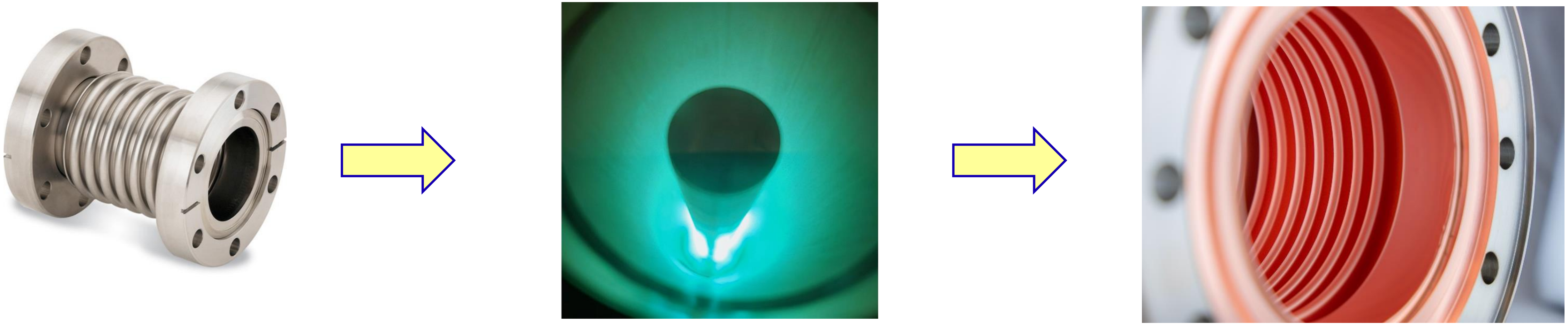

Defect Free, Conformal and Stable Coatings of Bellows and Waveguides for Accelerators Using Ultra-Fast HiPIMS Through Controlling Ion Energy

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starfire industries

DOE 2024 SBIR/STTR EXCHANGE PI MEETING
AWARD NO: DE-SC0020481

About Starfire Industries

Champaign, IL USA (near the University of Illinois)

- Vertical integration from R&D, manufacturing, applications testing and customer support
- “Deep Tech” nuclear, plasma and radiological team

Particle Accelerator Solutions:

- **nGen® portable neutron generators**
- **Centurion® ultra-compact MeV particle accelerators**

Plasma Processing Solutions:

- **IMPULSE® pulsed power modules for sputter/etch**
- **RADION™ microwave plasma sources for PECVD/etch**

Two Business Groups Within One Organization

Products on 6 Continents + Space!

Patent Portfolio Across Products

Facility Expansion

Starfire has recently relocated all operations to a 194,000 sq.ft. facility in Champaign, IL

- **Upgraded power infrastructure turning unused parking into solar for lower operating cost**

Design/Engineering

R&D Prototyping

Plenty of space for expansion

- Impulse Coatings & Materials Processing and Services
- Particle Accelerator Testing/Integration
- Manufacturing/Production
- Warehouse/Shipping

Starfire's Product Families

PARTICLE ACCELERATOR SOLUTIONS

nGen®

- Build/Sell Sealed Neutron Generators
- Design/Build Hardware For Custom OEM Solutions
- nGen® Systems Solutions For End-Users

Centurion®

- Build/Sell LINACS
- Custom OEM Sales To Development Partners
- Protons/Neutrons "As A Service"

PLASMA SOURCE SOLUTIONS

IMPULSE®

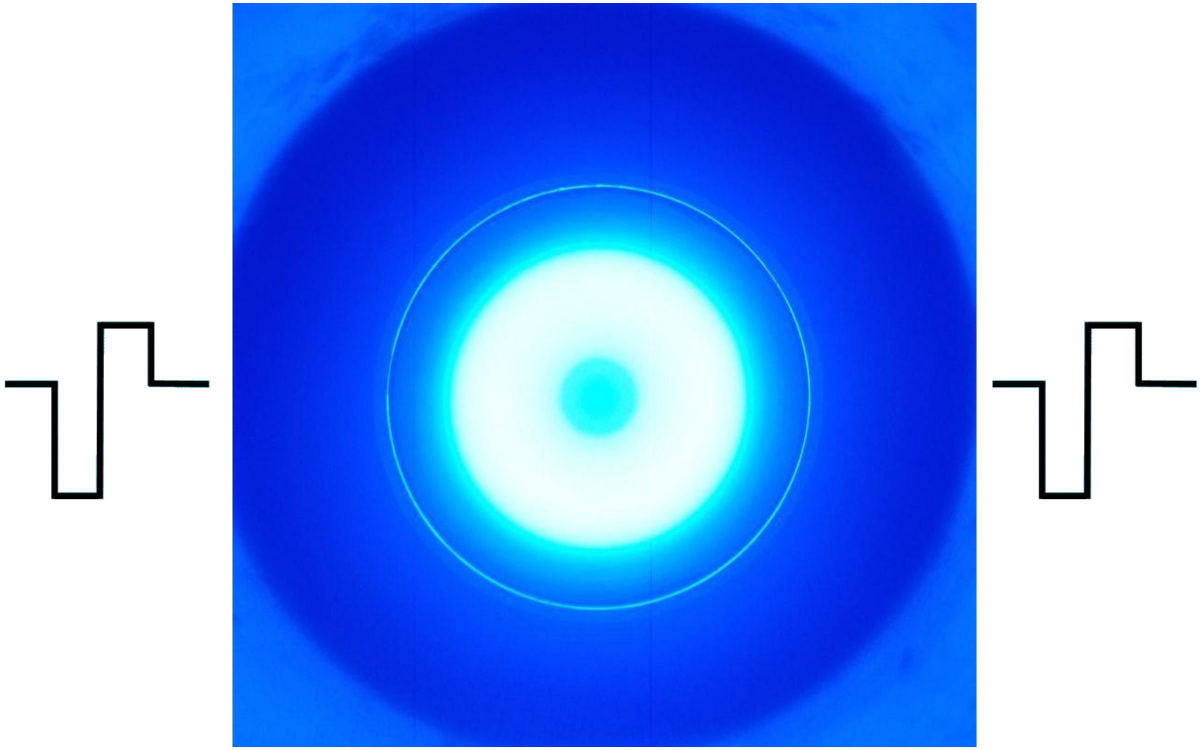
- Build/Sell Pulse Modules
- Design/Build Systems Using IMPULSE®
- Coatings "As A Service"

RADION™

- Build Microwave Plasma Sources
- Atmospheric Coating System Solutions
- Vacuum Etching/PECVD Systems Solutions

Hi Power Impulse Magnetron Sputtering (HiPIMS)

