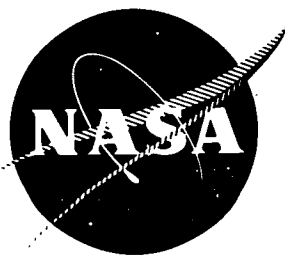




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DEVELOPMENT OF SPUTTERED TECHNIQUES FOR THRUST CHAMBERS

FINAL REPORT

MARCH 1979

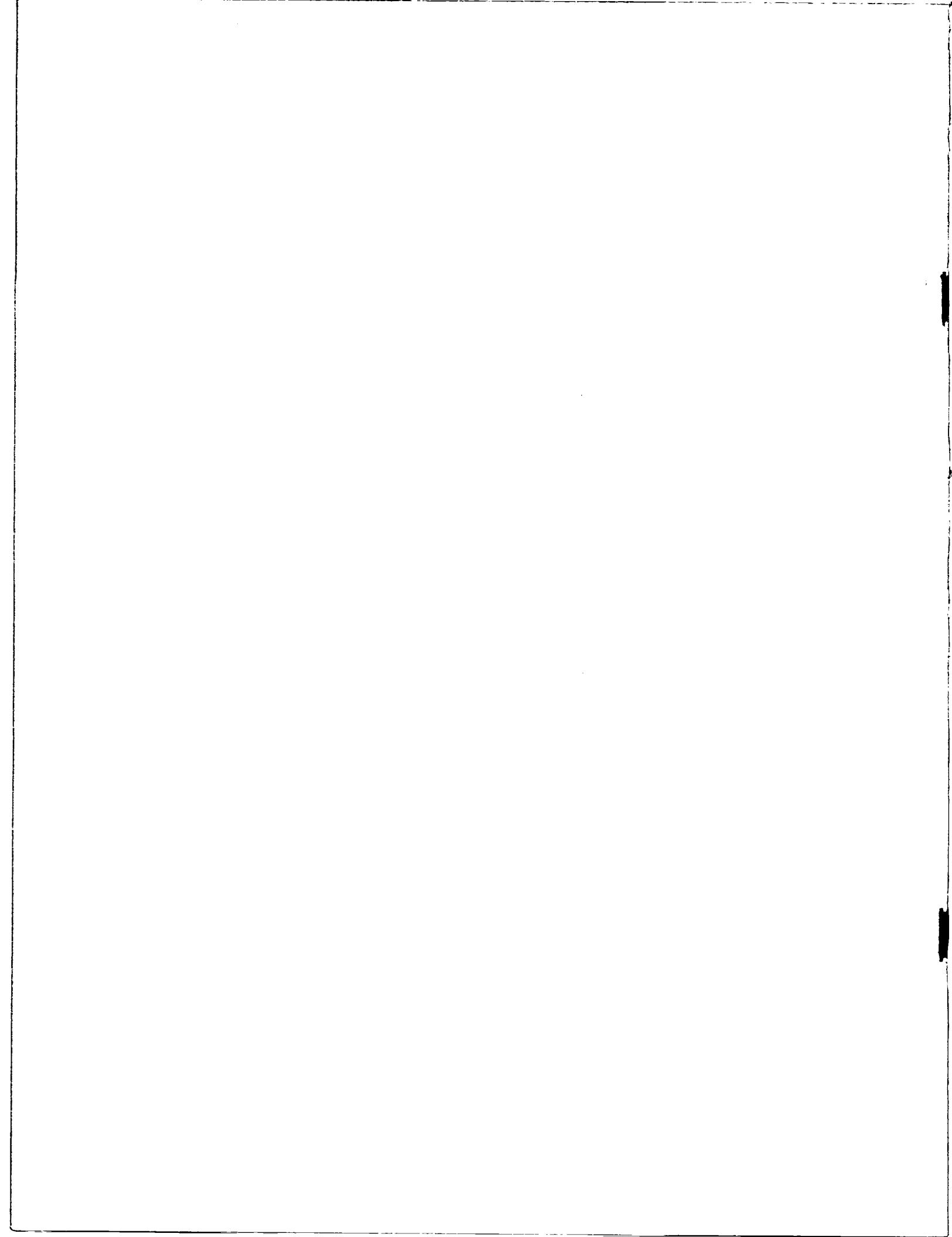
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21000 Brookpark Road
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Subject: Submittal of the approved Final Report, FR-11409

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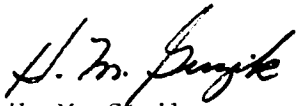
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16. Abstract The 0.152 cm thick sputtered OFHC and AMZIRC Copper deposits were electron beam welded to wrought copper. Tensile specimens were machined from the weld assemblies and tested at room temperature. Tensile strength approached the strength of wrought material. Elongations up to 25% were measured. Sputtered aluminum was used to fill 0.157 cm wide by 0.127 cm deep grooves in thrust chamber spool piece liners. The liners were closed out by sputtering AMZIRC copper from post and hollow cathodes.					
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CONTENTS

<i>Section</i>		<i>Page</i>
I	SUMMARY.....	1
II	INTRODUCTION.....	2
III	TASK I — SPUTTERING PARAMETER AND FABRICATION PROCESS- ING EFFECTS ON SPOOL PIECE CONSTRUCTION.....	3
	A. Equipment and Procedures.....	3
	B. Deposition Parameters.....	9
	C. Results.....	9
	D. Conclusions.....	39
IV	TASK II — SPOOL PIECE LINER FABRICATION.....	40
	A. Equipment and Procedures.....	40
	B. Sputtered Aluminum and Sputtered Copper Closeout Layer Deposition Parameters.....	45
	C. Results.....	45
	D. Conclusions.....	84
	REFERENCES.....	87

ILLUSTRATIONS

<i>Figure</i>		<i>Page</i>
1	Shaped Post Cathode.....	4
2	Orientation of Weld Pieces on Post and Hollow Cathode Substrates.....	7
3	Machining Details for Weld Sections.....	7
4	Machining Details for Weld Matching Pieces.....	8
5	Machining Details for Weld Assemblies. Gage Surface $\sigma_1 \sqrt{\quad}$	10
6	Machining Details for Cutting Hollow Cathode Tensile Specimens from Weld Assemblies.....	11
7	Hollow Cathode Burst Specimen.....	12
8	Post Cathode Burst Specimen.....	13
9	Variation of OFHC Copper Deposit Thickness for Hollow Cathode Substrates in a 15.24 cm ID Cathode (System No. 3).....	16
10	Variation of AMZIRC Copper Deposit Thickness for Hollow Cathode Substrates in a 10.16 cm ID Cathode (System No. 2). The 10.16 cm Was the Starting ID. All Three Substrates Were Coated With the Same Cathode.....	17
11	Variation of Copper Deposit Thickness for Hollow Cathode Substrates in 10.16 cm ID Cathodes (System No. 2). I-32 and I-33 Were Coated in the Same Cathode as I-29, I-30, and I-31. I-34 Was Coated in a New 10.16 cm ID Hollow Cathode.....	18
12	Variation of AMZIRC Copper Deposit Thickness for Post Cathode Substrates (System No. 2).....	20
13	Variation of OFHC Copper Deposit Thickness for Post Cathode Substrates (System No. 2).....	20
14	Typical Microstructure of Sputtered OFHC Copper Deposits from System No. 3 (Substrate I-26, Top of Section No. 1).....	21
15	Typical Microstructure of Sputtered AMZIRC Copper Deposits from System No. 2 (Substrate I-29, Bottom of Section No. 3).....	22
16	Typical Microstructure of Post Cathode Sputtered Deposits on Substrates I-8P, I-10P and Ends of I-13P (I-8P, Top of Section No. 1).....	22
17	Variation of Microstructure for OFHC Copper Sputtered from Post Cathodes. The Recrystallization May Depend on Parameters Other Than Substrate Bias.....	24

ILLUSTRATIONS (Continued)

<i>Figure</i>		<i>Page</i>
18	Example of Substrate Influence on Recrystallization of Sputtered OFHC Copper (I-12P, Top of Section No. 1).....	25
19	Layered Microstructure of Sputtered AMZIRC Copper (I-15P, Bottom of Section No. 3).....	25
20	Typical Appearance of Electron Beam Welds in the O.K. Condition. The Material Shown in the Left Photo Has Been Over Etched To Show Weld Detail. The Sputtered AMZIRC Copper Is Not Porous nor Is the Weld Defective.....	28
21	Side View of Tensile Specimen Fractured Ends After Room Temperature Testing. Hollow Cathode Sputtered Deposit Welded to Wrought OFHC Copper.....	29
22	Side View of Tensile Specimen Fractured Ends After Room Temperature Testing. Hollow Cathode Sputtered Deposit Welded to Wrought AMZIRC Copper.....	30
23	Side View of Tensile Specimen Fractured Ends After Room Temperature Testing Post Cathode Sputtered Deposits.....	31
24	Side View of Tensile Specimen Fractured Ends After Room Temperature Testing Wrought Copper TIG = Tungsten Inert Gas Weld EB = Electron Beam Weld	32
25	Examples of "O.K." Welds After Tensile Testing. Post Cathode.....	33
26	Examples of Welds After Tensile Testing Hollow Cathode.....	34
27	End View of Specimen I-28-1-1 After Tensile Testing. This Is an Example of a Porous Weld. The As-Tested Ends (10X) Were Polished Back and Over-Etched To Show Pore Formation During Welding. The Pores Formed Roughly in Equal Numbers in the Sputtered and Wrought Material.....	35
28	Examples of Tensile Specimens That Ruptured in the Sputtered Material Due to Open Columnar Growth. The Fractured Ends Were Photographed and Then the Weld Areas Were Polished for the Photomicrographs.	36
29	Hardness Test Pattern on a Piece of Weld Section from I-15P-1 Piece Heated 1 hr in Argon at 316°C. VPN. 0.2 kg Load.....	38
30	Machining Details for Test Grooved Hollow Cathode Substrates (I-18, I-19 and I-20).....	42
31	Slotted GF Cylindrical Specimen for Hollow Cathode Closeout Redrawn from NASA CC622357.....	43

ILLUSTRATIONS (Continued)

<i>Figure</i>		<i>Page</i>
32	Slotted GF Cylindrical Specimen for Post Cathode Closeout Redrawn from NASA CC622358.....	44
33	Variation of Aluminum Deposit Thickness for Test Grooved Hollow Cathode Substrates.....	53
34	Variation of Aluminum Deposit Thickness for Test Grooved Hollow Cathode Substrates.....	54
35	Growth Characteristics of Aluminum Sputtered from a Hollow Cathode into Nominal 0.152 cm Wide by 0.127 cm Deep (0.062 by 0.050 in.) Grooves as a Function of Substrate Bias Voltage.....	55
36	Growth Characteristics of Aluminum Sputtered from a Hollow Cathode into Flat and Round Bottomed Grooves -125 Volt Bias.....	56
37	Growth Characteristics of Aluminum Sputtered from a Hollow Cathode into Flat and Round Bottom Vertical Grooves With -125 Volt Bias. The Grooves on Substrate I-18 Were Nearly Filled on the First Deposition Cycle.....	57
38	Growth Characteristics of Hollow Cathode Sputtered Aluminum at the End of Flat and Round Bottom Vertical Grooves. Substrate I-18.....	58
39	Schematic of the Topography of Sputtered Aluminum in a Groove. This Schematic Defines the Angle θ and the Factors Used in Calculating Groove Filling Efficiency for Table 21.....	59
40	Hollow Cathode Burst Test Specimen Filled With Sputtered Aluminum. As-Machined and Hand Sanded With 600 Grit SiC Paper.....	63
41	Growth Characteristics of Aluminum Sputtered from a Hollow Cathode Onto Half-Round and Round Wires. The As-Sputtered Surface Shows the Smoothing Effect of the -125v Substrate Bias Voltage. Substrate I-20	64
42	Details of Growth Characteristics of Sputtered Aluminum Around Half-Round and Round Wires. Substrate I-20.....	65
43	Variation of Aluminum Deposit Thickness for the First Four Deposition Cycles on GF Hollow Cathode Substrate I-25. The Aluminum Was Machined Back to the Substrate OD After Each Deposition Cycle...	67
44	Variation of Aluminum Deposit Thickness for GF Hollow Cathode Substrate I-27. The Aluminum Was Machined Back to the Substrate OD After Each Deposition Cycle.....	68
45	Variation of Aluminum Deposit Thickness for GF Hollow Cathode Substrate I-35. The Aluminum Was Machined Back to the Substrate OD After Each Deposition Cycle.....	69

ILLUSTRATIONS (Continued)

<i>Figure</i>		<i>Page</i>
46	Variation of Aluminum Deposit Thickness for GF Hollow Cathode Substrate I-36. The Aluminum Was Machined Back to the Substrate OD After Each Deposition Cycle.....	69
47	Variation of Aluminum Deposit Thickness for GF Post Cathode Substrate II-4P Aluminum Machined Back to Substrate OD After Each Deposition Cycle.....	70
48	Sputtered Aluminum on GF Substrate I-35 After Deposition Cycle 2.....	71
49	Characteristics of Sputtered Aluminum Over Grooves in GF Hollow Cathode Substrate I-35 After Deposition Cycle No. 3.....	72
50	Appearance of Grooves After Machining Back the Sputtered Aluminum on GF Substrate I-35. Machining Has Been Stopped Just Short of the Copper Surface.....	73
51	Variation of Copper Closeout Thickness for GF Hollow Cathode Substrate I-25 Closed Out in Several Sizes of Cathodes.....	76
52	Variation of Copper Closeout Thickness for GF Hollow Cathode Substrate I-27 Closed Out in 10.80 cm ID Hollow AMZIRC Cathodes.....	76
53	Variation of Copper Closeout Thickness for GF Hollow Cathode Substrate I-36 Closed Out in a 12.7 cm ID Hollow AMZIRC Cathode.....	77
54	Variation of Copper Closeout Thickness for GF Post Cathode Substrates Using a Shaped Post Cathode. Deposit Not Machined Back Between Layers for II-4P.....	78
55	Cathode Erosion and Substrate Deposit Profiles Measured After Closing Out Substrate I-36. Approximately 318 cm ³ of AMZIRC Were Sputtered from the Cathode and 194 cm ³ Collected on the Substrate for a 61% Collection Efficiency.....	79
56	Erosion from Three Sizes of Hollow Cathodes.....	80
57	Change in AMZIRC Shaped Post Cathode vs Deposition Time for Closeout Layer Deposition on Substrate II-5P.....	81
58	Machining Detail for Closed Out GF Substrates. Redrawn from NASA CF621930.....	83
59	GF Substrates After Machining and Leaching Aluminum Filler.....	85

TABLES

<i>Table</i>		<i>Page</i>
1	Summary of Task I Hollow Cathode Substrate Sputter Cleaning Parameters	5
2	Summary of Task I Post Cathode Substrate Sputter Cleaning Parameters (System Number 2).....	6
3	Summary of Task I Hollow Cathode Deposition Parameters.....	14
4	Summary of Task I Post Cathode Deposition Parameters (System Number 2)	15
5	Deposition Rate, Composition, and Vickers Hardness for Hollow Cathode As- Sputtered Copper. 10.0 kg Load.....	15
6	Deposition Rate, Composition, and Vickers Hardness for Post Cathode As- Sputtered Copper. 10.0 kg Load.....	15
7	Deposit Thickness of Hollow Cathode Substrates in System No. 2.....	18
8	Deposit Thickness of Post Cathode Substrates.....	21
9	Room Temperature Tensile Properties of Hollow Cathode Weld Assemblies (Sputtered Copper EB Welded to Wrought Copper).....	26
10	Room Temperature Tensile Properties of Post Cathode Weld Assemblies (Sputtered Copper EB Welded to Wrought Copper).....	27
11	Room Temperature Tensile Properties of Hollow and Post Cathode Weld Assemblies (Wrought Copper EB and TIG Welded to Wrought OFHC Copper).....	37
12	Hardness (VPN) in and Near the Weld Zone for Material from Weld Assembly I-15P-1 as Welded and After Heating for 1 Hour in Argon at the Indicated Temperature. 0.2 kg Load.....	38
13	Sputtering Systems and Cathodes Used in Task II Substrate Filling and Closeout Layer Deposition.....	41
14	Summary of Sputter Cleaning Parameters for Test Grooved Substrates Prior to First Aluminum Depositions.....	46
15	Summary of Sputter Cleaning Parameters for GF Substrates Prior to First Aluminum Depositions.....	46
16	Summary of Sputter Cleaning Parameters for Filled GF Substrates Prior to Start of Copper Closeout Layer Deposition.....	47
17	Summary of Deposition Parameters for Filling Test Grooved and Wire Wrapped Cylindrical Substrates by Sputtering from Aluminum Hollow Cathodes.....	48

TABLES (Continued)

<i>Table</i>		<i>Page</i>
18	Summary of Deposition Parameters for Filling GF Fully Grooved Post and Hollow Cathode Substrates With Sputtered Aluminum.....	49
19	Summary of Task II Post and Hollow Cathode Closeout Layer Deposition Parameters.....	50
20	Deposition Rate and Substrate Appearance for Aluminum Sputtered Onto Test Grooved Substrates.....	52
21	Summary of Measurements Taken from Cross Sections of 0.152 cm Wide by 0.127 cm Deep Grooves Filled With Sputtered Aluminum. (See Figure 39 for Explanation of Terms).....	60
22	Deposition Rate and Substrate Appearance for Aluminum Sputtered Onto GF Substrates.....	66
23	Aluminum Filled GF Substrate Diameters After Final Machining.....	74
24	Deposition Rate and Substrate Appearance for Copper Closeout Layers on Aluminum Filled GF Substrates.....	75
25	Sputtered Copper Collection Efficiency.....	82

SECTION I SUMMARY

The purpose of this program was to establish the final dc triode sputtering techniques for the fabrication of high performance, long life, regeneratively-cooled thrust chambers. The program was divided into two work tasks.

In Task I OFHC® and AMIZRC® (Cu - 0.15% Zn) copper was sputtered from post and hollow cathodes onto smooth OFHC copper cylinders. Sputtering was done at different cathode and substrate potentials bias. The sputtered deposits were machined and electron beam welded to wrought OFHC copper. Tensile specimens were machined from the welded assemblies with the weld zone in the gauge section of the tensile specimen.

Excellent sputtered-wrought material welds were made with standard welding techniques. The tensile strengths approached those of the wrought material and elongations were equivalent to elongations obtained on sputtered material as established in the preceding contract for Development of Sputtered Techniques for Thrust Chambers, NAS3-17792 (Reference 1).

Based on the Task I accomplishments, the sputtering parameters used for Task II were established. All Task II copper deposition was done with a 50-volt substrate bias. Hollow cathodes were operated at -1000 volts, while post cathodes were operated at -500 volts.

Before sputter depositing the copper closeout layers, the study on groove filling with sputtered aluminum was continued as started by the proceeding contract. Groove filling efficiency was measured as a function of substrate bias voltage. Grooves 0.152-cm wide by 0.127-cm deep were filled with a single deposition cycle with a -125 volt substrate bias. Growth of aluminum in grooves was characterized by the angle made by the surface of the aluminum deposit and the bottom of the groove. The growth characteristics were similar to those for material growth over shallow steps on a substrate. Growth characteristics of sputtered aluminum over reinforcing wires were also obtained.

Five regenerative cooled spool piece liners were closed out by sputtered from hollow and post cathodes. The closedout liners were machined and the sputtered aluminum filler leached from the grooves. Sputtered material collection efficiencies were calculated for several hollow and post cathodes.

The program demonstrated that sputtered copper can be a viable engineering material. The cumulative total of the average sputtered deposit thicknesses for the program was over 10 cm.

SECTION II INTRODUCTION

In the development of advanced chambers for programs such as the Space Tug Experimental Engine Program, new fabrication techniques and/or materials will be needed to meet the projected chamber requirements. Of the techniques currently available for fabrication, deposition by sputtering offers the most potential for meeting the demands of the advanced designs. The application of sputtering techniques to the fabrication of thrust chambers permits relative freedom in materials selection for the chamber designer. Not being limited by the inability to electrodeposit a material and not having to sacrifice the material properties by elevated temperature joining operations, the chambers can be fabricated from practically any alloy or combination of alloys desired. Furthermore, the improved bonding obtainable with sputtering provides increased low-cycle fatigue life through improved materials and an elimination of joining materials at the bond interface.

Previous work by Mullaly, Hecht, Broch, and Allard (Reference 1) and McCalanahan, Busch and Moss (Reference 3) has shown that precipitation-hardened copper alloys synthesized by sputtering offer potential as materials for fabricating regeneratively-cooled thrust chambers. Furthermore, it was shown that sputtered AMZIRC copper can be stronger than the same alloy produced by conventional primary forming techniques.

The proposed method for chamber fabrication involves the sputtering of an inner or outer chamber wall, with subsequent machining to a ribbed wall configuration. The chamber channels are then filled with a suitable material and the final closeout layer applied. Fabrication by this approach requires that the filler material be compatible with the vacuum sputtering environment. The filler must be capable of being applied to the channeled configuration and completely removed without degrading the chamber material properties. The objectives of this program were (1) to final develop procedures for closing out the chamber channels by triode sputtering OFHC and AMZIRC copper from both a post and hollow cathode, and (2) to fabricate four spool piece liners by sputtering from post and hollow cathodes.

The investigation performed in the first task of this program involved developing the final procedures for fabrication of thrust chamber spool pieces. The procedure development included determining the effect of sputtering parameters on the weldability of AMZIRC copper and OFHC copper and the effects of thermal treatments on weld integrity and the closeout layer bond strength.

In the second task, four spool piece liners were fabricated by sputtering AMZIRC copper from hollow and post cathodes using Task I and previously developed procedures.

This report covers all the work performed in Tasks I and II of Contract NAS3-20377.

SECTION III
TASK I
SPUTTERING PARAMETER AND FABRICATION PROCESSING
EFFECTS ON SPOOL PIECE
CONSTRUCTION

The objective of Task I was to establish the final procedures for fabrication of thrust chamber spool pieces (NASA drawing No. CF621930). The development included determining the effect of sputtering parameters on the weldability of 0.15% zirconium-copper alloy and OFHC copper and the effects of thermal treatments or cycles on weld integrity and the closeout layer strength.

A. EQUIPMENT AND PROCEDURES

The two vacuum chambers used for sputter deposition from post and hollow cathodes have been shown schematically and described in Reference 1 as systems No. 2 and 3. System No. 2 was used as described. System No. 3 was modified to have a directly cooled hollow cathode of larger inside diameter which is 15.25 cm. One deposition was made with a shaped post cathode which is shown in figure 1.

All substrates were directly water-cooled OFHC copper cylinders 12.7 cm in length. Hollow cathode substrates were 6.604 cm ID by 7.036 cm OD (2.600 ± 0.005 ID by 2.770 ± 0.002 in. OD). Post cathode substrates were 7.036 cm ID by 8.026 cm OD (2.770 ± 0.002 ID by 3.160 ± 0.005 in. OD).

A general procedure was used for all depositions performed in this evaluation. The as-machined substrates were scrubbed with a commercial scouring powder to remove the visible oxide, then rinsed in tap water to remove the scouring powder. After a final rinse with ethyl alcohol, the substrates were installed on holders and checked for vacuum tightness with a helium mass spectrometer type leak detector. The systems were pumped to the low 10^{-7} Torr range or lower with the filaments on. Research grade krypton gas was bled into the system and sputter cleaning of the substrate started. Usually several cycles of sputter cleaning, each followed by pump down to 1.333×10^{-5} N/m² (10^{-7} Torr), were necessary to reduce substrate and system out gassing to an acceptable level. Sputter cleaning of the cathode was done simultaneously with substrate cleaning and at the same voltage. Whenever the deposition cycle was stopped for more than approximately a minute, a sputter cleaning cycle was performed before continuing the deposition. The cleaning cycle parameters are summarized for hollow and post cathodes in tables 1 and 2, respectively.

Deposition was done with varying sets of sputtering parameters as summarized in the next section of this report.

After a substrate was removed from the system, the thickest part of the deposit was permanently marked on the end of the cylinder. Each coated substrate was machined to the required diameter and then cut into three sections and numbered as shown in figure 2.

Each section was finish-machined into a weld section as shown in figure 3. Each end of the weld sections machined from Sections 1 and 3 were polished and etched for metallographic examination. Hardness readings were also taken on the sputtered deposit at these ends. All data were obtained from the area corresponding to the thickest part of the deposit.

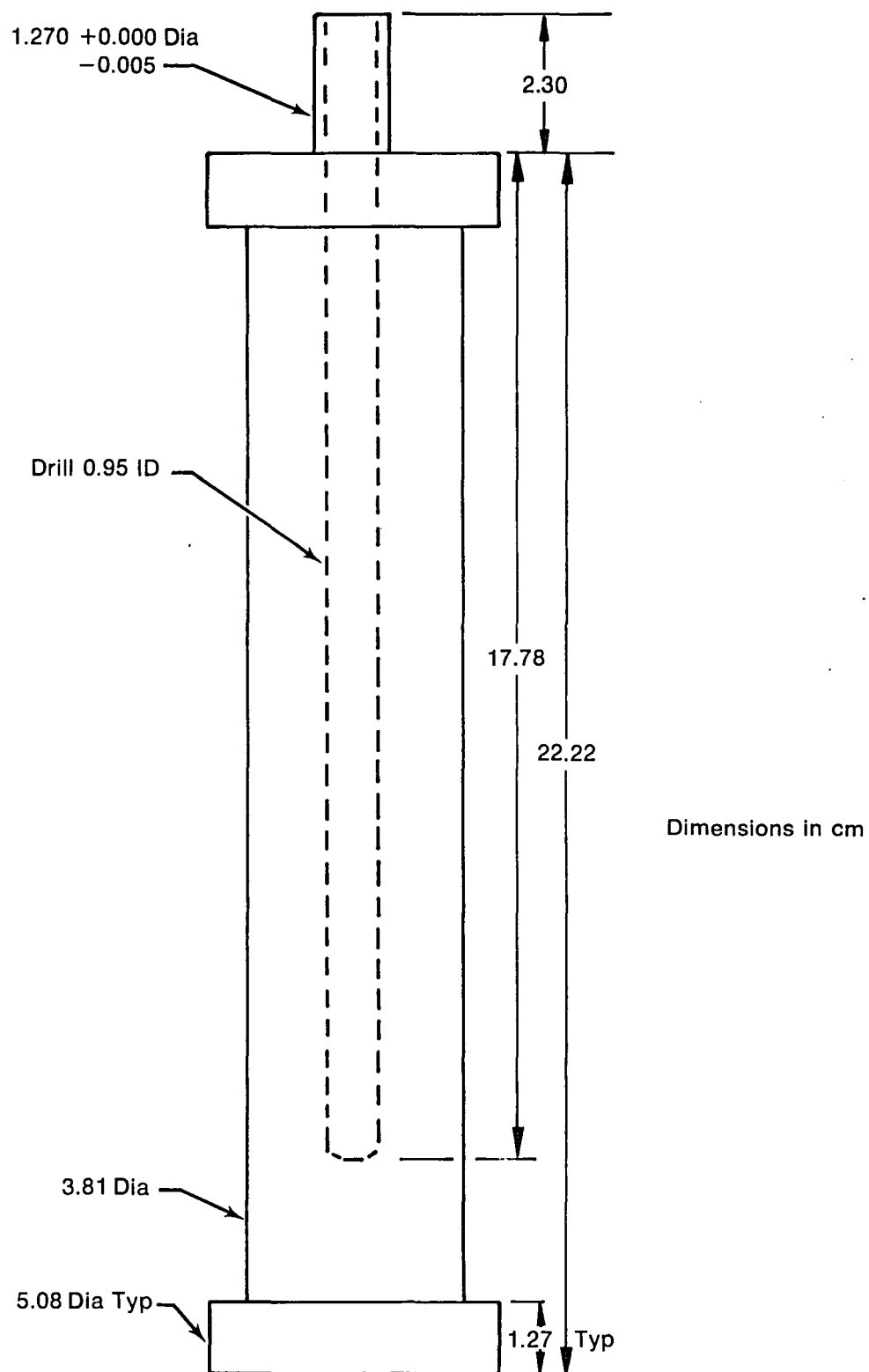


Figure 1. Shaped Post Cathode

TABLE 1. SUMMARY OF TASK I HOLLOW CATHODE SUBSTRATE SPUTTER CLEANING PARAMETERS

Substrate Number	System Number	Substrate		Time		Pressure		Cathode	Anode			
		Current (ma/cm ²)	Voltage (volts)	(min)	(ks)	(μ) ³	(N/m ²)	Current (ma/cm ²)	Current (amps)	Voltage (volts)		
I-24	3 ¹	4.3	-25	5	0.3	4.8	0.62	4.5	60.0	30.0		
		4.5	-50	30	1.8	4.4	0.57	4.7	62.0	30.0		
I-26	3 ¹	3.5	-25	5	0.3	4.9	0.64	3.6	58.0	30.0		
		2.0	-50	5	0.3	4.4	0.57	1.7	64.0	30.0		
		4.0	-50	5	0.3	8.5	1.11	3.8	71.0	30.0		
		6.3	-50	30	1.8	7.6	0.99	4.6	80.0	33.8		
		System Vented After 39.6 ks of Deposition										
I-28	3 ¹	3.6	-25	5	0.3	4.8	0.62	3.3	56.5	30.0		
		5.7	-50	30	1.8	6.1	0.79	5.7	68.0	40.0		
		1.1	-25	5	0.3	6.8	0.88	1.1	30.0	25.0		
		3.0	-25	5	0.3	6.5	0.84	3.1	47.0	30.0		
		5.4	-50	5	0.3	6.2	0.81	5.6	59.0	40.0		
		6.6	-50	30	1.8	6.5	0.84	7.0	70.2	40.0		
		6.7	-50	10	0.6	6.0	0.78	7.0	70.0	40.0		
		6.8	-50	5	0.3	5.9	0.77	7.4	72.5	40.0		
		I-29 ²	2	9.5	-25	5	0.3	6.1	0.79	5.5	30.0	42.8
				11.5	-50	5	0.3	6.0	0.78	6.3	33.0	46.0
10.9	-50			30	1.8	6.1	0.79	6.0	32.0	46.0		
System Vented After 100.8 ks of Deposition												
10.9	-25			5	0.3	6.1	0.79	5.7	30.0	43.5		
I-30 ²	2	10.9	-50	30	1.8	7.7	1.00	6.1	32.3	44.0		
		System Vented After 43.2 ks of Deposition										
		12.2	-25	5	0.3	6.4	0.83	7.1	35.5	45.0		
		10.9	-50	30	1.8	6.5	0.84	6.8	34.5	45.0		
		8.8	-25	5	0.3	5.1	0.66	6.5	29.0	46.0		
		10.2	-50	30	1.8	6.5	0.84	7.4	31.6	45.0		
		I-31 ²	2	8.2	-25	5	0.3	5.1	0.66	5.0	27.5	46.0
				10.6	-50	5	0.3	6.0	0.78	5.3	30.0	46.0
				10.9	-50	30	1.8	6.5	0.84	6.3	34.0	46.0
				10.5	-50	30	1.8	5.4	0.70	6.1	34.0	46.0
I-32 ²	2	10.2	-25	5	0.3	6.0	0.78	5.2	28.0	46.0		
		10.9	-50	30	1.8	5.4	0.70	6.6	34.3	46.0		
		System Vented After 97.2 ks of Deposition										
		13.6	-25	5	0.3	6.0	0.78	7.2	45.5	46.0		
I-33 ²	2	16.0	-50	30	1.8	6.7	0.87	8.2	44.8	46.0		
		13.6	-25	5	0.3	15.0	1.95	7.2	50.5	44.5		
		13.6	-50	5	0.3	15.0	1.95	7.8	49.8	44.0		
		14.4	-50	30	1.8	15.0	1.95	8.0	52.4	44.0		
		System Vented After 151.2 ks of Deposition										
		17.0	-25	5	0.3	6.1	0.79	7.6	41.0	46.0		
		18.3	-50	5	0.3	13.8	1.79	8.4	52.0	47.0		
		19.7	-50	5	0.3	14.3	1.86	9.9	56.5	46.0		
		18.7	-50	30	1.8	13.9	1.81	9.9	53.8	46.0		
		19.0	-50	55	3.3	14.6	1.90	9.6	54.2	45.0		
I-34 ²	2	21.7	-50	5	0.3	14.4	1.87	9.2	57.8	44.0		
		14.3	-25	5	0.3	6.5	0.84	7.6	41.0	46.0		
		13.6	-50	30	1.8	6.7	0.87	8.1	41.4	46.0		

¹Anode Current is Combined Current to Both Anodes

²3φ DC Power Supply Used for Substrate

³1μ = 1 micron = 1 × 10⁻³ mm Hg = 0.1333 N/m²

TABLE 2. SUMMARY OF TASK I POST CATHODE SUBSTRATE SPUTTER CLEANING PARAMETERS (SYSTEM NUMBER 2)

Substrate Number	Substrate		Time		Pressure		Cathode	Anode	
	Current (ma/cm ²)	Voltage (volts)	(min)	(ks)	(μ) ²	(N/m ²)	Current (ma/cm ²)	Current (amps)	Voltage (volts)
I-8P	6.2	-25	5	0.3	4.6	0.60	8.8	27.0	45.0
	6.3	-50	30	1.8	5.4	0.70	8.2	26.9	45.0
	6.5	-50	10	0.6	5.4	0.70	8.6	27.5	45.0
I-9P	6.5	-25	5	0.3	5.1	0.66	9.0	27.5	45.5
	6.7	-50	30	1.8	6.0	0.78	9.8	30.0	45.5
	6.7	-50	10	0.6	5.4	0.70	10.1	30.3	45.7
	6.7	-50	10	0.6	6.0	0.78	10.1	30.3	46.0
I-10P	5.6	-25	5	0.3	8.6	1.12	9.3	25.5	45.0
	6.5	-50	5	0.3	6.8	0.88	8.8	30.0	45.0
	6.7	-50	5	0.3	5.8	0.75	8.6	30.0	45.5
	6.7	-50	10	0.6	6.6	0.86	8.8	29.7	46.0
	6.3	-50	30	1.8	6.8	0.88	8.8	28.8	46.0
	6.6	-50	20	1.2	7.3	0.95	8.7	30.2	42.0
	6.5	-50	60	3.6	9.2	1.20	9.8	30.9	45.3
	6.6	-50	30	1.8	6.5	0.84	9.6	31.0	45.5
	6.5	-50	30	1.8	7.0	0.91	9.6	31.0	46.0
	6.6	-50	5	0.3	6.5	0.84	10.0	31.0	46.0
I-11P	6.3	-25	5	0.3	6.5	0.84	9.3	30.0	46.0
	6.7	-50	5	0.3	7.5	0.98	10.1	30.0	46.5
	6.5	-50	5	0.3	7.6	0.99	9.5	29.0	45.0
	6.7	-50	5	0.3	7.2	0.94	10.2	30.0	46.0
	6.4	-50	10	0.6	7.5	0.98	9.3	28.7	48.0
	6.5	-50	30	1.8	4.8	0.62	9.5	29.2	47.0
	6.3	-50	30	1.8	8.2	1.07	10.5	31.0	45.0
I-12P	6.7	-25	5	0.3	8.3	1.08	11.0	32.0	46.0
	6.7	-50	5	0.3	7.1	0.92	10.2	30.5	45.5
	6.7	-50	5	0.3	6.8	0.88	10.0	30.5	45.0
	6.7	-50	5	0.3	5.4	0.70	9.5	29.5	46.0
System Vented to Change Leaking AMZIRC Target (No Deposition Had Taken Place)									
	6.7	-25	5	0.3	7.1	0.92	11.2	30.8	48.0
	6.7	-25	5	0.3	7.8	1.01	12.7	31.0	46.5
	6.4	-50	5	0.3	6.8	0.88	11.4	30.0	46.0
	6.7	-50	5	0.3	8.5	1.11	12.9	31.0	46.0
	6.6	-50	5	0.3	6.8	0.88	12.1	30.0	45.0
	6.7	-50	5	0.3	6.8	0.88	12.1	31.0	45.5
I-13P ¹	7.8	-25	5	0.3	6.1	0.79	13.2	32.5	48.0
	7.2	-50	15	0.9	6.1	0.79	12.6	31.3	46.3
	7.5	-50	30	1.8	6.5	0.84	12.6	33.2	46.8
	7.4	-50	5	0.3	6.5	0.84	13.0	31.5	46.0
	7.5	-50	5	0.3	5.6	0.73	13.4	33.0	46.0
	7.3	-50	5	0.3	5.4	0.70	13.2	32.0	46.0
	6.8	-50	5	0.3	5.4	0.70	12.0	31.5	44.0
I-14P ¹	6.7	-25	5	0.3	6.8	0.88	11.4	31.0	48.0
	7.2	-50	5	0.3	7.1	0.92	12.7	33.0	47.0
	7.7	-50	5	0.3	6.6	0.86	14.1	35.0	46.5
	7.3	-50	15	0.9	6.3	0.82	13.5	33.3	46.0
	7.3	-50	12	0.7	5.4	0.70	14.3	34.7	43.7
	8.1	-50	15	0.9	6.1	0.79	15.6	37.3	43.7
System Vented After Powder Outage									
	6.1	-25	5	0.3	5.1	0.66	10.9	30.0	48.0
	7.7	-50	5	0.3	7.5	0.98	13.8	34.0	46.0
	8.1	-50	5	0.3	7.1	0.92	15.3	37.5	45.0
	7.5	-50	20	1.2	6.5	0.84	13.8	34.5	45.5
	7.2	-50	5	0.3	6.8	0.88	13.2	34.2	44.0
I-15P ¹	6.5	-25	5	0.3	6.5	0.84	9.8	30.2	49.0
	7.3	-50	5	0.3	6.5	0.84	9.8	34.0	45.5
	6.8	-50	50	3.0	5.6	0.73	9.7	31.3	47.0

