ENERGETIC COPPER COATING ON STAINLESS STEEL POWER COUPLERS FOR SRF APPLICATION*

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Abstract

Delivering RF power from the outside (at room temperature) to the inside of SRF cavities (at ~4 K temperature), requires a power coupler to be thermally isolating, while still electrically conducting on the inside. Stainless steel parts that are coated on the insides with a few skin depths of copper can meet these conflicting requirements. The challenge has been the adhesion strength of copper coating on stainless steel coupler parts when using electroplating methods. These methods also require a nickel flash layer that is magnetic and can therefore pose problems. Alameda Applied Sciences Corporation (AASC) uses Coaxial Energetic Deposition (CED) from a cathodic arc plasma to grow copper films directly on stainless steel coupler parts with no Ni layer and no electrochemistry. The vacuum arc plasma consists of ~100 eV Cu ions that penetrate a few monolayers into the stainless steel substrate to promote growth of highly adhesive films with crystalline structure. Adhesion strength and coating quality of copper coatings on complex stainless steel tubes, bellows, mock coupler parts and an actual Tesla Test Facility (TTF) type coupler part, are discussed.

INTRODUCTION

In the present work our objective is to demonstrate the potential of the CED process to coat copper films on SRF power coupler parts. The details of CED process are published elsewhere [1]. We begin by demonstrating crucial requirements, such as RRR and adhesion strength that need to be fulfilled before the CED process can be considered a serious alternative to electroplating. Next, we demonstrate the capability of CED to coat complex parts. Finally, we address other issues such as thickness uniformity and average surface smoothness and establish that the CED process is ready to coat coupler parts for RF tests.

RRR OF CED COPPER FILMS

The ratio of the resistance of a film at room temperature to the resistance near 4 K temperature is called Residual Resistance Ratio (RRR) [2]. RRR is one of the crucial parameters to indirectly gauge the quality of any coated film in the SRF community [1-3]. Standards at the European X-Ray Free Electron Laser (E-XFEL) for an acceptable copper coating require RRR in the range of 30 to 80 [4]. In this section we describe the method used to measure the RRR of the CED coated copper films on stainless steel (SS) substrates.

Measuring the resistance of a metal film (Cu in this case) deposited on another metal substrate (SS in this case) is not straightforward, but we can use the formula [5] described in eqn. (1), to calculate the resistance of a copper film (R_{Cu}) at a given temperature:

$$R_{Cu} = \frac{R_{st}.R}{(R_{st} - R)} . \qquad (1)$$

Where, R is the resistance of the copper coated substrate (coating plus substrate) and R_{st} is the resistance of the stainless steel substrate.

To measure the RRR, we used four stainless steel strips of dimension 3 mm x 75 mm. Two strips were 25 µm thick and the other two were 100 µm thick. The strips were cleaned in a sonicator with trichloroethylene (TCE), acetone, and isoporpylalcohol (IPA) for about 15 minutes in each chemical. One strip of each thickness was mounted in a CED chamber for the coating. We deposited a 28 µm Cu film at ~300 ºC on both strips. After the coating, the coated and the uncoated strips were sent to Fermi National Accelerator Laboratory (FNAL) for the RRR measurement. The resistance values of all four strips (two coated and two uncoated) measured at 298 K and 4.26 K are presented in the Table 1. With these resistance values we can calculate the R_{Cu} values using eqn. 1. By taking the ratio of R_{Cu} at 298 K to that at 4.26 K, we have deduced the RRR values of CED copper films.

Table 1: RRR Values of 28 µm Cu Films on 25 µm (Sub1) and 100 µm (Sub2) Stainless Steel Substrates

#	**T(K)**	**R (Ω)**	**R_{Cu} (Ω)**	**RRR**
Sub1	298	0.308796		
Sub2	298	0.073348		
Cu/Sub1	298	0.009492	0.009793	**50.9**
Cu/Sub2	298	0.008752	0.009938	**50.9**
Sub1	4.26	0.216840		
Sub2	4.26	0.050480		
Cu/Sub1	4.26	0.000192	0.000192	
Cu/Sub2	4.26	0.000195	0.000195	

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The RRR values of the CED coated copper film were measured near 50 and are in the range required by the E-XFEL standard.

