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HIGH-QUALITY, CONFORMAL BELLOWS COATINGS USING ULTRA-FAST HiPIMS WITH PRECISION ION ENERGY CONTROL

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Abstract

In this paper we demonstrate a replacement for traditional 'wet' chemical deposition processes using a vacuum, ionized physical vapor deposition (iPVD) process that results in a conformal metal film, capable of coating complex, convoluted parts that are common in modern particle accelerators (e.g., bellows, RF cavities). Results are presented for a process utilizing the combined deposition and etching that are achieved using ultra-fast high-power impulse magnetron sputtering (HiPIMS) coupled with precision control of the ion energy using a positive voltage reversal. This process results in a conformal film and has been used to coat both test coupons and full bellows assemblies. The resulting Cu films, which are 5-10 µm in thickness, exhibit excellent adhesion. Further, they have been shown to tolerate temperature extremes ranging from 77 K to a 400 C vacuum bakeout as well as extreme plastic deformation of the substrate without any buckling, cracking, or delamination.

INTRODUCTION

This work investigates the use of a conformal iPVD process and a novel radial magnetron design to replace wet chemical electroplating (e.g. Cu) for stainless-steel bellows and other specialty vacuum components used on accelerator structures. Wet chemical electroplating is being progressively phased out due to its damaging environmental impact, hazardous chemical handling, high cost, and lack of experienced tradespeople in the field. Further, years of attenuation have left only a small handful companies in the US that perform such coatings. Often multiple customer parts will be run using the same tanks, electrodes, and recirculating chemical baths creating embedded impurities, non-conformal deposition and delamination leading to scrapped assemblies, rework, additional cost, and timeline growth.

Jefferson Lab has recently faced this challenge for coating high-conductivity copper onto flexible de-coupling bellows for the LCLS-II cryomodule upgrade, and indeed there is an abundance of literature surrounding the bellows themselves that expresses difficulty with the electroplated Cu films. [1-3] These issues include surface finish/roughness (including macroscopically visible striations in the plating), [1, 2] inclusions, [2, 3] particulates from both the copper plating itself, as well as those potentially introduced during the electroplating or subsequent surface smoothing steps (e.g. Mo-wool polishing or bead blasting). [1, 3] Repeated electroplated part failures have driven up cost, introduced risk and extended timelines for introduction of components. Starfire has similar experiences working with plating vendors for high-conductivity copper on aluminum for accelerator structures. The desired characteristics of the coated film are that it is thick, adherent, and electrically conductive.

EXPERIMENT

Magnetron Design

A custom magnetron was designed to perform depositions on the inner surface of tube-like structures with ID's above 1.5" and extended lengths. This design is shown during an *in-situ* etch/pre-clean step and during a Cu deposition in Fig. 1. It employs a compact magnet pack that produces a serpentine racetrack on the outer diameter of a cylindrical target. The single, continuous racetrack helps to ensure a uniform plasma. The coolant connections (in/out) and the HV contact are all located at one end of the magnetron, leaving the other end open for mounting/dismounting cylindrical objects. The magnetron shown in Fig. 1 has an outer diameter (OD) of 1.0" and an active region that is approximately 6" in length. The target material is C10100 (OFE Cu).

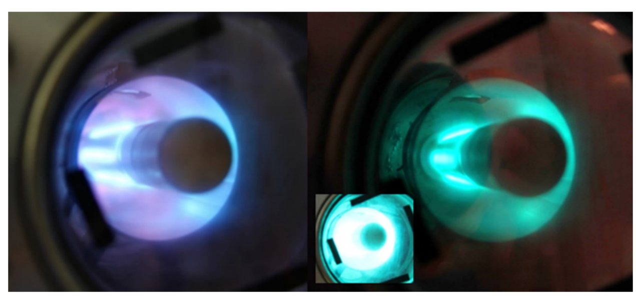

Figure 1: The first of two prototype magnetrons used for this work, shown here during an in-situ plasma etch/pre-clean step (left) and during a Cu dep (right) with a reduced exposure time due to the brightness. The inset photograph was taken with no reduction in exposure time relative to the photograph of the in-situ pre-clean step.

A second, revised design was constructed with a rotatable magnet pack. The magnet pack design was further altered to place the three end regions (where the racetrack turns around) at slightly different axial positions. The combination of these two design features was intended to spread the erosive load during operation and result in a dramatic increase in target lifetime/utilization and a dramatic reduction in the drift in operating parameters due to aging effects (see Ref. [4] for a discussion of these effects). Figure 2 shows the erosion patterns produced by the stationary (top) and rotating (bottom) magnet packs. The sta-

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tionary magnet pack results in a serpentine erosion pattern, with the greatest erosion occurring at the end regions. In stark contrast to this is the erosion profile of the rotating design, which is extremely uniform and contains a series of steps at each end corresponding to the staggered locations of the racetrack end regions. Based on the measured erosion profile, the target utilization of the rotating design is estimated to exceed 90%.