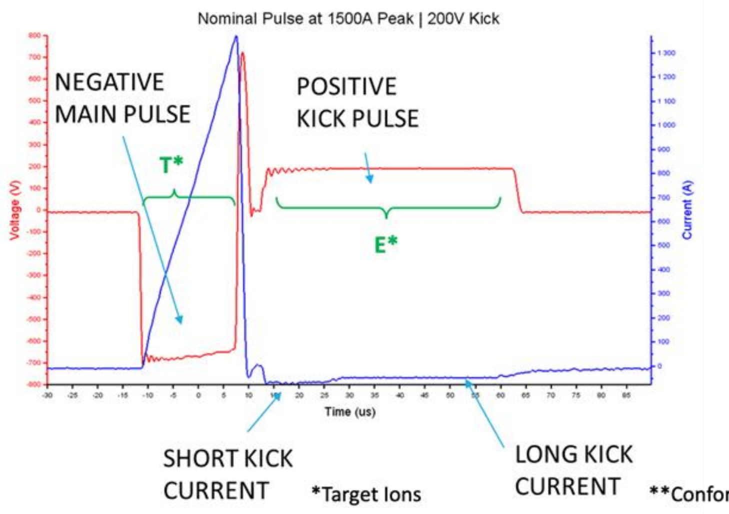
IMPULSE® + Positive Kick™

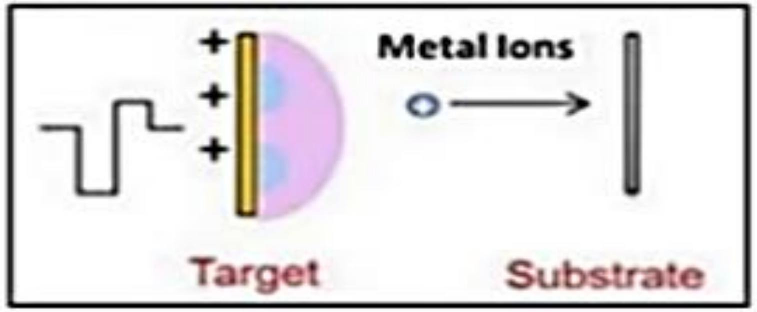


Next-Generation HiPIMS For PVD & Etch

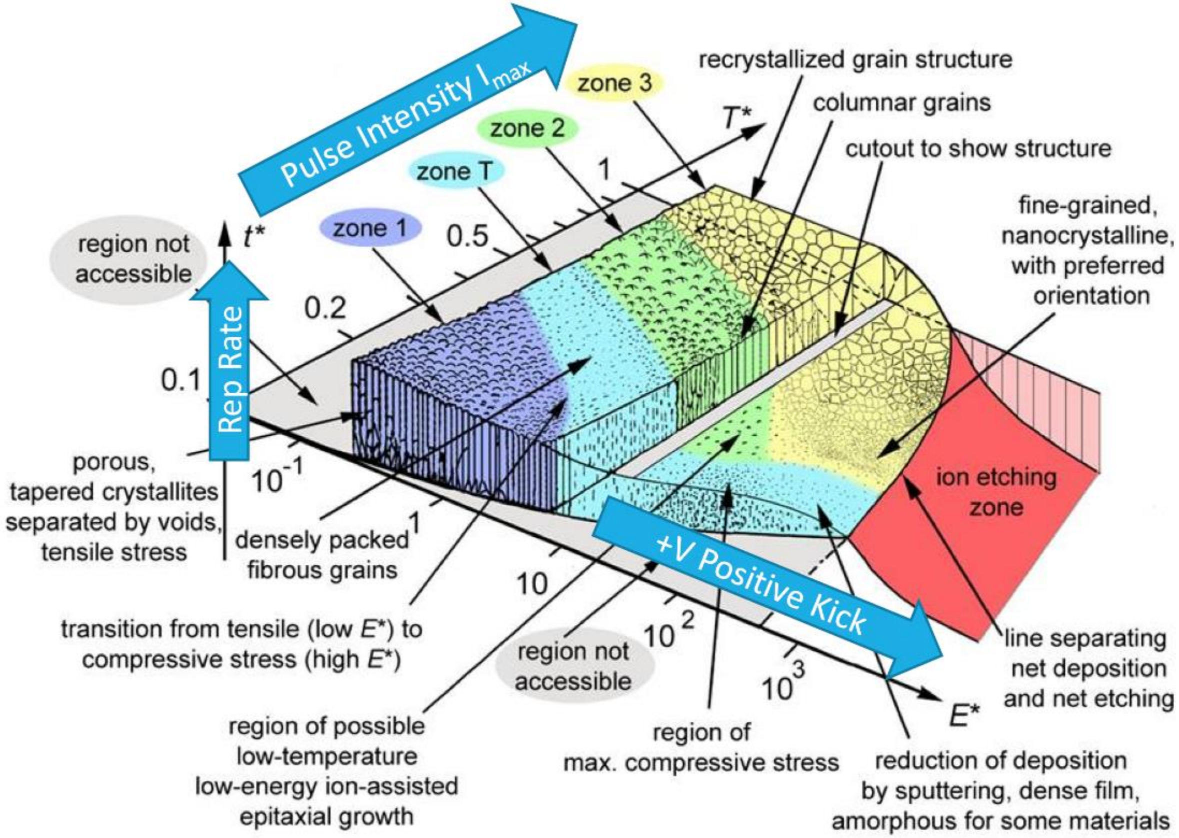
Controlling ion energy with IMPULSE® + Positive Kick™

IMPULSE® + Positive Kick™
Control T*, E*, t*, % ions



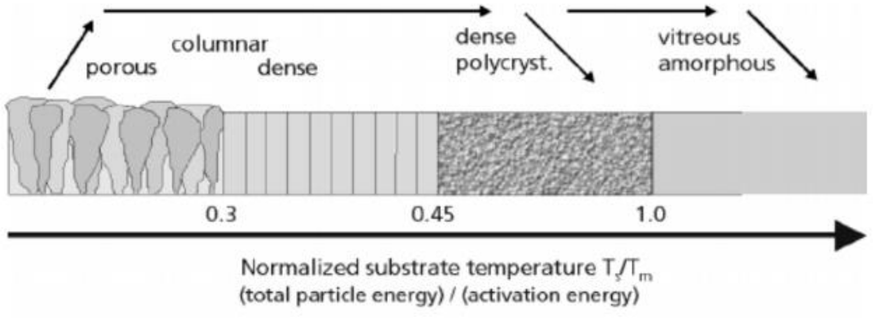


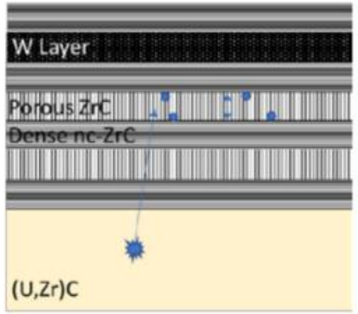
Nano Precision Surface Engineering
Access More Of The Structure Zone Diagram



Controlling Coating Properties in Wide Range

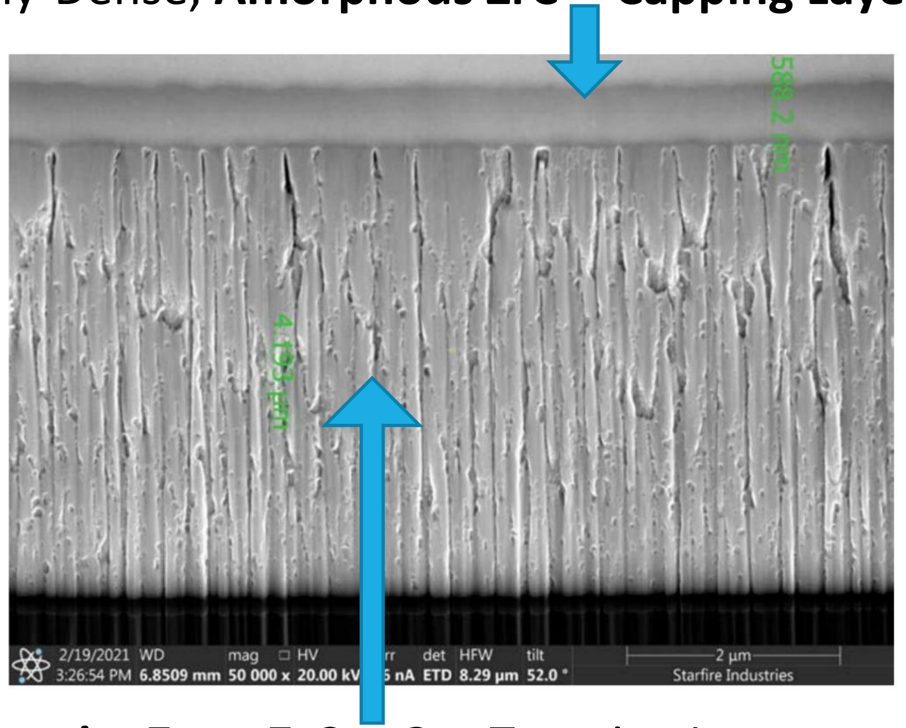
IMPULSE® + Positive Kick™
Adjust Layer Properties In Real-Time





Functionally Grade Coatings
CTE, Stress, Porosity, Phase, Texture
Even Same Material

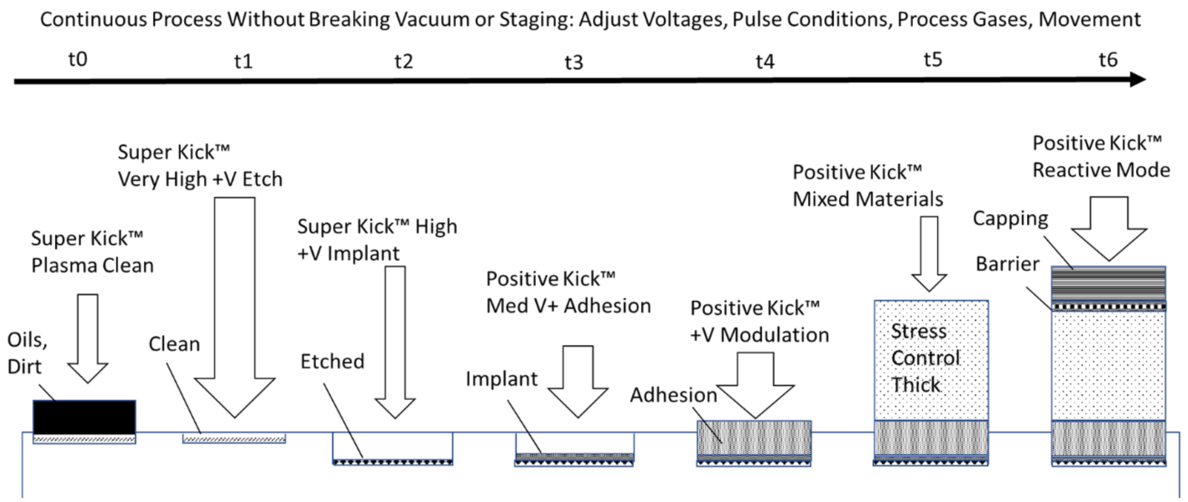
Fully-Dense, **Amorphous ZrC -- Capping Layer**

