is also increasing its operation specification to higher gradient enable the need for further R&D in order to increase the accelerating gradient with high $Q_0$ SRF cavities.

A. High Temperature Nitrogen Doping

One of the route to increase the accelerating gradient currently being pursued is to use of the modified nitrogen doping recipe with varying the nitrogen diffusion and annealing time. Once again with the LCLS-HE project driven R&D conducted independently at Jefferson Lab and Fermi Lab are currently pursuing a different doping recipe. Jefferson Lab explored the doping recipe where the nitrogen was introduced in the furnace for short period of time (2-3 minutes) followed by longer annealing time (~60 mins). The hypothesis behind this recipe is that the nitrogen will diffuse deeper into the bulk with uniform concentration, leading to high accelerating gradient. Recipe applied to single cell cavities reached ~35 MV/m, however with the high field $Q$-slope [75]. Further optimization on doping infrastructure with capping cavities opening with Nb foils, smoother pre-doping surface and colder electropolishing led to initial results on 9-cell cavities accelerating gradient closer to 30 MV/m [62,67].

Fermi Lab explored the recipe different than that was being considered by Jefferson Lab in order to increase the accelerating gradient on doped cavities. The proposed recipe include the short nitrogen diffusion time (~2 mins) with no post annealing time. The preliminary results on single cell showed the accelerating gradient above 25 MV/m with high field $Q$-slope, similar to that has been observed with Jefferson Lab 3N60 recipe as shown in Fig. 9. With available 9-cell cavity test results, using 2N0 recipe, there is small increase in accelerating gradient (~ 3 MV/m) over the earlier 2N6 recipe [76]. Further R&D are being planned to increase the accelerating with high $Q_0$ in doped cavities in order to realization of LCLS high energy upgrade [77].

![Figure 9](image_url)

**Figure 9.** $Q_0(E_{\text{acc}})$ of single cell cavities with modified recipe (a) 2N0 and (b) 3N60, in order to achieve high $Q_0$, high gradient SRF cavities aim for LCLS-II-HE project [61].
B. Recipe Development at Low Temperature

Alternative to the nitrogen doping at high temperature followed by the electropolishing to control the optimal nitrogen concentration within the rf penetration depth was realized using nitrogen with the recipe involved at low temperature baking [10]. In these new nitrogen “infusion” cavity processing recipes, cavities were heat treated at 800 °C for ~3 hours and the furnace temperature is reduced to 120-300 °C. The nitrogen is introduced into the furnace at a partial pressure of ~ 25 mTorr for several hours (24-96 hours) in the temperature range of 120-200 °C. This process has shown an improvement in $Q_0$ over the baseline measurements, without the need for post-furnace electropolishing [11,12,78, 79,80,81,82]. The summary of single cell cavity results is shown in Fig. 10.

In particular at Jefferson lab, the benefits in nitrogen doping was observed when the nitrogen gas in injected into the furnace at elevated temperature (~ 250-300 °C) and let the cavity cooldown to lower temperature (120-200 °C) and hold at that temperature for extended period of times (24 - 48 hours) [12, 83]. Even though diffusion of the nitrogen into the bulk of the SRF cavity is limited in depth at these low temperatures (120-200 °C), the introduction of nitrogen is sufficient to modify the cavity surface within the rf penetration depth as seen from cavities’ test results, which are similar to those previously reported for high-temperature nitrogen doped cavities. The nitridation and diffusion of nitrogen into the bulk is expected when the Nb surface is free of Nb$_2$O$_5$ which occurs >300 °C [84,85], but the infusion was done when the cavity was fully annealed at 800 °C/3hrs and cooldown to desired temperature under UHV conditions before injecting nitrogen into the furnace. While post-doping electropolishing is required to remove coarse nitrides from the surfaces of high-temperature nitrogen doped cavities, no further processing is required for the low-temperature “infusion” recipe showing a clear benefit in reducing processing steps as well as keeping higher gradient with high $Q_0$ values. In some cases, the increase in accelerating gradient over the baseline was also reported, although the actual mechanism isn’t fully understood [10,83,86]. One of the critical requirement for these low temperature recipes is that special cleaning and preparation is needed before loading the cavity into the furnace. The cavity is high pressure rinsed (HPR) and then dried in an ISO 4/5 cleanroom. While in the cleanroom, special caps made from niobium foils were placed to cover the cavity flange openings. The cavity was then transported to the furnace inside a clean, sealed plastic bag. To this date, the recipe was successfully applied to single cell cavities by several research groups around world, but no clear advantage was demonstrated in multi cell cavities.

