RF AND STRUCTURAL CHARACTERIZATION OF SRF THIN FILMS *

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Abstract

In the past years, energetic vacuum deposition methods have been developed in different laboratories to improve Nb/Cu technology for superconducting cavities. JLab is pursuing energetic condensation deposition via Electron Cyclotron Resonance. As part of this study, the influence of the deposition energy on the material and RF properties of the Nb thin film is investigated. The film surface and structure analyses are conducted with various techniques like X-ray diffraction, Transmission Electron Microscopy, Auger Electron Spectroscopy and RHEED. The microwave properties of the films are characterized on 50 mm disk samples with a 7.5 GHz surface impedance characterization system. This paper presents early results on surface impedance measurements in correlation with surface and material characterization for Nb films produced on sapphire and copper substrates.

INTRODUCTION

Due to the very shallow penetration depth of RF fields (only ~40nm for Nb), SRF properties are inherently a surface phenomenon, involving a material thickness of less than 1 micron. One can then foresee the merits of depositing a Nb film on the inner surface of a castable cavity structure made of copper (Cu) or aluminium (Al). At the system design level, this would exploit the freedom to decouple the active SRF surface from the accelerating structure definition and its cooling, opening the possibility to dramatically change the cost framework of SRF accelerators.

CERN has conducted pioneered studies [1-3] in the field of SRF Nb films on Cu (Nb/Cu) applied to cavities and successfully implemented this technology in LEP-2. Although 1.5 GHz cavities achieving gradients up around 25MV/m [4] were produced, these cavities suffered from significant losses resulting in the significant reduction of Q at accelerating gradients above 15MV/m. Some of the defects were inherent to the magnetron sputtering technique used to produce these cavities.

While tight correlation with the characterization of real materials has yet to be described, there exists a theoretical framework describing the relevant material parameters of surfaces as they influence SRF properties. Several material factors, highly dependent upon the surface creation conditions, contribute to degraded SRF performance with respect to ideal surfaces. These limiting factors such as intra-granular impurities and lattice defect density, inter-granular impurities and oxidation, surface topography and chemistry, may lead to the reduction of the electron mean free path, thus the reduction of the lower critical field H_{c1}. The relative contribution of each of these factors needs to be examined for any method used to produce SRF thin films. Fundamental work is needed to establish the correlation of detailed material characteristics with the consequent SRF performance.

Understanding the characteristics of the films produced, the nucleation and influence of the diverse deposition parameters, substrate nature, temperature and morphology on the final RF surface for Nb films is the focus of an on-going study at JLab and neighbouring partners.

TOWARDS BULK-LIKE NIOBIUM FILMS

The challenge is to develop an understanding of the film growth dynamics from nucleation to final exposed surface. What matters most is the defect density (which determines the electron mean free path) within the rf penetration depth. This defect density is certainly affected by intragrain contaminants incorporated during the final stage film growth, but it is also strongly affected by the underlying crystal texture, which is in turn developed from the initial film nucleation process, which necessarily is strongly influenced by the substrate. The development of every stage can be expected to depend strongly on the kinetic energy distribution of the arriving Nb ions.

With the objective of growing and characterizing Nb films with controlled deposition energy and substrate temperature to minimize the defect density and achieve bulk-like performance, studies including systematic assessment of the RF surface impedance and other parameters like the London penetration depth λ are conducted on two fronts: film growth via energetic condensation and nucleation studies.

Energetic Condensation via ECR

With the availability of techniques for energetic condensation in vacuum, Nb films with a wide range of microstructural properties and features believed to be relevant to RF performance can be produced, characterized and RF tested. Properties such as film purity, stress, texture, and grain size can be measured over a wide range of values not accessible using conventional sputtering techniques. One of the energetic condensation techniques JLab is pursuing is the use of an Electron Cyclotron Resonance (ECR) Nb ion source in ultra high

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vacuum (UHV). The process principle and the coating system are described in detail elsewhere [6]. The main advantages of this technique are the production of a higher flux of singly charged ions with controllable incident angle and kinetic energy and the absence of macroparticle production.

Nucleation Studies

In parallel, nucleation studies are underway within the frame work of a collaboration with the College William & Mary using a UHV sputtering system equipped with in-situ reflection high-energy electron diffraction (RHEED) and scanning tunnelling microscope (STM) to monitor in-situ the crystal character dependence on substrate properties and deposition parameters (temperature, working gas, intermediate annealing , etc.).