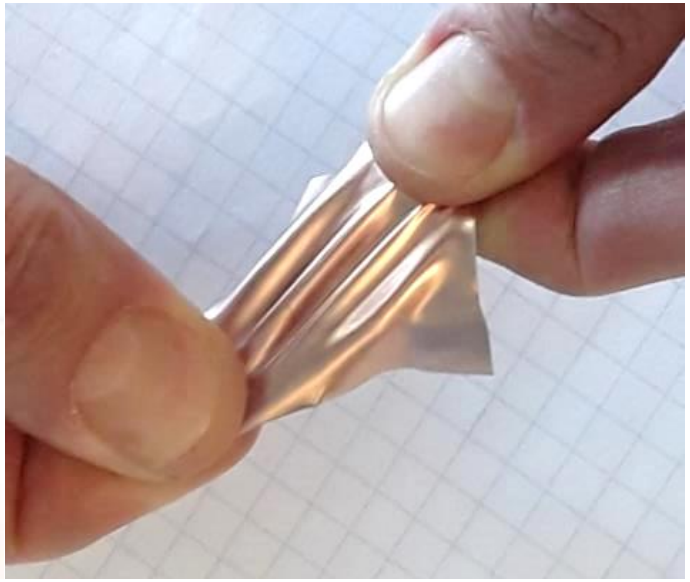


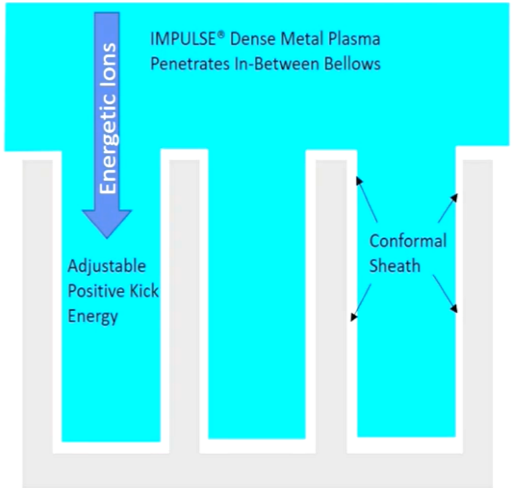
Porosity Zone ZrC -- Gas Trapping Layer

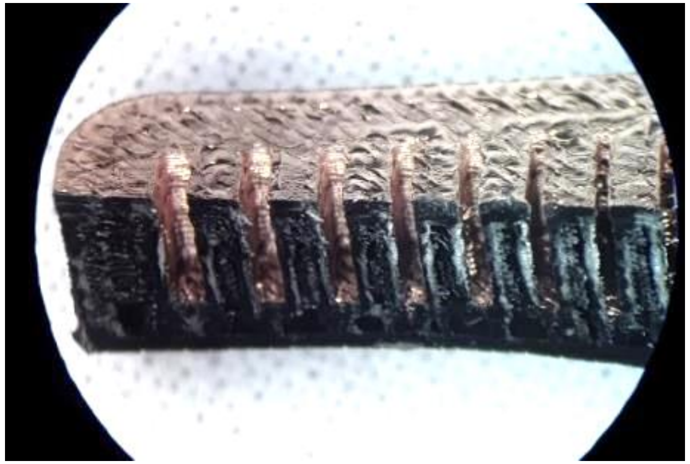
Positive Kick™ Dep/Etch + Dense Plasma

Conformality = 3D Coatings on Plastics

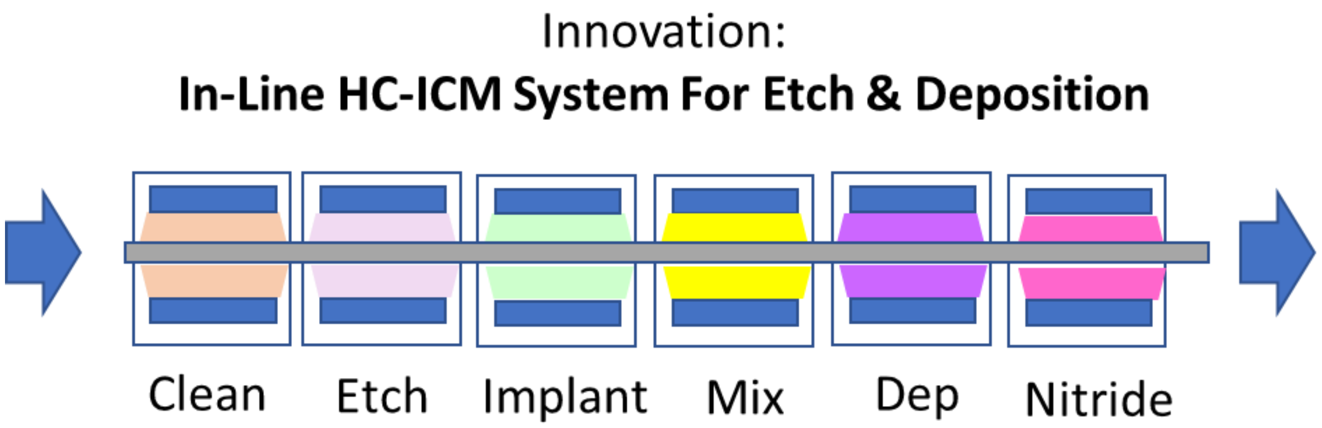


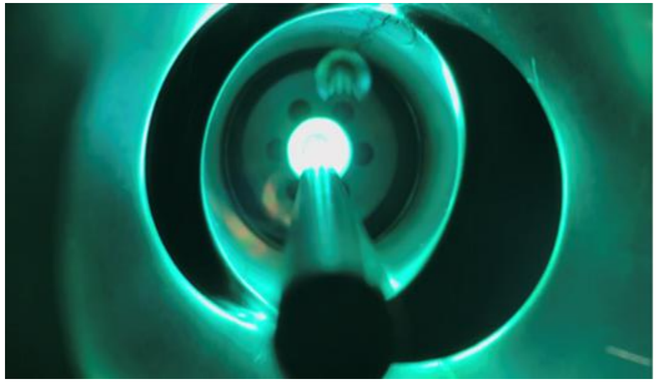


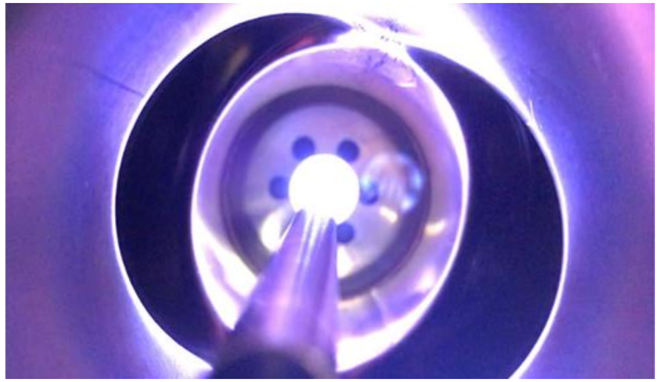




Hollow-Cathode Inverted Cylindrical Magnetron using IMPULSE® + Positive Kick™

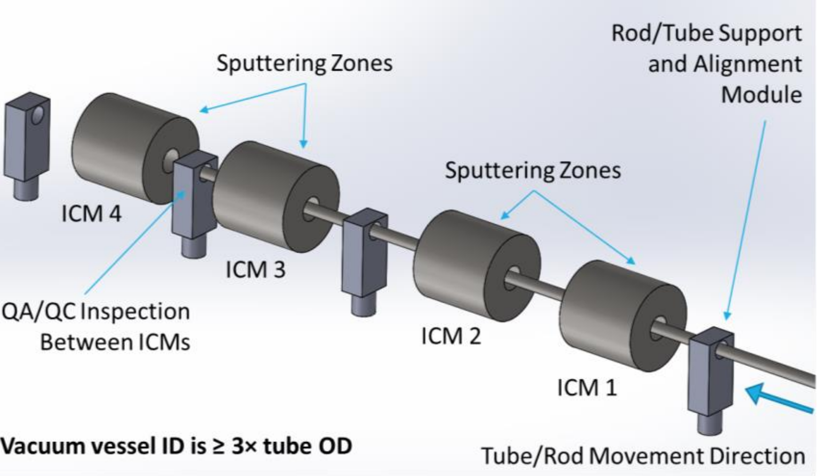






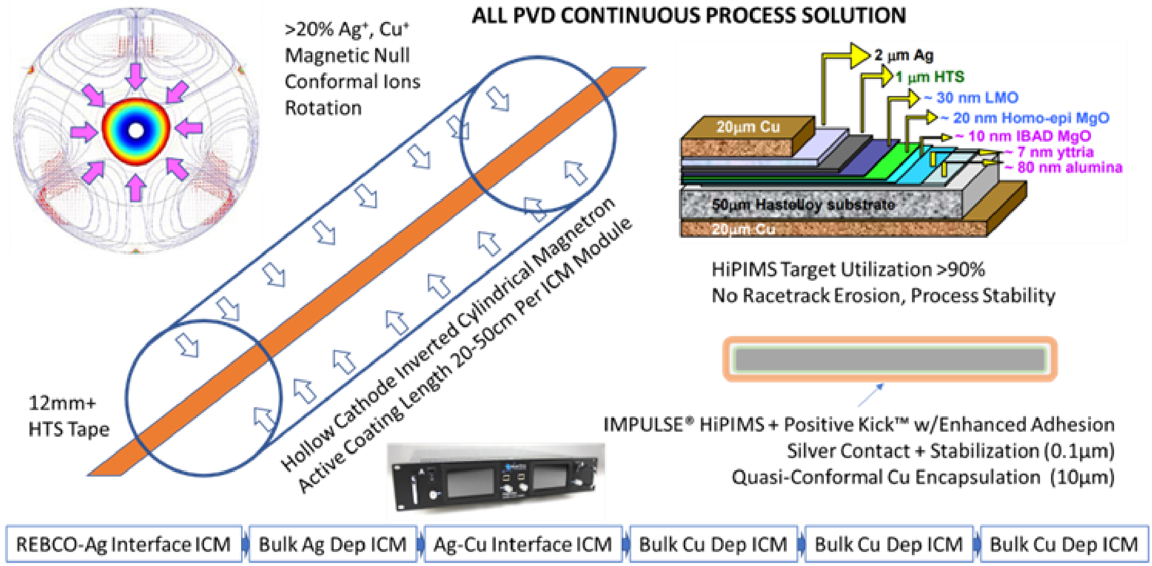
- Patented HiPIMS design for Positive Kick™
- Scalable in-line process for high mfg. throughput
- Large surface area → high deposition rate
- External magnet pack rotates for uniformity
- Geometry favorable for >90% target utilization
- Mitigates particles, flakes and debris!
- Etching, implantation, deposition, layering, stress control