In the quest for high gradient, high quality factor SRF cavities, a two-step low temperature baking process showed an increase in accelerating gradient as well as quality factor well above 40 MV/m [87,88,89] in single cell cavities. Furthermore, the medium temperature baking also showed the improve quality factor with high accelerating gradient [90]. Future accelerating projects need the continuous research and development towards the high gradient, high quality factor of SRF cavities that can be reliably produced in large numbers.
Figure 10. The quality factor of single cell SRF cavities with nitrogen infusion, at (a) Fermi Lab [10], (b) Jefferson Lab [12], (c) Cornell University [11] and (d) KEK [80].

VI. Sample Coupon Studies on Doping and Infusion

SRF cavities are much bigger structure and study of sample coupons treated along with SRF cavities provided very useful information about the mechanism behind rf performance of SRF cavities. Several surface sensitive techniques are: Scanning electron microscope, Electron diffraction X-ray, Transmission electron microscopy, Secondary ion mass spectroscopy, X-ray photoelectron spectroscopy, Magnetometry and Tunneling.

A. Surface Morphology

Surface imaging with SEMs and also with EDX allowed looking into the surface of niobium after the nitrogen doping. Almost all high temperature doping recipe produces the triangular or star like shaped structures on the surfaces of niobium (Fig. 11). These structures are identified as normal conducting nitride, mainly $\beta$-Nb$_2$N [45], which are removed using electropolishing in order to achieve the high $Q_0$ in SRF cavities. It was found that the density and size of the normal conducting niobium nitrides depends on the temperature, duration of nitrogen doping and grain orientation of niobium [91].
Figure 11. (a) SEM image of nitrogen doped (2N6) Nb sample at 800 °C [53], (b) SEM and EDX image (inset) elemental map for NKα of nitrogen doped (20N30) sample at 800 °C [45] and (c) TEM image of Nb sample of nitrogen doped (20N0) at 800 °C with nano electron diffraction image of Nb, [113] zone axis (upper) and of Nb₂N close to [310] zone axis (lower) [44].

A. Secondary ion mass spectroscopy (SIMS)

SIMS has been an excellent tool in order to measure the elemental concentration on the surface of the nitrogen doped Nb samples. Ions beams, primarily \(^{16}\text{O}_2^+\), \(^{16}\text{O}^-\), \(^{133}\text{Cs}^+\) or \(\text{B}_{n}m^+\) \((n=1-5, m=1,2)\) are used to bombard on the sample surface at an energy that is enough to eject secondary ions from the surface of the Nb. The ejected ions are detected and analyzed by the mass spectrometers to infer the composition of the sample [92]. Concentration of nitrogen as a function of depth profiles and other impurities such as oxygen, hydrogen, carbon dioxides, and carbon monoxides are typically measured during the SIMS study depending on the detection limit of the instrumentation. It is to be remembered that the quality factor of SRF cavities depends on the elemental compositions (impurities) within the rf penetration depth. The additional rf losses can be anticipated due to the large segregation of impurities even deeper than the rf penetration, since those sites acts as the effective pinning centers for the residual magnetic field and thereby contributing to the vortex related rf loss [93]. The concentration profiles measured on nitrogen doping seems to follow the simulation results using Fick’s laws [42]. As expected, the variation on nitrogen concentration is observed depending on the nitrogen partial pressure [43], temperature of diffusion [50], and duration of nitrogen doping [61] as shown in Fig. 12.

