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SURFACE STUDIES OF MATERIALS FOR SUPERCONDUCTING CAVITIES*

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Abstract

A multitechnique system has been constructed to study materials and processes used for producing high Q superconducting cavities, while constantly maintaining UHV environment. Characterization of a small disc of superconducting material, e.g. Nb, is done by a variety of methods, including AES, XPS, EID, ellipsometry, sputter profiling, and secondary electron yield measurements. The samples may be processed in situ by rf and electron bombardment heating, and ion sputtering. Sample temperatures may be held from 77K to 2500K. Coating by sputtering and evaporation, and oxidizing and nitriding are incorporated. Both AES and secondary yield measurements are accomplished using very low current electron beams and counting electronics to minimize the reduction of oxide surfaces by electrons. Computer-controlled ellipsometry allows monitoring of the temporal growth of surface layers during controlled exposure to gases. Extensive measurements have been carried out on Au, C, Pt, Nb and its oxides, nitride, and carbide. We present AES, secondary yield, and other measurements, and indicate trends which may enable the production of stable cavity surfaces and their simple, effective in situ regeneration while installed as accelerating cavity surfaces.

1. Introduction

The field of rf superconductivity has, for the last decade, been bedeviled by individual tantalizing results which have failed to develop into a mature technology. For each failure some very reasonable explanation (dust, oxide layer charging, single surface multipactor, etc.) is advanced, all too often not to be definitively investigated. It is now generally recognized that surface effects are a major barrier to the maximum electric field gradient attainable in multicell structures, and that performance generally degrades upon atmospheric exposure. At SLAC we have initiated a detailed application of sophisticated surface physics techniques to the study of niobium and its possible surface coatings. The objective is to produce a surface which is stable upon atmospheric exposure and has a low enough secondary emission coefficient to preclude multipactor.

2. Experimental System

The surface studies program is being carried out in a multisample ultrahigh vacuum system (Fig. 1) incorporating an Auger electron spectrometer (AES), an x-ray photoelectron spectrometer (XPS), an ellipsometer, a quadrupole mass spectrometer (RGA) with plans to add secondary ion mass spectrometry (SIMS), and electron impact desorption (EID). Samples can be transported to all positions of the apparatus while maintaining ultrahigh vacuum; for example, from the analytical apparatus to a process chamber, permitting ion etching, rf heating, resistive and electron beam evaporation, sputter deposition, and reactive sublimation coating. The samples can also be moved into the computer-controlled ellipsometer apparatus for in-situ film growth studies with reactive gases, or into the rf furnace chamber for high temperature outgassing with concurrent electron bombardment.

The computer-controlled ellipsometer allows real-time monitoring of surface adsorption and desorption using sampling intervals of 10 msec, and can be used at pressures up to atmospheric. In addition, the ellipsometer can scan in wavelength under

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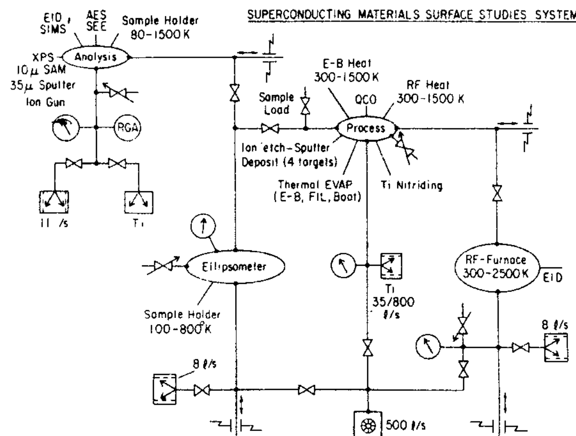

Fig. 1

computer control, providing measurements of the complex dielectric constant, thickness, and reflectivity of the surface under study.

