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COMPATIBILITY OF COLUMBIUM BASE ALLOYS WITH LITHIUM FLUORIDE

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Prepared by
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for Lewis Research Center

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16. Abstract

The compatibility of lithium fluoride with several columbium base alloys was studied by testing capsules under the cyclic thermal conditions which simulate the sun-shade cycle of a heat receiver in a 300-nautical mile orbit. Capsules fabricated from the columbium alloys Cb-1Zr, FS-85 (28Ta-10.5W-0.9Zr-bal. Cb) and SCb-291 (10Ta-10W-bal. Cb) containing lithium fluoride were cycled between 1500° and 1900° F for times up to 5000 hours in a high vacuum environment.

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FOREWORD

The work described herein was done at the General Electric Company, Space Division, under NASA Contract NAS3-8523 with Mr. J. A. Milko, Space Power Systems Division, NASA-Lewis Research Center, as Technical Manager.

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I. SUMMARY

Posttest evaluation of capsules fabricated from the columbium alloys Cb-1Zr, FS-85 (28Ta-10.5W-0.9Zr-Bal Cb) and SCb-291 (10Ta-10W-Bal. Cb) which were cycled between 1500° and 1900°F for times up to 5000 hours revealed no evidence of corrosion. No significant change in microstructure, chemical analysis, or tensile strength was observed. The only significant change was the ductile-to-brittle transition temperature of the FS-85 alloy. A comparison of the data obtained on the specimens which were exposed to vacuum and specimens removed from the capsule (shown in Table XII) indicate the lithium fluoride exposure has a definite effect on raising the ductile-to-brittle transition temperature FS-85.

Although no detrimental effects of lithium fluoride on Cb-1Zr were observed with respect to corrosion, chemistry, or physical properties, the capsules were severely distorted after 5000 hours of testing. It is believed the deformation occurred as a result of the melting and freezing of the lithium fluoride and the concomitant 30 percent change in volume during each thermal cycle.

In conclusion, the columbium alloys Cb-1Zr, FS-85, and SCb-291 are compatible with lithium fluoride at the temperatures tested, but strength requirements should be given special consideration when parts are to see thermal cycles between 1500°F and 1900°F for extended periods of time.

II. INTRODUCTION

A study of the compatibility of lithium fluoride with several columbium base alloys was conducted by testing capsules under the cyclic thermal conditions which simulate the sun-shade cycle of a heat receiver in a 300-nautical mile orbit. Capsules fabricated from the columbium alloys Cb-1Zr, FS-85 (28Ta-10.5W-0.9Zr-Bal. Cb) and SCb-291 (10Ta-10W-Bal. Cb) containing lithium fluoride were cycled between 1500° and 1900°F for times up to 5000 hours in a high vacuum environment.

The effects of exposure to lithium fluoride under these conditions were determined by chemical analysis, metallography, tensile strength, and determination of ductile-to-brittle transition temperature of weld specimens removed from the capsules and specimens exposed in vacuum to the same thermal conditions as the capsules. The capsules were so constructed, that welds as well as the parent metal could be evaluated.

The columbium alloys Cb-1Zr, FS-85, and SCb-291 were found to be compatible with lithium fluoride at the temperatures tested.

III. MATERIAL PROCUREMENT

A. Capsule Material

Specifications for the procurement of Cb-1Zr and FS-85 alloy were updated to reflect the latest developments in the state-of-the-art and a new specification was prepared for the SCb-291 alloy.

The SCb-291 alloy sheet material was produced by Wah Chang Corporation, Albany, Oregon. The chemical analysis and mechanical properties (vendor's test) of the 0.030-inch SCb-291 alloy sheet are presented in Tables I and II, respectively. Similar data for the 0.030-inch Cb-1Zr sheet produced by Kawecki Chemical Company, New York, are presented in Tables III and IV.

Two 24-inch x 24-inch x 0.030-inch sheets of FS-85 alloy were obtained from the Navy. This material was produced by Fansteel Metallurgical Corporation, North Chicago, Illinois, under Navy Contract NOw 65-0498-f. Complete processing information for this material, Heat No. 85D1525, is presented in the final report of the program.[(1)] The chemical analysis, Table V, and mechanical properties, Table VI, of this sheet conform to the allowable limits in the GE-NSP specification for FS-85, 01-0020-00-C.

B. Lithium Fluoride

The lithium fluoride for this program was ordered from the Optical Crystal Department, Harshaw Chemical Company, Cleveland, Ohio. The material was in the form of cleaved, single-crystal pieces (5 to 15 mm on an edge) taken from the centers of three optical grade single-crystal ingots which had been grown in platinum molds. Cleaving was performed under an inert gas cover. The material was shipped, double packaged

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(1) Fabrication of Fansteel 85 Metal Sheet, Fansteel Metallurgical Corporation, Contract NOw 65-0498-f, March 1966.

TABLE I. CHEMICAL ANALYSIS OF SCb-291 ALLOY[(a)] 0.030-INCH SHEET

(Wah Chang Heat 94047)

Element	Vendor Analysis	GE-NSP Analysis[(b)]
	Concentration, ppm (Unless Otherwise Noted)	
Oxygen	< 50	38, 20
Carbon	< 40	9, 20
Nitrogen	< 25	3, 4
Hydrogen	3	< 1, < 1
Aluminum	< 20	
Boron	< 1	
Cadmium	< 5	
Cobalt	< 10	
Chromium	< 20	
Copper	< 40	
Iron	50	
Magnesium	< 20	
Manganese	< 20	
Nickel	50	
Lead	< 20	
Silicon	< 50	
Tin	< 10	
Titanium	< 40	
Vanadium	< 20	
Hafnium	< 80	
Tungsten	10.1 w/o	
Tantalum	11.66 w/o	
Columbium	Bal.	

(a) Cb-10W-10Ta, recrystallized, ASTM grain size 6.5.

(b) Duplicate analysis.

TABLE II. MECHANICAL PROPERTIES OF SCb-291 ALLOY[a] 0.030-INCH SHEET

(Wah Chang Heat 94047)

Tensile Tests

Ultimate Tensile Strength psi	Yield Strength psi	Elongation %
59,200	40,500	25
58,200	39,900	24

Stress-Rupture Tests

Temperature, °F	Stress, psi	Time, Hours	Elongation, %
2200	10,000	2.3	50
2200	10,000	3.4	66

[a] Cb-10W-10Ta, recrystallized, ASTM grain size 6.5.

TABLE III. CHEMICAL ANALYSIS OF Cb-1Zr 0.030-INCH SHEET[a]

(Kawecki Heat 37507)

Element	Concentration, ppm (Unless Otherwise Noted) Ingot	Finished Product
Oxygen	120	100
Carbon	35	45
Nitrogen	< 10	10
Hydrogen	3	6
Iron	10	
Tantalum	800	
Titanium	< 10	
Silicon	10	
Zirconium	1.04 w/o	

(a) Recrystallized, ASTM grain size 7.5, vendor's analysis.

TABLE IV. MECHANICAL PROPERTIES OF Cb-1Zr 0.030-INCH SHEET[a]

(Kawecki Heat 37507)

Tensile Tests

Ultimate Tensile Strength psi	Yield Strength psi	Elongation %
37,600	25,200	28
37,700	27,700	37

Stress-Rupture Tests

Temperature, °F	Stress, psi	Time, Hours	Elongation, %
2200	10,000	15.6	39
2200	10,000	15.1	48

(a) Recrystallized, ASTM grain size 7.5.

TABLE V. CHEMICAL ANALYSIS OF FS-85 SHEET[a]

	(Fansteel Heat 85D1525)				
	Concentration, ppm (Unless Otherwise Noted)				
				Allowable in Specification[b]	
Element	Ingot	Sheet, Bar	Sheet	Minimum	Maximum
Carbon	10	10	22	--	100
Nitrogen	50	30	36	--	75
Oxygen	50	59	55	--	100
Hydrogen	5	8	1	--	10
Nickel	--	10	--	--	50
Iron	50	70	70	--	100
Chromium	--	10	--	--	100
Silicon	50	100	50	--	100
Tantalum	26.88 w/o	27.98 w/o	27.72 w/o	26 w/o	29 w/o
Tungsten	10.10 w/o	10.4 w/o	10.58 w/o	10 w/o	12 w/o
Zirconium	0.87 w/o	0.95 w/o	0.94 w/o	0.6 w/o	1.1 w/o

(a) Fabrication of Fansteel 85 Metal Sheet, Fansteel Metallurgical Corporation, Contract NOw-65-0498-f, March 1966.

(b) NSP Specification Number 01-0020-00-C, Sheet, Plate, and Strip: FS-85 (Cb-28Ta-10.5W-0.9Zr) Alloy, July 18, 1966.

TABLE VI. ROOM TEMPERATURE TENSILE PROPERTIES OF FS-85 SHEET[a]

(Fansteel 85D1525)

Specimen	Ultimate Tensile Strength psi	Yield Strength psi	Elong. %	$E \times 10^{-6}$ psi
Longitudinal	86,800	67,400	24	19.5
	85,500	71,400	21	20.1
Transverse	89,200	73,800	23	20.2
	89,400	74,100	21	20.3
Minimum Allowable in Specification[b]	70,000	50,000	20	--
Maximum Allowable in Specification[b]	100,000	80,000	--	--

(a) Fabrication of Fansteel 85 Metal Sheet, Fansteel Metallurgical Corporation, Contract NOw 65-0498-F, March 1966.

(b) NSP Specification Number 01-0020-00-C, "Sheet, Plate, and Strip: FS-85 (Cb-28Ta-10.5W-0.9Zr) Alloy," July 18, 1966.

in polyethylene, in a metal container containing a silica gel dessicant. Emission spectrographic analysis supplied on each ingot from which the material was taken is shown in Table VII.

A stainless steel container of high-vacuum design was constructed for storage of the lithium fluoride. This container was fitted with a Varian copper-gasketed flange and a bakeable valve. Subsequently, the container was sealed, and the lithium fluoride crystals were heated and outgassed under vacuum

IV. EXPERIMENTAL PROCEDURE

A. Capsule Fabrication

A 40° circular ring sector capsule is shown schematically in Figure 1.

Selection of this design was based on the ability of the capsule arrangement, shown in Figure 2, to simulate the sun-shade cycle. The weld specimen shown between the capsules was comparatively evaluated with the capsule material after testing. The formed capsule segments and weld specimen blanks are shown in Figure 3. After forming, the capsule segments were pickled in accordance with NSP Specification number 03-0021-00-A. They were then welded longitudinally by the automatic tungsten inert gas process. The end caps were welded in place manually. Sheet specimens of each alloy were similarly cleaned and welded along with, and in the same manner as the capsule segments.

All welding was performed in accordance with NSP Specification No. 03-0025-00-A, "Welding of Columbium, Tantalum, and Their Alloys by the Inert Gas Tungsten Arc Process." The welds were visually and radiographically inspected as described in the welding specification and found to be sound. All the capsules passed helium mass spectrometer leak testing according to NSP Specification No. 03-0013-00-B. Following welding, the Cb-1Zr and SCb-291 alloy capsules were annealed at 2200°F for 1 hour, and the FS-85 alloy capsules were annealed at 2400°F for 1 hour. The weld specimens were annealed along with the capsules.

Two ASTM standard tensile specimens of the design shown in Figure 4 were installed in each capsule. Wire holders, made of the same material as the capsule, were used to support the specimens.

TABLE VII. LITHIUM FLUORIDE SPECTROGRAPHIC ANALYSIS

Element	Ingot 1 ppm	Ingot 2 ppm	Ingot 3 ppm	Detection Limits ppm
Al	3	1-2	1-2	-
Sb	ND*	ND	ND	10
As	ND	ND	ND	100
Ba	ND	ND	ND	100
Be	ND	ND	ND	1
Bi	ND	ND	ND	1
B	ND	ND	ND	1
Cd	ND	ND	ND	10
Ca	1-2	3	1-2	-
Cr	< 1	< 1	< 1	-
Co	ND	ND	ND	1
Cu	ND	ND	ND	1
Ga	ND	ND	ND	1
Ge	ND	ND	ND	1
Au	ND	ND	ND	1
In	ND	ND	ND	1
Fe	3	ND	1-2	-
La	ND	ND	ND	10
Pb	ND	ND	ND	1
Mg	3	3	3	-
Mn	ND	ND	ND	1
Hg	ND	ND	ND	10
Mo	ND	ND	ND	1
Ni	ND	ND	ND	1
P	ND	ND	ND	1000
Pt	ND	ND	ND	10
Si	3	3	3	-
Ag	ND	ND	ND	1
Na	ND	ND	ND	100
Sr	ND	ND	ND	10
Ti	ND	ND	ND	1
Sn	ND	ND	ND	1
W	ND	ND	ND	100
V	ND	ND	ND	1
Zn	ND	ND	ND	100
Zr	ND	ND	ND	1

* ND - Not Detected

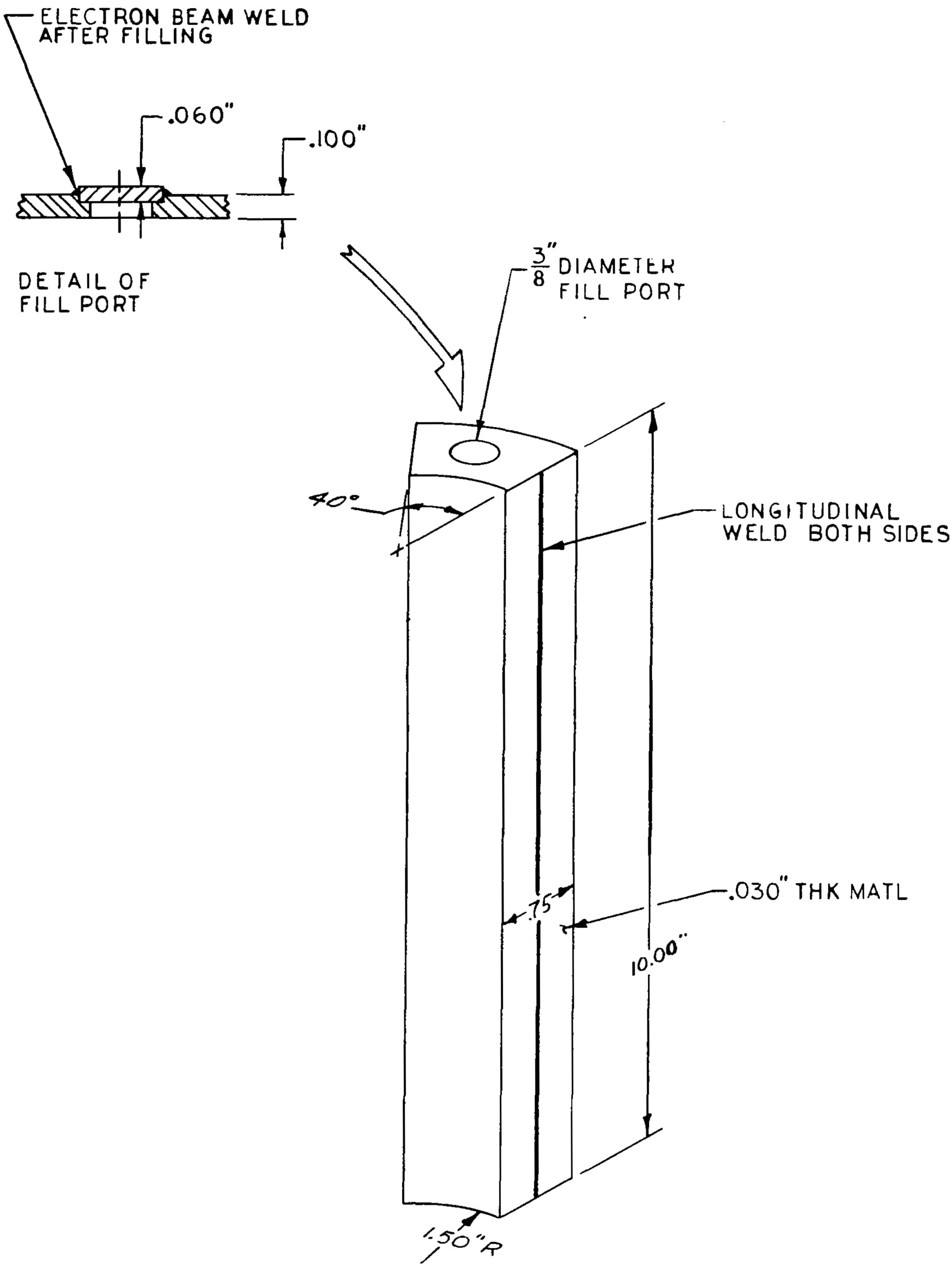


Figure 1. Lithium Fluoride-Columbium Base Alloy 40° Circular Ring Sector Corrosion Capsule.