ADHESION STREGTH OF THE CED COPPER COATING

In order to demonstrate the potential of CED copper coatings in SRF applications, the key issue to be addressed is the adhesion of the coating. According to some reports [6-8], one of the major shortcomings of the electroplated copper onto SS, is inadequate adhesion strength. Several methods are employed to gauge the adhesion strength of the copper. Popular methods in the SRF community include, the High Pressure Water Rinse (HPWR) test and the cold shock test. In this section we will describe the performance of CED copper coatings subjected to these adhesion tests.

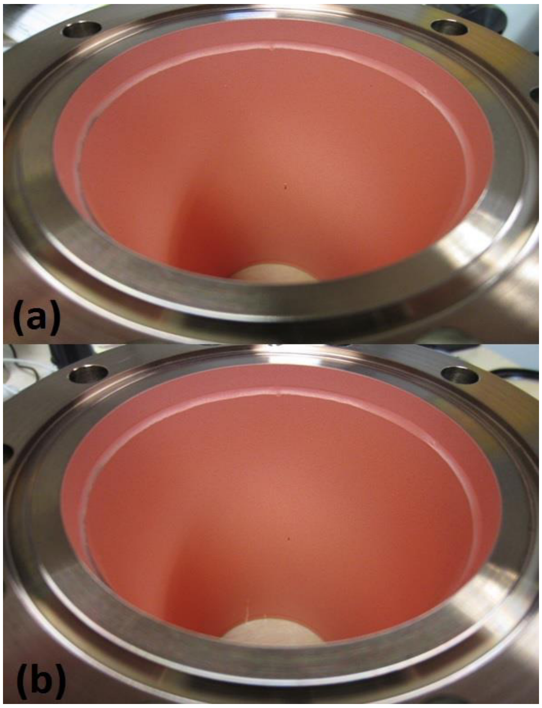

Figure 1: Lesker's off the shelf 3" ID, 7" long stainless steel tube coated with Copper. (a) Before and (b) After 10 minutes of 1250 psi HPWR test.

We purchased an off the shelf 7" long and ~3" ID SS 304L tube from Kurt J Lesker. There was no surface pre-treatment performed on the tube except TCE, acetone and IPA spray on the inside of the tube and a gentle wiping with Kimwipes. We coated it with a 50 µm thick copper film, the temperature maintained during the entire coating process was about 300 ºC and the tube itself served as anode. The coated tube is shown in Fig. 1a. After the coating the tube was sent to FNAL for the HPWR test. After 10 minutes of 1250 psi HPWR, no deleterious effects of the rinse were observed on the coating, as exhibited in Fig. 1b. This HPWR qualification encouraged us to demonstrate the adhesion strength of our coating process on more composite parts that are similar to coupler hardware.

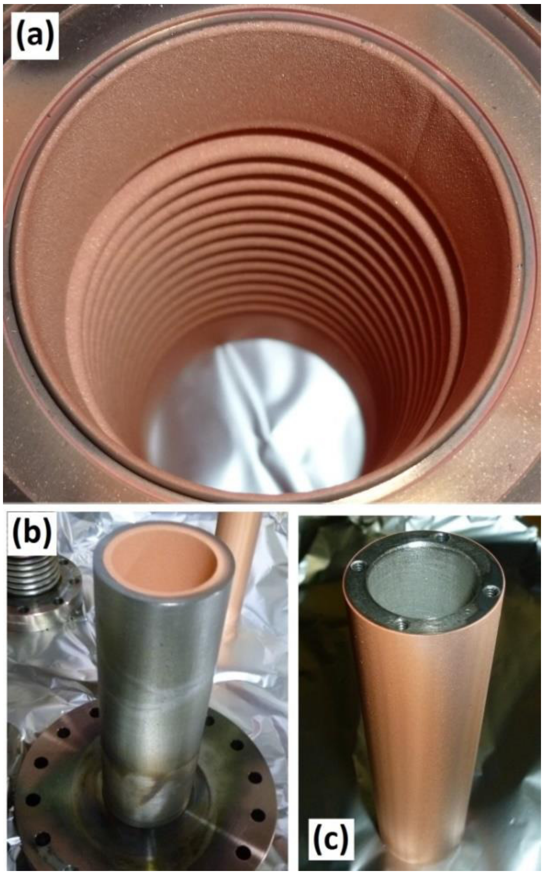

Figure 2: Copper coating on various stainless steel parts. (a) Bellows, (b) Internally coated Mock_CEC, and (c) externally coated Mock_WIC_1 tube.

