SURFACE SCIENCE LABORATORY FOR STUDYING THE SURFACES OF SUPERCONDUCTING RADIO FREQUENCY CAVITIES*

Andy T. Wu[+]

Thomas Jefferson National Accelerator Facility, 12000 Jefferson Avenue, Newport News, VA 23606, USA

Abstract

A Surface Science Laboratory (SSL) has been established at JLab to study surfaces relevant to superconducting radio frequency (SRF) cavities. Current operational facilities include a scanning electron microscope equipped with energy dispersive x-ray analysis, a secondary ion mass spectrometry, a metallographic optical microscope, a transmission electron microscope, a high precision and large scan area 3-D profilometer, a scanning field emission microscope, and a fully equipped sample preparation room. A scanning Auger microscope is being commissioned, and will be available for routine usage soon. Results from typical examples of the R&D projects on SRF cavities that were supported in the past through the use of the facilities in the SSL will be briefly reported.

INTRODUCTION

There has been increasing interest in using Superconducting Radio Frequency (SRF) cavities in high energy and larger scale particle accelerators such as free electron lasers (FELs), linear colliders, energy recovery linacs, and high current storage rings for providing high gradient acceleration. In order to reach high gradient acceleration routinely, a better understanding of the surfaces of SRF cavities is crucial. This general rule is also applicable to SRF cavities based on Nb.

Currently, JLab's Continues Electron Beam Accelerator Facility (CEBAF) and FEL use Nb SRF cavities. Both CEBAF 12GeV upgrade and possible high current FEL require the production of reliable SRF cavities with highest performance at the lowest possible cost. To achieve this goal, recently the Institute for SRF Science and Technology (ISRFST) at JLab has launched a program aiming at a better understanding of the Nb surfaces produced by our current cavity fabrication techniques through studies of possible correlations between RF performance of Nb cavities and their various surface and bulk properties using samples from the same batch throughout the program [1]. Obviously, we need appropriate instruments in order to make this program successful. With strong support from management of various levels and some friendly colleagues at JLab, recently a Surface Science Laboratory (SSL) has been established as a result of almost one and a half years effort. Currently our SSL has the following systems:

Scanning Auger Microscope (SAM), Scanning Electron Microscope (SEM), Energy Dispersive X-ray (EDX), Secondary Ion Mass Spectrometry (SIMS), Transmission Electron Microscope (TEM) and Scanning Transmission Electron Microscope (STEM), Scanning Field Emission Microscope (SFEM), Metallographic Optical Microscope (MOM), and 3-D profilometer. Most of the listed systems are operational. SAM is waiting for baking to reach appropriate vacuum before being commissioned. At this moment, I am dealing with problems associated with the ion gun unit of the SIMS. I also set up a fully equipped sample preparation room that can do all sample preparations required by our SSL except ion polishing for TEM. An ion polishing system will be acquired in fiscal year 2004 to complete the set-up of the sample preparation room.

In this paper, I will describe these instruments and their functions. In the meantime of setting up this SSL, some of the operational instruments have been used for supporting different needs across JLab from various groups such as, for instance, mechanical engineering group, safety group, injector group, coupler group, cryomodule group, FEL group, cavity production group, S&T group, infrastructure group, physics division, etc…. Typical examples of the supported R&D projects relevant to SRF science and technology will be briefly reported here. It is hoped that this paper will provide some information regarding the instruments that we have in the SSL at JLab and show some typical work that I did through the use of the facilities in the SSL. This will hopefully lead to possible collaborations on mutually interested R&D projects in future.

INSTRUMENTS AND THEIR FUNCTIONS

It is well-known that the performance of an Nb cavity is mainly determined by the surface of the cavity, provided that the other parameters, such as, for instance the shape of the cavity and the bulk properties of Nb etc., are all optimized. It is the surface that finally determines the performance of an Nb cavity. Considerable understanding of the surfaces of Nb SRF cavities has been gained over the past decade. However, a systematic study of a particular unsettled problem is still needed. Take the surface oxide structure as an example, although a rough idea about it has been illustrated [2] a detailed understanding is still lacking. The top layer is clearly Nb_2O_5. What are the sub-oxides in between Nb_2O_5 and the pure Nb? How thick exactly is the sub-oxide layer? There is evidence that the thickness of the oxide structure

on Nb varies with the surface treatment procedures. Do the variations in the thickness come mainly from the Nb_2O_5 layer or from the sub-oxide layer and why? Is it true that an Nb surface prepared by the standard BCP method has some Nb_2O interfacing the pure Nb metal and the Nb_2O_5 layer [3]. What is the oxide structure on the Nb surfaces prepared by the KEK electropolishing (EP) method? Recently an alternative electrolyte recipe has been used in our lab to electropolish Nb and a superior surface finish in terms of smoothness has been obtained [4]. Will the oxide structure thus obtained using the alternative recipe differs from that obtained using the KEK EP method? Finally, how do all these variations affect the performance of a cavity? Clearly the surfaces of Nb SRF cavities can only be obtained after a set of surface treatment procedures. Variations in procedures create variations in performance and affect the final oxide structure of an Nb cavity. Our R&D tasks are therefore to understand how variations in procedures affect the final surface finish of an Nb cavity in terms of texture, oxide structure, chemical composition, contamination, etc. using various surface experimental tools.

We know the results quoted above are from different labs and their Nb samples were prepared in different sets of procedures. Even if their samples were prepared in exactly the same set of procedures, they might still have reached different conclusions if the starting Nb materials were different. The advantage of having an on-site SSL is that we can do all sample preparations and measurements using well-defined sets of procedures on exactly the same samples under the same measurement parameters. In this way, hopefully artificial variations of various sources can be minimized so that highly reliable experimental results can be obtained.

Our SSL was set up with the above indicated problems and ideas in mind. Whatever and whenever it was possible based on the available resources and manpower, I tried to build surface instruments with functions that could deal with typical problems in the field of SRF cavities. In the following, I try to give you some ideas about our systems.

SAM

Fig.1 shows a picture of the main observation chamber of the SAM. Due to the limited funding, this SAM was built based on parts purchased from different companies. Some parts were either donated by friendly colleagues or found in some corners of our TestLab. Major analytical heads and associated controllers were bought from ThermoVG Scientific. This system is similar in functions to the commercial product "MultiLab" of ThermoVG Scientific with some modifications based on our needs.

