ATOM PROBE TOMOGRAPHY STUDIES OF RF MATERIALS*

J. Norem#, ANL, Argonne, IL, 60439 USA
P. Bauer, Fermilab, Batavia, IL 60510, USA
J. Sebastian, D. N. Seidman, Northwestern University, Evanston IL, 60208 USA

Abstract

We are constructing a facility that combines an atom probe field ion microscope with a multi-element in-situ deposition and surface modification capability. This system is dedicated to rf studies and the initial goal will be to understand the properties of evaporative coatings: field emission, bonding, interdiffusion, etc., to suppress breakdown and dark currents in normal cavities. We also hope to use this system to look more generally at interactions of surface structure and high rf fields. We will present preliminary data on structures relevant to normal and superconducting rf systems.

INTRODUCTION

Although breakdown in rf systems has been studied for many years, the process occurs so rapidly and is generally so destructive that it has been very difficult to determine the trigger mechanism, or how the properties of the surface contribute to the trigger process. A number of experiments, looking at the production of field emitted electron beams, have shown that local asperities on the surface of these structures operate at very high local fields, from 5 – 10 GV/m, fields that produce tensile stresses greater than the macroscopic tensile strength of the materials. Although this environment cannot be easily studied using rf systems, there is extensive experience in materials science using Atom Probe Tomography, and Field Ion Microscopy to study surfaces that involve surface fields of this magnitude.

We are starting a program that will look at the near surface region of metals, primarily copper and niobium, which should give us useful information on failure modes of high field cavities. We also hope to obtain useful information on the properties of a variety of surface treatments that are relevant to accelerator cavities. The initial problems we will be studying are field emission induced backgrounds in muon cooling systems, enhanced breakdown and stability problems with high magnetic fields and contamination and Q slope in superconducting rf materials.

In addition to analytical sensitivity, spatial resolution and excellent statistics, this technique offers a number of advantages in studying rf problems. The surface fields seen in cavities seem to be well within the range used in atom probe tomography. All ions from hydrogen to uranium can be detected with equal detection efficiency. A wide range of surface phenomena are accessible. This method of analysis proceeds at a high enough rate so that surface contamination from the vacuum can, in principle, be understood.

While the environment seen by samples is very similar to that seen in highly stressed areas of cavities, materials used in rf systems, (primarily copper and niobium) are not commonly studied using this method, and there is limited experience with surface and near surface analysis using these techniques.

ATOM PROBE TOMOGRAPHY

Atom probe tomography, which evolved from atom probe field ion microscopy, can produce images of many atoms in materials has developed rapidly in the past 10 years[1][2][3]. These devices have been constructed for many years by materials scientists for their own use, however the field is rapidly evolving with the appearance of the first commercial device, the LEAP Microscope, by Imago Scientific Instruments, which combines high resolution, sensitivity and data collection rate [4][5]. We have been using the instrumentation at the Northwestern University Center for Atom Probe Tomography, lead by David Seidman [6].

Figure 1: The Imago LEAP Microscope

Atom probe tomography uses high electric fields to field evaporate atoms as charged ions one-by-one from the surface of highly sharpened needles. Using mass spectroscopy and position sensitive detectors, the mass and charge of these ions can be measured and the original position from which the atom field evaporated can be determined, which allow reconstruction of the original material using computer. The LEAP tomograph can produce images of ~72 M atoms/hr, with position resolution less than 1 Angstrom and mass resolution on the order of $M/\Delta M \approx 500$. The machine is shown in Fig 1 and the basic principle is shown in Fig 2. Pulsing the

*Supported in part by the NSF and US DOE
norem@anl.gov

sharp, needle shaped target causes atoms to be evaporated as ions, which can be identified by time of flight. The magnification is primarily a function of the geometry, and is determined by ratio of the tip to detector distance (~10 cm), to the tip radius, (~30 nm), giving values greater than 10^6.

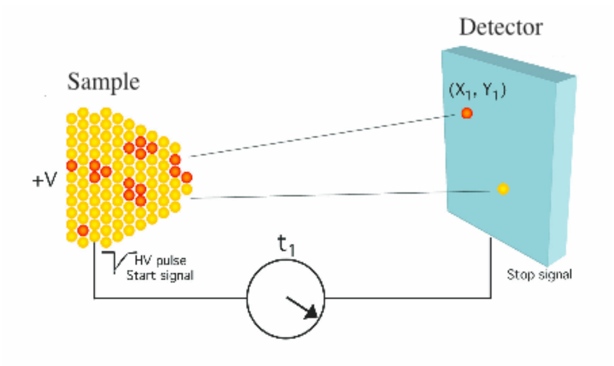

Figure 2: The principle of the atom probe tomography

In addition to work with the LEAP tomograph, we are constructing a facility to use atom probe tomography to study coatings and field emission in-situ. This will permit study of the properties of coatings that are relevant to the rf environment such as the field emission coefficient, bonding, interdiffusion, hardness and contamination.

