# EVALUATION OF SURFACE CONTAMINATION AND CLEANING TECHNIQUES ON SUPERCONDUCTING RF CAVITIES*

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## Abstract

Surface contamination is a major factor influencing field performance in superconducting rf cavities. Present processing techniques used to clean niobium surfaces leave substantial particulate densities in the 20 to 100 µm range. A capability to quantify contamination from cavity-cleaning processes has been established using optical and SEM microscopy on niobium samples which can be run in parallel to cavity-processing steps. Laser surface scanning has also been used on silicon samples to quantify noncorrosive steps. Niobium cavities at 3 GHz and 805 MHz processed with various cleaning procedures have obtained peak surface electric fields as high as 78 and 44 MV/m, respectively. Results from the evaluations and the cavity tests will be presented.

## Introduction

Superconducting cavities typically have two characteristic behaviors which limit performance: quenching and field emission. Quenching at low field levels was minimized by using higher RRR (>250) niobium to thermally stabilize hot spots. Today, field emission is the major limit to performance in superconducting rf cavities.

Studies of field emission at Cornell[1] indicate that particulates on the surface play a role in limiting cavity performance. Extensive SEM/EDS microscopy of emitter sites indicates that cavity fields strongly interact with particulates, leading to vaporized contaminants, melted parent material, and arc-damaged zones.

Cavity performance tests at 3 GHz at Los Alamos show data distributions with a deviation of ± 26% about the mean. Particulates are felt to play a role in establishing the deviations, as shown in Figures 1 and 2. Evaluating the magnitude of the contribution is one aim of the surface contamination work. Data has been taken with single-cell cavities at 805 and 3000 MHz.

In an attempt to improve cavity performance and decrease deviation, work has been done to characterize and better control surface and bulk contaminants. These studies are being used to develop an improved quality assurance plan to enhance reliability and consistency.

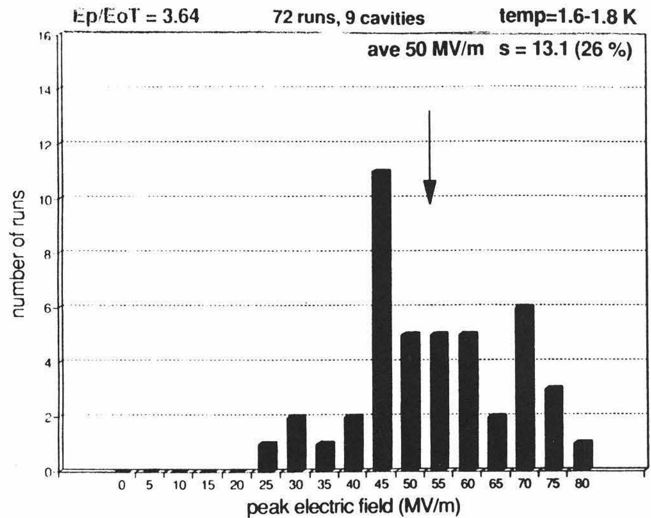


Fig. 1 3000 MHz performance distribution for nitric+111 chemistry

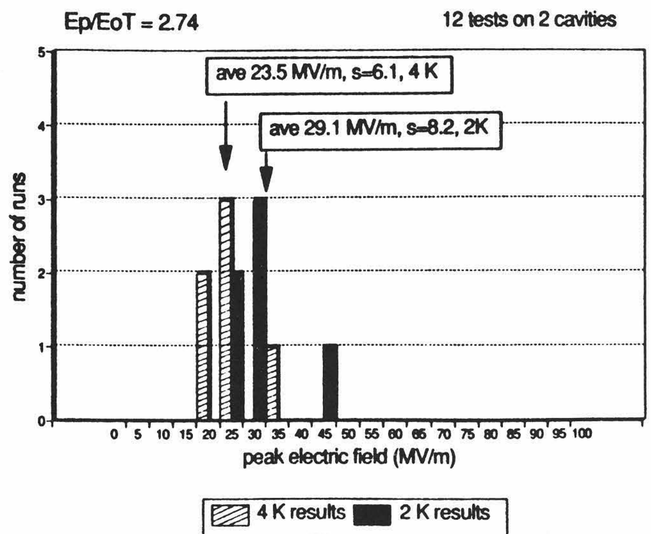


Fig. 2 805 MHz performance distribution for nitric+111 chemistry.

## Surface Contaminant Evaluation and Cleaning

Surface contamination studies were done in collaboration with the Microelectronics Development Laboratory at Sandia National Laboratory. Their experience with microelectronics fabrication in a class 1 environment was invaluable. Initial work focused on evaluation of contamination from the "standard" chemical treatment. This was done in two ways. First, blank silicon wafers were exposed to noncorrosive processing steps such as rinsing and drying, then analyzed with a laser surface scanner, similar to work done at Saclay[2]. This approach was very useful, but had two drawbacks: the surface scanner gave an integrated result for particles 5 µm and larger and 111 bcp aggressively etches silicon, so that the scanner no longer works.

