FNAL 3-year Single-Cell SRF Cavity Program

Lance Cooley, SRF Materials Group Leader February 23, 2009

Introduction

This document describes a program to conduct single-cell SRF cavity R&D at Fermilab. **The timeframe for this program is the 3-year period starting mid-FY2009 (3/1/09) through mid-FY2012 (ending 2/29/12).** The program is intended to serve several objectives:

- 1. Carry forward the single-cell S0 program for the Americas Region under the ILC GDE and transform it into a program with a long horizon. This objective seeks improvement in the baseline cavity process through statistical observations and processing trials. The results of R&D can be readily implemented into design efforts for Project X, ILC, and other TESLA-type SRF cavity applications.
- 2. Initiate a coordinated program to address generic topics for SRF, in particular the development of an understanding of performance limits on the basis of materials and processing. This objective seeks *transformational* improvement through *basic materials science*, which could have tremendous impact on SRF cavity performance, yield, and cost, but could also necessitate cavity designs or processes that deviate largely from existing baselines.
- 3. Integrate *regional* expertise near Fermilab to produce a complete and effective world class singlecell research effort. This includes our partners at Cabot, Able, ANL, MSU, FSU, UC, NWU, IIT, and others.

Single-cell cavity programs have always been an integral part of SRF projects. Most performance records are associated with single-cell cavities, and processing breakthroughs have been pioneered first on singlecell cavities. The relatively low cost of single-cell cavities allows them to serve as the pivotal link between basic materials studies, which often requires destructive characterization, and actual behavior relevant to present technology, which involves 9-cell cavities in cryomodules. The relative ease of fabrication has been vital for the qualification of vendors. The smaller size of single-cell cavities means that tools and facilities can be qualified while still minimizing quantities of acid, liquid helium, etc.

The work plan will be partly driven by priorities set by TTC, ILC-GDE, and other SRF programs. Close communication with these programs will be essential to assure appropriate overlap and prevent unnecessary duplication. In addition, much work will be driven by scientific curiosity and the ability to leverage Fermilab's personnel and unique capabilities. These include the recent installation of an optical inspection system and its planned automation, a new temperature-mapping (Tmap) system, a custom designed and built tumbling machine, the availability of a class IV laser for spot re-melting, the development of coupon electropolishing and process witness capabilities, and the anticipated start of a processing apparatus solely dedicated to single-cell work. The work plan will further include strong interactions with university research groups, in particular the research consortia recently proposed to DOE-HEP (ANL / UC / IIT / FSU / NW and FNAL / UC / FSU / IIT / NW).

The work plan will be modified as necessary based on results obtained and new information. A central source of feedback will be reporting to TTC and ILC-GDE projects. In addition, Fermilab plans to continue hosting an annual SRF Materials Workshop as well as be a central participant in international SRF workshops and other superconductivity and accelerator conferences (ASC, PAC, etc). Reporting of results and feedback from peers at these venues will further serve to update the work plan.

Depending on the labor and resources available to the program, some or all of the milestones below could be met by FY2012. This includes achievements by collaborators enabled by this program.

- Tumbling and chemical-mechanical polishing recipes
- Safer and more environmentally benign chemical processing, rinsing, and degreasing routes
- Improved material specifications
- Pit-free welded cavities
- EP processes tuned to in-line fluorine monitors, oxidation kinetics, and material texture
- Remediation strategies for various performance limitations
- Feasibility of niobium-on-copper cavities by new routes
- Hydroformed single-cell cavities

Scientific Approach

This research will be carried out primarily by the SRF Materials Group in Technical Division. A significant major role will be played by the A0 vertical test stand in Accelerator Division, with its personnel. As discussed later, the program assumes that the A0 VTS will become entirely dedicated to single-cell work, and that a vigorous development of support continues, such as continued development of Tmap systems. Also, some work will be carried out using the FNAL/ANL processing facility and the vertical test facility in IB1, after coordination with 9-cell processing and testing. The ANL and IB1 work is anticipated to occur at a 20% level.