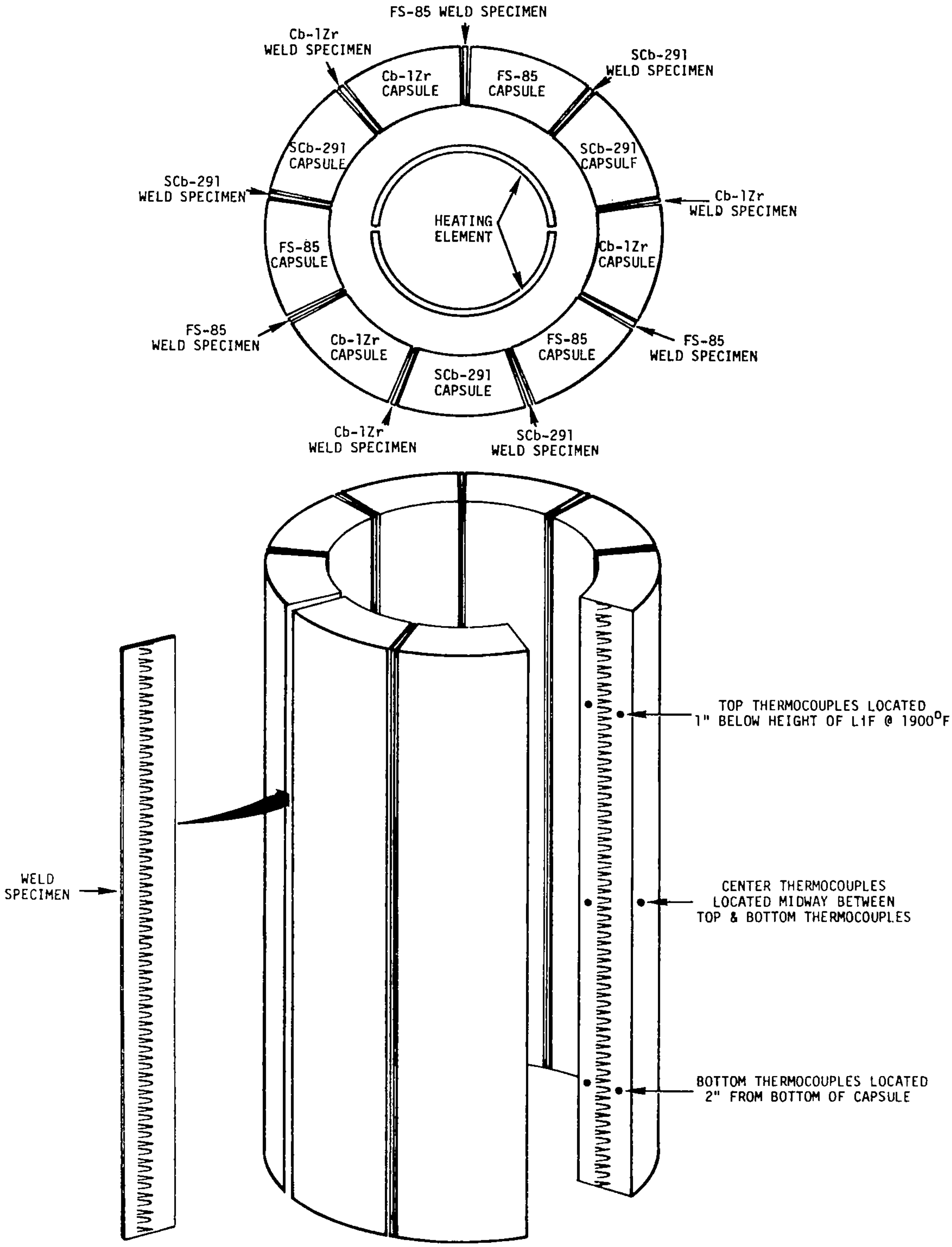


Figure 2. Lithium Fluoride 40° Circular Ring Sector Capsule Test.

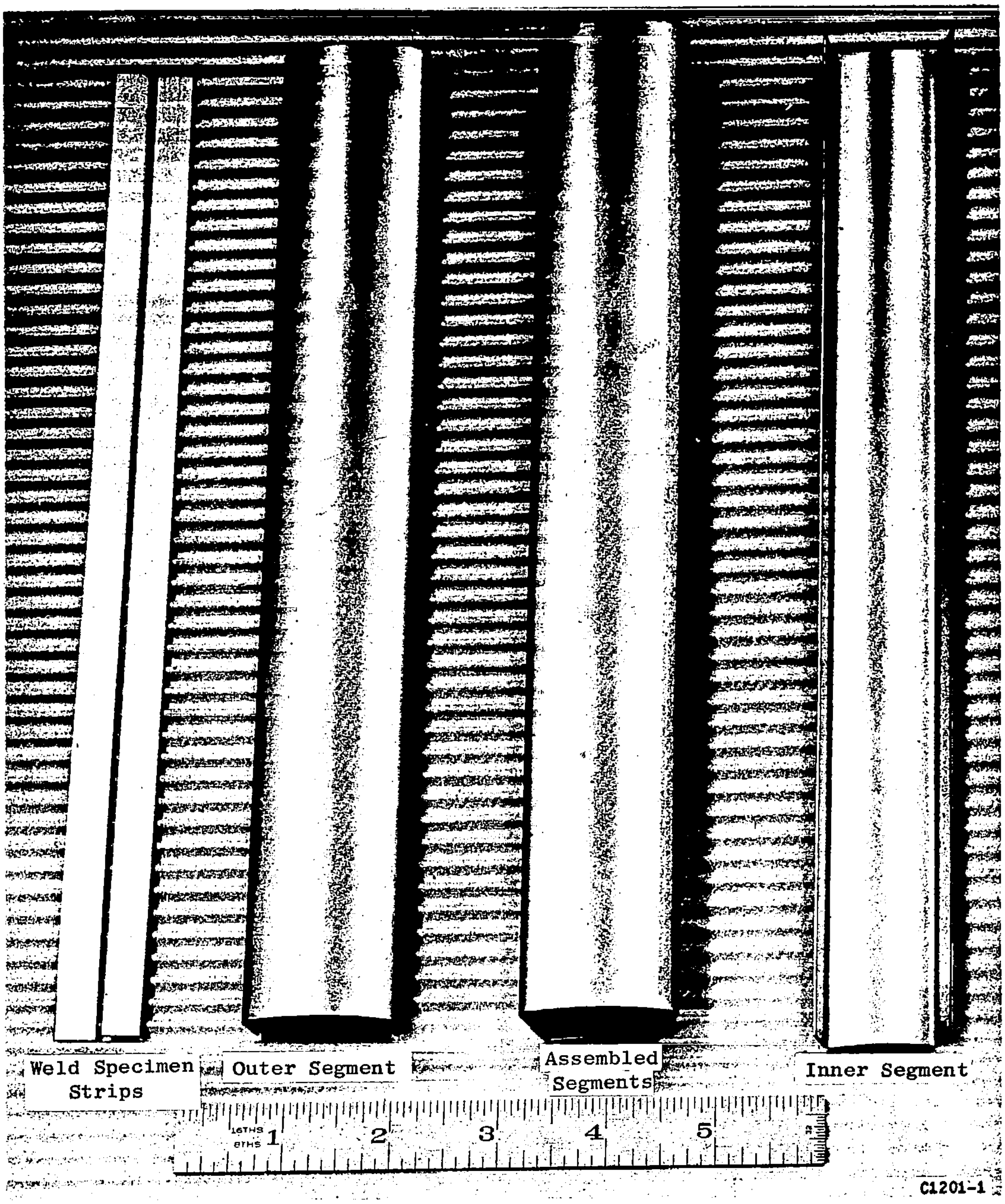


Figure 3. Lithium Fluoride 40° Ring Sector Capsule Segments and Weld Specimen Blank Before Welding. (Orig. C67021526)

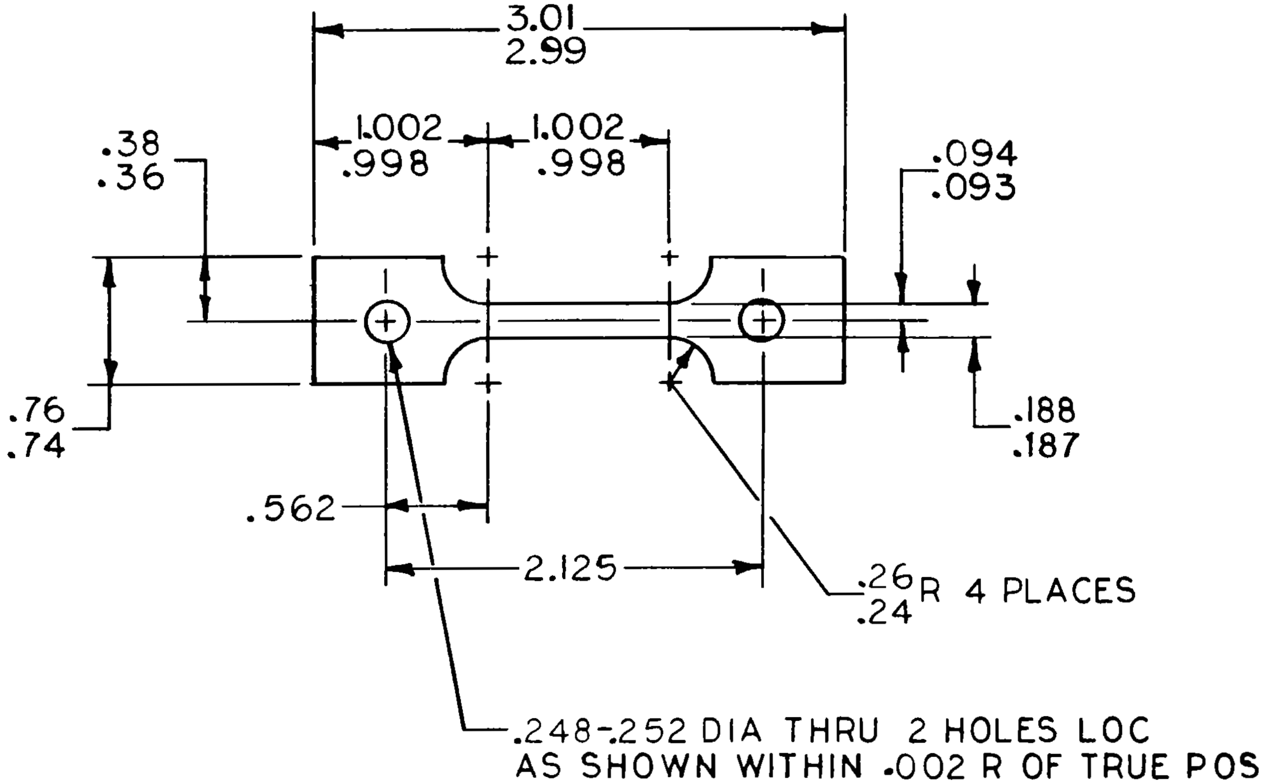


Figure 4. ASTM Standard Tensile Test Specimen.

B. Capsule Filling

Following welding and annealing, the 40° ring sector capsules were filled with lithium fluoride and subsequently sealed by electron beam welding.

The capsule filling apparatus shown assembled in the welding chamber in Figure 5 and schematically in Figure 6 employed two separately controlled split tantalum resistance heating elements. The lower capsule heater maintained the capsules during filling between 1550°F and 1650°F to keep the lithium fluoride from solidifying while the upper funnel heater supplied heat to melt the lithium fluoride crystals into each capsule.

The capsule and associated shielding rotated around the heater on a ball bearing supported table. Each capsule was positioned beneath the funnel by means of a positioning pin inserted in holes drilled in the revolving table. This method was required as the position of the capsules could not be determined visually inside the welding chamber, and precise positioning of the capsule under the funnel was critical.

The pressure in the chamber was maintained below 5×10^{-5} torr during melting of the lithium fluoride. As a result, loading the lithium fluoride crystals into the funnel and rotating the capsules had to be performed with the manipulators shown in Figure 7. The lithium fluoride crystals were preweighed into stainless steel beakers which were held on a rotating table in a position accessible with the manipulator. The foil shown in Figure 7 protected the gloves and welding hose from excessive heating by radiation from the filling facility. The filled capsules were allowed to cool before the chamber was backfilled with helium. The capsules were removed from the filling apparatus, sealed in the transfer container, and transferred to the electron beam welding chamber where they were subsequently sealed by electron beam welding at a vacuum of 1×10^{-5} torr.

C. Test Facility

The lithium fluoride 40° ring sector capsule test was conducted in an 18-inch-diameter Varian ultrahigh vacuum chamber. The complete 40° ring sector capsule test facility is shown in Figure 8.

