SURFACE STUDY OF Nb/Cu FILMS FOR CAVITY DEPOSITION BY ECR PLASMA*

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Abstract

Niobium (Nb) thin film deposited on copper (Cu) cavities through electron cyclotron resonance (ECR) plasma appears to be an attractive alternative technique for fabricating superconducting radio frequency cavities to be used in particle accelerators. The performance of these obtained Nb/Cu cavities is expected to depend on the surface characteristics of the Nb films. In this report, we investigate the influence of deposition energy on surface morphology, microstructure, and chemical composition of Nb films deposited on small Cu disks employing a metallographic optical microscope, a 3-D profilometer, a scanning electron microscope, and a dynamic secondary ion mass spectrometry. The results will be compared with those obtained on Nb surfaces treated by BCP, EP, and BEP.

INTRODUCTION

Niobium (Nb) coating on copper (Cu) cavities is an attractive technique for fabricating superconducting radio frequency cavities in comparison with other techniques based on bulk Nb for the application in particle accelerators, due to a significant material cost reduction and a better thermal stability at temperatures near 4.2 K. However, the commonly used cylindrical magnetron sputtering technique for depositing Nb film as developed by CERN for LEP2 suffers from a rapid increase of residual resistance [1] at accelerating gradients above 15 MV/m at 1.7 K, leading to the degradation in the quality factor Q of the coated Cu cavities. One of the major drawbacks of cylindrical magnetron sputtering technique is that the arrival angle of Nb flux on the inner surface of a Cu cavity is not constant. The angle varies from beam pipe to equator. In some cases [2], the angle variation can be continuously from 0^o to 90^o. Simulation using 3-D film growth code SIMBAD [3] and real experiment [4] have demonstrated that films grow at oblique incident angles are highly porous and the porosity can be as high as 80% [4] due to the well-known shadowing effect. Besides the adatom mobility of this technique is limited and the distance between cathode and anode is variable, porous columnar growth of Nb is inevitable in some areas on the inner surface of a Cu cavity. Therefore surface of the Nb film can be very rough that may result in an increase in surface residual resistance [5]. Most importantly, perhaps, is that porous columnar microstructure may provide an easy channel for oxygen to penetrate into Nb film, leading to the formation of some sub-oxides that may not be superconductors or may be superconductors at lower temperatures than that of Nb and causing RF losses. These drawbacks can, in principal, be overcome by an energetic thin film deposition technique [6] through generating Nb plasma via the electron cyclotron resonance method [7,8].

In this paper, we report our preliminary results on surface characterization on Nb films deposited on flat Cu coupons using the energetic deposition technique [6]. The aim is to study how the variations of deposition energy can affect morphology and surface oxide layer structure of Nb films, in preparation for actual deposition on a 500 MHz single cell elliptical Cu cavity. Due to the limited space, results are briefly presented.

EXPERIMENT

Four samples were used in this study. They were electro polished Cu disks of 2.5" diameter and coated with Nb films using the energetic deposition technique [6]. Deposition was done at a base pressure of $3X10^{-6}$ (Pa). About 150 μm was removed by electro polishing (55% H_3PO_4 and 45% $C_4H_{10}O$) from each Cu disk. Deposition energy was controlled by biasing the Cu disks with voltages of 60, 70, 80, and 90 V respectively (for simplicity, the corresponding samples are denoted as 60Cu, 70Cu, 80Cu, and 90Cu). A retarding field energy analyzer mounted on the substrate without bias voltage showed that arriving Nb ions had a kinetic energy of 63 V with energy spread of 20 eV full width at half maximum.

Optical measurements were done using a computer controlled metallographic optical microscope (MOM) made by Carl Zeiss. An Amray 1830 scanning electron microscope (SEM) was using for surface observation at larger magnifications. Surface roughness of the samples was quantitatively evaluated by a KLA-Tencor P-15 profilometer. This profilometer can do 3-D scans over an area as large as 80X200 mm^2 with a guaranteed vertical repeatability of 0.75 nm. Surface chemical information and depth profile were measured via a home-made dynamic secondary ion mass spectrometer (SIMS). For the details about the characterization devices used in this study, please see reference 9.