Figure 12. Diffusion depth profile of nitrogen with respect to the (a) partial pressure of nitrogen [43], (b) diffusion temperature [50] and (c) duration of time [61].
SIMS measurements were also carried out samples treated in nitrogen environments at low temperatures and also with titanium doped samples [94]. The diffusion of nitrogen with in the rf penetration depth (~50 nm) was observed when the baking temperature was kept 150-200 °C [10,12, 45,95]. Some SIMS measurements suggested the increase concentration of carbon and oxygen is responsible for the increased Q when cavities were heat treated at low temperature [96]. In fact, SIMS studies showed higher concentration of oxygen and carbon as a result of heat treatments above 120 °C [8, 11, 53, 68, 94, 97].

B. X-ray photoelectron spectroscopy (XPS)

XPS studies are done in order to identify the near surface composition on SRF niobium. Historically there has been several XPS studies done in order to understand the loss mechanism of SRF cavities on EP, BCP and Low temperature baked SRF niobium [98,99]. XPS studies on those treatments reveals the complex oxide substructures (Nb2O5, NbO, NbO2, NbO1.x) within the rf penetration depth (~10 nm) are highly responsible to the rf performance of SRF cavities (Fig. 13) [12, 45, 53, 69].

Figure 13. (a) Nb 3d XPS spectra (takeoff angle of 75°) of N-doped (2N6) at 800 °C Nb samples before and after the ~6 μm surface removal with EP [53]. (b) Deconvoluted Nb 3d lines measured for the Nb samples after nitridation at 800 °C [45]. (c) Background subtracted XPS spectra on sample F9 (800 °C/3 hrs, no doping) and F11 (800 °C=3 hrs +140 °C/48 hrs at 25 mTorr N2) at 45° takeoff angle. And, (d) Angle resolved XPS on sample F11 (low temperature nitrogen infusion sample) [12].
C. DC Magnetization and AC Susceptibility

The superconducting properties such as the transition temperature and critical fields can be extracted from the dc magnetization and susceptibility measurements. In particular, dc magnetization can be used to estimate the field of first flux penetration \( (H_{ffp}) \), which is believed to be one of the cause of cavity quench. The measurement showed that \( H_{ffp} \) is lowered by \( \sim 15\% \) due to the nitrogen doping (Fig. 14) [100, 101,102] with no significant change in the bulk superconducting properties, confirming that the doping volume is smaller compared to the sample size.

**Figure 14.** Isothermal dc magnetic hysteresis of samples (a) several surface treatments including nitrogen doped and followed by EP [101]. (b) Sample with nitrogen infusion at low temperature [12]. About 15% reduction in first flux penetration was observed after nitrogen doped samples, whereas no significant change in \( H_{ffp} \) was observed in low temperature nitrogen infused samples.

**Figure 15.** Imaginary part, \( \chi' \), of dc magnetic field-swept-mode ac susceptibilities of nitrogen doped sample with initial surface (a) EP, (b) BCP, followed by EP [101]. And (c) the surface critical field of clean Nb (solid line), nitrogen doped Nb followed by EP and low temperature nitrogen infused Nb samples [103].
The ac susceptibility measurement technique has been used to study the pinning and vortex motions in many superconductors. The real part of the susceptibility is the result of the diamagnetic shielding of the magnetic field where as the imaginary parts give the information about the loss mechanism. The external magnetic field dependence of ac susceptibility can be used to extract the surface critical fields $H_{c1}$ and $H_{c2}$. In some geometrical condition, when the external magnetic field is parallel to the sample, the nucleation of the superconducting phase in a thin surface sheath was first discovered by Saint-James and de Gennes [104] up to surface critical field $H_{c3}$. The ratio $H_{c3}/H_{c2}$ was estimated to be $\sim 1.7$ using Ginzburg-Landau theory. It has been previously reported that the experimentally measured ratio is higher than theoretical number for several Nb samples [105, 106] and the ratio increases with decreasing the electronic mean free path. The ratio for nitrogen doped niobium was found to be closer to the theoretical limit [100,101], even though, the electronic mean free path was assumed to be low due to the nitrogen diffusion into the Nb surface.