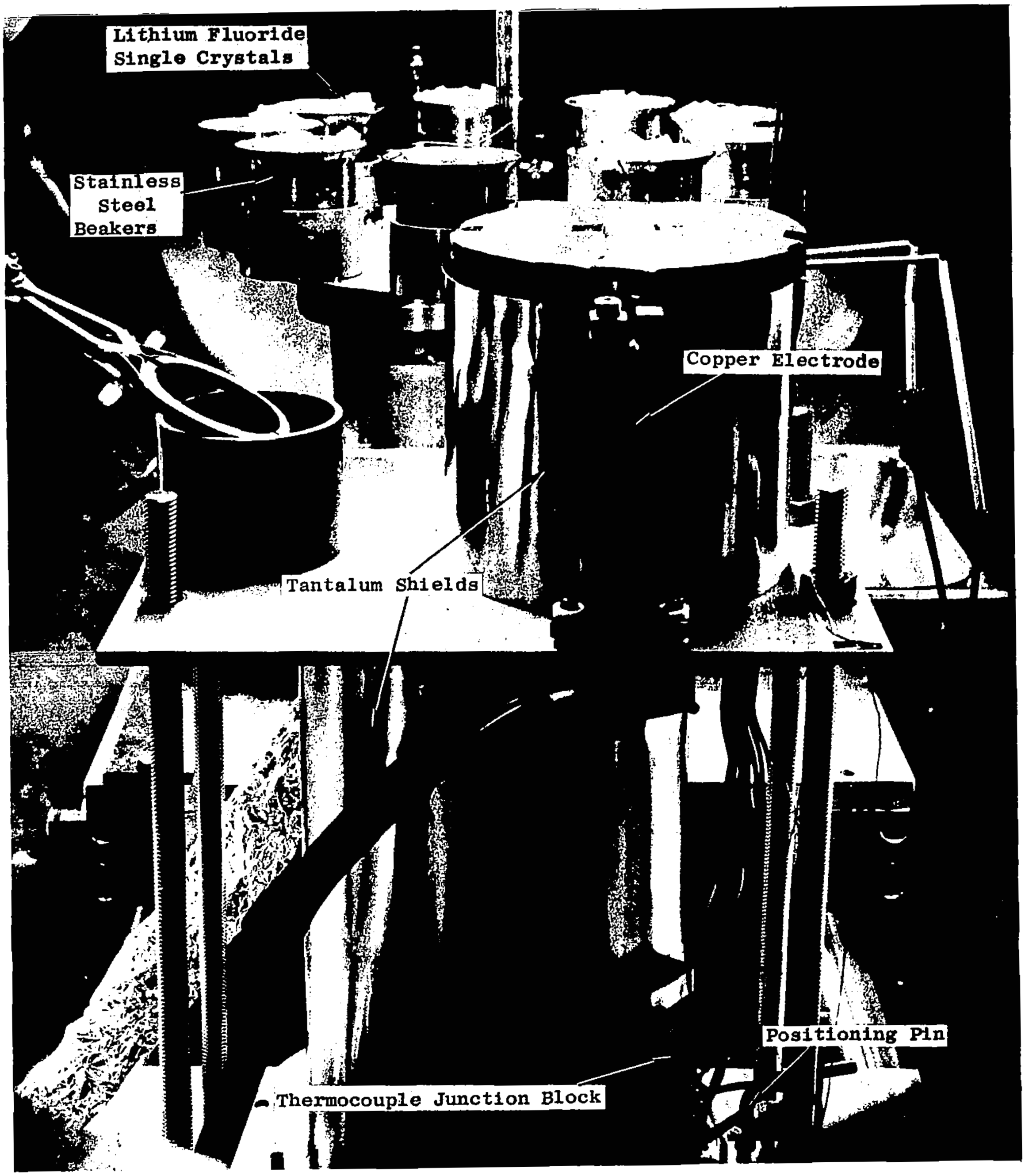


Figure 5. The Lithium Fluoride 40° Ring Sector Capsule Filling Facility. (Orig. C67040548)

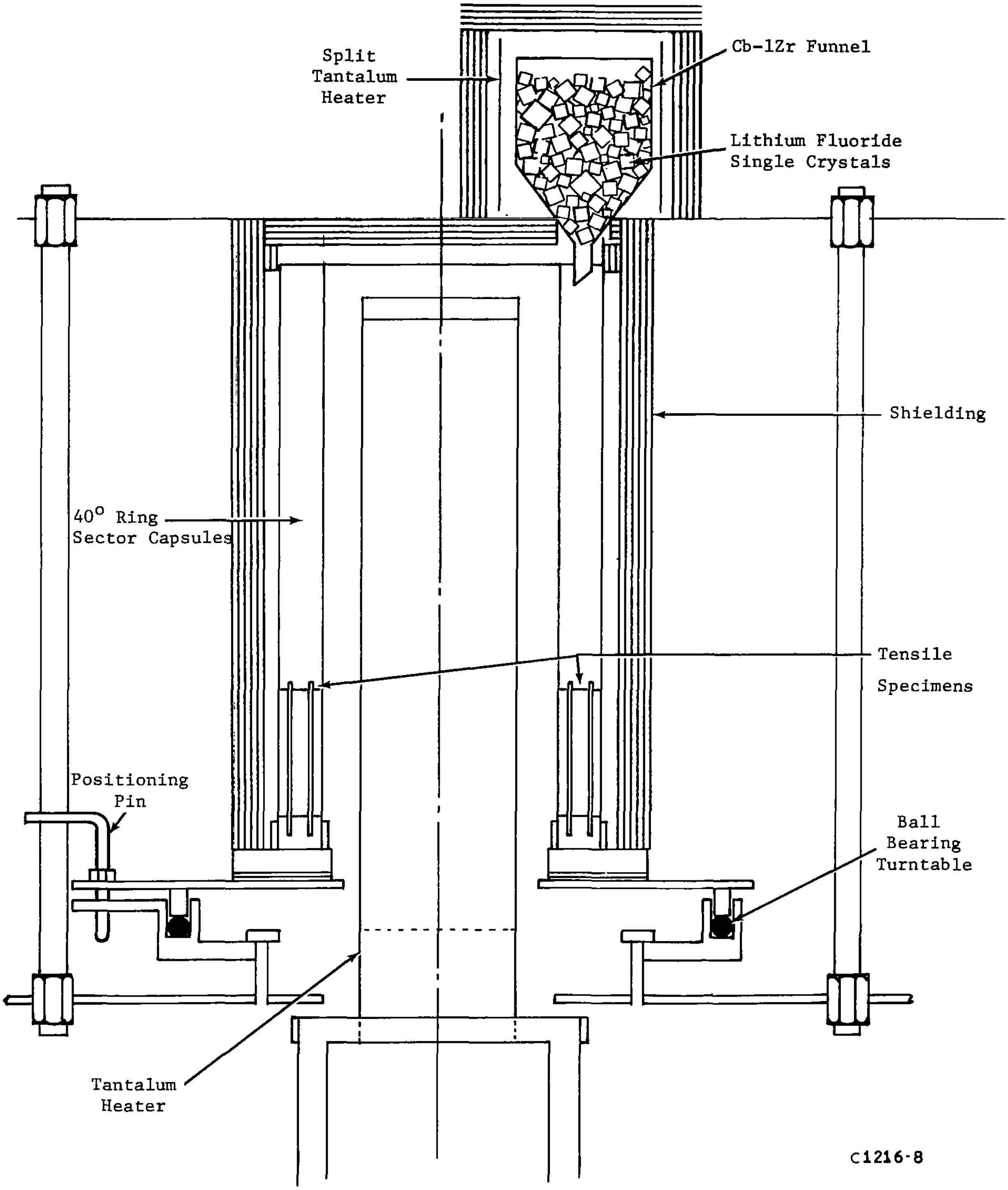


Figure 6. The Lithium Fluoride Capsule Filling Facility Schematic Diagram.

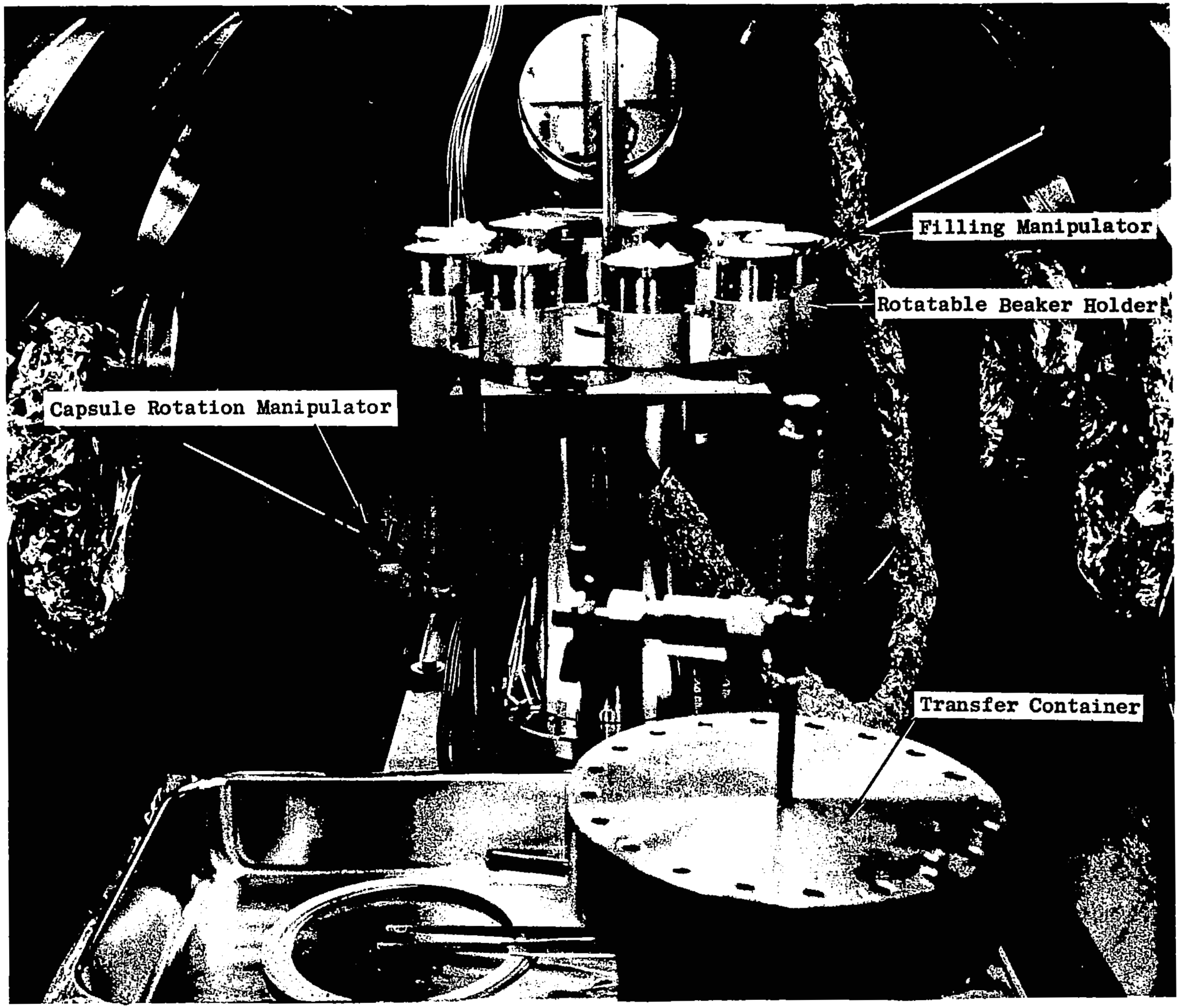


Figure 7. The Lithium Fluoride 40° Ring Sector Capsule Filling Facility. (Orig. C67040546)

C1216-9

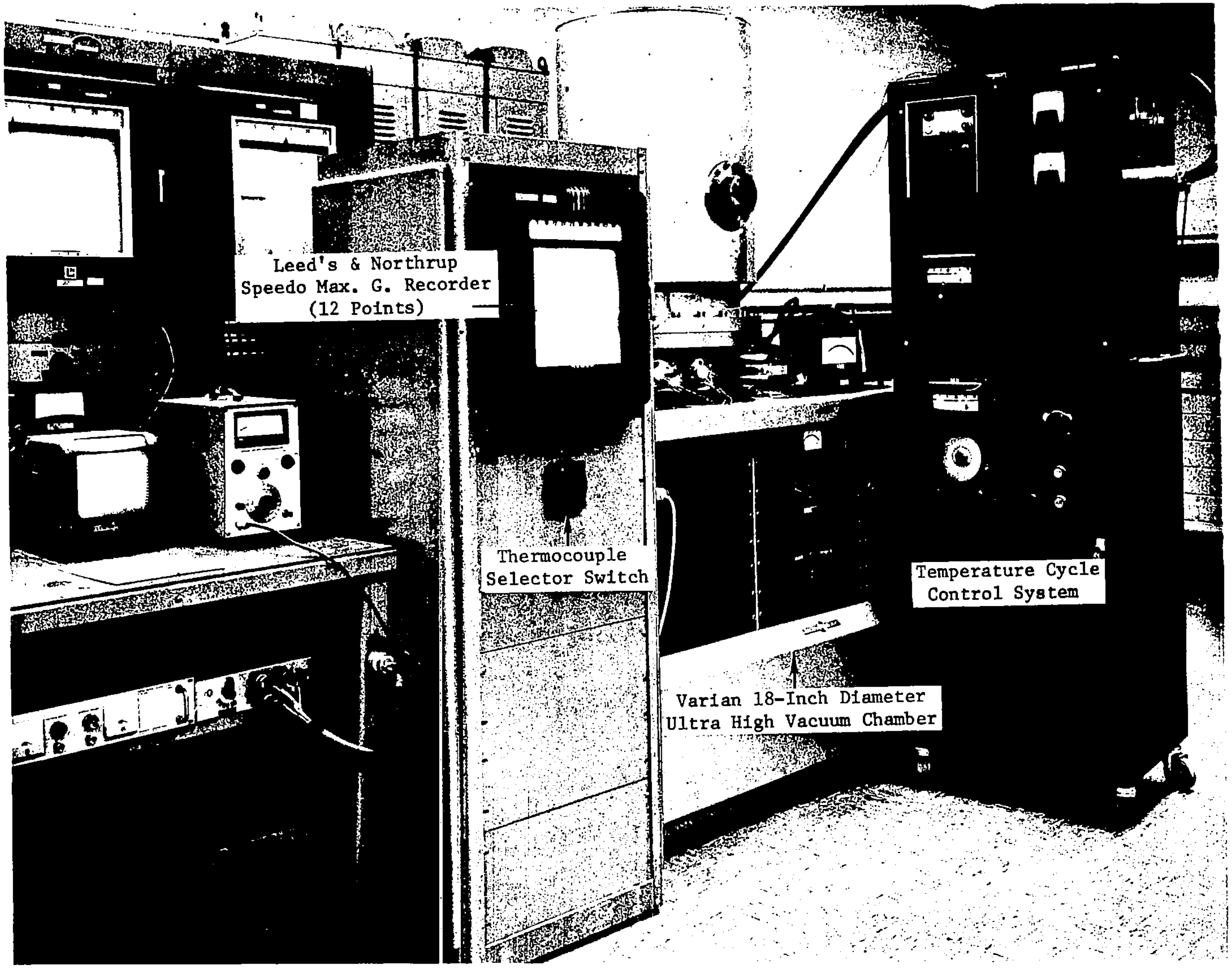


Figure 8. The Lithium Fluoride 40° Ring Sector Capsule Test Facility. (Orig. C68022941)

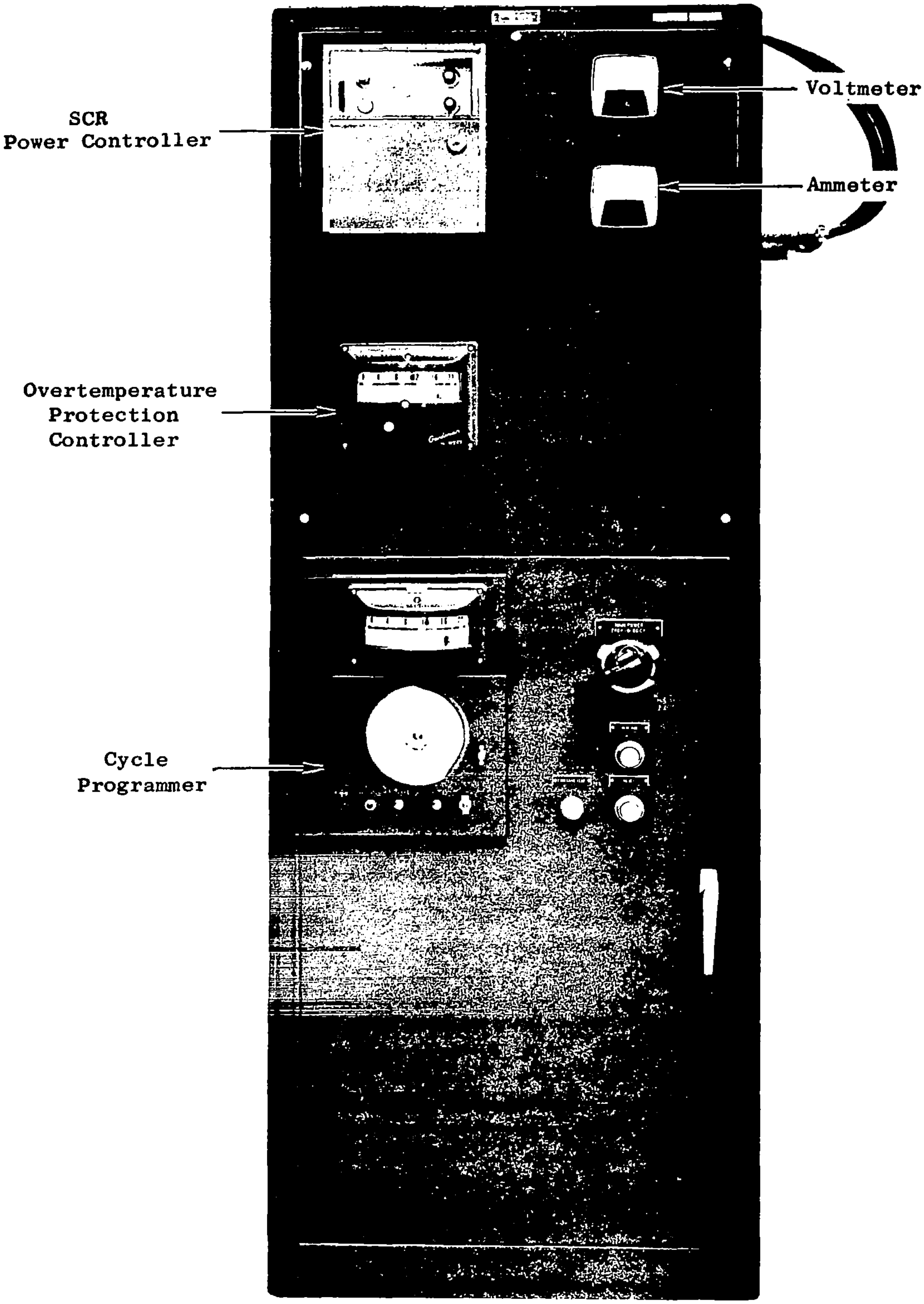


Figure 9. Temperature Cycle Control System Lithium Fluoride 40° Ring Sector Capsule Test. (Orig. C68022940)

