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Cu/Nb QPR SURFACE PREPARATION PROTOCOL IN THE FRAMEWORK OF ARIES PROJECT*

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Abstract

The Quadrupole Resonator is a powerful tool for SRF R&D on thin films. It allows to perform Q vs Eacc measurements on flat samples rather than a curved surface of a cavity. For the investigation of super conductive (SC) coatings on copper substrates, e-beam welded Cu/Nb samples have been prepared for the QPR. However, the presence of two metals, in particular at the interface, makes proper polishing of both surfaces challenging, due the different chemical behaviour of both components. In this work we present the protocol developed for the surface preparation of the coexisting Cu and Nb phases and the results obtained for 5 different samples. The work was performed in the framework of the ARIES project.

INTRODUCTION

ARIES is an international collaboration between research groups from CEA (France), CERN (Switzerland), INFN/LNL (Italy), HZB and USI (Germany), IEE (Slovakia), RTU (Latvia) and STFC/DL (UK) that are working on the improvement of the superconductive thin films for SRF cavities [1]. The work package 15, and in particular task 15.2, is focused on the substrate surface preparation.

After a study of electropolishing, chemical polishing (SUBU5), tumbling and its combination on the planar samples [2, 3], the protocol had to be adjusted for the QPR samples preparation. Subsequently, five Cu/Nb QPR samples were treated to study then SC thin films, described in detail in [1, 4-9]. In this work, the optimized protocol of the QPR samples is shown, and a brief description of all treatments and some variable parameters are given.

SURFACE PREPARATION PROTOCOL

A protocol consists of a series of steps, described below in the Table 1. Initially, the protocol included either electropolishing or chemical polishing (SUBU5) treatments as a main polishing step. In the last version, it was decided to do only electropolishing, as it is a more stable and potent technique. For the new QPR sample, the protocol starts from the 1a step and do not include step 3*- Indium removal process. Instead, for the previously sputtered QPR samples, no machining is done, but the stripping process that removes Nb thin film from the Cu disk part of the QPR sample. Some of the samples after measuring process had residuals of Indium. To remove it, relatively fast treatment was done, that chemically pills the Indium material from the flange after 5-10 minutes, and then can be easily removed with a plastic object.

Table 1: Complete Protocol of the Cu/Nb QPR Samples Treatment

#	Step	Solution	Parameters	Time
1a	Lathe machining	-	270 RPM, 40 µm a time	
1b	Stripping	100 g/l powder in HF:HFB_4	Applied only locally on the SC film	<30 min
2	Ultrasound cleaning	GP-1740 10g/l soap solution	40°C	1 h
3*	Indium removal	20% HCl	Until In detach	5-30 min
4	Etching	20 g/l $(NH_4)_2S_2O_8$		15 min
5	EP	3:2 v.r. H_3PO_4:n-Butanol	2-3 V, 40°C	15-30 min
6	Rinsing, Ultrasound	Distilled water		30 min
7	Passivation	10 g/l Sulfamic acid		1-3 min
8	High Pressure rinsing	Distilled water	150 atm	1 min
9	Rinsing, drying	Distilled water, ethanol, Nitrogen		2 min
10	Vacuum chamber transfer	-	Vacuuming, Ar fluxing up to the 1,1 atm	

Lathe Machining

The initial roughness of the commercially produced samples after milling was too high (Ra ~1.6 µm, Rz ~ 12 µm) to apply polishing recipes. That is why it was decided to do a uniform polishing by lathe machine at LNL mechanical workshop using a diamond tool that should not contaminate the surface. The first machining processes were optimized later, as initially processing caused troubles on the surface due to the low removing thickness (1 µm) (e.g. Fig. 1). Only an average removal of 40 µm has led to defect-free surface.

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Figure 1: The QPR sample surface. 1A – after non-optimised lathe; 1B – same QPR polished with pitting close to the border; 1C – optimised lathe polishing.

Stripping

As described earlier, to fulfil the workgroup sample exchange routine, some of the QPR workpieces were meant to be retreated, and thus the removal of the deposited SC film was needed. Most of the films had a relatively low thickness of 4 μm. An industrial stripping solution can selectively remove niobium-based films, without etching the Cu. In fact, a low time of stripping in some cases leads to the still reflective Cu surface. However, initially stripping was applied to all QPR samples and in some cases, the treatment was not able to efficiently remove the deposit, instead the Nb was visibly etched (e.g. Fig. 2). Moreover, sometimes, few small spots of coating after stripping, remain and are not visible, appearing only after EP. To deal with that issue, it was proposed to do a light etching in ammonium persulfate solution (see next sub-section).