¹3φ DC Power Supply Used for Substrates

²1μ = 1 micron = 1 × 10⁻³ mm Hg = 0.1333 N/m²

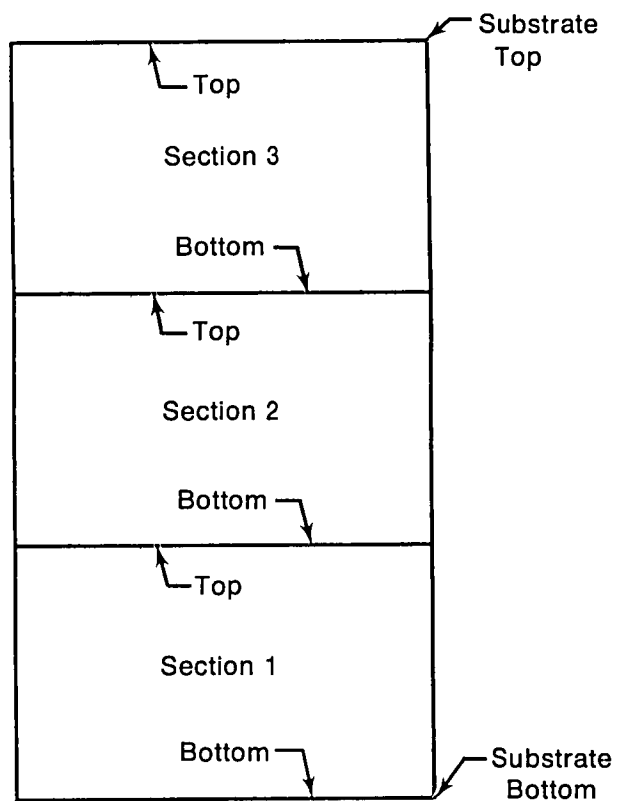
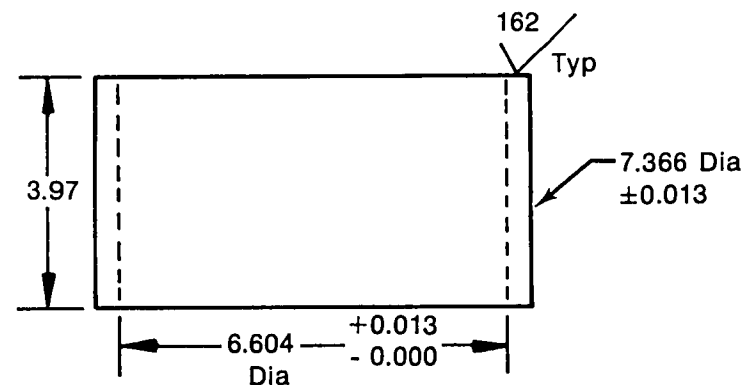
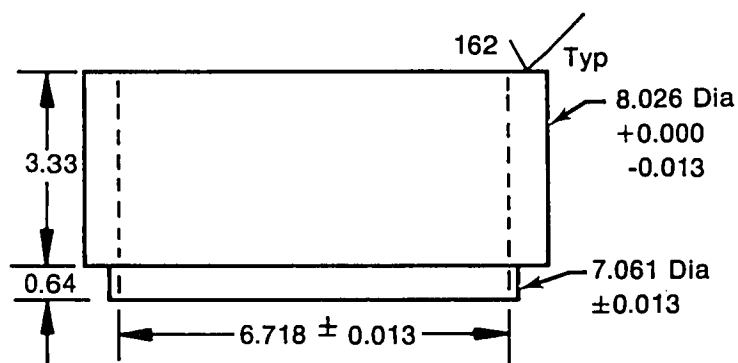


Figure 2. Orientation of Weld Pieces on Post and Hollow Cathode Substrates



Hollow Cathode Weld Section



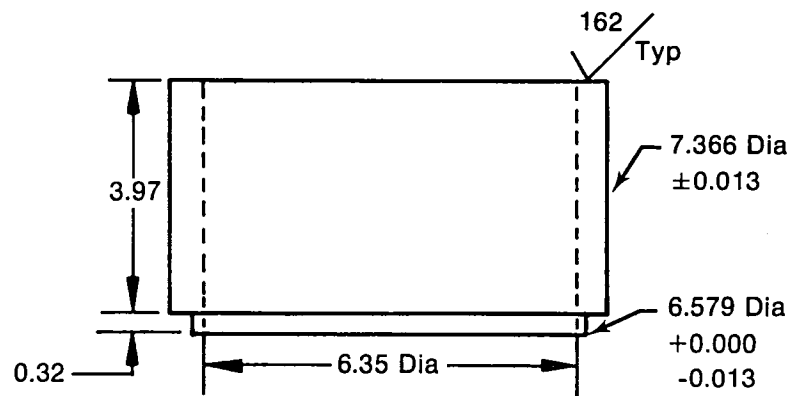
Post Cathode Weld Section

Dimensions in cm

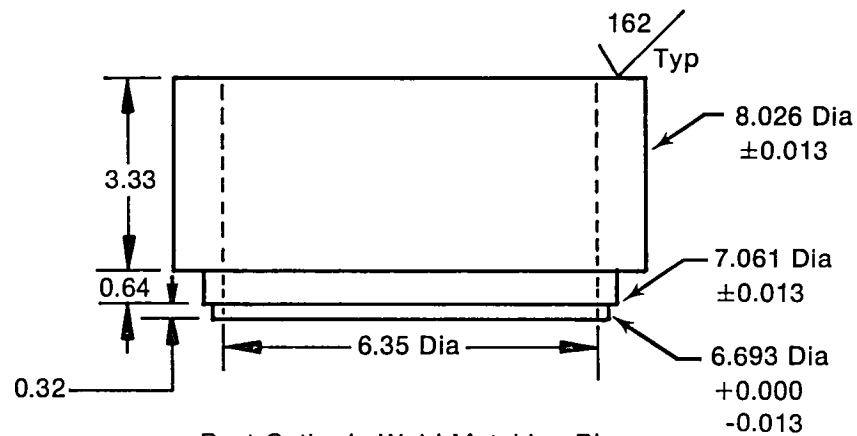
Figure 3. Machining Details for Weld Sections

Prior to welding, all sputtered and wrought copper parts were cleaned in a PS4-F solution and neutralized in PS52-A. The PS4-F solution is a bright dip solution with three parts sulfuric acid, two parts nitric acid, and one part water (by volume). The PS52-A solution is a 3% caustic soda solution.

The weld pieces from Sections 1 and 2 were electron beam welded to weld matching pieces which are shown in figure 4. The two different weld preps and matching pieces were necessitated by the different sizes of the hollow and post cathode substrates. The top end of the weld pieces from the No. 1 sections were welded to the OFHC copper weld matching pieces and the bottom ends of the weld pieces from the No. 2 sections were welded to the AMZIRC copper weld matching pieces. Welding was done with an 13,800 v, 11 ma beam in two passes.



Hollow Cathode Weld Matching Piece

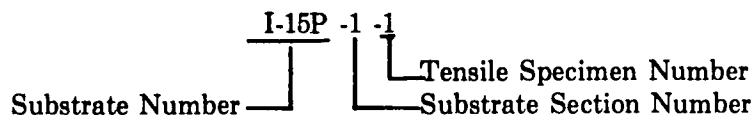


Post Cathode Weld Matching Piece

Dimension in cm

Figure 4. Machining Details for Weld Matching Pieces

The welded assemblies were machined to size as shown in figure 5. Six tensile specimens were cut from each weld assembly as shown in figure 6. The actual gage section could have been slightly shorter than the dimension specified on figure 6 due to polishing of the weld section for metallographic examination. The specimens were X-rayed to show the condition of the weld. The specimens cut from the area corresponding to the thickest part of the deposit was designated No. 1. The other five specimens were arbitrarily numbered from two through six. The complete numbering system for tensile specimens is illustrated by the following example.



The zirconium content of the sputtered AMZIRC copper was determined by electron microprobe analysis of the tensile specimens (see tables 5 and 6).

Small sections of welded material were salvaged from the specimen cut-out operation on some of the post cathode deposits. These small sections were used in the thermal treatment investigations.

Burst specimens were machined as shown on figures 7 and 8. These specimens were to be used in place of the normal end caps on the substrate holders to determine bond strength of the sputtered closeout layer. However, the sputtered aluminum filling process (see Task II) revealed that these specimens were not receiving sufficient cooling to be indicative of substrate material closeout layer combinations. Therefore, these specimens were not overcoated with copper. Although these specimens were not completed, their description is included in this report for possible future reference.

B. DEPOSITION PARAMETERS

The parameters used in Task I depositions are given in tables 3 and 4 for the hollow and post cathodes, respectively. All data are the average of values recorded periodically during the deposition cycle. The current densities given (ma/cm^2) were obtained by dividing the total substrate or cathode current by the respective electrode area. This does not imply that these electrodes were evenly bombarded by ions. The system pressure is krypton pressure, i.e., the pirani gauge reading has been corrected for krypton.

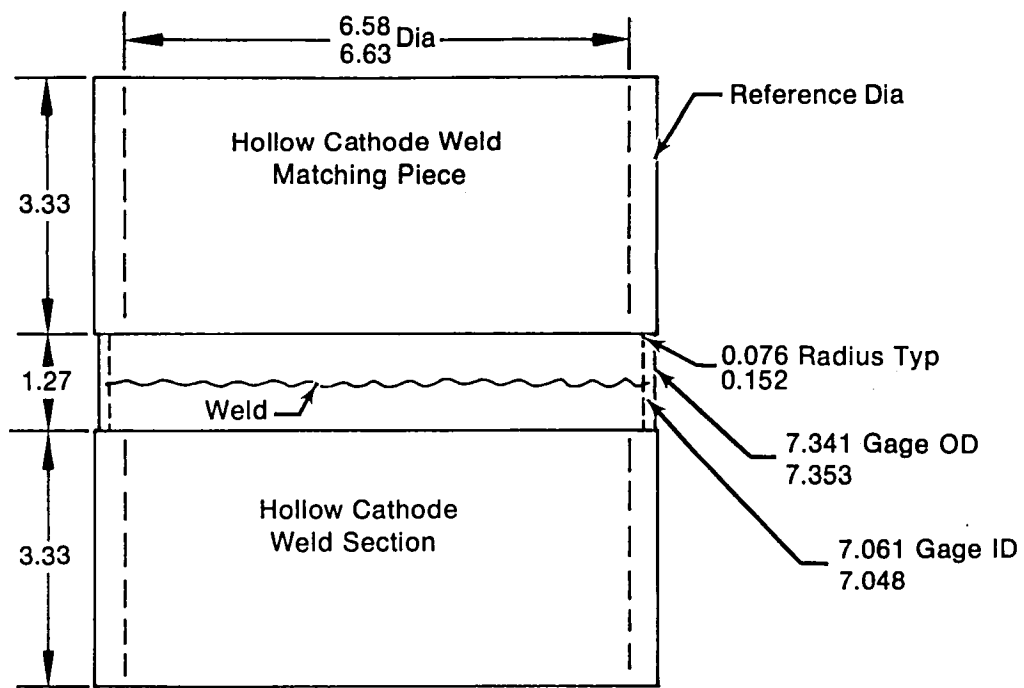
The expected effect of deposition parameters has been discussed in Reference 1. In this work, substrate bias and cathode voltage were chosen as the parameters *most likely* to affect weldability of the sputtered material. However, it should be added to the effect of cathode voltage discussion given in Reference 1, that an increase in cathode voltage will increase the average energy of the sputtered atoms arriving at the substrate.

C. RESULTS

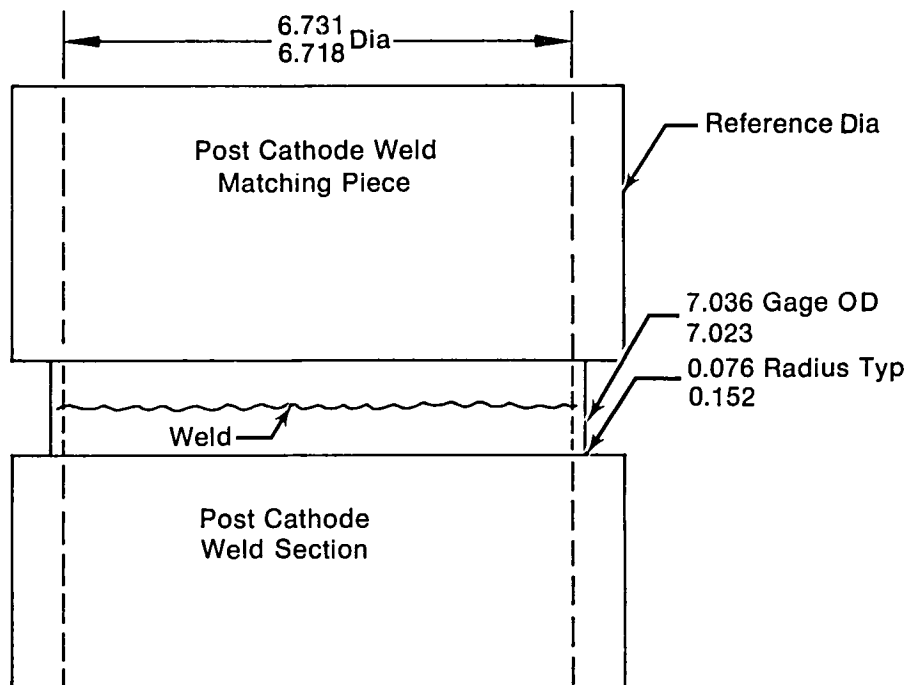
The deposition rate, AMZIRC composition, and Vickers hardness for hollow and post cathode depositions are summarized in tables 5 and 6. These results will be discussed first and then the results of welding will be presented.

1. Characterization of Sputtered Deposits

The deposit thicknesses given in the tables are determined by micrometer readings taken before and after deposition. This gives an average thickness since the deposits were not always even around the circumference of a substrate. This unevenness is caused by system geometry and has been discussed in Reference 1. Geometry effects for the hollow and post cathodes will be discussed further for this work.



Hollow Cathode Weld Assembly



Post Cathode Weld Assembly

Dimensions in cm

Figure 5. Machining Details for Weld Assemblies. Gage Surface $\sqrt{}$

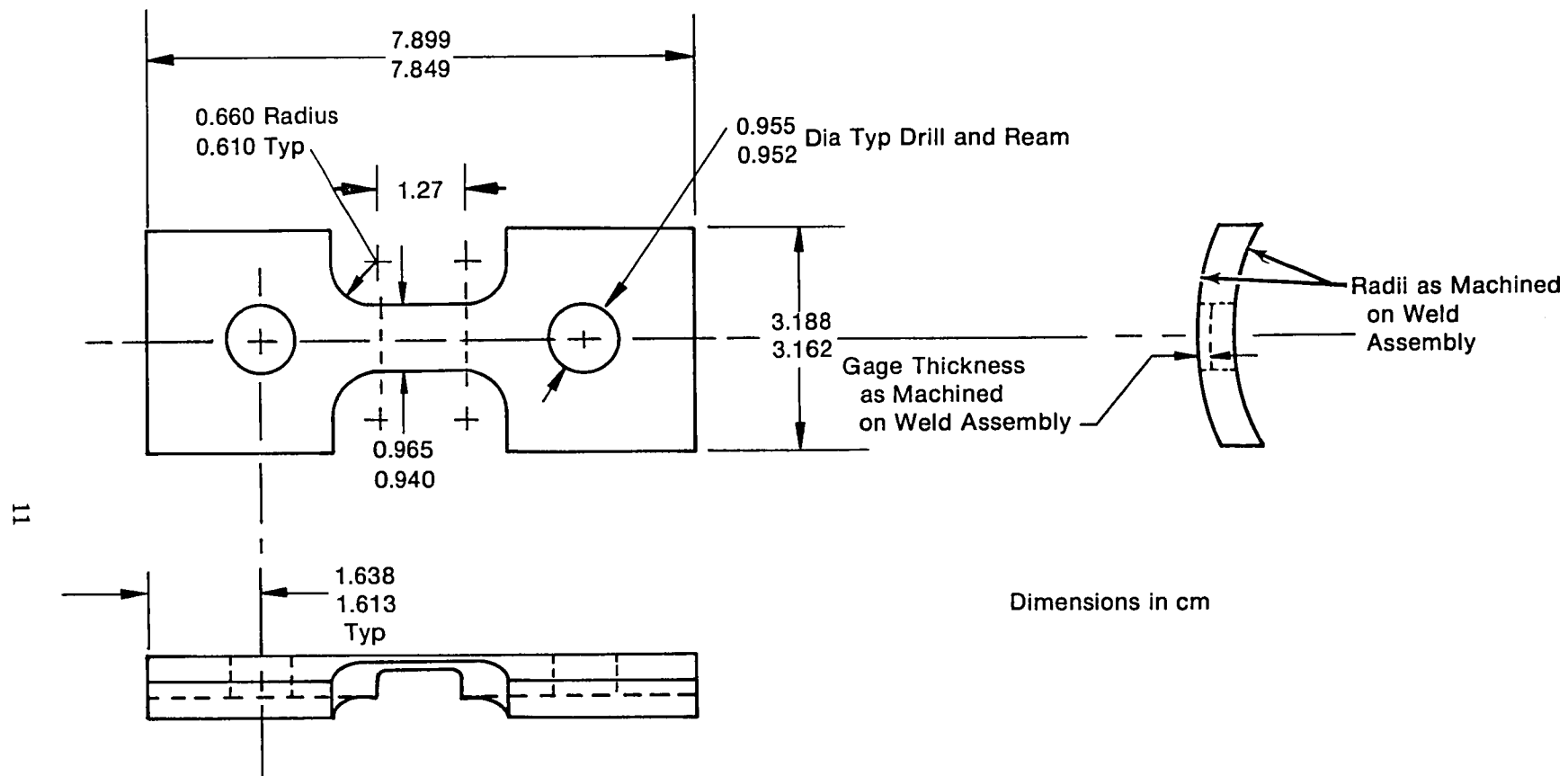


Figure 6. Machining Details for Cutting Hollow Cathode Tensile Specimens from Weld Assemblies

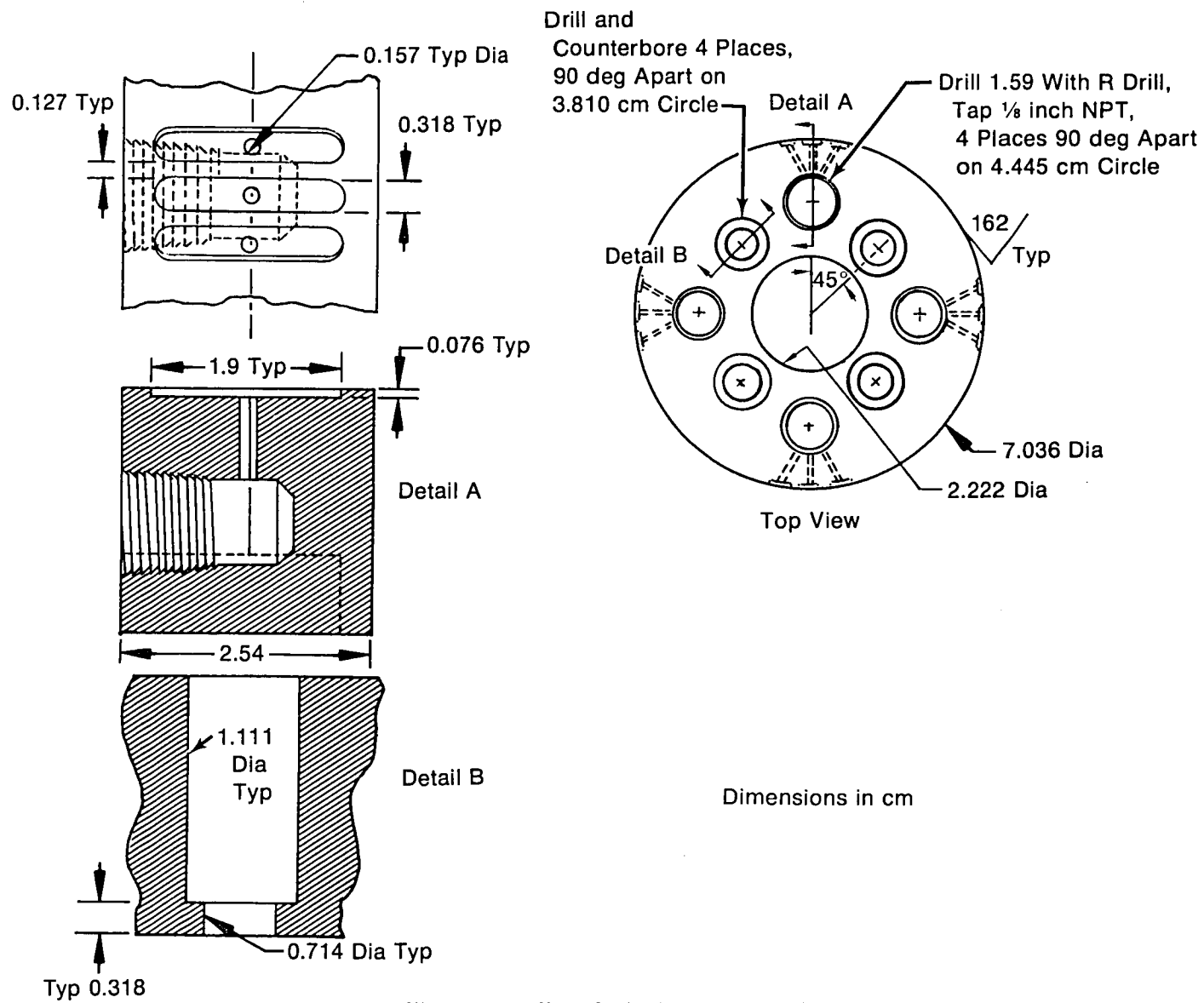


Figure 7. Hollow Cathode Burst Specimen

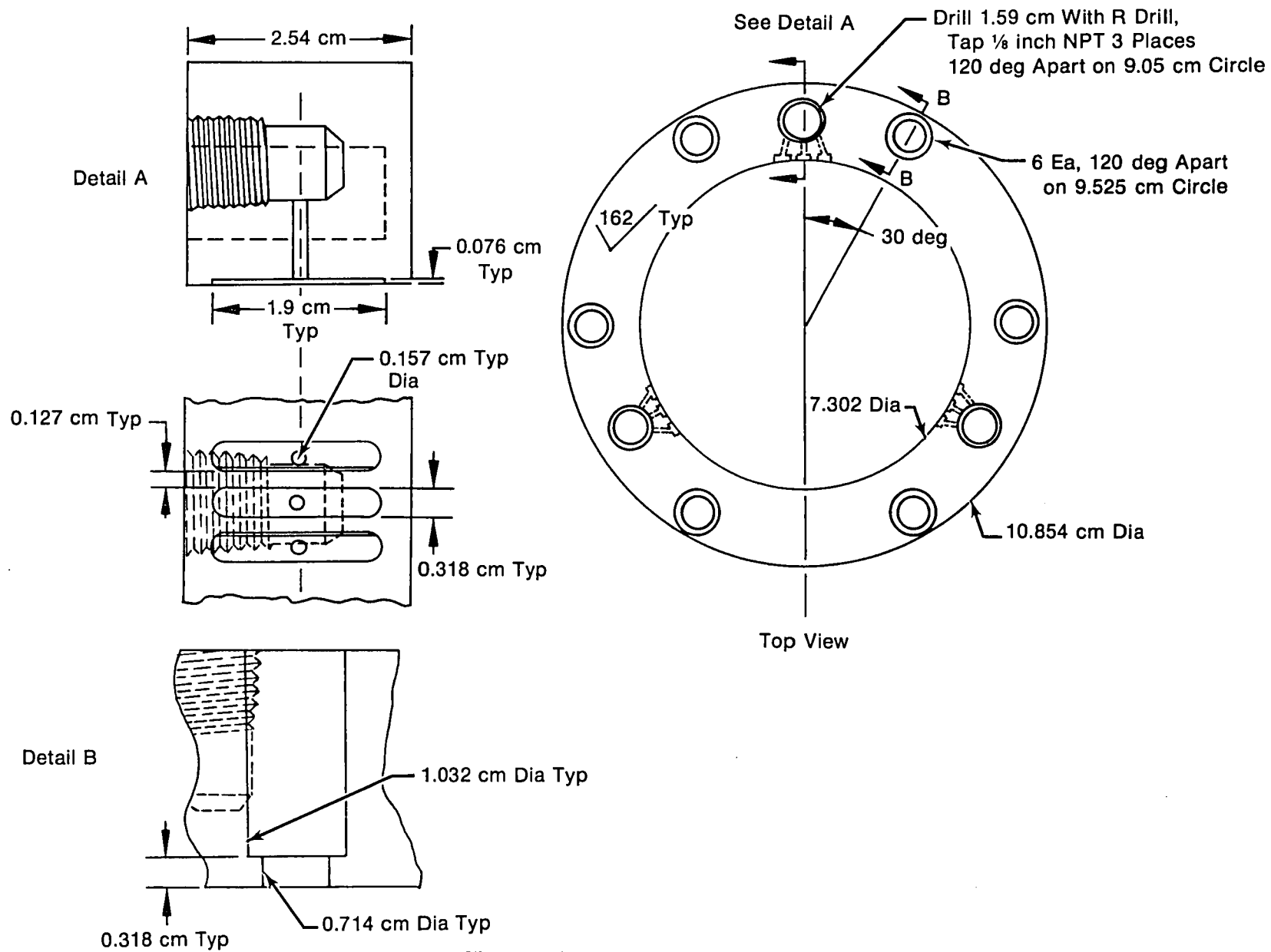


Figure 8. Post Cathode Burst Specimen

TABLE 3. SUMMARY OF TASK I HOLLOW CATHODE DEPOSITION PARAMETERS

Substrate Number	Substrate		Cathode		Material Copper	Anode		Deposition Time		Pressure	
	Current (ma/cm ²)	Voltage (volts)	Current (ma/cm ²)	Voltage (volts)		Current (amps)	Voltage (volts)	(hr)	(ks)	(μ) ³	(N/m ²)
I-24 ¹	4.4	-50	4.3	-1000	OFHC	65.0	30.0	104.0	374.4	3.8	0.49
I-26 ¹	6.3	-50	5.4	-1000	OFHC	78.4	33.4	11.0	39.6	7.2	0.94
	System Vented										
	6.7	-50	6.3	-1000	OFHC	71.6	45.0	46.0	165.6	6.0	0.78
I-28 ¹	6.1	-50	6.1	-1500	OFHC	68.3	38.2	0.3	1.2	5.6	0.73
	0.4	0	6.3	-1500	OFHC	70.5	40.0	1.2	4.5	6.3	0.82
	0.4	0	6.5	-1500	OFHC	70.0	40.0	2.5	9.0	5.8	0.75
	4.7	0	5.4	-1500	OFHC	64.6	36.3	11.5	41.4	5.7	0.74
	6.3	-50	5.8	-1500	OFHC	66.2	40.0	36.0	129.6	4.9	0.64
I-29 ²	9.2	-50	5.7	-1000	AMZIRC	33.2	44.0	6.0	21.6	5.6	0.73
	9.5	-50	5.7	-1000	AMZIRC	32.9	44.0	22.0	79.2	5.9	0.77
	System Vented										
	10.4	-50	5.6	-1000	AMZIRC	33.3	44.0	12.0	43.2	6.3	0.82
	System Vented										
	10.9	-50	5.9	-1000	AMZIRC	34.0	44.0	18.0	64.8	5.7	0.74
I-30 ²	9.1	-25	6.7	-1000	AMZIRC	33.9	44.0	32.0	115.2	5.0	0.65
	10.9	-25	6.7	-1000	AMZIRC	34.0	44.0	30.0	108.0	6.8	0.88
I-31 ²	8.2	0	5.4	-1000	AMZIRC	34.7	44.0	21.0	75.6	4.9	0.64
	8.2	0	5.4	-1000	AMZIRC	34.2	44.0	19.0	68.4	5.1	0.66
	9.5	0	5.3	-1000	AMZIRC	34.7	44.0	10.0	36.0	6.1	0.79
I-32 ²	10.9	-25	7.5	-500	AMZIRC	42.9	44.0	19.0	68.4	6.3	0.82
	13.1	-25	8.3	-500	AMZIRC	47.3	44.0	8.0	28.8	6.4	0.83
	System Vented										
	13.6	-25	7.7	-500	AMZIRC	44.2	44.0	17.0	61.2	6.2	0.81
I-33 ²	13.6	-50	8.2	-500	AMZIRC	51.0	44.0	42.0	151.2	17.1	2.22
	System Vented and Substrate Rotated 180 deg										
	18.7	-50	9.2	-500	AMZIRC	57.4	44.0	9.0	32.4	16.0	2.08
	16.3	-25	8.6	-500	AMZIRC	49.8	44.0	35.0	126.0	15.8	2.05
I-34 ²	13.1	-25	7.7	-1000	OFHC	42.7	46.2	42.0	151.2	9.1	1.18

¹System Number 3. Anode Current is Combined Current to Both Anodes²3φ DC Power Supply Used for Substrate³1μ = 1 micron = 1 × 10⁻³ mm Hg = 0.1333 N/m²

TABLE 4. SUMMARY OF TASK I POST CATHODE DEPOSITION PARAMETERS (SYSTEM NUMBER 2)

Substrate Number	Substrate		Cathode		Material Copper	Anode		Deposition Time		Pressure	
	Current (ma/cm ²)	Voltage (volts)	Current (ma/cm ²)	Voltage (volts)		Current (amps)	Voltage (volts)	(hr)	(ks)	(μ) ³	(N/m ²)
I-8P	6.5	-50	7.5	-500	AMZIRC	30.5	42.0	85	306.0	5.6	0.73
I-9P	6.6	-50	9.6	-500	OFHC ¹	30.2	46.4	36	129.6	7.0	0.91
I-10P	5.4	-25	8.2	-500	AMZIRC	31.1	42.0	63	226.8	6.1	0.79
	5.9	-25	6.5	-500	AMZIRC	30.2	42.0	30	108.0	6.6	0.86
I-11P	5.4	0	8.4	-500	AMZIRC	31.6	42.0	89	320.4	6.8	0.88
I-12P	5.9	-25	9.2	-1000	OFHC	30.5	44.0	72	259.2	5.7	0.74
I-13P ²	7.1	-50 ¹	10.7	-1000	OFHC	34.1	44.0	59	212.4	5.9	0.77
I-14P ²	5.9	0	11.7	-1000	OFHC	32.2	44.0	3	10.8	5.9	0.77
	6.3	0	9.7	-1000	OFHC	34.1	44.0	69	248.4	5.2	0.68
I-15P ²	7.3	-25	10.6	-1000	AMZIRC	37.4	45.4	48	172.8	5.5	0.72
	8.0	-25	8.0	-1000	AMZIRC	38.0	46.0	12	43.2	5.1	0.66

¹Spool Shaped Target (Figure 1)

²3φ DC Power Supply Used for Substrate

³1μ = 1 micron = 1 × 10⁻³ mm Hg = 0.1333 N/m²

TABLE 5. DEPOSITION RATE, COMPOSITION, AND VICKERS HARDNESS FOR HOLLOW CATHODE AS-SPUTTERED COPPER. 10.0 kg LOAD

Substrate Number	Sputtered Material (copper)	Deposit Thickness		Deposition Rate		Zirconium Content (wt %)	Substrate Hardness (VPN)	Deposit Hardness (VPN)			
		At Center of Substrate		At Center of Deposit				Section No. 1		Section No. 3	
		(mils)	(mm)	(mils/hr)	(nm/sec)			Top	Bottom	Top	Bottom
I-24	OFHC	103.0	2.62	0.99	7.0	—	117	174	173	181	177
I-26	OFHC	63.5	1.61	1.11	7.8	—	105	186	190	195	190
I-28	OFHC	84.5	2.17	1.62	11.4	—	121	181	188	195	184
I-29	AMZIRC	86.0	2.18	1.48	10.4	0.12	123	251	247	251	258
I-30	AMZIRC	86.0	2.18	1.39	9.8	0.14	120	245	240	245	245
I-31	AMZIRC	104.5	2.65	2.09	14.7	0.19	122	234	247	243	258
I-32	AMZIRC	78.5	1.99	1.78	12.6	0.12	121	240	243	237	240
I-33	AMZIRC	153.5	3.90	1.78	12.6	0.08	121	237	240	241	230
I-34	OFHC	118.5	3.01	2.82	19.9	—	122	109	162	88	111

TABLE 6. DEPOSITION RATE, COMPOSITION, AND VICKERS HARDNESS FOR POST CATHODE AS-SPUTTERED COPPER. 10.0 kg LOAD

Substrate Number	Sputtered Material (copper)	Deposit Thickness		Deposition Rate		Zirconium Content (wt %)	Substrate Hardness (VPN)	Deposit Hardness (VPN)	
		At Center of Substrate		At Center of Deposit				Section No. 1	Section No. 3
		(mils)	(mm)	(mils/hr)	(nm/sec)				
I-8P	AMZIRC	83.0	2.11	0.98	6.9	0.18	101	237	237
I-9P	OFHC	24.0	0.61	0.67	4.7	—	—	—	—
I-10P	AMZIRC	93.5	2.37	1.01	7.1	0.19	94	247	244
I-11P	AMZIRC	106.0	2.69	1.19	8.4	0.18	96	244	253
I-12P	OFHC	140.7	3.57	1.95	13.8	—	103	82-188	70-181
I-13P	OFHC	110.0	2.79	1.86	13.1	—	103	158	166
I-14P	OFHC	135.7	3.45	1.88	13.3	—	96	50	61
I-15P	AMZIRC	135.0	3.43	2.25	15.9	0.06	100	235	232

The larger ID cathode used with system No. 3 was designed specifically to improve the deposit evenness on hollow cathode substrates. The deposit thickness for substrates I-24, I-26, and I-28 are plotted vs substrate length in normalized units in figure 9. The measured thickness can be calculated by multiplying the normalized unit by the center thickness given in tables 5 and 6. The deposits were very uniform over the central section of the substrate. This is a considerable improvement over the deposits obtained in the same system with a smaller diameter cathode as those shown in Reference 1, figure 1C. The evenness around the circumference was within $\pm 5\%$ of perfect evenness. A vacuum integrity problem with the water cooled anodes on system No. 3 precluded doing all of Task I depositions in this system.

The deposit profiles for the remaining hollow cathode work are shown in figures 10 and 11. These profiles are essentially the same as shown in Reference 1, figure 1B. The thickness measurements for these profiles were made across the diameter connecting the thickest and thinnest part of the deposit (viewing the substrate end-on). This variation in thickness around the circumference and the change in cathode size and shape due to intensive sputtering precludes a correlation of deposit profiles with sputtering parameters. Some of these thickness variations are given in table 7. The unevenness is more pronounced at the top of each cylinder.

Deposit profiles for post cathode work are shown in figures 12 and 13. The deposits are characterized by being thicker at the centers than at the ends. Substrate I-9P was coated with the spool shaped cathode shown in figure 1. The I-9P deposit was considerably different, being thicker at each end of the substrate. This shows it may be possible to obtain an even deposit over the ID of a cylinder by using a shaped cathode. This will be discussed further in Task II. Note that the ID of the post cathode substrates were only measured at 2.54 cm intervals whereas the hollow cathode substrate OD's were measured every 1.27 cm.