1. Temperature Control System

The power for the tantalum heater was provided by means of a 10 kva step down transformer which reduced the line voltage from 220 v to 12 v. The line voltage which regulates the temperature was controlled by the temperature cycle control system, shown in Figure 9 which consisted of a cam-operated temperature programmer driving a silicon-controlled rectifier power regulator. The cam, shown in the temperature programmer in Figure 10, made one complete revolution every 96 minutes and was cut to provide a power on - power off cycle. During the melting and heating cycle, a constant power value was applied, capable of heating the capsule to 1900°F in one hour. This was accomplished by having the cam-actuated control set point well above the temperature indicator during the heating cycle, thereby, the programmer was constantly calling for power. The power level was in turn set by the current limiter adjustment in the silicon-controlled rectifier power controller. By applying a constant power throughout the heating cycle, the thermal conditions of the capsule better simulated the sun cycle of a heat receiver in a 300-nautical-mile orbit. After 60 minutes the cam lowered the set point to well below the indicated millivolt output of the control thermocouple and shut the power off. The low point of the cycle was selected to assure that all the lithium fluoride in the capsule had frozen before the power came on for the next cycle.

Power to the capsule heating element was determined by measuring the voltage with a standard voltmeter (± 3 percent) and measuring the current with a Weston current transformer and an ammeter (± 0.5 percent) as shown in Figure 11.

2. Thermocouple Instrumentation

By incorporating a thermoelectrically cooled constant temperature (32°F) reference junction in the thermocouple circuitry, as shown in Figure 12, corrected temperatures were plotted on the recorders. Temperatures were periodically checked with a potentiometer by means of the thermocouple switches, shown in Figure 13, which switch the thermocouple input from the recorder into the potentiometer.

Each of the nine 40° ring sector capsules was instrumented with three W-3Re/W-25Re thermocouples as shown in Figure 14. The thermocouple

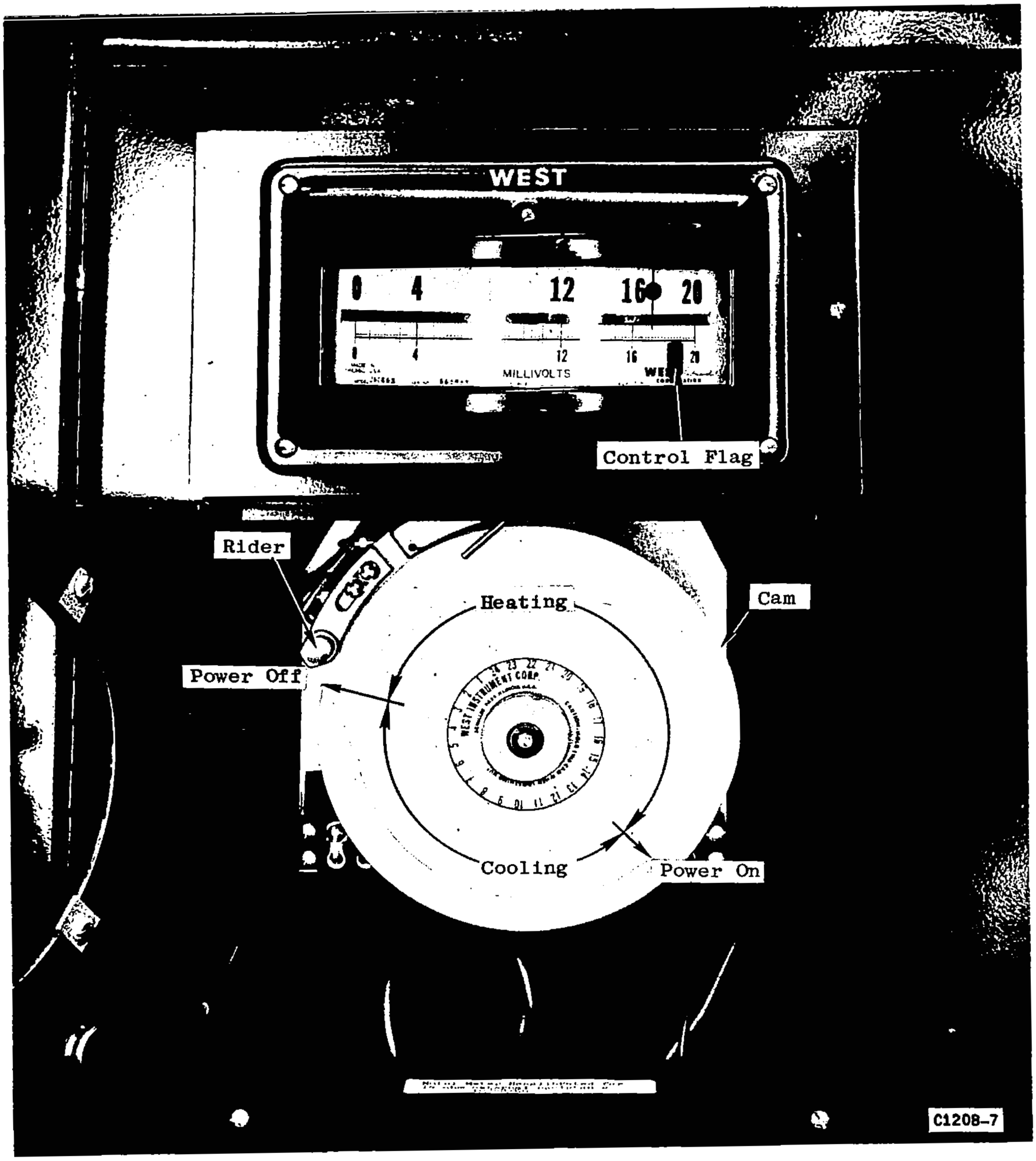


Figure 10. Cam Controlled Temperature Programmer - Lithium Fluoride 40° Ring Sector Capsule Test. (Orig. C67031427)

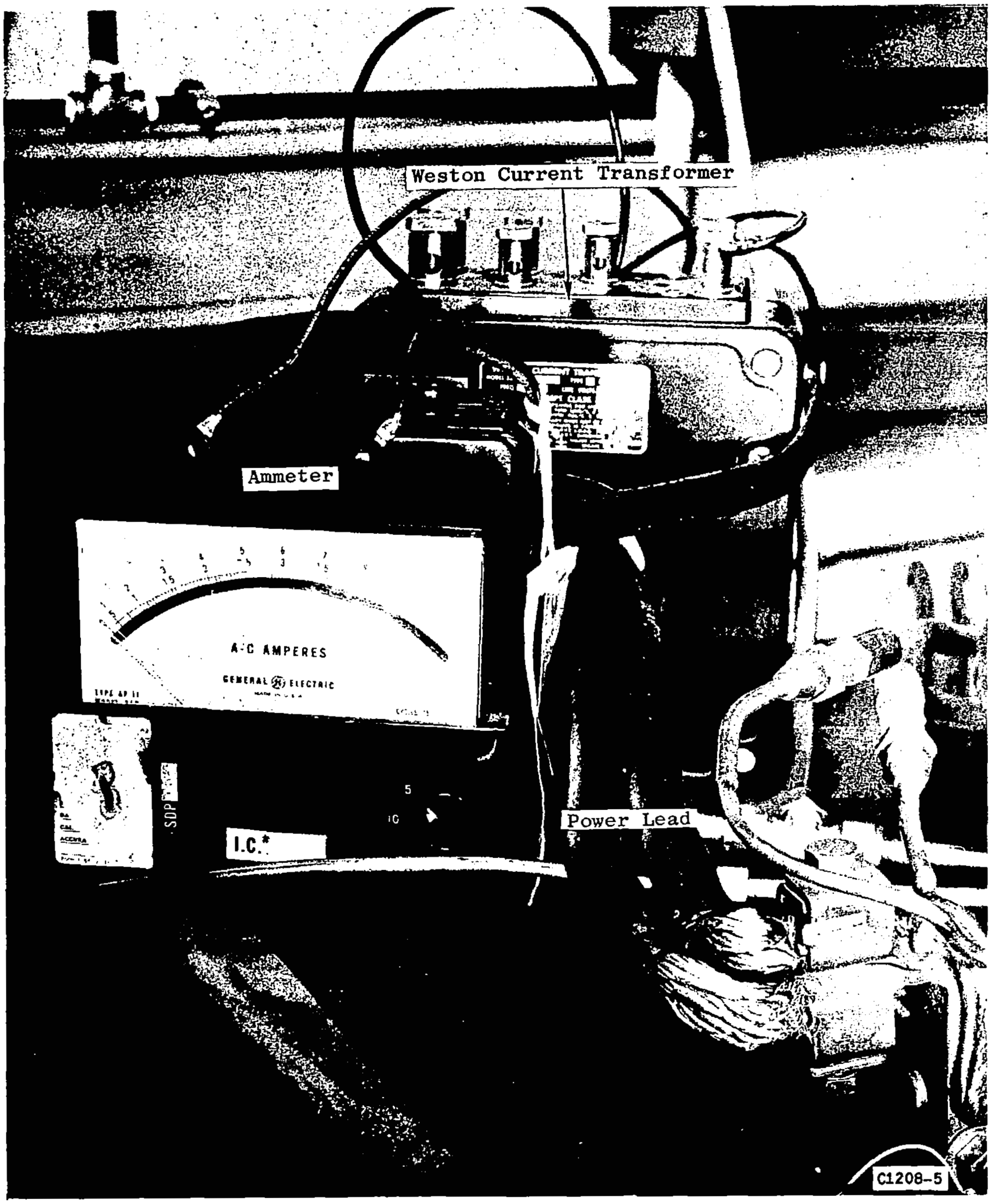


Figure 11. Current Measuring Instruments Employed in the Lithium Fluoride 40° Ring Sector Capsule Test.

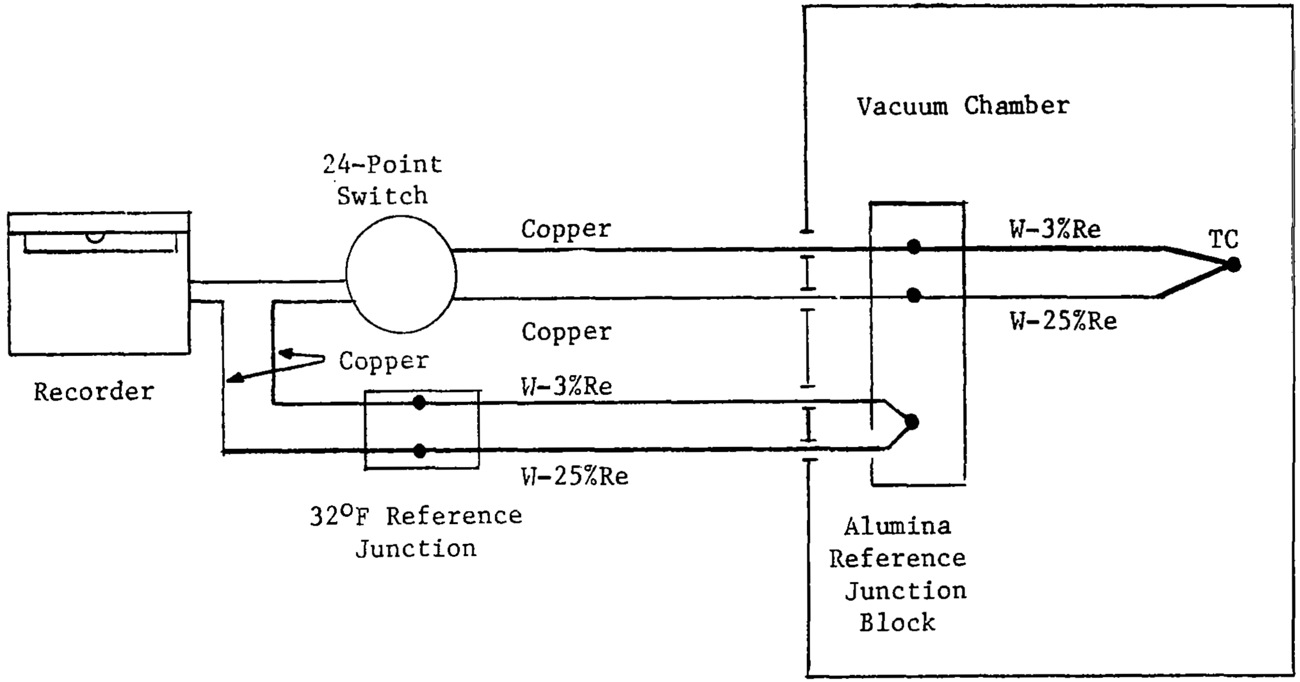


Figure 12. Thermocouple Circuitry Employed in the Lithium Fluoride Capsule Test.