25m-Long Pilot Line

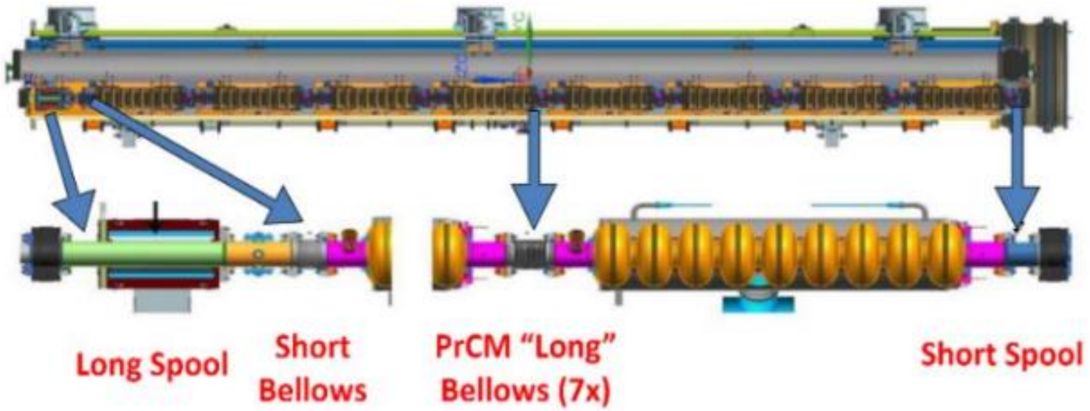


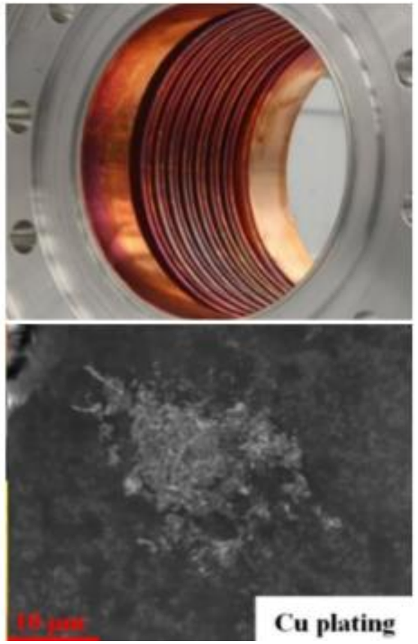
In-Line Coatings using Hollow-Cathode Inverted Cylindrical Magnetron + IMPULSE®

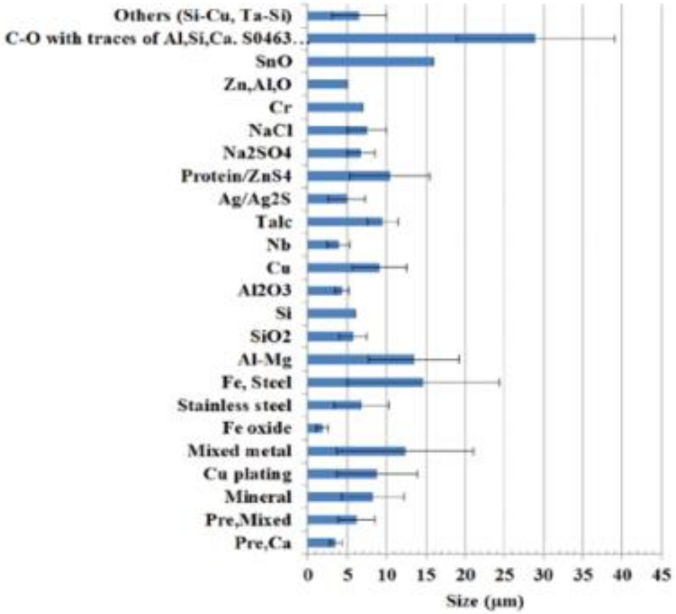
Coat km's HTS Tape



Cu Coating in Bellows and Waveguides







One cryomodule section in an accelerator
There may be 10s or 100s of these with SRF cavities, beam pipes and decoupling bellows

Bellows & beam pipes are electroplated with copper for high conductivity so when the "beam" passes there is minimal loss

Defects and inclusions from the Cu plating bath cause resistivity at cryogenic temperatures = Contaminants are BAD!

Corners are bad for electroplating and delaminate after thermal cycling causing major downtime for an accelerator

High-purity PVD solution with excellent adhesion is needed!

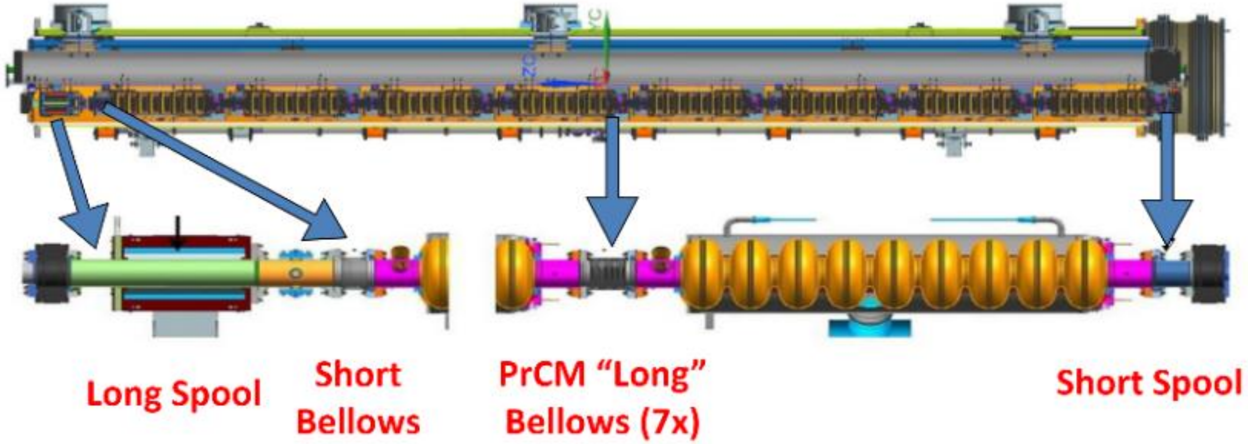
The Problem

Modern particle accelerators employ numerous specialized components requiring metal films

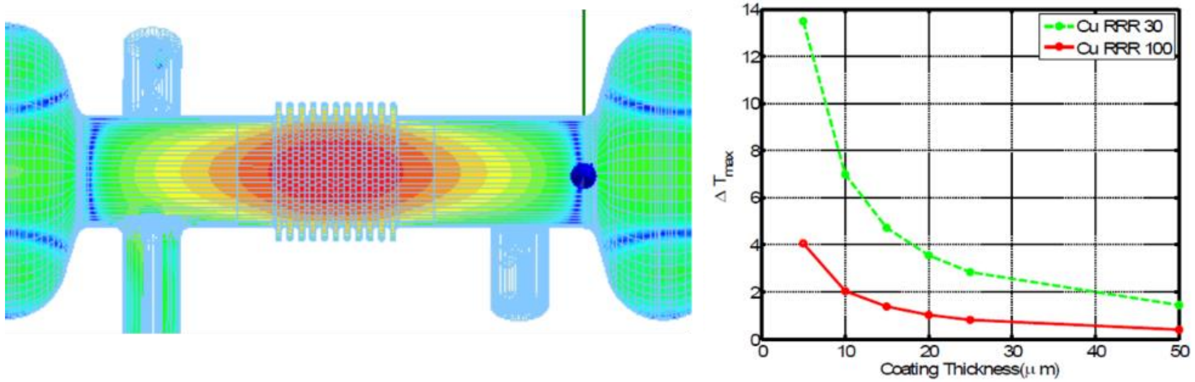
Bellows sections are a great example (and the focus of this work)

- Want high electrical conductivity on the inner surface for low beam-losses
- … & low thermal conductivity in the bulk for low thermal losses

So, a stainless-steel bellows w/ a copper film on the inner surface solves this problem



Some of the LCLS-II cryomodule bellows and spool sections that see RF energy and beam. Additionally, there are many other feedthroughs and bellows on the accelerator platform that are plated and material controlled. Figure from Ref [1].



(left) A simulated trapped RF mode that exists within the bellows that couples two cryomodules (Figure from Ref. [2]). (right) Calculated maximum rise in temperature as a function of layer thicknesses for Cu layers having RRR values of 30 and 100 (Figure from Ref. [3]).

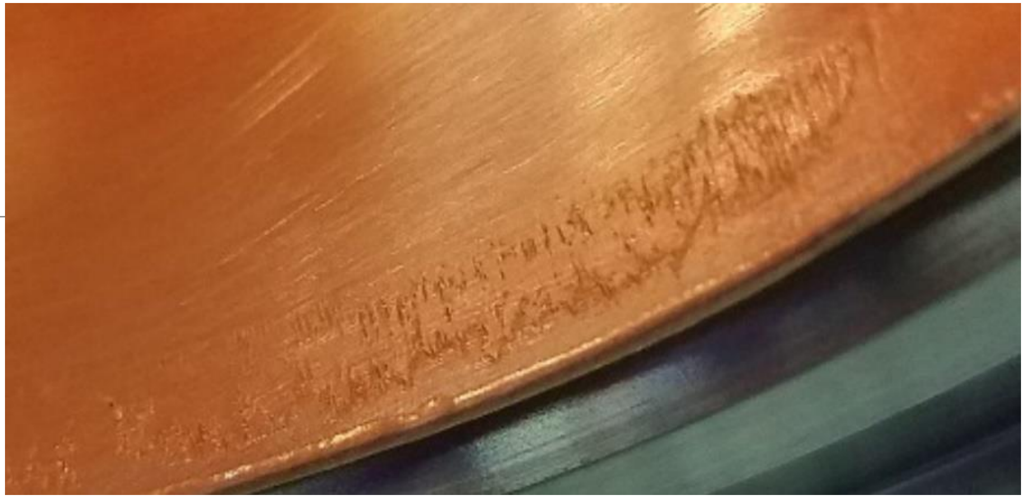
[1]: K. Wilson et al., "Production of Copper-Plated Beamline Bellows and Spools for LCLS-II," in 8th International Particle Accelerator Conference (IPAC 2017), Copenhagen, Denmark, 2017.
[2]: A. Saini et al., "RF Losses in 1.3 GHz Cryomodule of The LCLS-II Superconducting CW Linac," in 28th International Linear Accelerator Conference (LINAC16) , East Lansing, Michigan, 2017.
[3]: A. Saini et al., "LCLS-II TECHNICAL NOTES: Temperature Rise in LCLS-II Cavity Bellows," 29 June 2015. [Online; Accessed 01 October 2019].

What is done now?

