PURDUE UNIVERSITY
School of Materials Engineering

Exploration of Cold Atmospheric Plasma Deposition of SiOx Layer to Passivate and Insulate Niobium Feedthrough Wires

Student Team: Daniel Susalla, Julia Long-Reiser, Jonah Brinkerhuff, Katrina Evancho
Faculty Advisor: Dr. Rahim Rahimi
Industrial Sponsor: Dr. Peter Tortorici

This study explores the use of Cold Atmospheric Plasma Deposition (CAPD) to apply silicon oxide (SiOx) coatings onto niobium feedthrough wires used in pacemakers. These coatings aim to improve electrical insulation and long-term reliability while maintaining biocompatibility. Using both standard and modified CAPD methods, SiOx was deposited on Medtronic-supplied components, and coating performance was evaluated through SEM, EDS, and breakdown voltage testing. Results showed that neither method alone significantly improved breakdown voltage with ten passes, but combining the two methods yielded the high performance, indicating potential synergy. Despite elevated breakdown voltage, the combined-method coating was powdery and lacked adhesive strength. The achieved maximum thickness of the CAPD tests was 44.6 µm for 55 passes. Future work could explore alternative precursors, mechanical adhesion tests, and improved coating uniformity to advance CAPD as a viable feedthrough insulation solution.

This work is sponsored by Medtronic, Minneapolis, MN
Medtronic

Background

Medtronic provides an array of medical device solutions to ensure better quality of life. Approximately 2 billion people worldwide rely on various medical devices, yet the industry remains relatively niche compared to others in the U.S. This creates challenges within the manufacturing of such devices, making incessant innovation a necessity.

A key aspect of pacemaker function involves feedthrough wires. Electrical feedthroughs create a circuit pathway that extends from the inside of a hermetically sealed metal case to an external point while preserving the case's airtight seal. Currently, a polymer and medical adhesive are used to seal and insulate wiring, however, different materials and methods could improve these needed barriers. Insulating feedthrough wires in pacemakers is crucial to prevent electrical leakage, avoid short circuits, and ensure safe, reliable transmission of signals to regulate heart rhythms without interference.

Cold atmospheric plasma deposition (CAPD) is a tool that leverages a partially or fully ionized gas operating at room temperature and atmospheric pressure. CAP creates a mixture of charged and neutral particles, including free electrons and ions, with electron temperatures typically higher than ion temperatures. This allows CAP to interact with material surfaces to coat, clean, or modify their properties, making it ideal for temperature/pressure-sensitive applications like electrical medical devices. Unlike traditional plasma methods, CAPD does not require a vacuum, reducing both cost and complexity. Over recent decades, CAP has gained attention in the biomedical field for applications such as sterilization, wound healing, blood coagulation, cancer therapy, and immunotherapy, offering energy-efficient and accessible solutions. Silicon oxide (SiO_x) is a versatile, biocompatible material commonly used in medical devices due to its resilience and electrical/chemical inertness. The CAPD process allows for uniform, low-temperature deposition of SiO_x, making this ideal for coating delicate components. Applying a SiO_x layer enhances the durability, corrosion resistance, and electrical insulation of the wires, which are critical for the long-term stability and safety of implantable devices like pacemakers.

Experimental Design

The first step for this experiment was to coat Medtronic-supplied printed circuit boards (PCBs) and niobium wires with an SiO_x layer.
Before coating, the spray nozzle was cleaned by running isopropyl alcohol through the tubing to prevent potential contamination. Once clean, the tubing was primed with the precursor hexamethyldisilazane (HMDSN) in preparation for deposition. The fixture containing the wires and PCBs was then secured to the baseplate of the CAPD machine using tape to prevent movement. The nozzle's height, start point, and end point were adjusted and tested to ensure the target areas were within the spray zone.

