ISBN: 978-3-95450-232-5 ISSN: 2673-7000 doi:10.18429/JACoW-NAPAC2022-MOPA62

HIGH QUALITY CONFORMAL COATINGS ON ACCELERATOR COMPONENTS VIA NOVEL RADIAL MAGNETRON WITH HIGH-POWER IMPULSE MAGNETRON SPUTTERING

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Abstract

In this work, we present two configurations of a novel radial magnetron design which are suitable for coating the complex inner surfaces of a variety of modern particle accelerator components. These devices have been used in conjunction with high-power impulse magnetron sputtering (HiPIMS) to deposit copper and niobium films onto the inner surfaces of bellows assemblies, waveguides, and SRF cavities. These films, with thicknesses of up to 3 µm and 40 µm for niobium and copper respectively, have been shown to be conformal, adherent, and conductive. In the case of copper, the post-bake residual resistivity ratio (RRR) values of the resulting films are well within the range specified for electroplating of the LCLS-II bellows and CEBAF waveguide assemblies. In addition to requiring no chemical processing beyond a detergent rinse and solvent degrease, this magnetron design exhibits over 80% target material utilization. Further, in the case of niobium, an enhancement in RRR over that of the bulk (target) material has been observed.

INTRODUCTION

This work continues the investigation of the use of ionized physical vapor deposition (iPVD) as an alternative process to wet chemical electroplating for depositing conformal coatings of various materials for use in accelerator components. Previous investigation utilized a novel radial magnetron design used for coating the inner diameter of LCLS-II bellows components which resulted in conformal, well adhered films with thicknesses of 5 – 10 µm. Resulting films were capable of withstanding extreme temperature fluctuations (77 K to 400 °C vacuum bake-out) and remained well adhered after plastic deformation [1]. Utilizing the results from this previous work, efforts were focused on optimizing RRR values for LCLS-II coatings, and translating similar process conditions for use in coating CEBAF waveguides – with the added challenge of determining process conditions that work well with the high aspect ratio of the waveguides.

As is the case for Cu coatings on other accelerator components, different methods for coating SRF cavities with Nb thin-films have been attempted in the past with varying results [2]. One of the key parameters that determines the effectiveness of Nb thin-fims for superconductivity applications, is the grain structure of the resulting film [2]. Part of this work focused on determining process conditions which result in well adhered, high-RRR Nb films on Cu test coupons.

EXPERIMENT

HiPIMS Operation

Each magnetron was driven by a Starfire Industries IMPULSE® 20-20 HiPIMS pulser module. Argon was used as the carrier gas at varying pressures during parameter exploration and optimization. Using HiPIMS with a Positive Kick™ allows ions to be accelerated to energies in the range of 0 – 400 eV. This allows the target material to be implanted into a substrate, resulting in a transition layer that produces better film adhesion. In addition, the Positive Kick enables the formation of fully dense thin-films by implanting metal ions into the bulk film [3]. This is in contrast to other deposition processes where metal ions implant to the surface of the film only, often leading to pinholes and large voids.

Magnetron Design

Two magnetron designs were built and tested: a 0.5" (1.27 cm) diameter magnetron with an azimuthal racetrack and a 1” (2.54 cm) diameter magnetron with an axial racetrack. Both designs were tested with various materials. Niobium and copper were tested on the 1" design, while silicon carbide and copper were tested on the 0.5" design. Both magnetrons are comprised of a copper body that facilitates heat transfer between coolant and the magnet pack(s). For copper magnetrons, the copper shell itself acts as the target material. Details regarding the design and construction of the 1" magnetron design are given elsewhere, in Ref. [1]. Figure 1 shows a schematic of the 0.5" magnetron used for coating CEBAF waveguides.

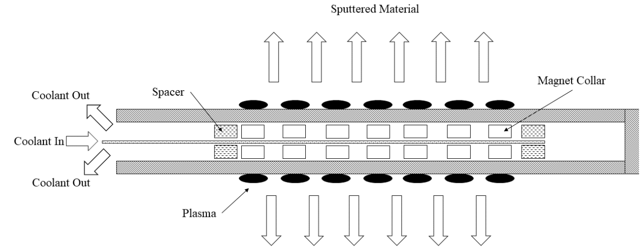

Figure 1: Diagram of 0.5" outer diameter magnetron for coating CEBAF waveguides. Magnet pack creates distinct erosion patterns around each stack of magnets.

