Power Spectral Analysis of Niobium Surfaces

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Abstract

Experiments at Jefferson Lab's Continuous Electron Beam Accelerator Facility (CE-BAF) have demonstrated that the performance of superconducting Niobium accelerators is limited by the roughness of the interior of the SRF cavities that power these accelerators. Attempts to improve the smoothness of these surfaces has created the necessity for a standardized metric by which both the size and shape of surface features may be recorded. We employ power spectral analysis in an attempt to achieve these goals.

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1 Background

Superconducting Radio Frequency (SRF) technologies are increasingly being used in linear accelerator applications that require a large, continuous accelerating field. Niobium SRF cavities offer distinct advantages over their non-superconducting copper counterparts by virtue of their lower surface resistance, which allows for long (continuous) RF pulses, high quality factors, and accelerating fields measuring in the range of millions of volts per meter (MVm^{-1}) . Improved gradients and quality factors permit the construction of more powerful accelerators in less space, and at a lower cost. Since the construction of the Continuous Electron Beam Accelerator Facility (CEBAF) at Jefferson Lab in 1995, SRF has emerged as the favored technology for large linear accelerators. Most notably, the proposed International Linear Collider (ILC) project aims to construct a 500GeV accelerator using SRF technology.[4]. To meet this ambitious goal within financial and logistical constraints, the performance of SRF cavities must be improved on several fronts.

The performance of an SRF cavity is most commonly described in terms of its quality factor, Q_0 , and its accelerating gradient, E_{acc} . Q_0 is the ratio of energy stored in an accelerating cavity to the energy dissipated by its walls, which can be effectively thought of as the cavity's overall efficiency.[9] This dissipated energy is released as heat, which must be removed by the accelerator's cryosystem so that the cavities remain in the superconducting state, at approximately 2 Kelvin[4]. Given the high costs associated with maintaining a 2K cryosystem, it becomes imperative to maximize this quality factor.

The accelerating gradient indicates the distance necessary to accelerate the particles by a given amount of energy; improving this gradient permits more powerful accelerators to be constructed over shorter distances, and (for our purposes) is most frequently given in terms of mega-volts per meter $(MV \cdot m^{-1})$. The SRF cavities currently operating at CEBAF at JLab currently produce an accelerating gradient of 6 $MV \cdot m^{-1}$, a modest improvement over the original design gradient of 4 $MV \cdot m^{-1}$ [2]. To facilitate the ILC's ambitious design goals, two 12km linear accelerators consisting of 8,000 each, with an accelerating gradient of approx. 35 $MV \cdot m^{-1}$ will be required[4]. Attempts to improve this accelerating gradient have met with some success, and accelerating gradients of 25-40 $MV \cdot m^{-1}$ have been demonstrated in a laboratory setting.[12]

As we attempt to push the accelerating gradient of our cavities higher and higher, we observe the decline of the quality factor of these cavities, before a critical point is reached, indicated by a rapid decline of Q_0 . Given our definition of Q_0 , we can infer that more heat is being dissipated through the walls of the cavity as we attempt to increase E_{acc} . Although superconductors have no measurable resistance, RF currents are still subject to a nonzero surface resistance due to the inertia of superconducting Cooper pairs.[11] Increased heat dissipation reduces the fraction of charge carriers bound as Cooper pairs, reducing the performance of the superconductor. In extreme cases, localized areas of the cavity will heat beyond the superconductor's critical temperature, returning the metal to its normal conducting state.[12]

SRF has been shown to primarily be a surface-bound phenomenon, as the penetration depth of the RF power has been measured to be approximately 40nm, indicating that cavity performance is primarily dependent upon material characteristics very close to the surface[10]. As a result, the interior surface of the cavities must resemble the ideal resonant structure as much as possible. Deviations from this structure, (ie. grain boundaries, surface defects, welds, pits, mechanical damage, etc.) no matter how small will cause the RF power to be dissipated to the body of the cavity, sometimes producing in an observable hot-spot on the exterior of the cavity. Although smaller defects might not produce an observable hot-spot, we can theorize that even extremely small defects will adversely affect the overall performance of the cavity by reducing the fraction of Cooper pairs within the material. Improvements in the manufacture and handling of cavities to reduce damage and contamination have already yielded promising results.