Once commissioned, the system should be able to perform following measurements: a) Auger Electron Spectroscopy (AES), b) depth profiling, c) SEM, d) surface chemical mapping - SAM, e) in-situ heat
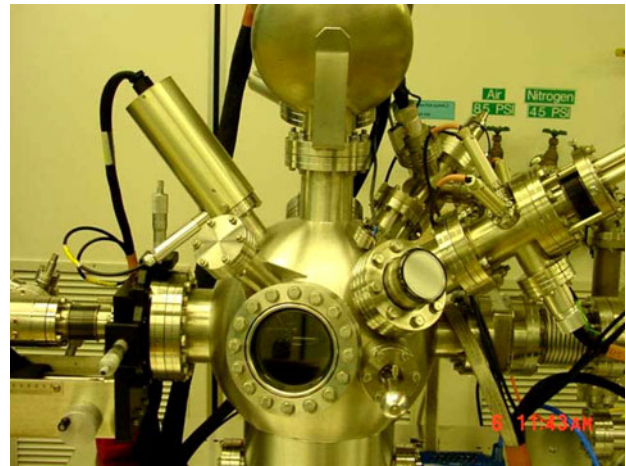

Fig.1: Observation chamber of our scanning Auger microscope.

treatments under different environments, and f) cooling down to –230 ^{0}C and heating up to 1000 ^{0}C. This system can be extended to include X-ray Photoelectron Spectroscopy (XPS) by incorporating a twin anode x-ray source into the main observation chamber.

The vacuum system of this SAM is a completely home-built one. Valves of the system are programmed to allow so-called "one-button" operations with interlocks. The operational pressure of the main observation chamber is designed to reach about 10^{-12} Torr. This system is powerful in studying low temperature baking effect, the effect of high temperature annealing, surface chemical compositions, difference in oxide structures at grain boundary and within a grain, etc.

SEM and EDX

Our SEM system is equipped with EDX (see Fig.2) to allow the identification of elements and their relatively concentrations of the materials observed under SEM. Since the typical probing depth of EDX is a few μm,

Fig.2 Scanning electron microscope equipped with energy dispersive x-ray analysis.

the revealed chemical information by this instrument is of bulk nature. This system is very useful for studying particulate collected from the inner surfaces of cavities as well as from other devices used in processing the surfaces of cavities as shown in the following Section 3. With suitable sample preparation, it can also be use to study topography and morphology of the surfaces and cross-sections of Nb samples such as, for instance, to inspect the quality of electron beam welding and to see the grain boundary shape.

The magnification of this SEM can reach 100,000X routinely. It can detect all elements with mass heavier than Boron with a sensitivity of ~0.5% atomic weight. It can also be extended to include the measurement of secondary electron yield.

TEM and STEM

This TEM was constructed based on components from two broken TEM systems. Some critical missing parts were either purchased or obtained free of charge from JEOL. Originally, I planned only to have the normal TEM functions from this system. In reality, however, the system (see Fig.3) can do not only normal TEM, but also STEM and SEM. It can be extended to have EDX, so that element analysis can also be done. A dark room has been built to accommodate this system.

The electron accelerating voltage of this system can be as high as 100 kV with nominal lattice resolution of 0.14 nm and point-to-point resolution of 0.3 nm. Magnification can be up to 800,000X.

With development of appropriate sample preparation procedures, this system can be powerful in studying the oxide structure on Nb surface in real and reciprocal spaces. It can be used for observing dislocation, grain structure, interstitial atom, stacking fault, secondary phase, grain orientation, crystal structure, texture, morphology etc. It can also be a very powerful tool in studying microstructure of Nb thin films.

Fig.3: Transmission electron microscope and scanning transmission electron microscope.

SFEM

As far as I know, this SFEM is one of the only two systems available in USA. The other system is at Cornell University, which has a smaller scanning size. Please see reference 5 for a detailed description of this system. An important feature of the SFEM is that it is coupled with our SEM and EDX systems. It uses the SEM chamber as a load-lock entrance for samples. Through appropriate marking, the coordinate of a sample can be transferred from the sample holder of SEM to that of SFEM, which allows an emitter to be checked at the same location before and after field emission.

The SFEM (see Fig.4) has the largest scanning diameter of 25 mm in the world. The spatial resolution of the system is 2.5 μm with an electric field gradient up to 140 MV/m. The apparatus is housed in a 10^{-9} Torr vacuum chamber with an in-situ heat treatment ability. Heating can be as high as 1400 ^{0}C. This system is very powerful and unique in studying field emission in Nb.
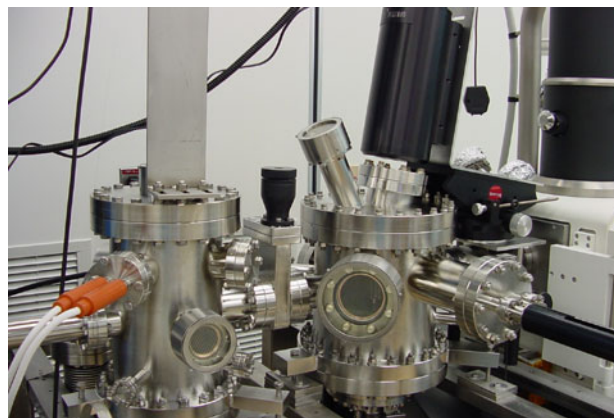

Fig.4: Scanning field emission microscope.

SIMS

This SIMS (see Fig.5) was built based on a home-made system which is more than two decades old. Before the system reached our hands, there were several intermediate owners. Presumably, each previous owner took some parts that they wanted from the system, ending up with an incomplete system in our hands. The worse thing is that we don't have any documentation on the system. It has been very difficult to make this system work. First of all, vacuum system was completely rebuilt. Ion pump was regenerated and two mechanical pumps and one turbo pump were added to the system. Several vacuum gauges and gauge controllers were installed. A getter pump located in ion gun assembly was also cleaned and regenerated. The gas inlet of the ion gun was redone and a digital needle valve for controlling gas flow was installed. Sample holder was modified and an ion beam monitor and detector unit was made. Ionizer near the top of mass analyzer assembly was replaced. A digital data acquisition unit was created so that we could get SIMS spectra using a computer

Fig.5: Home-made secondary ion mass spectrometry.

instead of using an x-y plotter. A preliminary program based on LabView has been written to allow control of the measurement process to a certain extend and to do data treatments on SIMS spectra. With some help from our electronic group, I expect that the program will be revised so that measurement of depth profile can be done using this computer control unit through GPIB interface.

