REVIEW OF SRF MATERIALS WORKSHOP

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Abstract

The performance of niobium cavities has approached the theoretical hard limit. Yet the consistent achievement of higher performing cavities remains the greatest challenge. To further understand the basic materials science, a workshop was held at Fermilab in May 2007 to present and discuss the fundamental and experimental limitations, and propose new ideas [1].

INTRODUCTION

As a niobium cavity undergoes material preparation, manufacturing, processing and final RF testing to its limit, many changes to the material could affect the final cavity performance. The workshop program focused on the niobium material production, innovative processing, surface analysis, RF superconducting properties and the fundamental RF superconductivity. The workshop also invited the presentation of research focused on new materials beyond the currently dominant material: niobium.

Overall issues encountered in SRF include materials and surfaces such as surface roughness, impurities, oxides, grain boundaries and thermal properties [2,3]. These affect the cavity performance in terms of breakdown field and surface resistance before field emission. While high field dissipation could be better explained by vortex dynamics [4], a careful calculation of superheating field suggests the current understanding of ultimate breakdown field could be greatly improved [5].

Experimental results from the high field region have not been well connected to many models including vortex model [3]. Many well developed surface analysis instruments and characterization techniques have been used to understand the material surface and its intrinsic physical properties [6]. New techniques such as laser surface interaction [7], tunneling spectroscopy [8] and near-field RF scanning [9] could help to further understand the subtle nature of RF surfaces. Temperature mapping studies concluded the cavity performance limitation comes from a few isolated hot spots. Unfortunately, the surface studies focusing on hot spots have been limited [10].

Looking beyond the hard limitation set by the intrinsic niobium critical magnetic field, experimental techniques and alternative materials have been proposed to extend the current SRF material studies well into the future [11]. Atomic layer deposition showed good promise to realize the multi-layer surface engineering [4]. Among the new materials, MgB2 and its fabricating technique seemed feasible [12].

Based on the nature of application-driven SRF technology, the surface studies historically provided more process verification than process guidance [13]. Decades of processing development successfully pushed the cavity performance close to the currently perceived theoretical limit [5]. The limitation to the state of art processing has been the poor reproducibility. Some effort has been devoted to less damaging chemical mechanical polishing [14], alternative electropolishing [15], plasma dry etching and cleaning [16, 17] and gas clustered ion beam processing [18].

In addition to the high field and processing limitations, the practical cavity performance can also benefit from the optimized niobium material manufacturing and well engineered cavity forming [19,20].

THEORETICAL ADVANCEMENT

A vortex could penetrate into the surface due to a remanent magnetic field, thermal electric currents, or local RF magnetic field which may be strong enough at high accelerating gradient. The vortices penetrate through the weakest spot or RF surface imperfections such as defects or grain boundaries. Once a vortex moves in, the subsequent oscillating field causes it to diffuse and annihilate with an anti-vortex. The vortex movement has a time scale the same as RF period; it could best be described as a "jump" at supersonic speed [4]. As Gurevich calculated, the "jump" is the dominating local heat source which comprises a hot spot. Since the vortex penetration is still considered to be happening around the critical field, one of the solutions to delay vortex penetration is to utilize the surface barrier feature of a very thin superconducting slab [4].

The remaining unknown is the exact magnetic field level at which a vortex starts to enter the superconductor and causes the loss. The past models reviewed by Padamsee [3] showed overly simplified formulae and temperature extension which resulted in pessimistic limitations for niobium, and are even worse in the case of high κ superconducting materials. As stated by Padamsee, the simple energy balance approach used in the line nucleation model overly simplified the meta-stable state under which the critical field is not the same as the equilibrium critical field. It does not bode well for uniform, flat and pure superconductors, such as the niobium typical for modern day cavities. Another inconsistency of the earlier line nucleation model was the simple linear extrapolation from a DC superheating field to an RF superheating field. Sethna proposed a more generic theoretical framework based on the Eilenberger equation to safely cover the lower temperature region where the energy barrier calculation and linear stability analysis can be worked out [5].