Since etching is a significant cleaning process, blanks of niobium were used to measure particulates coming from blocks of processing steps, e.g, etching, short rinsing, and long rinsing. On etched niobium, the surface topography turned particle counting on the SEM into an extremely

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turned particle counting on the SEM into an extremely tedious exercise. Particle counts obtained for niobium, then, are only indications of particle densities. Even so, the levels are on the order of a particle per square centimeter, which would provide significant emission sites. This is taken as a significantly high count for particles above 5 µm, because silicon-wafer cleaning technology gives densities typically less than 0.02 particles/cm$^2$ greater than 5 µm.

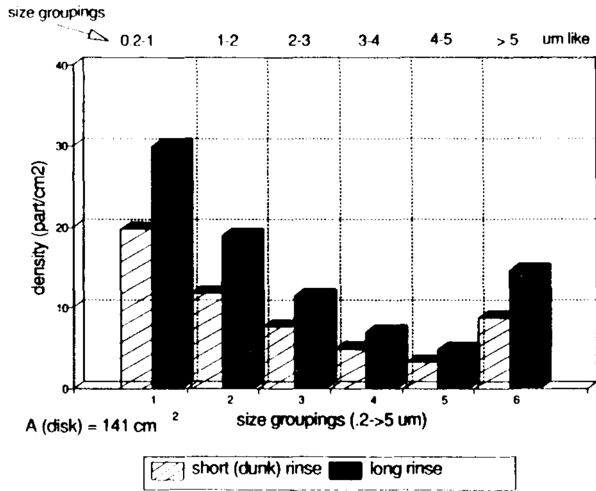


Fig. 3 Laser surfscan of particulates on silicon obtained from chemistry room, and the effect of short and long rinse on removing the dirt.

As shown in Figure 4, significant particle densities remain after a "standard" chemical etch treatment. The sources for these particles are many. Analytical grade chemicals abound in particulates [3]. The area where we do chemistry is a normal "dirty" room, with more than $10^6$ particles per cubic foot. Containers are wiped with regular wipers and ethanol. The data shows that processing through etching is dirty, and that taking the cavity into a clean room and rinsing it with water does not reduce particulate levels.

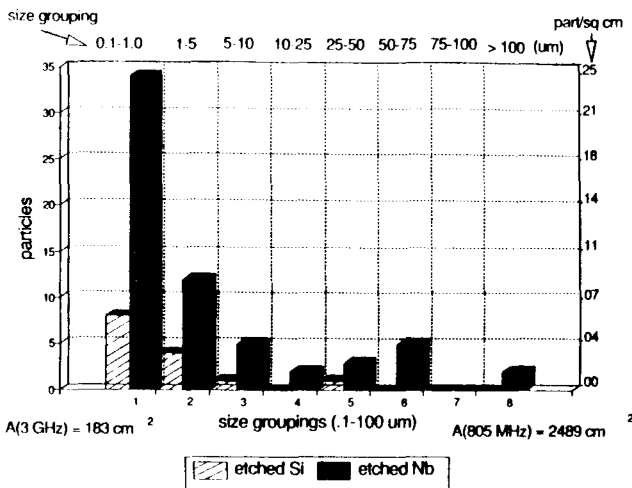


Fig. 4 SEM microscopy of particles on niobium from standard bcp chemistry and long rinse.

The particle densities indicated are for fairly large particles (>5 µm) from a microcontamination point of view. Particles of this size can be cleaned by various techniques including ultrasound, megasound, surfactant sprays, lower pressure gas and liquid jets, and wiping [4]. This is encouraging, because many of these techniques are not extremely high cost or high tech, but they must be done properly.

## Cleaner Chemistry

Two experiments were performed to investigate how cleaning up the chemical processing would influence cavity performance. In the first, a HEPA module was installed over our chemistry area, as a mini-clean room. Analytical grade chemicals were used. The results of eight tests were not significantly higher than the standard distribution, as shown in Figure 5. This could be attributed to the particulate levels present in the analytical grade chemicals.

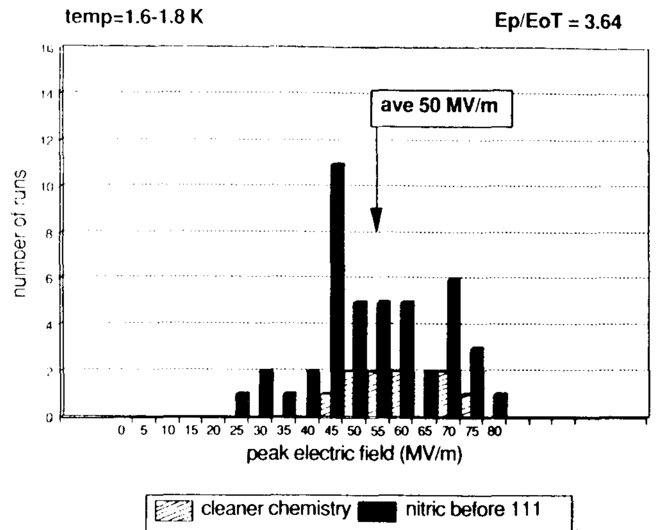


Fig. 5 Distribution showing clean chemistry results.