Like other old systems, the system is unstable and may stop working any time although I have gained substantial understanding of the hardware of this system up to now. Currently, the system is functioning with manual operation mainly.

Despite many drawbacks as a home-built old SIMS system, it can still do normal SIMS measurements with a typical AMU resolution of unity or better which is
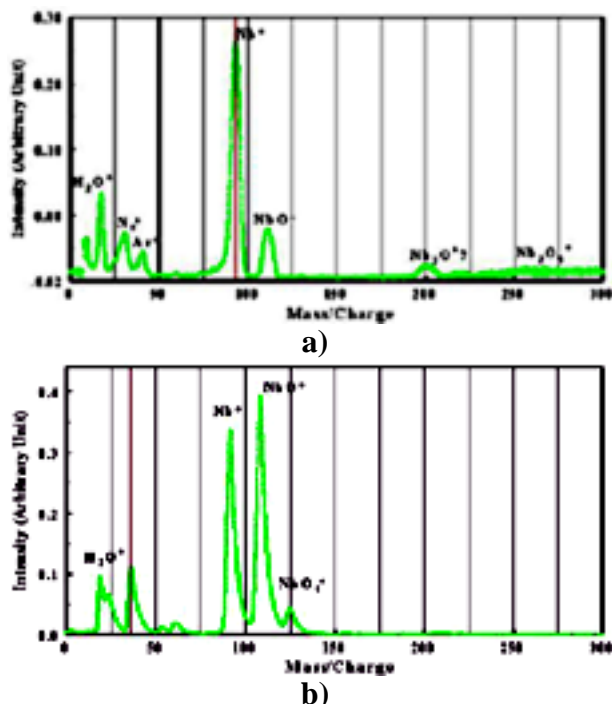
probably sufficient for our application. The mass analyzer of the SIMS has a quadrupole filter. This allows different peaks in an SIMS spectrum to be monitored rapidly in a depth profile experiment. Coupled with our advanced 3-D profilometer, a depth resolution better than 3 nm is possible. The system can also be used as a residual gas analyzer. Therefore background gas species can be determined before an SIMS experiment starts. It has the advantage of the ability to detect all elements, including hydrogen that is known to have a significant effect on the performance of an Nb SRF cavity. The nominal sensitivity of this system can also be as high as 1 ppm and better when the computer control unit is fully functional. Typical preliminary spectra obtained on an as-received and an electropolished Nb samples are shown in Fig.6. It is safe to conclude here that despite many drawbacks this system still contains all the essential features that we want to have from an SIMS for our applications.

3-D Profilometer

Our profilometer is one of the most advanced products from KLA-Tencor (see Fig.7). It has a vertical resolution as high as 8 nm and a guaranteed reproducibility of 0.75 nm. The instrument can scan over a large area of 80X200 mm and the resulted data can be plotted in three dimensions. This is not only a critical system in studying depth profile of Nb materials but also a very powerful tool in studying their surface topography and microstructure. Although surface roughness may not be the most important factor controlling the performance of an Nb cavity, it is still largely unknown as to what extent the performance is affected by roughness. It has been illustrated [6], however, that a sharp step at a grain boundary may produce local enhancement in magnetic field. An example of profilometer images is shown in Fig.8 where grains are clearly seen on a cross-section of an Nb sample.

a)

b)

Fig.6: Typical SIMS spectra obtained on a) as-received Nb, b) electropolished Nb.

Fig.7: 3-dimension large scan area profilometer with a vertical resolution as high as 0.1 nm
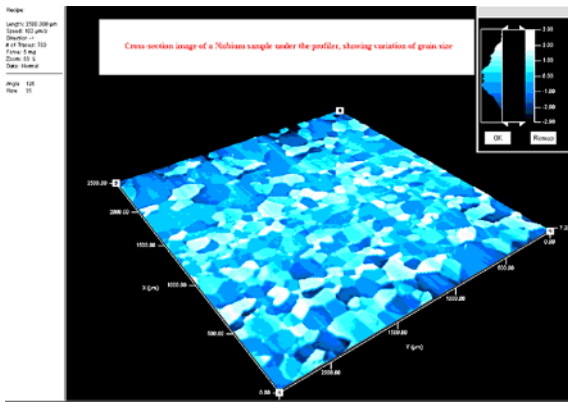
Fig.8: Typical profilometer image showing the grain structure on an Nb surface.

MOM

This MOM was made by Carl Zeiss (see Fig.9). It has all functions that a common MOM has and is a very useful tool for studying surface or cross-section microstructure of an Nb sample. To some extent, it is complementary to SEM due to its shallow depth of field.

Image capture and processing of the MOM are done by a computer. Magnification can go up to 8000 times routinely with a resolution about 150 nm.
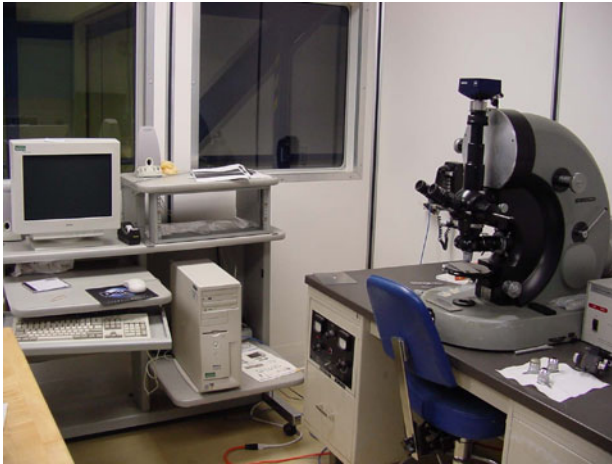

Fig.9: Metallographic optical microscope made by Carl Zeiss.