For the non-modified coating method, the plasma and precursor pump were activated, and coating began at a flow rate of 10 g/hr. Each run of the program deposited one layer of SiO_x. In the modified coating method, the same setup was used, with the addition of a syringe filled with precursor taped to the nozzle. The syringe released the precursor directly into the plasma stream using a mechanical plunger, delivering at a rate of 19.584 g/hr. Combined with the existing 10 g/hr flow, the total precursor flow rate for the modified method was 29.584 g/hr. The number of coating passes applied to each board and wire sample is detailed in the accompanying table.

Following coating, the PCBs and wires were analyzed under a scanning electron microscope (SEM). SEM imaging focused on the PCB holes to assess coating thickness. ImageJ software was used to compare hole diameters and determine the thickness added per layer. Energy-dispersive X-ray spectroscopy (EDS) was also performed on the same regions to determine the elemental composition of the coated surfaces.

The final test measured the breakdown voltage of each coated wire. A variable power supply capable of delivering up to 10,000 V was connected to the sample wire and a 3900 Ohm resistor. The resistor was then brought into contact with the coated wire to complete the circuit. Voltage was slowly increased until the circuit breaker tripped, and the voltage dropped. The voltage immediately before the drop was recorded as the breakdown voltage. This procedure was repeated for each coated wire sample.

Sample	Non-Modified Passes	Modified Passes
1	10	0
2	15	0
3	55	0
4	0	10
5	0	15
6	0	55
7	5	5

* For the combination-coated wires, the modified method was used for the first 5 layers, followed by 5 layers using the non-modified method. Each sample included one scrap circuit board with three niobium wires inserted through three separate gaps.