D. Tunneling Measurements

The BCS surface resistance depends on many superconducting parameters and among them are the superconducting gap and density of states (DOS) of the quasi-particles at the Fermi level. The tunneling spectroscopy such as point contact tunneling (PCT) and scanning tunneling microscope (STM) are particularly useful in determining the DOS of quasi-particles, superconducting gaps, local distribution of the DOS and gaps as well as imagining vortices. PCT measurements done on nitrogen doped Nb showed the homogeneous distribution of the superconducting gap compared to non-doped samples and also the better surface oxidation states on surface [107]. The oxide structures is particularly beneficial in reducing the loss related to the two level system [108]. Recent STM measurements on cut out samples from nitrogen doped cavities showed the homogeneity of the superconducting gap with broadening of DOS [109] predicted for the dirty superconductors [110]. Furthermore, the vortex imaging with STM showed the slightly reduced but homogenous superconducting gap and lower coherence length compared to the traditionally prepared niobium cavities.

![Figure 16](image.png)

Figure 16. (a) Temperature dependence of $\Delta$ for typical tunnel junction measured using point contact tunneling (PCT) shown in the insert [107]. (b) Scanning tunneling spectrum (dots) and BCS-Dynes fit (red line) acquired on Nb and N-doped Nb cavity cutout samples, respectively, at T=1.5 K [109].
VII. Theoretical Models

As mentioned earlier, the increase in quality factor can be realized by minimizing the surface resistance, either residual or BCS or both residual and BCS resistance. Statistically, it is found that the residual resistance of doped SRF cavities is lower than non-doped counterpart tested in the low residual magnetic field environment. It is to be noted that the residual magnetic field has a severe effect on doped cavities compared to non-doped cavities [66,111,112, 113]. Historically, it is believed that the hydrogen in niobium is responsible for higher residual resistance due to the precipitation of hydrogen forming normal conducting hydrides with in the rf penetration depth [114,115,116,117]. Some improvement in quality factor at low accelerating gradient and a significant improvement in quality factor with increased accelerating gradient was observed when cavities were baked at low temperature. Even though no unanimous theory or model exist at this time, several evidence shows that the impurities (O and H) as well as their interactions with vacancies play a role in order to minimize the rf loss in SRF cavities due to low temperature baking [118,119,120,121,122]. The presence of nitrogen (or even titanium) traps the hydrogen, preventing the formation of lossy hydrides probably leading to the lower residual resistance in doped SRF cavities [123,124,125, 126, 127,128].

One of the peculiar observations in doped cavity is the increase in quality factor as a function of the microwave field tangential to the inner surface of cavity. This means that the surface resistance decreases with the rf field, contrary to perception that the microwave field suppressed the superconductivity leading to the decrease in quality factor $Q_0$ with accelerating gradient $E_{acc} \propto H_{rf}$ [129]. The quality factor of doped SRF cavities increase with the increase in rf field up to $H_{rf} \sim 100$ mT and is then limited by either quenched or by high field $Q$-slope, typically observed in non-doped BCP or EP cavities.

The original BCS theory was developed without taking in to account of the amplitude of rf field and this theory explains well the temperature dependence of surface resistance at low rf field [130,131]. The theory was extended to explain the high field $Q$-slope in SRF cavities, which may be related to the non-linear BCS resistance [132,133]. Soon after the first demonstration of $Q$-rise phenomena after the titanium doping in SRF cavities [8], the BCS theory was extended taking into account of the moving Cooper pairs in the presence of rf field cavities [134]. The calculation reproduced the rf field dependence surface resistance of titanium doped cavity and consequently the nitrogen doped cavities [135].