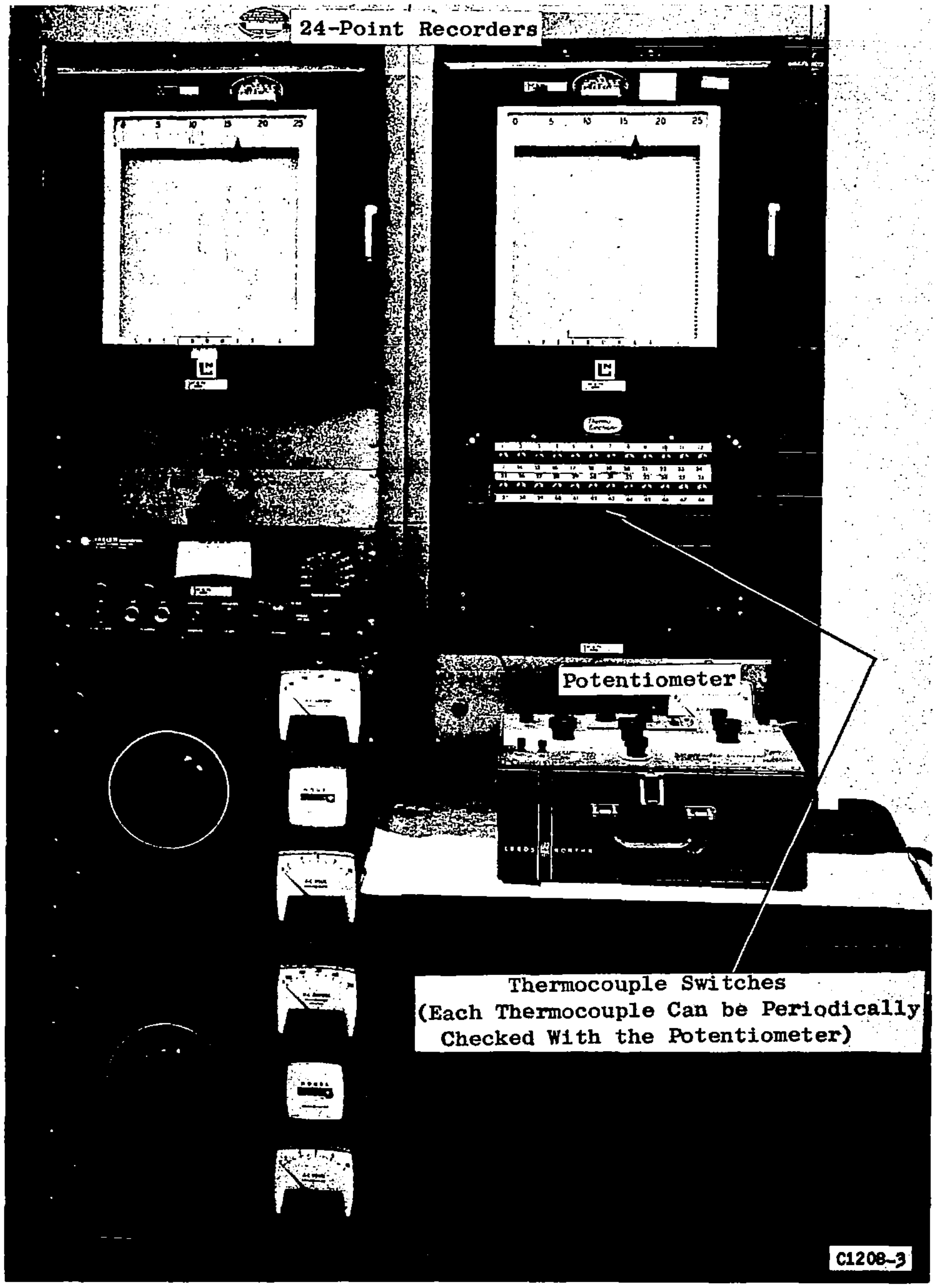


Figure 13. Temperature Measuring and Recording Instrumentation - Lithium Fluoride 40° Ring Sector Capsule Test.

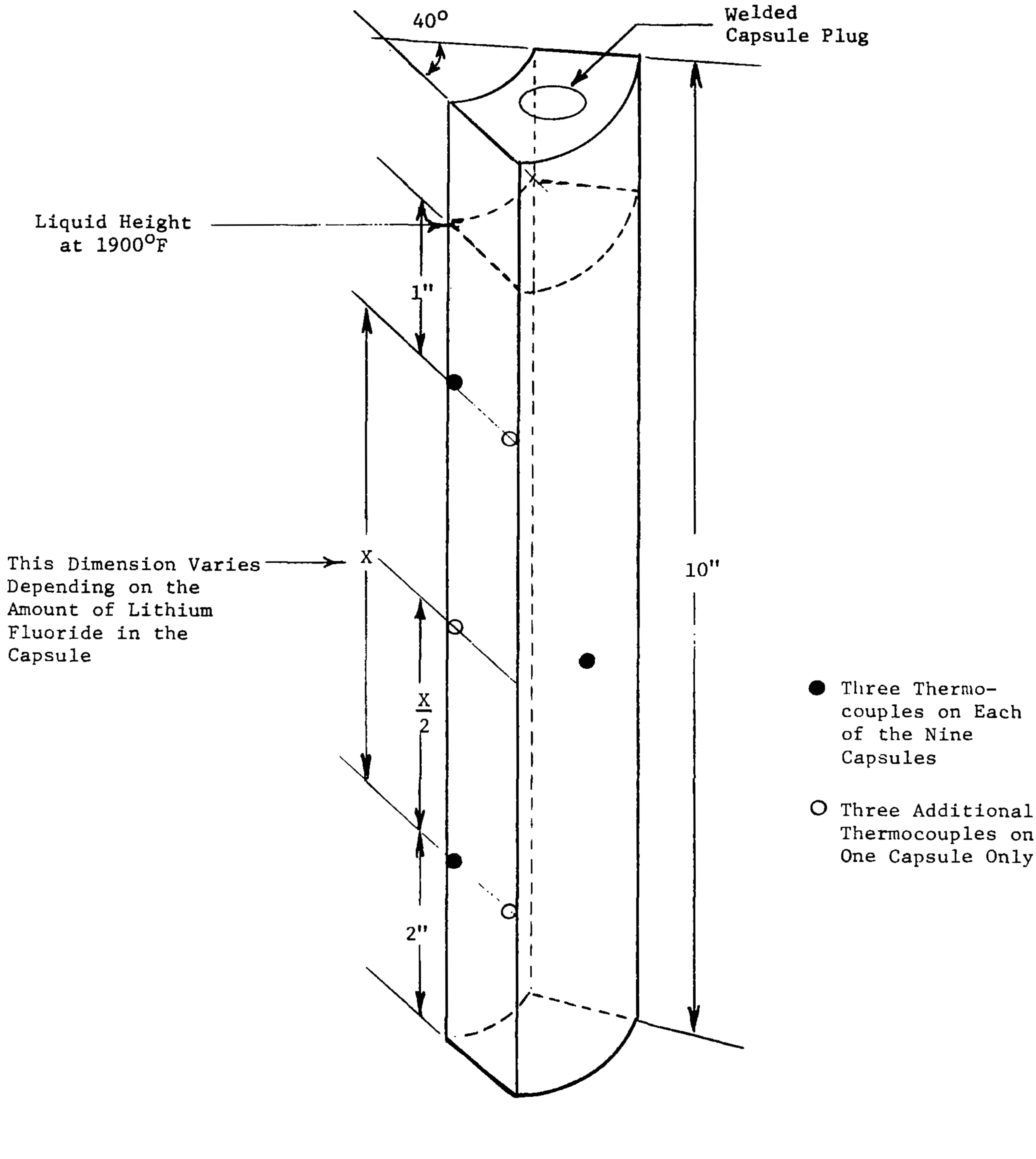


Figure 14. Thermocouple Location - 40° Ring Sector Capsule Test.

on the outside radius of each capsule was positioned vertically halfway between the other two thermocouples and horizontally in the center of the capsule. One Cb-1Zr capsule was instrumented with three additional thermocouples in the same horizontal planes as the other three thermocouples as shown in Figure 14. All the thermocouples were attached according to NSP Specification No. 03-0019-00-A, "Thermocouple Installation Refractory Loop Systems in High Vacuum Environment." Instrumentation of the capsule test facility was completed as shown in Figure 15.

3. Lithium Fluoride Leak Detector

Lithium fluoride leak detection devices were employed on the 40° ring sector capsule test. They are surface ionization detectors. The operation of such a detector depends on the conversion of an atom of low ionization potential into a positive ion on a surface with a high work function. The detector shown in Figure 16 employs a rhenium filament and a tantalum collector. Lithium fluoride molecules impinging on the heated rhenium filament will be dissociated to atoms and the lithium atoms subsequently will be ionized. The ionized lithium atoms will be drawn to the tantalum collector which is electrically negative. The electrical signal produced by these ions can be detected with an electrometer, as shown in the wiring schematic, Figure 17.

D. Capsule Testing Procedure

Upon completing the instrumentation and assembly of 40° ring sector capsules as shown in Figure 15, the bell jar was secured and pumpdown was initiated. The facility passed helium mass spectrometer leak checking to NSP Specification No. 03-0013-00-B and was subsequently evaluated by sorption and getter-ion pumping. Following a 12-hour bakeout of the vacuum system, the chamber pressure was 10^{-9} torr. At this time power was applied to the tantalum heating element at a rate to maintain the chamber pressure in the 10^{-7} torr range. When the capsule's temperature reached 1500°F, the temperature cycle control system was put in the automatic position and temperature cycling commenced. The capsules were heated from 1500°F to 1900°F in one hour with 4.1 kw power applied throughout the heating cycle. The capsules cooled to the lithium fluoride freezing temperature in eight minutes, rejected the heat of solidification in 23 minutes, and cooled to 1500°F in five minutes. At that time,

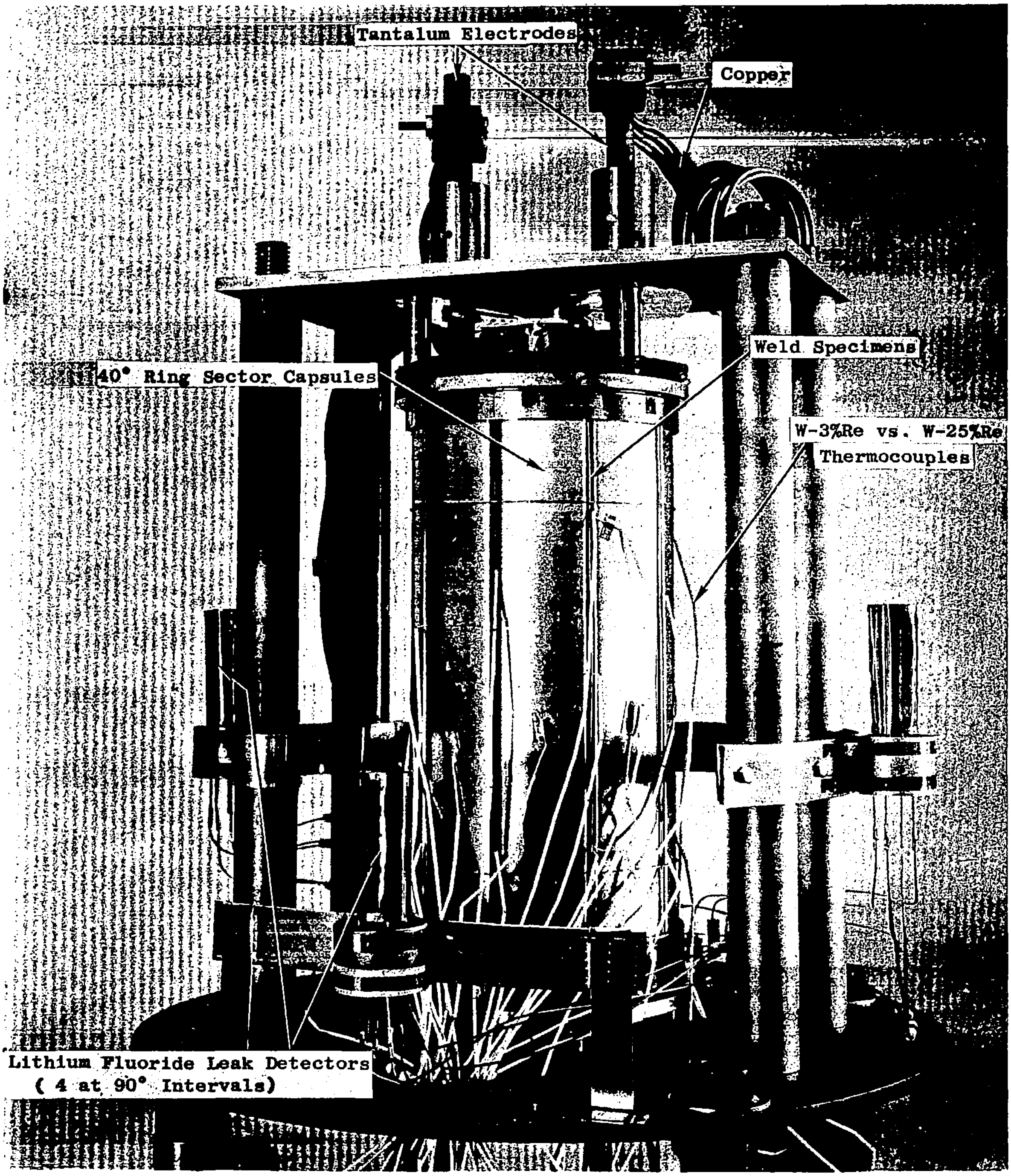
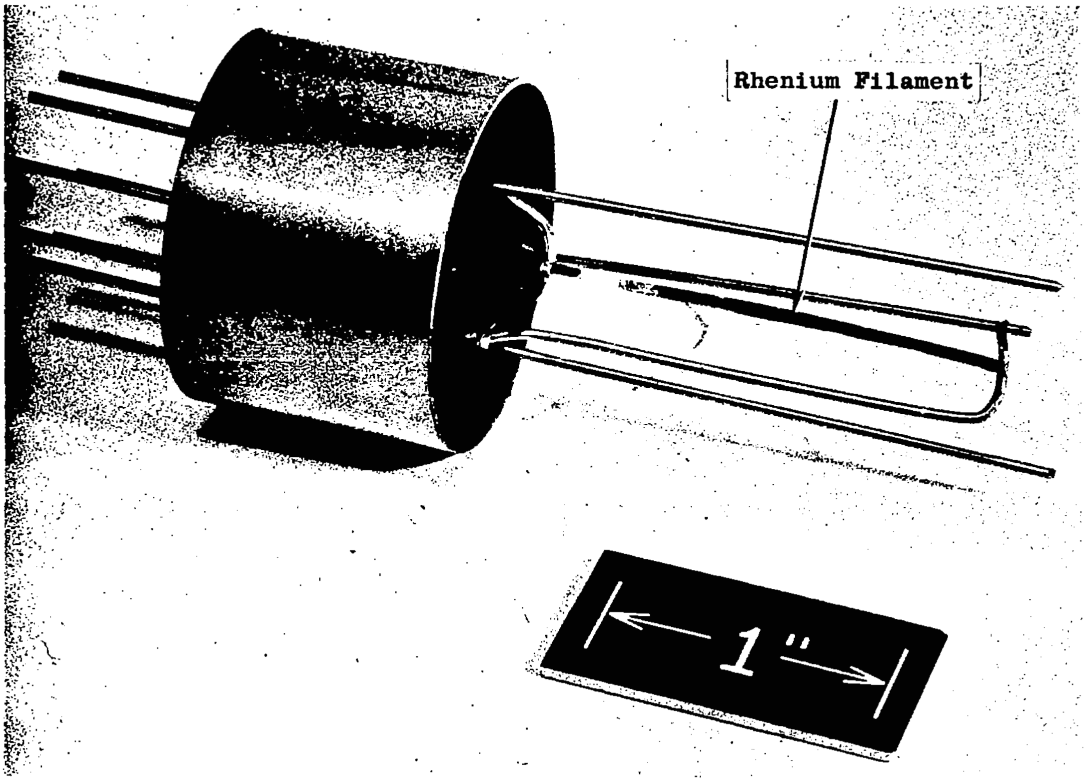
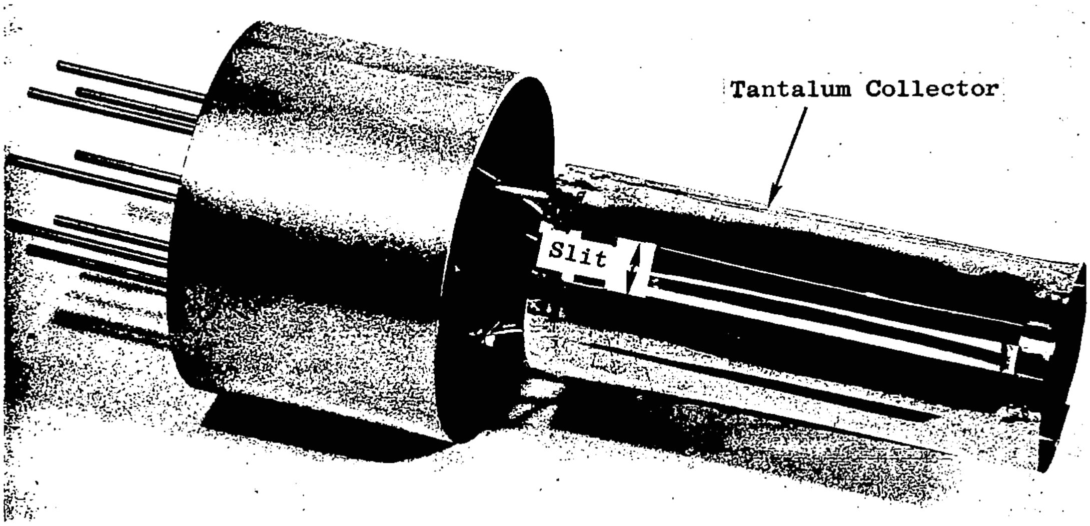


Figure 15. The Lithium Fluoride 40° Ring Sector Capsule Test Facility. (Orig. C67052544)

C1230-4



Before Attachment Of The Tantalum Collector



Tantalum Collector In Place

C1166-1

Figure 16. Lithium Fluoride Leak Detector Filament And Collector Assembly.