Researchers at Jefferson Lab have explored various techniques to polish of these Niobium cavities, to remove as many surface defects as possible. Buffered Chemical Polishing (BCP) is used to remove bulk surface defects and mechanical damage remaining from the fabrication and initial mechanical polishing of the cavities via a mixture of HNO_3 , HF, and H_3PO_4 that is washed through the cavity. Although this technique is commonly used to remove large

defects, it etches pits and other surface features of its own, as we can easily see in the optical micrograph below. As we've noted that the optimal cavity surface must be smooth to within nanometers, any surface features visible at optical wavelengths (> $0.2\mu m$) are undesirable. Electropolishing (EP) has been proposed as an additional step to remove these surface features by circulating HF and H₂SO₄ through the cavity, while applying a voltage of 10-20V. [15]



(a) BCP-Polished Niobium



(b) Electropolished Niobium

Figure 1: Comparison of optical micrographs of BCP and EP-polished surfaces. Images are approx. $800 \mu m$ across.

Although we can subjectively observe that electropolishing more effectively removes surface features present by observing optical micrographs, we must develop a method by which we can quantitatively characterize the smoothness of these surfaces, particularly with regard to surface features that are too small to be resolved with optical methods. Additionally, as these polishing processes can be adjusted and optimized, we would like to develop a diagnostic to test the effectiveness of a particular polishing routine *prior* to the construction of a cavity, given that cavity construction and testing is an expensive and lengthy process.

2 Surface Metrology

To evaluate the effectiveness of the polishing processes, we must collect data about the surface topography of our niobium samples after they have been subjected to a polishing regimen. Because we are interested in surface features over a wide range of dimensions, we must employ a variety of methods to collect topographical data.

As demonstrated in the previous section, optical microscopy is used when possible to develop an intuitive understanding of the evolution of the polishing process, but is fundamentally unable to reveal details smaller than 0.2μ m, which are of particular interest to us when evaluating electropolished surfaces. Furthermore, optical micrographs are unable to provide numeric topographic data to help us quantitatively evaluate surfaces. However, optical micrographs can be produced considerably more rapidly and inexpensively than any other method described in this paper, and will quickly reveal any major defects or damage on the surface to the examining technician.

To develop a true topographic model of the surface, we must resort to Scanning Probe Microscopy (SPM), which directly collects numeric data about the two or three-dimensional surface of our sample. In particular, we will employ Stylus Profilometry and Atomic Force Microscopy to collect topographic data.

Stylus Profilometry (SP) is able to produce a two-dimensional surface "profile" of a surface by dragging a stylus across the surface, and measuring the deflection of the stylus, producing a two-dimensional surface profile. The KLA Tencor P-15 profilometer available at Jefferson Lab is capable of collecting scans up to $1000\mu m$ in length, with lateral resolution limited primarily by the $2\mu m$ radius of the stylus tip. In our experience, technique has proved to be fast and reliable. Because the stylus is in direct contact with the surface, physical damage can potentially occur as a result of the measurements being taken.

Atomic Force Microscopy (AFM) is conceptually similar, but offers improved resolution by positioning a microscale probe/tip just above the surface of the sample, and measuring the deflection of the probe due to atomic forces between the probe and the surface being measured. Through the use of a piezoelectric positioning system, AFM can very accurately scan the tip over a designated area, and construct a true 3-dimensional profile of a surface.[13] However, as these piezoelectric devices can only operate over a very small range, the largest area that our AFM is able to scan is on the order of $100 \times 100 \mu$ m, although the instrument is frequently used to scan much smaller areas. Like SP, the lateral resolution of surface features able to be resolved is limited by the radius of the tip being used; the tips used by our lab have an advertised radius of less than 10nm, and a maximum height of $17 \pm 2\mu m$ [1]. The height of the tip limits the maximum peak-to-valley distance that can be measured in a single scan, which can cause difficulty when measuring across a large area on a rough surface. Although the improved resolution of the AFM is desirable, data collection is time-consuming, and requires an experienced technician; ideally, we would like to limit our reliance upon AFM data as much as possible.