To demonstrate the compatibility of our process in coating actual coupler parts we procured three different SS structures. We purchased a 6" long 2" ID hydraulically formed SS 300 series bellows from Kurt J Lesker (Part # MH-CF-D06). We designed a mock cold external conductor (CEC) coupler part and a mock warm inner conductor (WIC) tube part. The mock CEC part nearly matches, length, ID, OD and end flanges of the E-XFEL CEC coupler part and the mock WIC_1 tube matches the ID and OD of the corresponding coupler part. We coated all three parts in a single run. The CEC part was coated from inside and the WIC_1 part was coated from outside. The mock CEC and the bellows acted as anode sections themselves, while the mock WIC_1 tube was outside a SS mesh anode with 50% transmission. In order to coat the WIC_1 from the outside, we attached the part to a rotating feedthrough outboard of the anode, allowing for a uniform coating. In other words the coating uniformity was dependent on rotation steps. We selected 4 positions, 90º apart and coated the WIC_1 in 8 steps. The three coated parts are shown in Fig. 2. The non-uniform copper coating visible on the WIC_1 is due to the stepwise coating. After the coating the parts were shipped to FNAL for adhesion tests. The results of adhesion tests are presented in Table 2.

Table 2: Adhesion Results on Various SS Part Coatings

Sample	77 K Cold Shock	1200 psi rinse
Mock WIC_1	Pass	Fail
Mock CEC	Pass	Pass
6” SS Bellow	Pass	Pass

The SS bellows and the mock CEC parts qualified both the HPWR (1200 psi) and the cold shock (77 K) test. The mock WIC_1 tube qualified the thermal shock test but failed to qualify the HPWR test. The HPWR test failure on WIC_1 forced us to develop a new strategy, where we can avoid rotating parts and achieve a smooth uniform coating on a tube coated from outside. Nevertheless, we have demonstrated the versatility of our coating process, and that multiple pieces can be stacked and coated in one single coating run. We also established that our coatings possess a high degree of adhesion on inside of a tube or a bellows.

MOCK WARM INNER CONDUCTOR COATING WITH A NEW CED CONFIGURATION

Figure 3: A coated mock WIC next to a TTF WIC.

As mentioned in the previous section, our conventional approach of coating mock WIC did not yield a uniform coating. To overcome nonuniformity and lack of adhesion, we designed a new configuration to coat WIC parts. In our conventional CED coating we induce an arc from a centrally placed cathode rod to a solid or perforated anode surrounding the cathode. The material eroded from the cathode is evenly deposited on the inside of the surrounding tube. In order to coat a tube from outside we rotated the tube outboard of the anode, only coating the small section facing the cathode at each rotation stage.

In a new configuration, we reversed this geometry by designing a large hollow cathode around a centrally placed smaller OD tube anode. In this case, the mock WIC acts as the anode. The arc is induced from inside the hollow cathode and the eroded material deposits on the outside of the central anode tube. We coated a mock WIC_2 with 30 μm copper as presented in Fig. 3. The tube was cleaned in TCE, acetone and IPA in a sonicator bath and dried with N_2 before being loaded in the CED chamber for the copper coating. A 2000 Watt, 10” long cartridge heater was placed inside the mock WIC_2 tube, towards the top side. The temperature was monitored at the bottom, 1” above the 2 3/4” CF flange. The temperature during the deposition was ~270 ºC. In the central tube region the temperature must have been higher. After coating, the chamber was vented to air. Due to an error, the chamber was vented while the central region was hotter than 150 ºC, resulting in surface oxide formation and a dark dull coloration on the coating. In Fig. 3 the coated mock WIC_2 is shown next to an actual Tesla Test Facility (TTF) WIC part for comparison. The hollow 10.5” long and 2” ID copper cathode is also presented in the figure. On the mock WIC_2 there is a greyish ring 10.5” from the top, which is due to partial erosion from the SS holder of the copper cathode. After coating, the mock WIC_2 was sent to FNAL for the adhesion test.