Key Results

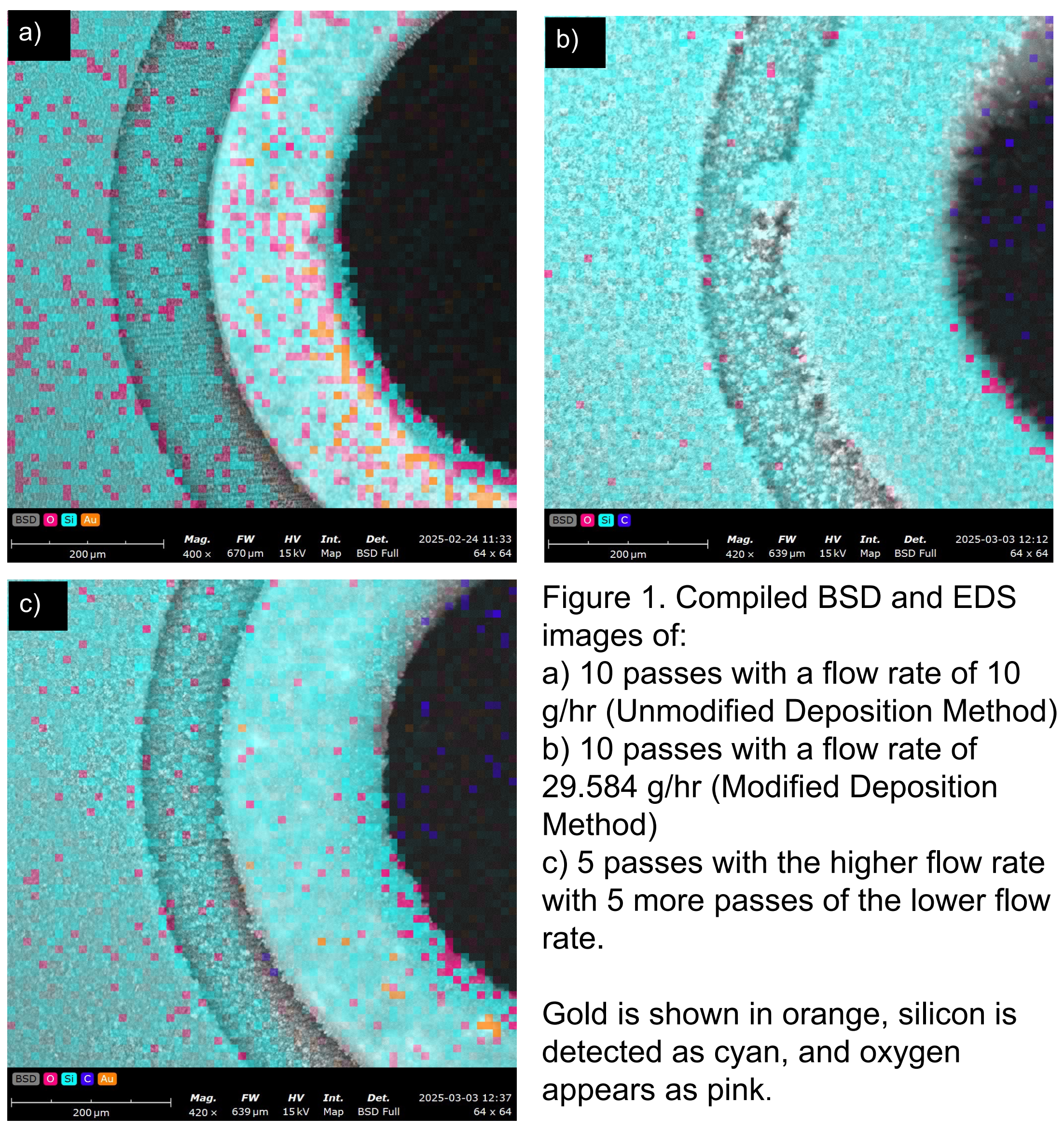

Figure 1. Compiled BSD and EDS images of:
a) 10 passes with a flow rate of 10 g/hr (Unmodified Deposition Method)
b) 10 passes with a flow rate of 29.584 g/hr (Modified Deposition Method)
c) 5 passes with the higher flow rate with 5 more passes of the lower flow rate.

Gold is shown in orange, silicon is detected as cyan, and oxygen appears as pink.