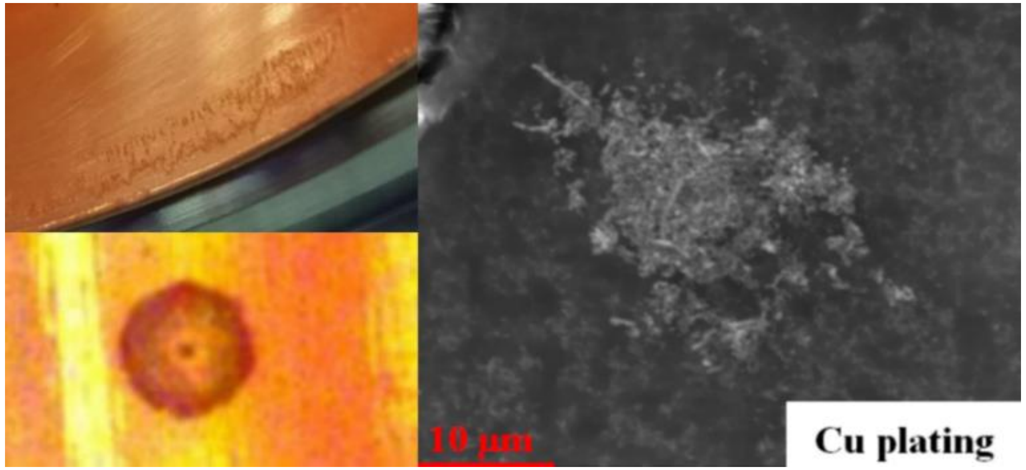
Copper films on stainless-steel bellows are presently deposited via a 'wet' electrochemical plating process

This has some inherent limitations/drawbacks:

- Defects/inclusions, flaking, etc. can lead to sparking or increased power deposition
- Hazardous waste streams are generated by these processes
  - These processes are being legislatively phased out in the EU where possible (e.g. where alternatives exist)
  - The US may be soon to follow
- Difficulty sourcing parts
  - Years of attenuation have left only a small handful companies in the US that perform such coatings
  - Infrequent orders result in plating shops having to re-learn some of the techniques and consideration that are lost with personnel turnover



A photograph showing striations in plating attributed to a leak in a seal during the plating process (Figure from Ref. [1]).



Images showing striations in plating (upper left, from Ref. [1]), a possible inclusion (lower left , from Ref. [1]), and a Cu plating particle (right, from Ref. [3]), plus distribution of particulate types by size Ref. [3].

[1]: K. Wilson et al., "Production of Copper-Plated Beamline Bellows and Spools for LCLS-II," in 8th International Particle Accelerator Conference (IPAC 2017), Copenhagen, Denmark, 2017.
[4]: L. Zhao et al., "Study on Cleaning of Copper Plated Bellows for LCLS-II," in 29th International Linear Accelerator Conference (LINAC18) , Beijing, China, 2018.

Clean Approach for Bellows Interior Coatings

Apply Impulse HiPIMS with positive kick and Radial Magnetron

A plasma generated on the OD of a Cu10100 cylinder sputters material radially outward

- HiPIMS w/ Positive Kick™ allows for both conformal Cu deposition AND *in-situ* plasma cleaning/etching

Designed to have a single, rotating serpentine racetrack

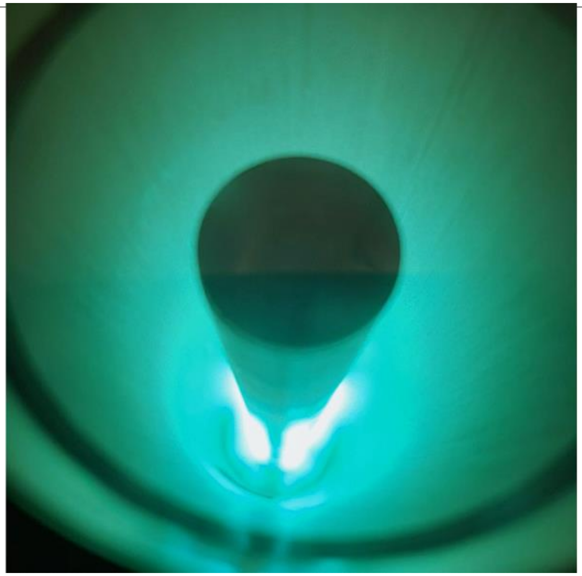
- A single racetrack ensures plasma uniformity
- Stationary cathode → No brushed electrical contacts
  - High pulse currents easily achievable
- A stationary cathode design also minimizes particle generation

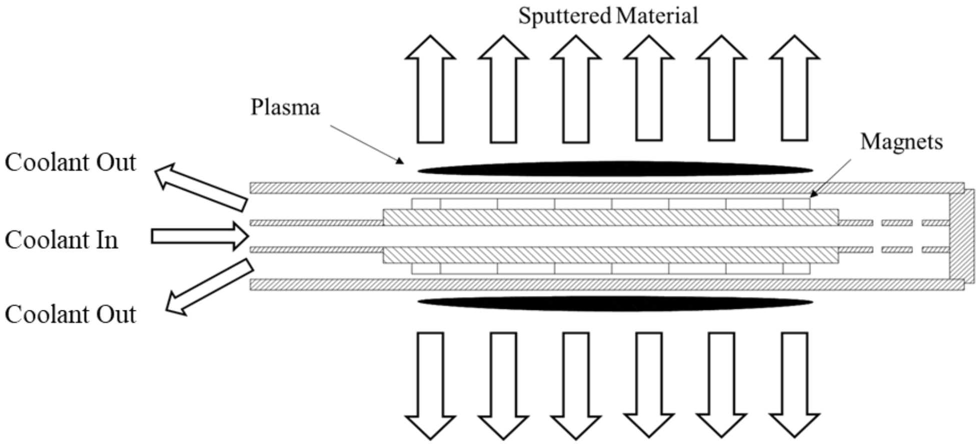
HV and cooling are at one end of the magnetron

- Facilitates easy loading/unloading of cylindrical parts at the other end

Plasma acts as our conformal anode

- Control of confomality by controlling pulse conditions
- Eliminates need for precision-machined conformal electrodes as used with electroplating

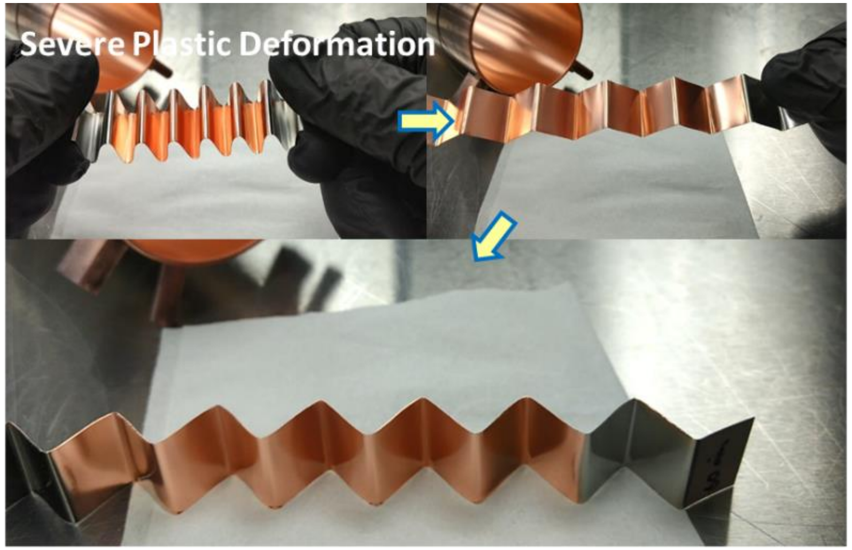




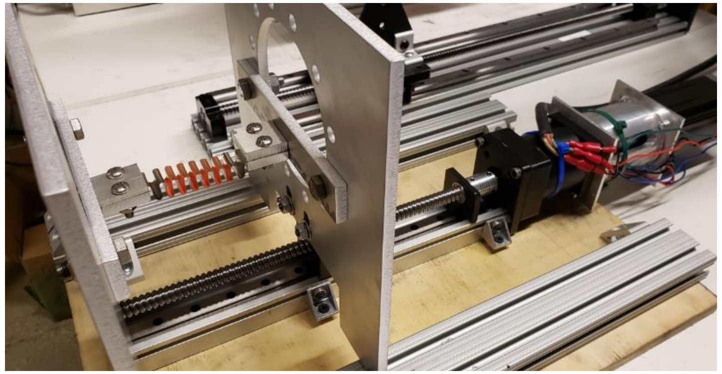
Experimental Results – Coupons and LCLS-II Bellows Sections

Adherent, thick (3—30 µm), conformal copper films were achieved on both test coupons and full bellows assemblies

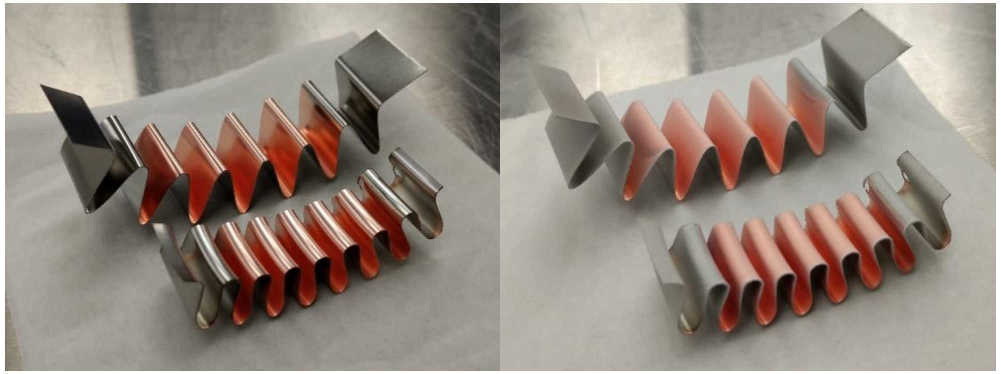
- Even after severe plastic deformation, the deposited films were not observed to buckle or delaminate
- Films did not delaminate after vacuum baking to 400 C and subsequent cooling to 77 K
- Fatigue testing had no observable effects
  - 2,000 cycles at ± 6 mm stroke, as per the LCLS-II bellows specifications
- A custom conductivity meter was used to record IACS conductivities
  - Values in the range of 70—90% IACS were typical



A test coupon with a 5 µm Cu film being plastically deformed. No buckling or delamination was observed in the Cu film.