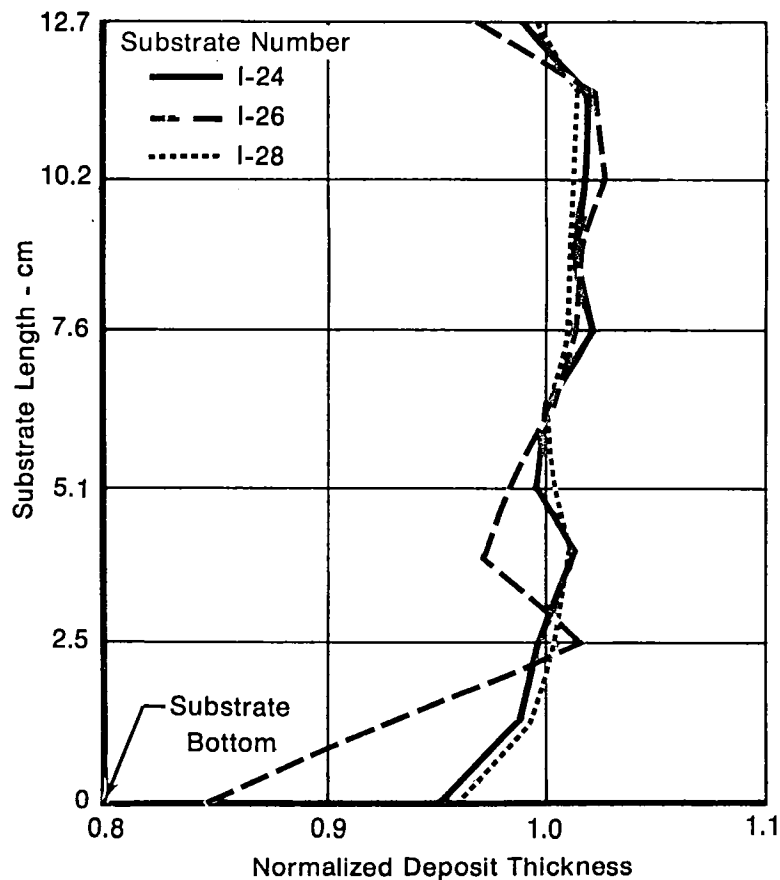


Figure 9. Variation of OFHC Copper Deposit Thickness for Hollow Cathode Substrates in a 15.24 cm ID Cathode (System No. 3)

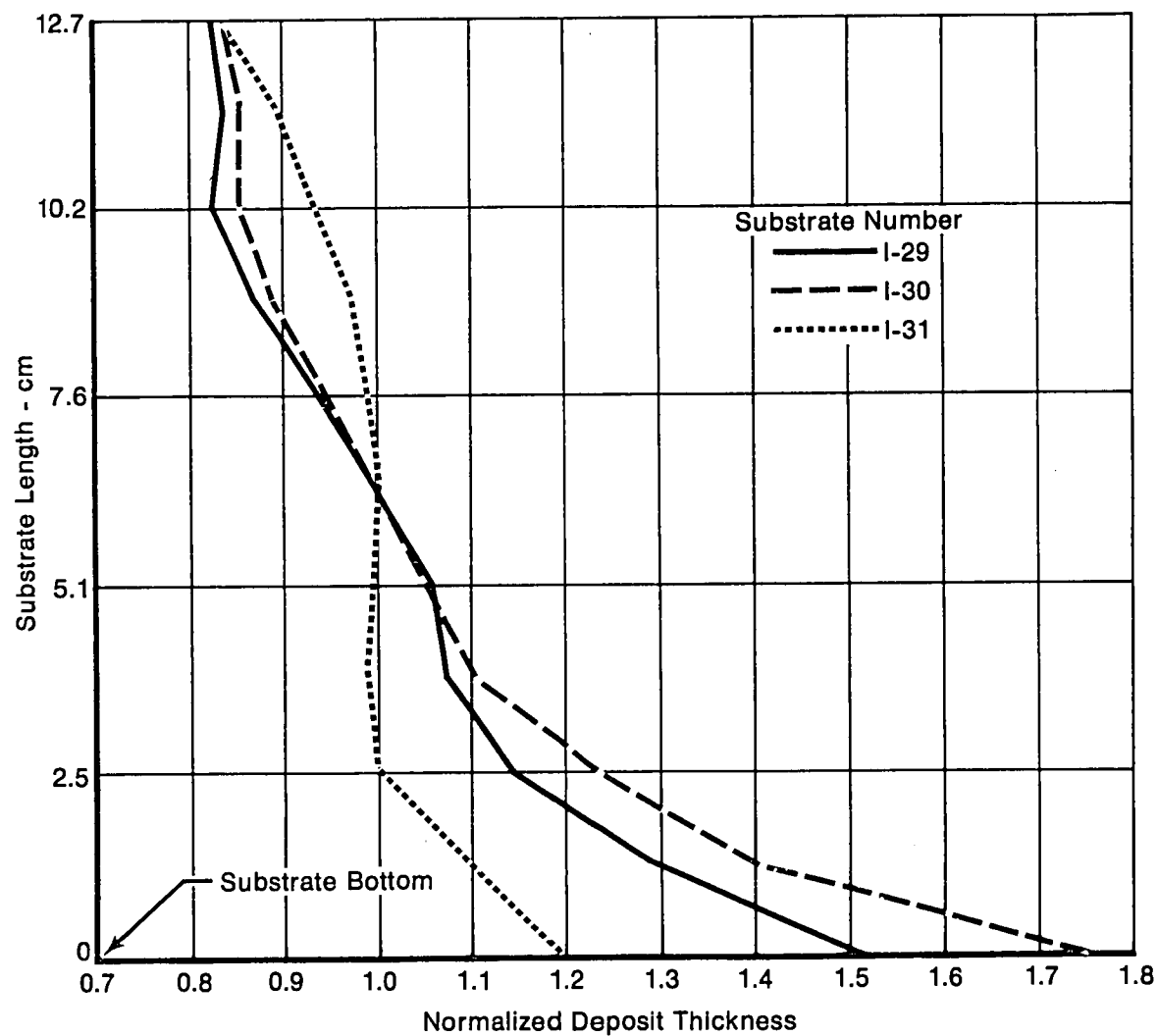


Figure 10. Variation of AMZIRC Copper Deposit Thickness for Hollow Cathode Substrates in a 10.16 cm ID Cathode (System No. 2). The 10.16 cm ID Was the Starting ID. All Three Substrates Were Coated With the Same Cathode

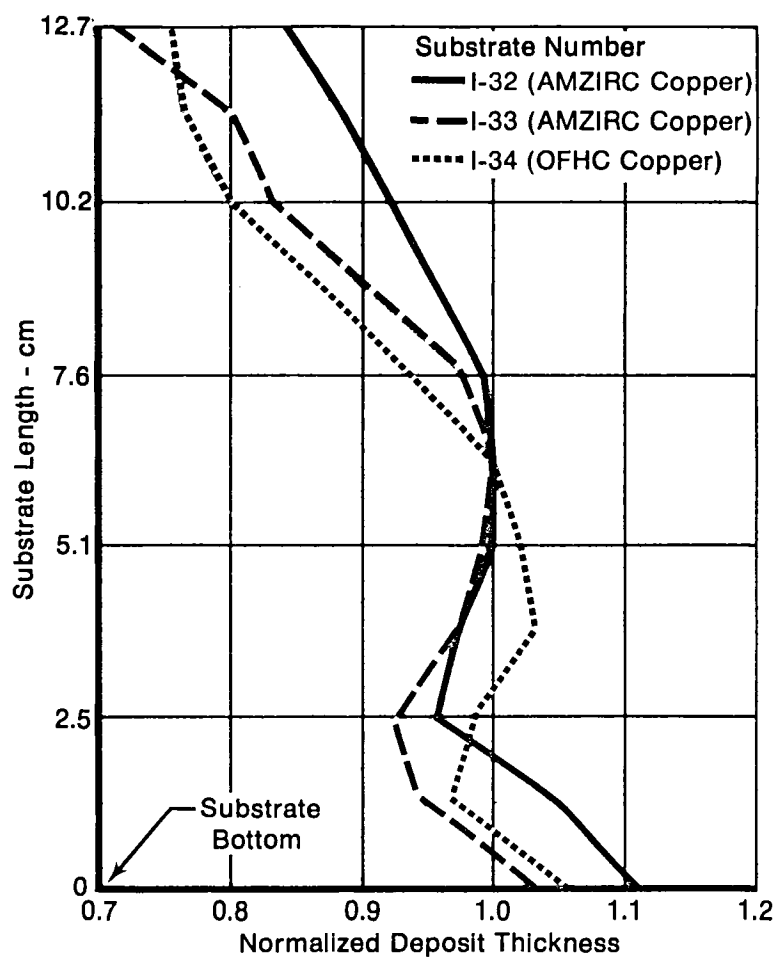


Figure 11. Variation of Copper Deposit Thickness for Hollow Cathode Substrates in 10.16 cm ID Cathodes (System No. 2). I-32 and I-33 Were Coated in the Same Cathode as I-29, I-30, and I-31. I-34 Was Coated in a New 10.16 cm ID Hollow Cathode

TABLE 7. DEPOSIT THICKNESS OF HOLLOW CATHODE SUBSTRATES IN SYSTEM NO. 2

Substrate Number	Position	
	Top	Bottom
I-31	0.348/0.079	0.368/0.307
I-32	0.364/0.063	0.257/0.188
I-33	0.401/0.155	0.457/0.389
I-34	0.348/0.114	0.363/0.282

Note: Dimensions are at max./min. cm.

The data in table 8 show that the deposit is even around the bottom of the post cathode substrates but not at the top. The ID of substrates I-14P and I-15P were not actually round, but had an octagonal shape when viewed end-on. The shaping was more pronounced for substrate I-15P. Similar shaping has been reported by McClanahan, Busch, and Moss (Reference 3).

The visual appearance of the sputtered deposits varied from bright (reflective) to matte (nonreflective) with only a rough correlation to sputtering parameters. Hollow cathode deposits at -1000 and -1500 volts had the matte finish while deposits at -500 volts varied from 25 to 80% bright. Post cathode deposits were bright at all voltages except when a 0 volt bias was used. The bright surfaces were not necessarily smooth, e.g., I-15P was bright but octagonal shaped. Some of the matte surfaces were very smooth and defect free.

Although the zirconium content of the sputtered AMZIRC cannot be correlated to sputtering parameters, the data in tables 5 and 6 clearly show that zirconium is transferred to the deposit. The correlation of zirconium content with sputtering parameters is complicated by the uneven distribution of zirconium in the cathodes.

Typical microstructures of hollow cathode deposits are shown on figures 14 and 15. All structures were dense with columnar growth habit and with the exception of I-34, no grains could be discerned at magnifications up to 500X. The first 1/3 of the coating on I-34 had a fine grain structure (still maintaining the columnar habit). All deposit to substrate bonds appeared sound at 500X.

Some of the structures had layered sawtooth patterns superimposed on the columnar structure. The patterns were similar to the patterns shown in Reference 1, figure 33 except that the patterns in the present work are all parallel with the surface of the substrates. The patterns were visible at the top and bottom ends on substrates I-26 and I-28 (none observed on I-24) and at the bottom end of the substrates I-29, I-31, and I-33 (none observed on I-32). The top of piece No. 1 from I-29, I-30, and I-31 also had the sawtooth pattern which became more pronounced from I-29 to I-31, seemingly a function of substrate bias. However, no change in the pattern was observed on either I-28 or I-33 when the bias was changed during the deposition. The significance of this pattern to deposit properties is not known.

Some void defect structures (termed "leaders" in Reference 1, figure 20) were also observed starting at defects at the substrate surface. The leader population increased with decreasing substrate bias, i.e., substrate bias could effectively stop the leaders from becoming cracks in the closeout layer.

Microstructures of post cathode deposits were different in several respects from hollow cathode deposits. No columnar habit was observed and a variety of microstructures were found.

Figure 16 shows a featureless microstructure typical of the deposit on substrates I-8P, I-10P, and the ends of I-13P.

Substrate I-11P was also similar to I-8P except for one sawtooth pattern running parallel to the substrate surface and approximately 0.9 mm from the substrate surface (observed on the bottom of piece No. 3). The pattern apparently divided regions of different hardness. The deposit had a hardness of 239 VPN near the substrate and 270 VPN near the edge. The mechanism responsible for this change in hardness is still not understood. This change in hardness was also characteristic of the post cathode deposits reported in Reference 1.

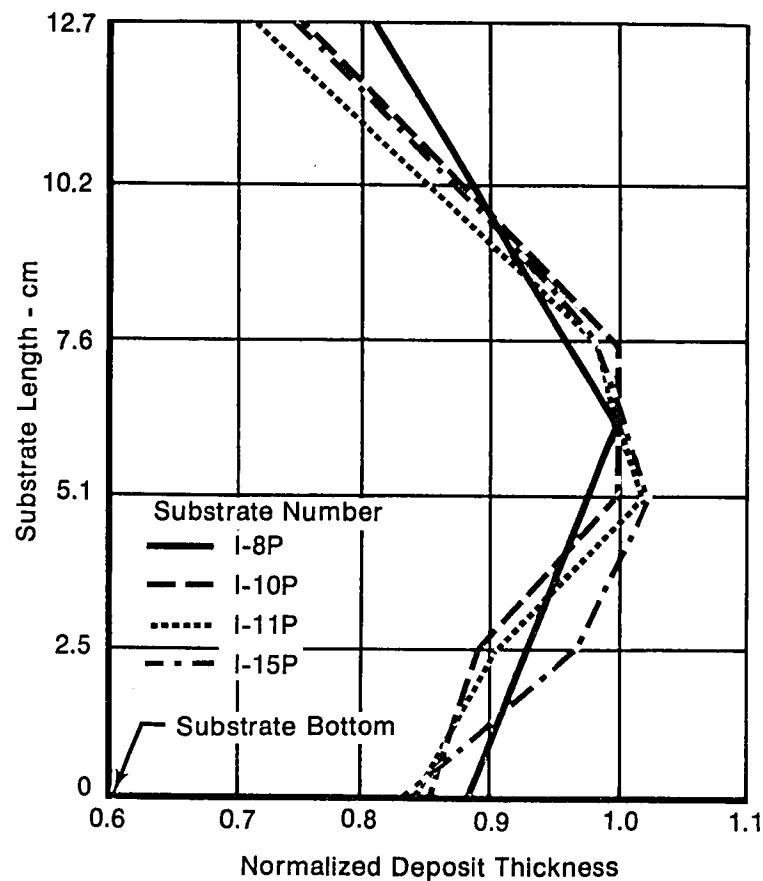


Figure 12. Variation of AMZIRC Copper Deposit Thickness for Post Cathode Substrates (System No. 2)

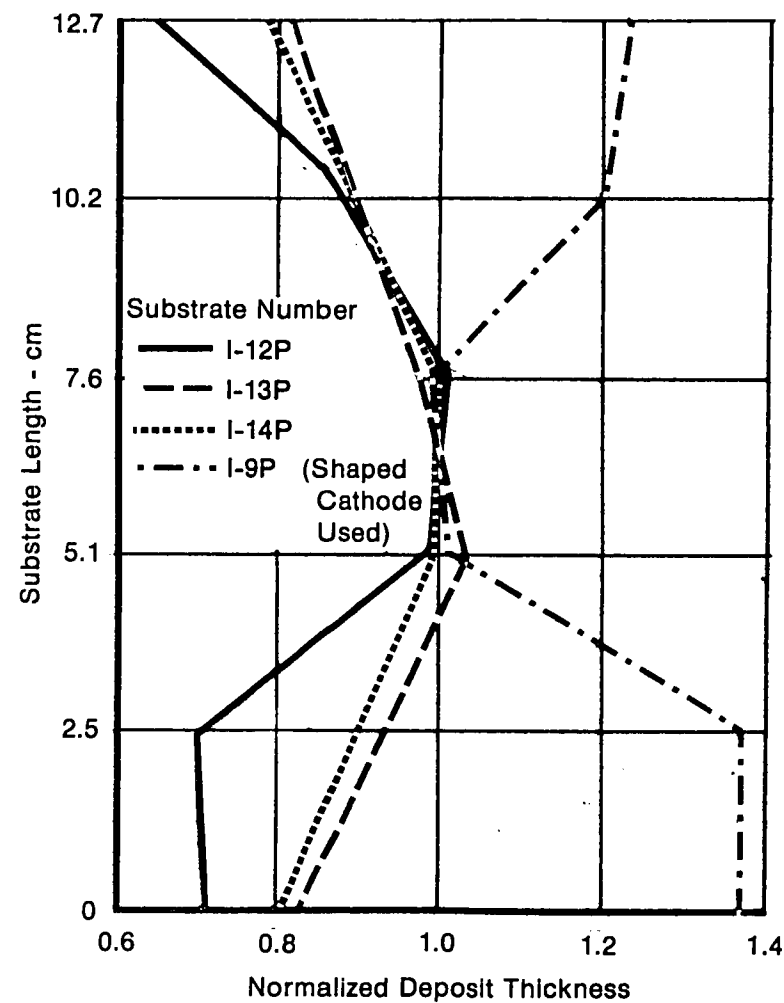
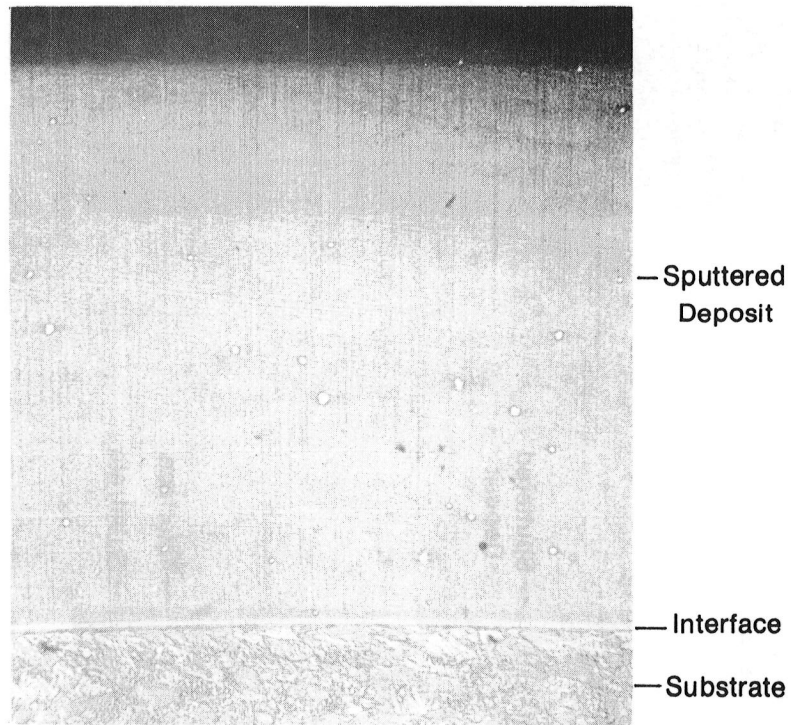


Figure 13. Variation of OFHC Copper Deposit Thickness for Post Cathode Substrates (System No. 2)

TABLE 8. DEPOSIT THICKNESS OF POST CATHODE SUBSTRATES

Substrate Number	Position	
	Top	Bottom
I-11P	0.254/0.127	0.226/0.226
I-12P	0.351/0.096	0.254/0.254
I-13P	0.318/0.178	0.228/0.228

Note: Dimensions are at max./min. cm.



Mag: 50X Etched

Recrystallization Starting

Figure 14. Typical Microstructure of Sputtered OFHC Copper Deposits from System No. 3 (Substrate I-26, Top of Section No. 1)

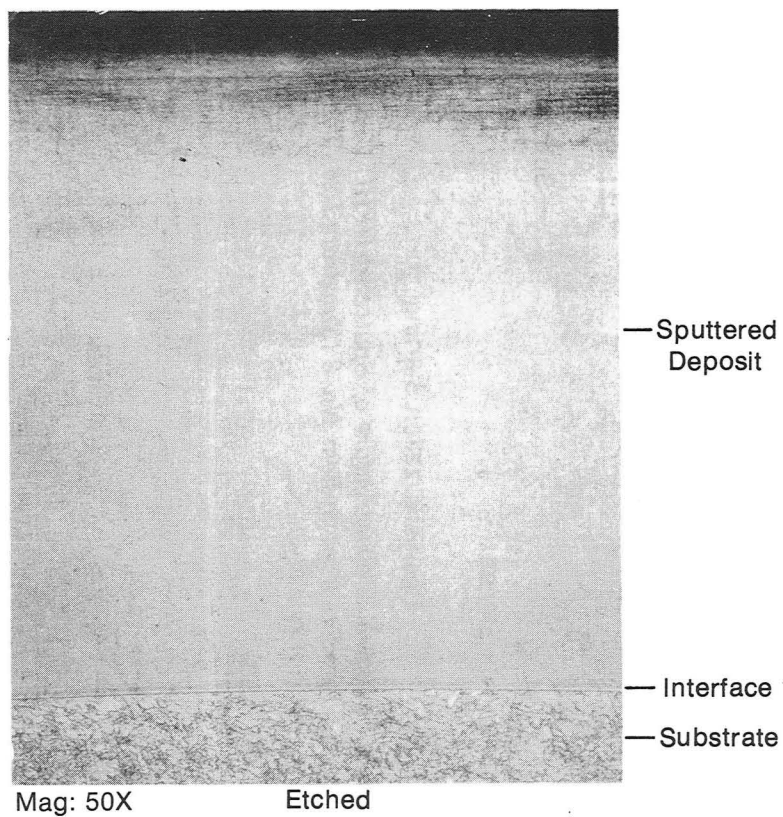


Figure 15. Typical Microstructure of Sputtered AMZIRC Copper Deposits from System No. 2 (Substrate I-29, Bottom of Section No. 3)

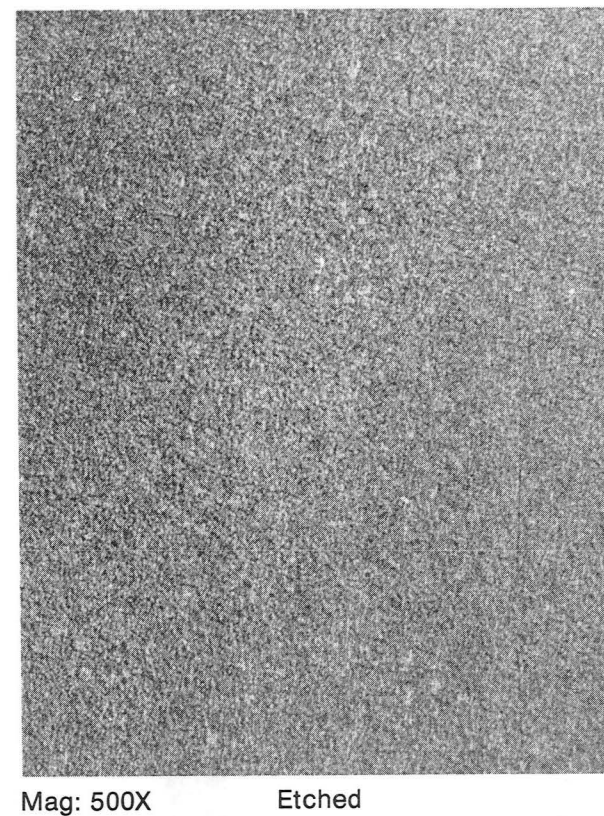


Figure 16. Typical Microstructure of Post Cathode Sputtered Deposits on Substrates I-8P, I-10P and Ends of I-13P (I-8P, Top of Section No. 1)

Deposits I-12P, I-13P, and I-14P, figure 17, exhibited a structure similar in appearance to the recrystallized structures reported by Patten, McClanahan and Johnston (Reference 9) for sputtered OFHC copper. The structures of I-12P and I-14P were typical of the entire deposit. (The inner 60% of the deposit on I-12P was fully recrystallized. Figure 17 also shows the structure near the substrate.) Substrate I-13P did not have the same structure over the entire cylinder. The structure shown on figure 17 was typical of the center of the cylinder on the high deposition rate side. No recrystallized structure was observed at the ends of the cylinder (piece No. 3, top and piece No. 1, bottom). The recrystallized structure rapidly disappeared on either side of the high rate area. The structure of I-12P, -13P and -14P may depend on other parameters such as deposition temperature and rate rather than on the substrate bias, as implied in figure 17.

An example of substrate influence on deposit structure is shown in figure 18. The grain structure of the OFHC copper substrate is completed in the deposit.

The deposit on substrate I-15 exhibited a layered structure (figure 19). The layers were more pronounced and more numerous at the top of the substrate.

2. Welding Sputtered Copper to Wrought Copper

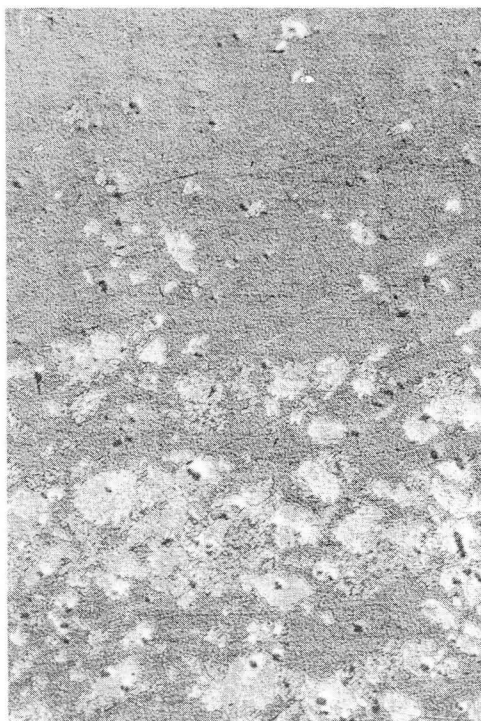
The weldability of sputtered copper was determined by X-ray inspection of the welded tensile specimens, by tensile testing, and by metallographic examination of the weld area on selected specimens. The results of X-ray inspection and tensile testing are given in tables 9 and 10 for the hollow and post cathode deposits, respectively. Photomicrographs of selected welds and pulled tensile specimens are shown in figures 20 through 28.

The photomicrographs of figure 20 show excellent quality of welds in the "OK" condition. Note that specimen I-15P-1 is heavily etched and what looks like a defect at the bottom of the weld is just a difference in surface level. Also, the apparent porosity in the sputtered AMZIRC is a zone of fine grain equiaxed structure.

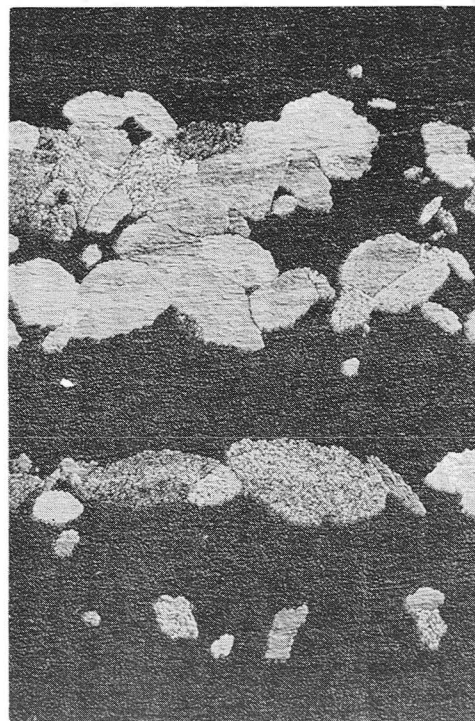
The data of table 9 seem to indicate poor weldability for the sputtered hollow cathode deposits. However, the welds of the wrought to wrought material were of the same general quality (see table 11) which strongly indicates that other mechanisms affected weldability. The welding was done in batches and the following factors may have played an important role in weld quality:

1. Uneven removal of the surface oxides by the chemical cleaning procedure used
2. Uneven regrowth of surface oxide due to variable time between cleaning and welding
3. Slight mismatch in mating the machined sections (due to tolerances in machining)
4. Different gas contaminant species in the welding chamber
5. Electron beam not centered on weld specimen joint
6. Difference in operator technique.

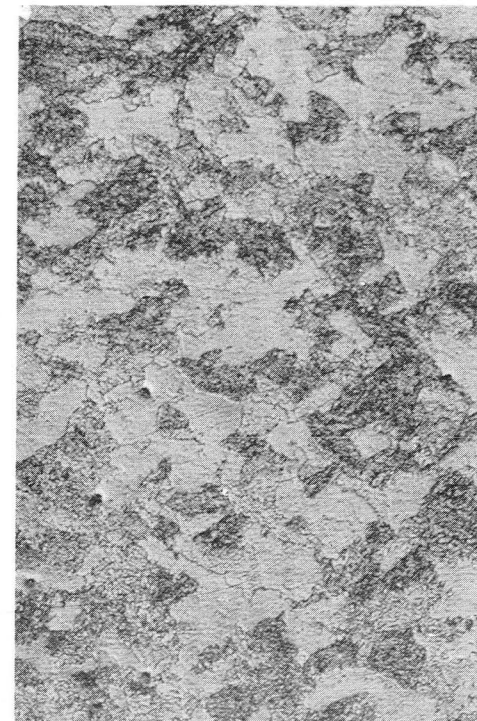
TIG welding was not investigated for welding sputtered material after welding wrought metal showed clearly that the welds (by hand) would not be reproducible.



Mag: 100X Etched
(I-13P, Top of Section No. 1)
-50V Bias

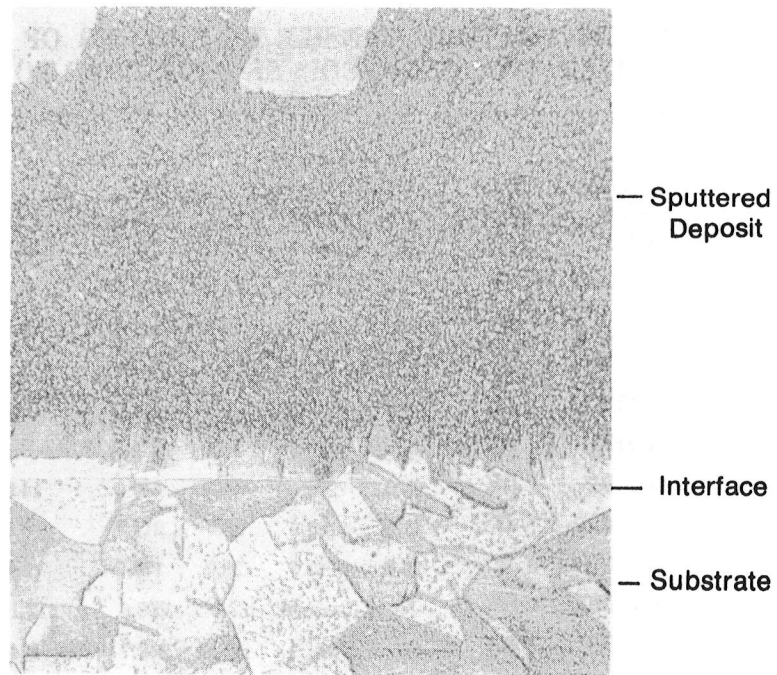


Mag: 100X Etched
(I-12P, Top of Section No. 1)
-25V Bias



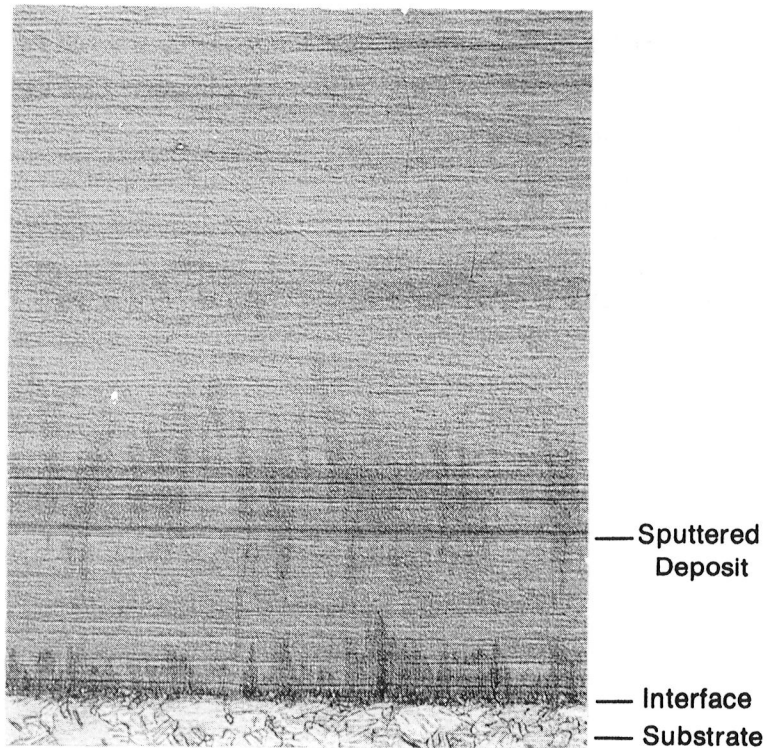
Mag: 100X Etched
(I-14P, Top of Section No. 3)
0V Bias

Figure 17. Variation of Microstructure for OFHC Copper Sputtered from Post Cathodes. The Recrystallization May Depend on Parameters Other Than Substrate Bias



Mag: 500X Etched

Figure 18. Example of Substrate Influence on Recrystallization of Sputtered OFHC Copper (I-12P, Top of Section No. 1)



Mag: 100X Etched

Figure 19. Layered Microstructure of Sputtered AMZIRC Copper (I-15P, Bottom of Section No. 3)

TABLE 9. ROOM TEMPERATURE TENSILE PROPERTIES OF HOLLOW CATHODE WELD ASSEMBLIES (SPUTTERED COPPER EB WELDED TO WROUGHT COPPER)

Specimen No.	Weld Section Sputtered Material	Weld Matching Piece	Weld Description ¹	0.2% Offset Yield Strength		Tensile Strength		Elongation (%)	Substrate Bias (volt)	Cathode Voltage (volt)	Fracture Position ²
				(ksi)	(Nm/m ²)	(ksi)	(Nm/m ²)				
I-24-1-2	OFHC	OFHC	OK	25.7	178.8	33.4	229.8	11.6	-50	-1000	WR
I-28-1-3	OFHC	OFHC	IP	10.9	74.9	18.6	128.0	5.4	-50	-1000	W
I-28-1-1	OFHC	OFHC	P	20.6	141.7	23.6	162.4	5.2	0 & -50	-1500	W
I-34-1-5	OFHC	OFHC	P	22.7	156.2	26.1	179.6	7.6	-25	-1000	W
I-29-1-4	AMZIRC	OFHC	OK	28.7	197.4	37.0	254.6	10.0	-50	-1000	WR
I-30-1-2	AMZIRC	OFHC	OK	30.2	207.8	36.7	252.5	8.1	-25	-1000	WR
I-31-1-2	AMZIRC	OFHC	OK	30.0	206.4	35.9	247.0	14.0	0	-1000	WR
I-32-1-1	AMZIRC	OFHC	IF	27.6	189.9	30.7	211.2	8.0	-25	-500	W
I-33-1-1	AMZIRC	OFHC	IF	21.6	148.6	26.0	178.9	6.2	-25 & -50	-500	W
I-24-2-2	OFHC	AMZIRC	OK	Not Obtained		37.3	256.6	17.1	-50	-1000	W
I-26-2-1	OFHC	AMZIRC	OK	29.4	202.3	36.3	249.7	8.4	-50	-1000	W
I-28-2-1	OFHC	AMZIRC	P	24.2	166.5	28.8	198.1	3.0	0 & -50	-1000	W
I-34-2-6	OFHC	AMZIRC	IF	23.1	158.9	28.8	198.1	8.9	-25	-1000	W
I-29-2-6	AMZIRC	AMZIRC	IP	17.4	119.7	17.4	119.7	3.0	-50	-1000	W
I-30-2-2	AMZIRC	AMZIRC	IP/IF	Broke	—	22.8	156.9	2.1	-25	-1000	S
I-31-2-1	AMZIRC	AMZIRC	IP	Broke	—	10.4	71.6	1.0	0	-1000	S
I-32-2-1	AMZIRC	AMZIRC	OK	20.6	141.7	20.6	141.7	4.0	-25	-500	W
I-33-2-1	AMZIRC	AMZIRC	OK	37.9	260.8	39.4	271.1	8.0	-25 & -50	-500	W

¹Condition of Weld As Read from X-ray

P = Porous

IF = Incomplete Fusion

IP = Incomplete Penetration and/or Notch

²W = In Weld Zone

S = In Sputtered Material

WR = In Wrought Material Adjacent to Weld Zone.

TABLE 10. ROOM TEMPERATURE TENSILE PROPERTIES OF POST CATHODE WELD ASSEMBLIES (SPUTTERED COPPER EB WELDED TO WROUGHT COPPER)

Specimen No.	Weld Section Sputtered Material	Weld Matching Piece	Weld Description ¹	0.2% Offset Yield Strength		Tensile Strength		Elongation (%)	Substrate Bias (volt)	Cathode Voltage (volt)	Fracture Position ²
				(ksi)	(Nm/m ²)	(ksi)	(Nm/m ²)				
I-8P-1-1	AMZIRC	OFHC	OK	31.8	218.8	38.1	262.1	10.3	-50	-500	WR
I-10P-1-1	AMZIRC	OFHC	OK	32.2	221.5	37.0	254.6	13.1	-25	-500	WR
I-11P-1-1	AMZIRC	OFHC	OK	31.0	213.3	35.3	242.9	11.5	0	-500	WR
I-15P-1-1	AMZIRC	OFHC	OK	33.0	227.0	36.9	253.9	9.9	-25	-1000	WR
I-12P-1-1	OFHC	OFHC	OK	21.9	150.7	28.1	193.3	20.1	-25	-1000	S
I-13P-1-2	OFHC	OFHC	OK	23.5	161.7	31.6	217.4	11.6	-50	-1000	W
I-14P-1-1	OFHC	OFHC	OK	18.7	128.7	26.0	178.9	29.0	0	-1000	S
I-8P-2-2	AMZIRC	AMZIRC	OK	34.1	234.6	42.6	293.1	10.1	-50	-500	W
I-10P-2-1	AMZIRC	AMZIRC	OK	32.4	222.9	37.3	256.6	7.0	-25	-500	W
I-11P-2-1	AMZIRC	AMZIRC	OK	39.1	269.0	45.5	313.0	9.1	0	-500	WR
I-15P-2-1	AMZIRC	AMZIRC	OK	37.6	258.7	42.6	293.1	8.8	-25	-1000	W
I-12P-2-1	OFHC	AMZIRC	OK	17.8	122.5	25.8	177.5	25.5	-25	-1000	S
I-13P-2-1	OFHC	AMZIRC	OK	23.7	163.1	30.1	207.1	20.4	-50	-1000	S
I-14P-2-1	OFHC	AMZIRC	OK	17.7	121.8	24.2	166.5	23.5	0	-1000	S

¹Condition of Weld As Read from X-ray

P = Porous

IF = Incomplete Fusion

IP = Incomplete Penetration and/or Notch

²W = In Weld Zone

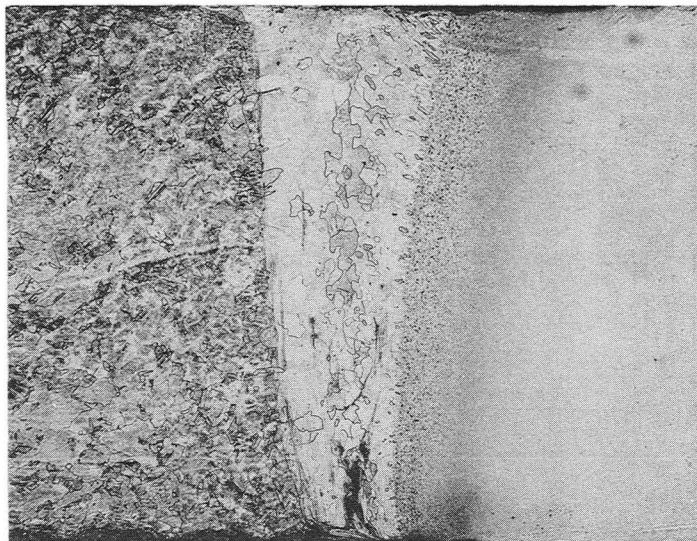
S = In Sputtered Material

WR = In Wrought Material Adjacent to Weld Zone.