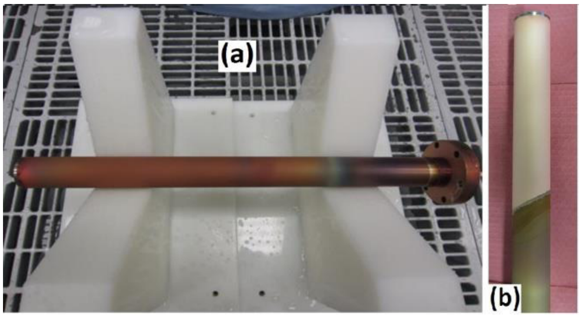

Figure 4: Mock WIC_2 at FNAL, (a) the HPWR test stand and (b) after a mild acid bath.

The FNAL HPWR test stand is shown in Fig. 4a. The mock WIC_2 was blasted with 1500 psi water. No delamination spots were observed in the top 10” of Cu coating. Two delamination spots of diameter 1-2 mm were observed in the greyish ring region, but no further peeling of the coating was observed when the 1500 psi water gun was directed along the edges of the delaminated spots. In Fig. 4b, the effect of soaking the mock WIC_2 tube in 1% Citronox Solution (a mild acid) is presented. The tube was taken out of the solution after 12 hours and a bright copper color was visible. It confirms that the discoloration occurred due to surface oxide formation.

COATING OF TTF COLD EXTERNAL CONDUCTOR PART WITH CED

To continue the momentum built with our early success in copper coating, we tried to procure currently used rf power coupler parts and coat those with the CED process. We received an old TTF CEC and an old TTF WIC from the LAL Orsay group. We decided to coat the CEC part. The CEC has an end flange of OD ~150 mm (similar to a 6" CF flange), an approximately 170 mm long tube of ID 40 mm, a 34 mm long bellows section with 9 convolutions, and a second end flange of OD 80 mm, as shown in Fig. 5. The CEC part is to be coated from inside with 10 μm copper film, including the face of the large flange all the way to just before the knife edge and on the face of the smaller OD flange, just to 3.5 mm beyond the ID of the CEC tube. We designed several custom adaptor pieces to meet these requirements.

Figure 5: TTF CEC coupler part.

The bellows on the CEC was stretched approximately 10 mm with the help of custom adaptors. The CEC coupler part was sprayed with IPA and wiped dry with Kimwipes. The temperature was maintained in the 275-300 ºC range during the coating. The inside of the CEC is shown in Fig. 6a before the coating and in Fig. 6b after the coating.

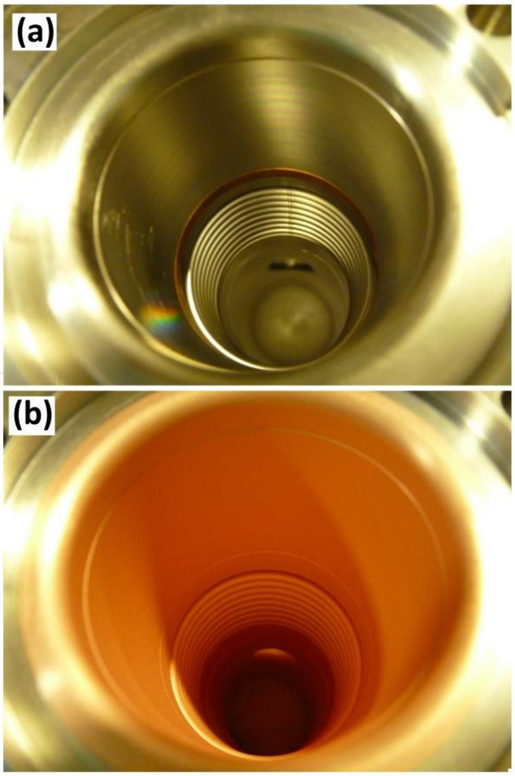

Figure 6: TTF CEC coupler part, (a) Before and (b) After copper coating.