Sample Preparation Room

This sample preparation room is one of the best equipped in this area. It has all the facilities for doing sample preparation required by our SSL. A precision ion polishing system for the final step of TEM sample preparation will be purchased soon to complete the set-up of the sample preparation room (see Fig.10).

TYPICAL R&D PROJECTS

The SSL has been set-up mainly to study the surfaces of SRF cavities. However, the SSL has been supporting

Fig.10: Our sample preparation room.

other relevant activities across JLab also since its establishment, which is considered as one of the SSL duties. So far, surface measurements have been done for various groups, including Physics, FEL, Mechanical Engineering, Safety Group, Infrastructure Group, Injector Group, affiliated University Users, etc. In this report, I will include only some examples that are relevant to SRF science and technology.

Mechanism of Softening of SNS Nb Cavities after Heat Treatments at 800 °C

During a recent heat treatment on an SNS superconducting rf cavity made from niobium with RRR value larger than 250, it was found that the mechanical properties of the cavity degraded seriously after the treatment at 800 ^{0}C for a time period as short as one hour. Particularly, the treatment decreased the yield strength, which would result in a "soft" cavity that will easily suffer from deformation. This may eventually affect the performance of the cavity due to the difficulties in monitoring the resonance frequency and field profile inside it.

Several test plans were arranged to study the mechanism responsible for the degradation and to find appropriate conditions of heat treatments that could satisfy both requirements for acceptable mechanical properties and a low hydrogen concentration below which the Q disease would not appear. As part of these plans, the SSL did microstructure observations on high RRR SNS niobium samples obtained from batches from which SRF cavities were made. Through comparison with results of tensile and hardness tests, our cross-section and surface observations revealed that changes in microstructure, especially grain size, were the major reasons responsible for the degradation. The detailed report of this study was published in Reference 7 that is accessible through Internet worldwide at <http://tnweb.jlab.org/tn/2002/>.

Samples used in this study came from three different branches, namely SNS prototype niobium (denoted as

WCL), high RRR niobium (denoted as WC), and RRR niobium (denoted as TD). WCL and WC were purchased from one vendor and had RRR value of approximately 400 whereas TD was from another vendor with RRR value of about 300. Six samples were obtained from WCL and WC respectively, while five samples were obtained from TD. These samples were then cut into the standard shape according to the ASTM standards for tensile test (gauge length 3.81 cm and cross section 20.2 mm^2). Different heat treatments were performed on these samples as summarized in Table 1.

Table 1: Summary of heat treatments performed on SNS niobium samples (that show softening after heat treatment at 800°C for as short as one hour) and their grain sizes extracted from cross section MOM observation

Sample Number	Sample Code	Heat Treatment		Grain um
		Temperature (^{o}C)	Time (hour)	
1	WCLASR	As prepared		51.3
2	WCL6006	600	6	44.9
3	WCL60010	600	10	47.2
4	WCL7006	700	6	56.2
5	WCL8001	800	1	66.8
6	WCL8003	800	3	89.1
7	WCASR	As prepared		45.3
8	WC6006	600	6	46.4
9	WC60010	600	10	47.2
10	WC7006	700	6	46.8
11	WC8001	800	1	83.8
12	WC8003	800	3	94.0
13	TDASR	As prepared		
14	TD6006	600	6	
15	TD7006	700	6	
16	TD8001	800	1	
17	TD8003	800	3	84.5

After tensile test, samples were cut from the head areas at least 5 mm away from the tensile test gauges using a diamond cutter (LECO VC-50). Mechanical and chemical polishings were done on these samples using a semi-automatic Buehler Variable Speed Grinder-Polisher and a standard BCP method before optical or scanning electron microscope observations. Since high RRR niobium is relatively soft, it was quite difficult to get a final surface or cross-section finish without any scratches on it. Detailed mechanical polishing procedure had to be adjusted according to the history of a particular sample in order to get a satisfactory surface finish. Final etching to reveal microstructure was done using a mixture of H_2SO_4, HNO_3, HF, and H_2O with appropriate ratio between them according to the procedure of heat treatment. Cross-section images were mainly taken using the MOM whereas surface observations were done using both the MOM and SEM.

From our accumulated knowledge about metals, we know that plastic deformation of a polycrystalline metal normally takes place via one of the two simple processes, namely slip or twinning. For a metal of b.c.c. structure such as niobium, twinning is relatively more difficult and deformation occurs mainly through slip. It is well-known that slip can be triggered by activating the movement of either screw or edge dislocations in the grains of the polycrystalline metal. To complete a slip along a particular direction, the movement of dislocations has to be propagated from one grain to the other. Therefore, the more the grain boundaries, the more difficult the slip is to occur. As a result, the more difficult the metal is to yield. Quantitatively, the yield strength of a polycrystalline metal is related to its grain size according to the following well-known relationship:

$$\sigma_y = \sigma_i + 1.414\sigma_D l^{1/2} d^{-(1/2)} \quad .$$

Here σ_y is lower yield stress, σ_i the shear stress resisting the movement of dislocations across a particular slip plane, σ_D is the shear stress to unpin a dislocation, l is the distance from piled-up dislocations at the head of the plastic front held up by the grain boundary to the nearest dislocation sources (Frank-Read sources) in the next grain, and d is the diameter of grain. Naturally, the primary information that needs to be extracted from microstructure measurements is grain size.