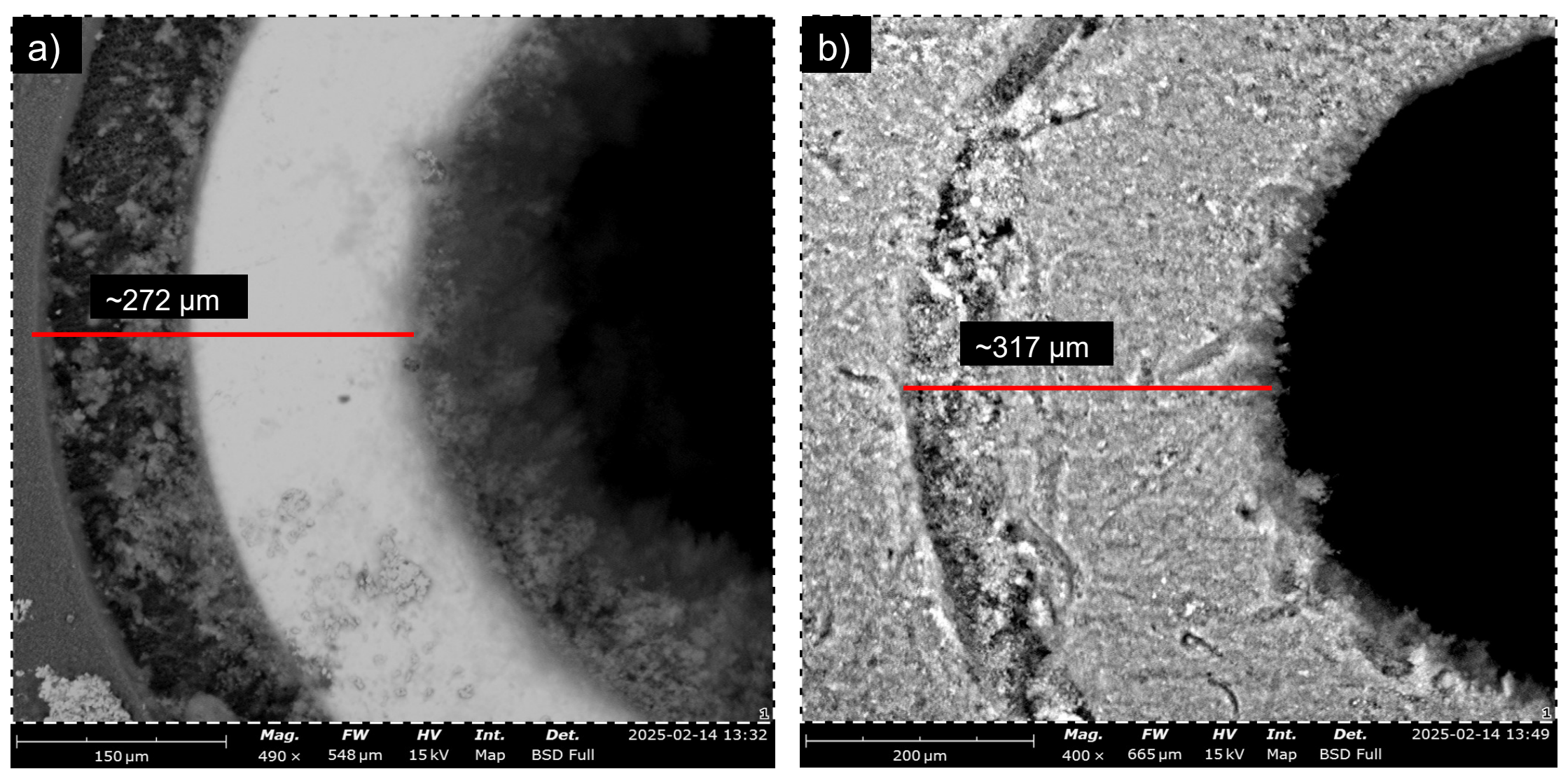

Figure 2. BSD images of the PCB a) Uncoated and b) Coated with the maximum flow rate for 55 passes. The thickness is measured as the change in thickness of an uncoated to coated feedthrough hole.

The EDS analysis of the PCBs resulted in only the conditions of Figure 1b fully coating the boards, which is shown with no gold from the circuit contacts being detected. The powder thickness is measured from the outer radius of the contact to the inner radius. A sample coated with the conditions of Figure 2b was on average 44.6 µm thicker than an uncoated wire with a coating rate of 0.81 µm/pass.

Breakdown Voltage of SiO_x Coated Niobium Wires (V)

No Coating	50	50	50

Non-Modified	1	2	3
10 passes	50	50	50
15 passes	400	80	200
55 passes	480	340	290

Modified	1	2	3
10 passes	50	50	50
15 passes	50	50	50
55 passes	460	240	520

Combination	1	2	3
5 non mod + 5 mod passes	550	400	410

The table above compares 22 niobium wires coated using two CAPD methods and their resulting breakdown voltages. The highest breakdown voltage, 550 V, was achieved using a combination coating process: 5 passes with the non-modified plasma method followed by 5 passes with the modified method. In contrast, coatings produced by 10 passes of either the modified or non-modified method alone showed little to no improvement in breakdown voltage compared to the uncoated wire. Notably, the modified plasma method did not yield a significant increase in performance until a much higher number of passes was applied (55 passes). This suggests that a synergistic effect may be present when combining methods, or that initial layers deposited by the non-modified plasma enhance adhesion or surface activation for subsequent modified passes. The observed variation in results may be attributed to nonuniform coating deposition, potentially due to fluctuations in plasma exposure, wire positioning during treatment, or inconsistencies in gas flow and power delivery.

