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Development of Spray Coated Cathodes for RITS-6

Sean Simpson, Joshua Leckbee, and Stephen Miller

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Sandia National Laboratories
Albuquerque, New Mexico 87185 and Livermore, California 94550

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Sean Simpson
Coating and Surface Engineering Department
Joshua Leckbee
Advanced Radiographic Technologies Department
Stephen Miller
Coating and Surface Engineering Department

Sandia National Laboratories
P.O. Box 5800
Albuquerque, New Mexico 87185-MS1130

Abstract

This report documents work conducted in FY13 to conduct a feasibility study on thermal spray coated cathodes to be used in the RITS-6 accelerator in an attempt to improve surface uniformity and repeatability. Currently, the cathodes are coated with colloidal silver by means of painting by hand. It is believed that improving the cathode coating process could simplify experimental setup and improve flash x-ray radiographic performance. This report documents the experimental setup and summarizes the results of our feasibility study. Lastly, it describes the path forward and potential challenges that must be overcome in order to improve the process for creating uniform and repeatable silver coatings for cathodes.

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NOMENCLATURE

Al	Aluminum
Cu	Copper
DOE	Department of Energy
EDS	Energy Dispersive X-ray Spectroscopy
FIB	Focused Ion Beam
NNSS	Nevada national Security Site
RITS-6	Radiographic Integrated Test Stand /w Six Inductive Voltage Adder Cells
SEM	Scanning Electron Microscope
SNL	Sandia National Laboratories
TSRL	Thermal Spray Research Lab

1. INTRODUCTION

The Radiographic Integrated Test Stand (RITS-6) diode program is focused on improving pulsed power-driven electron beam sources used for intense flash x-ray radiography [1]. It is believed that improvements to the surface preparation and uniformity of a cathode could improve the radiographic performance of these electron beam diodes by reducing the spot size of the beam and possibly increase the x-ray yield. Currently, the cathodes are hand-painted using a colloidal silver solution which inevitably introduces variability in the coating thickness, uniformity and possibly in the coating density [2]. Also, it is not yet understood what effect the coating conductivity, microstructure features, coating density or coating material may have on the performance of the cathode.

This report presents the experimental results of coating a typical aluminum cathode used in the RITS-6 accelerator with pure aluminum (Al) via the use of a plasma spray torch. An aluminum coating was chosen for the initial feasibility study due to the well understood nature of spraying aluminum with the Triplex plasma spray torch and it was readily available.

2. EXPERIMENTAL SETUP

The setup used for spray coating the cathode is depicted in Fig. 1. The torch parameters were determined from a cut sheet provided by Sulzer-Metco for spraying aluminum. The geometric parameters for the torch and the rotational stage were based on previous experience with similar parts at the Thermal Spray Research Lab (TSRL). The spray geometry was configured for a conical part with a constant 30 degree inclination with respect to the axis of the cathode. This implies that the standoff distance was not constant across the entire sprayed area, but given the small part size, the error was expected to be negligible. The coating uniformity could likely be improved by implementing a hemispherical or arc geometry.

Figure 1: Experimental setup for coating the cathode. Pictured is the Triplex with the cathode (wrapped with red tape) mounted to a rotational stage. The rate of rotation and the trajectory of the torch are calculated such that the coating is near uniform.

Prior to coating the cathode, it is masked using Kapton tape, grit blasted using the Washington Mills using Duralum Special White, then cleaned with acetone, and rinsed with isopropanol as depicted in Fig. 2 and the corresponding SEM image depicted in Fig. 3. The grit blaster pressure was set to ~60 psi, which should result in an average surface roughness of $\sim 6 \mu\text{m}$ on aluminum (based on previous characterization of the grit blaster). This is a typical process for thermally sprayed coatings at TSRL. For completeness, the cathode depicted in Figures 2 (top) and 3 was prepared using only the grit blasting process and delivered for future testing on the accelerator.