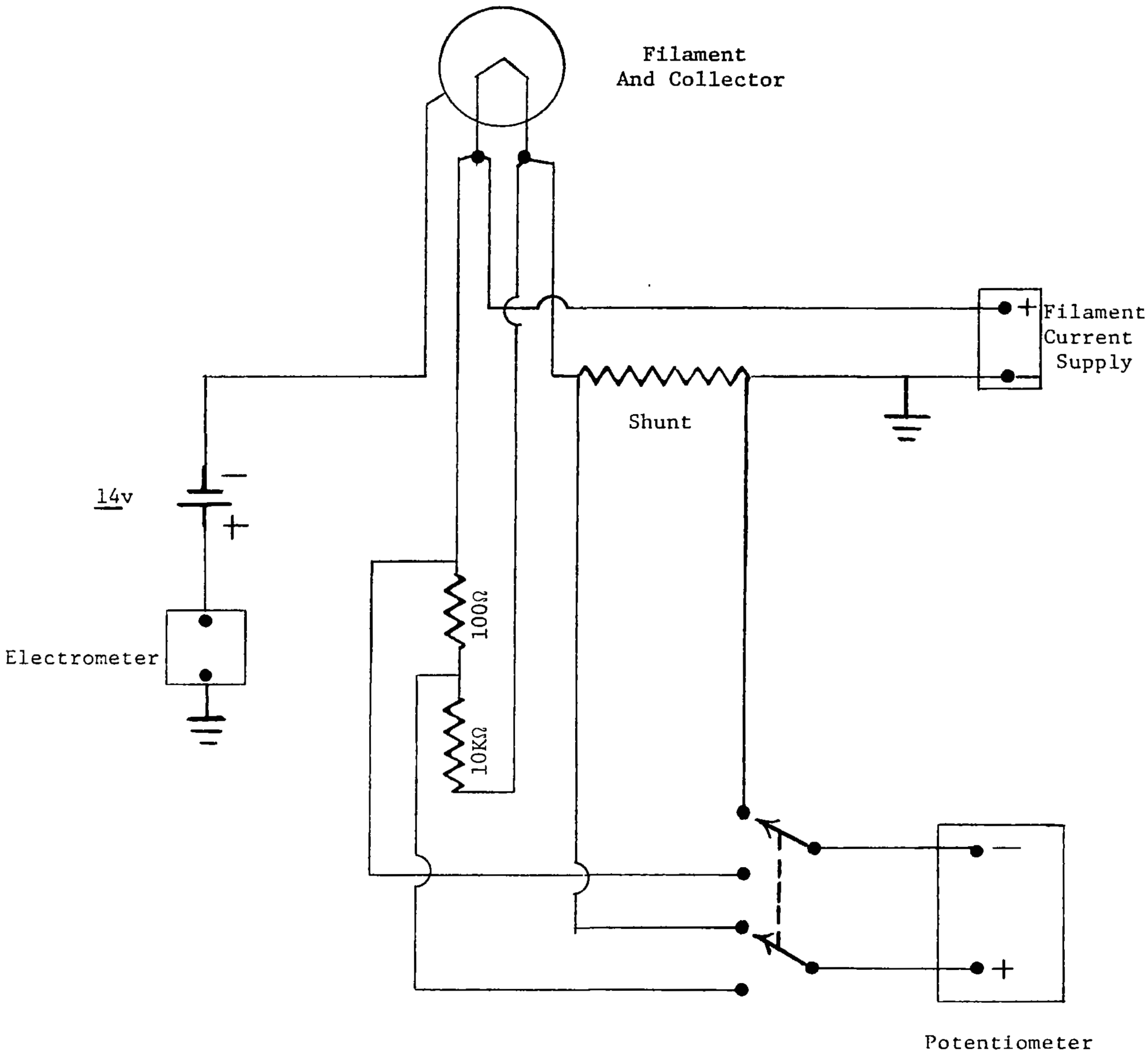


Figure 17. Lithium Fluoride Leak Detector Wiring Schematic Diagram.

the chamber pressure was 2 x 10^{-7} torr with the capsules at 1900°F. After 300 hours the chamber pressure was in the 10^{-8} torr range with the capsules at 1900°F and after 5000 hours the pressure was 1 x 10^{-8} torr.

A typical capsule temperature profile during cycling, shown in Figure 18, was measured on the 320th cycle. At the start of the cycle, the area adjacent to the top thermocouple was void of lithium fluoride, and its temperature rose rapidly until lithium fluoride began to melt in the bottom of the capsule. Melting at the outside wall of the capsule occurred soon after with all the lithium fluoride being molten thirty minutes after heating was initiated. The area adjacent to the upper thermocouple was covered with molten fluoride at this time reaching a maximum temperature of 1885°F, sixty minutes after the cycle was initiated. The power was turned off at this time, and the capsule cooled to the lithium fluoride freezing point in approximately eight minutes. Once lithium fluoride began to freeze, the concomitant volume reduction lowered the liquid level below the area adjacent to the top thermocouple. This area, void of lithium fluoride cooled rapidly reaching a minimum temperature of 1240°F. The lithium fluoride adjacent to the bottom thermocouple rejected the heat of solidification in twenty-two minutes and cooled 25°F below the freezing point before the next cycle was initiated.

After 1000 hours of testing, the capsules were allowed to cool to room temperature. The chamber was opened, and one capsule each of Cb-1Zr, FS-85, and SCb-291 alloys were removed. The vacated capsule space was filled with a tantalum foil insulation assembly and the chamber reclosed. Following ion pump confinement and bakeout, capsule heating was initiated. The remaining six capsules were returned to the test conditions and tested for an additional 4000 hours (5000 hours total test duration).

Cb-1Zr capsules removed after 5000 hours of testing were severely distorted. Capsules fabricated from the stronger columbium-base alloys, SCb-291 and FS-85, showed little change. The deformation observed in the Cb-1Zr capsules is compared with the unchanged appearance of the FS-85 capsules in Figure 19.

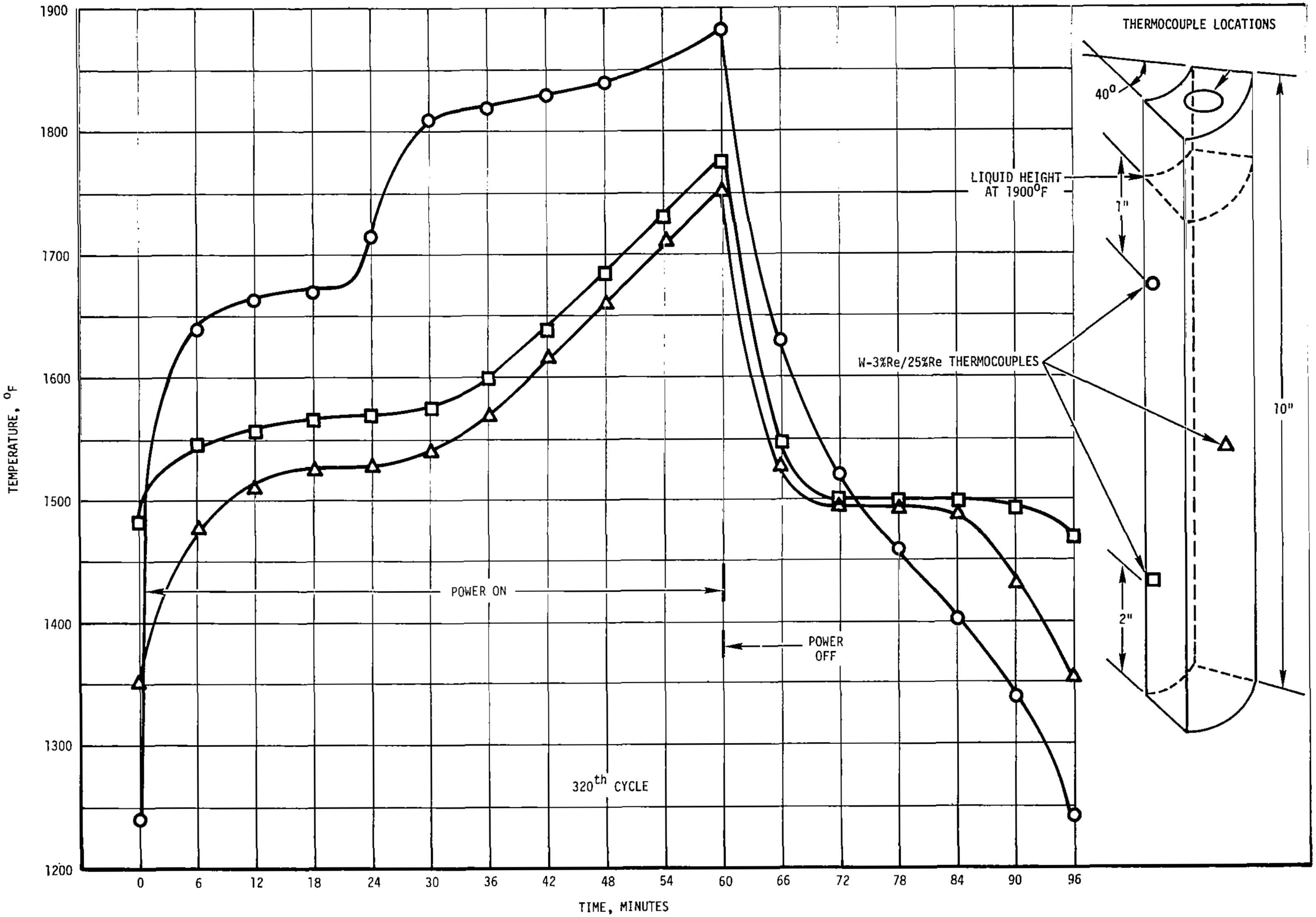


Figure 18. Typical Thermal Cycle of a 40° Ring Sector Lithium Fluoride Test Capsule.

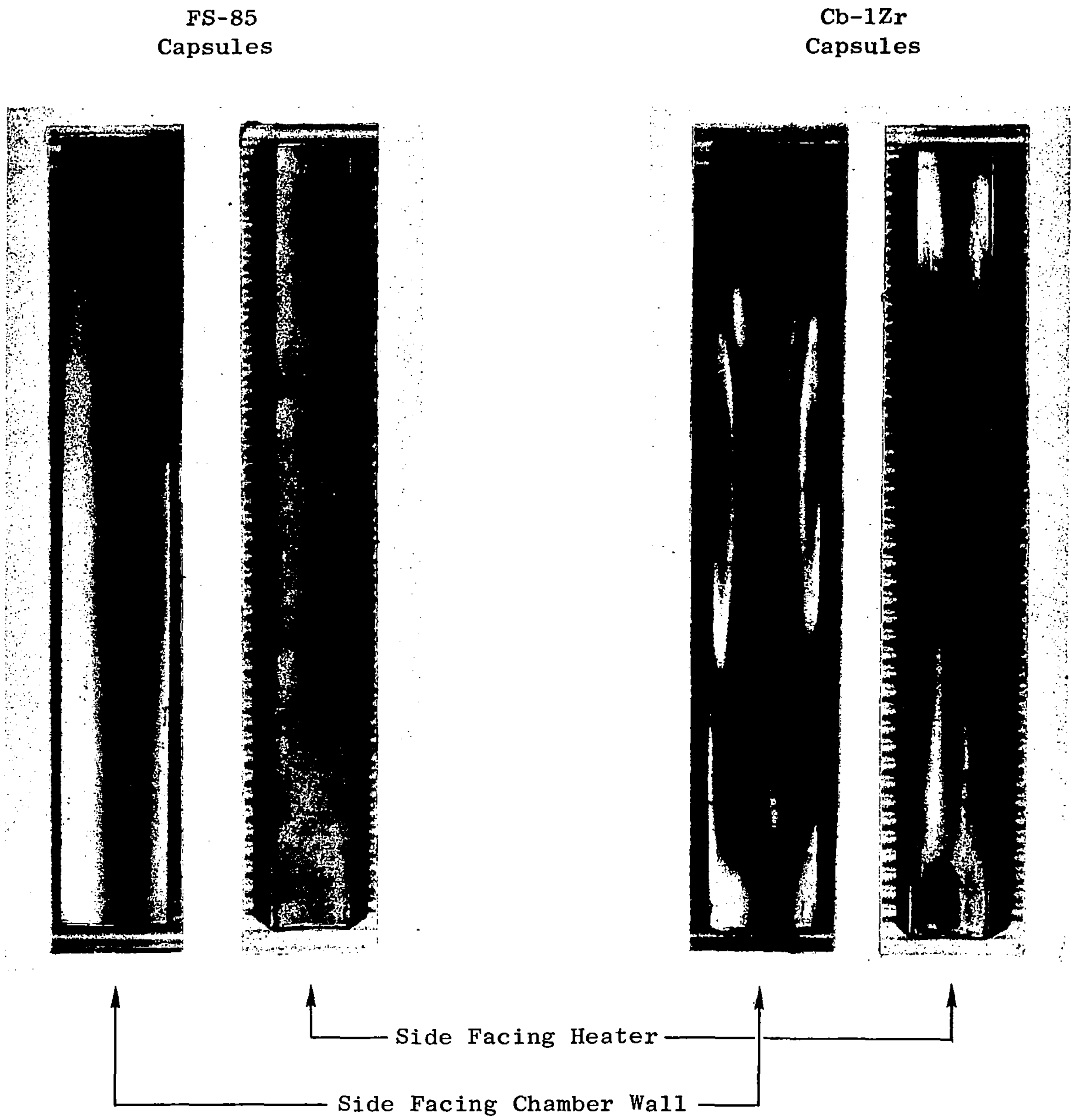


Figure 19. 40^{o} Ring Sector Capsule Following 5000 Hours of Testing. The Capsules Were Cycled Between 1500^{o} and $1900^{o}F$. The FS-85 Capsules Were Unchanged in Appearance Whereas Severe Distortion Was Observed in the Cb-1Zr Capsules. (Orig. C68061208 and C68061209)

V. EVALUATION OF CAPSULES AND TEST RESULTS

The evaluation of the capsules and weld specimens consisted of chemistry, metallography, and determination of ductile-to-brittle transition temperature.

Room temperature tensile properties were determined for specimens exposed to lithium fluoride in 40° ring sector capsules. The weight of the tensile specimens before and after test was also determined.

A. Chemical Analysis

Chemical analyses results of the 40° ring sector capsules and weld specimens are shown in Table VIII. No significant changes in chemical analysis were found. Semiquantitative spectrographic analysis of the lithium fluoride removed from each of the capsules (FS-85, Cb-1Zr, SCb-291) after 1000 hours and 5000 hours showed no increase in Cb, W, Ta, or Zr concentrations beyond the limits of detection (< 10 ppm Cb, < 50 ppm W, < 100 ppm Ta, and < 10 ppm Zr).

B. Weight Change

Weight measurements of the tensile specimens which were removed from the capsules after exposure to lithium fluoride for 1000 and 5000 hours indicated no significant weight change (± 0.003 g 0.05 percent).

C. Hardness Surveys

Hardness surveys across the capsule wall specimens showned no discernible hardness gradient.