LCLS-II Bellows

Optimization was carried over a large parameter space of operating pressures and pulse settings. Efforts focused

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5th North American Particle Accel. Conf. NAPAC2022, Albuquerque, NM, USA JACoW Publishing
ISBN: 978-3-95450-232-5 ISSN: 2673-7000 doi:10.18429/JACoW-NAPAC2022-MOPA62

on maximizing the residual resistivity ratio (RRR), conformality, and adhesion. RRR values were measured using the Dynacom Physical Property Measurement System (PPMS). Measurements were performed using a four point probe in a cryogenic chamber. Conformality was determined based on visual inspection of scanning electron microscope images of sample cross sections. Adhesion was measured by successive pass/fail tests consisting of (1) baking samples at 400 °C in vacuum for one hour followed by (2) submersion in a bath of liquid nitrogen (77 K).

These depositions were performed using a 1" diameter radial magnetron. Each deposition process was comprised of a plasma clean step, two ion-implant steps, and a main deposition step. The plasma clean step is used to prepare the substrate by etching away oxides and adsorbed gasses. The first implant step is used to create an intermixing layer between the sample and the film, while the second implant step creates a material gradient to aid in Cu adhesion to the substrate. Both steps utilize high positive pulse voltages in the approximate 150 – 300 V. The process conditions for the final deposition step were adjusted to maximize conformality, adhesion, and RRR.

CEBAF Waveguides

Stainless steel and silicon witness coupons were mounted to a test fixture approximating the CEBAF waveguide geometry, which is shown in Fig. 2. Following a deposition, the witness coupons were inspected via SEM to extract film thickness as a function of position within the high-aspect-ratio waveguide structure.

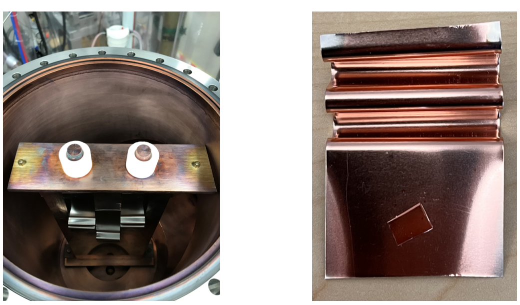

Figure 2: Stand-in volume for CEBAF waveguide (left). Example test coupon used for characterizing film uniformity (right).

SRF Cavities

A 1" radial magnetron was jacketed in niobium for depositing films onto the inner surface of copper SRF cavities. To keep the magnetron free of exposure to atmosphere, a retracting magnetron design was used to keep the it at vacuum during sample cycling. This magnetron totaled 8'(2.44 m) in length and had a 21" (53.34 cm) rotating magnet pack. Sapphire and electropolished copper (C10100) coupons were used to explore the parameter space. Additionally the chamber was heated to the approximate range of 200 – 400 °C.

RESULTS AND DISCUSSION

LCLS-II Bellows

Overall, RRR values in the approximate range of 10–70 were achieved, although higher RRR values correlated with reduced conformality. RRR measurements of the best films (best being a subjective mix conformality, adhesion, and RRR) were 45 at the peak of the corrugation (nearest to plasma) and 43 at the trough (furthest from plasma). For these same films, thickness of 42 µm and 20 µm were measured at the peak and trough, respectively.

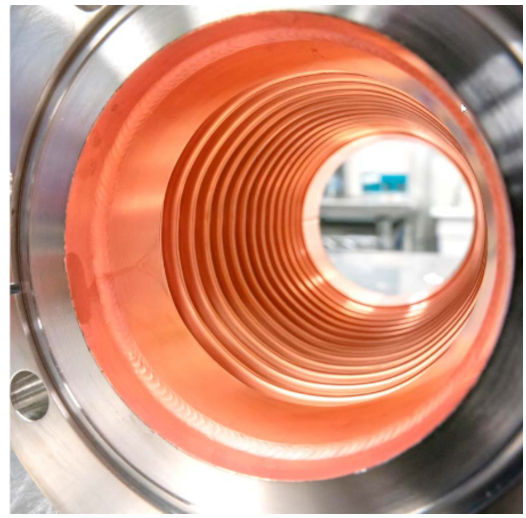

Figure 3: Coated LCLS-II bellows section using 1" magnetron design.

The process conditions for the best films produced a high density discharge which aided in creating a densely packed film. Too high of a plasma density, however, resulted in a drop in RRR. This threshold occurred at around 2 A/cm^2 of peak current density at the magnetron. This is believed to be caused by damage to the underlying crystal structure due to the increasing density of doubly ionized copper in the plasma. Additionally, lower positive pulse voltage may be beneficial for balancing conformality and ion energy with the other plasma properties. An example coated bellows section is shown in Fig. 3.