NEW RESULTS AND SURFACE CHARACTERIZATION TECHNIQUES

An oxygen diffusion model suggested the region right below the surface oxide layer would be improved during the low temperature baking [2, 21] which has been responsible for removing high field Q-slope. A 400°C insitu baking showed that nearly all the oxide was gone and the oxygen content at surface region would be greatly improved [22]. Yet the cavity test results showed the Q-slope remained. Eremeev concluded that the surface oxide may follow a different diffusion process than those previously modeled or that there may be other impurities which can be further studied using cut-out samples from these 400°C baked cavities.

For those cavities showing high field Q-slope (without baking), the sample was cut out following T-map indication for further analysis. The x-ray photoelectron spectroscopy (XPS) results revealed no oxide difference between the hot spot and regular surface samples [10]. Surface roughness and grain distribution also remained indistinguishable. The only difference picked up by Auger electron spectroscopy (AES) was the slight nitrogen signal in four hot spot samples while no such signal was seen in regular samples.

Surface oxide studies by Variable Photon Energy XPS on non cavity samples also concluded the oxide layer modified by low temperature baking did not seem to improve the high field Q-slope [23].

Other than oxide studies, several other new techniques may be helpful such as laser scanning microscopy, near field scanning microwave microscopy, tunneling spectroscopy and grain boundary studies using combined tools such as focused ion beam cutting and high resolution transmission electron microscopy (TEM) and electron energy loss spectrometry (EELS) [24].

A laser scanning microscope uses a laser to heat the superconducting resonator surface to induce a resonant signal change which would be proportional to local RF currents. Such a technique could be used to identify defects and study the surface current enhancement at grain boundaries [7].

Near field microwave microscopy may be able to use the same RF frequency as in a cavity to study a sample, especially at a grain boundary [9]. The important issue would be to keep the necessary resolution at higher frequency and increase the field strength comparable to that at a cavity surface.

Tunneling spectroscopy measures the tunneling current between a sample and the point contacting probe tip in order to calculate the electron state density and band gap. The comparison between measurements on baked and unbaked niobium sample showed no apparent change in band gap, except the inelastic behavior of the interface [8]. Careful theoretical work and further experiment are under development.

Many devices were built to directly measure surface resistance on a small sample with limited success [25]. The newly designed mushroom cavity was the latest effort

to measure surface resistance and the super heating critical field [26]. The base cavity was constructed from copper to reduce the cost and allow quick heat dissipation in a high power pulsing test.

The phonon peak study of differently treated niobium could provide ways to bring back the phonon peak, enhancing the thermal conductivity of niobium to provide extra capacity to ward off potential hot spot induced quenches [27].

NEW MATERIALS

For a material to be better suited for SRF cavities than pure solid niobium it has to have higher critical field than niobium to sustain higher local magnetic field; it also has to have a higher transition temperature and lower normal state resistivity to have lower surface resistance [11]. Several compound superconductors in the A-15 and B-1 series all satisfy the above requirement with Nb3Sn and MgB2 exceptionally well positioned in Vaglio's chart [28].

Ultra high quality MgB2 film has been achieved through hybrid physical-chemical vapor deposition [12]. A two-stage process has been proposed to coat a single cell cavity [12]. First, a boron film will be coated to the cavity inner surface by heating the cavity to 400-500°C during the gas flow of B2H6 and H2. The second stage requires the cavity to be heated to ~850-900°C while Mg vapor reacts with the boron film.

Atomic layer deposition (ALD) is a staged chemical vapor deposition self limited by surface reaction [29]. A typical deposition uses two chemical precursors one at a time. Each step limits the surface growth by one atomic layer. ALD is conformal to the substrate, pin-hole free and can achieve coating with very uniform thickness. These advantages make it very attractive to achieve the high-quality multilayer film proposed by Gurevich [4]. It can also be used to cap the niobium surface with layers such as Al2O3 to prevent oxygen diffusion during baking. This allows one to study the oxygen effect in niobium cavities.