RESULT AND DISCUSSION

A typical sample is shown in Fig.1. Arrows in Fig.1 indicate Cu surface that is used for MOM observations to check the smoothness of the Cu substrate. Generally speaking, all surfaces of the electro polished Cu disks look similar under MOM. Observations at larger magnifications reveal that the Cu surfaces of 50Cu and 90Cu are a little smoother than those of the other two. Quantitative information regarding the smoothness of the

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deposited Nb surfaces is obtained through 3-D scans on the surfaces using the P-15 profilometer. Surface roughness is found to vary dramatically from one area to the other. Scans over areas smaller than 4000X4000 μm^2 show a standard deviation larger than 10%. Fig.2 summarizes the values of average root mean square (RMS) extracted from profilometer measurements. Each RMS value shown in Fig.2 is an average of five scans over areas of 4000X4000 μm^2 randomly selected on the surface of a sample. Typical profilometer images for 70Cu and 90Cu are shown in Fig.3

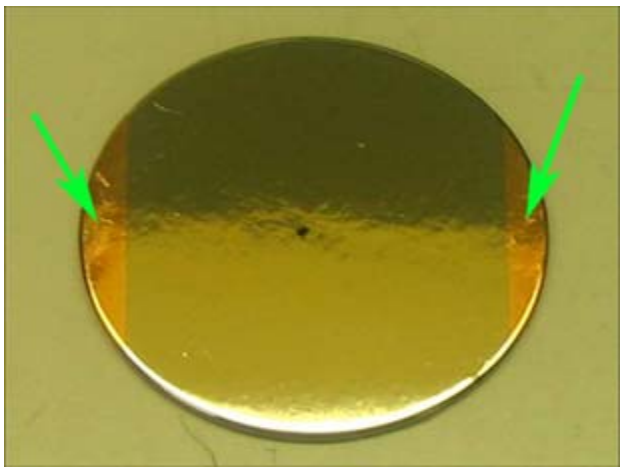

Figure 1: A typical sample.

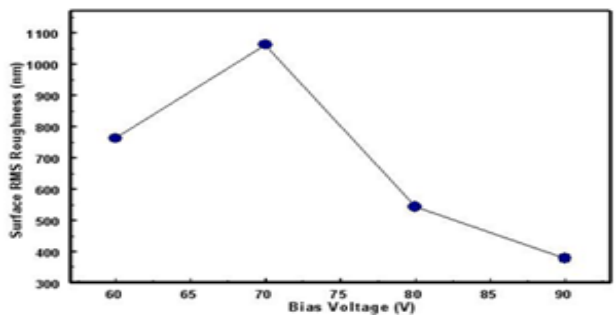

Figure 2: Average surface RMS roughness Vs the bias voltage extracted from 3-D profilometer scans over areas of 4000X4000 μm^2 on Nb/Cu samples (see the text for details).

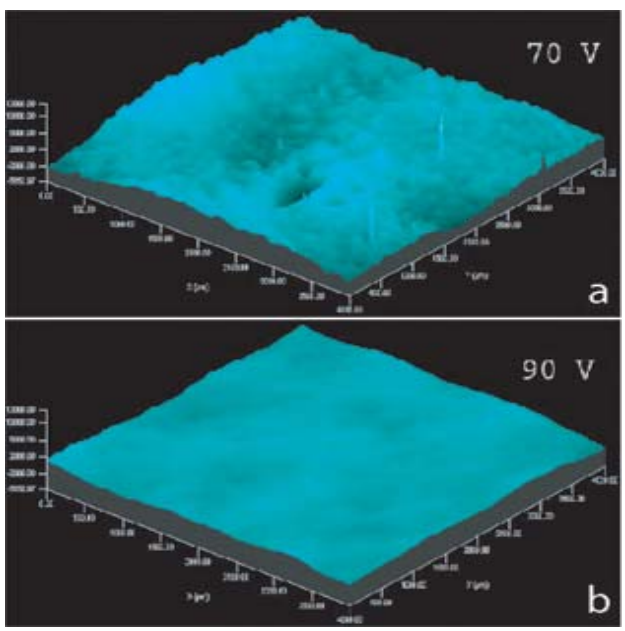

Figure 3: Typical profilometer images of Nb films deposited at bias voltages of a) 70V and b) 90V.