D. Metallography

Specimens were removed from each capsule and examined for evidence of corrosion. The side wall weld area exposed to lithium fluoride was compared in appearance with the corresponding weld specimen exposed to the same thermal and vacuum environment along with the capsule but not to lithium fluoride. No corrosion was observed as can be seen in the photomicrographs of specimens of Cb-1Zr, FS-85, and SCb-291 alloys shown in Figures 20-22.

As described previously, the Cb-1Zr 40° ring sector capsules were severely distorted following the 5000-hour-test exposure.

Surface discontinuities, shown in Figure 23, were observed in the

TABLE VIII. CHEMICAL ANALYSIS OF SECTIONS REMOVED FROM THE WELDED SIDE WALL OF LITHIUM FLUORIDE 40° RING SECTOR CAPSULES AND WELD SPECIMENS AFTER 1000 HOURS AND 5000 HOURS OF TESTING[a]

		Concentration, ppm								
			Capsule				Weld Specimens			
			After 1000 Hours		After 5000 Hours		After 1000 Hours		After 5000 Hours	
Capsule Material[b]	Element[c]	Before Test[d]	Weld	Parent Metal	Weld	Parent Metal	Weld	Parent Metal	Weld	Parent Metal
Cb-1Zr	O	168, 157[e]	211, 227	138, 154	237, 232	141, 129	129, 124	109, 102	165, 168	122, 134
	N	8, 10	16, 19	8, 15	11, 11	4, 3	9, 11	2, 4	12, 12	6, 8
	H	2, 1	4, 2	4, 5	1, 1	<1, <1	1, 2	<1, <1	3, 2	2, 3
	C	48, 54	76	44, 54	70	65, 70	80	41, 59, 86	85	67, 70
SCb-291 (10Ta-10W-Bal. Cb)	O	38, 20	32, 30	28, 28	38, 35	38, 38	38, 40	30, 39	51, 82	53, 50
	N	3, 4	17, 9	13, 10	7, 5	6, 6	2, 3	1, 4	16, 13	9, 7
	H	1, 1	2, 2	<1, <1	2, 1	1, 1	<1, <1	<1, <1	1, 3	1, 2
	C	9, 20	85	31, 37	46	34, 38	40	26, 30	53	18, 20
FS-85 (28Ta-10.5W-0.9Zr-Bal. Cb)	O	154, 120	154, 153	140, 153	150, 167	155, 155	134, 149	108, 120	132, 133	151, 148
	N	22, 114	33, 35	34, 41	27, 18	17, 17	20, 16	13, 15	10, 18	18, 18
	H	<1, <1	<1, <1	<1, 5	<1, <1	<1, <1	3, 4	1, <1	1, 1	<1, <1
	C	15, 28	41, 53	36, 48	55, 56	53, 54	50, 97	40, 45	56, 71	51, 51

[a] Capsules and weld specimens were cycled between 1500°F and 1900°F; chamber pressure at the maximum cycle temperature decreased from 3.7 x 10^{-7} torr after 50 hours of operation to 4 x 10^{-9} torr after 5000 hours of operation.

[b] As-Received 0.030-inch thick sheet.

[c] Analytical Methods: O, N, and H; Vacuum Fusion C; Combustion Conductometric

[d] Analysis following welding and postweld annealing of FS-85 for 1 hour at 2400°F. The Cb-1Zr and SCb-291 were postweld annealed for 1 hour at 2200°F.

[e] Duplicate Analyses.

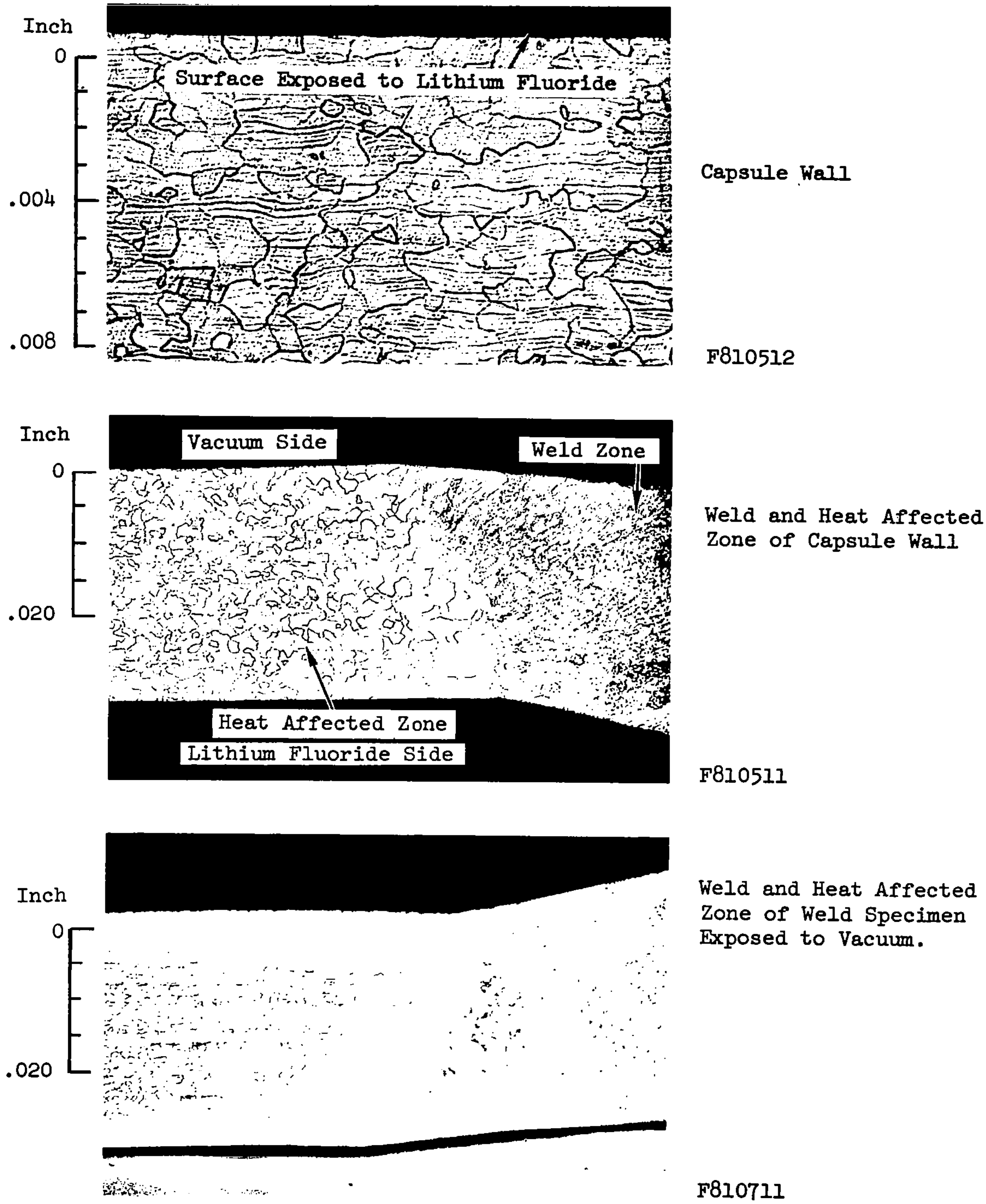


Figure 20. Photomicrographs of the FS-85 40^{o} Ring Sector Capsule and Weld Specimen Following 5000 Hours of Testing.

Etchant: 60% Glycerin, 20% HNO_3, 20% HF.

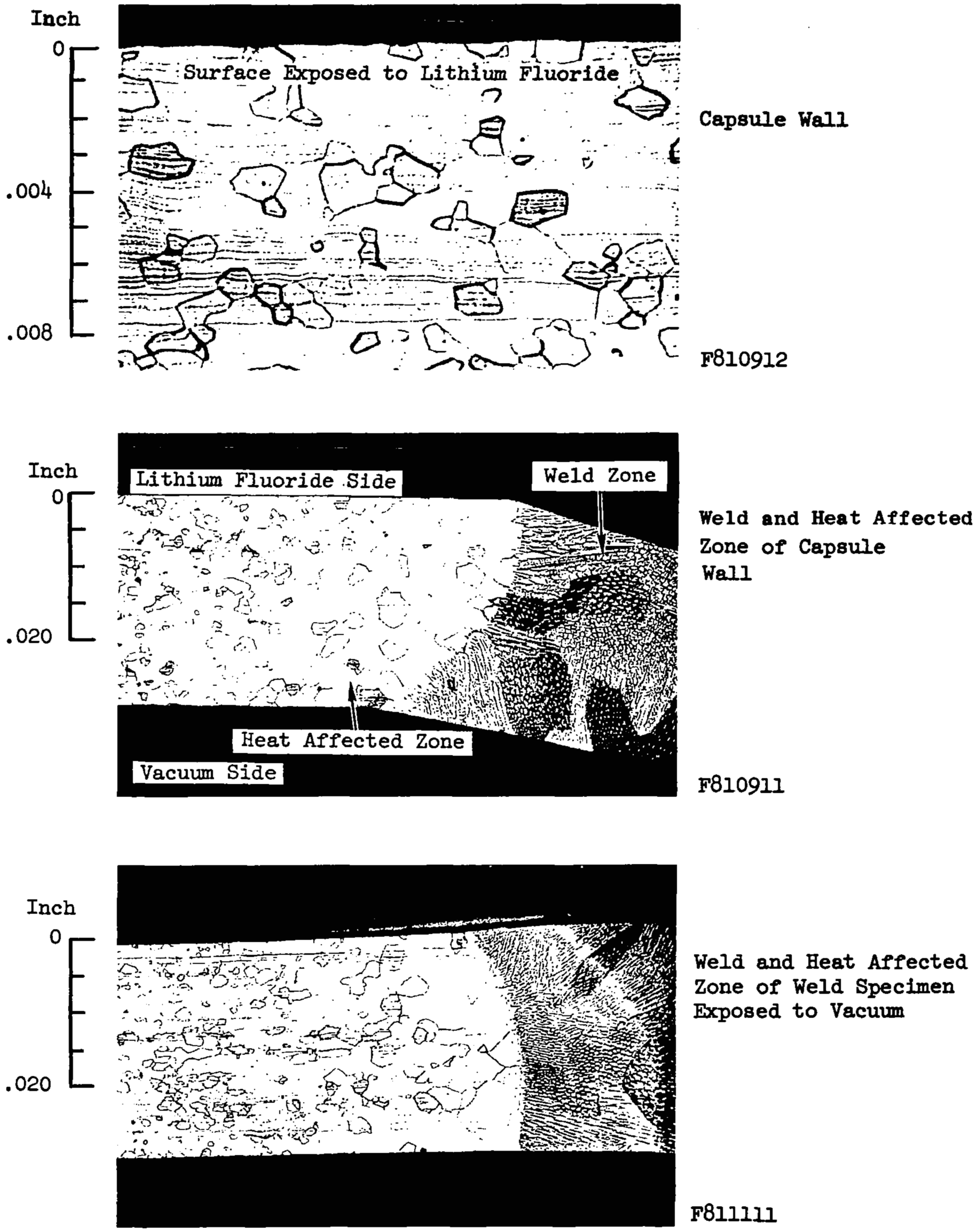
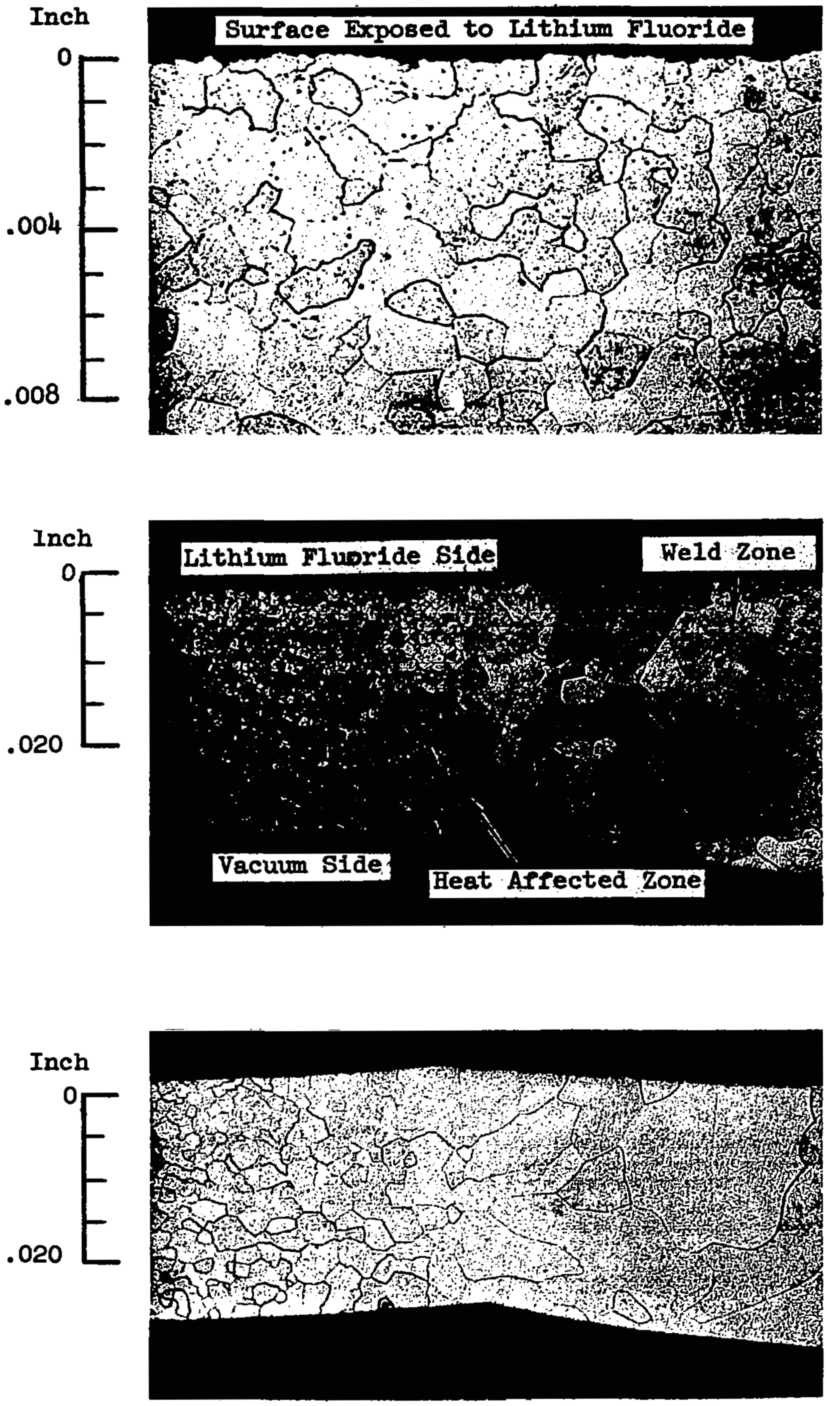


Figure 21. Photomicrographs of the SCb-291 40° Ring Sector Capsule and Weld Specimen Following 5000 Hours of Testing.

Etchant: 60% Glycerin, 20% HNO_3, 20% HF.