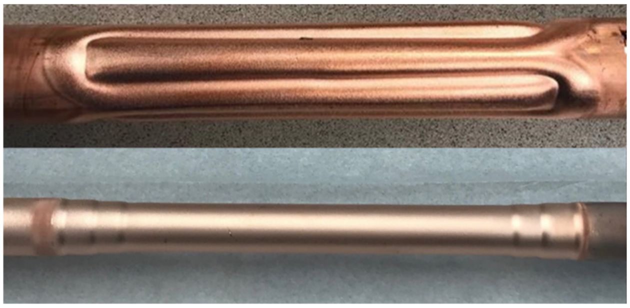

Figure 2: The erosion patterns produced by the stationary (top) and rotating (bottom) magnet packs.

In addition to its small size, an important distinguishing feature between this magnetron and other rotating radial magnetrons is that this design has no moving, vacuum-facing components. Whereas typical rotating magnetrons employ a mobile (rotating) cathode surface with brushed electrodes, [4] this design instead makes use of a rotating, internal magnet pack and a fixed electrical contact. This allows for kA-level currents, far exceeding what is required for HiPIMS operation. Additionally, particle generation is minimized by the fact that none of the vacuum facing surfaces/components move.

Sample Fixturing

Three types of substrates were used for these experiments: 1) sample coupons formed into the approximate shape of the hydro-formed bellows, 2) wire-EDM-cut sections of an LCLS-II bellows assembly, and 3) off-the-shelf (OTS), 4.5" CF-flanged bellows assemblies having dimensions approximately equal to the LCLS-II bellows. A cylindrical holder, shown in Fig. 3, was designed for the sample coupons and LCLS-II bellows sections.

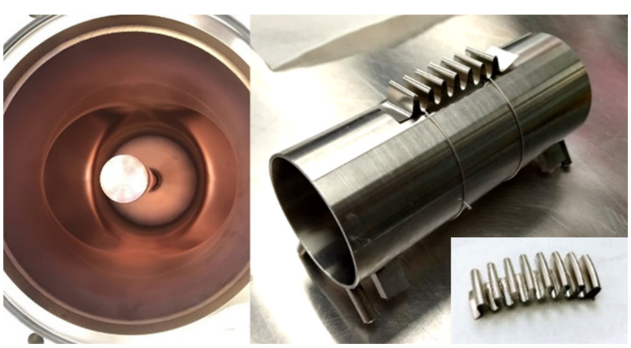

Figure 3: The sample holder (right) and the magnetron around which it mounted (left).

It is a 316 stainless steel cylinder having an inner diameter (ID) of 2.35" with a 1.5" × 0.75" window cut into the wall. The sample coupon or LCLS-II bellows section is strapped over the window and held in place with steel lock-wire, as shown in Fig. 3. The fixtured assembly is then moved into position, such that the active region of the radial magnetron is centered within the cylindrical sample holder.

HiPIMS Operation

Depositions were performed using a Starfire Industries IMPULSE® 20-20 HiPIMS pulser module and two Magna-Power SL-series DC power supplies. Argon was used as the process gas at pressures of 0.1-4 Pa. Pulse voltages, widths, and repetition rates were varied over a large range for this exploratory work. In general, the deposition process was performed in four phases:

1. **In-situ plasma clean (~20 minutes):** A short negative pulse strikes a plasma followed by a longer high-voltage (≥ 300 V) positive pulse to etch the surface of the substrate.
2. **Implantation/intermixing (~5 minutes):** The negative pulse is lengthened to rarefy the plasma. A high-voltage positive pulse is applied to drive the metal ions into the surface of the substrate. Etching also occurs during this step.
3. **Transition layer (~5 minutes):** Pulse settings are transitioned from the implantation/intermixing process conditions to the thick-film process conditions.
4. **Thick film (~100 nm/min):** Low- to moderate-voltage (50-100 V) positive pulses are used for this step. Pulse repetition frequency and peak current are adjusted as needed.

Fatigue Tester and Conductivity Measurements

A custom fatigue tester, shown loaded with a Cu-coated sample in Fig. 4, was constructed for this work. Technical specifications for LCLS-II bellows require that they survive 6,000 cycles at a stroke of ± 6 mm. The fatigue tester was set to cycle at 3 Hz for these experiments. A custom pulsed-DC resistivity meter was also built for this work. This meter simply pulsed a current of approximately 10 A through the series combination of the sample and a current-sense resistor. A ratio of the two voltages then yields a ratio of two resistances.