Work areas and anticipated experiments fall into 6 categories as detailed below. Where appropriate, the scientific issues being addressed are stated. **The baseline cavity processing sequence, testing sequence, diagnosis sequence, and performance benchmarks are attached as Appendix A. As stated there, a "qualified" single-cavity shall exceed 30 MV/m in vertical test at 2 K.**

- **1. Qualify new vendors of SRF cavities.**
	- **PAVAC**: Anticipated purchase of ~6 cavities late FY09 to early FY10.
	- **HC Starck sheets:** Fabricate 2-6 cavities using sheets from this new vendor. The manufacturer is to be determined.
- **2. Use proof-of-concept experiments to test process improvements.**
	- **Tumbling**: Develop and apply a tumbling regimen to \sim 2 qualified cavities and \sim 2 unqualified cavities, then (a) compare results for tumbling to baseline EP process, (b) compare whether tumbling improves or degrades process. The actual process may be an integration of the baseline with tumbling, e.g. tumble for 100 µm material removal followed by 20 µm EP. Then extend method to new or unqualified cavities. *Issue: Does tumbling provide comparable or better performance with reduced reliance on hydrofluoric acid?*
	- **High fluorine-concentration processing (FNAL + JLab):** (a.k.a. "flash" processing): Compare performance for \sim 4 cavities processed under EP conditions that use fresh acid, or under other procedures to maintain a high fluorine gradient, to results for baseline processing. This includes developing a method to assess fluorine gradient during the EP process (presently a Raman spectrometer is sought for this purpose, as discussed later). *Issues: Are the benefits of "flash" processing compelling enough to offset increased complexity and acid use? Can we understand the mechanism why "flash" processing works?*
	- **ECS validation:** Compare performance for \sim 2 cavities that have been fabricated from sheets with known eddy-current scan defects and have been given a baseline process to assess whether the ECS control is useful. *Issue: The ECS defect cavities have been completed. The impact of defects on cavity performance is not known but is suspected to be bad.*

• Large grain (Niowave): Contract with Niowave / MSU to produce 2-3 cavities (1.3 GHz) from large ingot to validate formability, weldability, and processing claims. Rinse, assemble and test cavities to evaluate performance. *Issues: Our direct interaction should give us better grasp of potential cost issues as well as a better understanding of their production claims.*

3. Supply reference cavities for qualification of tools, procedures, processes, test stands, equipment, and so on.

- **ANL facility**: Processing to qualify the ANL facility is underway at the time of drafting this program. It will be assumed that the facility will be fully qualified when this is implemented.
- **A0 VTS:** Qualify this test facility using cavities with known low, medium, and high gradient.
- **ICPA**: ICPA operation is expected mid-FY2010. Repeat ~6 EP processing cycles on previously qualified cavity(ies) to demonstrate repeatability. Attain qualifying gradient \sim 3 consecutive times.
- **IB4 oven:** Qualify planned upgrade (FY2010 or later) to IB4 high-temperature bake oven to verify that gradient can be maintained in ~3 previously measured cavities.
- **High-resolution Tmap system (FNAL + JLab + Cornell)**: Provide cavities with known hot spots to qualify Tmap system (Muckerjee) and facilitate development of new systems (FY2010 and beyond) with JLab and Cornell (second sound). Explore whether highresolution mapping combined with cavity modeling can be used to triangulate the location of field emitters.

4. (a) Supply benchmark cavities to serve as a baseline for subsequent experiments done by collaborators. (b) Also, accommodate process / test requests from collaborators.