The resulting time of stripping in the protocol was decreased down to 30 minutes with a partial processing of a Cu disk part, excluding the bulk Nb workpiece.

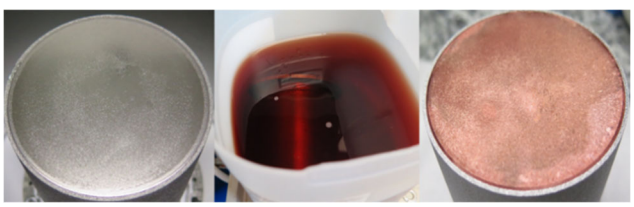

Figure 2: Stripping of the QPR B4 sample.

Etching

An intermediate step before the polishing – ammonium persulfate solution etching - was used. The purpose of this decision is related to the fact that reflective copper surface cannot be a reliable tool to ensure complete Nb stripping. As a result, during electropolishing black points might appear, and the only way to deal with that is to repeat the protocol from step 1b.

A simple treatment is highlighting copper grains, and additionally can be useful to understand if the grains are the same size across the surface. In B4 sample (e.g. Fig 3.), it was noticed that polishing and resulting sputtering leads to the odd effect of the surface. In the next processing of the workpiece, during persulfate etching (in the same position of the defect) higher grain size, which potentially was formed due to the local overheating, was noticed.

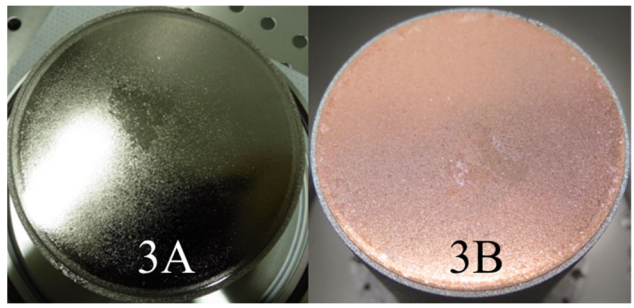

Figure 3: The B4 QPR sample surface look. 3A – After sputtering; 3B – next round after stripping and etching.

Electropolishing

The electropolishing remains the best known way to prepare the surface removing contaminations and smoothing microroughness of the surface. As a solution, a standard recipe of the LNL Cu 6 GHz cavities treatment was used protocol; it included 3:2 volume ratio of Phosphoric acid and n-Butanol. This solution can polish the surface up to the mirror-like grade. The controlling of the power supply was done through the custom written software. As a working voltage load, initially it was chosen a point in which the conductivity of the solution is minimal, based on the curve dI/dV vs V. However, this standard approach was changed to the manual voltage selection in the lower values of voltage at 2-3 V. The reason that motivated it was the pitting, that damages the surface with the processing time. Additionally, higher working voltages may oxidize the Nb forming a thin layer of Niobium oxide. The last is easy to remove with diluted hydrofluoric acid but may consequently decrease the quality of copper in case of more than 10 m of treatment.

Three main setups for the EP (e.g. Fig. 4) were tested and all of them had their drawbacks. Finally, only 4B was applied onto workpieces. The 4A was discarded, due to the non-uniform polishing across the disk, caused by the non-uniformity of the viscous layer and additional complexity of the sample fixing. In 4C, the sample placement has a significant impact on the cathode produced gasses. Instead, 4B position showed stability, reproducibility, and enough uniform layer creation with a relative ease of sample operation.

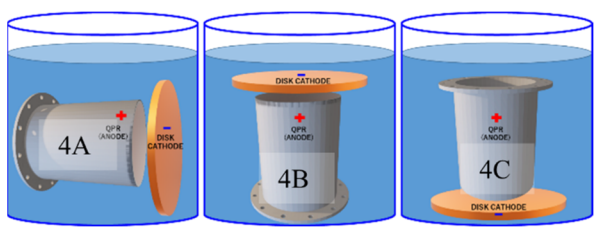

Figure 4: Tested EP setups. 4A – horizontal; 4B – upper vertical, 4C – down vertical placement.

The oxygen production during the electrochemical process is almost impossible to avoid in case of Cu EP., so that it can be decreased, and in fact, lower voltage is the right tool in case of treatment lasting more than 10 minutes. A

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secondary approach was based on the viscosity of the solution and surface adsorption, which is in a direct contact with the temperature. Decreasing the viscosity can help the O_2 detaching from the surface. A heating of the solution to the 40°C has made an improvement on the quantity of pitting and gas trapped inside the viscous layer. As a positive side effect, it was noticed an increment of the working current density almost two times higher respect to the ambient temperature.

CONCLUSIONS

A study on the small sample surface preparation was successfully scaled to the QPR samples. A list of issues was assessed and settled.

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