Wrought
OFHC Cu

Weld
Zone

Sputtered
AMZIRC Cu



Mag: 50X

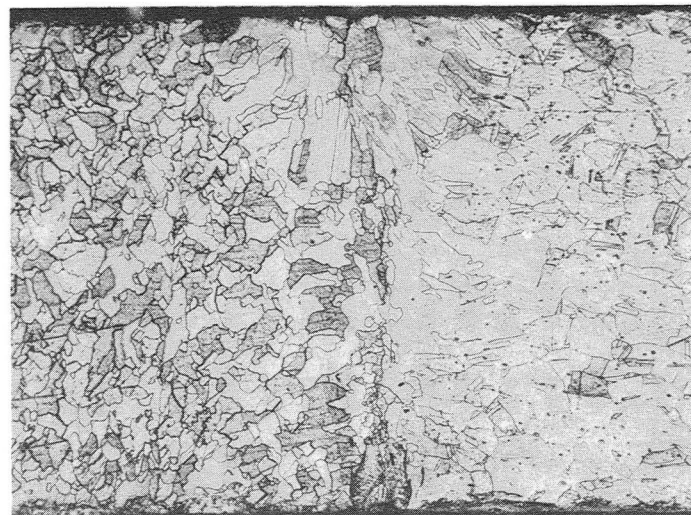
Etched

I-15-P-1

Sputtered
OFHC Cu

Weld
Zone

Wrought
AMZIRC Cu



Mag: 50X

Etched

I-14P-2

Figure 20. Typical Appearance of Electron Beam Welds in the O.K. Condition. The Material Shown in the Left Photo Has Been Over Etched To Show Weld Detail. The Sputtered AMZIRC Copper Is Not Porous nor Is the Weld Defective

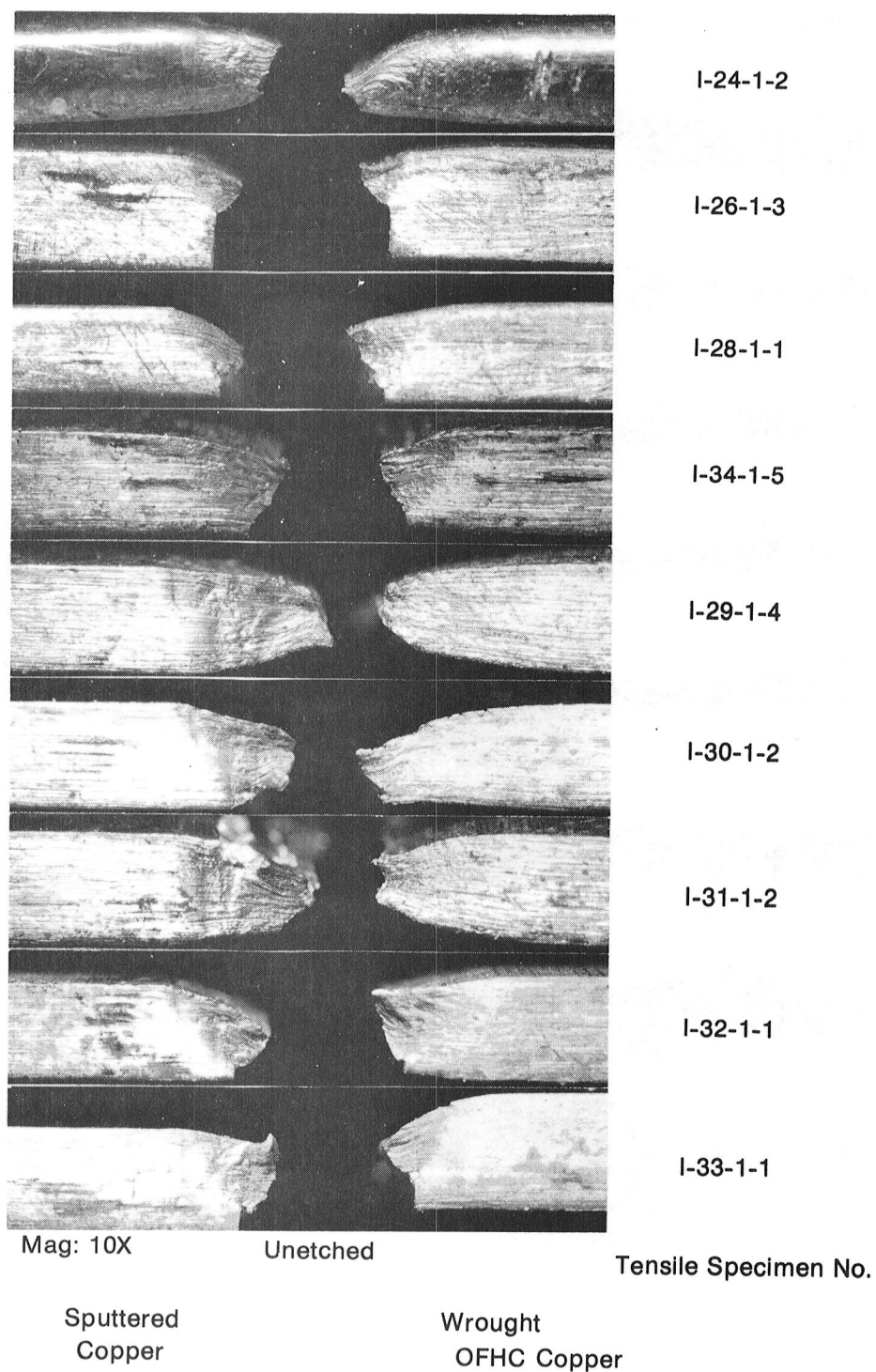


Figure 21. Side View of Tensile Specimen Fractured Ends After Room Temperature Testing. Hollow Cathode Sputtered Deposit Welded to Wrought OFHC Copper

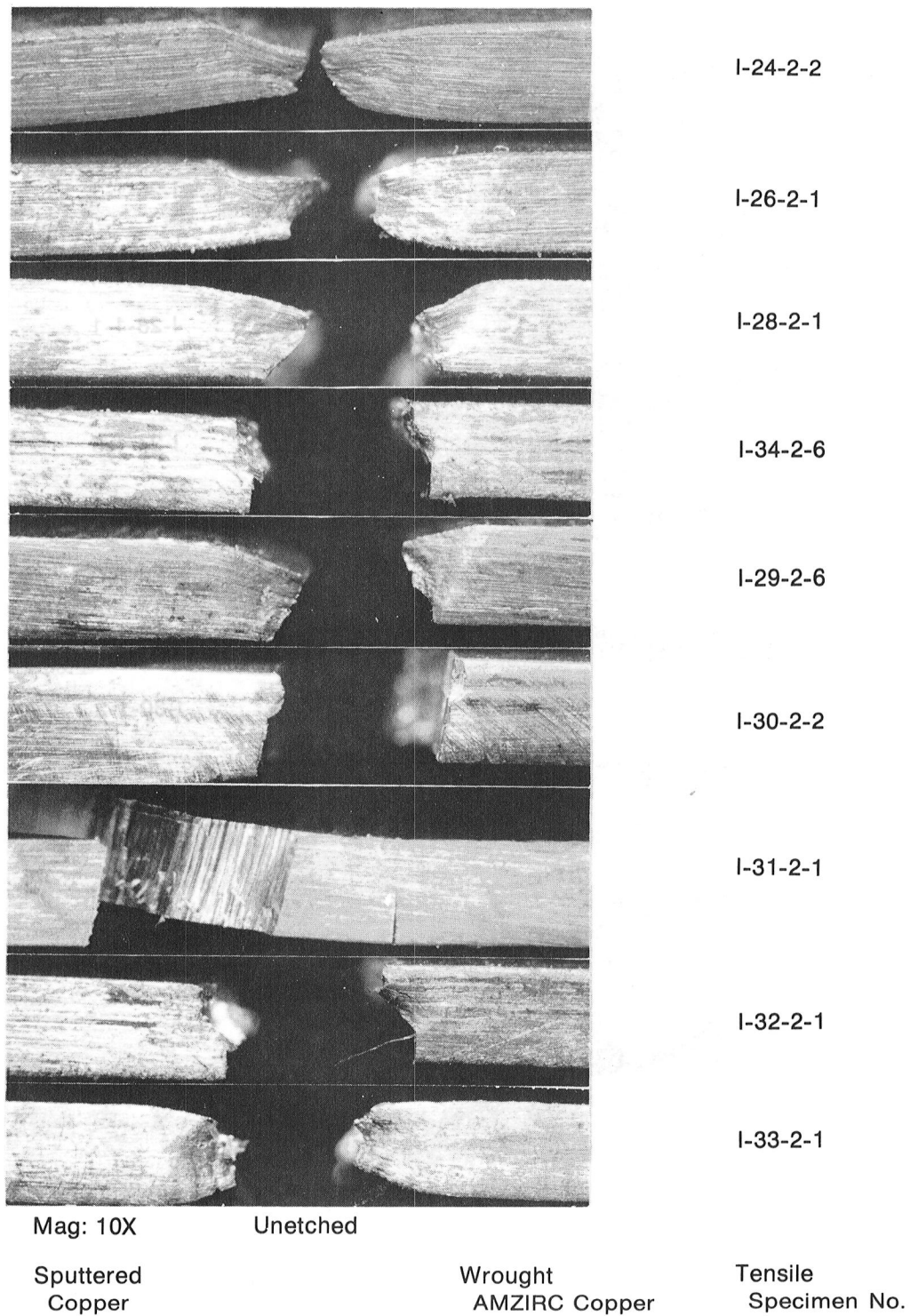


Figure 22. Side View of Tensile Specimen Fractured Ends After Room Temperature Testing. Hollow Cathode Sputtered Deposit Welded to Wrought AMZIRC Copper

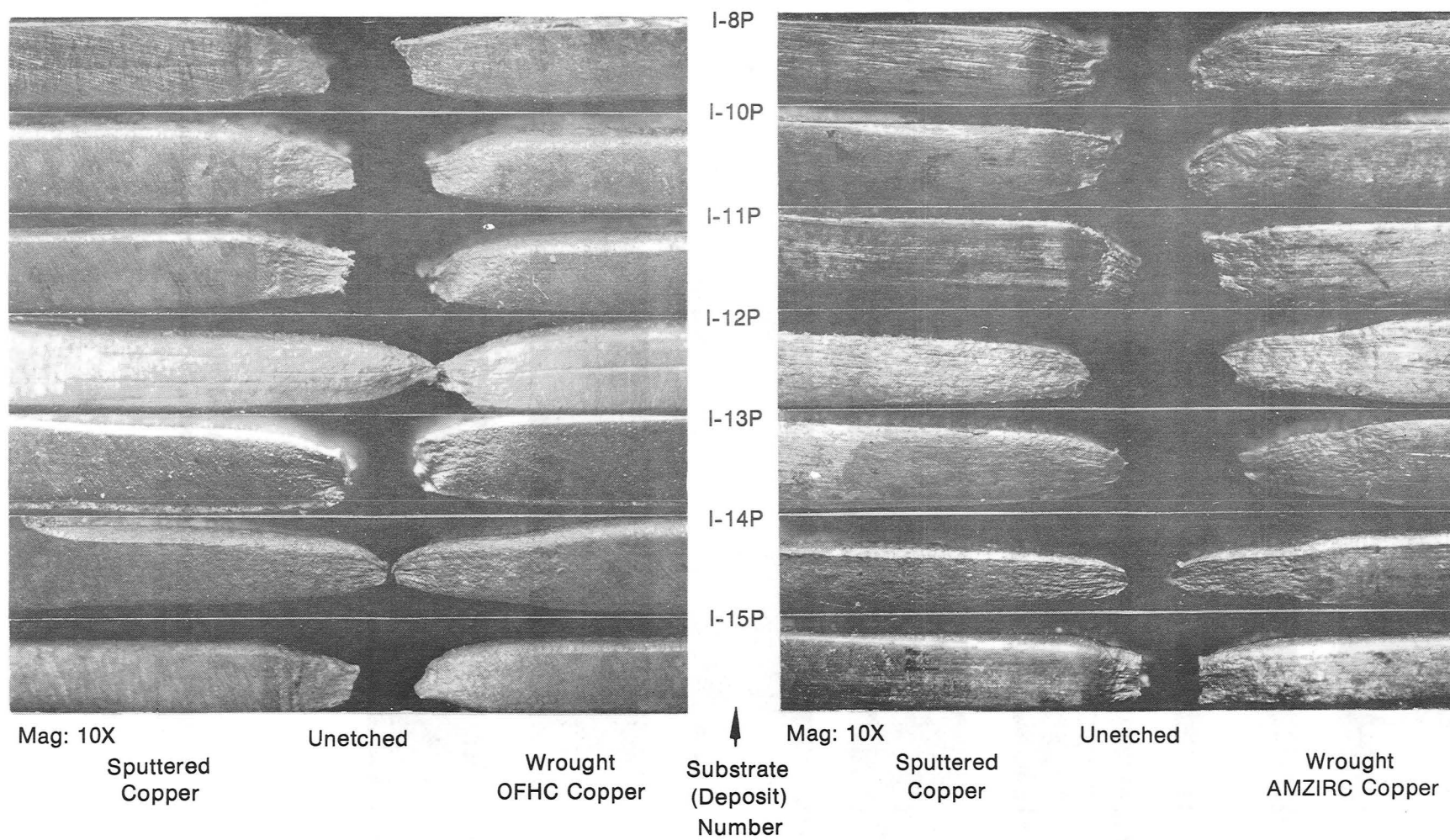
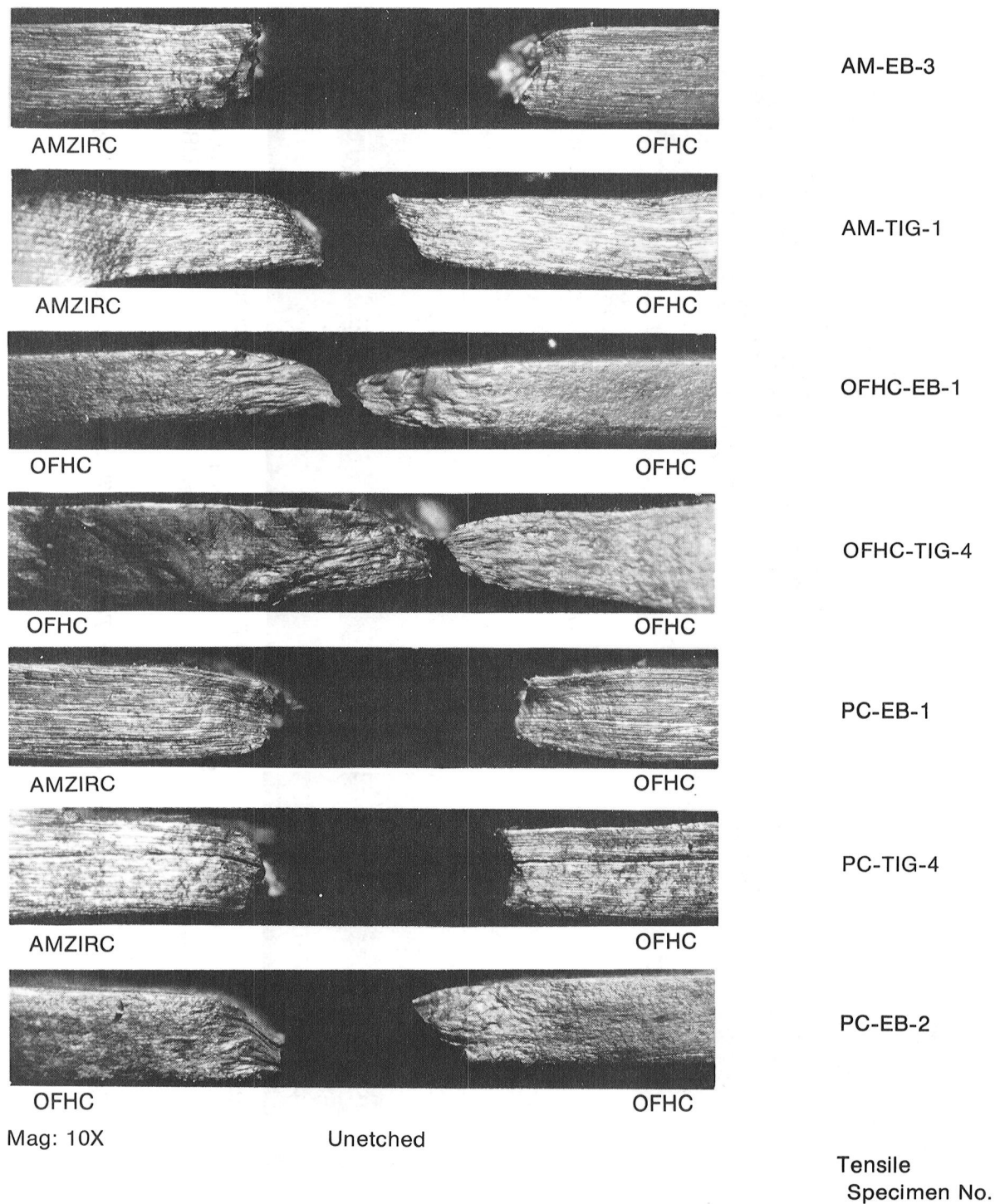


Figure 23. Side View of Tensile Specimen Fractured Ends After Room Temperature Testing Post Cathode Sputtered Deposits



*Figure 24. Side View of Tensile Specimen Fractured Ends After Room Temperature Testing Wrought Copper TIG = Tungsten Inert Gas Weld
EB = Electron Beam Weld*

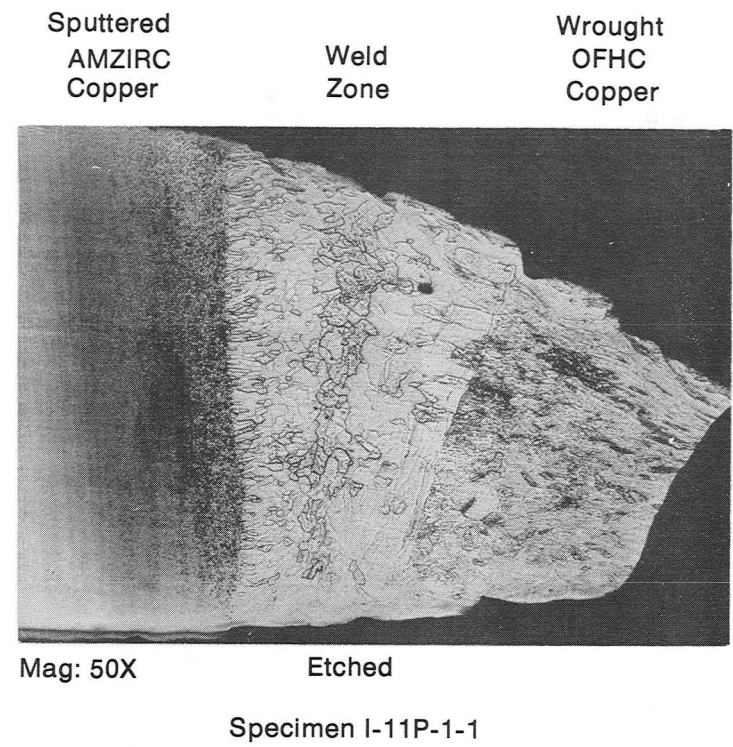
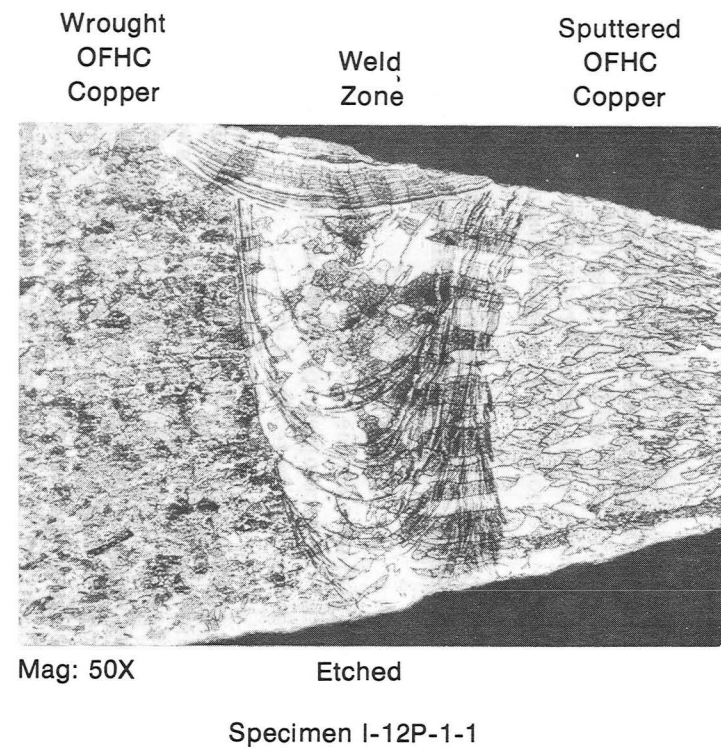


Figure 25. Examples of "O.K." Welds After Tensile Testing. Post Cathode

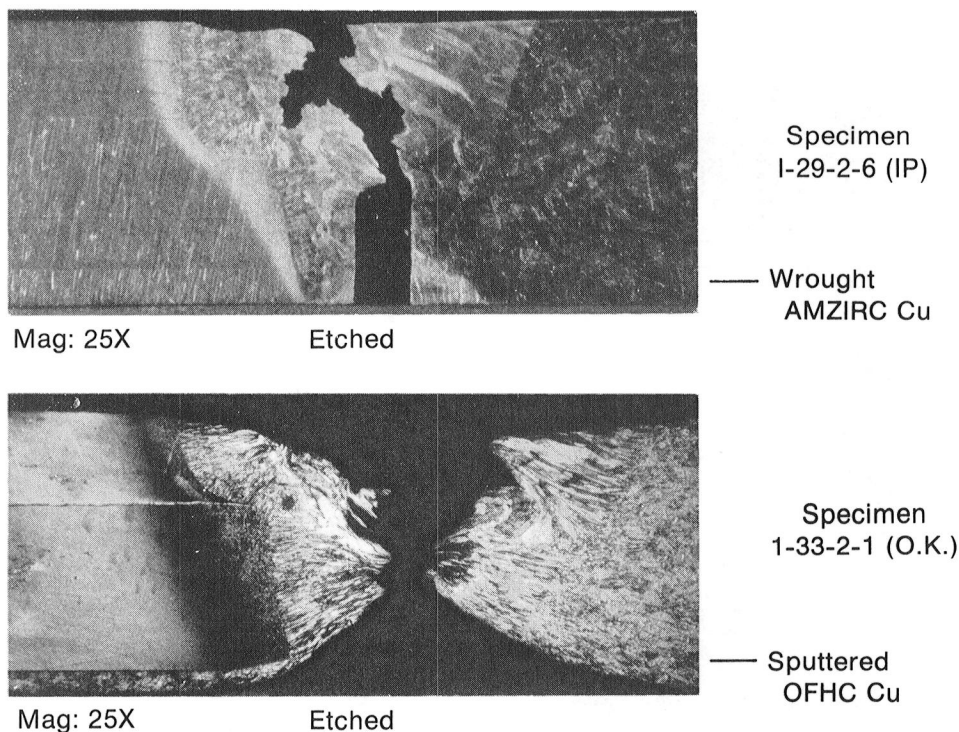


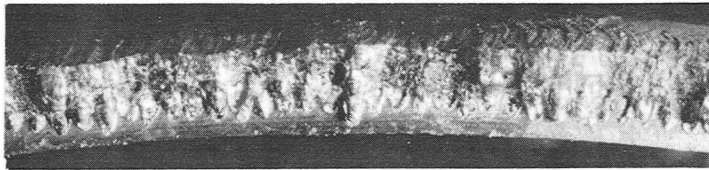
Figure 26. Examples of Welds After Tensile Testing Hollow Cathode

Although the post cathode weld assemblies yielded a higher percentage of completely welded tensile specimens, incomplete fusion and porosity was still observed on some of the specimens not tested. The increase in percentage of good welds was most likely due to increased experience in welding this cylindrical geometry and not due to sputtered material properties. The post cathode weld assemblies were welded after the hollow cathode weld assemblies.

The excellent weld quality obtained on sputtered material is shown again in figure 25, this time on the cross section of tested tensile specimens. Rupture of the specimens occurred outside of the weld zone.

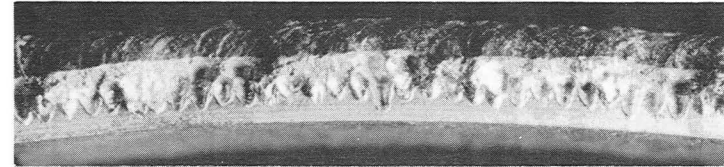
Three tensile specimens that ruptured in the weld zone are shown on figures 26 and 27. One weld was in the OK condition, one weld showed incomplete penetration and another showed porosity (and incomplete penetration which wasn't detected by X-ray).

Figure 28 illustrates open columnar growth of sputtered deposits as well as weld defects. Tensile specimens I-30-2-2 and I-31-2-1 ruptured in the sputtered material well away from the weld zone. This open columnar structure was not found in the structure survey done on the ends of the cylindrical sections, No. 1 and 3 indicating that deposit structure worsened toward the center of the substrate. Note that the cross section of specimen I-30-2-2 shows a considerably different deposition rate and growth direction for two nearly equal deposition times and nearly equal deposition parameters. The only recorded sputtering parameter changes were a 0.23 N/m^2 increase in krypton pressure and a 1.8 ma/cm^2 increase in substrate current density (see table 3).



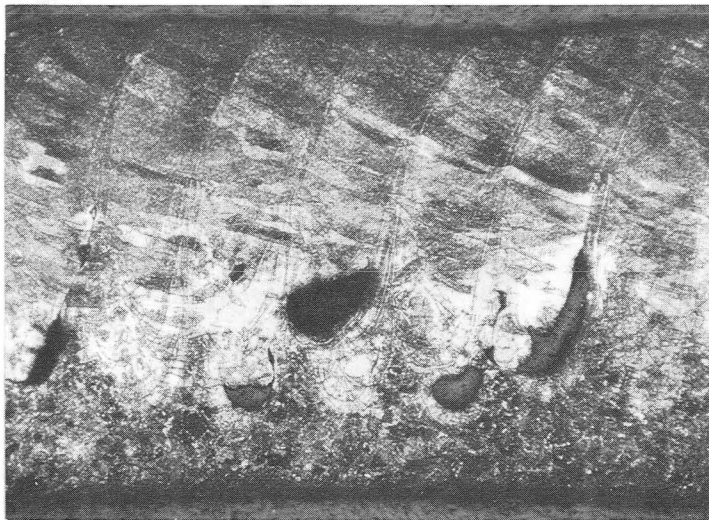
Mag: 10X Unetched

As Tested



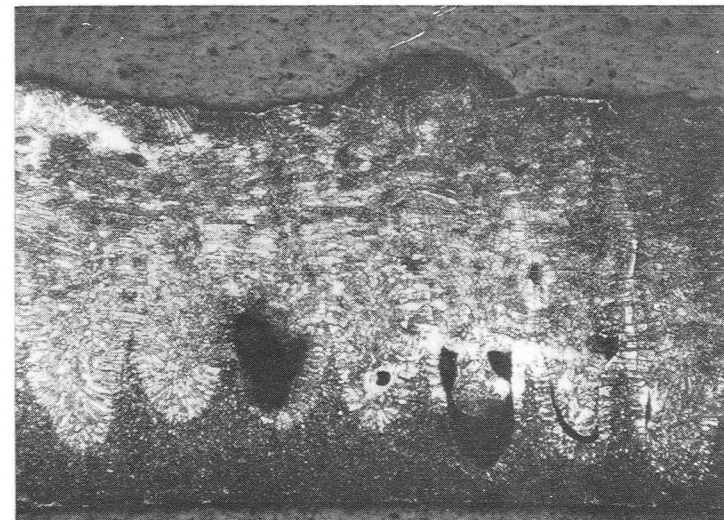
Mag: 10X Unetched

As Tested



Mag: 50X Etched

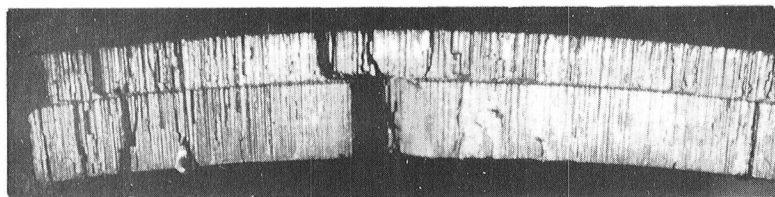
Wrought OFHC Copper



Mag: 50X Etched

Sputtered OFHC Copper

Figure 27. End View of Specimen I-28-1-1 After Tensile Testing. This Is an Example of a Porous Weld. The As-Tested Ends (10X) Were Polished Back and Over-Etched To Show Pore Formation During Welding. The Pores Formed Roughly in Equal Numbers in the Sputtered and Wrought Material

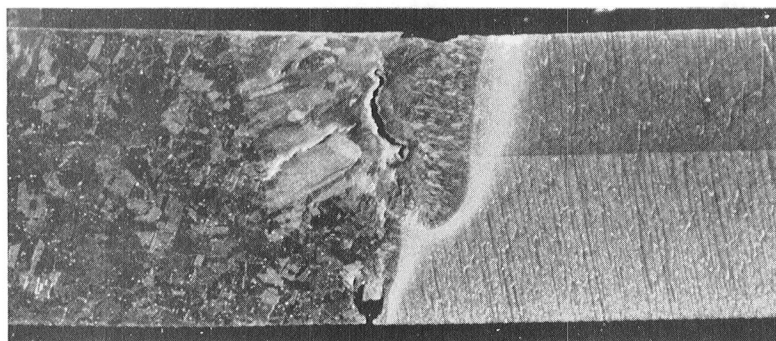


Mag: 10X

Unetched As-Pulled

End View
Sputtered Material

Specimen
I-30-2-2
(Incomplete Penetration/Fusion)

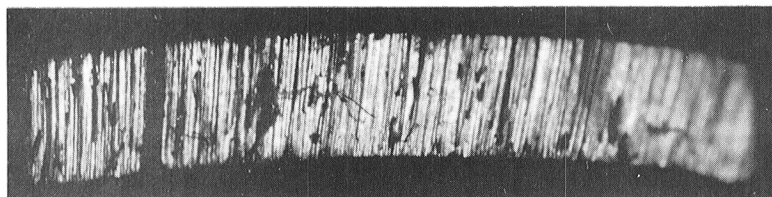


Mag: 25X

Etched

Side View
Weld Area

Sputtered AMZIRC Cu

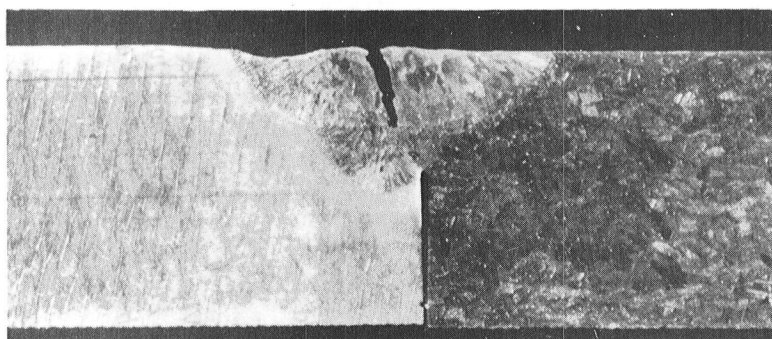


Mag: 10X

Unetched As-Pulled

End View
Sputtered Material

Specimen
I-31-2-1
(Incomplete Penetration)



Mag: 25X

Etched

Side View
Weld Area

Wrought AMZIRC Cu



Figure 28. Examples of Tensile Specimens That Ruptured in the Sputtered Material Due to Open Columnar Growth. The Fractured Ends Were Photographed and Then the Weld Areas Were Polished for the Photomicrographs

TABLE 11. ROOM TEMPERATURE TENSILE PROPERTIES OF HOLLOW AND POST CATHODE WELD ASSEMBLIES (WROUGHT COPPER EB AND TIG WELDED TO WROUGHT OFHC COPPER)

Specimen No.	Weld Section Wrought Material	Weld Matching Piece	Weld Description ¹	0.2% Offset Yield Strength		Tensile Strength		Elongation (%)	Fracture Position ²
				(ksi)	(Nm/m ²)	(ksi)	(Nm/m ²)		
Hollow Cathode Sized Tensile Specimens									
AM-EB-3	AMZIRC	OFHC	P/IF	34.4	236.7	37.3	256.6	5.9	W
AM-TIG-1	AMZIRC	OFHC	OK	28.9	198.8	36.4	250.4	30.8	WR(OFHC)
OFHC-EB-1	OFHC	OFHC	OK	16.1	110.8	32.4	222.9	36.0	W
OFHC-TIG-4	OFHC	OFHC	P	16.4	112.8	33.9	233.2	35.4	WR
Post Cathode Sized Tensile Specimens									
PC-EB-1	AMZIRC	OFHC	OK	35.8	246.3	41.6	286.2	12.2	W
PC-TIG-4	AMZIRC	OFHC	OK	18.1	124.5	24.8	170.6	10.2	WR(AMZIRC)
PC-EB-2	OFHC	OFHC	OK	20.8	143.1	32.5	223.6	17.2	W

¹Condition of Weld As Read from X-ray
P = Porous
IF = Incomplete Fusion
IP = Incomplete Penetration and/or Notch

²W = In Weld Zone
WR = In Wrought Material Adjacent to Weld Zone.

3. Thermal Treatment of Sputtered/Welded Material

Hardness determinations were made on scrap gauge sections of weld section I-15P-1. The gauge sections were cut out during the tensile machining operation. Hardness measurements for different temperature heat treatments are given in table 12. The position pattern used in the hardness survey on each test piece is given in figure 29.

The purpose of this survey was to determine if any gross structural changes would occur with temperature which would be indicative of a change in tensile strength. Within experimental error, no changes were observed. Factors influencing the hardness results were:

1. Exact location of the weld center reference line
2. Each section surveyed was from a different area around the circumference of the weld assembly which, due to the geometry effect previously discussed, could mean that starting material properties were not exactly the same. No differences in microstructure were observed for any of the sections tested.

A detailed study, which would be beyond the scope of this program, would be needed to determine heat treatment schedule needed to increase tensile strength of welded material. Low-cycle fatigue (LCF) tests would also have to be done to determine the effects of heating cycles on closeout layer to substrate bond strength. Also, welding operations would have to be studied in detail to determine effects of rate of energy input to the weld.

TABLE 12. HARDNESS (VPN) IN AND NEAR THE WELD ZONE FOR MATERIAL FROM WELD ASSEMBLY I-15P-1 AS WELDED AND AFTER HEATING FOR 1 HOUR IN ARGON AT THE INDICATED TEMPERATURE. 0.2 kg LOAD

Location	Wrought OFHC Cu			Weld Center	Sputtered AMZIRC Cu			Heat Treat Temperature	Micro No.
	-1.3 mm	-0.8 mm	-0.3 mm		+0.3 mm	+0.8 mm	+1.3 mm		
T	90	90	90	114	231	285	320	As Welded	MN39785
C	93	78	73	118	256	320	320	As Welded	MN39785
B	103	85	80	142	285	320	320	As Welded	MN39785
T	88	73	73	78	161	285	302	177°C	MN40048
C	85	80	78	78	183	320	320	177°C	MN40048
B	90	93	96	100	200	320	320	177°C	MN40048
T	88	76	71	80	106	270	285	232°C	MN40049
C	90	76	71	71	110	285	302	232°C	MN40049
B	—	88	83	83	168	302	320	232°C	MN40049
T	80	71	70	78	118	270	285	288°C	MN40205
C	85	80	62	88	114	302	302	288°C	MN40205
B	85	85	76	71	168	302	302	288°C	MN40205
T	83	66	61	88	110	256	285	316°C	MN40208
C	83	70	66	71	93	270	302	316°C	MN40208
B	96	88	76	76	114	285	302	316°C	MN40208
Second Determination ^a									
T	88	68	66	73	96	270	302	316°C	MN40208
C	83	83	66	62	88	270	302	316°C	MN40208
B	85	85	76	76	100	270	285	316°C	MN40208
T	85	68	66	76	114	285	302	343°C	MN40114
C	93	73	66	71	96	285	302	343°C	MN40114
B	85	85	71	80	118	302	320	343°C	MN40114

¹See Figure 29 for Location of Map of Test Locations.

^aMicro No. 40280 Was Re-Polished Between Determinations.

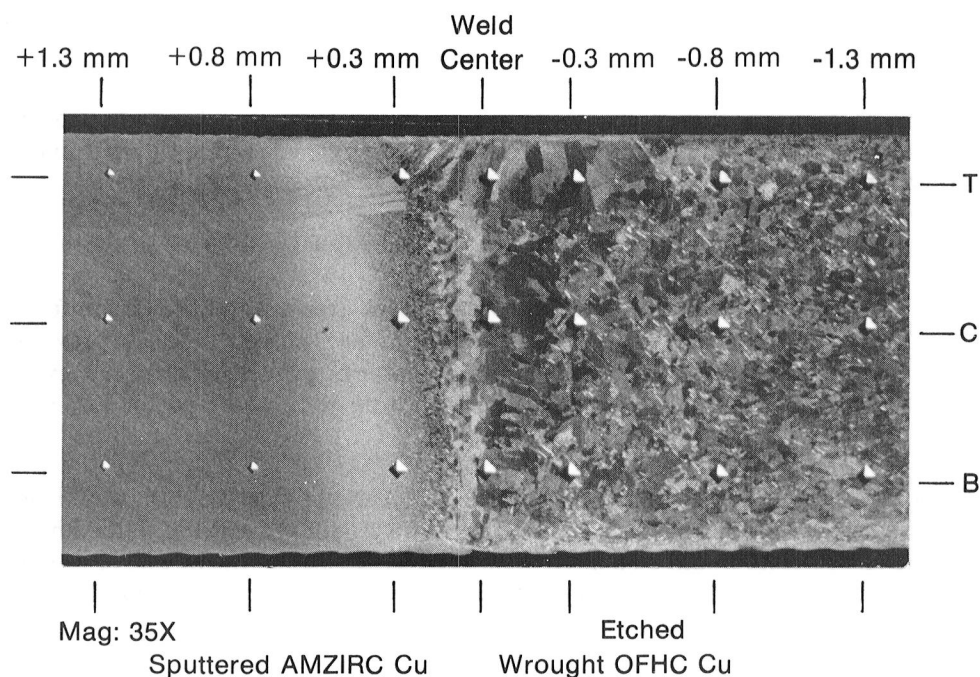


Figure 29. Hardness Test Pattern on a Piece of Weld Section from I-15P-1 Piece Heated 1 hr in Argon at 316°C. VPN. 0.2 kg Load

D. CONCLUSIONS

Excellent welds were obtained between sputtered copper and wrought copper. No special welding techniques were needed. The key to obtaining good welds is to use high-quality sputtered material which can be obtained with a fairly wide range of sputtering parameters. The most important consideration appears to be sufficient ion current density and energy to the substrate.