- **ALD** cap and bake (ANL and JLab): Provide 1-2 cavities (1.3 GHz) after they have been given a baseline process and have passed a qualifying performance test. If requested, perform high-temperature or low-temperature vacuum baking. Then, perform rinsing and vertical testing. *Issues: Capping with aluminum oxide prevents oxygen from re-attacking the niobium surface. Baking decomposes niobium oxides and drives the oxygen down deep into the niobium bulk. Do these steps improve both Q and the quench field by removing "pollution" and possible contact with magnetic scattering sites?*
- **ALD multilayer on niobium (ANL):** Provide 1-2 cavities (either 1.3 or 3.9 GHz) after they have been given a baseline process and have passed a qualifying performance test. Then, perform rinsing and vertical testing after multilayers have been grown. The first step is a "niobium on niobium" cavity, upon which subsequent layers will be grown. *Issues: This is an explicit test of Alex Gurevich's theory that the limits of niobium bulk can be surpassed by multilayer geometries. Note: multilayer deposition requires development of a plasma-aided process at ANL, to be developed FY2010.*
- **ALD niobium on copper (ANL + India + UES)**: Provide 2-3 copper cavities, which have been fabricated and qualified through India collaboration, to ANL for coating with niobium. As an alternative, or in addition, provide cavities to a new vendor (UES, Inc.) for coating via chemical vapor deposition. Then receive coated cavities, rinse, and assess performance with vertical test. Possibly re-process cavities with ICPA. *Issues: plasma-enhanced atomic layer deposition (PE-ALD) promises to be a very clean niobium deposition route, similar to chemical vapor deposition. Is this so?*
- **MgB₂** (Penn State): Provide 1 cavity (3.9 GHz) with special end tubes to accommodate PSU's chemical vapor deposition system. Receive back a $MgB₂$ coated cavity. Possibly rinse with alcohol or CO_2 ; this will not be water rinsed because water attacks MgB_2 . Vertical test to assess gradient and residual resistance at 2 K and 4.2 K. Possibly this experiment could be repeated. *Issue: Is MgB₂ a viable SRF material due to its much higher critical temperature of 39 K?*
- **EP (ABLE):** Provide ~6 qualified cavities to evaluate industrial electropolishing at ABLE Electropolishing. This consists both of 1.3 and 3.9 GHz cavities. *Issues: Can an industrial vendor provide sufficient quality, or does EP always have to be a processing activity that is coordinated with a lab? Will ABLE repeat solving vertical EP issues already solved by Cornell?*
- **Chemical-mechanical polishing (CMP Cabot and Northeastern):** Provide ~3 qualified cavities to Cabot to see if their proprietary slurries can achieve superior surface finishes without need for HF. Provide rinse and VTS for these cavities. Then, implement their slurries together with tumbling studies at FNAL on another ~3 cavities (FY 2010-2011). Also, accept ~3 cavities from Northeastern, who plan to apply CMP to sheets and half-cells *prior to welding* at AES, for possible process, test and analysis. *Issues: Is a nanometer RMS roughness achievable? Is non-HF processing viable?*
- **Rx and Rv half cells (FNAL and Black Labs and vendor TBD):** At Fermilab, revisit experiments to recrystallized (Rx) or recover (Rv) deep-drawn half cells prior to welding. Include coupons in the study. Fabricate 2 to 3 cavities and assess shrinkage, mechanical property sacrifices, possible changes to compromise, etc. Then, assess RRR and chemistry near welds by cutting apart one single-cell (3.9). Process the others using a baseline followed by rinse and test. Also, supply niobium sheet to Black Labs and another vendor for half cell processing and then pre-weld heat treatment. Then provide baseline processes and test welded cavities (2 to 4 in total). *Issues: Weld pitting may arise from dislocation clusters or interstitial atom clusters. Does pre-conditioning the material to alleviate the formation of such clusters reduce the tendency for pitting?*
- **Textured sheets and single crystals (FNAL + MSU, JLab + Black Labs):** Complete fabrication of single-crystal cavities at FNAL (two 3.9 GHz cavities). *Issues: Besides advantages for etching and smoothness, are there advantages for avoiding weld pits? Or, will dislocation pitting show up?* Also, conduct two experiments in parallel with textured sheet (\sim 3 to 5 cavities each team, FY2010-2012): working with material vendors (Starck, Wah Chang), obtain niobium sheet with different crystallographic textures. Verify textures at MSU and Black Labs prior to, and after, deep-drawing. Weld half cells, analyze rotations of grains post-weld. Process per baseline, rinse, assemble, test. *Issues: These cavities should expose whether orientation aff*
- **Hydroformed cavities (Ohio State, Texas A&M, MSU, Black Labs):** Complete fabrication of 3.9 GHz hydroformed copper and niobium cavities at MSU, then process and test. Consider option for scaling up the MSU program to 1.3 GHz cavities at \$200k cost. Also, consider single-crystal cavities if \$300k funds for single-crystal tube refining (Nevada-Reno) becomes available. An additional benefit of the MSU program is the installation of a bulge test there during FY2010. Also, TAMU have demonstrated the grain refining of the weld zone of a seamed niobium tube. If funds are available, hydroform additional \sim 2 cavities at MSU using TAMU-refined tubes. Also, anticipate coordination of raw materials studies and provide cavity rinsing, assembly and testing to Black Labs for 2 to 3 three-cell cavities (or extract single cells from these 3-cell cavities), as part of SBIR work using the DESY facility (expected late FY2009 thru FY2011). Finally, Ohio State is fabricating extrusionbonded niobium-copper tubes under SBIR work. This work should be completed during mid to late FY2010 and these tubes should then be sent to MSU or to another institution (e.g. DESY, via Black Labs) for hydroforming. *Issues: These experiments should help us understand better the wide range of raw materials issues, formability issues, and QA issues (e.g. bulge test) beyond the initial scope of the pioneering DESY work. In addition, feasibility of this process, in particular the raw materials supply chain, could be demonstrated.*