Gurevich [110] proposed that microwave suppression of surface resistance comes from the current-induced broadening of the quasiparticle density of states. The BCS surface resistance takes the logarithmic dependence with the microwave field in dirty limit, as experimentally observed in Ti [136] and nitrogen doped cavity [51,137,138, 139]. The microwave suppression of surface resistance was also previously observed in thin films and explained due to the non-equilibrium quasi-particle distribution function leading to the enhancement in supercurrent [140]. A theoretical model in which the surface resistance of a superconductor coated with a thin normal metal was recently presented and showed that the $R_s(B_p)$ behavior observed in SRF cavities following different surface preparations can be explained with changes in the thickness of the normal layer and of the interface boundary resistance [141]. A recent theoretical model proposed by Gurevich extends the zero-field BCS surface resistance to high rf fields, in the dirty limit [142]. Such a model calculates $R_s(H)$ from the nonlinear quasiparticle conductivity $\sigma_1(H)$, which requires knowledge of the quasiparticles’ distribution function. The calculation was done for two cases, one which assumes the equilibrium Fermi-Dirac distribution function and one for a non-
equilibrium frozen density of quasiparticles. A non-equilibrium distribution function is appropriate when the rf period is shorter than the quasiparticles’ relaxation time. $R_s(H)$ is calculated numerically for these two cases and it depends on a single parameter, $\alpha$, which is related to the heat transfer across the cavity wall, the Nb-He interface and between quasiparticles and phonons. The theory explained well the microwave surface resistance in cavities with low temperature nitrogen infusion cavities [12].

![Figure 17](image)

**Figure 17.** Normalized surface resistance as a function of peak surface magnetic field on cavity surface (a) Ti-doped cavity [110] and (b) Nitrogen infused srf cavities [12], the solid lines are calculated using the model described in ref. [142].

Several other models are reported in order to understand the microwave resistance in SRF cavities extending the theory to clean, dirty, equilibrium and non-equilibrium superconductors [143,144,145,146,147]. The nitrogen doping and infusion studies were also extended to different frequency cavities [148,149], showing that the frequency might play the role in the field dependence of microwave surface resistance [146]. In addition, two-fluid models taking into account the surface impurities on SRF cavities were extended in order to fit the field dependent surface resistance in order to extract the superconducting parameters responsible for the reduced rf loss in doped SRF cavities [150,151].

**VIII. Summary and Future Outlook**

Engineering the concentration of nitrogen within the rf penetration depth in SRF cavities have been extremely beneficial in order to achieve the high quality factor, potentially cost saving on current and future SRF based accelerators. SRF cavities with high quality factor ($Q_0>3\times10^{10}$) at 2.0 K in accelerating gradient $\sim 25$ MV/m has been successfully produced commercially and is in process of installation in LCLS-II. Efforts are being made in order to extend the high quality factors towards higher accelerating gradient with modification of existing recipe as well as exploring the new recipes. High $Q_0$ and high gradient SRF cavities would be of great interest for lowering the cryogenic heat load of future high-energy accelerators.

High quality factor SRF cavities are also drawing interest in several other applications. For example, the quantum computing with microwave resonators [152,153,154] and axion dark matter research [155]. Extremely high quality factor with decay time of the order of few seconds were
measured in SRF cavities in quantum temperature limit [156]. In addition to Nb cavities, the alternative materials Nb$_3$Sn are being explored in order to achieve high quality factor, however so far the accelerating gradient are limited to below 20 MV/m [157,158,159]. Thin Nb$_3$Sn cavities are fabricated using thermal diffusion of Sn on Nb cavities, potential for industrial applications [160]. For high accelerating gradient cavities, multilayers of superconducting are being considered [161,162].

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