While studying film growth in homo-epitaxy and hetero-epitaxy on both single crystal and polycrystalline substrates, particular attention is given to three sequential phases: (1) film nucleation on the substrate, (2) growth of an appropriate template for subsequent deposition of the final rf surface, and (3) deposition of the final surface optimized for minimum defect density.

Material and RF Characterization

To inform the interpretation of the integrated film growth dynamics and influence of various deposition parameters, the standard surface and structural characterization includes X-ray diffraction (XRD), Electron Backscatter Diffraction (EBSD), Scanning Auger Electron Microscopy (SAM), high resolution Secondary Electron Microscopy (HR-SEM), High Resolution Transmission Electron Microscopy (HR-TEM), and Secondary Ion Mass Spectroscopy (SIMS).

The superconducting surface impedance, the penetration depth (λ) and the temperature dependence of H_{c1} is investigated on 50 mm disc samples with the use of the Surface Impedance Cavity (SIC), a 7.5 GHz TE_{011} sapphire-loaded Nb cavity [7].

The cryogenic performance is investigated with the 4-point-probe method to measure Tc and RRR.

HETERO-EPITAXIAL NIOBIUM FILMS

This section presents early results in the study of the correlation of the surface resistance of Nb/Cu and Nb on sapphire (Nb/Al_2O_3) films coated in both deposition systems with surface and material properties (structure, morphology, impurity content…) of the film as a function of deposition energy and substrate temperature.

Nb Hetero-epitaxy on Sapphire

Although sapphire is not a practical substrate for SRF cavities, it constitutes a good ground to study Nb nucleation due to its relatively small lattice mismatch with Nb (1.9% for (11-20) Al_2O_3). All Nb films produced on (11-20) sapphire in various conditions by ECR and magnetron sputtering are single crystals with the orientation (110) Nb//(11-20) Al_2O_3 as revealed with EBSD and XRD measurements. However, XRD pole figures along the (110) Nb plane (Fig. 1), show a texture for 200nm films which is strongly attenuated for 2µm thick films deposited in the same conditions.

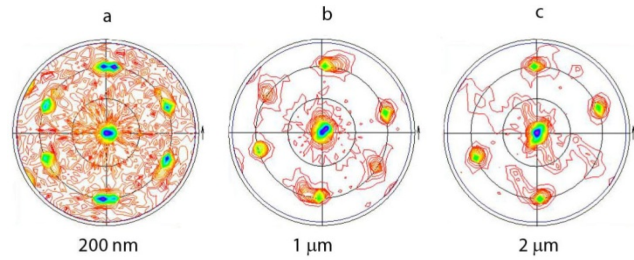

Figure 1: (110) pole figures for ECR Nb films coated with -120V bias voltage and different thickness: (a) 200nm, (b) 1 µm and (c) 2 µm.

AFM scans (Fig. 2) reveal some anisotropy in the morphology of the films with the presence of 2 growth domains for the thinnest Nb film (50nm). The anisotropy is accentuated with the prevalence of one growth domain for the thicker film (600nm). This film has a RRR value of 97 and T_c of 9.29K.

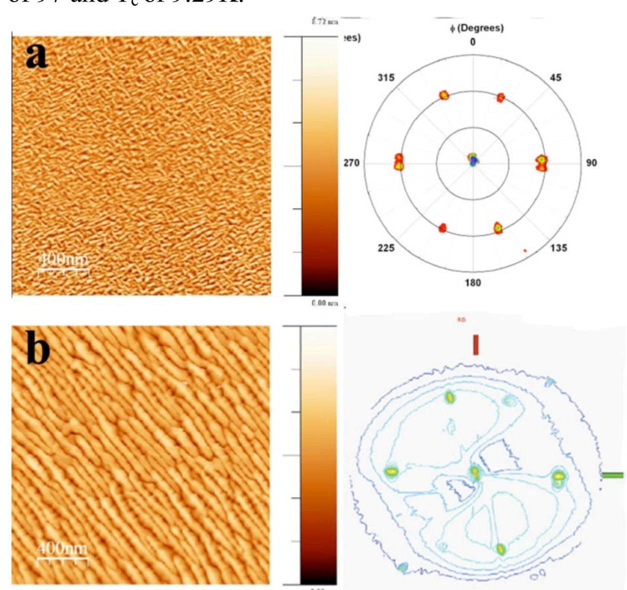

Figure 2: AFM scans (50µm x50 µm)and (110) pole figures for magnetron sputtered films deposited at 600ºC with a thickness of (a) 50nm and (b) 600nm.