5. Test new cavity ideas:

- In-situ remediation: Complete several tests of laser re-melting using known pits in \sim 3 cavities. Reprocess cavities, either EP + Ultrasonic + HPR or just Ultrasonic + HPR. Assess feasibility of this process. Then (FY2011), consider mounting a reprocessing laser and mirror and gas purge in-line with optical inspection system for search-and-destroy remediation tool. *Issue: Using the laser may be more tractable and elegant than either small-pad grinding (Kyoto) or e-beam re-melting (JLab).*
- **Plasma post-processing:** Complete further tests to understand the viability of cavity postprocessing once assembly is complete. This will likely be the summer intern work of a student.
- **"Quench" cavity:** Fabricate 1 cavity with altered shape that reduces the ratio of Eacc to B. In this case, field emitters are suppressed and sources of quenching are amplified. Reevaluate the usefulness of this task after initial demonstration (FY2010).
- **Polyhedral cavity:** Working with Texas A&M, fabricate and test a longitudinal seam cavity as a demonstration of concept. If TAMU gain funding, it may be possible to extend this to a real cavity process and test.

6. Develop characterization tools

- **High-resolution temperature mapping**: *A robust T-mapping effort in parallel with the single cavity program is essential. One Tmap system is urgently needed for the A0 VTS, so this will be a high priority for the single-cell program.* Two to three cavities will be made available for continual development and refining of temperature mapping systems. One of these cavities should have a known hot-spot arrangement and therefore will serve as a calibration for new Tmap systems. In particular, diode thermometry has the capability to increase the number of sensors placed on a cavity. Thereby, the amount of information we can gain increases to the point where it may become possible to triangulate on field emitters or assess Kapitza resistance at the outside of a cavity.
- **Optical inspection standards**: Cavities with calibrated pits, hand-made defects, positioning grids, and other features are needed for standards and calibrations for the optical inspection systems. This will require 2 to 3 non-qualified cavities.

Work Plan

 \overline{a}

The metric of planned work is the number of expected cavity EP processes and test cycles. **The work plan assumes 100% use of ICPA and up to 20% use of ANL as primary processing facilities. The work plan also assumes 100% use of the A0 vertical test stand and up to 20% use of the IB1 vertical test stand.** The work plan assumes no use of facilities at JLab or Cornell; however, these facilities will be relied upon to provide additional resources to alleviate schedule delays.