Increasing weld and closeout layer strength by heat treatments will be a formidable task. McClanahan, et al (Reference 3), showed that the tensile strength of sputtered AMZIRC copper (on a cold substrate) could be increased by heating to 325°C for 1 hr. However, at higher temperatures the tensile strength dropped rapidly. In the electron beam welding process the part temperature easily exceeds 325°C near the weld zone. The problem is compounded for OFHC copper where tensile strength drops immediately with any heat treatment.

Improving on the properties of welded sputtered material may be possible with a program high-energy rapid welding and/or energetic particle irradiation after welding.

Additional conclusions, which have application to Task I, are listed in the Conclusion Section of Task II.

SECTION IV

TASK II

SPOOL PIECE LINER FABRICATION

The objectives of Task II were: (1) Close out two Government-furnished (GF) slotted cylinders (NASA Drawing CC 622357) by sputtering AMZIRC copper from a hollow cathode, (2) Close out two GF slotted cylinders (NASA Drawing CC 622358) by sputtering AMZIRC copper from a post cathode, (3) Machine all of the closeout cylinders per NASA Drawing CF 621930, detail C, on both ends, and (4) Remove filler material from all channels of each closed out cylinder.

Before the GF cylinders were filled and closeout layers applied, additional study was done on the filling of grooves by sputtering from an aluminum hollow cathode. Part of this filling study was a continuation of work started in the previous program on the effect of negative substrate bias voltage on the growth characteristics of aluminum in various size grooves (substrates I-10 and I-17). The other part of this filling study was confined to groove sizes nearly equal to the groove in the GF cylinders (substrates I-18, I-19 and I-20).

Due to the technological importance of wire reinforcing in a closeout layer (Reference 4), substrates I-18, I-19 and I-20 were wrapped with half-round and round wire at the ends.

A. EQUIPMENT AND PROCEDURES

1. General Comments

The equipment and general procedures for Task II were the same as those for Task I. A summary of the substrates, cathodes and sputtering systems used is given in table 13. Note that several additional size hollow cathodes were used. Also, cathodes and substrates were sometimes turned end-for-end in an attempt to increase deposit uniformity.

2. Substrates

Substrates I-10 and I-17 were grooved as described previously in figure 8 of Reference 1. Substrate I-10 had been used in the previous program, but all previously deposited aluminum was leached out before starting depositions for this program.

Substrates I-18, I-19 and I-20 were grooved as shown in figure 30. The primary purpose of these substrates was to study aluminum growth at the ends of a truncated groove of the same size as those machined in the GF cylinders. In the previous program, grooves of this size either ran the full length of the substrate or had a gentle slope made by sawing the ends. The round bottom grooves were included to study characteristics of sputtered aluminum in a differently shaped groove. The horizontal grooves around the circumference at each end of the cylinder were included in an attempt to determine whether or not most of the flux of sputtered material was arriving from the part of the cathode beyond the end planes of the substrate. Stainless steel half-round and round wire was wrapped around these substrates at approximately 2.5 cm from the ends.

The GF cylinders were designated post cathode substrates II-4P and II-5P and hollow cathode substrates I-25, I-27, I-35 and I-36. The dimensions of these substrates are shown on figures 31 and 32. The groove widths varied from 0.165 to 0.183 cm (0.065 to 0.072 in.). Groove details varied widely, i.e., some grooves had rough walls and bottoms from machine chatter. All of the GF substrates, except II-5P, were filled completely by sputter deposition of aluminum. Substrate II-5P was filled by sputter deposition of aluminum over inlaid aluminum wire.

TABLE 13. SPUTTERING SYSTEMS AND CATHODES USED IN TASK II SUBSTRATE FILLING AND CLOSEOUT LAYER DEPOSITION

Substrate Number	Shape	Material	Cathode				Length		System Number	Lab Record	
			ID		OD					Book No.	Pages
			(cm)	(in.)	(cm)	(in.)	(cm)	(in.)			
Test Grooved Substrates (9 Combinations of Width and Depths) ^(a)											
I-10	Hollow	6061 Al	10.16	4.0	12.70	5.0	22.86	9.0	2	6	80-85
I-17	Hollow	6061 Al	10.16	4.0	12.70	5.0	22.86	9.0	2	6	88-94
Test Grooved Substrates (Vertical and Horizontal Grooves) ^(a)											
I-18	Hollow	6061 Al	10.16	4.0	12.70	5.0	22.86	9.0	2&1 ⁽¹⁾	6/10	94-97/11-13
I-19	Hollow	6061 Al	10.16	4.0	12.70	5.0	22.86	9.0	1	10	8-11
I-20	Hollow	6061 Al	10.16	4.0	12.70	5.0	22.86	9.0	1	10	13-15
Government Furnished Fully Grooved Substrates (OD Grooved) ^(a)											
I-25	Hollow	6061 Al	10.16	4.0	12.70	5.0	22.86	9.0	1	10	21-55
I-27	Hollow	6061 Al	10.16	4.0	12.70	5.0	22.86	9.0	1	10	23-52
I-35	Hollow	6061 Al	10.16	4.0	12.70	5.0	22.86	9.0	1&2 ⁽²⁾	10/12	35-42/2-7
I-36	Hollow	6061 Al	12.70	5.0	17.78	7.0	22.86	9.0	3	8	21-34
Government Furnished Fully Grooved Substrate (ID Grooved) ^(a)											
II-4P	Post	1100 Al	0.95	0.375	3.81	1.5	17.15 ⁽³⁾	6.75	2	6/12	137-152/1-2
II-5P	Post	1100 Al	0.95	0.375	3.81	1.5	17.15	6.75	2	12	15-20
Closeout Deposition on Government Furnished Substrates											
I-25	Hollow	AMZIRC	10.80 ⁽⁴⁾	4.25	12.70	5.0	22.86	9.0	1	10	58-72
I-27	Hollow	AMZIRC	10.80	4.25	12.70	5.0	22.86	9.0	1	10	73-79
I-36	Hollow	AMZIRC	12.70	5.0	17.78	7.0	22.86	9.0	3	8	34-41
II-4P	Shaped Post ⁽⁵⁾	OFHC	0.95	0.375	3.81	1.5	22.22	8.75	2	12	8-15
II-5P	Shaped Post	AMZIRC	0.95	0.375	3.81	1.5	22.22	8.75	2	12	21-32

⁽¹⁾First deposition cycle done in System No. 2. Work finished in System No. 1.

⁽²⁾First 4 deposition cycles done in System No. 1. Last 2 cycles done in System No. 2.

⁽³⁾First Deposition cycle done with 22.22 cm long post cathode.

⁽⁴⁾First of three cathodes used. See table 19.

⁽⁵⁾Refer to figure 1 for size and shape.

⁽⁶⁾Shown in figure 8, Reference 1.

⁽⁷⁾Shown in figure 31 and wire wrapped as explained in text.

⁽⁸⁾Shown in figure 31.

⁽⁹⁾Shown in figure 32.

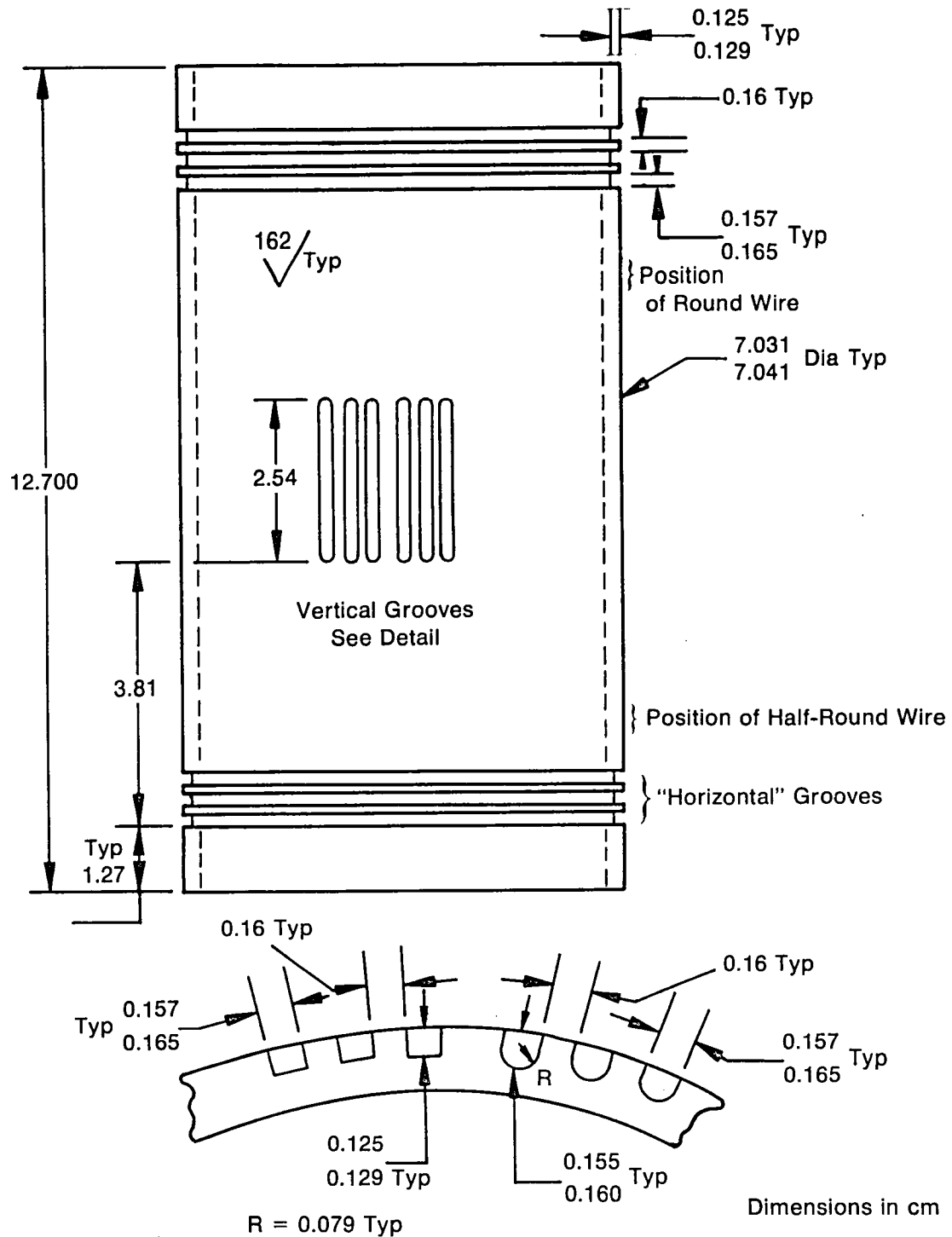


Figure 30. Machining Details for Test Grooved Hollow Cathode Substrates (I-18, I-19 and I-20)

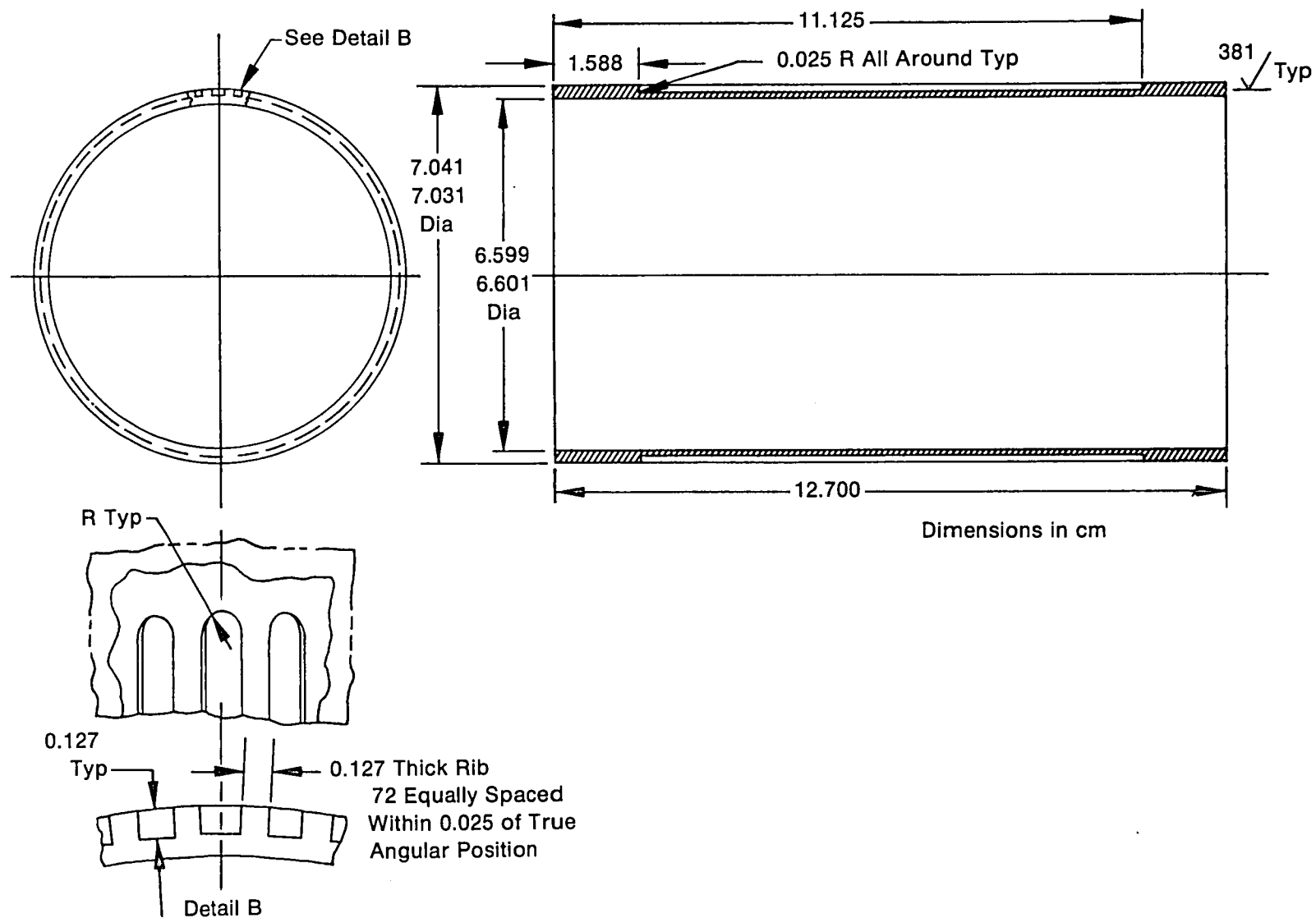


Figure 31. Slotted GF Cylindrical Specimen for Hollow Cathode Closeout Redrawn from NASA CC622357

Figure 32. Slotted GF Cylindrical Specimen for Post Cathode Closeout Redrawn from NASA CC622358

The post and hollow cathode burst test specimens were filled simultaneously with substrates II-4P and I-27, respectively. These specimens replaced the regular end caps used to contain the substrates on their mandrels.

3. Substrate Cleaning

Prior to the first aluminum deposition each substrate was chemically and sputter cleaned in the same manner as the smooth substrates of Task I. Sputter cleaning parameters are given in tables 14 and 15. On the substrates where the aluminum was machined back between deposition cycles, a more rigorous cleaning was done to remove cutting fluids. This cleaning consisted of ultrasonic agitation for 2 hr in freon and ½ hr in ethyl alcohol after a preliminary chlorinated cleanser scrub. Sputter cleaning was done with a -50v bias on the substrate for a time sufficiently long to achieve outgassing as determined by discharge and pressure stability. Usually, several cycles of sputter cleaning and system pumpdown were necessary.

The aluminum filled GF substrates were hand sanded lengthwise with No. 600 grit SiC paper after the final machining off of the sputter aluminum and then chemically cleaned like the smooth substrates for Task I. Sputter cleaning cycles are given in table 16. When a GF substrate was machined before the completion of a closeout layer, the sputtered copper was also hand sanded, to partially remove machine marks, and then cleaned in the usual manner.

B. SPUTTERED ALUMINUM AND SPUTTERED COPPER CLOSEOUT LAYER DEPOSITION PARAMETERS

The sputtering parameters used in Task II depositions are given in tables 17 through 19, respectively, for filling test grooved substrates, filling GF substrates and for closing out GF substrates. These data were obtained from the data recorded during the depositions as explained for Task I. Additional explanatory notes have been included on the tables.

C. RESULTS

Task II results will be discussed in the following order:

1. Characteristics of sputtered aluminum in test grooves and over wires
2. Filling GF substrates with sputtered aluminum
3. Copper closeout layer deposition.

1. Characteristics of Sputtered Aluminum in Test Grooves and Over Wires

In the previous program (Reference 1) grooves in cylindrical substrates were filled with aluminum sputtered from post and hollow cathodes. Although grooves in a limited size range were completely filled, the role played by the substrate bias was not understood. The bias voltage was expected to accelerate a sufficient number of ions to the top of the groove walls to keep the deposit from bridging over and shielding the bottom of the groove from the sputtered aluminum flux. In addition, by the mechanism of "forward sputtering," the aluminum resputtered from the groove walls was expected to greatly increase groove filling rate.

The ions accelerated to the groove walls by the bias potential were either deficient in density (number of ions per square centimeter) or in energy (electron volts) or in both. In the triode discharges used in these sputtering systems an increase in ion density is accomplished by increasing filament temperature or increasing anode voltage or both. Either way of increasing ion density increases the thermal load to the discharge system. Also, the ion density to the cathode is increased resulting in an increased sputtering rate which may mask the effect of bias on the

TABLE 14. SUMMARY OF SPUTTER CLEANING PARAMETERS FOR TEST GROOVED SUBSTRATES PRIOR TO FIRST ALUMINUM DEPOSITIONS

Substrate Number	Substrate		Cathode		Time		Pressure		Anode	
	Current (ma/cm ²)	Voltage (volts)	Current (ma/cm ²)	Voltage (volts)	(min)	(ks)	(μ) ¹	(N/m ²)	Current (amps)	Voltage (volts)
I-10	6.2	-25	5.8	-25	5	0.30	9.1	1.18	25.0	44
	6.1	-50	5.5	-50	30	1.80	9.0	1.17	24.4	40
I-17	9.0	-25	7.9	-25	5	0.30	9.4	1.22	36.5	45
	9.0	-50	9.1	-50	30	1.80	10.9	1.42	39.0	40
I-18	6.8	-25	6.8	-25	5	0.30	7.8	1.01	28.5	45
	7.3	-50	7.4	-50	14	0.84	7.9	1.03	30.0	45
	7.5	-50	7.7	-50	30	1.80	7.0	0.91	30.0	45
I-19	3.2	-25	2.5	-25	5	0.30	3.7	0.48	18.5	34
	3.9	-50	3.2	-50	30	1.80	2.7	0.35	21.8	34
	4.6	-50	4.0	-50	20	1.20	3.6	0.47	24.3	34
I-20	4.4	-25	3.5	-25	5	0.30	5.5	0.72	25.0	30
	4.7	-50	3.9	-50	30	1.80	4.6	0.60	26.0	30

¹1μ = 1 micron = 1 × 10⁻³ mm Hg = 0.1333 N/m²

TABLE 15. SUMMARY OF SPUTTER CLEANING PARAMETERS FOR GF SUBSTRATES PRIOR TO FIRST ALUMINUM DEPOSITIONS

Substrate Number	Substrate		Cathode		Time		Pressure		Anode	
	Current (ma/cm ²)	Voltage (volts)	Current (ma/cm ²)	Voltage (volts)	(min)	(ks)	(μ) ¹	(N/m ²)	Current (amps)	Voltage (volts)
I-25	2.0	-25	4.2	-25	5	0.3	3.1	0.40	16.8	35
	2.5	-50	5.6	-50	30	1.8	2.5	0.32	20.8	30
I-27	2.2	-25	5.3	-25	5	0.3	4.0	0.52	20.3	30
	3.5	-50	8.2	-50	30	1.8	4.1	0.53	26.4	40
	3.5	-100	7.9	-50	5	0.3	4.5	0.58	26.0	40
	3.4	-50	8.0	-50	15	0.9	4.1	0.53	26.3	40
I-35	3.9	-25	9.8	-25	5	0.3	23.0	2.99	30.0	34
	4.4	-50	10.8	-50	5	0.3	25.4	3.30	31.0	30
I-36	1.3	-25	3.9	-25	5	0.3	6.5	0.84	49.0	30
	1.5	-25	4.3	-25	5	0.3	6.7	0.87	50.0	30
	1.7	-50	5.3	-50	30	1.8	6.7	0.87	53.0	35
II-4P	8.3	-25	11.4	-25	5	0.3	11.6	1.51	40.0	46
	9.3	-50	12.7	-50	8	0.5	5.9	0.77	42.0	46
	8.5	-50	11.8	-50	30	1.8	11.6	1.51	41.4	46
	9.0	-50	12.5	-50	10	0.6	11.9	1.55	41.5	46
	9.0	-50	12.2	-50	5	0.3	11.6	1.51	40.0	46
II-5P	7.0	-25	10.9	-25	5	0.3	4.2	0.55	30.0	45
	6.8	-50	10.2	-50	30	1.8	5.5	0.72	28.9	45
	7.1	-50	11.4	-50	10	0.6	3.1	0.40	28.7	45
	8.0	-50	12.7	-50	5	0.3	3.7	0.48	31.0	45

¹1μ = 1 micron = 1 × 10⁻³ mm Hg = 0.1333 N/m²

TABLE 16. SUMMARY OF SPUTTER CLEANING PARAMETERS FOR FILLED GF SUBSTRATES PRIOR TO START OF COPPER CLOSEOUT LAYER DEPOSITION

Substrate Number	Substrate		Cathode		Time		Pressure		Anode	
	Current (ma/cm ²)	Voltage (volts)	Current (ma/cm ²)	Voltage (volts)	(min)	(ks)	(μ)	(N/m ²)	Current (amps)	Voltage (volts)
I-25	3.3	-25	2.8	-25	5	0.3	4.2	0.55	18.8	34
	3.9	-50	3.2	-50	5	0.3	5.0	0.65	20.3	34
I-27	3.5	-25	2.5	-25	5	0.3	5.0	0.65	18.5	35
	4.5	-50	3.4	-25	30	1.8	5.1	0.66	22.6	40
	4.5	-50	3.3	-50	5	0.3	5.0	0.65	22.2	40
I-36	2.9	-25	1.6	-25	5	0.3	4.1	0.53	43.5	35
	3.0	-50	1.6	-50	15	0.9	4.2	0.55	44.0	35
	3.0	-50	1.7	-50	15	0.9	4.6	0.60	45.7	35
	3.1	-50	1.6	-25	30	1.8	4.5	0.58	44.5	35
	3.0	-50	1.6	-25	5	0.3	4.3	0.56	43.0	35
II-4P	5.3	-25	8.8	-25	30	1.8	3.4	0.44	25.0	40
	5.3	-50	9.0	-25	5	0.3	3.0	0.39	25.8	40
II-5P	7.7	-25	9.0	-25	5	0.3	4.1	0.53	31.5	44.5
	7.7	-50	10.0	-25	5	0.3	4.9	0.64	33.5	45
	7.0	-50	9.2	-25	30	1.8	4.5	0.58	32.8	45
	7.7	-50	9.7	-25	5	0.3	3.4	0.44	33.5	45
	8.0	-50	9.9	-25	5	0.3	3.6	0.47	36.5	45
	8.0	-50	9.4	-25	20	1.2	3.5	0.46	33.5	45
	8.7	-50	11.7	-25	5	0.3	4.5	0.58	38.5	44.5

TABLE 17. SUMMARY OF DEPOSITION PARAMETERS FOR FILLING TEST
GROOVED AND WIRE WRAPPED CYLINDRICAL SUBSTRATES BY
SPUTTERING FROM ALUMINUM HOLLOW CATHODES

Substrate Number (Cycle No.) ¹	Substrate		Cathode		Anode		Deposition Time		Pressure	
	Current (ma/cm ²)	Voltage (volts)	Current (ma/cm ²)	Voltage (volts)	Current (amps)	Voltage (volts)	(hr)	(ks)	(μ)	(N/m ²)
I-10 (1)	5.7	-50	5.2	-500	21.3	40.0	11.5	41.4	3.8	0.49
	5.7	-50	5.1	-500	20.0	40.0	17.0	61.2	4.0	0.52
	5.4	-50	5.0	-500	20.0	40.0	18.0	64.8	3.2	0.42
	5.7	-50	5.1	-500	20.0	40.0	15.0	54.0	3.2	0.42
	8.7	-50	8.1	-500	31.8	40.0	20.0	72.0	3.7	0.48
	9.5	-50	8.8	-500	34.1	40.0	16.0	57.6	4.0	0.52
I-17 (1)	7.9	-75	7.9	-500	29.8	40.0	15.0	54.0	5.3	0.69
	7.9	-75	7.9	-500	29.8	40.0	20.0	72.0	4.4	0.57
	8.2	-75	8.2	-500	31.0	40.0	20.0	72.0	4.1	0.53
	8.2	-75	7.9	-500	30.0	40.0	8.0	28.8	4.1	0.53
	8.4	-75	6.2	-500	29.8	40.0	2.5	9.0	4.6	0.60
	8.4	-75	6.1	-500	29.8	40.0	1.0	3.6	2.9	0.38
	8.4	-75	5.9	-500	31.0	40.0	1.0	3.6	3.7	0.48
	7.3	-75	5.4	-500	30.0	40.0	4.0	14.4	4.0	0.52
	7.1	-75	5.4	-500	31.5	40.0	7.5	27.0	2.9	0.38
	6.8	-75	5.1	-500	29.7	40.0	10.0	36.0	3.0	0.39
	6.8	-125	7.1	-500	28.0	40.0	1.5	5.4	4.1	0.53
	6.0	-125	5.9	-500	24.6	40.0	13.0	46.8	3.8	0.49
I-18 (1)	6.2	-125	5.9	-500	24.7	40.0	3.0	10.8	4.8	0.62
	6.0	-125	6.2	-500	23.5	40.0	2.0	7.2	4.0	0.52
	6.0	-125	5.8	-500	23.0	40.0	2.0	7.2	4.0	0.52
	6.0	-125	5.9	-500	22.0	40.0	3.0	10.8	3.7	0.48
	5.0	-125	5.7	-500	20.6	40.0	12.0	43.2	4.2	0.55
	6.0	-125	6.1	-500	22.0	40.0	10.5	37.8	4.8	0.62
	5.7	-125	4.8	-500	27.9	30.0	10.0	36.0	3.9	0.51
	6.2	-125	5.4	-500	29.8	30.0	16.0	57.6	4.1	0.53
	6.0	-125	5.2	-500	29.0	30.0	15.0	54.0	4.5	0.58
	6.0	-125	5.0	-500	28.2	30.0	24.0	86.4	4.1	0.53
	5.2	-100	4.5	-500	25.6	34.0	3.0	10.8	3.3	0.43
	5.7	-100	5.0	-500	28.6	30.0	13.0	46.8	4.7	0.61
I-19 (1)	5.7	-100	4.8	-500	28.0	30.0	11.0	39.6	4.7	0.61
	6.0	-100	5.1	-500	28.4	30.0	10.0	36.0	4.6	0.60
	6.2	-100	5.1	-500	29.4	30.0	20.0	72.0	4.3	0.56
	6.2	-100	5.2	-500	29.7	30.0	8.0	28.8	4.3	0.56
	5.7	-125	5.1	-1000	29.1	30.0	14.0	50.4	4.7	0.61
I-20 (1)	6.0	-125	5.1	-1000	29.4	30.0	24.0	86.4	4.7	0.61
	6.0	-125	4.8	-1000	29.2	30.0	22.0	79.2	4.6	0.60
	5.7	-125	4.4	-1000	27.3	30.0	12.0	43.2	4.2	0.55

¹ Aluminum machined back to substrate OD at end of each cycle and cylinder orientation changed end for end. Note that the wire wrapped substrates I-18, I-19 and I-20 were not machined after deposition over the wires.

² Stainless steel wire wrapped on I-18 before start of deposition cycle No. 2.

TABLE 18. SUMMARY OF DEPOSITION PARAMETERS FOR FILLING GF FULLY GROOVED POST AND HOLLOW CATHODE SUBSTRATES WITH SPUTTERED ALUMINUM

Substrate Number (Cycle No.) ¹	Substrate		Cathode		Anode		Deposition Time		Pressure	
	Current (ma/cm ²)	Voltage (volts)	Current (ma/cm ²)	Voltage (volts)	Current (amps)	Voltage (volts)	(hr)	(ks)	(μ)	(N/m ²)
Hollow Cathode										
I-25 (1)	7.6	-100	5.5	-1000	27.8	32.0	34.0	122.4	20.1	2.61
(2)	7.3	-100	5.2	-1000	23.5	32.0	50.0	180.0	18.2	2.37
(3)	7.3	-100	5.4	-1000	23.2	32.0	51.0	183.6	18.2	2.37
(4)	7.3	-200	5.5	-1000	25.2	32.0	33.0	118.8	17.1	2.22
(5)	7.1	-200	5.1	-1000	28.1	32.3	21.0	75.6	16.8	2.18
(6)	7.1	-200	4.9	-1000	27.2	32.0	32.0	115.2	16.1	2.09
(6)	8.4	-200	5.8	-1000	29.1	30.1	16.0	57.6	19.7	2.56
I-27 (1)	7.3	-50	4.8	-1000	27.6	40.0	21.0	75.6	4.3	0.56
(1) ^a	6.6	-50	4.0	-1000	24.8	40.0	73.2	363.5	4.4	0.57
(2)	5.4	-50	4.5	-1000	25.2	40.0	56.0	201.6	3.4	0.44
(3)	7.9	-60	5.8	-1000	31.9	26.3	73.0	262.8	23.5	3.06
(4)	6.8	-200	5.0	-1000	31.6	26.8	33.7	121.3	17.7	2.30
(5)	7.3	-200	5.1	-1000	32.0	26.2	32.5	117.0	19.1	2.48
I-35 (1)	9.0	-50	6.2	-1000	28.6	24.2	75.0	270.0	21.4	2.78
(2)	8.4	-50	6.1	-1000	32.0	25.2	75.5	271.8	19.7	2.56
(3)	7.9	-100	5.8	-1000	32.0	26.6	35.5	127.8	20.6	2.68
(4)	7.1	-100	5.1	-1000	32.0	27.2	27.0	97.2	20.1	2.61
(5)	6.8	-50	6.9	-500	33.7	38.6	26.5	95.4	12.2	1.59
(6)	6.0	-50	7.6	-500	25.6	40.0	48.0	172.8	4.0	0.52
I-36 (1)	4.1	-75	2.4	-1000	51.9	35.0	65.0	234.0	4.2	0.55
(1)	4.2	-75	2.4	-1000	42.1 ^c	35.0	36.3	130.7	4.5	0.59
(2)	4.1	-100	2.3	-1000	47.3	36.7	54.0	194.4	4.6	0.60
(3)	3.7	-100	2.0	-1000	50.9	35.0	51.5	185.4	4.0	0.52
Post Cathode										
II-4P (1)	7.5	-50	9.9	-1000	31.3	40.0	41.0	147.6	11.2	1.46
(2) ^a	7.0	-50	10.2	-1000	34.0	40.0	38.0	136.8	13.1	1.70
(3)	8.0	-50	10.9	-1000	34.0	40.0	64.5	232.2	11.4	1.48
(4)	8.0	-50	11.2	-1000	33.8	40.0	65.0	234.0	11.1	1.44
(5)	7.5	-50	9.3	-1000	34.0	40.0	45.0	162.0	12.9	1.72
(6)	6.4	-50	10.3	-500	33.9	40.0	65.0	234.0	14.6	1.90
II-5P (1)	8.4	-50	10.0	-500	32.0	44.5	18.0	64.8	4.3	0.56
(2)	8.2	-25	9.1	-500	34.5	45.0	10.0	36.0	2.9	0.38

¹ Aluminum machined back to substrate ID at end of each cycle and substrate turned end for end on each installation.

^a Substrate rotated 180 deg before resumption of this part of the deposition cycle.

^b Cathode rotated 180 deg before start of deposition cycle.

^c Reduced current due to open circuit in top filaments for 56.9 ks.

TABLE 19. SUMMARY OF TASK II POST AND HOLLOW CATHODE
CLOSEOUT LAYER DEPOSITION PARAMETERS

Substrate Number (Cycle No.) ¹	Substrate		Cathode		Anode		Deposition Time		Pressure	
	Current (ma/cm ²)	Voltage (volts)	Current (ma/cm ²)	Voltage (volts)	Current (amps)	Voltage (volts)	(hr)	(kg)	(μ)	(N/m ²)
Hollow Cathode										
I-25 (1)	5.0	-50	4.1 ^a	-1000	33.7	28.9	76	273.6	3.1	0.40
(1) ^a	4.6	-50	3.7	-1000	34.0	25.1	37	133.2	3.1	0.40
(1)	5.4	-50	3.9	-1000	34.0	27.0	48	172.8	3.6	0.47
(1)	6.6	-50	4.8 ^a	-1000	34.0	28.2	32	115.2	5.0	0.65
(1) ^a	5.8	-50	4.2	-1000	34.0	25.5	129	464.4	3.5	0.46
(2)	5.3	-50	3.3 ^a	-1000	37.0	26.3	127	457.2	5.4	0.70
(2) ^{3,4}	6.2	-50	3.9	-1000	40.0	28.8	37	133.2	4.7	0.61
(3)	6.7	-50	4.6 ^a	-1000	40.0	26.6	68	244.8	4.1	0.53
I-27 (1)	4.6	-50	3.3	-1000	40.0	23.3	95	342.0	4.3	0.56
(1) ^{3,5}	4.3	-50	3.0	-1000	40.0	21.3	31.8	114.5	4.4	0.57
(1) ^a	5.0	-50	3.2	-1000	40.0	23.4	75	270.0	5.0	0.65
(1) ^{2,3,5}	7.0	-50	4.4 ^a	-1000	40.0	28.0	149	536.4	4.3	0.56
I-36 (1)	4.6	-50	3.0	-1000	45.0	63.0	52.3	188.3	4.1	0.53
(1) ^a	4.1	-50	2.4	-1000	41.0	61.5	116.0	417.6	3.9	0.51
(1) ^a	3.5	-50	1.8	-1000	35.0	58.8	82.8	298.1	3.5	0.46
Post Cathode										
II-4P (1)	5.6	-50	11.4	-500	44.0	27.3	93.8	337.7	2.6	0.34
(1)	6.4	-50	11.2	-500	44.0	27.8	93.0	334.8	3.6	0.47
(1)	4.7	-50	13.3	-500	44.5	29.5	113.2	407.5	3.1	0.43
II-5P (1)	7.9	-50	8.9	-500	44.9	34.4	35.0	126.0	3.5	0.46
(1)	7.9	-50	7.0	-500	45.0	34.1	63.5	228.6	3.2	0.42
(1)	8.0	-50	9.8	-500	45.0	34.7	47.0	169.2	3.7	0.48
(2)	7.5	-50	6.0	-500	45.0	33.6	51.0	183.6	3.0	0.39

¹Substrate machined to a true diameter before reinstallation for the next cycle.

^aSubstrate was turned end for end before reinstallation for this part of the deposition cycle.

^bCathode turned end for end before starting this part of the deposition.

^cSubstrate rotated 180° before starting this part of the deposition cycle.

^dSubstrate rotated 90° before starting this part of the deposition cycle.

^e10.16 cm ID AMZIRC Cu cathode used to start deposition.

^fA new 10.16 cm ID AMZIRC Cu cathode installed.

^g11.43 cm ID cathode installed for this cycle.

^h10.80 cm ID cathode installed for this cycle.

substrate. In these experiments, the decision was made to increase ion energy by using more negative bias voltages, in order to more clearly define the function of ion bombardment on groove filling.

Before reporting the results of this work, reference works which may apply will be noted. Seeman (Reference 5) has shown the effect of bias on lining narrow grooves in steel with sputtered chrome. Thornton and Hedgcoth (Reference 6) have shown how bias affects the structure and thickness of coating in various sized grooves in a hollow magnetron cathode. The characteristics of ceramic coatings deposited over a step on a substrate, as a function of bias on the substrate, have been shown by Kennedy (Reference 7).

The deposit thickness at the center of each test substrate, deposition rate and substrate appearance are given in table 20. The deposit profiles along the length of each substrate are given in figures 33 and 34. The thickness around the circumference at each end was not measured. Photomicrographs which illustrate the findings of this study are shown on figures 35 through 38. The topography of a typical aluminum deposit in a groove has been illustrated schematically in figure 39. The photomicrographs have been analyzed with the aid of this schematic and data on θ , the angle of the aluminum on the substrate wall, plus data used to calculate groove filling efficiency are entered in table 21.

Even though the topography of the sputtered aluminum is more clearly defined with the more negative bias, a comparison of data from different substrates or from different deposition cycles is made difficult by the obvious differences in ion current density and in the arrival rate of the sputtered aluminum at different sections of the substrate. The results would have been easier to interpret had the ion current density been the same over all parts of the substrate and the depositions had been all stopped when either the deposit thickness or the groove deposit thickness (figure 39) had reached the same thickness on each substrate. Also, a new cathode for each substrate would have eliminated ambiguities due to the change in cathode surface profile with deposition time.