INNOVATIVE PROCESSING

Standard processing has been very helpful to bring the current record breaking cavity performance. Yet the processes have been expensive, time consuming and less reliable in achieving high performance, especially in multi-cell niobium cavities. As summarized by Antoine, alternative processes are available and being perfected [13]. Three processes were designed to combat field emission. Dry ice (CO2) removes the surface particles by mechanical force much like high pressure water rinsing, but does not leave a residue. It has the potential to be applied to cavities already assembled in a cryomodule. Helium processing has been demonstrated to reduce field emission moderately [30]. High power processing improves the performance by burning out field emitters. It successfully raised a multicell cavity gradient from 12 MV/m to 25 MV/m [31]. Ultrasonic and megasonic rinsing have been successful at removing some strongly bonded sulfur particles in electropolished cavities [32]. Alternative acid-free electropolishing solutions do exist as demonstrated by Palmieri [33] and Crooks [15]. Another idea to avoid surface defects is not through aggressive etching but applying a high purity film to bring the RF surface layer to highest quality.

Gas clustered ion beam (GCIB) is another tool to combat field emission. A sample treated by GCIB of oxygen showed not only the field emitters were removed but also the surface was greatly smoothed [18]. The concerns about the surface oxide modification and crystal lattice damage need to be carefully tested. Both cavity testing and sample analysis are being conducted.

Plasma etching and cleaning are also pursued to avoid hazardous acid handling in standard processing. Preliminary experiments with reactive gas plasma such as chloride and boron trifluoride have been successful [16]. The limitation for this process is the undesired chemicals such as niobium borides left on the etched surface. Careful control of the sample temperature may help.

Electron cyclotron resonance (ECR) plasma has the potential to convert the plasma processing to a truly insitu post processing [17]. An external magnetic field is applied to the cell region in a resonating cavity as such the plasma is limited with a cavity cell but not in vulnerable RF input coupler. Upon finishing processing, the cavity can remain sealed until connected to beam line. Preliminary results showed the process is feasible and effective in sulfur removal.

Chemical mechanical polishing (CMP) uses abrasive and reactive slurry to remove the surface layer. Initial studies showed a niobium sample at 1 μm roughness (Ra) improved to 10 nm in 70 minutes [14]. However, the polishing could be labor intensive in a cavity environment. At least, this technique could have the benefit of a very smooth surface with a thin damage layer. With the reduced electropolishing time, cavity performance could improve.

NIOBIUM PRODUCTION

As niobium material is being prepared, several preferred conditions could be beneficial for final cavity performance. As summarized by Singer [19], purity, grain distribution and mechanical properties are important factors for successful cavity production. Tantalum is the most concentrated impurity in niobium. Current experimental data showed the high tantalum content could negatively affect the performance of cavities reaching gradients above 30 MV/m. Fine grain material puts a burden on sheet manufacturers to obtain uniformly distributed grain size throughout the bulk of the material [34]. The current vendor processes are relatively costly and prone to introduce contamination and defects. Equal Channel Angle Exchange (ECAE) is proposed to reliably achieve uniform fine grain niobium. Preliminary results showed the possibility to obtain a niobium texture which is favorable for deep drawing [35]. Large grain niobium

sheet was claimed to use much less processing to produce. The limitations were unexpected forming behavior and rough grain boundaries. The single crystal proves to be best suited for cavity production [20], yet the extra cost based on current forming technology [36] did not justify the performance gained over fine grain niobium.

To understand niobium deformation and recrystalization, extensive simulation models have been established, and the work is under development [37].

TIG welding has been proposed to reduce the welding cost compared to electron beam welding. Current results showed the cleanliness of the welding environment needs to be optimized [38].

CONCLUSIONS

The SRF materials workshop has brought in many parties who became interested in SRF technology. Many collaborations and experiments have been initiated after the materials workshop. Many were reporting their new findings in this workshop. We expect another productive SRF materials meeting in the near future.