Influence of Bias Voltage

In hetero-epitaxy of Nb on Cu, the bias voltage seems to have a drastic influence on grain growth. Figure 3 represents EBSD maps for Nb/Cu films deposited at -120V and 0V bias respectively. Both Cu substrates were heat treated to 450ºC for 18 hours to completely reduce the native Cu oxide and allow hetero-epitaxy of Nb on Cu.

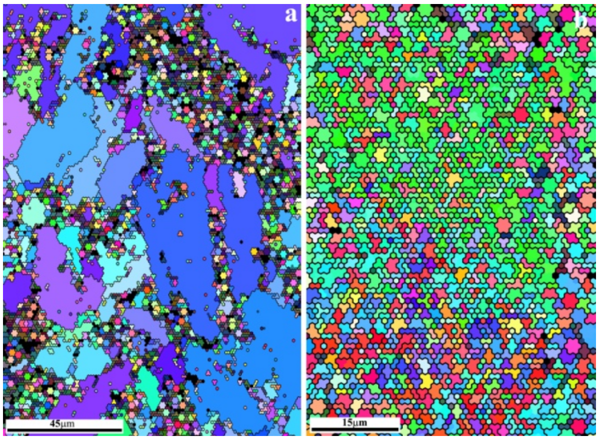

Figure 3: EBSD maps of ECR Nb/Cu films coated at 450ºC with -120V (a) and 0V (b) bias voltage.

Influence of Substrate Treatment

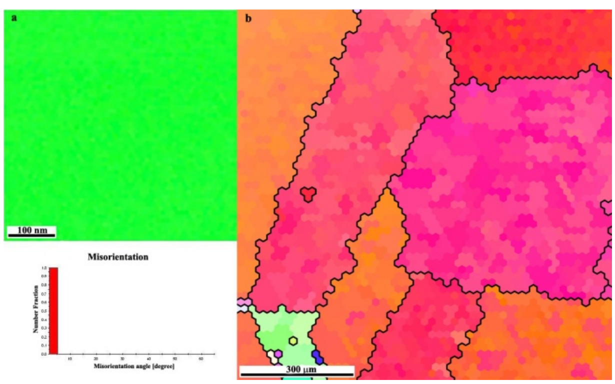

Figure 4: EBSD maps of ECR Nb films on Al_2O_3 (a) and Cu (b) coated with -120V bias and 450°C after heat treatment of the substrates at more than 900°C.

The quality of the substrates has also a great influence on the quality of the film. High quality Nb films were produced on substrates heat treated in-situ to a temperature higher than 900°C prior to coating which fully re-crystallized the Cu substrate and annealed the sapphire surface. Figure 4 shows an EBSD maps for both Nb/Cu and Nb/(11-20) Al_2O_3 films. The Nb/(11-20) Al_2O_3 film, only 232 nm thick, exhibits a very high level of crystallinity and has a RRR of 71.5 and T_c of 9.25K. One needs to keep in mind that, because of the small thickness of the film, boundary scattering is severely limiting the RRR value.

RF Characterization of ECR Samples

Several ECR films have been measured with the SIC cavity. Figure 5 compares the surface resistance as a function of temperature for ECR films coated at 0V, -90V and -120V bias voltages with a fine grain bulk Nb sample.

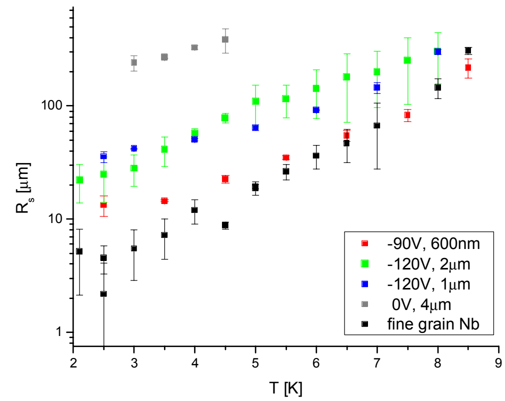

Figure 5: Surface Impedance R_s with SIC measurements for different ECR films as a function of temperature T.

CONCLUDING REMARKS

JLab in collaboration with neighbouring partners is pursuing the opportunity to understand and develop niobium films with bulk-like performance by elucidating the functional dependence of film-grown niobium crystal texture, intra-grain defect density, and grain boundary impurities on SRF performance.

Early results on the influence of the bias voltage, substrate interface and RF characterization have been obtained. Films with good RRR values have been produced by both deposition techniques employed. More detailed and extensive studies are underway.

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