Our study [7] reveals that grain size extracted from surface measurement is insufficient for explaining the softening. For Nb materials, it is often the case that microstructure is not homogeneous. Therefore, to explain bulk mechanical properties cross-section study has to be done although it is much more time consuming. Fig.11 shows a plot of lower yield stress as a function of the inverse of the square root of the diameter of grain for all samples that are fully crystallized and have clearly defined grain boundaries. From this figure, we may extract the following information: a) Generally, yield strength decreases with increasing grain size. b) Regular
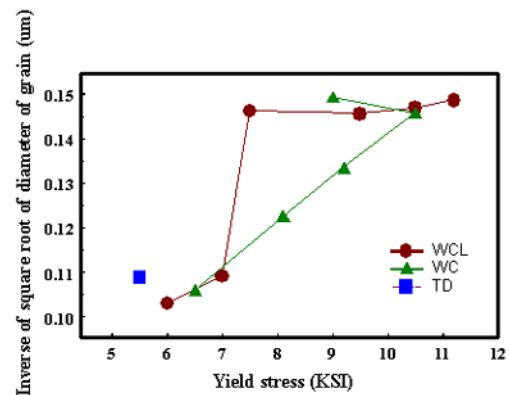

Fig.11: Yield stress measured as a function of grain size that is extracted from cross section microstructure observation.

linear dependence between lower yield stress and diameter of grain seems to be followed only when the grain growth mechanism is the same. Undoubtedly, mechanical properties of metals are mainly related to the crystal and micro structures of the metals. This is especially true for the samples under study, since they are highly pure niobium. From Fig.11, we can see the average grain sizes in WC and WCL samples are in the order of 85 μm after the heat treatment at $800^{o}C$ for one hour while the average grain sizes of Sample No. 1 and 8 are only 51.3 μm and 45.3 μm respectively. Although we could not get an average grain size for Sample No. 16, some of the grains in this sample are abnormally large (see Fig.12 for an example). Therefore increase in grain size is the major mechanism responsible for the softening. This does not necessarily imply that we can neglect completely the effect from possible impurities inside the niobium which can serve as pinning centers for dislocations, creating therefore many shorter Frank-Read dislocation sources. Since the force required to activate the dislocation sources is inversely proportional to the length of the Frank-Read sources, this leads to a higher yield strength. For pure niobium, hydrogen is known to be readily dissolved and can diffuse into the bulk at a speed of approximately several mm/hour at room temperature if the oxide layer on the niobium surface is defective. Hydrogen atoms in niobium act in a similar way as those of carbon and silicon in ion. Heat treatment at $800\ ^{0}C$ also removes hydrogen away from niobium substantially, which may also contribute to the softening. What the significance of the contribution from hydrogen removal is remains unclear at this moment and requires further study.
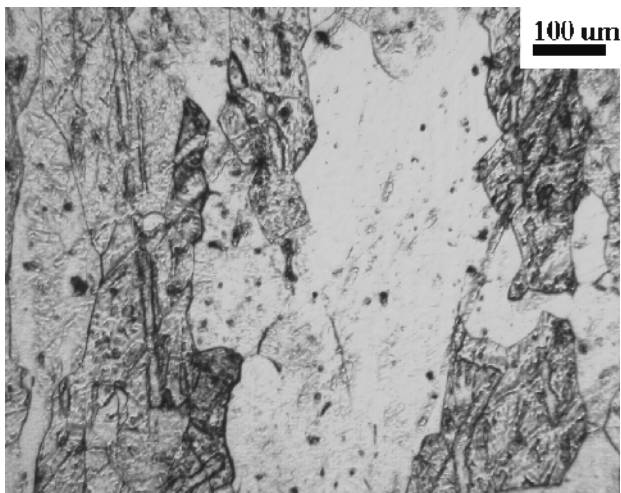

Fig.12: Typical MOM image obtained on Sample No. 16, showing some super-sized grains due to the heat treatment at $800\ ^{0}C$ for one hour.

TEM Observation of Surface Oxide Structure of Nb

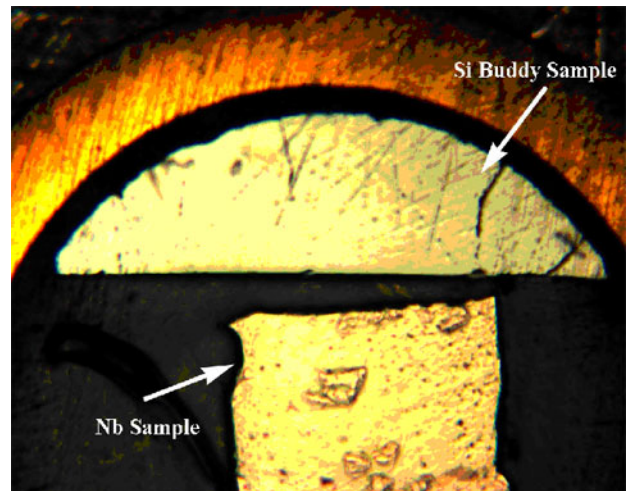As discussed in the introduction part of this paper, it is

Fig.13: A sample preparation procedure for doing TEM cross-section observation on the surface oxide structure of Nb has been developed [8].
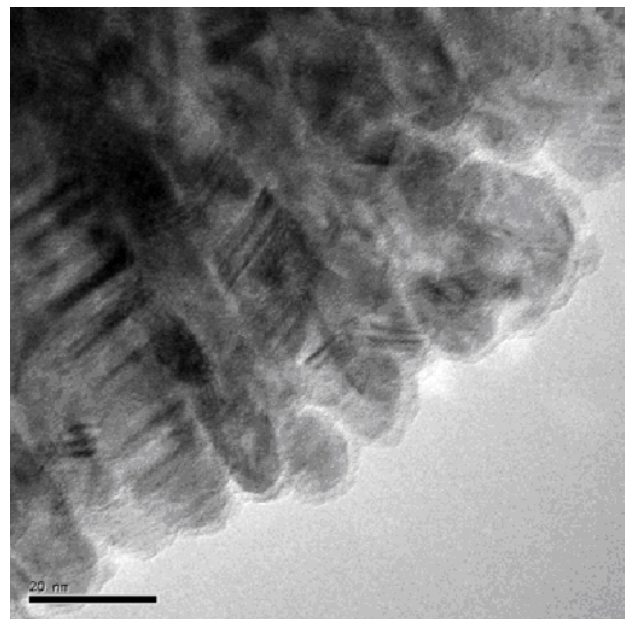

Fig.14: Typical TEM cross-section image on the surface of an electropolished Nb sample.

well-known that Nb surfaces are covered by an oxide layer. On the very top of the oxide layer, it is Nb_2O_5. In between Nb_2O_5 and pure Nb, there are sub-oxides with various oxidation states. Some of the sub-oxides may be metallic or semiconducting, and they are either not superconductors or superconductors with low transition temperatures, which may be the source of energy losses at low temperatures. Therefore knowing the exact chemical formulae of the sub-oxides is the first step towards controlling them.