Figure 2: 3-Dimensional view of an AFM image of BCP-Polished niobium. Scanned area measures $20\mu m \ge 20\mu m$.

White Light Interferometry has been used[6] as an additional method for obtaining surface profiles, and is capable of rapidly recording 3-dimensional profiles with a vertical resolution of <0.1nm, and a lateral resolution of approximately 0.5μ m.[3] Although this method shows promise, particularly for its ability to capture 3-dimensional profiles much more rapidly than by AFM, we were unable to obtain reliable access to one of these devices or obtain a sufficiently large set of data to draw any meaningful conclusions. We suggest that these techniques be explored in the future.

3 Measurement & Analysis

Because we are interested in identifying and measuring surface features across a wide range of sizes, it becomes necessary to incorporate measurements from several types of instrumentation. The most common measurement of the smoothness of a given surface is the roughness parameter, frequently given following the form of:

$$R_{a} = \frac{1}{n} \sum_{i=1}^{n} |y_{i}| \tag{1}$$

$$R_q = \sqrt{\frac{1}{n} \sum_{i=1}^n y_i^2} \tag{2}$$

where R_a is the arithmetic average of absolute values of the distance of each sampled point above/below the mean height of the surface, while R_q is the root mean squared average of these values. Numerous other roughness parameters exist. Although this measurement is adequate for comparisons between measurements taken by similar instrumentation, it cannot be used to compare data between different types of instrumentation.[7] A surface may appear smooth to an instrument with a low lateral resolution, but may still contain small surface defects that can only be detected by an instrument with a higher lateral resolution. In this situation, one instrument would measure a small roughness parameter, while the other would measure a large roughness parameter on the same surface.

To reconcile this gap, we must develop a standard for analysis that is able to compensate for spatial resolution of the instrumentation being used to measure the surface profile. For this purpose, we propose the Power Spectral Density (PSD) as a means to determine the presence of surface features based upon the size of those features (or more specifically, their spatial frequency), and to reveal any hidden periodicities of features on the surface. To estimate the Power Spectral Density from a set of discrete data points, we employ what is known as the Periodogram method for spectral estimation[14]:

$$\hat{\phi}_p(\omega) = \frac{1}{N} \left| \sum_{t=1}^{N-1} z(t) e^{-i\omega t} \right|^2 \tag{3}$$

In this instance, ω is equal to angular frequency, N is the number of points being sampled in our signal, and z(t) is our signal itself. This may be more easily recognized as the square of the Fourier transform of a given signal. In practice, these calculations are computed using a Fast Fourier Transform (FFT). Because we are computing the PSD from surface profile data, rather than a time-variant signal, we must rewrite this formula in the spatial domain[5]:

$$\hat{\phi}_p(m) = \frac{\Delta x}{N} \left| \sum_{n=1}^{N-1} z(n) e^{-i2\pi n m/N} \right|^2$$

In this instance, Δx is our sampling resolution, and *n* has replaced *t* as the indexing variable. For a more in-depth derivation and analysis, see [5, 14]. Through this technique, we are provided with information about both the lateral size (spatial frequency) and height (power) of surface features. In practice, these calculations are performed via a Fast Fourier Transform (FFT). Previous attempts to apply PSD-based analysis of Niobium surfaces by H. Tian, et al. have employed the periodogram method for their calculations [8].