REFERENCES

- [1] SRF Materials Workshop,
- http://tdserver1.fnal.gov/project/workshops/RF_Materials/
- [2] G. Ciovati, Introduction talk on SRF issues about materials and surfaces, SRF Materials Workshop.
- [3] H. Padamsee, Introduction: Opening remarks to update the experimental and theoretical situation for the High-Field dissipation (high field Q-Slope), SRF Materials Workshop.
- [4] A. Gurevich, High-field surface resistance and RF breakdown in multilayer coatings, SRF Materials Workshop.
- [5] H. Padamsee and J. Sethna, Physics of the Ultimate RF critical magnetic field (superheating field), SRF Materials Workshop.
- [6] M. Kelley, Overview: Survey of characterization methods, XPS, SIMS, TEM..., SRF Materials Workshop.
- [7] S. Anlage et al., Imaging of Microwave Currents and Microscopic Sources of Nonlinearities in Superconducting Resonators, SRF Materials Workshop.
- [8] J. Zasadzinski et al., Tunneling Spectroscopy and Surface Modification of Nb for SRF Cavity Development, SRF Materials Workshop.
- [9] J. Wu, Overview: Scanning RF Microscopy, SRF Materials Workshop.
- [10] A. Romanenko, Surface Analysis of samples dissected from a cavity with high-field Q slope (XPS, Auger, EBSD, SIMS, Optical Profilometry), SRF Materials Workshop.
- [11] A.-M. Valente-Feliciano, Overview: New materials for SRF cavities, SRF Materials Workshop.

- [12] X.X. Xi, MgB2 thin film and its application to RF cavities, SRF Materials Workshop.
- [13] C. Antoine, Overview: Unconventional cavity processing techniques, SRF Materials Workshop.
- [14] S. Muftu et al., Chemical Mechanical Polishing for Manufacturing of Smooth Nb Surfaces, SRF Materials Workshop.
- [15] R. Crooks et al., Novel Surface Treatments for RRR Niobium, SRF Materials Workshop.
- [16] M. Raskovic et al., Plasma etching of Niobium surface, SRF Materials Workshop.
- [17] G. Wu et al., ECR plasma: a possible in-situ cavity processing technique, SRF Materials Workshop.
- [18] D. Swenson, In-situ surface treatment of SRF cavities with Gas Cluster Ion Beams and the effect on the maximum gradient and Q-slope of the cavity, SRF Materials Workshop.
- [19] W. Singer, Introduction: Metallurgical and Technological Request for High Purity Niobium in SRF Application, SRF Materials Workshop.
- [20] G. R. Myneni, Overview of the RRR Nb Specifications and the Evolution of SRF Technology, SRF Materials Workshop.
- [21] K. Yoon et al., Atomic scale chemical analyses of niobium superconducting radio frequency cavity, SRF Materials Workshop.
- [22] G. Eremeev, Correlation between XPS and temperature maps for nearly oxide-free niobium, SRF Materials Workshop.
- [23] H. Tian et al., Surface oxide studies on solid Niobium for Superconducting RF Accelerators using variable photon energy XPS, SRF Materials Workshop.
- [24] Z. Sun, Analytical electron microscopy studies and transport characteristics of large grain niobium for SRF cavity, SRF Materials Workshop.
- [25] C. Reece, Overview: Review of RF measurement of samples, SRF Materials Workshop.
- [26] S. Tantawi et al., Critical magnetic field measurement of MgB2, SRF Materials Workshop.
- [27] S.K. Chandrasekaran et al., Heat transfer measurements of niobium for SRF cavities, SRF Materials Workshop.
- [28] R. Vaglio, Particle Accelerators 61, 391 (1998).
- [29] M. Pellin et al., Atomic layer deposition, SRF Materials Workshop.
- [30] C. Reece et al., Improvement of the operational performance of SRF cavities via in-situ helium processing and waveguide vacuum processing, Proc. Of PAC 1997.
- [31] H.Padamsee, Jens Knobloch and Tom Hays, RF Superconductivity for Accelerators, Wiley-Interscience, 1998.
- [32] K. Saito, TTC meeting at Fermilab, 2007.
- [33] V. Palmieri, Alternative Electropolishing of Niobium, The International Workshop on Thin Films and new ideas for pushing the limits of RF superconductivity, Padua, Italy, 2006

- [34] P. Jepson, Textures of Niobium Sheet, SRF Materials Workshop.
- [35] K. T. Hartwig, Microstructural Refinement of Niobium for Superconducting RF Cavities, SRF Materials Workshop.
- [36] V. Levit, Orientation Effect on Recovery and Recrystallization of Deformed Niobium Single Crystals for Superconducting RF Cavities, SRF Materials Workshop.
- [37] D. Baars, Preliminary investigation of a model for predicting recreytallization in Nb, SRF Materials Workshop.
- [38] C. Compton, The Potential of TIG Welding Technology For SRF Cavity Fabrication, SRF Materials Workshop.