Conventionally, the surface oxide layer is studied by XPS or sometimes SAM and SIMS. Chemical information and the thickness of the oxide layer are "indirectly" extracted from XPS, SAM and SIMS data. I

have been exploring a new approach to study the oxide layer. We know that nowadays TEM is quite powerful. A normal TEM with an accelerating voltage of 100 kV can, in principal, be able to resolve a spacing of 0.3 nm. With a suitable sample preparation procedure and an experienced TEM operator, it should be possible to see the oxide layer directly in real space. Furthermore, oxidation information of the oxide layer should be able to be extracted from electron energy loss spectroscopy function of TEM.

Recently, a procedure has been developed at JLab to prepare Nb samples for cross-section TEM observation [8]. A typical example of an Nb sample prepared by the developed procedure is shown in Fig.13. Preliminary high resolution TEM observation has been done on the sample. A typical TEM image is shown in Fig.14. Much more work is needed before meaningful conclusions can be drawn from this study.

Electropolish on Nb Using an Alternative Electrolyte Composition

It is well-known that a thickness more than 150 μm has to be removed from the surface of an Nb cavity in order to have good performance. Conventionally, BCP was the standard technique used in SRF community for getting this done as the final step of chemical treatments. However, it has become more and more popular to use electropolish (EP) as the final step of chemical treatments on Nb SRF cavities after the pioneer investigation [9] done at KEK. To the best of our knowledge, all the reported electropolishing on Nb SRF cavities has been done using exactly the same electrolyte recipe first used by Diepers et al. [10] and subsequently adopted by Saito and his coworkers [9].

As part of our R&D effort, JLab has initiated a study to explore other alternatives. Some preliminary results have been published in Reference 4. We have tried various recipes including the one used by KEK. One particular recipe, consisting of appropriate mixture of lactic, sulfuric, and hydrofluoric acids, has shown very encouraging results in terms of surface smoothness. I used this recipe for EP on Nb about more than six years ago for the purpose of getting a smooth surface for making high temperature superconductor devices. This type of acid mixture for electropolishing on Nb was first briefly described by Monti [11] in a French journal *Métaux Corrosion Industries* in 1958 for corrosion protection, although the ratio of the mixture described there is not the same as the one that I used six years ago.

Generally speaking, any electropolishing process can be characterized by plotting its anode current density (I_d) against its voltage (V) drop between cathode and anode. The detailed shape of each I_d vs. V curve depends on the configuration of each polishing setup and is largely influenced by the distance between cathode and anode. For a simple EP setup as shown in reference 4, the I_d vs. V curve was determined as Fig.15. Through surface observation using MOM and SEM, four regions as
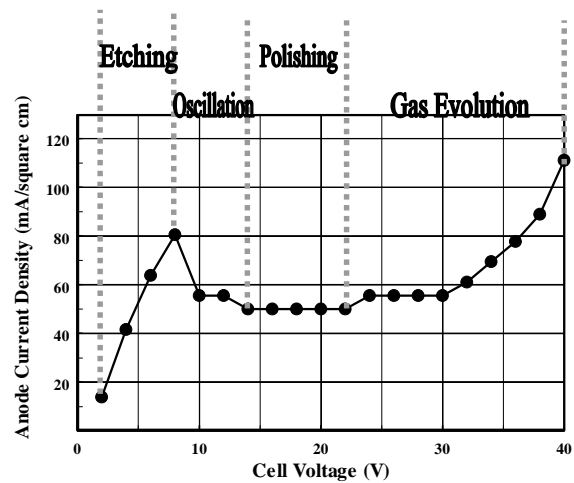

Fig.15: Anode current density measured as a function of applied voltage when using the alternative acid mixture for electropolishing on Nb. Four areas are identified.
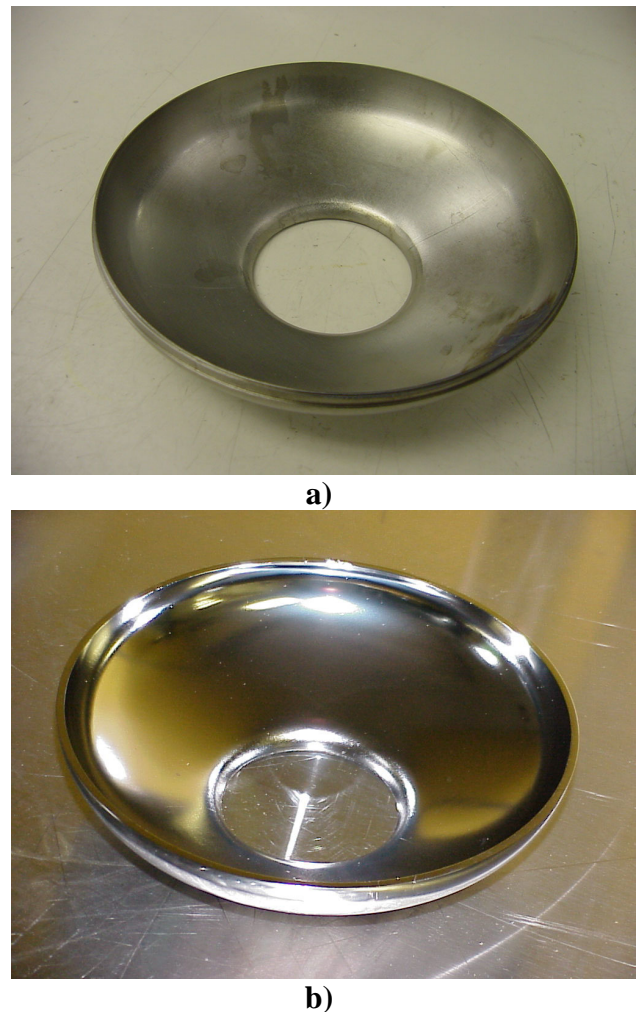

a)

b)

Fig.16: Pictures of a half-cell Nb cavity a) before electropolishing and b) after electropolishing for 30 minutes using a special shape of cathode which has a more uniform distance between cathode and anode.