At present, the ANL facility can handle 4 cavity processes per month* with current manpower (Wu and Bice), or 48 processes per year. Adding a second work team (Scott Gerbick and Wade Muranyi) could increase this capability to 2 cavity processes per week or at maximum 100 processes per year. However, it should be stressed that the ANL facility is a *processing* facility, especially in the sense that once the processing parameters are established they should not be varied to maintain quality. That is, t**he ANL facility can** *only* **serve the single-cell tasks of providing benchmark cavity processing**. Given pending cavity orders, and assuming that similar cavity purchases in out years will continue to drive the ANL

^{*} Mike Kelly (ANL) estimates 4 processes per month, not 3 per 2 weeks.

schedule, approximately 40 processes at ANL are tied up by 9-cell work. The remaining ~ 8 processes (given present manpower) will be available to serve benchmark single-cell functions.

ICPA is expected to become operational in mid FY2010. **The purpose of this facility is an** *R&D* **facility, providing the means to vary processing intelligently as well as handle non-standard cavity processing situations.** Baseline processing can also be accomplished. It is expected that a 2-man team will be required to operate this apparatus, and staff is already here for that purpose (Thompson, Schuessler). The initial scope for this apparatus is 1 single-cell cavity process per week, or a maximum of 50 processes per year. The full capacity is intended to be 100 processes per year.

The vertical test stand (VTS) at A0 has entered the operational readiness clearance phase and should be ready for cavity testing at the start of this program. For purposes of scheduling, a 50% capacity (i.e. 25 tests) is assumed for the first year of operations at A0. Tests will be led by N. Dharanaj, and we will rely upon A0 technicians for operation and cryogenics at the facility. The vertical test stand at IB1 will continue to provide testing services for this program as well.

Figure 1. Breakdown of cavity EP and test processes.

The total number of planned cavity processes over the 3-year program is 80, and the total number of vertical tests is 118. A breakdown of each cavity task is shown in fig. 1, and a further item-by-item breakdown is presented in Appendix B. The present cavity inventory is attached in Appendix C. As shown in Appendix B, new cavities will be added to the inventory as part of the R&D that will be performed. **The present inventory, along with the anticipated additions, is sufficient to sustain the program as planned.** Under the assumptions above, the ANL facility could provide 24-30 processes over the course of the program, while ICPA could provide approximately 100 processes. **Note that several cavities in the inventory have already been given qualifying processes, so the oversubscription of FY09-FY10 EP processes is offset by the number of qualified cavities (~30) in the present inventory.** Also, IB1 VTS could provide 24-30 vertical tests, and A0 VTS could provide approximately 125 vertical tests. **Therefore, the work plan can be met with 10-20% reserve if the available facilities are fully utilized.**

Budget and development of resources

The following estimates are made, based on present information (discussed in more detail below):

- **1 cavity EP process per week at a cost of \$5k (acid, waste, maintenance) with a 2-man team at 0.5 FTE**
- **1 rinse process per week (same week as cavity) at a cost of \$1k (hardware, water plant maintenance, not incl. clean room costs) using clean-room 1-man team at 0.5 FTE**
- **1 test cycle per week at a cost of \$15k (helium, maintenance) using 3-man (operator, cryo tech, data analyst) team at 0.5 FTE**

Development of resources will also be a key part of the program. Planned development includes:

- Purchase of a small quantity of niobium sheets, or use of excess niobium sheets, and using these sheets for custom cavity fabrication experiments
- Continued access to Sciaky for e-beam welding and training of Mike Foley's successor
- Support and maintenance of ICPA, with its clean room and HPR. (Note, support of A0 clean room with HPR is NOT considered).
- Tumbling, with tooling upgrade capable of chemical-mechanical polish
- Development of a Tmap system for A0
- Purchase of an additional optical inspection system and development of automation
- Upgrades to class IV laser to permit defect re-melting (mirrors, lenses)
- 120 °C oven retrofit at MDTL for low-T bake
- Purchase of new or rebuild of existing single-cell 1100 °C oven
- Seeding and support for university work

Synergistic activities

The single-cell program will benefit greatly from programs in place within the SRF Materials Group and the proposed university collaborations to conduct basic materials science. The SRF Materials Group presently conducts studies in the following areas:

- Weld coupons of niobium in different metallurgical states;
- Coupon-scale EP, fully instrumented for reproducibility or for process control with intelligent variation of parameters;
- Witness EP coupons that are put in-line with standard 1-cell EP and processed identically
- Optical, 3-D optical, and electron microscopy;
- Metallurgical testing.