INITIAL RESULTS

We are looking primarily at three problems: 1) optimizing copper surfaces for low field emission, 2) studying mechanisms which are relevant to rf breakdown, and 3) understanding the niobium surface present in superconducting rf systems and how it depends on surface treatments. Our initial experiments have been productive and we have found that data from the surface regions are accessible and relevant, and the technology seems flexible enough to explore a relevant parameter space; there have been some challenges, however.

It has been necessary to understand how copper and niobium samples perform, and initial work has been directed at sample preparation and observing how copper and niobium initially behave during field evaporation. Since copper cavities run at or above room temperature, while the resolution of atom probe systems requires low temperature operation, (20 to 80 K), it has been necessary to verify that these devices can operate usefully at high temperature.

Sample preparation is another challenge. Atom probe samples for metallurgical studies are made by etching thin wires in a drop of acid, in air, under a microscope. This produces a surface that can be contaminated by the acid, air and transport to the machine. In practice we find that the acids and techniques used to produce and sharpen samples are similar to those used for superconducting systems, (electropolishing and buffered chemical polishing), so the surface treatments used should be relevant to the rf environment. We used 10% HF and 90% HNO_3 for electropolishing, and a mixture of HF, HNO_3 and H_3PO_4 for buffered chemical polishing.

Initial Results from Copper

The primary uncertainty in operation with copper is whether the system would be able to produce useful data at room temperatures. Using a sample made of a random copper wire, we were able to sharpen and operate samples at 300 K, using standard techniques. Figure 3, a screen shot from the LEAP tomography during this operation, gives the initial stages of a run, showing an initial period of running with low voltage and uneven evaporation rate, ultimately terminated by a "flash", followed by a period where the voltage is rising with no field evaporation, and a long period of stable operation with stable evaporation at two preset rates. We estimate that the surface field during the final period of operation in on the order of 30 GV/m, based on the mass plot, which shows only $^{63}Cu^+$ and $^{65}Cu^+$. It is possible that the initial operation period may show emission typical of the behaviour of asperities in cavities at surfaces fields of 5-10 GV/m, and may be associated with oxides or surface contamination.

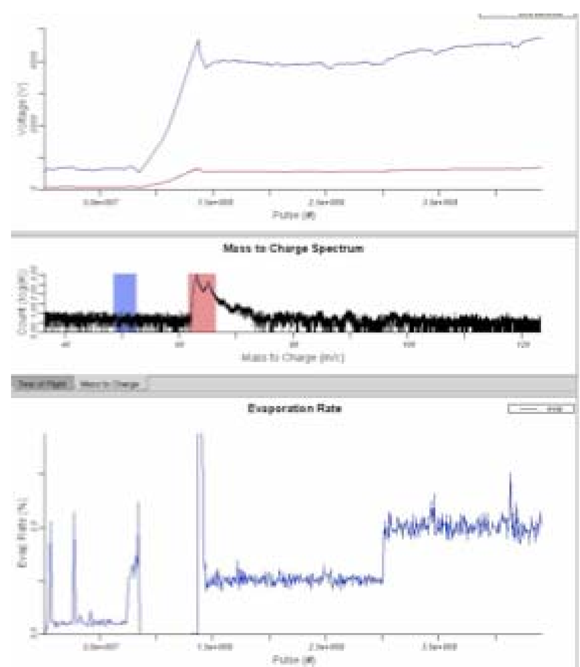

Figure 3: A screen shot of V, m/q and evap, rate for Cu.

Niobium

While the etching required to produce sharp samples for atom probe tomography can be done using similar techniques to those used to prepare surfaces for rf cavities, the nature of the surface that was produced was unknown. In initial tests, we found that the niobium surface produced with a buffered chemical polish produced a useful sample tip, and even the initial data from the extreme surface seemed to be relevant. An example of surface data from an early run is shown in Fig 4, which shows a few fluorine atoms from the buffered

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chemical polish on the surface of a much larger number of niobium atoms.