Although this method appears ideal, providing us with the information that we are interested in, Stoica and Moses's analysis[14] indicates that the the PSD estimates produced by this method have a high statistical variance that does not decrease with large values of N. High variability makes it difficult to extract meaningful information from a PSD estimate, making comparisons between the PSD estimates of two samples difficult. Although this statistical variance can be reduced by averaging the PSDs of several surface profiles, we would like to limit the amount of data necessary to perform our analysis.

Specific care must be taken when considering PSD estimations produced from scanningprobe instruments. Although the spectral range of our PSD estimate will be determined by the sampling length and resolution of the instrument, the radius of the probe itself may be larger than the instrument's sampling resolution, producing inaccurate or incomplete PSD estimates at high spatial frequencies.

3.1 Improved PSD Calculation Methods

To produce a "smoother" PSD estimate that is more representative of actual surface features, we employ Bartlett's method for power spectral estimation. Bartlett's method modifies the periodogram method by splitting our signal into several segments of L points each, calculating the periodogram of each segment, $\hat{\phi}_i$, and averaging the results. [14]

$$\hat{\phi}_B(\nu) = \frac{1}{L} \sum_{j=1}^{L} \hat{\phi}_j(\nu)$$
(4)

Although the Bartlett method reduces the spatial frequency resolution of the PSD estimate by reducing the Nyquist limits of our signal, it offers a dramatically smoother PSD estimate that can more readily be used to identify surface features, or more importantly, be compared to PSD estimates of other surfaces. Unlike an unaveraged periodogram, this method allows us to improve the quality of our results by simply recording more data points per surface profile, rather than recording several individual profiles. Surface Profilometry methods particularly benefit from this, as it is often trivial to increase the length of a surface profile scan, with modern profilometers able to scan across lateral distances far greater than the size of our polished Niobium samples. Stoica and Moses indicate that though this method does decrease statistical variance, a bias error may be introduced.

P.D. Welch proposes two improvements to Bartlett's method, in which a windowing function is applied to each segment of data, and the segments are allowed to overlap[16]. Although most common windowing functions, including the Hamming window that we use for our calculations, give more weight to data points near the center of each segment[14], the fact that the segments are allowed to overlap will mitigate these effects, provided that a sufficiently large overlap is selected. These optimizations further reduce the statistical variance of the PSD estimate over the Bartlett method, without sacrificing any additional spatial resolution. For these reasons, the Welch method is one of the most frequently-used PSD estimation methods, and has gained some acceptance in the optics community for use in surface metrology.[5]

To demonstrate the smoothing characteristics of the Welch PSD, we take a single pro-

filometry trace from a polycrystalline mechano-chemical polished niobium sample, and compute the PSD via the periodogram method and the Welch method, along the same axes:



Figure 3: Comparison of Welch and periodogram methods on a single surface profile.

In the above figure, we use a 250 point window, with a 125 point overlap to generate the Welch PSD from a 1,000 point surface profile. As we can clearly observe from the graph, the Welch method generates a smoother PSD estimate, particularly at lower frequencies. Although we are concerned about the potential loss of information when using the Welch method, the extremely high variance of the periodogram makes it difficult to distinguish actual surface features from statistical noise.

The advantages of the Welch method are not initially as clear for use on AFM data, as we treat these scans as 512 individual surface profiles. The variance inherent in the periodogram method should be greatly reduced by averaging of 512 individual periodograms, which we do indeed observe. However, as we also observe in the plot below, there is a considerable bias gap between our PSD estimates. Although this discrepancy appears great enough to be the result of a bad dataset, additional AFM measurements from the same sample produced similar results. To produce this PSD, we use a 200 point window, with a 100-point overlap:



Figure 4: Comparison of Welch and periodogram methods on 512-line AFM scan

To determine which PSD estimate is more likely to be representative of the actual surface, we combine the data from the previous two figures, and observe that the Welch PSD estimates from both instruments align with each other, indicating a continuous spectrum of frequencies, as we'd expect to observe on a physical surface in nature. In light of this alignment, we can conclude that a large bias error must be present in the periodogram of the AFM scan. Although we present a somewhat extreme example, previous studies have indeed noted the disparity between PSD estimates produced by different instrumentation, although there are also instances in which the averaged periodogram method has produced acceptable results.[15]