Discussion

Unmodified Deposition Method:

With a small number of CAPD passes, the coating appeared thin and glassy compared to the coating on the modified samples. Compared to the modified samples, the average breakdown voltage was greater for the wires coated with 15 passes but lesser for the wires coated with 55 passes. As the number of CAPD passes increased, the coating became more powdery, less glassy, and easier to remove with minimal abrasion. This indicates that the SiO_x coating may have less of an affinity for itself than it does for the niobium wires, as the coating grew thicker with an increased number passes, the adhesive strength of the coating decreased.

Modified Deposition Method:

To try to seal the gap between the wires and circuit board, a modified deposition method was implemented to increase the flow rate of precursor, ideally increasing the thickness of the coating per pass. From Figure 1, it was determined that the modified deposition method did maximize the coverage of the coating, as no substrate was detected. However, even with less than ten passes, the SiO_x coating was powdery and not glassy like the coating deposited via less than ten passes of the unmodified deposition method.

Combination of Modified and Unmodified Deposition Methods:

The two deposition methods were combined to attempt to create thicker coatings with a glassy surface. The coating was deposited using the modified deposition method for thickness, and the unmodified deposition method was used to ideally add a glassy "capping" layer to the thicker coating. The results showed that despite the different flow between methods, the surface was still powdery and minimally adhesive, again, likely due to the precursor's greater affinity to polymerize onto the niobium wires than onto itself. Despite the failure to create a glassy surface, the wires coated using the combined method had the highest breakdown voltage of all the tested wires.