Fig.17: One of the setups for electropolishing on Nb single cell cavities.

indicated in Fig.15 were identified. The optimum polishing conditions were determined [4] as a cell voltage of 23 V and an anode current density of 50 mA/cm^2. Since we are going to use current density as the control parameter for polishing, we can discard the voltage. Employing the parameters obtained with the help of surface observation instruments in the SSL and the alternative EP recipe, a half-cell Nb cavity was polished (see Fig. 16b). The polished surface appears to be rather smooth and shiny. This polishing process was named [4] as "Buffered ElectroPolishing" (BEP). It seems that BEP is a very promising method for treating Nb cavities.

More setups for EP on Nb single cell cavities have been made (see Fig. 17 for an example) by J. Mammosser, L. Phillips, and A.T. Wu and further investigation will be followed.

Particulate Analysis on Samples Collected from Nb Cavities, High Pressure Water Rinse Filters, Scroll Pump, and Vacuum Pump Line

Field emission (FE) loading has been and still is a major obstacle for Nb SRF cavities to achieve high accelerating gradient at JLab. Contaminants of micron or submicron sizes on cavity surface are believed to be mainly responsible for it, although surface geometrical scratches, imperfections, damages may also contribute to FE. Those micron or submicron-scale contaminants may enter a single or multiple-cell cavity during any one of the various cavity handling, transporting, treating, assembling, and pumping processes. Particulate control has become an important issue needed to be addressed at JLab.

As the first step for investigating the cause of contamination, particulate samples have to be collected and then analyzed by SEM and EDX to check their shapes and compositions. Recently, a simple particulate collection technique using a double sided carbon tape (DSCT) has been developed (see reference 12 for details

which is accessible via internet worldwide at <http://tnweb.jlab.org/tn/2002/>). This technique has been applied to collect samples from cavity surfaces, surfaces of disassembled high pressure water rinse (HPWR) filters, surfaces of a disassembled scroll pump used for pumping cavities, and surface of a vacuum pump line for pumping cavities. For the detailed procedure, please see reference 12. Sustained SEM and EDX analysis requires dedicated skilled effort. These measurements were time consuming. Some of the results of this study from cavity surfaces and HPWR filters were published in reference 12. Here let me report briefly the results obtained on samples from the scroll pump and the vacuum pump line.

The results for the front side of the vacuum pump line that is mounted to the pump flange of a cavity are summarized in Fig.18. Eleven elements were detected from twenty particles analyzed using SEM and EDX on a DSCT. Interestingly, the most frequently found element was again Si. Most of the other elements such as S, Cl, Fe, Ca, Ti, K, Al, and Na that were found on the JL009 cavity [12] also appeared here. On the other hand, more elements were detected on the rear side of the vacuum pump line where it was attached to a vacuum
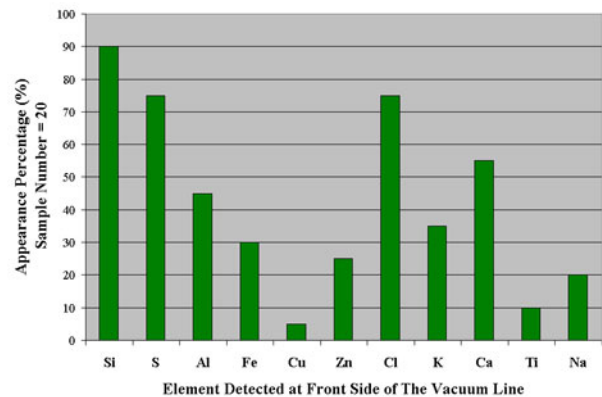

Fig.18: Element appearance frequency detected at the front side of vacuum pump line based on 20 particles analyzed using SEM and EDX on a DSCT.
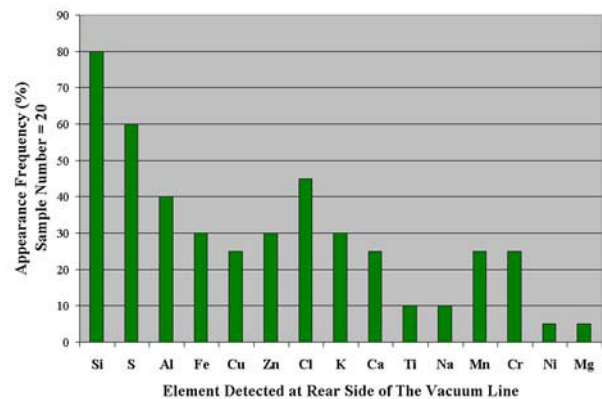

Fig.19: Element appearance frequency detected at the rear side of vacuum pump line based on 20 particles analyzed using SEM and EDX on a DSCT.
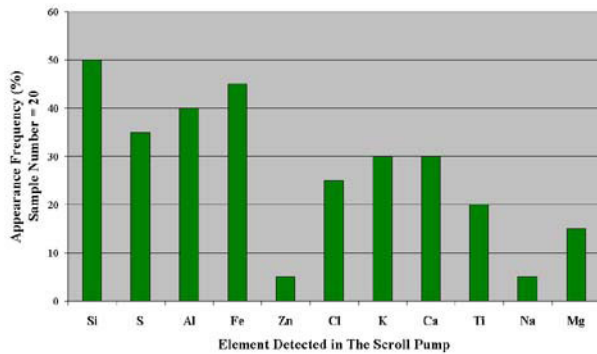
Fig.20: Element appearance frequency detected on the interior surface of a scroll pump based on 20 particles analyzed using SEM and EDX on a DSCT.

pump as shown in Fig.19. Surprisingly, Si is the most abundant element as in the other cases. Four more elements Cu, Cr, Ni, and Mn were detected here. Among them, all except Mn were also found on the JL009 cavity.