As a consequence of the uncertainty in interpretation, the values for θ and filling efficiency can have a considerable range. This is compared on table 21 for substrates I-10 and I-19. The origins of the range of values reported is explained on the table.

The data from different systems will also be difficult to compare since the bias voltages are measured with respect to ground (zero volts) and not with respect to the plasma potential. At zero volts the substrate is still being bombarded by ions with energy determined by the potential difference between the plasma and the substrate.

Although the quantitative data lack high precision, the following conclusions may be clearly drawn from this filling study: (Note that the following conclusions apply only when the ion current density is sufficient to cause efficient forward sputtering.)

1. The filling efficiency of all sized grooves was improved with increasing negative bias. This is illustrated for 0.157-cm wide by 0.127-cm deep grooves on figures 35 and 36. Figures 36 and 37 shown that this sized groove is easily filled in one deposition cycle with a -125v substrate bias.
2. The angle θ increased with increasing negative substrate bias voltage. The importance of a large θ for efficient groove filling can be summarized as follows (Reference 7). As θ increases, the sputtering yield (number of atoms removed per bombarding ion) increases and therefore forward sputtering is more rapid. This not only redeposits more material at the bottom of the groove, but the wider groove opening can accept sputtered flux from a wider angle.

TABLE 20. DEPOSITION RATE AND SUBSTRATE APPEARANCE FOR ALUMINUM SPUTTERED ONTO TEST GROOVED SUBSTRATES

Substrate Number (Cycle No.)	Deposit Thickness at Center of Substrate		Deposition Rate at Center of Deposit		Substrate Appearance
	(mils)	(mm)	(mils/hr)	(nm/sec)	
I-10					
(1)	21.5	0.55	0.35	2.47	Bright
(2)	20.0	0.51	0.56	3.95	Bright
I-17					
(1)	27.0	0.69	0.43	3.03	Bright
(2)	—	—	—	—	—
I-18					
(1)	16.5	0.42	0.35	2.47	—
(2)	20.0	0.51	0.31	2.19	Bright/Matte
I-19					
(1)	28.0	0.71	0.49	3.46	Bright
I-20					
(1)	48.5	1.23	0.67	4.73	Bright ⁽¹⁾

⁽¹⁾Except for a 2-in. wide strip running the length of the substrate.

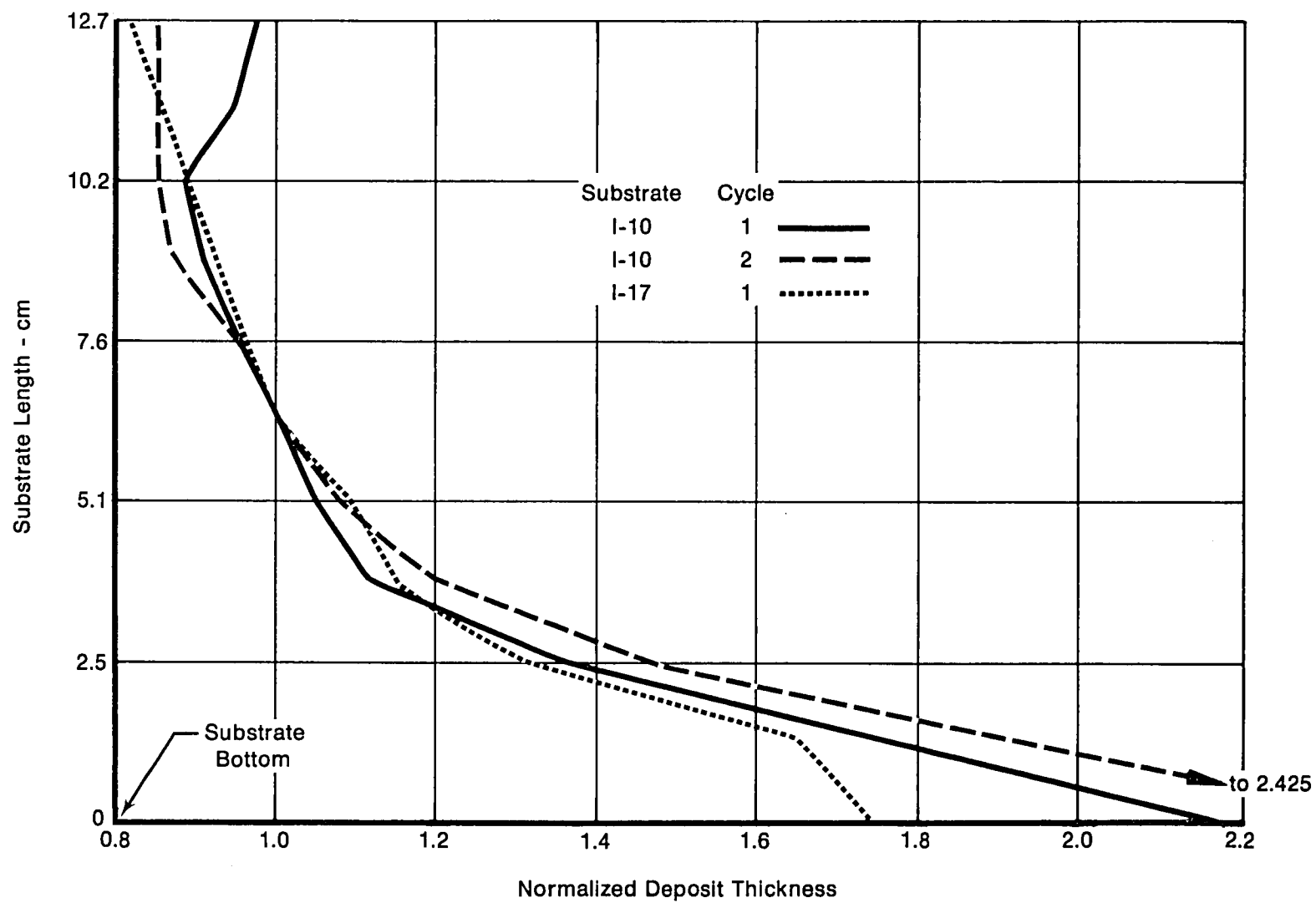


Figure 33. Variation of Aluminum Deposit Thickness for Test Grooved Hollow Cathode Substrates

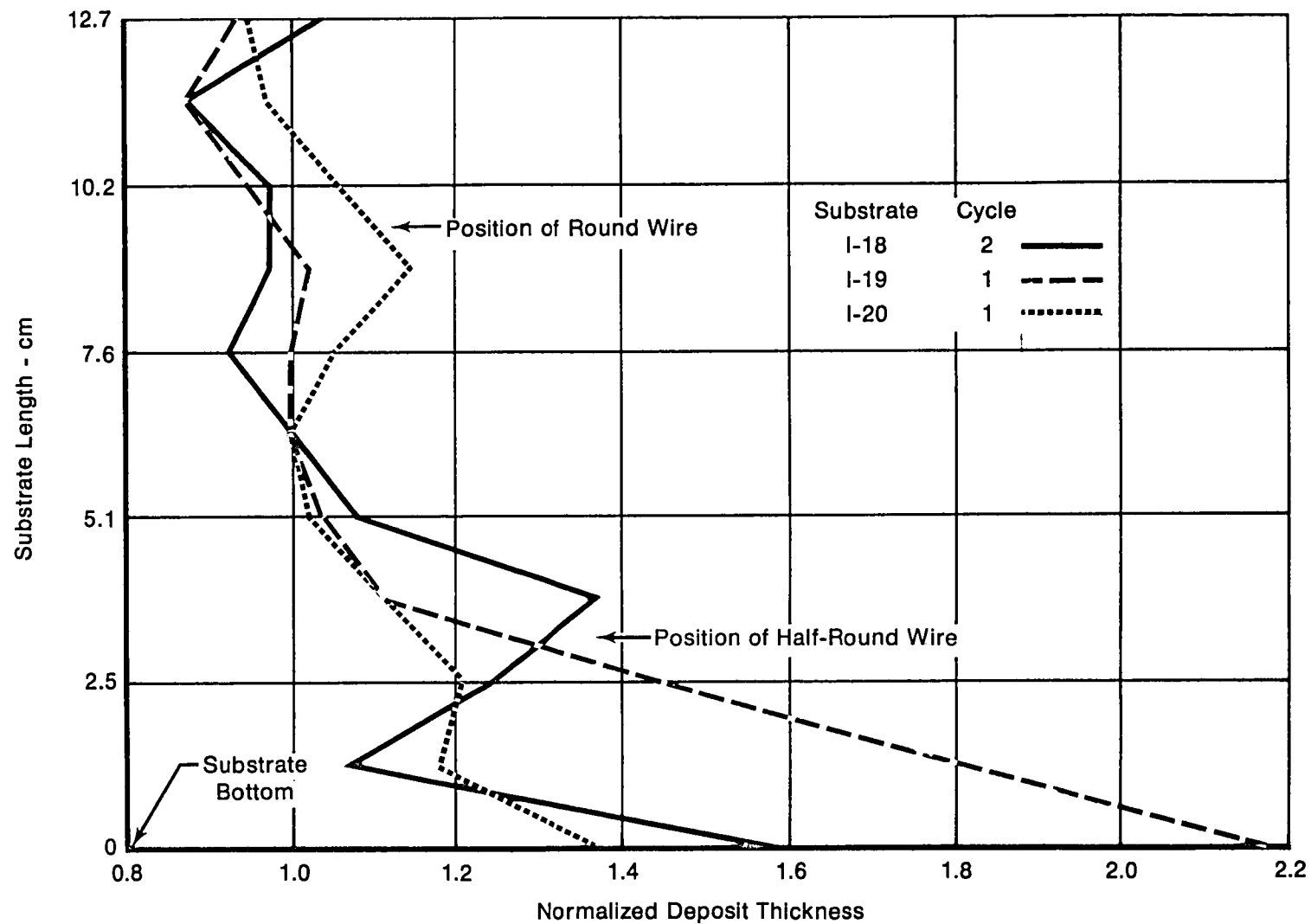
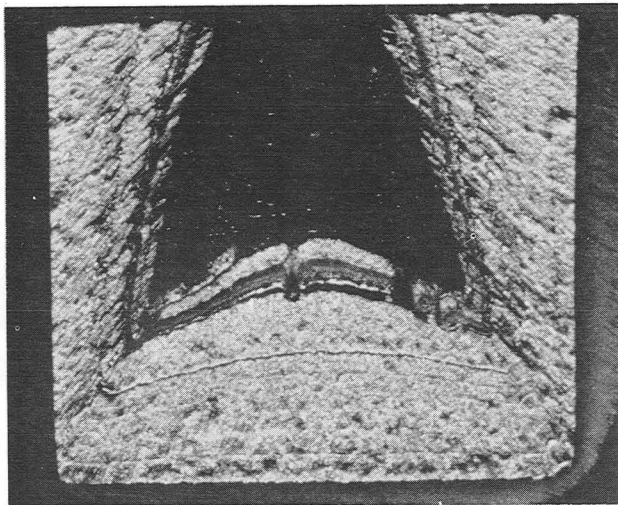
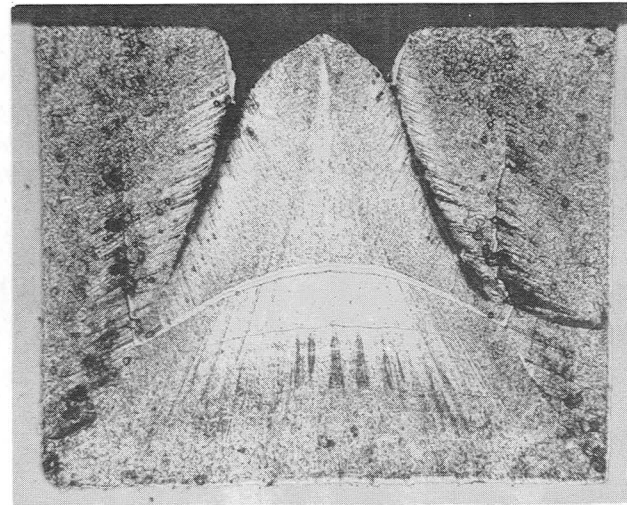


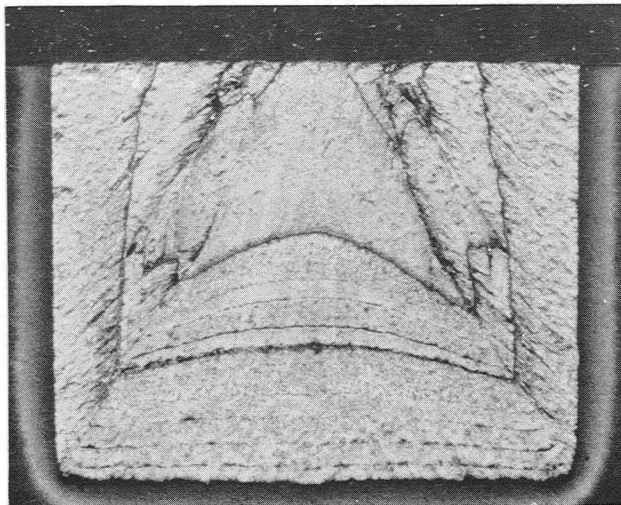
Figure 34. Variation of Aluminum Deposit Thickness for Test Grooved Hollow Cathode Substrates



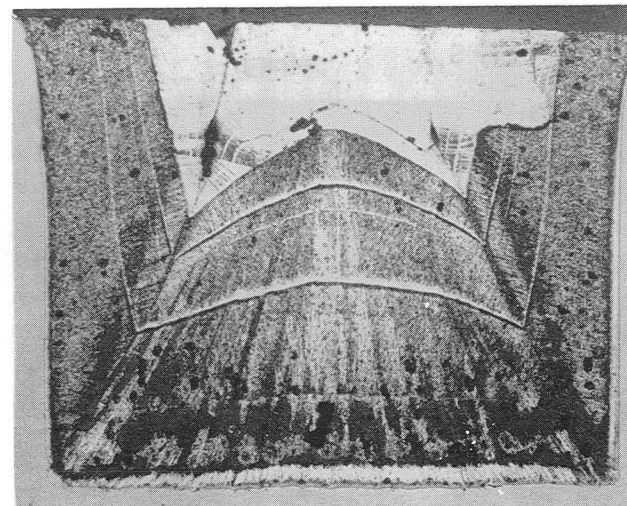
Substrate I-2 (0V Bias)
Etched



Substrate I-10 (-50V Bias)



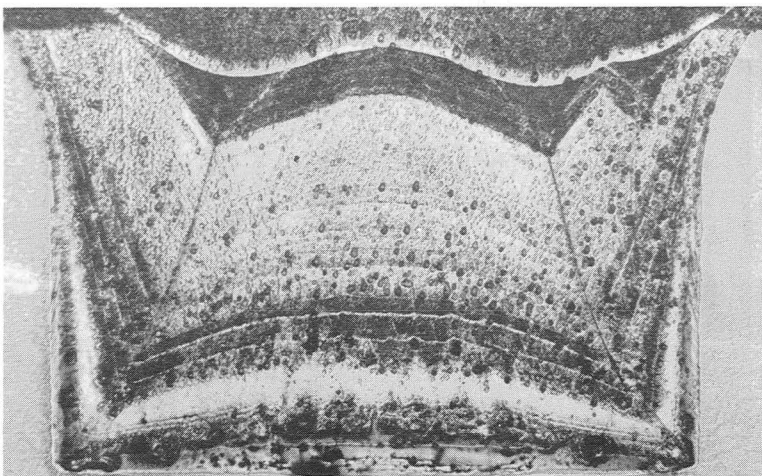
Substrate I-1 (-25V Bias)
Etched



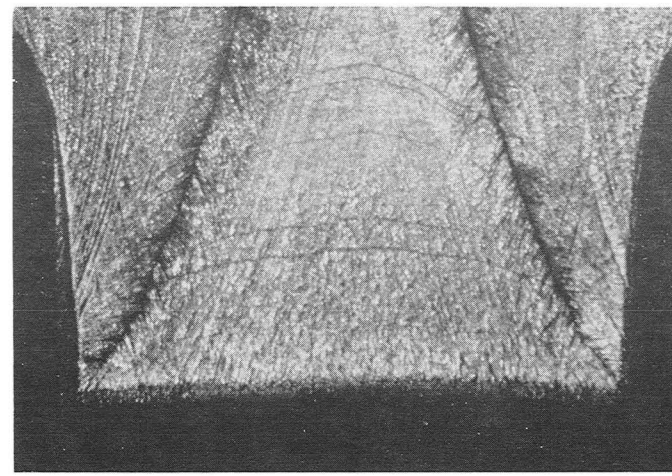
Substrate I-17 (-75V Bias)

Mag: Nominal 50X

Figure 35. Growth Characteristics of Aluminum Sputtered from a Hollow Cathode into Nominal 0.152 cm Wide by 0.127 cm Deep (0.062 by 0.050 in.) Grooves as a Function of Substrate Bias Voltage

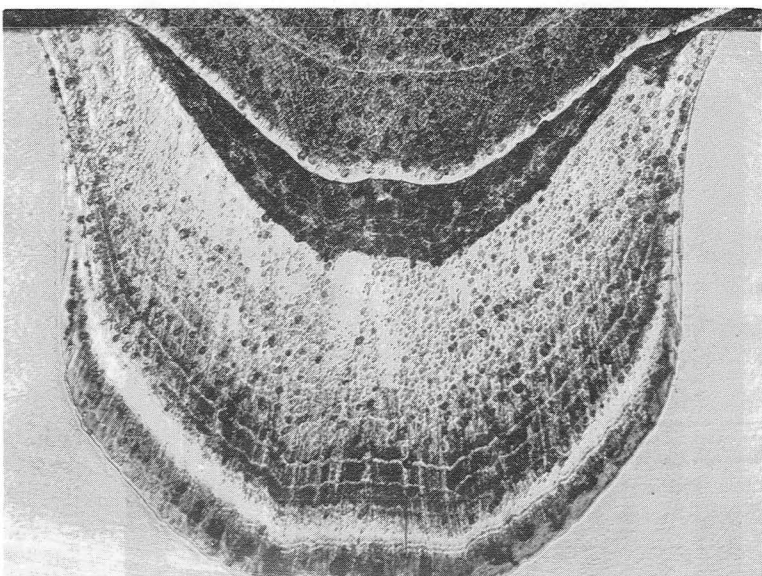


FAL 41942
Substrate I-18

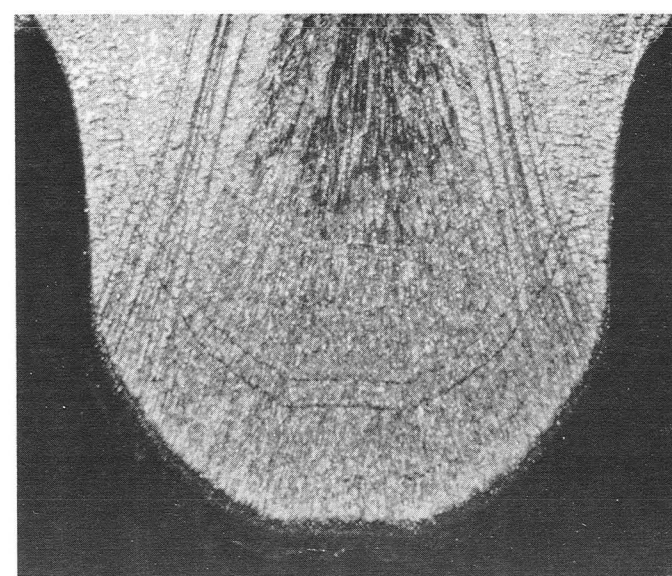


Etched

Substrate I-20

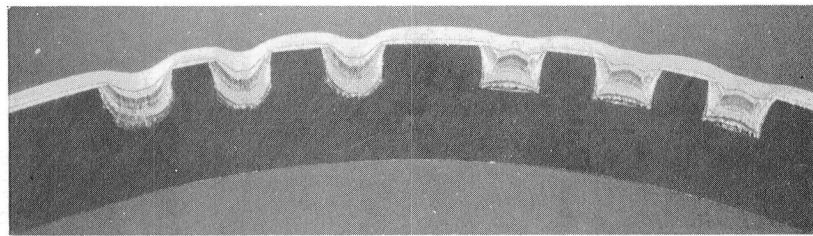


FAL 41943



Etched

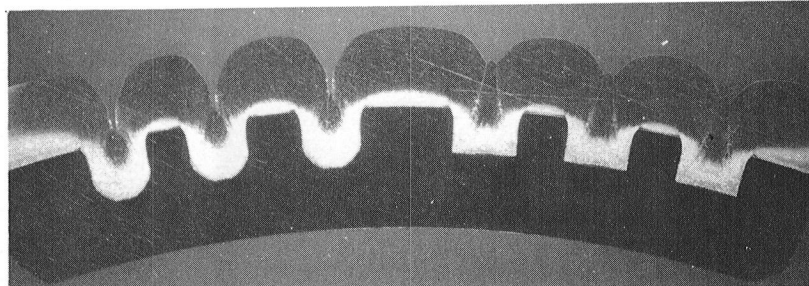
Figure 36. Growth Characteristics of Aluminum Sputtered from a Hollow Cathode into Flat and Round Bottomed Grooves. -125 Volt Bias



Mag: 5X

I-18

FAL 41934

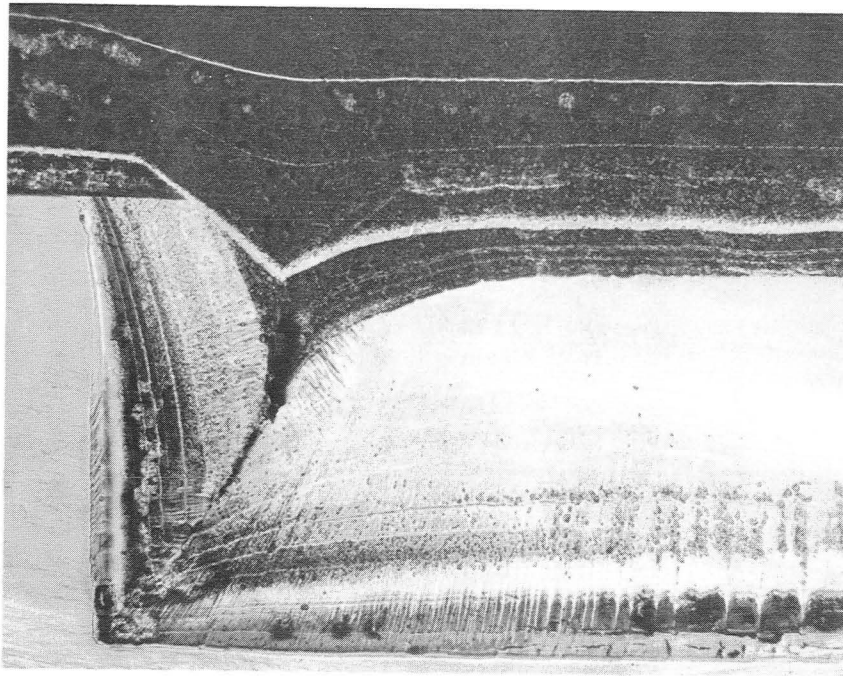


Mag: 5X

I-20

FAL 42155

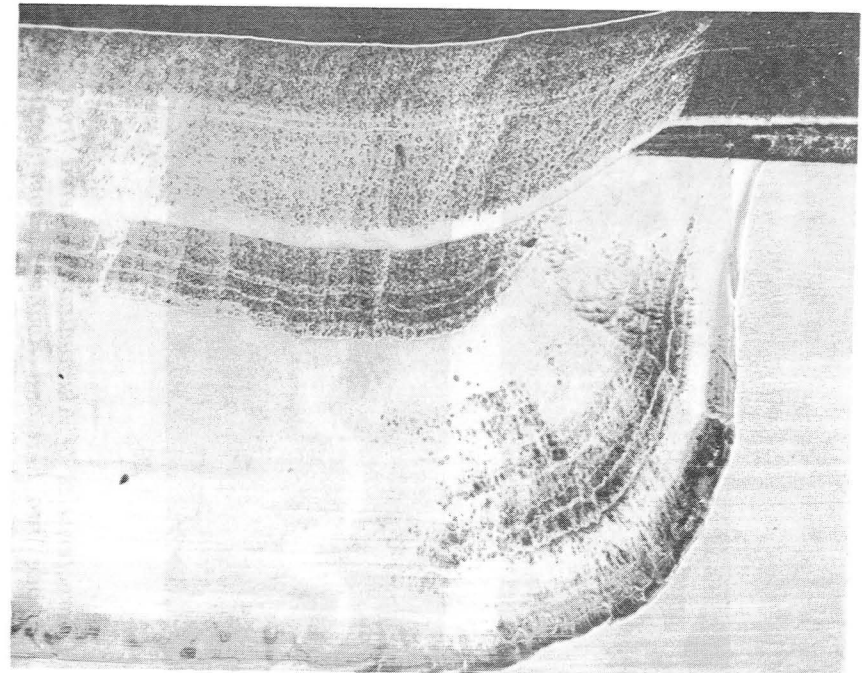
Figure 37. Growth Characteristics of Aluminum Sputtered from a Hollow Cathode into Flat and Round Bottom Vertical Grooves With -125 Volt Bias. The Grooves on Substrate I-18 Were Nearly Filled on the First Deposition Cycle



Mag: 50X

End of Flat Bottom Groove

FAL 41944



Mag: 50X

End of Round Bottom Groove

FAL 41945

Unetched

Figure 38. Growth Characteristics of Hollow Cathode Sputtered Aluminum at the End of Flat and Round Bottom Vertical Grooves. Substrate I-18

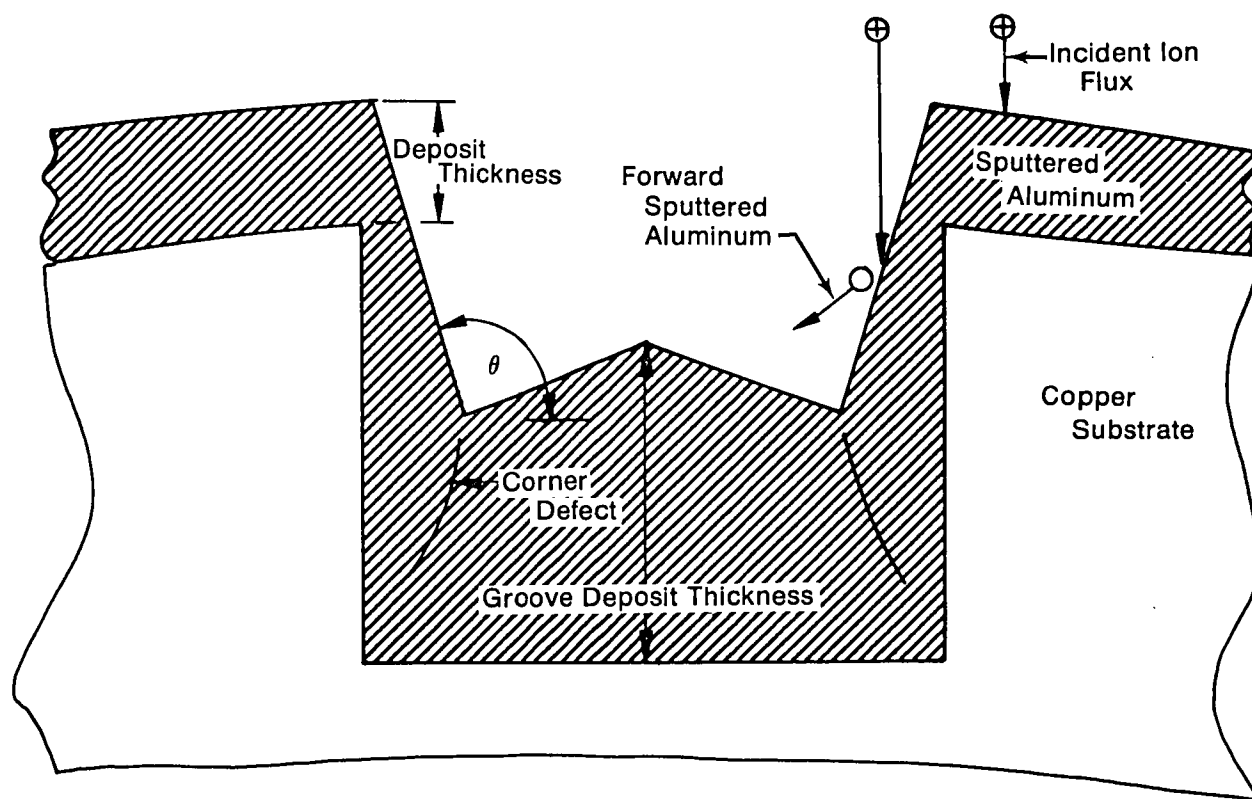


Figure 39. Schematic of the Topography of Sputtered Aluminum in a Groove. This Schematic Defines the Angle θ and the Factors Used in Calculating Groove Filling Efficiency for Table 21

TABLE 21. SUMMARY OF MEASUREMENTS TAKEN FROM CROSS SECTIONS OF 0.152 cm WIDE BY 0.127 cm DEEP GROOVES FILLED WITH SPUTTERED ALUMINUM. (See Figure 39 for Explanation of Terms)

Substrate Number	Substrate Bias (volts)	θ (deg)	Groove Deposit Thickness		Center Deposit Thickness		Filling Efficiency (%) ¹	Deposition Time		Deposition Rate	
			(mils)	(mm)	(mils)	(mm)		(hr)	(ks)	(mil/hr)	(nm/sec)
I-2 ²	0	80	8.0	0.20	43.0	1.09	19	68	244.8	0.63	4.44
I-1 ²	-25	82	15.0	0.38	46.0	1.17	33	88	316.8	0.52	3.67
I-10	-50	92	15.5	0.39	21.5	0.55	72	61.5	221.4	0.35	2.47
I-10 ²	-50	Not Measurable	25.0	0.64	75.0	1.91	33	169.0	608.4	0.44	3.10
I-17	-75	102	33.0	0.84	27.0	0.68	122	63.0	226.8	0.43	3.03
I-19 ³	-100	90-97	16.0	0.41	14.0	0.36	114	65.0	234.0	0.22	1.55
I-19 ⁴	-100	100-116	49.0	1.24	42.0	1.07	117	65.0	234.0	0.65	4.59
I-19 ⁵	-100	118-130	57.5	1.46	36.0	0.91	160	65.0	234.0	0.55	3.88
I-18	-125	103	40.0	1.02	16.5	0.42	242	47.0	169.2	0.35	2.47
I-20	-125	106	—	—	28.5	0.72	120	72.0	259.2	0.40	2.82

¹ Filling Efficiency is the quotient of groove deposit thickness and (center) deposit thickness on the substrate.

² Data taken from work done for previous contract (Reference 1).

³ Vertical grooves were in a very low rate area. Deposit thicknesses scaled from photomicrograph.

⁴ Deposit thicknesses scaled from photomicrograph of bottom horizontal groove from a low rate region.

⁵ Deposit thicknesses were scaled from photomicrograph of top horizontal groove from a high rate region.

3. Increasing ion current density at constant bias voltage causes a rapid increase in θ and filling efficiency. The wide range of data obtained for substrate I-19 illustrates this (table 21, notes 3, 4 and 5). (The highest ion current density corresponds to the highest deposition rate.)
4. The angle θ increases with deposition time for bias voltages more negative than -50v . This is shown on figure 36 for the rectangular groove from substrate I-18. The change with time is seen in the demarkation lines which are formed every time the deposition was stopped.
5. The structure of the aluminum deposit was improved with increasing negative bias.
6. The corner defect is eliminated at higher negative bias voltages. The apparent contradiction shown in figure 35 for substrate I-10 can be explained by assuming that the grooves received a lower bias current density for deposition cycle number 2 than for cycle 1. The ion current density was not sufficient to do any appreciable forward sputtering. (The explanation of how the arrival rate of the sputtered aluminum can be appreciable at a place on the substrate which has a low ion current density has been given previously (Reference 1, pages 44-45) in reference to the deposit on substrate I-14.) The resultant deposit is similar in appearance to the deposits formed in smaller grooves at low substrate bias (Reference 1, figure 12).
7. Deposit characteristics for round-bottomed grooves were similar to the deposits in rectangular-bottomed grooves except for the absence of the corner defect. The round bottom had no effect on θ .
8. The deposit characteristics at the end of the truncated grooves were identical to the deposits seen in the cross sections (figure 38). The corner defect, when present, degenerates to a pinhole defect. This type of defect is illustrated in figure 39 for substrate I-35.

The previous conclusions provide sufficient evidence to say that Kennedy's step coating model (Reference 7) may be applied to groove filling and that additional conclusions may be drawn.

9. The amount of bias voltage needed to achieve groove filling is dependent on the sputtering yield and deposition rate of the filling material. That is, a less negative bias voltage would be needed to fill a groove with copper than with aluminum assuming the same arrival rate of sputtered material and the same ion current density. Evidence of the effectiveness of the bias voltages for sputtering copper can be seen on figures 35 and 36. The groove walls are heavily rounded at the top (substrates I-17, I-18 and I-20) from the relatively short bombardment carried out for sputter cleaning. The second part of this conclusion implies that the deposition rate and resputtering rate must be balanced for efficient groove filling.

The last conclusion predicts that a more efficient method of fabricating a regeneratively cooled thrust chamber would be to sputter copper onto an aluminum substrate which had properly machined lands. The aluminum would be machined back to leave "aluminum filled grooves" which would be closed out in the usual manner.

The deposits in the horizontal grooves had the same characteristics as the deposits in the vertical grooves. Conclusions as to the directions of incoming sputter aluminum could not be made.

Note that the relatively shallow grooves on the burst test specimens filled completely with no defects when the grooves were in a region of adequate ion current density (figure 40). Grooves in a region of low ion current density were filled only after several cycles of back machining and deposition. Indications of high temperatures were noticed in several of the burst test specimens. The sputter aluminum formed a gold colored alloy with the copper.

The deposits over wires had many of the characteristics of deposits in grooves. The results shown on figure 41 for substrate I-20 are also typical of the results obtained for substrates I-18 and I-19. The half-round wires were filled over completely and were apparently well "bonded" to the substrates. As shown at the higher magnification on figure 42, the round wire had the typical "corner" growth defect on each side.

The effectiveness of the substrate bias in smoothing closeout layers is seen previously on figure 41. The smoothing was more rapid on substrate I-18 and I-19.

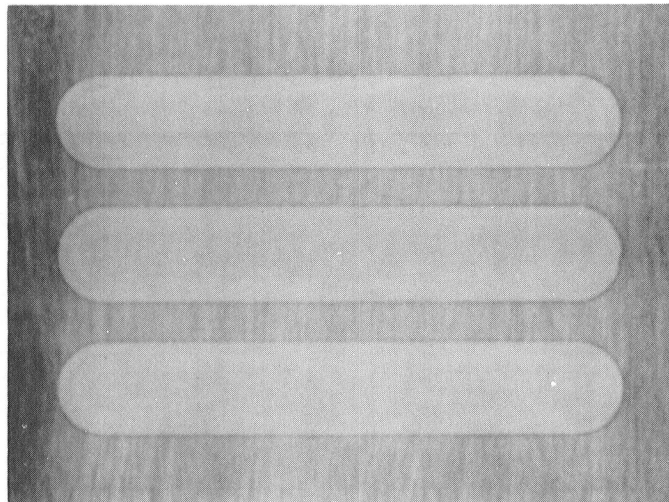
The difference in smoothing over the rounded and half-round wires may be due partly to the smaller ion current density at the top of the substrate. Round wires may be suitable for reinforcing if adequate bias ion current density and/or slightly increased wire spacing is used.

2. Filling GF Substrates With Sputtered Aluminum

Measurements on deposit thickness and evenness of sputtered aluminum deposits on GF substrates are given in table 22 and figures 43 through 47. Data in table 20 show that several deposition/machining cycles were necessary to fill grooves completely despite the high negative bias voltages used. After the first deposition only those grooves in a region of high ion current density were filled completely. The remaining grooves were only partially filled and had the corner defect structure. The additional cycles were required to fill over these grooves.

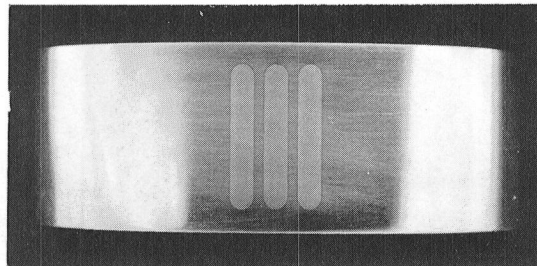
Typical filling results are shown for various filling stages for substrate I-35 on figures 48 through 50. The overall view of the substrate with the aluminum as-deposited (on the water-cooling mandrel) and as-machined is shown in figure 48 after the second deposition cycle. Figure 49 shows a higher magnification of the as-sputtered groove appearance after the third deposition cycle. Pinhole defects can be seen at the end of the grooves on the top end of the substrate. These grooves were at the top of the substrate in the initial deposition cycle, at the bottom for the second cycle and then back at the top for the third cycle. (The substrate was machined back after each cycle.) The higher ion current density at the bottom of the substrate during the second cycle failed to close up the defects which were started on the first cycle. The growth habit over the grooves is typical of the growth habit over defect structures (Reference 8).

Details of the grooves after machining substrate I-35, after the second and third depositions, are shown on figure 50. Machining was stopped just short of the copper surface. Some breakout of the sputter aluminum is evident for the second cycle machining. An example of this breakout is shown previously in figure 35 for substrate I-17.