CEBAF Waveguides

Results show that the best azimuthal uniformity for the dual magnetron configuration was achieved with short negative pulse widths and high negative pulse voltages. Figure 4 shows the effects of negative pulse length on the deposition rate at various points on the waveguide. The high aspect ratio of the CEBAF waveguide results in a widely varying gap between the magnetrons and the walls of the sample ranging from 0.25" – 1.25". At the energies expected during HiPIMS for copper neutrals (0.1 – 0.25 eV), transit time from target to substrate is on the order of a few microseconds. With negative pulse widths above 5 µs, copper neutrals expelled form the surface of the magnetron collide with the waveguide before there is time for the positive pulse to ionize

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them and redistribute them. This is similar to the results obtained during LCLS-II bellows coatings – indicating good portability of process conditions for coating both geometries. Further optimization will continue after a third magnetron is installed to increase conformality.

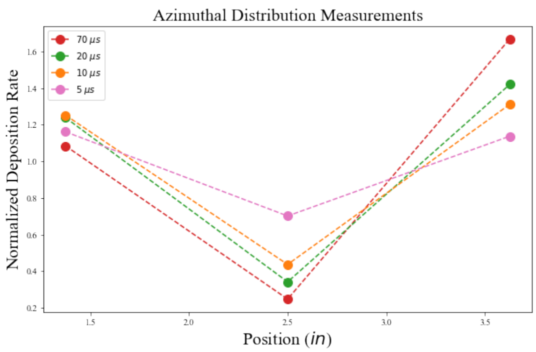

Figure 4: Deposition rate measurements at various negative pulse conditions. Measurements were taken directly in front of each magnetron and at a half-way point between them.

SRF Cavities

RRR of niobium on copper films varied little and stayed consistently in the approximate range of 30 – 40 range. Investigation revealed the target material used in the magnetron was the limiting factor, having a RRR of 13. Table 1 lists process conditions and RRR values for the bulk Nb and two select process conditions that achieved the highest RRR values.

Table 1: Niobium RRR of Films and Magnetron Target

Sample	Peak Current (A)	Kick Voltage (V)	Power (W)	RRR
Bulk Material	N/A	N/A	N/A	13
Best Film #1	16	90	700	40
Best Film #2	24	90	1100	39

These results suggest that creating SRF niobium films is feasible with HiPIMS and the radial magnetron presented in this work, and that these methods might be suitable even for improving RRR above that of the bulk material. A photo of this deposition process in progress is shown in Fig. 5. To achieve RRR's greater than 100 it would be necessary to use niobium target material with a RRR in that range. The main difficulty is sourcing high-RRR (RRR300) niobium in the required cylindrical geometry for magnetron fabrication.

CONCLUSION

Using the novel radial magnetrons presented here in conjunction with HiPIMS with Positive Kick(TM), adherent, conformal, and high quality films have been developed for

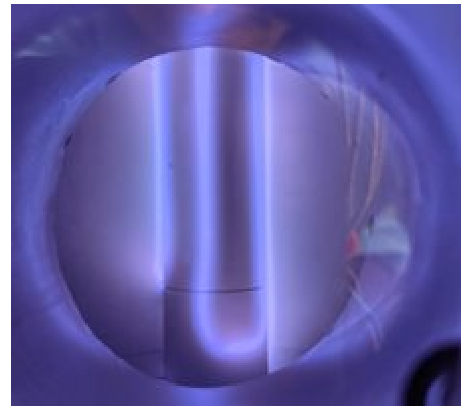

Figure 5: The bottom turn-around region of the niobium radial magnetron while actively depositing material with a HiPIMS plasma.

accelerator components. This work has focused on optimizing film adhesion, conformality, and RRR values for copper films intended for LCLS-II bellows assemblies and CEBAF waveguides, and for Nb films intended for SRF cavities. Future work will focus on further improving these values in copper and niobium, the latter of which will almost certainly require procuring RRR300 Nb in a cylindrical form factor.

ACKNOWLEDGEMENTS

This material is based upon work supported by the U.S. Department of Energy under Award Numbers DE-SC0019784 and DE-SC0020481. The views and conclusions contained in this document are those of the authors and should not be interpreted as representing the official policies, either expressly or implied, of the DOE or the U.S. Government.

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