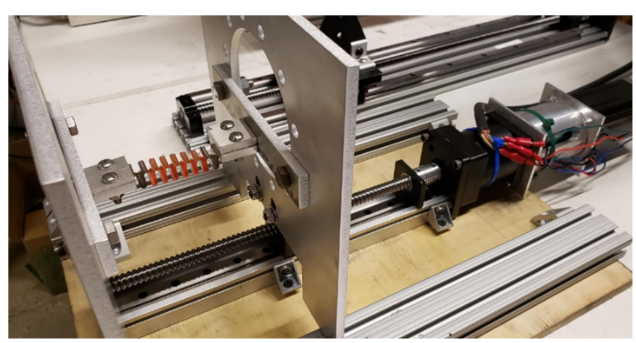

Figure 4: A custom fatigue tester constructed for this work.

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RESULTS AND DISCUSSION

Test Coupons

Most of the process development for this work was conducted using test coupons and LCLS-II bellows sections. Under certain process conditions, it was possible to repeatably achieve adherent, thick, conductive films. Table 1 lists conductivities (%IACS) that were obtained under various pulse conditions. The symbols (*) and (**) in the table denote minor and major delamination, respectively, upon plastic deformation of the substrate. The process gas used for these data was Ar at a pressure of 0.5 Pa. In most cases, these films were adherent enough to survive severe plastic deformation, as shown for a 5 µm Cu film in Fig. 5. Even after the coupon was stretched into a flattened state, no buckling, cracking, or delamination was observed in the film. In general, Positive Kick™ voltages in the range of 50-100 V resulted in the best adhesion over a wide range of peak currents. For voltages significantly above 100 V, the films were consistently observed to crack and delaminate, often even under elastic deformation.

Table 1: Cu Film Conductivities (%IACS)

Peak Current (A)	Kick Voltage (V)		
	25	50	100
50	70%	67%	69%
100	73%*	65%	86%
150	54%**	90%	69%

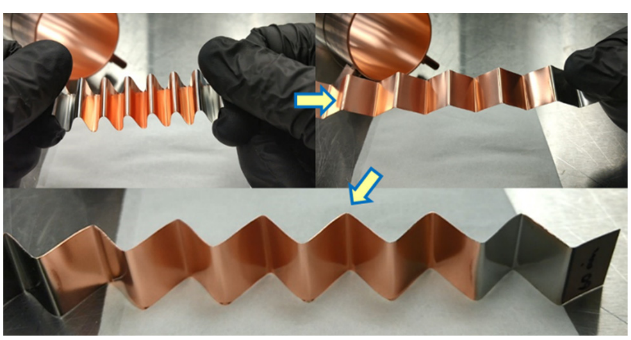

Figure 5: A sequence of images showing the plastic deformation of a substrate with a 5 µm Cu film.

Bellows Assemblies

Following the process development on test coupons, two processes were selected for deposition onto full bellows assemblies—one at 0.53 Pa and one at 2.7 Pa. A photograph of the resulting film and a cross section of the bellows is shown in Fig. 6. As with the test coupons, these films were again adherent, conformal, and conductive.

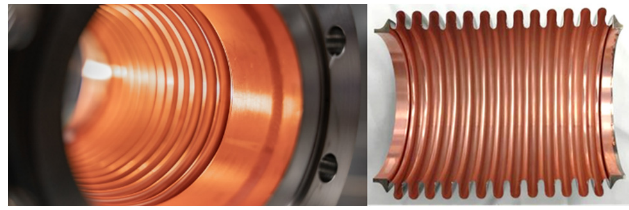

Figure 6: A photograph of the Cu-coated bellows and a cross section of the bellows prepared via wire-EDM.

After being cross sectioned, samples of the Cu film were peeled off of the substrate for Residual Resistance Ratio (RRR) measurement. These samples were taken from four locations: the apex, trough, and wall of a bellows cycle, and the tube section (flat) which connects the bellows to the flange. The measured RRR values are given in Table 2. While the 2.7 Pa process resulted in significantly higher RRR values than the 0.5 Pa process, the anomalously low values recorded in the trough in both cases are presently unexplained.

Table 2: Measured RRR Values for Cu Films

Location	Process Pressure 0.5 Pa	Process Pressure 2.7 Pa
Apex	14.33	22.93
Wall	9.53	23.42
Trough	6.07	6.74
Flat	10.35	24.86

CONCLUSION

A novel, radial magnetron suitable for small-ID parts was demonstrated and driven via HiPIMS w/ Positive Kick™ to produce adherent, conformal, conductive films. This capability was demonstrated both on test coupons and on full bellows assemblies. The measured RRR values were somewhat lower than expected, especially in the trough of the bellows. The reason for this is not yet known. Future efforts will be directed towards identifying any contaminants or impurities that might result in the low RRR as well as towards a full HiPIMS parameter optimization to maximize this value.

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