THICKNESS UNIFORMITY AND SURFACE ROUGHNESS

The thickness uniformity required by E-XFEL standard is ±20% in the tube region and ±30% in the bellows region [9]. AASC has an optical 100x Nikon Optishot microscope, which is capable of measuring thickness uniformity in the desired region. Fig. 7 shows an optical micrograph of a copper film with an average thickness of 20 μm on a Si crystal. The image was taken from one side and the edge was cleaned with a razor blade. The striation marks on the film are visible due to the edge cleaning with the razor blade. We are in a process of obtaining multiple laser cut samples that would reveal much sharper details in optical micrographs.

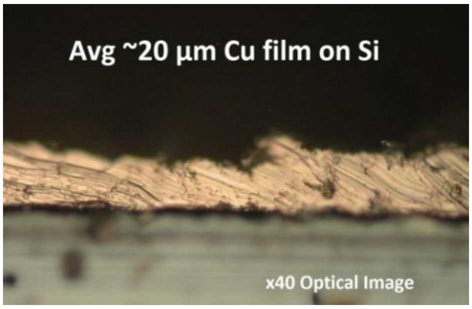

Figure 7: Optical arrangement for thickness uniformity measurement at AASC.

The CED coating has multiple macro particles on the surface. Our ongoing parallel efforts to filter these particles have resulted in a success in terms of absolute surface roughness. In Fig. 8, an atomic force microscopy (AFM) image of a thin copper film is presented. The selected film area is 2 µm x 2 µm, with an average root mean square (RMS) surface roughness of 5 nm. The efficiency of the filter is currently 10% but if we relax the surface roughness to a few hundred nm (requirement by XFEL standard is <1.6 µm [10]), the efficiency will be improved. The optimization of the macro particle filter is under progress at AASC.

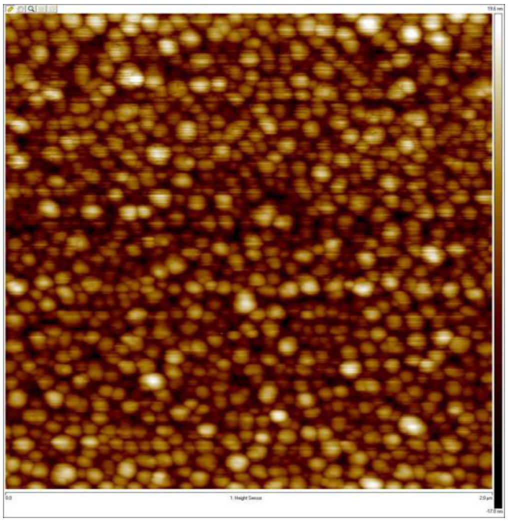

Figure 8: AFM image of CED coated Cu film. 2 x 2 µm area with an average RMS roughness of 5 nm.

We have also coated SS 316L strips of dimension 80 mm x 5 mm x 0.5 mm. In Fig. 9a and 9b, the mounted strips before and after the copper coating are shown. These strips will be tested for thickness uniformity, average surface roughness and RRR at LAL Orsay.

Figure 9: SS 316L strips, (a) before and (b) after 30 µm copper coated film.

CONCLUSION

In conclusion, we have demonstrated the potential of the CED copper films in SRF power coupler parts. The quality of the CED copper films has surpassed the minimum RRR requirement of 30. The adhesion strength of the CED copper films is well above 1000 psi water rinse and thermal shock of (room temperature to) 77 K. It is important to mention that the high adhesion strength of the CED copper films is achieved without using deleterious nickel flash layer. We have demonstrated copper films with very smooth surface finish using a macro particle filter. We have also successfully demonstrated coating of actual coupler parts with the CED process. With our early success, we are poised to take the CED coating to the next level and coat coupler parts for RF tests.

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