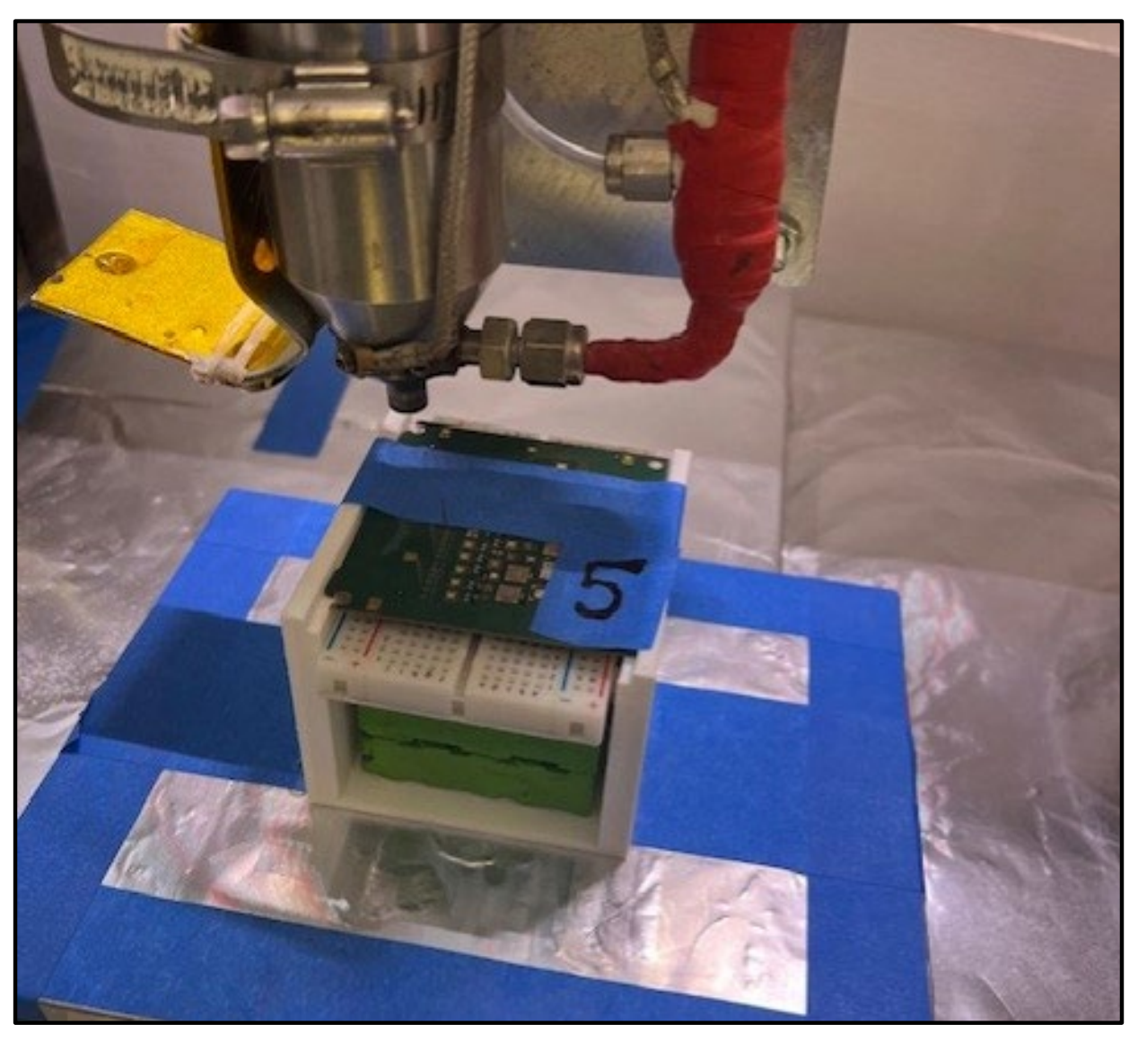

Figure 3. Image of the CAPD deposition nozzle during an unmodified coating test. The PCB is fixed on to a 3d-printed base for secure deposition. The niobium wires are threaded through the PCB and into a solderless breadboard.

Recommendations for Future Work

For these trials, HDMSN was used as the precursor for the CAPD. It would be interesting to try other precursors to see how they would affect the breakdown voltage of the wires. Additionally, combining different precursors by depositing multiple layers using two or more precursors may help determine if one precursor may form a glassier coating atop another precursor to create a stronger, thicker layer that may even be able to seal the gap between the wires and circuit boards. To experimentally determine how glassy or powdery a coating is, a mechanical testing method should be devised to apply force to the wires to determine how much force is required to remove them from the board after they have been coated. Mounting and polishing of cross-sectional samples in epoxy could also be useful to continue characterization. The breakdown voltage testing revealed inconsistent results caused by inconsistent coating of the niobium wires. A new method for wire coating should be devised to create a more consistent and even coating.

Acknowledgements & References

We would like to express our gratitude to our sponsor from Medtronic, Dr. Peter Tortorici, our Purdue faculty advisor, Dr. Rahim Rahimi, and Dr. Rahimi's graduate students, Advika Vidhyadhiraja and Rahman Mahabubur for their continuous help, support, and contributions to our project.

References:
1. Chen, Z., Chen, G., Obenchain, R., Zhang, R., Bai, F., Fang, T., Wang, H., Lu, Y., Wirz, R. E., & Gu, Z. (2022). Cold atmospheric plasma delivery for biomedical applications. *Materials Today*, *54*, 153–188. https://doi.org/10.1016/j.mattod.2022.03.001
2. Bárdos, L., & Baránková, H. (2010). Cold atmospheric plasma: Sources, processes, and applications. *Thin Solid Films*, *518*(23), 6705–6713. https://doi.org/10.1016/j.tsf.2010.07.044
3. Fenner, A. A., Larson, L. R., Greeninger, D. R., Thompson, D. L., & Inc, M. (n.d.). *US6052623A - Feedthrough assembly for implantable medical devices and methods for providing same - Google Patents*. https://patents.google.com/patent/US6052623A/en
4. *Electric breakdown*. IEEE Technology Navigator. (n.d.). https://technav.ieee.org/area/electric-breakdown

MSE Senior Design