3. Measurements

Calibration of sputter etch rates was accomplished using anodically prepared Nb_2O_5 coatings of known thickness over the range 100Å to 4000Å. The coatings were prepared in a 3% NH_4OH solution using a niobium cathode. X-ray diffraction measurements confirmed the thickest film to be Nb_2O_5. A typical sputter etch profile is shown in Fig. 2, and the sputter etch calibration curve in Fig. 3.

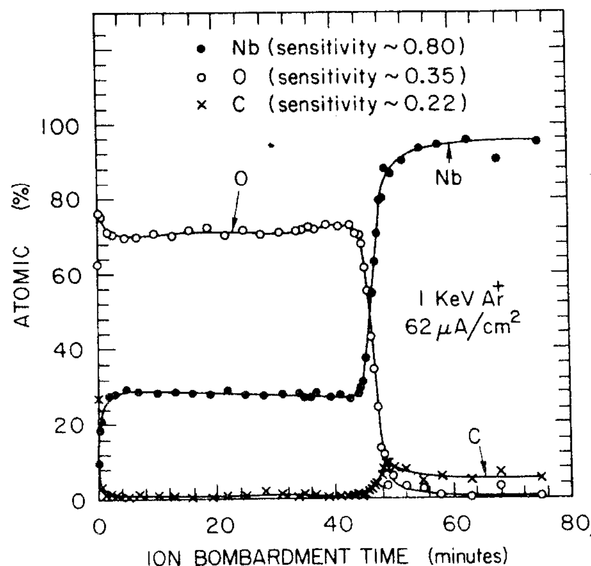

Fig. 2. Sputter profile - 1000Å anodic Nb_2O_5.

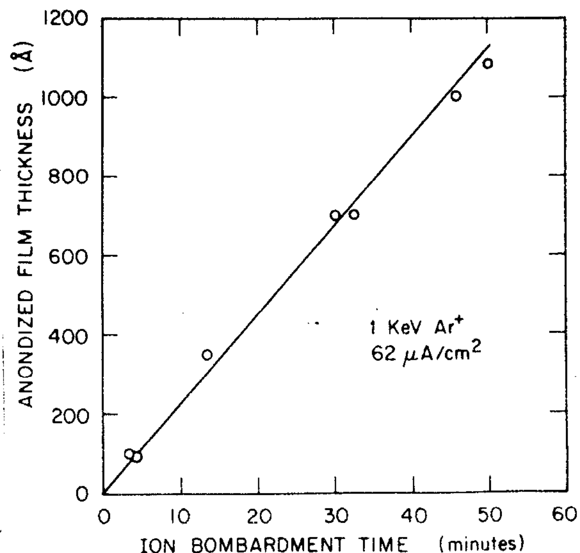

Fig. 3. Sputter etch calibration.

Figure 4 shows typical Auger spectra obtained for niobium surfaces before and after removing an anodic (Nb_2O_5) layer. Lin and Lichtman {1} have identified certain Nb peaks as being metal-like or oxide-like. We have extended their method to obtain quantitative information on oxide stoichiometry by normalizing these peaks to the $M_{4,5}N_1N_{2,3}$ 110 eV peak and plotting the resulting relative peak heights against the O/Nb ratio. These results are shown in Fig. 5, which includes with our powder data both anodized Nb_2O_5 and bulk Nb metal.

Secondary electron emission (SEE) coefficients were measured using the retarding potential method {2, 3} at nanoampere incident currents. The results for a typical anodized niobium sample are displayed in Fig. 6, which shows the changes in σ as the oxide is first cleaned and then progressively removed by sputter etching. Secondary electron yield versus primary energy for eight different sputter-cleaned surfaces is shown in Fig. 7. Two separate sets of samples were run and the reproducibility was excellent. Notice that the yields are much lower for the powder samples. We attribute this to trapping of the secondaries in the rough powder matrix. There were no measurable differences in yield for Nb samples with particle sizes > 47 and < 200 mesh. The values ($\sigma_{max} \sim 0.8$) obtained for NbC and NbN are significantly lower