It appears that the large standard deviation for scans smaller than 4000X4000 μm^2 is mainly a result of a relatively large smoothness variation on the Cu substrates as shown in an extreme case in Fig.4. On the other hand, it is difficult to attribute the variation in Fig.2 to the smoothness variation in substrates alone. For instance, the RMS roughness of 60Cu is almost twice as much as that of 90Cu. But the Cu surface of 60Cu looks almost the same or even a little smoother than that of 90Cu under MOM. Therefore, our results seem to show that a higher deposition energy does tend to result in a smoother Nb surface due to a higher adatom mobility. SEM measurements do not give any additional meaningful information due to its deep depth of field.

A comparison of surface RMS roughness of 90Cu with a typical BCP, EP, BEP, and Nb single crystal samples is given in Table 1.

Table 1: RMS roughness extracted from 3-D profilimeter scans over areas of 200X200 μm^2 for typical 90Cu, Nb single crystal (NbSC), Nb surfaces treated by BCP, EP, and buffered EP (BEP)

Preparation Method	BCP	EP	BEP [10]	NbSC [11]	90Cu
RMS (nm)	1274	251	35	27	94

SIMS measurements are done at a base pressure of $9.5X10^{-9}$ Torr with an Ar primary ion beam at an ion beam energy of 2.7 KeV. A typical SIMS spectrum from 1 to 300 AMU obtained on 90Cu is shown in Fig.5. Fig.6 is a typical SIMS spectrum on a Nb surface treated by the conventional 112 BCP. From the comparison between Figs. 5 and 6, we can see that the cracking pattern of surface oxide layer of the Nb film is different from that of BCP treated Nb surface as judging from the ratio between the intensities of Nb^+, NbO^+, and NbO_2^+ peaks, implying

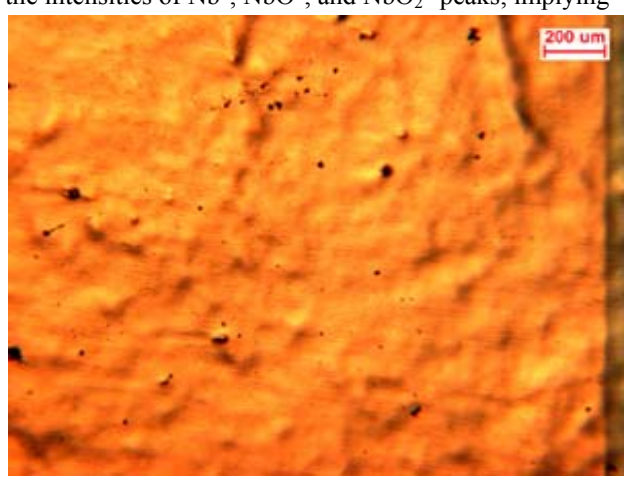

Figure 4: MOM image of the Cu surface of the Nb film deposited at a bias voltage of 70V.

therefore that surface oxide layer structure may not be the same. The surface of Nb film also appears to be much less contaminated. The same observations are found on all other Nb/Cu samples. Hydrogen may not be the major concern on our Nb films. SIMS depth profile measurements reveal that oxygen content is close or more in the interior of the films as compared with those of BCP and EP, except 90Cu (see Fig.7). If oxygen is the main impurity of the Nb films as implied from our SIMS data, this result should indicate that the superconducting transition width should be the narrowest for 90Cu, which is not what observed [6]. Therefore, other small amounts of impurities may play some roles here and have to be measured by SIMS depth profiling too, if microstructures are similar. More SIMS measurements are underway. Detailed SIMS results will be published elsewhere.

