

Reclamation of niobium compounds from ionic liquid electrochemical polishing of superconducting radio frequency cavities

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ABSTRACT

Recent research has shown that choline chloride (vitamin B₄)-based solutions can be used as a greener alternative to acid-based electrochemical polishing solutions. This study demonstrated a successful method for electrochemical deposition of niobium compounds onto the surface of copper substrates using a novel choline chloride-based ionic liquid. Niobium ions present in the ionic liquid solution were dissolved into the solution prior to deposition via electrochemical polishing of solid niobium. A black coating was clearly visible on the surface of the Cu following deposition. This coating was analyzed using scanning electron microscopy (SEM), electron dispersive X-ray spectroscopy (EDX), atomic force microscopy (AFM), and X-ray fluorescence spectroscopy (XRF). This ionic liquid-based electrochemical deposition method effectively recycles previously dissolved niobium from electrochemical polishing of superconducting radio frequency (SRF) cavities.

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Introduction

Modern particle accelerators require many large superconducting radio frequency (SRF) cavities, typically fabricated from solid niobium metal. Although niobium is an ideal material for use in SRF cavities, high-purity metal Nb is very expensive, making large-scale SRF cavity fabrication a sizable financial burden. Current methods for polishing Nb cavities bound for SRF accelerators use mixtures of concentrated acids including sulfuric (H₂SO₄) and hydrofluoric (HF) acid [1–3]. An alternative to these methods, described by Wixtrom et al. [4], instead utilizes a choline chloride-based ionic liquid. This “green” polishing method is more environmentally friendly and cost-effective due to the lack of acid-related hazards and waste disposal [5–8]. A further key feature of this alternative polishing method is that the Nb ions electrochemically dissolved from the solid Nb being polished remain in the ionic solution. This Nb in solution is able to be recycled, using electrochemical deposition to coat a solid Cu substrate. This deposition method, when coupled with ionic liquid-based polishing of Nb, is very economical, with successful deposition achieved with little-to-no additional Nb dissolved in solution.

Previous methods for depositing Nb onto various substrates have included atomic layer deposition of Nb₂O₅ thin films [9], high vacuum chemical vapor deposition of Nb oxide [10], and high power impulse magnetron sputtering (HiPIMS) of Nb₂O₅ [11]. These methods have had various levels of success in achieving successful deposition of Nb, though each has disadvantages, foremost among these being the expensive instrumentation required for deposition. One study specifically focused on preparing solid Cu SRF cavities coated with Nb, using yet another method for deposition, based on a cathodic arc system [12]. This study demonstrated the ability of Nb-coated solid Cu SRF cavities to perform similarly to solid Nb cavities, provided the quality of the coating is sufficient [12]. Materials and preparation costs for an Nb-coated solid Cu SRF cavity would be significantly lower than the cost of a similarly sized solid Nb cavity.

The primary goal of this study was recovery of electrochemically dissolved Nb ions from solution via deposition onto the surface of Cu substrates using a VB₄-based ionic liquid and a three-electrode system. Other research has explored the use of choline chloride/ethylene glycol-based and other similar solutions accepted as ionic liquids for electrochemical deposition of metal ions, including much of A.P. Abbott's research [13–17]. These studies focused on metals other than Nb (Ni, Mg, Se and others), but used ionic liquid solutions to perform deposition, demonstrating the viability for metal ion recovery from ionic liquid solutions. The purpose of this research was to combine the benefits of ionic liquid-based deposition methods, including lower costs associated

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with the technique and the ability to recycle Nb from used EP solutions [4], with the potential for producing viable Nb-coated solid Cu SRF cavities.

Experimental

The VB_4 ionic liquid was prepared using the same procedures detailed in Wixtrom et al. [4], by mixing ammonium fluoride (AF, 98+% – Acros Organics), choline chloride (VB_4 , MP Biomedicals), and ethylene glycol (EG, 99+% – Acros Organics) in a 1:1:2 molar ratio. The mixture was heated to $\sim 70^\circ\text{C}$ with stirring until the solution became homogenous. The total volume of the solution used for deposition onto a 2.9-cm^2 Cu surface area was ~ 80 ml. Niobium was dissolved into the deposition solution according to the polishing methods detailed in Wixtrom et al. in addition to using higher voltages for accelerated dissolution of Nb into solution when required [4].

A Gamry PCI-4G750 potentiostat was used to perform electrochemical deposition of Nb compounds onto the surface of Cu substrates. Prior to deposition, each Cu piece was sanded, dipped for 10–20 s in a minimal volume of a 1:3 mixture of nitric and hydrochloric acid, cleaned using soap and deionized water, dried, and finally rinsed with acetone before weighing. A standard three-electrode system was used, with initial trials utilizing an Nb counter electrode and a Cu substrate working electrode, with a glassy carbon reference electrode, and all subsequent trials using a carbon counter electrode. Glassy carbon counter and reference electrodes were chosen to recover and deposit dissolved Nb ions directly from solution without contaminating the composition of the solution by introducing additional metal ions [18,19]. All trials were performed at $\sim 70^\circ\text{C}$ with a typical separation of ~ 1.5 cm between the anode and cathode. The deposition was performed by running chronoamperometry and fixing the voltage at various negative voltages from -0.8 V to -7.0 V, with deposition duration ranging from 0.5 to 8 h. Coated Cu substrates were weighed after drying and stored in a vacuum box for further analysis. Cyclic voltammetry was also performed using the same 3-electrode system (Cu working, glassy carbon counter and reference), with a potential sweep rate of 50 mV/s.