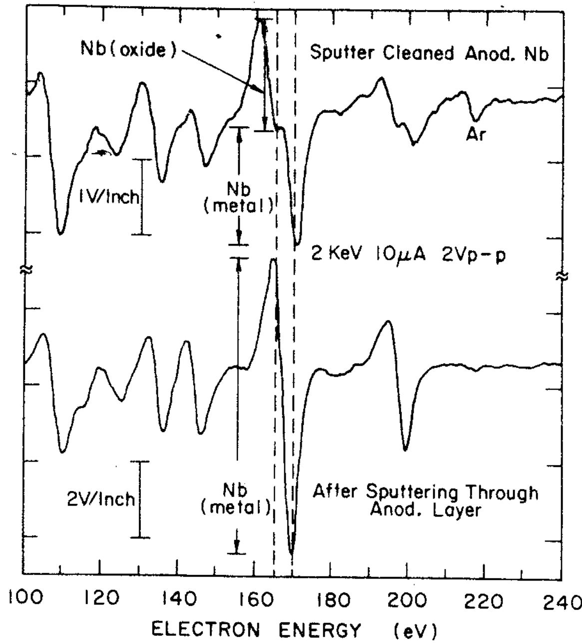

Fig. 4. Nb Auger peaks for Nb_2O_5 and Nb.

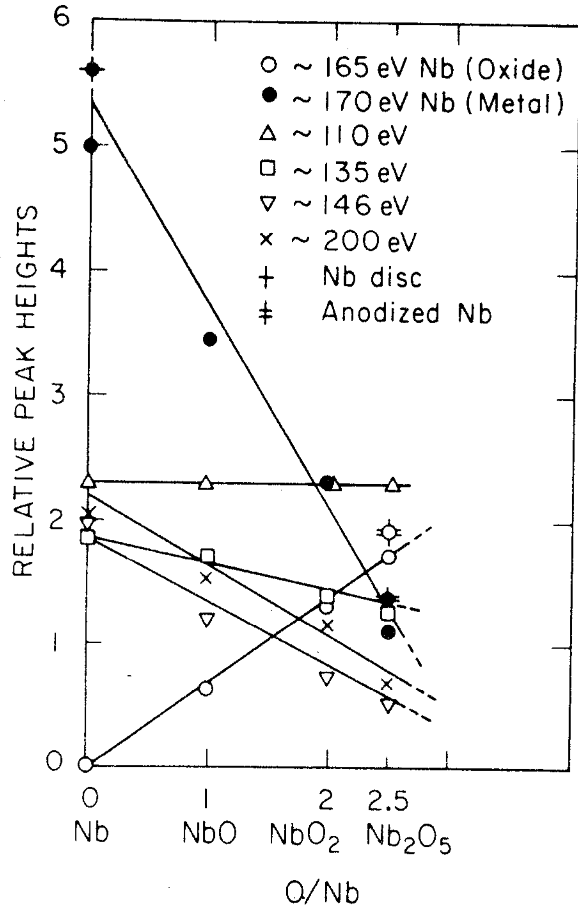

Fig. 5. Nb peaks normalized to 110 eV transition vs oxide composition.

than for the other materials, thus these two materials are likely candidates for cavity coatings in the near future.

Previous studies of electron beam effects on surface layers by Benninghoven et al. {4} and Lin and Lichtman {1} indicate that low beam current exposures should be used during AES and SEE to prevent damage, particularly on oxides. In Fig. 8 we present results for EID modification to an anodized Nb_2O_5 surface using a typical AES beam of 10 μA current. Clear modification to the surface layer has occurred within one minute exposure (exposure of 2 C/cm^2). SEE measurements presented in this paper used an electron beam having a current density a thousand times smaller than that of Fig. 8, with AES and SEE analysis being done on adjacent areas of the samples to prevent influence by the AES beam on SEE analysis. We have now converted AES to low current analysis by incorporating pulse counting for signal processing. The N(E) spectrum so produced is stored in a dedicated minicomputer, where it may be numerically differentiated for the $\frac{dN(E)}{dE}$ spectrum without information loss and without introducing phase distortion effects {5} common to CMA's run in the electronically differentiated mode.