In addition, collaborations with university groups can provide:

- Surface-science studies, including x-ray photoelectron spectroscopy, Auger electron spectroscopy, Raman spectroscopy;
- Chemical-kinetic studies, including real-time oxidation structures from scanning tunneling microscopy and 3-D atomic profiling;
- Structure-property studies, including scanning laser confocal microscopy, scanning electron microscopy with element identification and orientation imaging, transmission electron microscopy with holography and electron energy-loss spectroscopy, point-contact tunnel-junction spectroscopy, and atomic force microscopy;
- RF studies, including point-RF probe and non-linear strip-line resonator studies.

Key interactions to support these synergistic activities have already been established:

- Michigan State: orientation-imaging microscopy, sheet texture analyses, metallurgical expertise, cryogenic and bulge testing, forming science, hydroforming, welding science
- Florida State: grain-by-grain electron microscopy, magneto-optical imaging, laser scanning confocal microscopy, focused ion-beam micromachining, superconducting property measurements
- IIT: superconducting tunnel-junction spectroscopy
- Maryland: point RF probe analyses
- Northwestern: 3D atom probe reconstruction, depth profiling of composition, FIB micromachining
- Chicago: molecular diffraction for dynamic surface imaging, in-situ XPS, scanning tunneling microscopy, surface chemical kinetics
- Nevada-Reno: Zone refining and single-crystal niobium tubes
- Texas A&M: textured niobium, fine-grain seamed tubes (cheap tubes)
- Able Electropolishing: Single-cell EP
- Black Labs: niobium texture control, hydroforming, flow-formed tubes
- Ohio State: Nb/Cu extrusion bonded tubes and bi-metal hydroforming
- VCU: Hydroforming modeling

Appendix A – Baseline techniques and performance standards

Italic font denotes optional steps.

Baseline *Single-Cell* **Processing Recipe**

- 1. Incoming cavity quality control checks.
- *2. Optical inspection of as-received cavity.*
- 3. Bulk electro-polishing of ~150 um.
- 4. Ultrasonic degreasing.
- 5. High-pressure rinsing.
- 6. Hydrogen degassing at 600-800 °C.
- *7. Optical inspection.*
- 8. 20 μ m electro-polishing.
- 9. Ultrasonic degreasing.
- 10. High-pressure rinsing.
- 11. Assembly and vacuum leak testing.
- 12. 120 °C bake.
- 13. Vertical test.

Standard Testing Recipe

- 1. Hold at ~100 K during cool down to check for Q disease.
- 2. Q vs. T measurement during cool down.
- 3. Q vs. E measurement. RF process as needed.
- 4. Final Q vs. E measurement.

Notes[.]

All Q vs. E measurements to include radiation data logging. Utilize temperature-mapping system if available.

Diagnostic Techniques

- 1. Apply thermometry to determine location of limiting defect.
- 2. Perform optical inspection of limiting defect.
- 3. *Remediate defect by grinding, additional EP, additional degreasing, additional HPR, or other technique.*

Performance Standards

- 1. A cavity shall be considered as "qualified" if it attains 30 MV/m after being given the baseline process above.
- 2. A cavity may be considered as "qualified" if it attains 25 MV/m after an alternate process where BCP is substituted for EP in the baseline above and no final etching (steps 7-10) is done.

Appendix B - Breakdown of individual single-cell experiments

APPENDIX C – PRESENT CAVITY INVENTORY

3.9GHz Single Cell Summary 3C: 3rd harmonic single cell center cell Last vectors and Last updated on 2/12/2009

1.3GHz Single Cell Summary TE: Tesla Endcell shape

FNAL_ANL facility cummulative RF test cycles

Many people contributed to various works listed here. If you want to quote the data here, please contact M. Champion or G. Wu to make sure proper credit will be given to those who did actual works.

Legal Notices