Figure 5: Comparison of periodogram and Welch method for AFM and SP data

As shown in the above figure, Welch's method of power spectral estimation enables measurements from SP and AFM to align, and produce a continuous spectrum of spatial frequencies, whilst producing a sufficiently smooth PSD estimate to enable meaningful comparisons between the datasets. Although we observe that the Welch PSD estimates from the profilometer and AFM do not perfectly overlap for frequencies above $1\mu m^{-1}$, this discrepancy is to be expected, and may be explained by noting that profilometer's stylus has a radius of approximately $2\mu m$. As previously mentioned, the profilometer cannot accurately resolve surface features smaller smaller than the radius of the stylus itself.

3.2 Optimization of the Welch Method

As we have previously mentioned, the Welch method provides us with two adjustable parameters: the size of the window, and the amount by which the windows are allowed to overlap. Selection of these parameters requires a compromise between spatial resolution and the smoothness of the PSD. We will compare the Welch PSDs of a 1000-point surface profile, with 1000, 500, 250, 100, 50, and 10 point windows, with an overlap of 0%, 25%, 50%, and 75%. Because this analysis is highly subjective, we present our complete set of data. For the purposes of comparison, the unaveraged periodogram (Figure 7A) is also included on each plot, colored light gray.



Figure 6: Welch PSD with windows overlapping by 0%



Figure 7: Welch PSD with windows overlapping by 25%



Figure 8: Welch PSD with windows overlapping by 50%



Figure 9: Welch PSD with windows overlapping by 75%

Unfortunately, Stoica and Moses note that a numerical or analytical analysis of the variance of the Welch spectral estimator is not possible, except for a small number of specially-constructed cases[14], requiring us to rely upon an empirical observation of the Welch estimates. Similarly, because have no alternate means of determining the frequency content of our surfaces, no meaningful statistical analysis can be performed, requiring us to subjectively select the parameters that produce the PSD estimate containing the most usable information.

Our observations indicate that the 500-point window with a 75% overlap provides the best compromise of smoothness and resolution for this sample. This is a promising result, as it indicates that we are able to produce a reasonably smooth PSD without having to sacrifice much resolution by allowing our segments to overlap. As we would expect, the resolution of the PSD is visibly reduced as we constrict the length of the segment (thus reducing the Nyquist limits of what the FFT is able to resolve in each segment). However, it is notable that the PSD produced from windows as small as 50 points follow the same general form as our original 1000-point periodogram, although minor frequency components, specifically those in the $\leq 10^{-2} \mu m^{-1}$ frequency region, are smoothed into the background.

In cases where a 500-point PSD might not produce a sufficiently smooth plot, we observe that a 250-point window with a 25% overlap still captures most major frequency components. However, unlike the case of the 500-point window, increasing the overlap beyond 25% appears to reduce resolution in the low-frequency range, and increase variance in the high-frequency region for all of our small windows.

4 Surface Features / PSD Signatures

It has been hypothesized that certain kinds of surface features (welds, grain boundaries, point defects) will have readily identifiable PSD signatures. The ability to identify these features solely based upon a PSD plot would be a useful diagnostic in a manufacturing environment, where we would like to minimize the amount of time spent testing and analyzing each cavity. Similarly, we would like to be able to identify the presence and relative size of these surface features at different stages of the polishing process.

To test this hypothesis, we will closely examine a surface containing large and easilyidentifiable grain boundaries, and digitally add a similar grain boundary to a monocrystalline surface known to contain no grain boundaries. Comparing the PSD plots of the monocrystalline surface, before and after the addition of the grain boundary could potentially show one of these hypothesized signatures.

We select a polycrystalline mechano-polished sample, as our analysis of optical images of this sample shows a smooth surface with notably distinct grain boundaries. We then take a cross-section of an AFM scan to get a clearer picture of the size and shape of the grain boundary.