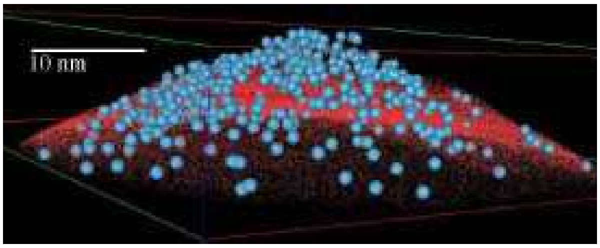

Figure 4: Fluorine atoms (blue) on niobium (red).

With both niobium and copper samples the ion field evaporation in the early stages was uneven and complex. A mass spectrum of ions emitted from the surface, (Fig 5), shows a variety of complex ions in different charge states being field evaporated in the early stages of analysis. We have made preliminary measurements of the concentrations of niobium, oxygen, hydrogen and a variety of contaminants as a function of distance from the surface, and are continuing to refine and develop surface preparation techniques.

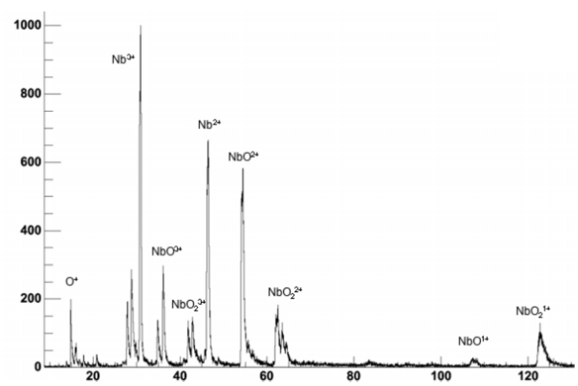

Figure 5: Ions initially field evaporated from niobium.

The goal of this work is to make systematic studies of how the near surface region depends on the temperature, impurity density, oxygen and hydrogen concentration and other variables to help understand questions like Q-slope in superconducting rf materials. We have preliminary measurements of the Nb/O ratios that seem to show how the density of niobium rises smoothly through the oxide layer until the oxygen density is only about 5 - 10%. There have been initial problems with samples flashing.

In-Situ Coating Tests

The system we are constructed is shown in Fig 6. The system uses a four-element coating head produced by MANTIS, operating with a pulsed laser atom probe system built at Northwestern [7]. The apparatus will permit measurements of field emission from samples with well-understood surface properties, with the ability to control and modify the surface using a variety of coating materials and techniques. This should allow us to understand the limits of control one has on the surface of cavities in rf environments.

One challenge of coatings is that the tensile stresses on asperities on the surface can be large enough to damage coating surfaces. This facility will permit us to understand the coating process on an atom-by-atom basis and to understand the physical metallurgy at the most fundamental level. Our initial tests will measure the stability and properties of high work function, low field emission, coatings which should help reduce x ray production from cavities and perhaps also retard breakdown in high magnetic fields.

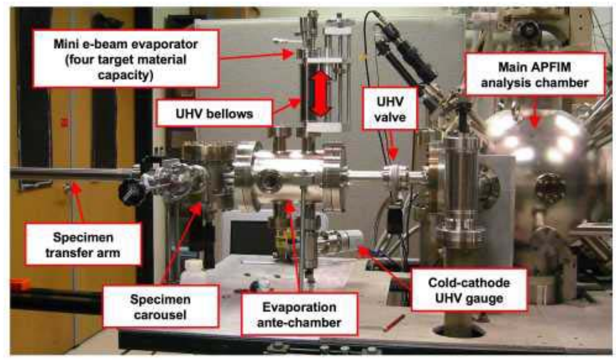

Figure 6: The coating test assembly.

SUMMARY

The near surface region of rf materials, and mechanisms operating at high electric fields have never been well understood. We are developing techniques using atom probe tomography, which will enable us to understand the nature of the surface and near surface environment in these systems and the limits of the control one can have using different materials and techniques. Surface analysis of copper and niobium has not been performed in these machines. Initial tests have shown that the system has very impressive sensitivity, resolution and data rates, and we are developing sample preparation techniques and gaining experience with these metals. We have discovered, however, that the surface regions of these materials are very complex and challanging to study because of a large number of compounds, oxides and contaminants that are present, which can have different mechanical properties from bare metals.

We would like to thank M. Zisman of LBNL for encouraging this work.

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