Analysis of the coated Cu pieces was performed using Scanning Electron Microscopy (SEM) coupled with Electron Dispersive X-ray Spectroscopy (EDX) on a Hitachi S-4700 SEM with an AMETEK EDAX attachment. SEM/EDX was used to view the interface of the coated and uncoated surfaces of the Cu substrate, to identify the presence of Nb in the coating, and to determine if any contaminants were present (or excess oxidation had occurred), which would render the coated Cu unsuitable for use in some applications. Atomic force microscopy (AFM) analysis was used to examine surface topography, specifically to determine the root mean squared roughness (R_q) of the coated surface of the Cu samples. AFM scans were taken using a DimensionTM 3100 atomic force microscope in tapping mode with a 300 kHz resonant frequency silicon tip. X-ray fluorescence (XRF) spectroscopy was performed on coated samples using a Bruker S4 Pioneer to determine the thickness and elemental composition of the coating, and to further confirm the presence of Nb on the coated Cu surface.

Results and discussion

Linear sweep voltammetry was performed to determine the ideal voltage for deposition. Due to the lack of a clearly defined peak, a range of voltages were used. Cyclic voltammetry indicated a reduction peak at -0.78 V in solution containing dissolved Nb ions (Fig. 1), and at -0.74 V in solution containing no dissolved Nb ions (Fig. 2). -0.8 to -1.0 V were found to be effective voltages for achieving successful deposition. A range of voltages and

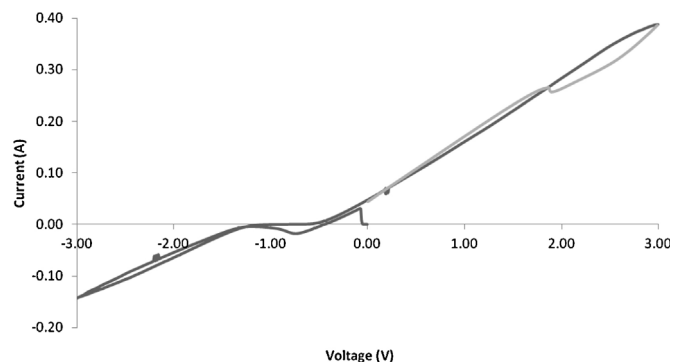


Fig. 1. Cyclic voltammogram of Cu in an ionic liquid solution (2EG:1AF:1 VB_4) containing dissolved Nb. Potential sweep range from -3 V to $+3$ V, starting in the negative direction with an initial voltage of 0.0 V.

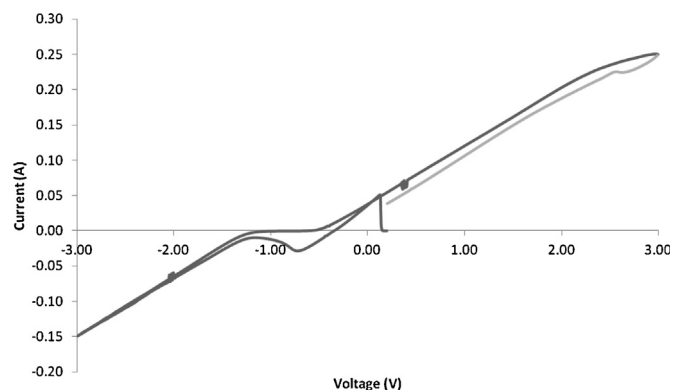


Fig. 2. Cyclic voltammogram of Cu in an ionic liquid solution (2EG:1AF:1 VB_4) without dissolved Nb. Potential sweep range from -3 V to $+3$ V, starting in the negative direction with an initial voltage of 0.2 V.

conditioning times for chronoamperometry were used to deposit Nb onto Cu substrates with both Nb and glassy carbon counter electrodes. Initially, deposition was performed with an Nb counter electrode in order to confirm that the system could successfully deposit Nb onto the Cu substrate with constant introduction of Nb into solution. After this was determined to be successful, all future trials utilized a glassy carbon counter electrode with Nb electrochemically dissolved into the solution prior to deposition, in order to determine if recycling of dissolved Nb from used electrochemical polishing solution could be achieved. Anodic dissolution of Nb into solution was performed using the optimal polishing parameters as detailed in Wixtrom et al. [4], or alternatively, using higher voltages due to the faster rate of Nb dissolution. Deposition using a glassy carbon counter electrode opposite the Cu substrate working electrode was successful in reclaiming the previously dissolved Nb in solution via deposition

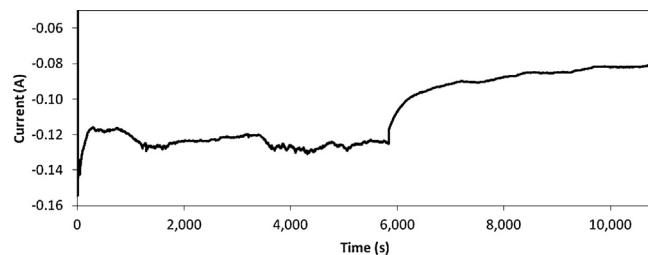


Fig. 3. Chronoamperometry scan of Nb deposition for 3 h at -1.5 V. The Cu substrate was the working electrode, with glassy carbon counter and reference electrodes.

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