Normally, cavity vacuum roughing down at JLab is done using Varian's TriScroll pumps. We happened to find that there were quite some debris inside a scroll pump assembly when the pump was disassembled during a failure, although the pump was supposed to be clean. Naturally, we would like to know what the particles are since they may have something to do with the particles found in Nb cavities. Particles on the internal surface of a scroll pump were collected using the technique described in reference 12 and then analyzed by SEM and EDX as shown in Fig.20. Fig.20 is similar to Fig.18 except Mg and the appearance frequencies for some elements. Most noticeable is the element Fe that may come from the bearing material 52100 steel of the pump. The lubricant used for the moving scrolls is krytox that contains mostly Si and a sodium-based additive for corrosion prevention. The moving scrolls are made of hard-coated Al (6061 alloy) that is the third most frequent found element in Fig.20. The 6061 alloy has some Mg and Si in it. Furthermore, the hard-coating is produced in a sulfuric acid bath, hence, perhaps, the appearance of S. Our SEM observation also found some large organic particles of a few hundred micrometers. A typical example is shown in Fig.21. It is highly plausible that the large particles are deformed polyethylene and Teflon that are used for tip seal in the scroll pump.

This study strongly suggests that the scroll pumps do generate particulates that may find their way into our cavities. Some common elements such as Si, Al, Fe, S, and Na found in the scroll pump, front and rear sides of the vacuum pump line, and the cavity [12] may partially come from the scroll pump and the contaminated vacuum pump line. If we assure that the vacuum pump line is clean initially, it may get contaminated from both cavities and scroll pumps after being used for a long time. Then the line itself becomes a source of particulate generation. Therefore, it may be useful to install a particle trap or filter at the front side of the vacuum pump line to prevent the possible migration
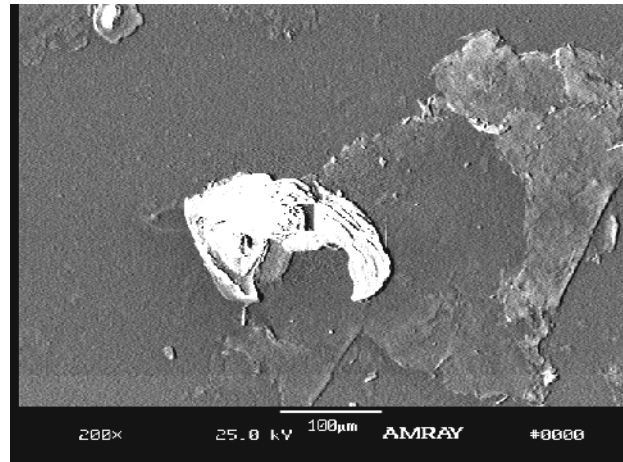

Fig.21: Typical organic particles detected on the interior surface of a Varian Triscroll pump by SEM.

of particles from the vacuum pump line to cavities. It is also beneficial to clean the vacuum pump line periodically so that contamination in the vacuum pump line can be minimized. Due to a relatively large amount of particles found in the vacuum pump line, one must be careful when the pump line is mounted to a cavity since mechanical vibration is know to activate the movement of particles. More study is needed to confirm the findings made here.

CONCLUSION

A surface science lab (SSL) has been established at JLab to study the surfaces of superconducting radio frequency (SRF) cavities. The characteristics of the each of the available facilities in the SSL are described briefly. Some typical examples of the R&D projects on Nb SRF cavities carried out through the partial use of the facilities in the SSL in the past couple of years while establishing the SSL are briefly described and reported. This paper may serve as the basic knowledge regarding our SSL for possible future collaboration on mutually interested R&D projects.

ACKNOWLEDGMENT

how I can survive and still be here working on the SSL without their valuable encouragements and advices during my desperate periods along the course of setting up this SSL. They also donated some parts and components. I would like to thank Joe Preble and Haipeng Wang who offered their hands at the time when I desperately needed help. Thanks are also due to Hari Ariti, Jean Delayen, Robert Rimmer, and Charles Reece for their support and encouragement. I am also indebted to Peter Kneisel for his words during my down periods that warmed my heart. Valuable support and assistance from Curt Hovater, John Musson, Tom Power, Hai Dong, and Christiana Grenoble are highly appreciated. Finally I would like to thank three students David Smith, Robert Ike, and David Erickson. Without their share of my workload and hard work, I would not have had the time to sit down and write this paper. It is virtually impossible to go through all the friendly colleagues who supported the SSL morally or physically in the past couple of years in the limited space here. Thank you all!!!

Samples from the scroll pump and the vacuum pump line as discussed in Section 3.4 were provided by John Mammosser.

Thank Charles Reece for critical review of this paper.

REFERENCES

1. C. Reece, J. Mammosser, L. Phillips, A.M. Valente, T. Wang, A.T. Wu, Correlation studies between material and surface characteristics and superconducting properties of Nb for RF cavities, this proceedings

2. C. Antoine, A. Aspart, S. Regnault, H. Safa, Surface studies: method of analysis and results, Proc. of the 10th workshop on RF superconductivity. (2001) P272

3. Qing Ma and R.A. Rosenberg, Thermal and electron-beam irradiation effects on the surfaces of niobium for RF cavity production, Proc. of the 10th workshop on RF superconductivity (2001) P368

4. J.R. Delayen, J. Mammosser, L. Phillips, A.T. Wu, Alternate electrolyte composition for electropolishing of niobium surfaces, Proc. of the 10th workshop on RF superconductivity (2001) P499

5. T. Wang, C. Reece, R. Sundelin, DC scanning field emission microscope integrated with existing scanning electron microscope, Review of Scientific Instruments 73 (2002) P3215

6. J. Knobloch, R.L. Geng, M. Liepe, H. Padamsee, High-field Q slope in superconducting cavities due to magnetic field enhancement at grain boundaries, Proc. of the 9th workshop on RF superconductivity (1999)

7. A.T. Wu and G. Myneni, Effect of Heat Treatments on Microstructure of High RRR SNS Niobium, (2002) JLab-TN-02-027

8. A.T. Wu, to be published

9. K. Saito, Y. Kojima, T. Furuya, S. Mitsunobu, S. Noguchi, K. Hosoyama, T. Nakazato, T. Tajima, and K. Asano, Proc. the workshop on RF superconductivity, (1989) P635

10. H. Diepers, O. Schmidt, H. Martens, F.S. Sun, Phys. Lett., 37A (1971) P139

11. H. Monti, Métaux Corrosion Industries, 33 (1958) P481

12. J. Mammosser and A.T. Wu, Preliminary Results from Cavity and Process Particulate Collection, (2002) JLab-TN-02-025