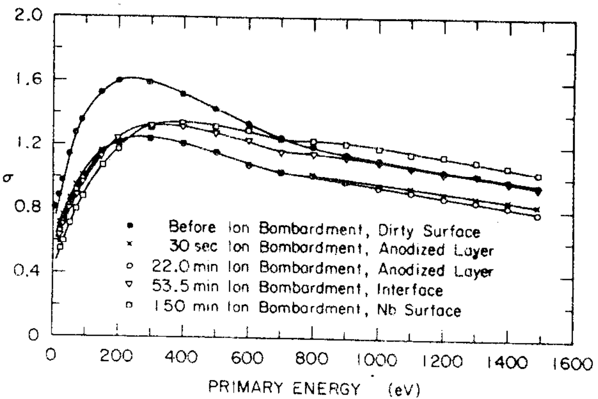

Fig. 6. Total secondary emission coeff. for various surface conditions.

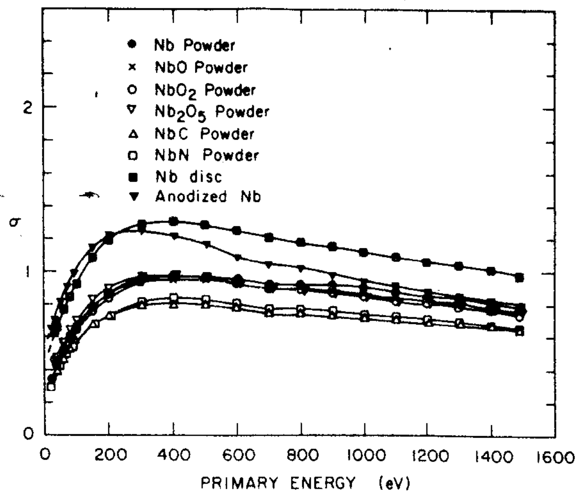

Fig. 7. Total secondary emission coefficients for sputter-cleaned Nb and various compounds.

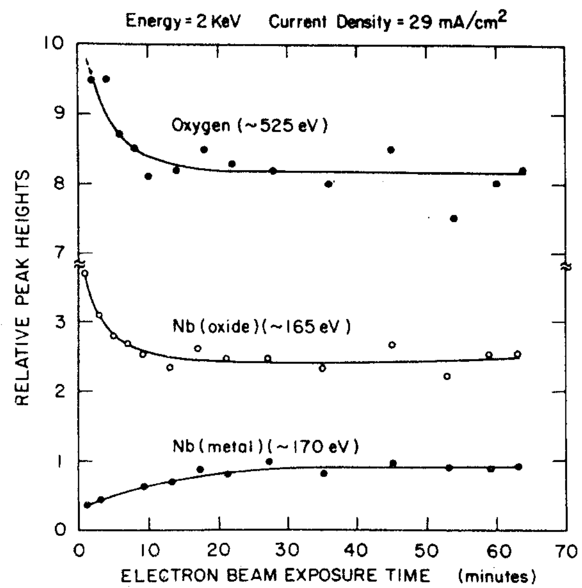

Fig. 8. Electron beam exposure modification of an anodized Nb surface.

4. Conclusion

A sophisticated surface analysis and treatment system has been described which is capable of surface cleaning and modification by a variety of methods and which, by dedicated computer, makes possible rapid ellipsometric measurements of surface adsorption-desorption in real time. In addition, meaningful static AES and SEE can be conducted at low enough primary currents to preclude dielectric surface damage.

References

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