The fatigue testing apparatus used for this work.



The test pieces after being subjected to a 400 C vacuum bake (left) and a 77 K LN2 bath (right).

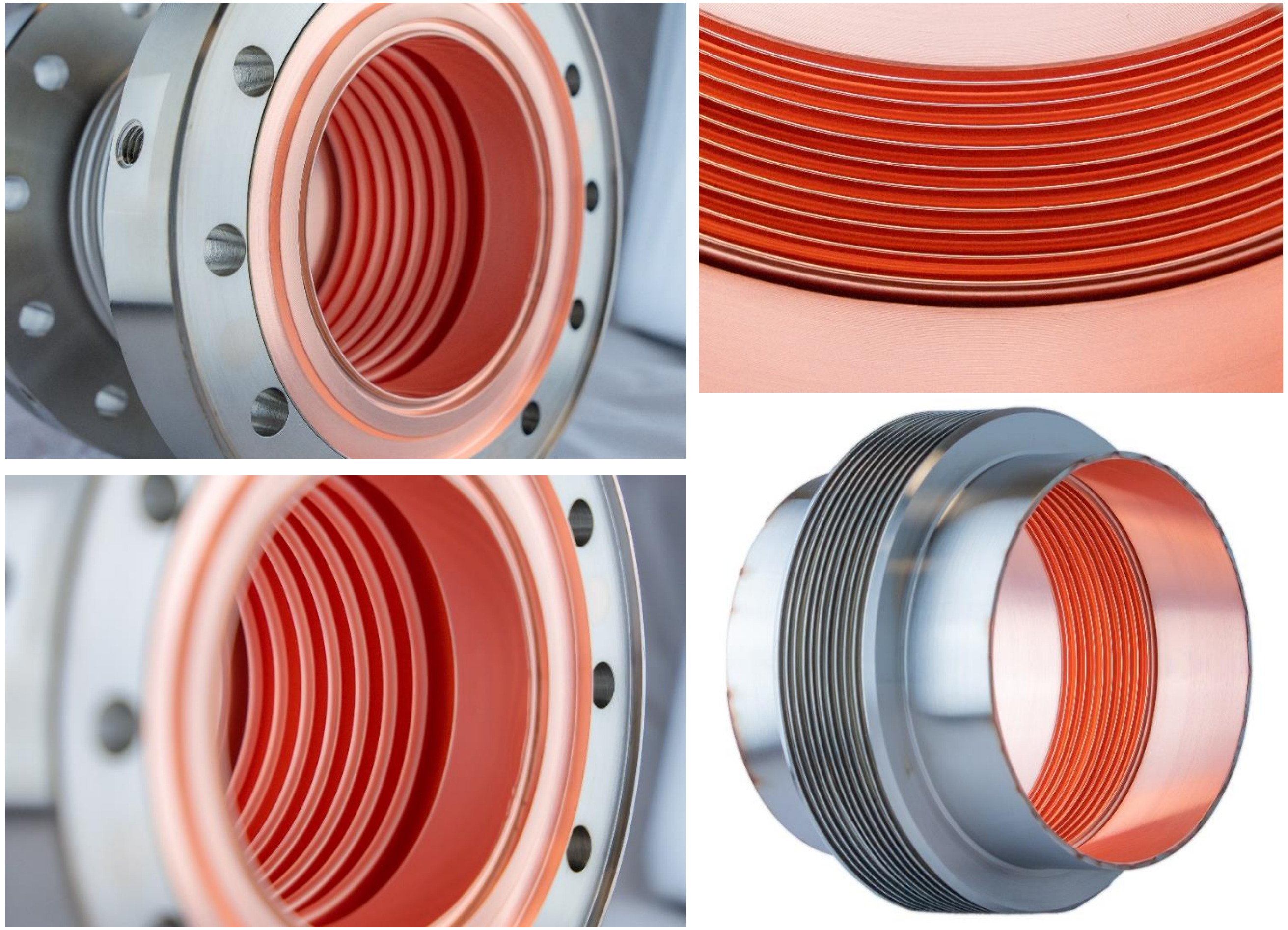
Cu-Coated Bellows

Stainless steel bellows were coated in Cu

- Optimum process conditions discussed on the previous slide were used for these depositions
- Both hydroformed and edge-welded bellows were coated
  - The edge-welded geometry makes this a more difficult deposition than for the hydroformed bellows

A rejected LCLS-II bellows (supplied by JLab) was coated and returned to JLab for analysis

- This sample was subjected to the same processing that an electroplated bellows would undergo



(Left) Photographs of a Cu-coated LCLS-II bellows section, which is presently being evaluated at JLab . (Right) Photographs of a Cu-coated edge-welded bellows.

Experimental Results – Coated Bellows Assemblies

Depositions on full bellows assemblies were also performed

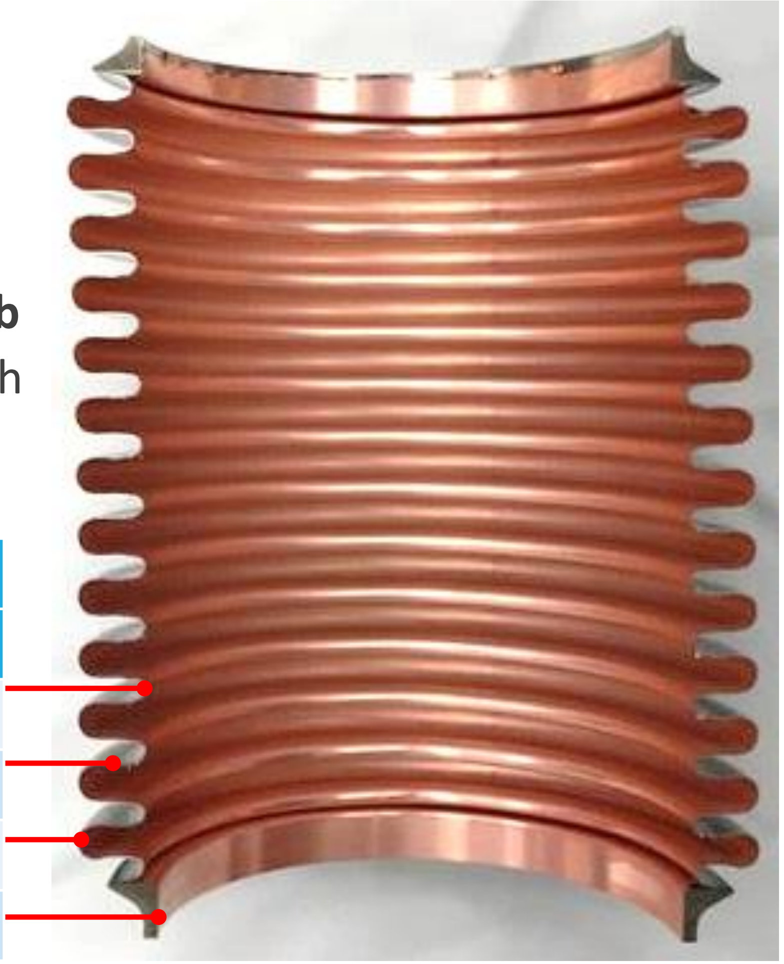
- The resulting films (~20—30 µm) were adherent, conductive, and conformal
- As with the coupons, fatigue testing (>20k cycles +/- 6mm) resulted in no observable changes

RRR values were measured at various places along the bellows at Jefferson Lab

- Bellows were sectioned by wire-EDM and sections of film were pulled off with tweezers for RRR measurement
- Residual Resistance Ratio, RRR = ρ_{300K} / ρ_{0K}

A Cu-coated bellows.

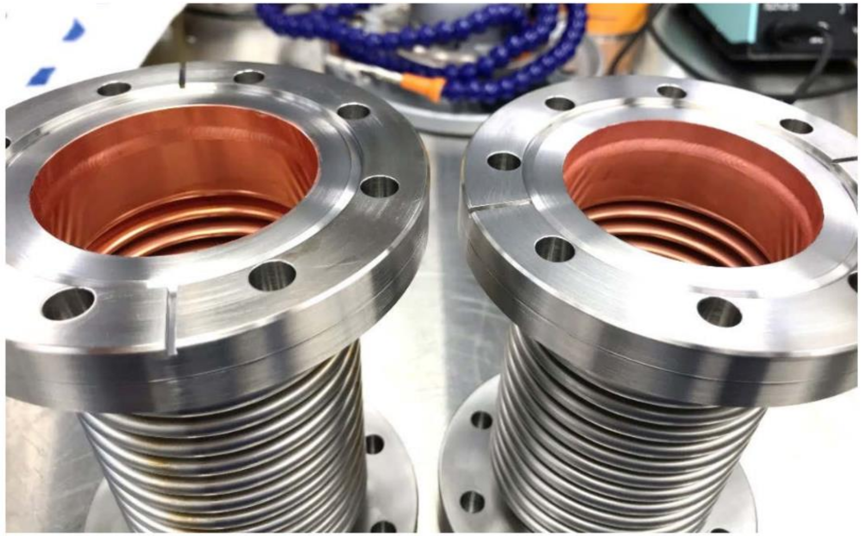
Location	RRR	
	4 mTorr	20 mTorr
Apex	14.33	22.93
Wall	9.53	23.42
Trough	6.07	6.74
Flat	10.35	24.86



A cross sectioned piece of a Cu-coated bellows (right). The apparent delamination at the top is where Cu was intentionally peeled off for RRR measurements. Measurements of the RRR for two processes (made at the indicated locations) are given here as well (left)