Mag: 4X

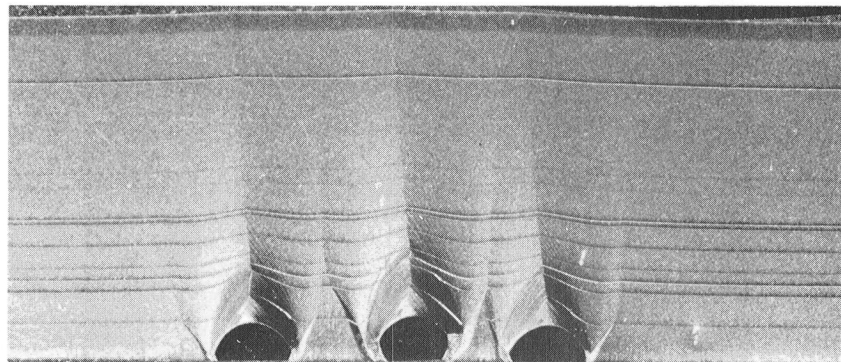
FAL 51366



Mag: 1X

FAL 51365

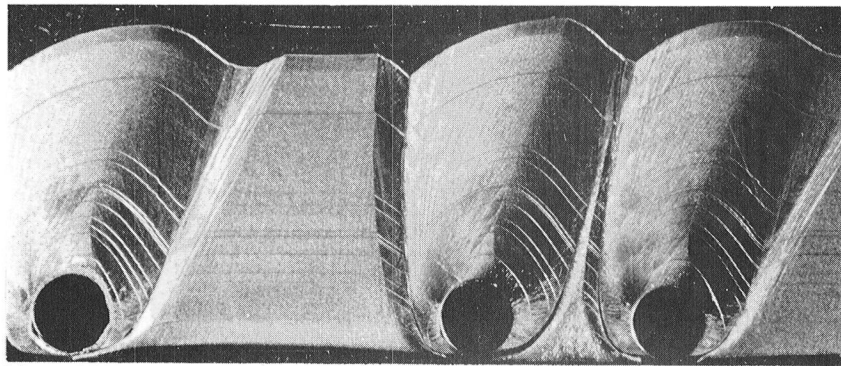
Figure 40. Hollow Cathode Burst Test Specimen Filled With Sputtered Aluminum. As-Machined and Hand Sanded With 600 Grit SiC Paper



Mag: 20X

Half-Round Wire

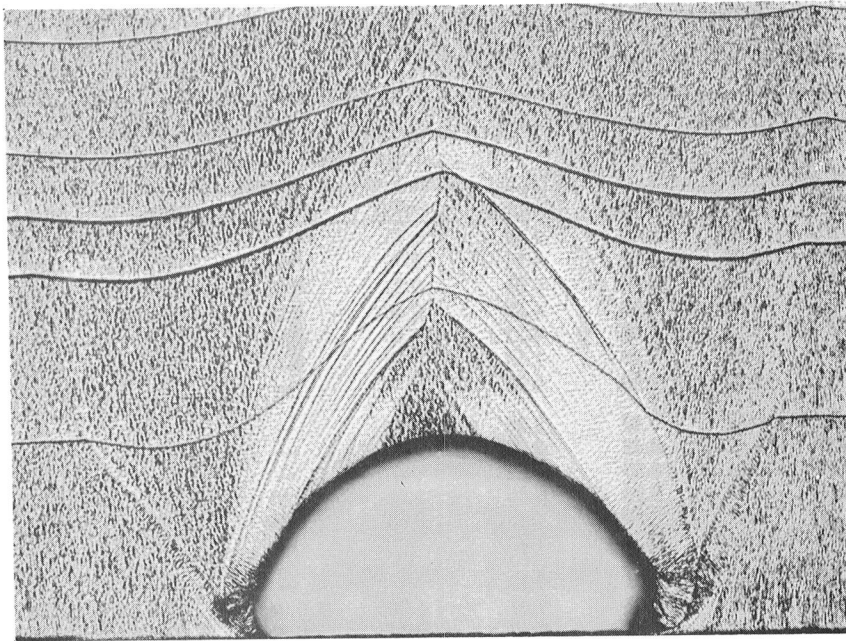
Interface With
Substrate



Mag: 20X

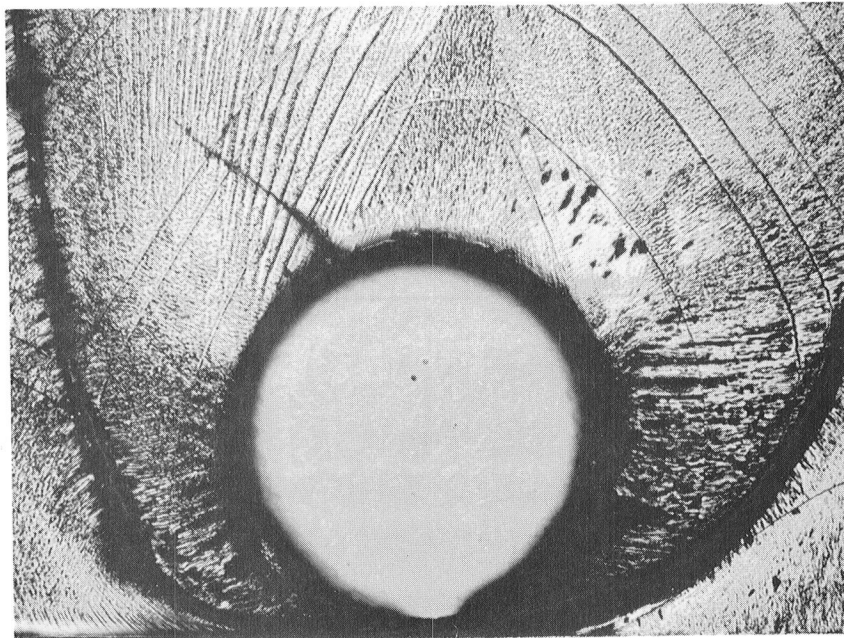
Round Wire

Figure 41. Growth Characteristics of Aluminum Sputtered from a Hollow Cathode Onto Half-Round and Round Wires. The As-Sputtered Surface Shows the Smoothing Effect of the $-125v$ Substrate Bias Voltage. Substrate I-20



Mag: 100X

Etched



Mag: 100X

Etched

Figure 42. Details of Growth Characteristics of Sputtered Aluminum Around Half-Round and Round Wires. Substrate I-20

TABLE 22. DEPOSITION RATE AND SUBSTRATE APPEARANCE FOR ALUMINUM SPUTTERED ONTO GF SUBSTRATES

Substrate Number (Cycle No.)	Deposit Thickness at Center of Substrate		Deposition Rate at Center of Deposit		Deposit Thickness				Substrate Appearance
	(mils)	(mm)	(mils/hr)	(nm/sec)	Top of Substrate		Bottom of Substrate		
					Maximum (mm)	Minimum (mm)	Maximum (mm)	Minimum (mm)	
I-25 (1)	15.0	0.38	0.44	3.10	0.61	0.18	0.69	0.28	Semibright
(2)	31.0	0.79	0.62	4.37	0.76	0.36	0.79	0.58	Bright
(3)	41.0	1.04	0.80	5.64	0.81	0.61	0.94	0.81	Bright
(4)	22.5	0.57	0.68	4.80	0.46	0.23	0.68	0.58	Matte/Bright ¹
(5)	13.0	0.33	0.62	4.37	0.46	0.06	0.38	0.36	Bright/Matte
(6)	5.0	0.13	0.16	1.13	0.18	0.13	0.20	0.15	Bright
(7)	8.0	0.20	0.50	3.53	—	—	—	—	Matte
I-27 (1)	17.5	0.44	0.83	5.86	—	—	—	—	Bright
(1)	40.0	1.02	0.42	2.96	—	—	—	—	Bright
(2)	32.0	0.81	0.57	4.02	—	—	—	—	Bright
(3)	68.0	1.73	0.93	6.56	1.52	0.91	1.75	1.57	Semibright
(4)	20.0	0.51	0.59	4.16	0.53	0.20	0.61	0.23	Bright
(5)	8.5	0.22	0.26	1.83	—	—	—	—	Bright
I-35 (1)	76.0	1.93	1.01	7.13	—	—	—	—	Semibright
(2)	79.0	2.00	1.05	7.41	1.30	0.94	1.78	1.73	Bright
(3)	40.0	1.02	1.13	7.97	0.71	0.51	0.91	0.81	Bright
(4)	15.0	0.38	0.56	3.95	0.38	0.05	0.56	0.29	Semibright
(5)	—	—	—	—	—	—	—	—	Bright
(6)	9.0	0.23	0.19	1.34	—	—	—	—	Bright
I-36 (1)	35.0	0.59	0.54	3.81	—	—	—	—	Matte
(1)	23.2	1.48	0.64	4.52	—	—	—	—	Matte/Bright
(2)	24.5	0.62	0.45	3.17	0.91	0.58	—	—	Bright
(3)	27.5	0.70	0.53	3.74	0.89	0.79	—	—	Bright
II-4P (1)	46.5	1.18	1.13	7.97	1.37	0.10	1.02	0.86	Matte
(2)	30.5	0.77	0.80	5.64	—	—	—	—	Bright
(3)	61.5	1.56	0.94	6.63	1.65	0.23	1.37	0.74	Semibright
(4)	63.0	1.60	0.97	6.84	—	—	—	—	Semibright
(5)	38.8	0.98	0.86	6.07	—	—	—	—	Bright
(6)	—	—	—	—	—	—	—	—	—

¹Matte Finish Over Grooves, Bright on Substrate Ends.

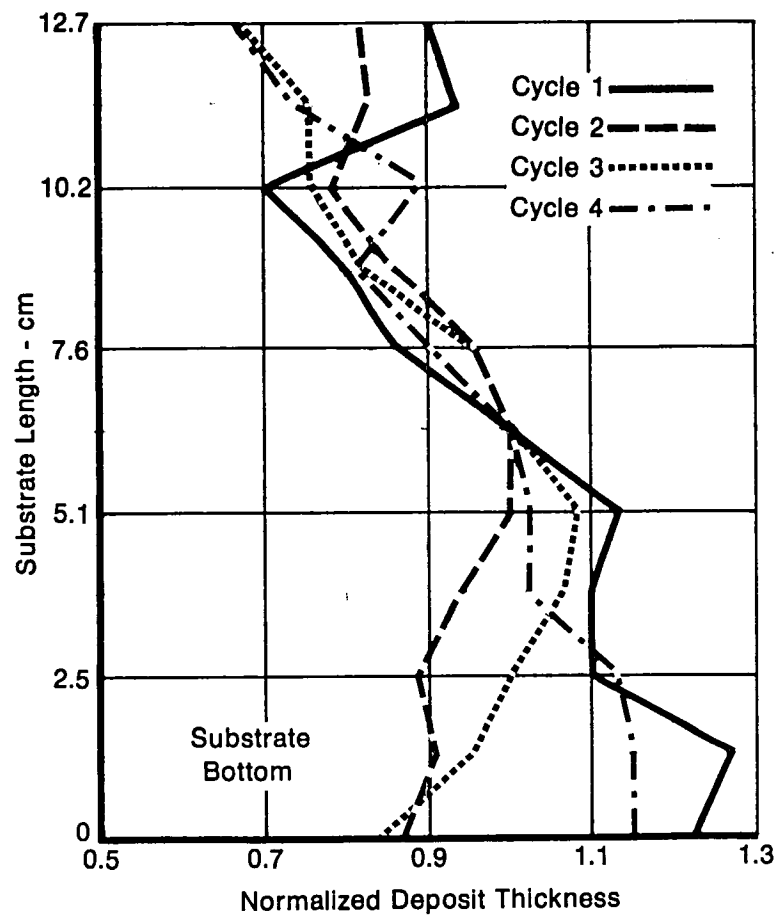


Figure 43. Variation of Aluminum Deposit Thickness for the First Four Deposition Cycles on GF Hollow Cathode Substrate I-25. The Aluminum Was Machined Back to the Substrate OD After Each Deposition Cycle

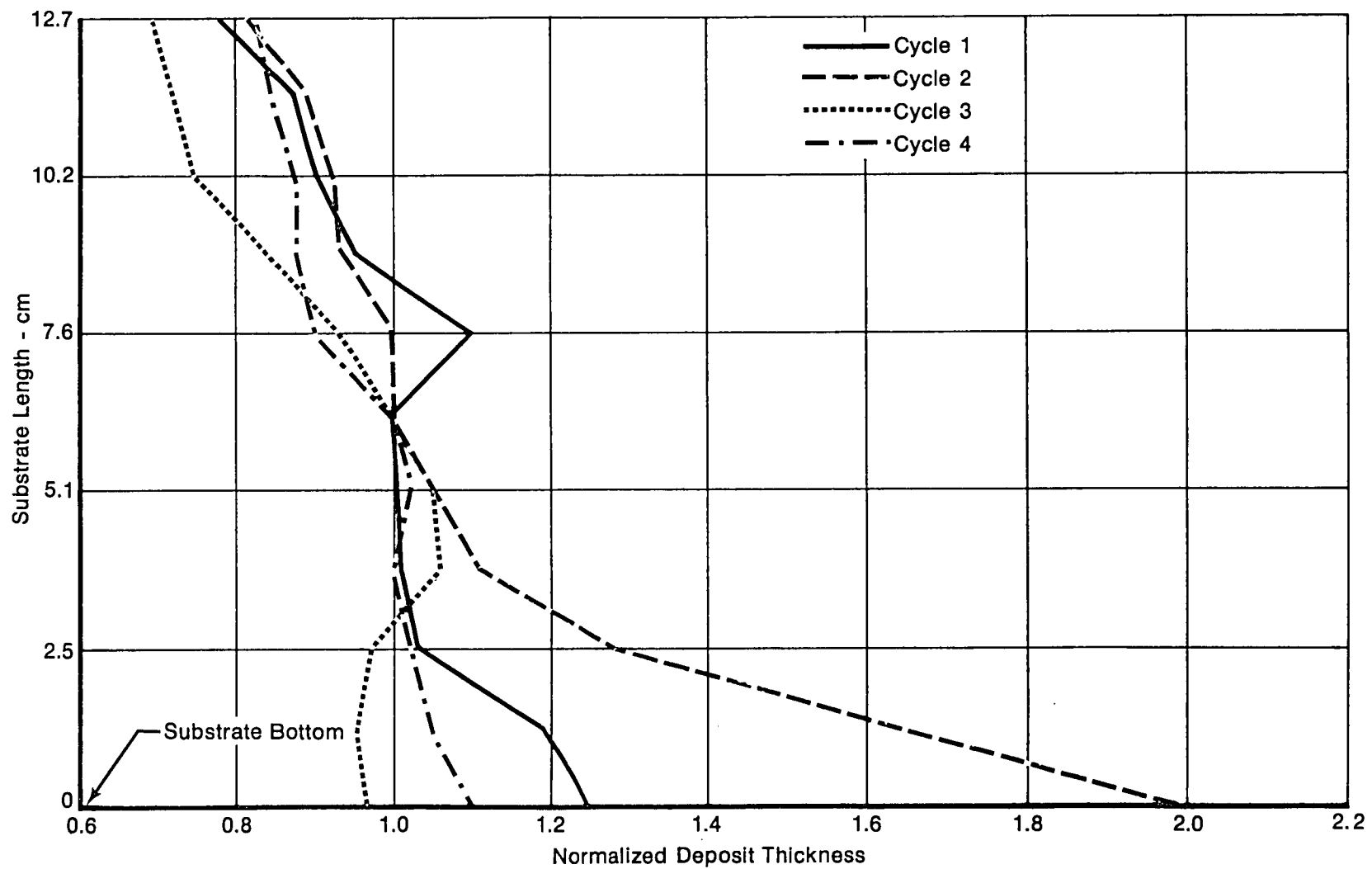


Figure 44. Variation of Aluminum Deposit Thickness for GF Hollow Cathode Substrate I-27. The Aluminum Was Machined Back to the Substrate OD After Each Deposition Cycle

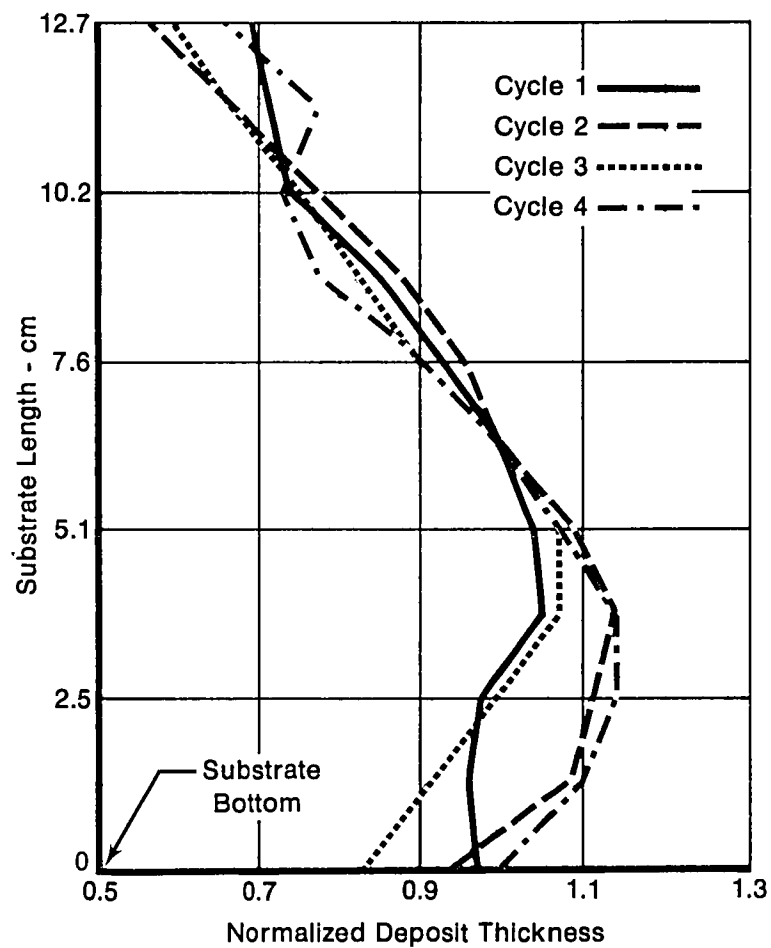


Figure 45. Variation of Aluminum Deposit Thickness for GF Hollow Cathode Substrate I-35. The Aluminum Was Machined Back to the Substrate OD After Each Deposition Cycle

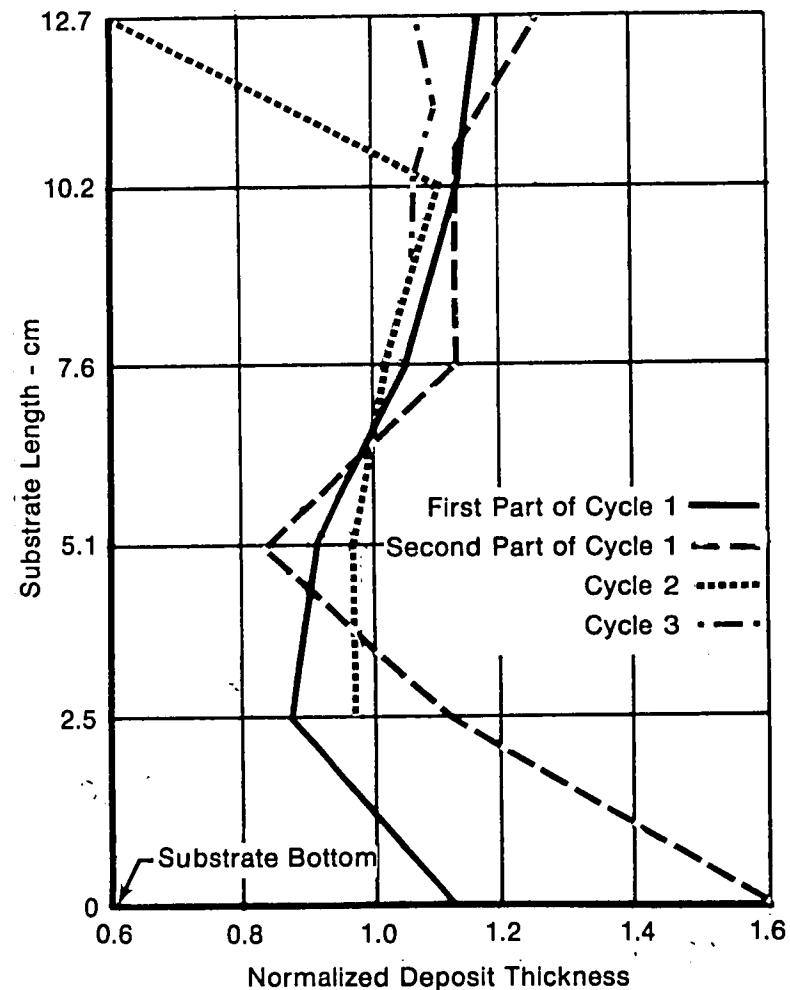


Figure 46. Variation of Aluminum Deposit Thickness for GF Hollow Cathode Substrate I-36. The Aluminum Was Machined Back to the Substrate OD After Each Deposition Cycle

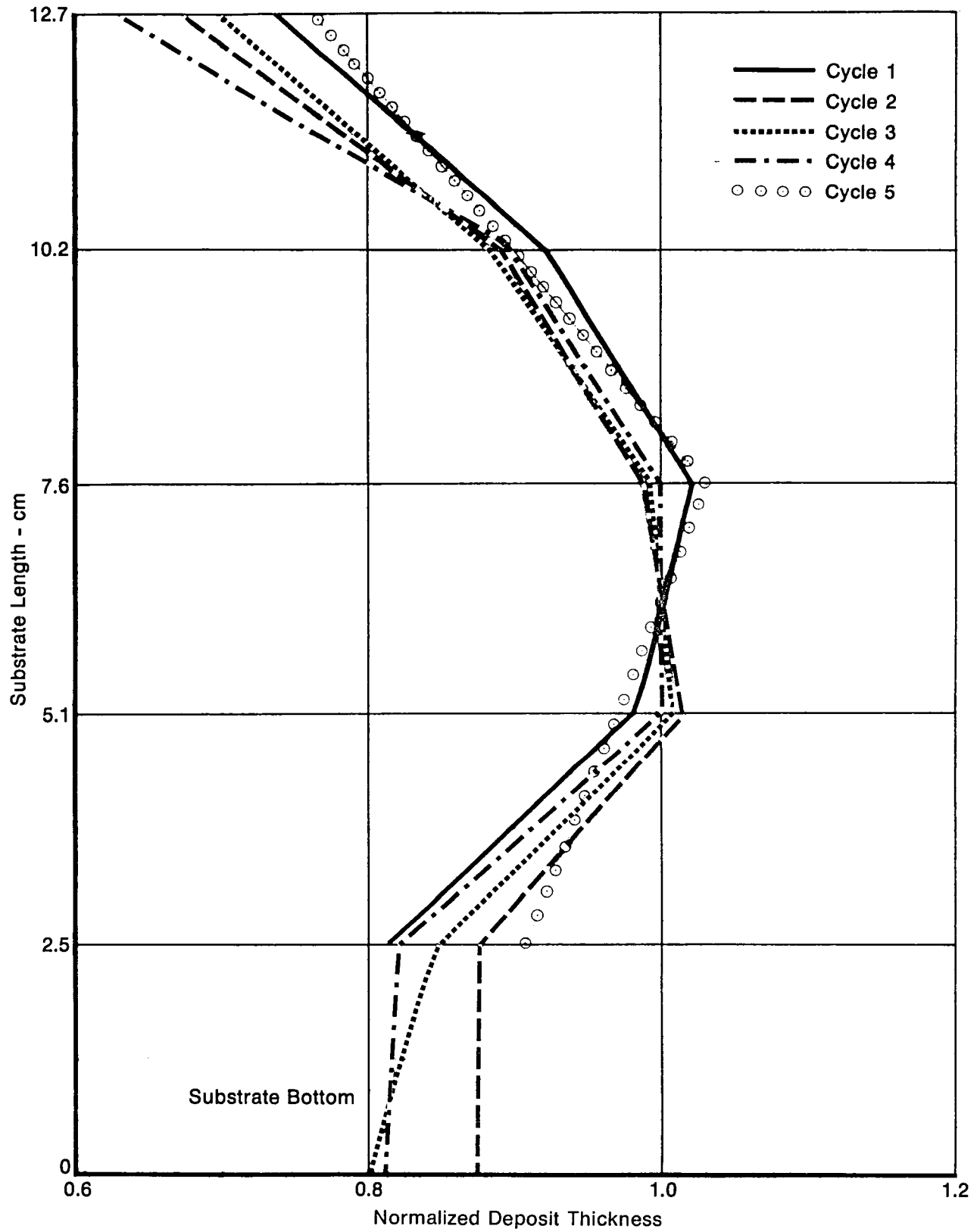
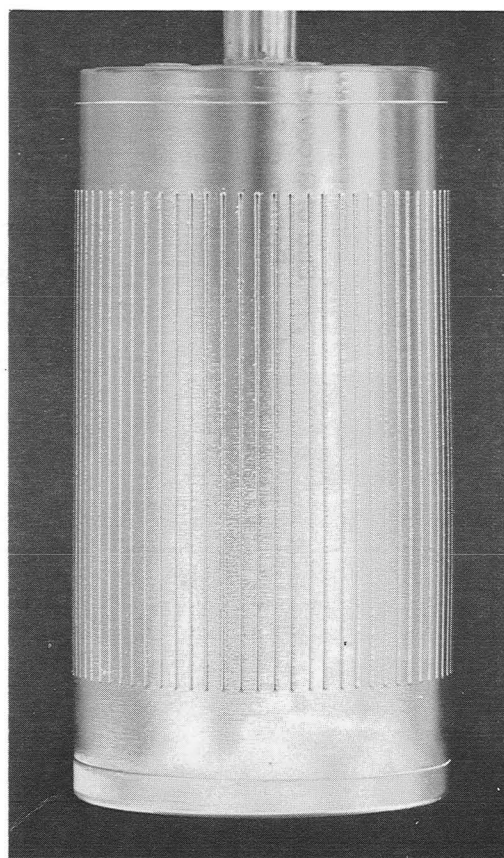


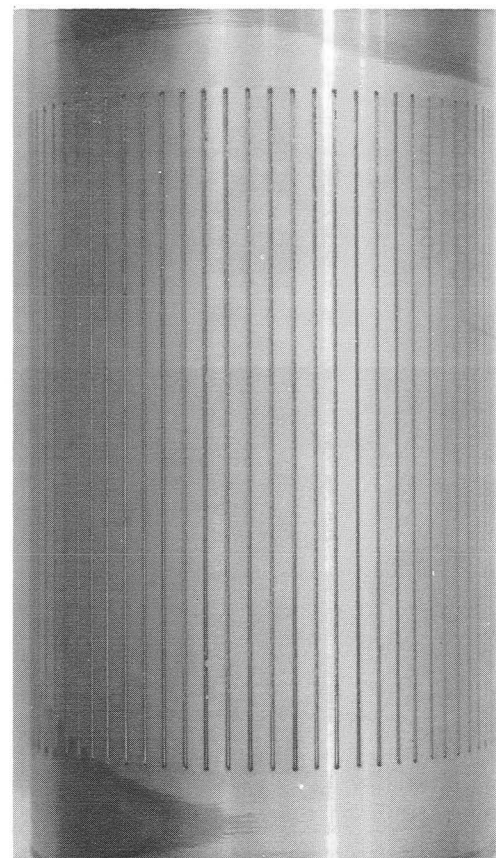
Figure 47. Variation of Aluminum Deposit Thickness for GF Post Cathode Substrate II-4P Aluminum Machined Back to Substrate OD After Each Deposition Cycle



As-Deposited

FAE 162555

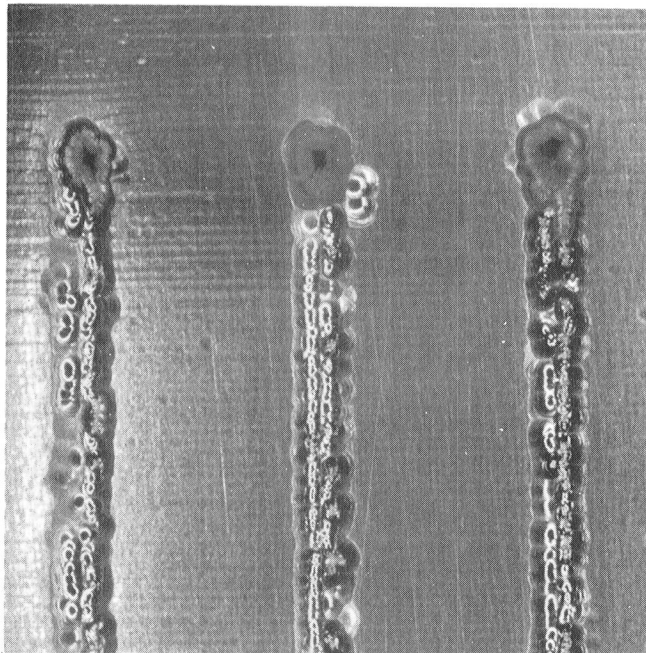
— Bottom End
of Substrate



As-Machined

FAL 44897

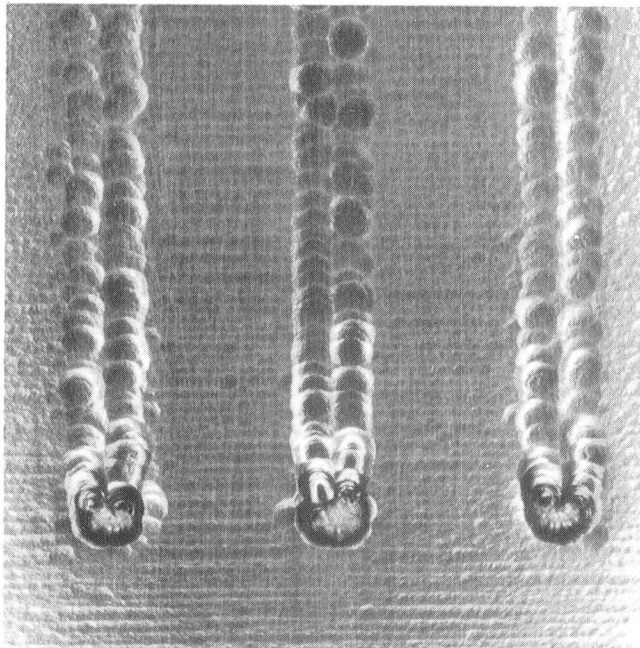
Figure 48. Sputtered Aluminum on GF Substrate I-35 After Deposition Cycle 2



End of Grooves at
Top of Substrate

Mag: 10X

FAL 45033

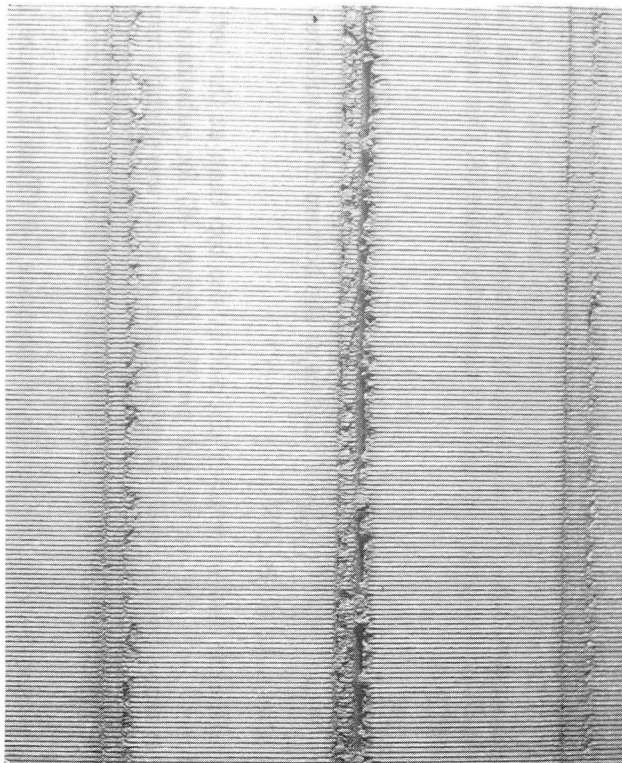


End of Grooves at
Bottom of Substrate

Mag: 10X

FAL 45032

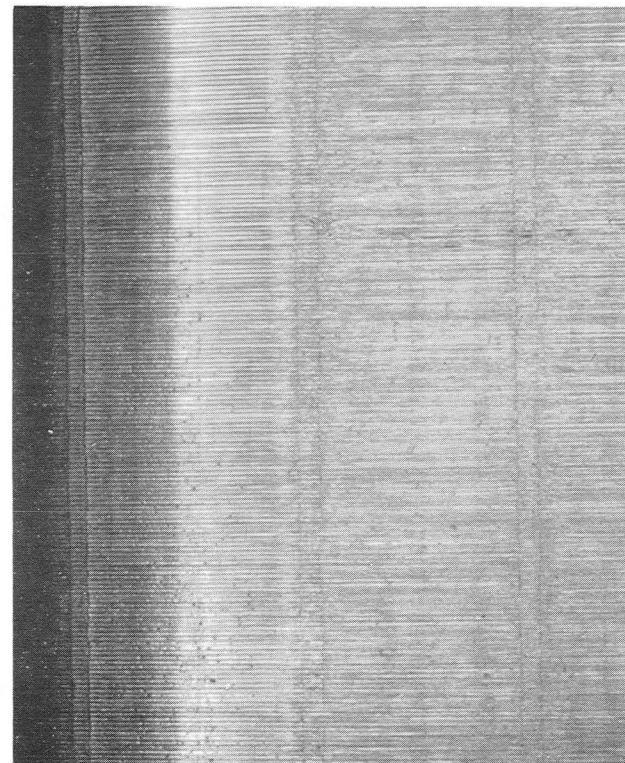
Figure 49. Characteristics of Sputtered Aluminum Over Grooves in GF Hollow Cathode Substrate I-35 After Deposition Cycle No. 3



Mag: 10X

FAL 44899

After Deposition Cycle No. 2



Mag: 10X

FAL 35038

After Deposition Cycle No. 3

Figure 50. Appearance of Grooves After Machining Back the Sputtered Aluminum on GF Substrate I-35. Machining Has Been Stopped Just Short of the Copper Surface

From the observation that the deposits in adjacent grooves could be quite different in appearance, e.g., a pinhole end between two completely filled ends, it was concluded that groove machining details may be important to groove filling. The varied groove widths and machining imperfections (chatter marks on bottom and sides) could have played a role in groove filling. Even though sputter filling was slow, this groove filling technique was used on all GF substrates except substrate II-5P due to the difficulty in filling over aluminum wire inlays. The variable groove widths caused some wire preforms to bulge out from the grooves at the center of the substrate when the wire expanded from heat-up after the start of deposition. These wires were subsequently torn out on the first machining cycle.

The diameters of the GF substrates after the final machining are given in table 23.

Some of the substrates were machined past tolerance since uneven aluminum layers were able to stress the substrates out-of-round.

TABLE 23. ALUMINUM FILLED GF SUBSTRATE DIAMETERS AFTER FINAL MACHINING

Substrate	Finished OD		Finished ID	
	(cm)	(in.)	(cm)	(in.)
I-25	6.985	2.750	—	—
I-27	6.985	2.750	—	—
I-35	6.985	2.750	—	—
I-36	7.036	2.770	—	—
II-4P	—	—	7.150	2.815
II-5P	—	—	7.036	2.770

3. Copper Closeout Layer Deposition

Dimensions and physical appearance of the as-sputtered copper closeout layers on the aluminum filled GF substrates are given in table 24 and figures 51 through 54. Cathode erosion profiles are shown on figures 55 through 57. Table 25 shows data on the amount of material collected on the substrates vs amount of material sputtered from the cathodes. Two examples of the final product of this program, i.e., closed out, fully grooved cylinders which have been machined and have had the filler material removed, are shown in figure 58. Due to lack of time, substrate I-35 was not closed out.

The program of changing substrate and cathode orientation and cathode ID for substrates I-25 and I-27 as described previously on table 19 did not help the deposit profiles. The deposits were still thicker and more even around the circumference at the bottom of the substrate than at the top.

Substrate I-36 had a thicker and more even deposit at the top than at the bottom. The deposit profiles were different from the aluminum profiles (figure 46) for substrate I-36 even though the same deposition geometry was used. The aluminum cathode was slightly more eroded at the bottom than at the top while the AMZIRC cathode was more eroded at the top than at the bottom. Cathode erosion will be discussed later in connection with deposition efficiency.