SUMMARY

Preliminary results of surface characterization on Nb films deposited on 2.5" electro polished Cu disks under four different deposition energies of 123, 133, 143, and 153 eV are reported. Measurements show that higher deposition energy can lead to a smoother Nb surface in the energy window studied. More Nb films should be made at deposition energy higher than 153 eV to find the optimal energy that may give us the smoothest surface. The Nb film surface deposited at a bias voltage of 90 V is smoother than typical Nb surfaces treated by the conventional BCP and EP techniques. The surfaces of Nb films have much less contaminations compared with those of BCP treated Nb. SIMS measurements also reveal that surface oxide layer structure of the Nb films may not be the same as that of BCP treated Nb bulk samples and oxygen content is higher for some Nb films in the interior of the films. A better base pressure may be necessary for the energetic deposition system in order to reduce oxygen content of the films. RF measurements are needed for comparison with the results from surface characterization.

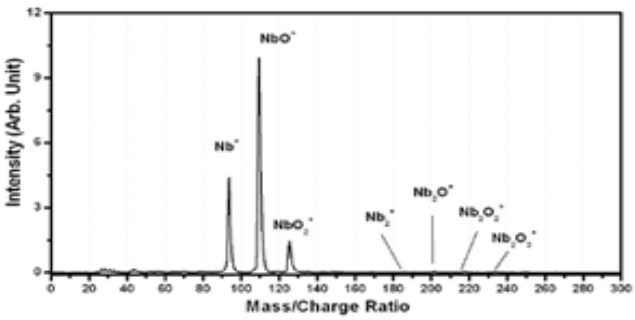

Figure 5: Typical SIMS spectrum on 90Cu surface.

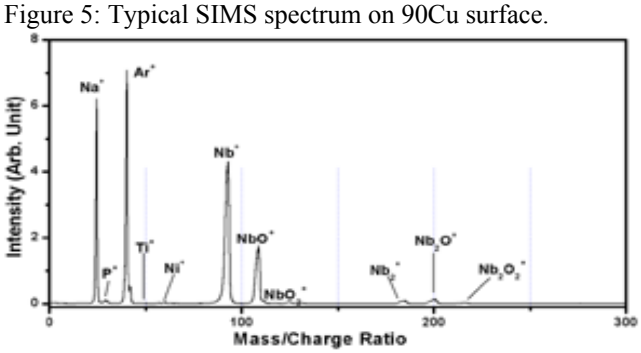

Figure 6: Typical SIMS spectrum on BCP treated Nb surface.

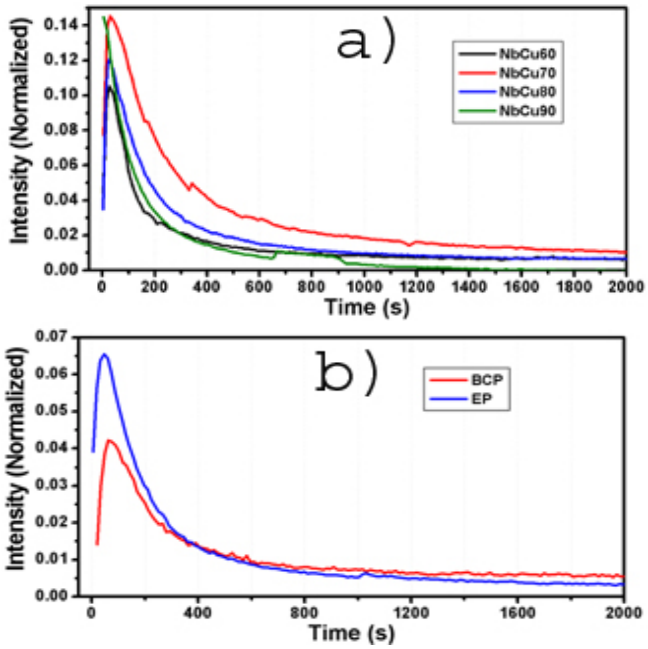

Figure 7: Typical SIMS oxygen depth profile plots for a) 60Cu, 70Cu, 80Cu, and 90Cu, b) BCP and EP.

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