KJLC® Bellows Coating and Conductivity Measurements

Cu Coated KJLC® Bellows Coated w/Kapton Edge Masking



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%

A Smaller (0.5”-OD) Radial Magnetron

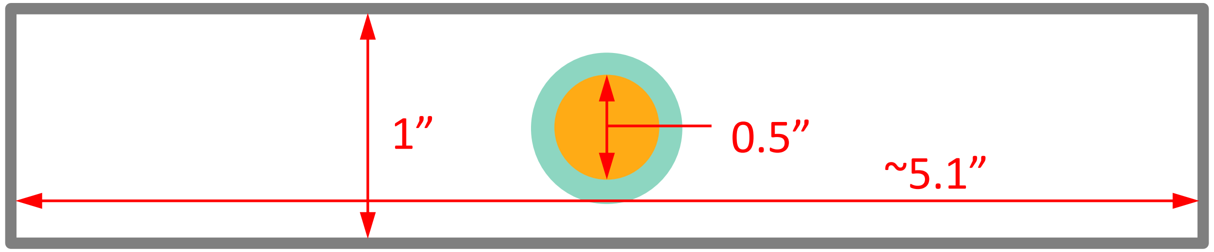
The CEBAF waveguide structure presents a much more difficult problem

- Low minimum internal clearance (1”)
- High aspect ratio (> 5:1)

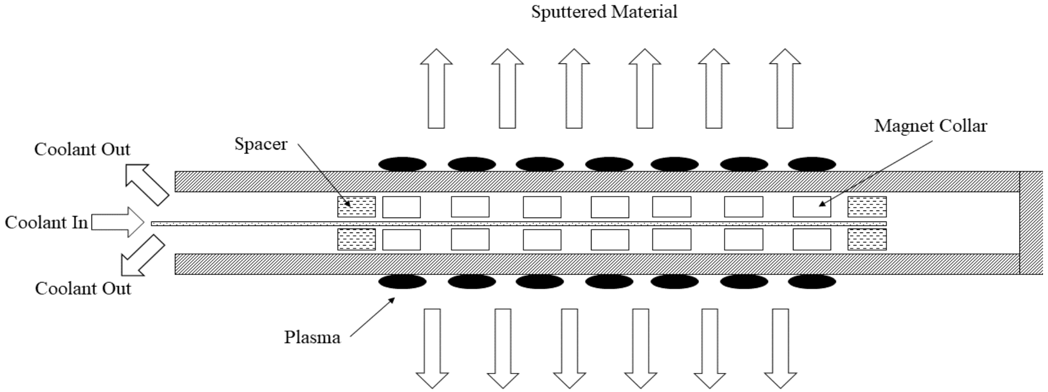
Required a new, smaller magnetron

- Same single-ended design as the 1”-OD version
- Plasma racetrack changed from a single serpentine racetrack to an array of annular (azimuthal) racetracks

We very quickly decided to make the jump to a dual-magnetron configuration to combat the challenging aspect ratio



The low internal clearance and high aspect ratio of the CEBAF waveguide structure present difficult technical challenges.



A cross-sectioned drawing of the 0.5”-OD radial magnetron design used for this work.

The prototype 0.5”-OD magnetron, which was made with an aluminum target.

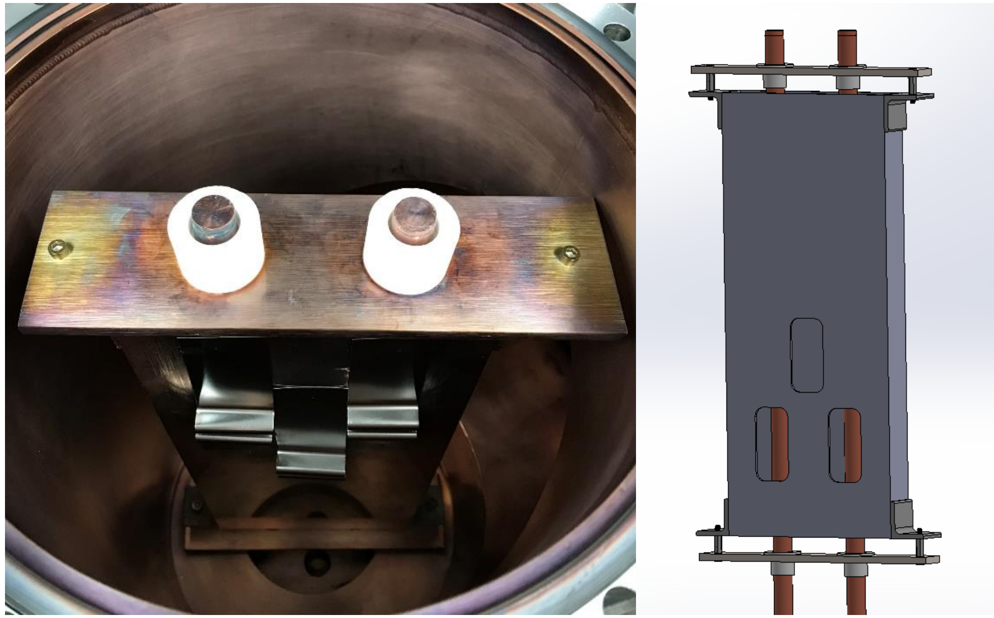
Test Reactor for Coating CEBAF Waveguides

A sample holder with dimensions representative of the CEBAF waveguide structure was constructed for this work

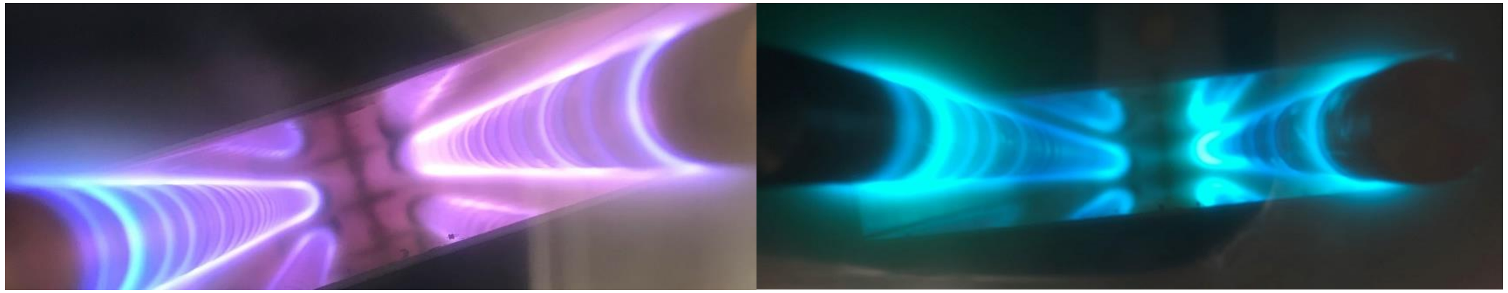
- Rectangular cutouts were included for mounting sample coupons
- Small holes were also included for mounting Si wafer pieces to serve as witness coupons for calculating local deposition rates

Fixturing was also made for holding/positioning the magnetrons themselves

- The small internal clearance requires more precision in magnetron placement than for the LCLS-II bellows



(Left) A photograph of the test-fixture used for these experiments. (Right) A CAD rendering of the same fixture.



Photographs of the dual-magnetron configuration in-operation during the plasma-etch step (left) and main deposition step (right), taken with the top of the test fixture removed.

A photograph of a coated sample coupon and Si wafer piece.

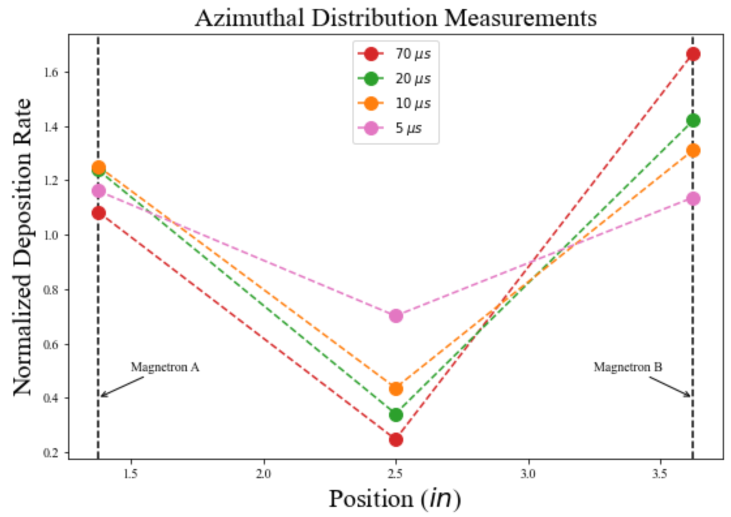
High-Aspect-Ratio Coating Challenges

Film thickness measurements at right were taken over each magnetron and half-way between them

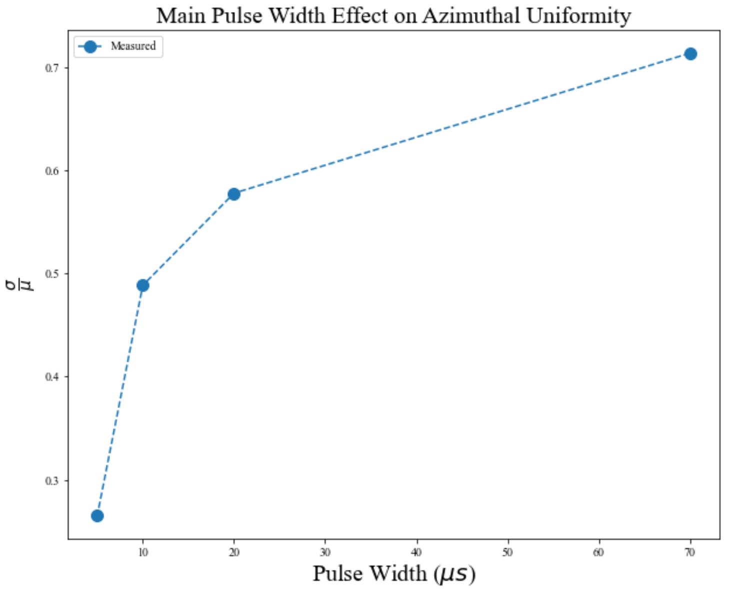
- Used as a quick evaluation of uniformity: lower peak/mid-point ratio is what we're looking for

In general, shorter main pulses are extremely beneficial for increasing azimuthal uniformity

  - If the main pulse is too long, too much mass is deposited in the form of Cu neutrals
    - Neutrals leave the target with an azimuthally uniform distribution
- Long kick pulses (100 µs or more) are beneficial
- Moderate kick voltages (~100 V) are good
  - Too low: insufficient ionization of the in-flight Cu neutrals occurs
  - Too high: results in a large sheath region that doesn't conformally cover the smaller features in the substrate surface



Normalized deposition rates at each magnetron and at the midpoint between the two for a variety of main-pulse widths.