(a) Optical Micrograph (350x Magnification. Image $~900\mu m$ across.)

(b) AFM Image ($80\mu m \ge 80\mu m$). Sectioned area highlighted



Figure 10: Grain boundary analysis of polycrystalline mechano-polished niobium

This grain boundary is measured to be approximately 100nm in height, over a lat-

eral distance of approximately $10\mu m$. Naturally, larger and smaller grain boundaries exist throughout the sample. Although a detailed statistical analysis of grain boundary sizes would make an interesting topic for study, this is not necessary for the purposes of our analysis until we establish that these PSD signatures exist at all.

For the sake of simplicity, we will be overlaying our artificially-constructed grain boundary on top of a 2-dimensional profilometry scan of an electropolished monocrystalline niobium sample. The artificial grain will be $40\mu m$ across, $0.2\mu m$ high, with a flat plateau, and a boundary approx. $10\mu m$ long. Although $10\mu m$ is a fairly short distance for our profilometer to measure, our profilometer's stylus has a radius of $2.5\mu m$, and is configured sample data at a resolution of $2\mu m$, which should be just barely sufficient to resolve the slope of this grain boundary.



(d) Power Spectral Density of perturbed and unperturbed surfaces

Figure 11: Grain boundary analysis of polycrystalline mechano-chemical polished niobium

As can be observed in the figure above, the PSD plot of the perturbed signal does indeed indicate the presence of additional surface features in the range of $10\mu m$ (spatial frequency of approx $0.1\mu \cdot m^{-1}$). The oscillatory pattern surrounding this peak has been observed to be a characteristic of nearly-vertical surface features. Given that this example of a grain boundary may be a somewhat extreme example, we suggest three additional perturbations to attempt: a single grain (as opposed to three); a set of 3 grains with a $30\mu m$ boundary, as opposed to the $10\mu m$ boundary we had simulated earlier; and finally, a set of three grains with a more realistic $10\mu m$ step height, as opposed to the $20\mu m$ we had been using earlier.



(a) Perturbed with single $0.2\mu m$ peak, $10\mu m$ boundary.

(b) Perturbed with three $0.2 \mu m$ peaks, $30 \mu m$ grain boundary



Figure 12: Power spectral analysis of artificial grain boundaries on mechano-chemical polished niobium

We can immediately notice from these three plots that the PSD of the perturbed surface only differs slightly from the PSD of the unperturbed surface, with very few (if any) discerning features. Based upon these plots, we arrive at the unsatisfying conclusion that grain boundaries and surface features do not have a significant (or identifiable) effect on the PSD of a given surface, unless those features have particularly sharp edges, or are particularly tall in comparison with the overall roughness of the surface.

5 Conclusions & Further work

We have demonstrated that power spectral analysis can be a powerful tool to analyze and characterize the features of a surface, especially for the purposes of combining results from multiple types of instrumentation. Through these methods and results, we hope to be able to fine-tune the processes by which the niobium surfaces are prepared, in an attempt to produce SRF cavities with higher acceleration gradients.

The employment of the Welch method for power spectral estimation has been shown to produce consistent, repeatable PSD estimates of surfaces, allowing for less ambiguous analysis and easier comparison of datasets from disparate sources. Although we were unable to positively confirm our theory that certain types of surface features can be identified from their PSD alone, we remain confident that the PSD is an accurate indicator of the surface's overall roughness.

Although progress was made to attempt to reduce the amount of time required for analysis, further steps must be taken, as AFM measurements remain time-consuming and unreliable. A better understanding of how differently-sized surface features affect SRF performance would greatly contribute to the optimization of our polishing and analysis processes, particularly if these features are large enough to be detected without the use of AFM. The use of white light interferometry should also be studied in-depth to adequately determine the applicability of this technique to our analysis. Work is currently underway at Jefferson Lab to develop techniques for non-destructively recording the surface of the interior of an SRF cavity after it has been manufactured.

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