The second test involved doing ultraclean chemistry and assembly, again at Sandia National Lab. In this test, two cavities were etched in nitric acid, then 111 bcp, both filtered to 0.5 µm. They were rinsed for 60 hours in ultrapure water, then assembled in a class 1 bay. These cavities also performed within the distribution, as shown in Figure 5.

Samples were run in parallel with the cavity processing. Optical microscopy on these samples did not show particulate matter as was seen on samples taken after unclean chemistry, but light scattering from surface topography precluded obtaining meaningful density values using this approach.

Even though the statistics of the tests are small, these results indicate that though particulates certainly affect performance at a certain level, they may not be the only limitation to cavity performance. Of the 25 tests at more than 50 MV/m, 36% were quench limited, supporting the idea that other limiting mechanisms are occurring. If particulates were a major factor in limiting cavity

performance, the cavities cleaned at Sandia should have performed significantly better than our distribution, because the particulate densities in the processing chemicals were lower by at least a factor of 100 than what is typically used. The fact that no electrons were observed in either test also supports this idea.

Work supplementing the aforementioned tests is being done with cavity cleaning techniques involving high-pressure spray rinse, surfactants, and sonics. These are being quantified with liquid-borne particle metrology.

### Fabrication Control

Investigation of new cavity fabrication on 3 GHz cavities has shown that removal of approximately 120 µm of material per side is required before the cavities perform optimally. A study of performance as a function of removal on 10 cavities indicates poorer performance between 20 and 100 µm removal and consistently higher performance above 100 µm as shown in Figure 6. This is in contrast to the opinions expressed by the TESLA superconducting technology working group at the 5th Workshop on RF Superconductivity, which held that 60 µm removal is sufficient for optimum cavity performance.

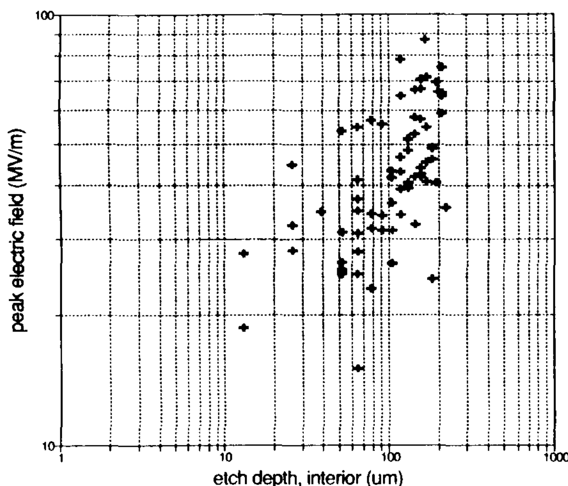


Fig. 6 3 GHz cavity performance as a function of material removed per side.

To account for the difference in removal, various analytical techniques have been used. Fixed thermometers on the cavities indicated heating in the equator-weld region of the cavity. This heating was the dominant limiting mechanism in a new set of cavities, where less than 100 µm removed. Analysis was done using SEM/EDS, SIMS, x-ray crystallography, Auger, and metallography, looking for obvious signs of impurities. The results so far are not obvious. Metallography has shown a large number of tiny pits throughout the bulk material. The density of these pits is enhanced by a factor of 3 in the weld melt region to a depth of around 100 µm. The nature of these pits and what caused them have yet to be determined. SEM analysis shows the pits are empty, indicating that either whatever was there was etched away or they are gas pockets.

The disparity in etch removal, combined with a desire to reduce the performance deviation and better understand the factors that contribute to the deviation has led us to institue a broad quality assurance plan to check, document, and control cavity fabrication from material purchase to cavity retirement. The goal of this program is to achieve a high level of consistency in cavity production and establish a detailed record of procedures followed, to better understand the factors that influence cavity performance. This will lead to a clear set of guidelines for production cavity work. In this way, we are building on work done by labs which are presently in production mode, in the hope of enhancing our procedures to give higher production gradients in the future.

### Conclusion

Cavity test results are yielding good gradients. In chemically treated 3 GHz cavities, peak fields as high as 78 MV/m have been seen. In chemically treated 805 MHz cavities, peak fields up to 44 MV/m have been observed. Testing continues to establish statistics.

Particulate contamination is a recognized problem in superconducting cavities. Measurements show large particle densities above 5 µm, compared to microelectronics-clean silicon. Experiments in cleaner chemistry do not show drastic improvements in performance, indicating that cavity performance is only partially affected by particulates in this size range. Since particulates in this size range are fairly easily removed by established cleaning technologies, work is continuing to enhance understanding. These results are prompting further investigation into the role of cleaning in performance, with emphasis on high-pressure rinsing, sonics, and surfactants. Additional surface analysis and evaluation are also planned.

To better control cavity production, processing, and testing, an improved quality assurance plan is being instituted. The goal of this plan is to more thoroughly document procedures to better understand the factors that affect cavity performance.

### References

[1] D. Moffat et al., Proc 5th Workshop on RF Superconductivity, DESY, Hamburg, Germany, August, 1991, v. 1, pp 245-284.

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[4] ibid. pp. 329-338.