Figure 2: The top cathode prepared for thermal spray coating. The amber layer is the kapton masking tape. The red layer is silicon tape used to cover the remainder of the cathode. The cathode was grit blasted using the Washington Mills, cleaned using acetone, and rinsed with isopropanol. The bottom cathode thermally sprayed with aluminum and has all of the masking removed.

Figure 3: SEM image of the cathode after grit blasting in the Washington Mills. This image depicts the surface features prior to coating.

The aluminum powder used for coating the cathodes in this feasibility study consisted of an in-house powder from Belmont, manufactured in 1998. The mean diameter was 51.8 μm with a standard deviation of 28.6 μm. The TSRL part number is P0479. An in-house measurement was performed using the Beckman Coulter Laser Diffraction Particle Size Analyzer. The results of this characterization are presented in Fig. 4.

Figure 4: Particle size distribution for the Belmont 50 μ m aluminum powder used to coat two cathodes. Analysis performed using the Beckman Coulter Laser Diffraction Particle Size Analyzer.

The powder gas consisted of argon set to a flow rate of 5 standard liters per minute (SLPM) at a pressure of 230 mBar. The hopper vibration was set to 2000 mbar and the feed rate set to 40 gr/min.

A copper (Cu) coupon was initially sprayed using the above configuration to determine how many passes of the torch were required to produce a fairly uniform coating. As can be seen in Figures 5, 6, and 7, a fairly uniform coating was created using just a single pass of the torch. Based on this result, two cathodes were sprayed using the above spray conditions. One cathode was destructively analyzed to determine the coating uniformity while the second cathode was delivered for future testing on the RITS-6 accelerator.

Figure 5: Image depicting aluminum coating on a copper coupon using only a single pass of the torch.

Figure 6: SEM image of aluminum sprayed on a copper coupon.

Al_on_Cu_map1

EDS spectrum taken over entire image area

Al_on_Cu_map1

Figure 7: EDS map of aluminum sprayed on copper coupon along with EDS spectrum. As can be seen, the aluminum coating is fairly uniform over the copper surface for a single pass of the torch. The aluminum is indicated in red whereas the copper substrate is indicated in blue.

3. RESULTS

The small diameter of the cathode proved a little difficult to spray. This particular geometry required a compromise of rotational speed of the spindle versus the linear speed of the torch in order to avoid “candy striping”, or an uneven axial distribution of the coating. The smaller the diameter of the part, the faster the spindle must turn in order to achieve the same equivalent speed in mm/s. The overall spray speed was lowered to 100 mm/s, which is much slower than typical spray speeds of 500-800 mm/s used for parts processed in this manner. Since the speed of the part inversely affects the thickness of the coating per pass of the torch, a thinner coating may not be achievable without reducing the flow rate of the powder into the plasma or by increasing the rotational speed of the spindle.

Figures 8 and 9 depict the aluminum coating at the boundary where the Kapton tape was used to mask the remainder of the cathode. As can be seen in Fig. 8, the spray penetrated ~200 μm under the Kapton tape resulting in a thin deposition of aluminum and possibly other contaminants such as the adhesive used on the Kapton tape.

Figure 8: SEM image of aluminum coating at the boundary where the kapton tape was used to mask the remainder of the cathode. The aluminum coating is the bottom half of the image, the upper half is the machined aluminum cathode as provided. Note the scale is 200 μm .

Figure 9: Zoomed in SEM image of Fig. 8 depicting the boundary between the cathode and the sprayed region. Note the scale is 100 μm.

Figure 10 depicts the coating morphology near the end of the cathode. Figures 11 and 12 are zoomed SEM images of the same region.

Figure 10: SEM image of the aluminum sprayed coating near the end of the cathode.

Figure 11: Zoomed SEM image of Fig. 10.

Figure 12: Zoomed SEM image of Fig. 11.