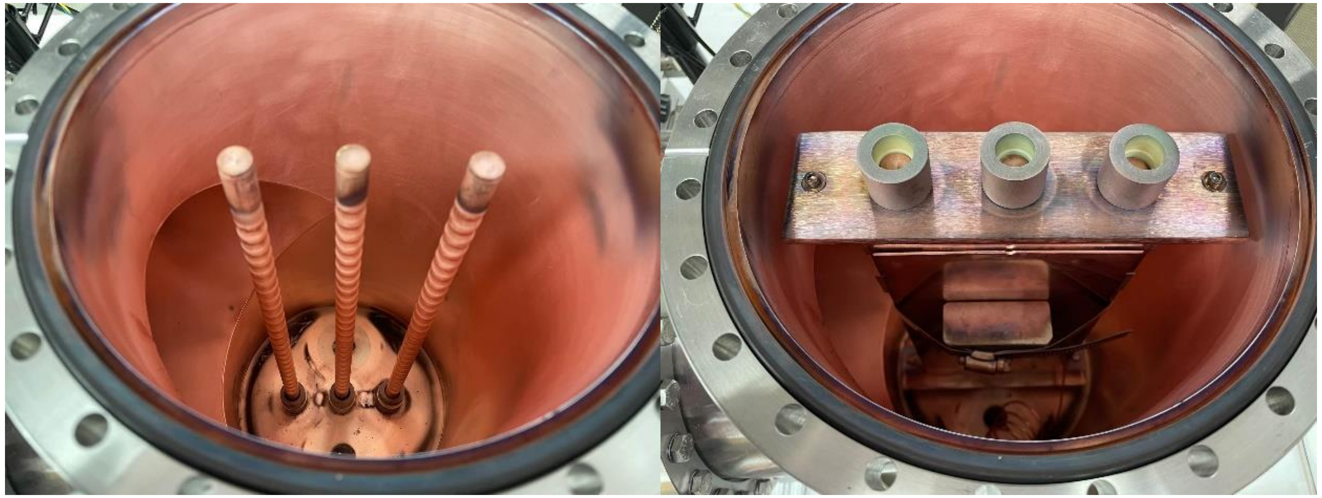


Uniformity as a function of main-pulse width.

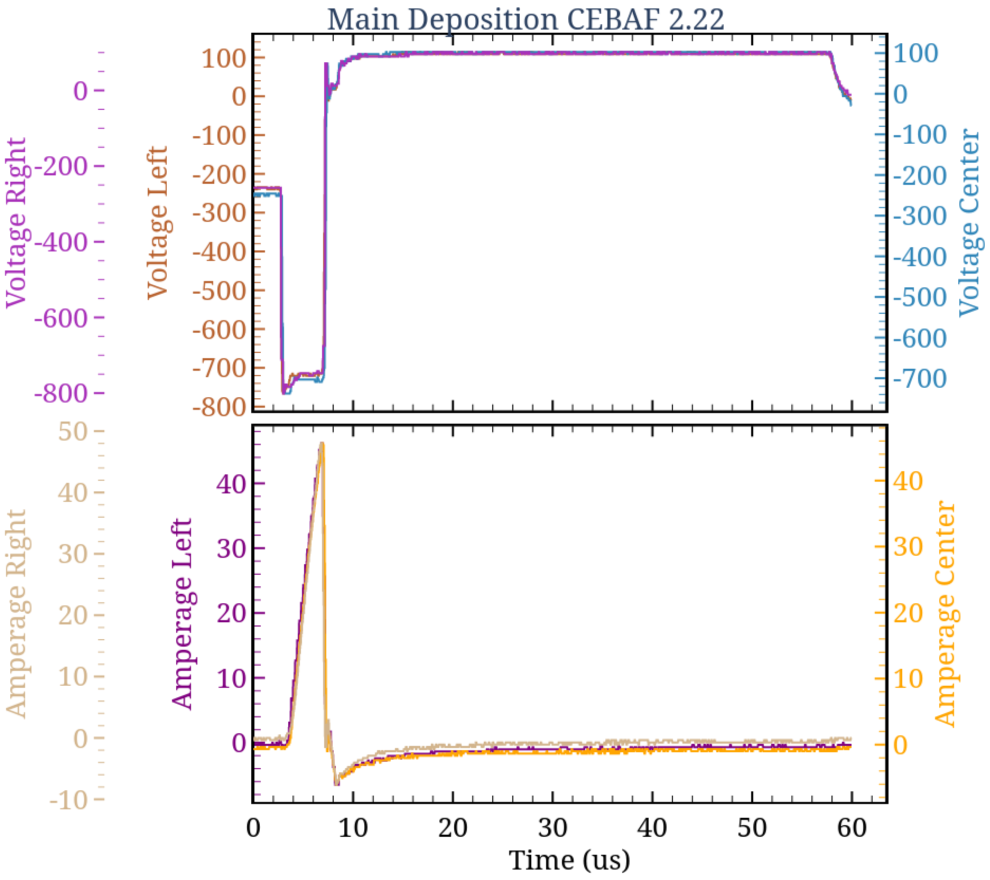
Three-Magnetron Deposition Setup

Cu is deposited onto a stainless steel strip mounted to a cutout in the sample holder

- Again, the sample holder approximates the inner dimensions of a CEBAF waveguide
  - The cutout is a strip along the entire long edge of the holder
- Pulsing of the three magnetrons is staggered to prevent current from commuting between them
  - Voltages are adjusted slightly to maintain equal peak currents



(Left) The three radial magnetrons shown with the sample holder unloaded from the chamber. (Right) The deposition chamber loaded with the sample holder and the three radial magnetrons.



A set of voltage (top) and current (bottom) waveform for each of the three magnetrons. Each of the waveforms here are plotted relative to the t_0 trigger time for the IMPULSE unit that produced it. So, while they are shown here plotted on top of one another, in reality they are evenly spaced out in time.

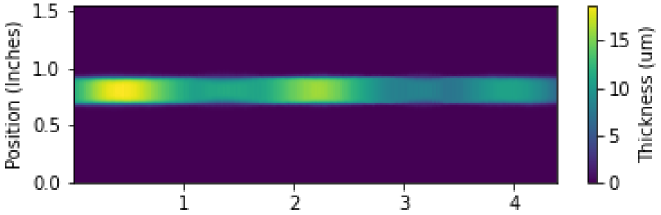
Present Effort: Cu coating inside C75 and C100 Waveguides

Recipe Development has been recent focus

- Uniform coating in high aspect-ratio waveguide
- Well adhered films
- Repeatability

Plan to deposit on actual hardware when possible

- Need slightly larger deposition chamber to accommodate larger size.



Best Repeatable Recipe

3 x 7 x 14”

5.5 x 8 x 9"

Recipe	Pressure (sccm)	MW (us)	Kick Voltage	Peak Cur (A)	PtTR
1.1	30 (22 mhPa)	4	100	50	2.06
1.2	80 (220 mhPa)	4	100	160	2.69
1.3	*58 (40 mhPa)*	*5*	*65(Low)*	*220*	*1.52*
1.4	70 (74 mhPa)	2	150	105	3.21
1.5	50 (34 mhPa)	4	100 (Low)	150	2.68
1.6	80 (220 mhPa)	4	100	200	???
1.7	15 (9mHpa)	12	90 (LOW)	170	1.91
1.8*	50 (34 mhPa)	2	150	70	1.50
2.2*	34 mhPa	2	150	70	1.74
2.3	42 mhPa	2	150	70	1.64
2.1	50 mhPA	2	150	70	1.46
2.4	60 mhPa	2	150	70	1.65
2.5	34 mhPa	5	150	70	1.53
2.6	50 mhPa	5	150	70	1.91
2.7	50 mhPa	5	30	70	1.74
2.9 E	50 mhPA	2	150	70	1.38*
2.9 E	50 mhPA	2	150	70	1.5
2.10 E	50 mhPA	2	150	70	1.38*

Conclusions, Next Steps, & Acknowledgements

LCLS 2 bellows coated with conductive, adherent, and conformal Cu film.

- The radial magnetron works and can be inserted from one end or fixed between two end blocks and pulled through pipes.
- These films, having thicknesses of ~5—30 µm, do not delaminate, often even after severe plastic deformation of the substrate
- Conformality appears strongly correlated to main-pulse width and kick-pulse voltage

Coated Proxy CEBAF rectangular waveguide

- Multiple magnetrons and an investigation into further shortening the main pulse helped to minimize the overall variation in film thickness
- Allows coating down to 1-inch ID
- Next steps are vacuum chamber upgrade and deposition on actual CEBAF waveguide

The developed technique can be utilized for various applications using other materials:

- We can get much better uniformity and adjust thickness with plasma vs traditional line of sight.
- High conductivity coatings for beampipes (e.g. in BNL EIC beamline components)
- Nb for SRF applications, porous gettering materials (Ti, Zr, Mo), etc.
- SiC coating on high aspect ratio substrates and structures

Starfire is submitting a Phase 2b

- Seeking lab partners that will be commercial customers to join
- Potential to be a strategic supplier or spin off a company that can do this.
- Impulse tech can touch many markets

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