The deposit profile measured along the length of substrate I-9 (Task I, figure 13), after sputtering from a shaped post cathode, was not reproduced in any of the deposits on substrates II-4P and II-5P (figure 54). The length profiles were only slightly more even than the profiles obtained with straight post cathodes. The shaped post cathode deposits were more even around

TABLE 24. DEPOSITION RATE AND SUBSTRATE APPEARANCE FOR COPPER CLOSEOUT LAYERS ON ALUMINUM FILLED GF SUBSTRATES

Substrate Number (Cycle No.)	Deposit Thickness at Center of Substrate		Deposition Rate at Center of Deposit		Deposit Thickness				Substrate Appearance
	(mils)	(mm)	(mils/hr)	(nm/sec)	Top of Substrate		Bottom of Substrate		
					Maximum (mm)	Minimum (mm)	Maximum (mm)	Minimum (mm)	
I-25 (1)	82.0	2.08	1.08	7.62	Hollow	Cathode	—	—	Matte
(1)	10.0	0.25	0.27	1.90	—	—	—	—	—
(1)	48.0	1.22	1.00	7.06	—	—	—	—	—
(1)	11.2	0.28	0.35	2.47	4.32	3.15	7.19	5.74	—
(1)	230.0	5.84	1.78	12.56	5.03	1.60	5.86	2.97	—
(2)	106.5	2.71	0.84	5.93	—	—	—	—	Matte
(2)	—	—	—	—	—	—	—	—	Matte
(3)	72.5	1.84	1.07	7.55	1.42	0.89	1.91	1.40	Matte
I-27 (1)	63.7	1.62	0.67	4.73	—	—	—	—	Matte
(1)	20.5	0.52	0.64	4.52	—	—	—	—	—
(1)	58.0	1.47	0.77	5.43	—	—	—	—	—
(1)	200.0	5.08	1.34	9.45	11.43	6.48	8.64	7.37	—
I-36 (1)	52.0	1.32	0.99	6.98	—	—	—	—	Matte
(1)	126.5	3.21	1.09	7.69	—	—	—	—	Matte
(1)	57.0	1.45	0.69	4.87	7.75	7.44	6.71	5.82	Matte
Post Cathode									
II-4P (1)	36.8	0.93	0.39	2.75	—	—	—	—	Matte
(1)	47.0	1.19	0.51	3.60	—	—	—	—	Matte
(1)	60.8	1.54	0.54	3.81	3.15	2.39	3.51	2.44	Matte
II-5P (1)	43.1	1.09	1.23	8.68	—	—	—	—	Bright
(1)	76.0	1.93	1.20	8.47	—	—	—	—	Bright (Octagon)
(1)	—	—	—	—	—	—	—	—	Debonded
(2)	50.5	1.28	0.99	6.98	1.27	0.89	1.52	1.09	Bright (Matte in High Rate Area)

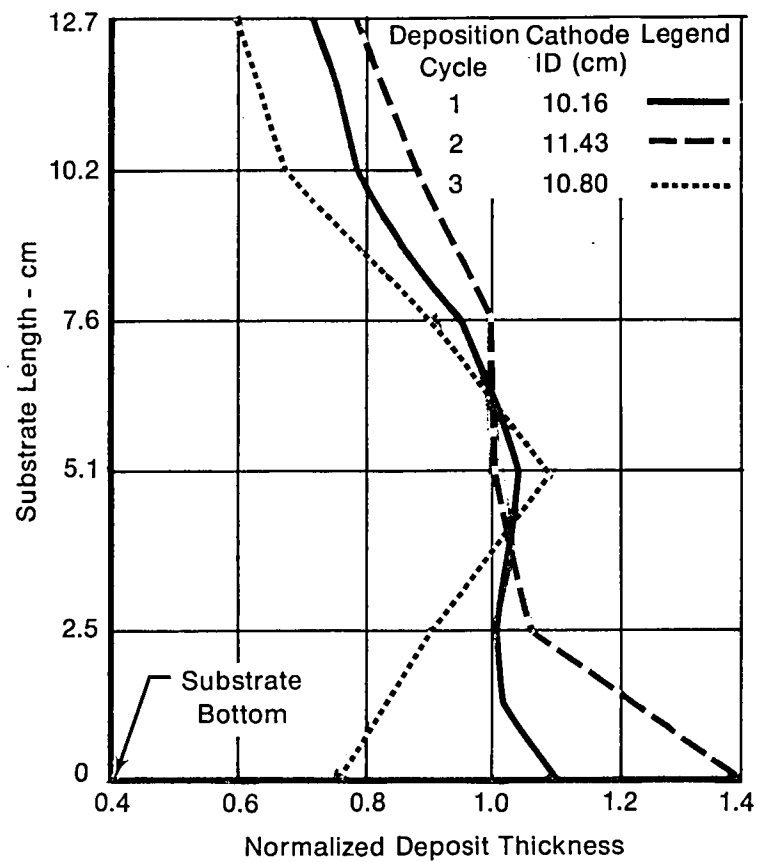


Figure 51. Variation of Copper Closeout Thickness for GF Hollow Cathode Substrate I-25 Closed Out in Several Sizes of Cathodes

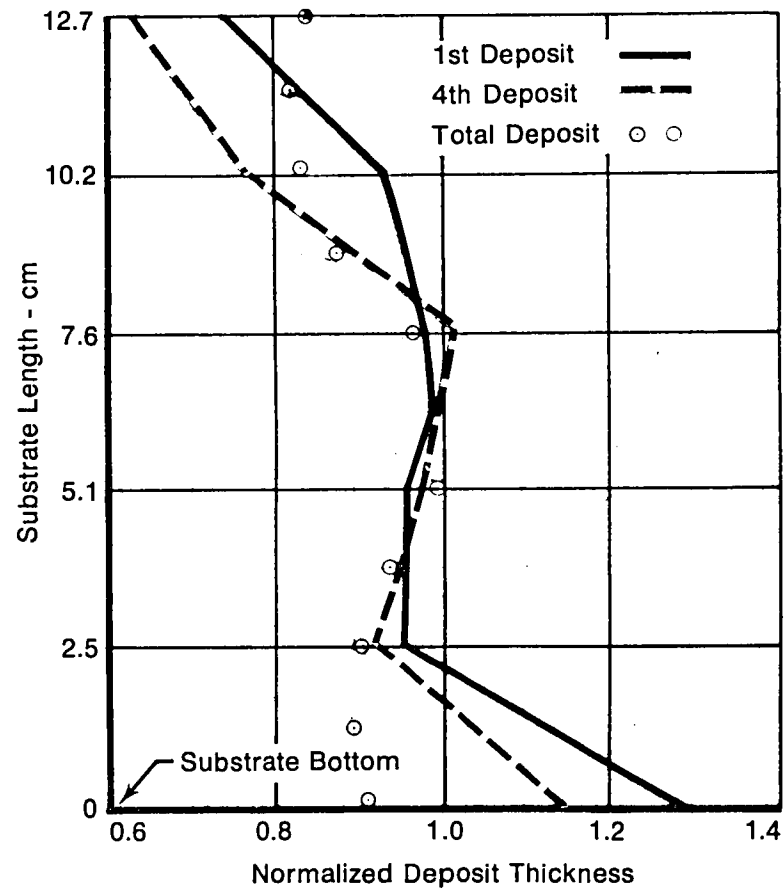


Figure 52. Variation of Copper Closeout Thickness for GF Hollow Cathode Substrate I-27 Closed Out in 10.80 cm ID Hollow AMZIRC Cathodes

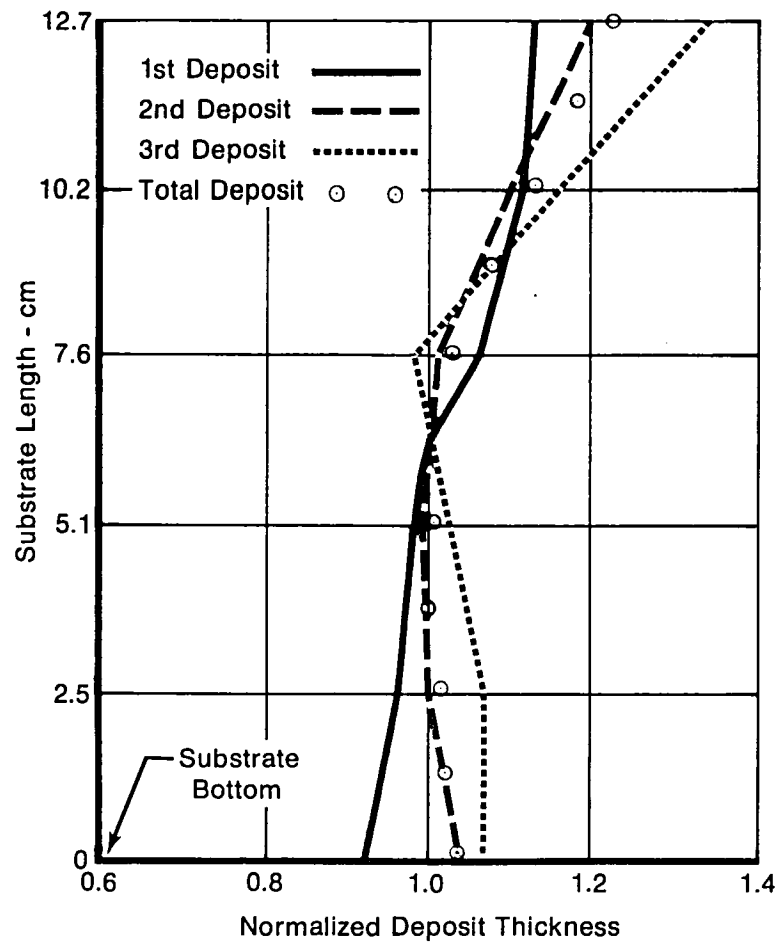


Figure 53. Variation of Copper Closeout Thickness for GF Hollow Cathode Substrate I-36 Closed Out in a 12.7 cm ID Hollow AMZIRC Cathode

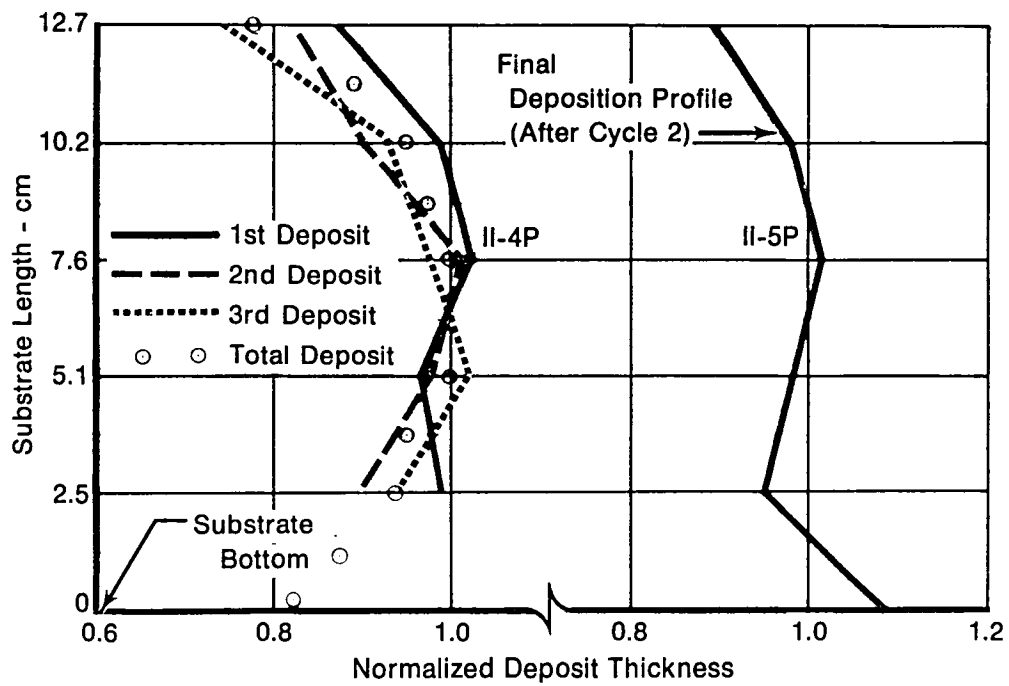


Figure 54. Variation of Copper Closeout Thickness for GF Post Cathode Substrates Using a Shaped Post Cathode. Deposit Not Machined Back Between Layers for II-4P

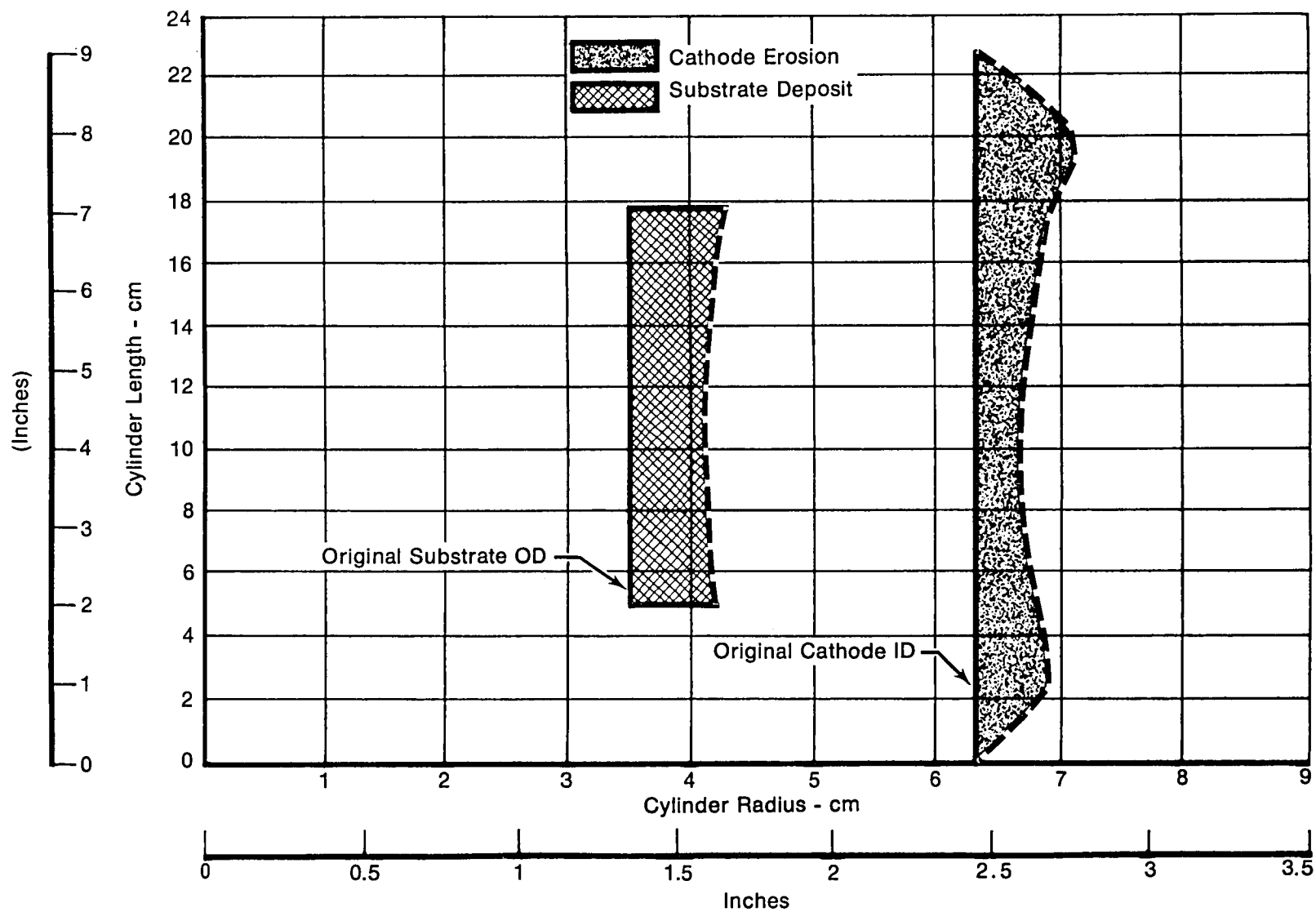


Figure 55. Cathode Erosion and Substrate Deposit Profiles Measured After Closing Out Substrate I-36. Approximately 318 cm³ of AMZIRC Were Sputtered from the Cathode and 194 cm³ Collected on the Substrate for a 61% Collection Efficiency

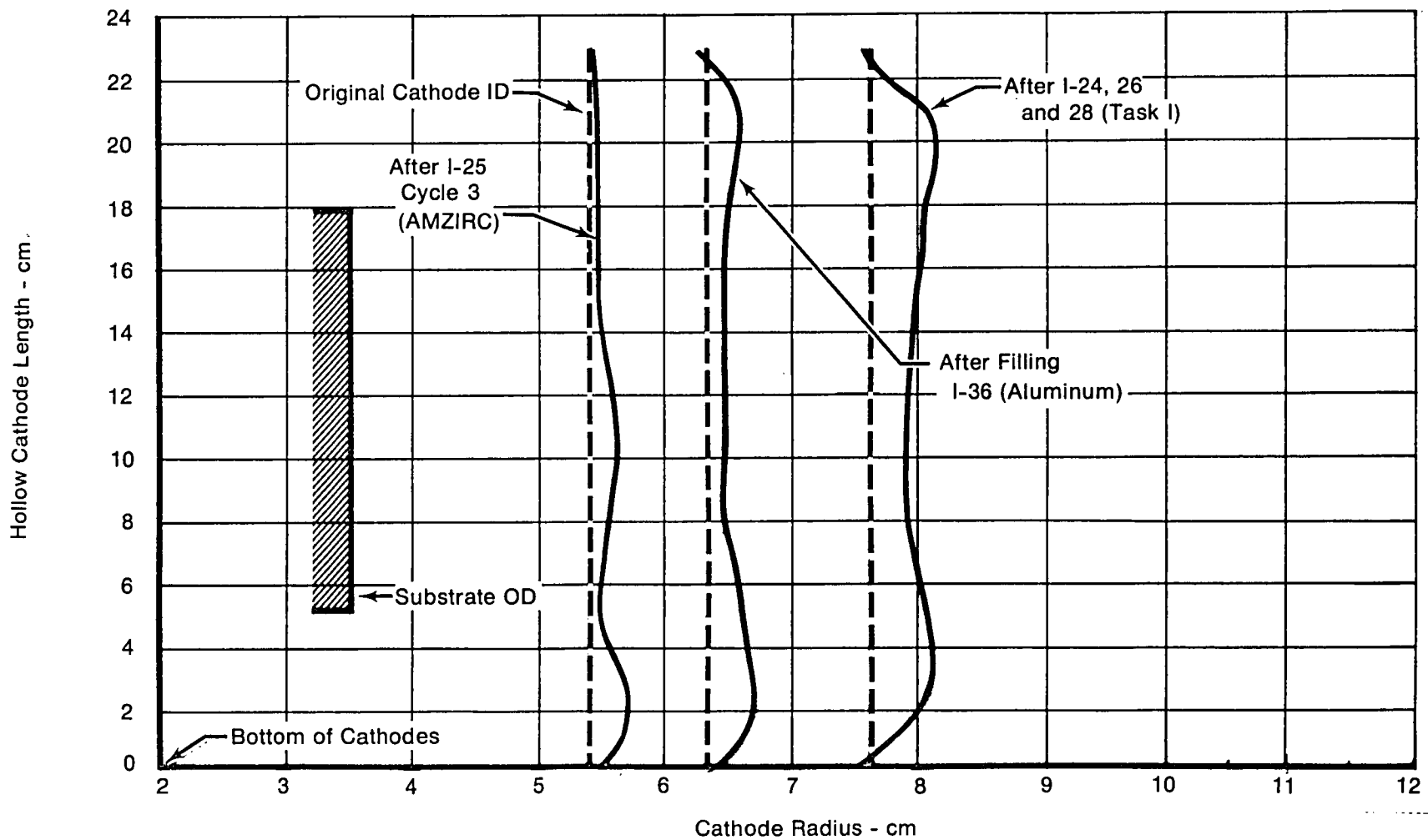


Figure 56. Erosion from Three Sizes of Hollow Cathodes

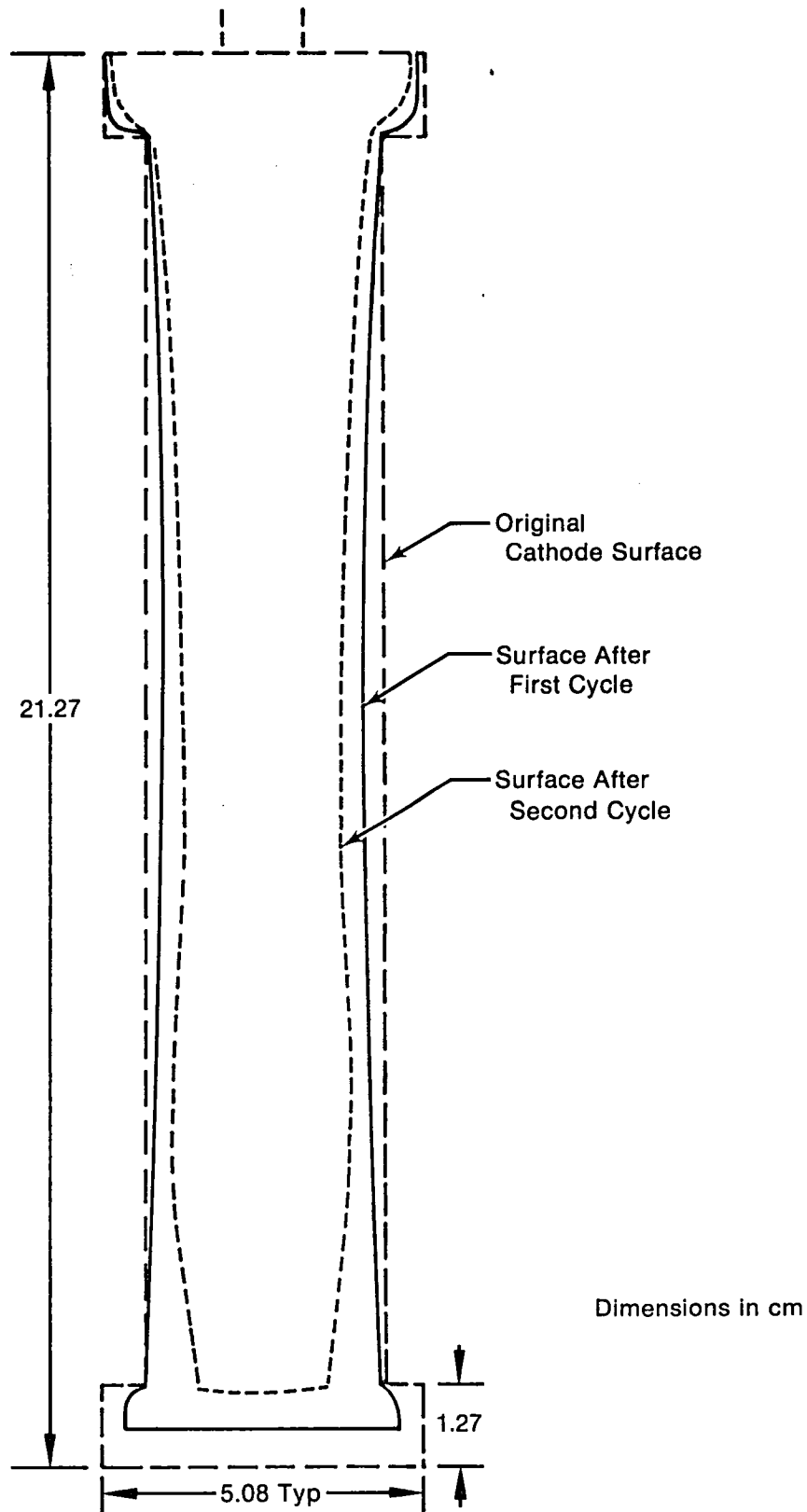


Figure 57. Change in AMZIRC Shaped Post Cathode vs Deposition Time for Closeout Layer Deposition on Substrate II-5P

TABLE 25. SPUTTERED COPPER COLLECTION EFFICIENCY

Substrate Number	Cathode			Amount of Cathode Material Sputtered (cm ³)	Substrate			Amount of Material Collected on substrate (cm ³)	Collection Efficiency (%) ²
	Eroded ID ¹ (cm)	Starting ID (cm)	(in.)		Coated Dia ¹ (cm)	Starting Dia (cm)	(in.)		
I-25 ³	11.092	10.80	4.25	117	8.395	8.026	3.160	60	51
I-25 ⁴	11.798	11.43	4.50	154	8.072	7.530	2.965	84	55
I-36	13.401	12.70	5.00	328	8.336	7.036	2.770	199	61
I-36 ⁵	12.977	12.70	5.00	128	7.673	7.036	2.770	93	73
I-24 ⁶	—	15.24	6.00	—	7.559	7.036	2.770	—	—
I-26 ⁶	—	—	—	—	7.353	7.036	2.770	—	—
I-28 ⁶	15.941	—	—	392	7.465	7.036	2.770	184	47
II-4P	Shaped Post			113	6.749	7.150	2.815	56	50
II-5P	Shaped Post			158	6.477	7.036	2.770	75	48

¹ Average of diameters measured along length of cathode or substrate.

² Quotient of volume of material collected and amount of material sputtered.

³ Measurements made after last deposition cycle (No. 3).

⁴ Measurements made after deposition cycle No. 2.

⁵ For aluminum cathode.

⁶ Deposits made in Task I.

the circumference at the top of the substrates (table 24) than the deposits from straight post cathodes (table 8), but not as even around the circumference at the bottom of the substrates.

Note that the inside diameter of substrate II-4P was measured in situ for deposits 1 and 2 and the bottom of the substrate could not be measured with any degree of accuracy. The octagonal shape of the deposit on substrate II-5P precluded any meaningful measurement during the first deposition cycle. The deposit after deposition cycle 2 on substrate II-5P was *not* octagonal in shape.

Since a relatively large amount of cathode material is needed to close out a substrate, the percentage of sputtered material collected on a substrate, collection efficiency, is of interest. The collection efficiency for several cathode-substrate combinations are shown on table 25. The amount of material collected on a substrate or eroded from a hollow cathode was calculated by using the average diameters of the substrate deposit and cathode erosion profile. The ID of hollow cathodes was measured at every 2.54 cm along the length. Substrate diameters were measured every 1.27 cm along the length. The accuracy of these diameter measurements was such that numerical integration of the profiles, to find volume of material sputtered or deposited, was not warranted. The volume of material sputtered from post cathodes was determined from the difference in the volume of water displaced by a sputtered cathode from that displaced by a new cathode. Note that the total of the average deposit thicknesses for Tasks I and II added up to over 10 cm.

The collection efficiency is shown graphically for substrate I-36 by the scale profiles of the eroded cathode and substrate deposit seen on figure 55.

The erosion profiles for three hollow cathodes are shown to scale on figure 56. The profile shown for the 10.8-cm diameter (5.4-cm radius) was typical of all the hollow cathodes used in systems 1 and 2 for Tasks I and II. The profile shown for the 12.7-cm diameter aluminum cathode is slightly more eroded at the bottom than at the top. This is in contrast to the erosion profile for the 12.7-cm diameter AMZIRC hollow cathode shown in figure 55 which was more heavily eroded at the top. The 15.2-cm diameter OFHC copper hollow cathode used in Task I had a symmetrical erosion profile.

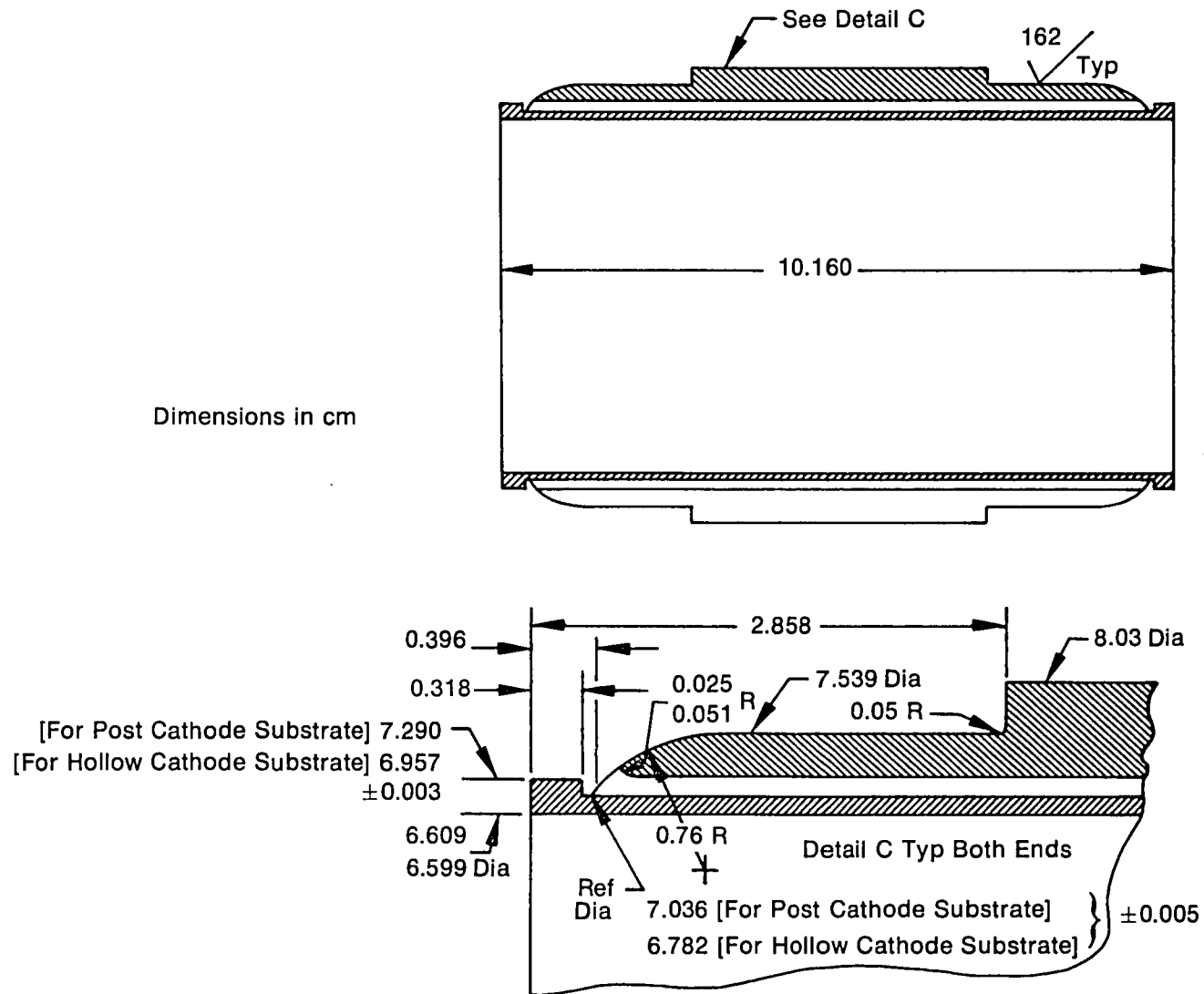


Figure 58. Machining Detail for Closed Out GF Substrates. Redrawn from NASA CF621930

The decrease in diameter shown for the ends of the 12.7- and 15.2-cm diameter cathodes is due to the ground potential shielding used at the end of each cathode. The shielding prevented sputtering of the cathode ends but did not shield the ends from sputtered material.

The erosion profile for a shaped post cathode is shown in figure 57. The profile has been reconstructed from micrometer measurements made on the cathode, and consequently, the skewed shape typical of all post cathodes is not shown. The skewed shape is due to uneven erosion around the circumference of the posts. However, the reconstructed profiles do show the main features of erosion. The final shape shown is also typical of the final shape of straight post cathodes.

The Zr content of the AMZIRC closeout layers on substrates I-25 and I-36 was measured by the electron microprobe. The Zr levels were within the range measured in Task I deposits (table 5). The values measured were 0.17 and 0.19 wt% for the top and bottom, respectively, of substrate I-25 and 0.08 and 0.13 wt% for the top and bottom, respectively, of substrate I-36.

The closed out GF substrates were machined to the dimensions shown on figure 58. These dimensions deviate from the dimensions shown on NASA drawing CF 621930 for the post cathode substrates. The reference diameter for the post cathode substrate was 7.036 cm to correspond to the ID of the GF slotted cylinders. This necessitated changing the OD of the end flanges to 7.29 cm. The dimensions used to machine hollow cathode substrates were as shown on NASA CF 621930.

Photographs of a finished hollow cathode substrate (I-36) and a finished post cathode substrate (II-5P) are shown on figure 59. Substrate I-25 exhibited some cracks in the sputtered material after machining. Cracks were visible in the wrought material on substrate II-4P after machining.

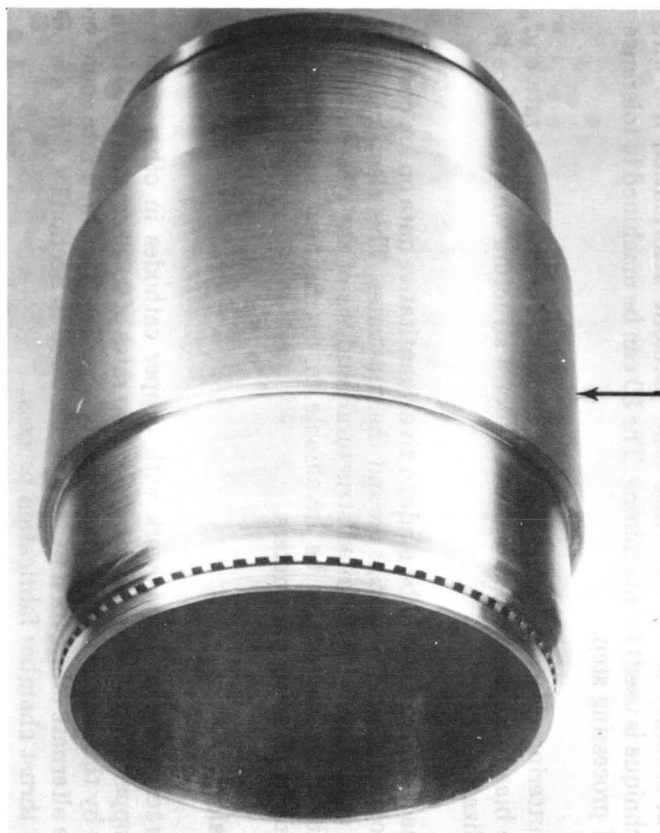
The aluminum filler was leached from the machined closeout substrates with a 7.0 molar NaOH solution at an average temperature of 328°K (130°F) using ultrasonic agitation. Due to the different structure (defects vs no-defects) of the aluminum filler, determination of leaching rate was not possible. However, all channels were cleared in 86.4 ks (24 hr).

D. CONCLUSIONS

Slotted cylinders were successfully closed out by sputtering from hollow and post cathodes. The following additional conclusions are drawn.

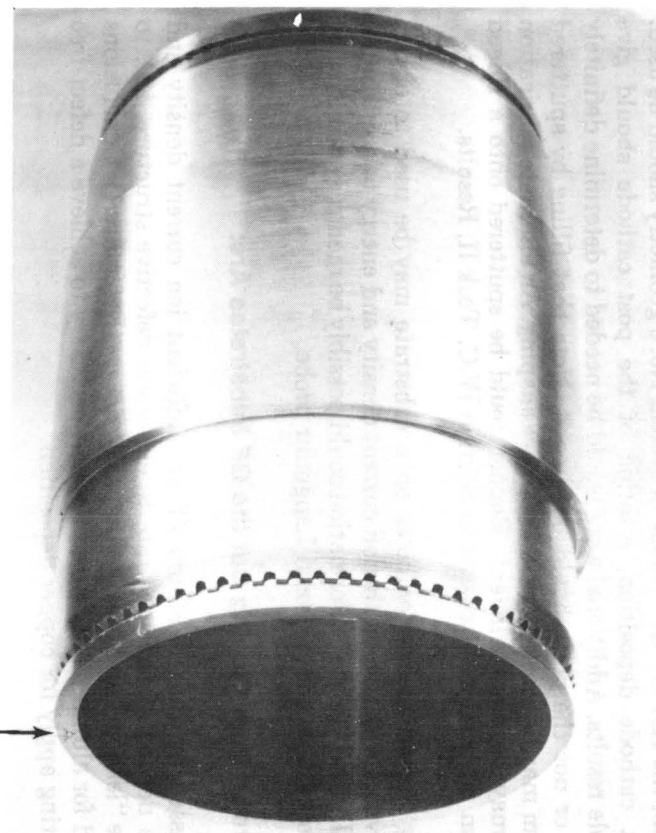
1. Conclusions Drawn from Sputtered Aluminum Filling Study Are:

1. Although it has been demonstrated that grooves may be filled with a single deposition of sputtered aluminum, the filling is time consuming and a more efficient system would make use of aluminum wire or extrusions shaped to fit the groove. Aluminum would be sputtered over the wire to fill the gap between the aluminum wire and the rib wall. The aluminum wire or extrusion filling scheme requires a uniform groove width and preferably grooves which run the full length of the substrate. This and other filling techniques are described in Reference 1.
2. If sputtered aluminum were used for groove filling, the arrival rate of the sputtered material must match the ion current density and energy available to the substrate to ensure a defect-free filling made in one deposition cycle. As a practical matter, this requirement dictates the use of sputtering geometry



FAL 51067

GF Substrate I-36



FAL 51066

GF Substrate II-5P

Sputtered
AMZIRC

Figure 59. GF Substrates After Machining and Leaching Aluminum Filler

that will give an even deposit thickness on the substrate. For hollow cathode deposition, the 12.7-cm ID cathode in system No. 3 geometry should be used. For post cathode deposition, rotation of the post cathode should give reasonable results. Additional work would be needed to determine definitely whether or not the post should be shaped. Note that filling by sputtered aluminum may be more efficient by groove shaping. An alternate fabrication process may be more efficient. Copper could be sputtered onto a ridged aluminum mandrel as discussed in Section IV.C, Task II, Results.

3. Specially shaped grooves or holes on a substrate may be used to obtain qualitative information on the ion current density and energy to a substrate. This qualitative "probe" information could possibly be made quantitative by calibration with a conventional Langmuir probe.

2. Conclusions Drawn from Closing Out the GF Substrates Are:

1. As discussed for the aluminum filling, sufficient ion current density and energy is needed at the substrate to eliminate defective structure, i.e., to eliminate "leaders" and open columnar structure (Reference 1). Conclusions discussed for aluminum filling equipment necessary to achieve a defect-free even coating apply for copper deposition.
2. An even coating is desirable on closeout layers for additional reasons. Copper substrates can be stressed out of shape by the difference in closeout layer thickness. End caps used to hold hollow cathode substrates on the water cooling mandrel, may be deformed away from the O-rings to cause loss of vacuum integrity. The deformation is caused by the growth of the thick sputtered deposit along the length of the substrate.
3. The 0.093-cm thick wall between the bottom of the grooves and the ID of a hollow cathode substrate should be increased in thickness to give additional mechanical stability to the substrate (and preclude deformation if a wire filling technique is used) for machining. The ID can be machined to tolerance in a final processing step.
4. When sputtering closeout layers of OFHC copper, the deposition rate and substrate bias must be chosen to give a uniform structure with reference to recrystallization.
5. Probe measurements should be made to give quantitative data on ion current density to and plasma potential around the substrates. This electrical data could be correlated with detailed structural analysis of the closeout layer. This probe data would also be invaluable in determining substrate ion bombardment cleaning parameters.

3. Further Conclusion

Since this contract was completed, work with copper cathodes in other equipment has demonstrated good copper deposit structure at deposition rates of 0.010 cm/hr (0.004 in./hr). The rate was limited only by the capacity of available power supplies. This high rate could probably be combined with the alternate fabrication technique discussed in Section IV.C for a more viable regeneratively cooled thrust chamber fabrication process.

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