Further observation of Fig. 12 reveals that there are several features smaller than 10 μm which may act as accumulation points or field enhancements for electrons. The submicron features, which appear as fuzz, may be a result of oxidation of the aluminum during the time of flight from the Triplex torch till deposition on the cathode sample. If the oxidation is determined to be undesirable, it may be possible to spray the aluminum using hydrogen which will act as an oxygen getter.

Figures 13 – 16 depict the aluminum coating morphology at the radius of the cathode at various levels of zoom.

Figure 13: SEM image of the aluminum coating at the radius of the cathode. The defocused image towards the bottom of the image is the interior of the cathode.

Figure 14: Zoomed SEM image of Fig. 13.

Figure 15: Zoomed SEM of Fig. 14.

Figure 16: Zoomed SEM image of Fig. 15.

The morphology depicted in Fig. 16 suggests that the aluminum powder is sufficiently heated during the transport time and is clearly molten upon deposition on the cathode. The small spherical particles are indication of splashing behavior of molten aluminum at impact, which is indicative of good coating adhesion.

As mentioned above, a second cathode was sprayed using similar conditions as the first. This cathode was used to perform focused ion beam (FIB) analysis. In this manner, the sample is precision etched allowing a view of the layers comprising a coating. For this sample, the FIB was limited to a maximum depth of $\sim 70\ \mu\text{m}$ into the coating as seen in Fig. 17 along with the corresponding EDS images in Fig. 18.

Figure 17: FIB analysis after etching the aluminum coating. The depth achieved was ~70 μm which is about the limit of the FIB device.

Figure 18: EDS analysis showing the layer is composed of pure aluminum. Also included is map of oxygen (red) and gallium (teal). The presence of gallium is due to the FIB process.

It is important to note that the FIB and EDS analysis indicates some porosity in the aluminum coating which could potentially have an effect on the overall conductivity of the cathode. Also note, the EDS results suggest the aluminum is quite pure with oxidation being localized primarily to the surface of the coating and at the boundaries between aluminum ‘splats’. If necessary, the oxidation of a molten metal can be reduced through other spray techniques.

A metallographic cross-section was prepared on the second cathode in order to get a macro scale characterization of the coating uniformity as depicted in Fig. 19. The top of this image corresponds to the outer diameter of the sprayed cathode. The mean coating thickness was found to be $89\text{ }\mu\text{m}$ with a standard deviation of $16.7\text{ }\mu\text{m}$ based on 20 measurements performed as depicted in Fig. 20. The maximum and minimum heights were $129\text{ }\mu\text{m}$ and $61\text{ }\mu\text{m}$ respectively; however, these do not accurately reflect the coating thickness since these measurements were made from an arbitrary line near-parallel to the original cathode surface.

Figure 19: Second cathode via metallographic cross-section. The top of the image represents the outer diameter of the cathode.

Figure 20: Analysis of surface roughness and coating thickness performed on a small region from Fig. 19.

4. PATH FORWARD

Future work is planned in which a batch of cathodes will be sprayed using a pure silver coating so that a direct comparison can be made with the hand-painted technique. This will require procuring a silver powder compatible with current thermal spray capability as well as developing a set of spray conditions due to silver's low vapor temperature, and the potential need for a denser coating. Development of spray conditions will include increasing the effective surface velocity of the cathodes by either improving the rotational velocity of the fixture or by reducing the flow rate of the powder. Additionally, fielding of the cathodes in the RITS-6 in FY14 will be required to provide a comparison between hand-painted and thermally sprayed coatings.

5. SUMMARY AND CONCLUSIONS

Coating a RITS-6 cathode with pure aluminum using plasma spray was successfully demonstrated at the Thermal Spray Research Lab. Two cathodes were sprayed with aluminum powder with mean particle diameter of 50 μm yielding a coating that was <150 μm thick with a standard deviation of 17 μm . FIB, SEM, EDS, and metallographic cross-section analysis were performed and reported. Lastly, a third cathode was prepared via means of grit blasting and analyzed using the SEM. Based on these results, it should be possible to spray a cathode with silver powder yielding improved surface uniformity and process repeatability.

6. REFERENCES

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