Capsule Wall

F810112

Weld and Heat Affected Zone of Capsule Wall

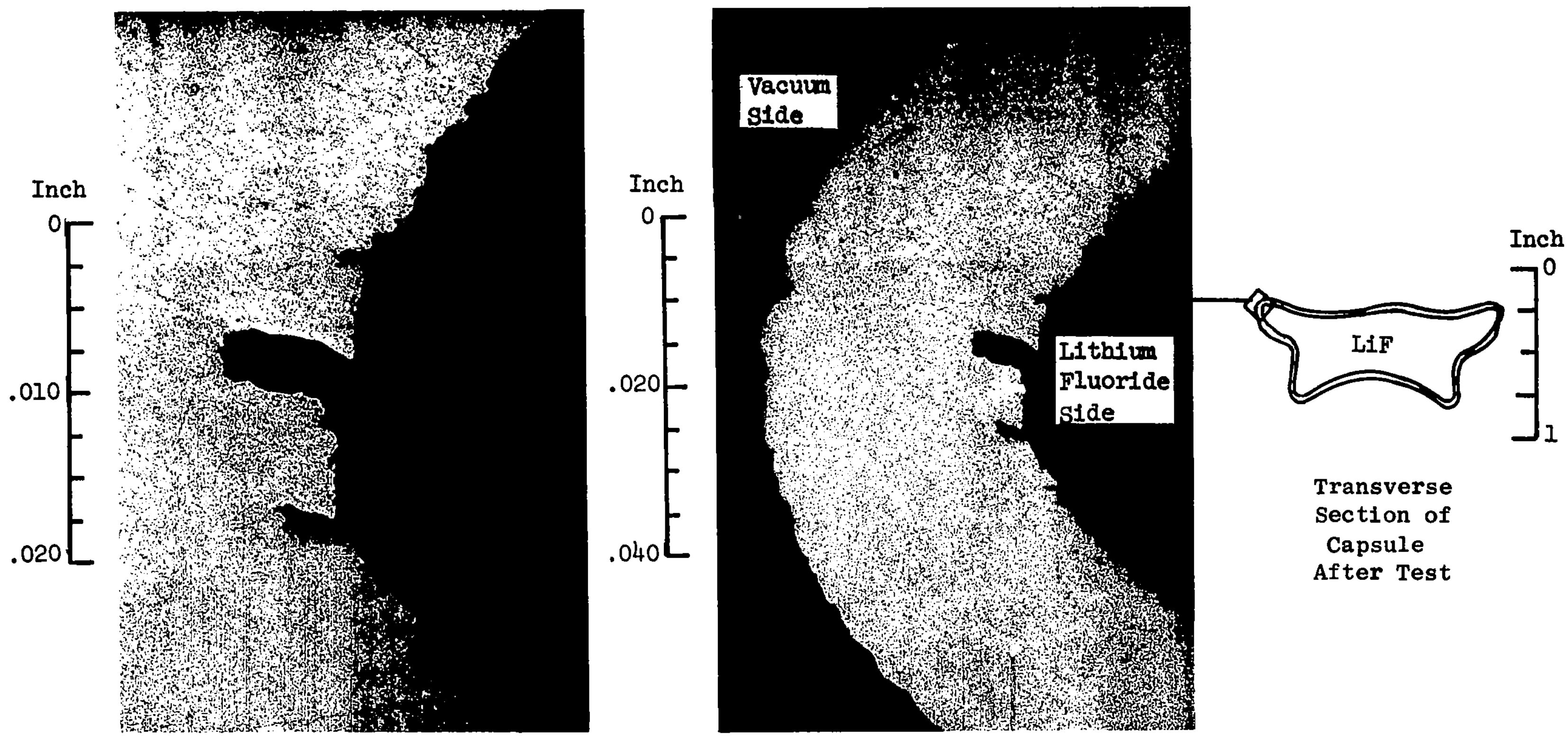
F810111

Weld and Heat Affected Zone of Weld Specimen Exposed to Vacuum.

F810311

Figure 22. Photomicrographs of the Cb-1Zr 40° Ring Sector Capsule and Weld Specimen Following 5000 Hours of Testing.

Etchant: 60% Glycerin, 20% HNO_3, 20% HF.



F810211

F810212

Figure 23. Photomicrographs of the Distorted Corner of the Cb-1Zr 40° Ring Sector Capsule. The Capsule Deformed as a Result of 5000 Hours of Thermal Cycling Between 1500°F and 1900°F.

As-Polished.

severely distorted corners of the Cb-1Zr capsule. Closer examination of these areas in the etched condition, shown in Figure 24, did not indicate a corrosion mechanism as a possible cause. The rough appearance of the outside surfaces of the distorted corners which were not in contact with lithium fluoride further suggests the discontinuities did not result from lithium fluoride corrosion. In this regard, analysis of the lithium fluoride removed from the capsules after testing did not indicate the presence of any elements present in the capsule materials.

A spare Cb-1Zr 40° ring sector capsule which had not been filled or tested was examined metallographically to ascertain that the discontinuities were not produced during the forming of the capsules. No discontinuities were observed, and both the inside and outside walls at the corners were smooth as shown in Figure 25.

It is postulated that the discontinuities observed in the Cb-1Zr capsule resulted from the deformations caused by the thermal cycles, and the discontinuities on the inside capsule wall in contact with lithium fluoride were enlarged as a result of the melting and freezing of the lithium fluoride and the concomitant 30 percent change in volume.

E. Tensile Tests

Room temperature tensile tests of Cb-1Zr, SCb-291, and FS-85 alloys exposed to lithium fluoride for 1000 hours and 5000 hours in 40° ring sector capsules were conducted along with the pretest specimens of each alloy. The test results are shown in Tables IX, X, and XI.

Graphical analyses of the data shown in Figures 26, 27, and 28 indicate small changes in mechanical properties as a result of the test exposure. The tensile strengths of all three alloys decreased slightly in relation to exposure hours with the gettered alloys, Cb-1Zr and FS-85, showing the most change. The changes in properties are most likely a result of the thermal effects of the exposure and not the effects of lithium fluoride since no significant changes in chemical analysis and microstructure were observed.

F. Bend Tests

Weld specimens of Cb-1Zr, SCb-291, and FS-85 of the following conditions were tested:

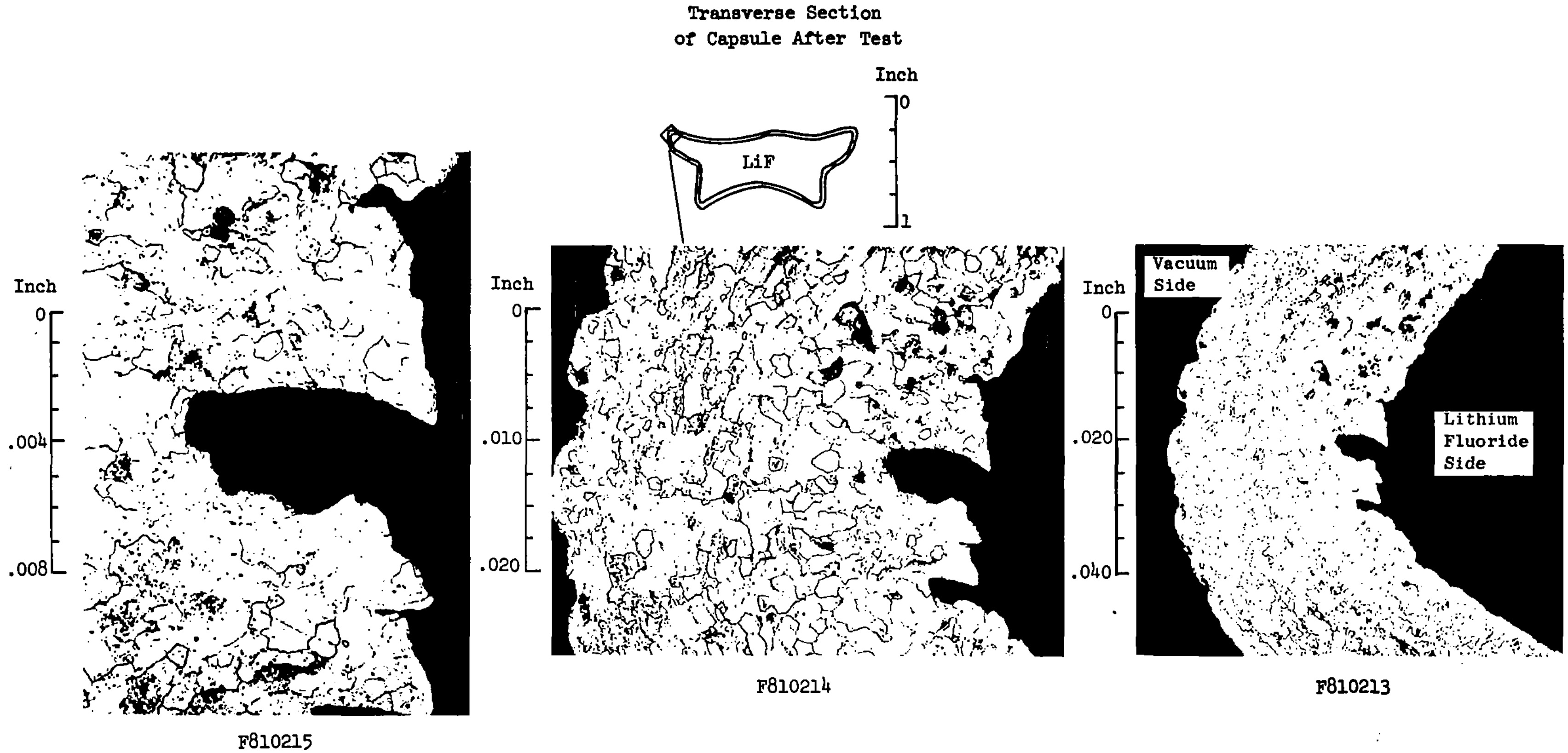


Figure 24. Photomicrographs of the Distorted Corner of the Cb-1Zr 40^{o} Ring Sector Capsule Showing the Discontinuities in the Surface. The Capsule Deformed as a Result of 5000 Hours of Thermal Cycling Between 1500^{o}F and 1900^{o}F.

Etchant: 60% Glycerin, 20% HNO_3, 20% HF

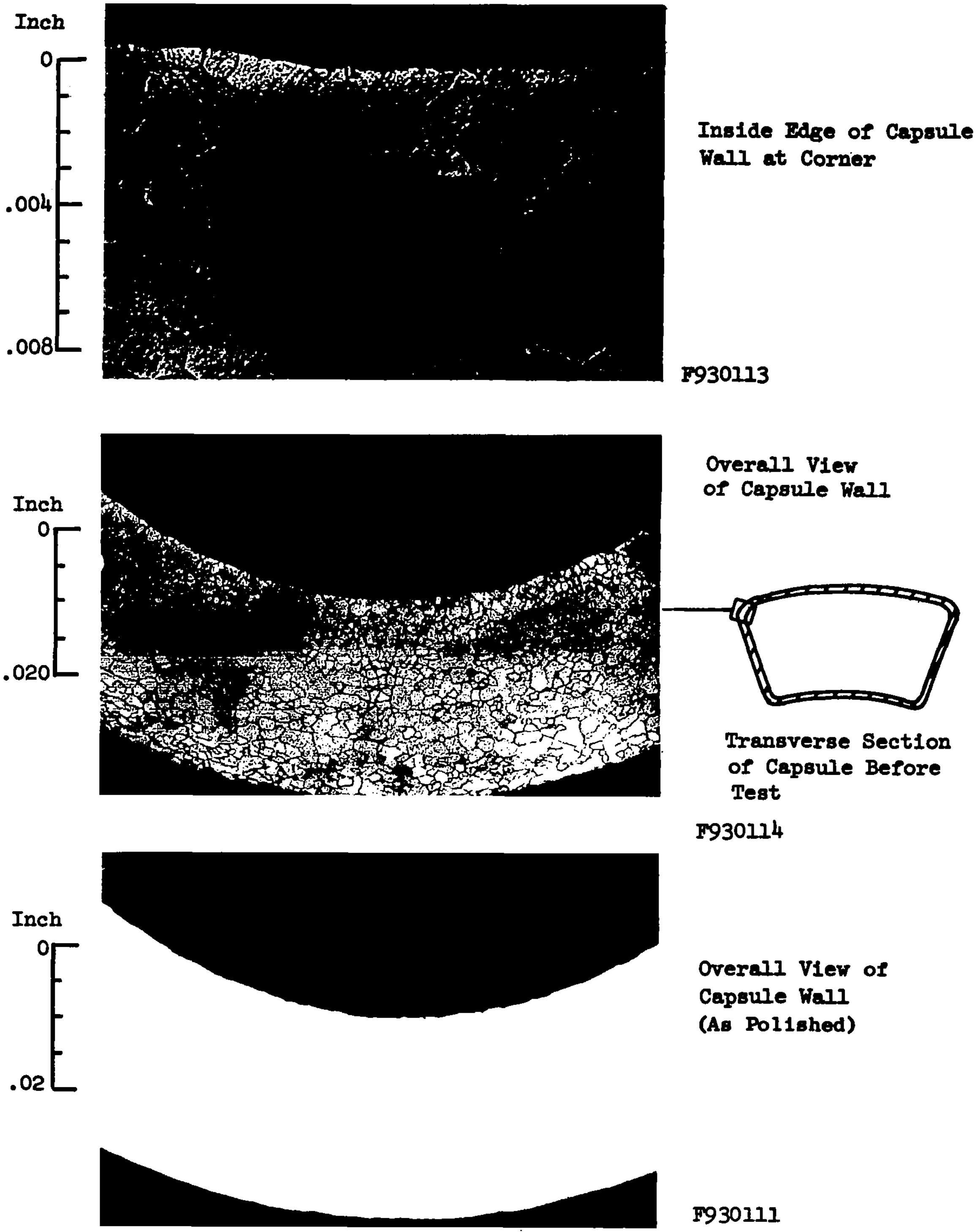


Figure 25. Corner of Cb-1Zr 40° Ring Sector Capsule Before Test.

Etchant: 60% Glycerin, 20% HNO_3, 20% HF.

TABLE IX. ROOM TEMPERATURE TENSILE PROPERTIES OF Cb-1Zr SPECIMENS EXPOSED TO LITHIUM FLUORIDE IN 40° RING SECTOR CAPSULES[a]

Condition	Ultimate Strength psi	0.2% Yield Strength psi	Elongation %
As Received	38,020	20,920	32.5
	38,000	21,120	34.1
	38,000 avg.	21,000 avg.	33 avg.
Exposed 1000 hours in lithium fluoride	37,200	23,740	28.7
	37,450	22,840	28.6
	37,300 avg.	23,300 avg.	28 avg.
Exposed 5000 hours in lithium fluoride	34,990	20,990	26.1
	35,630	20,290	26.6
	35,860	22,000	27.2
	36,857	23,370	26.2
	35,800 avg.	21,700 avg.	26 avg.

(a) The capsules were cycled between 1500°F and 1900°F.

TABLE X. ROOM TEMPERATURE TENSILE PROPERTIES OF SCb-291 SPECIMENS EXPOSED TO LITHIUM FLUORIDE IN 40° RING SECTOR CAPSULES[a]

Condition	Ultimate Strength psi	0.2% Yield Strength psi	Elongation %
As Received	56,890	43,260	34.0
	57,110	42,510	32.7
	57,000 avg.	43,300 avg.	33 avg.
Exposed 1000 hours in lithium fluoride	57,020	41,950	33.5
	55,500	39,350	27.5
	56,300 avg.	40,700 avg.	31 avg.
Exposed 5000 hours in lithium fluoride	56,880	40,060	27.0
	56,950	40,090	29.6
	55,397	39,900	25.2
	55,349	39,200	28.5
	56,100 avg.	39,800 avg.	28 avg.

(a) The capsules were cycled between 1500°F and 1900°F.

TABLE XI. ROOM TEMPERATURE TENSILE PROPERTIES OF FS-85 SPECIMENS EXPOSED TO LITHIUM FLUORIDE IN 40° RING SECTOR CAPSULES[a]

Condition	Ultimate Strength psi	0.2% Yield Strength psi	Elongation %
As Received	84,560	68,800	24.0
	83,790	64,860	25.9
	84,200 avg.	66,800 avg.	25 avg.
Exposed 1000 hours in lithium fluoride	81,753	61,440	25.8
	81,415	62,796	26.7
	81,600 avg.	62,100 avg.	26 avg.
Exposed 5000 hours in lithium fluoride	81,120	61,010	27.4
	81,270	60,910	26.1
	81,230	61,170	26.5
	81,150	60,560	26.8
	81,200 avg.	60,900 avg.	27 avg.

(a) The capsules were cycled between 1500°F and 1900°F.

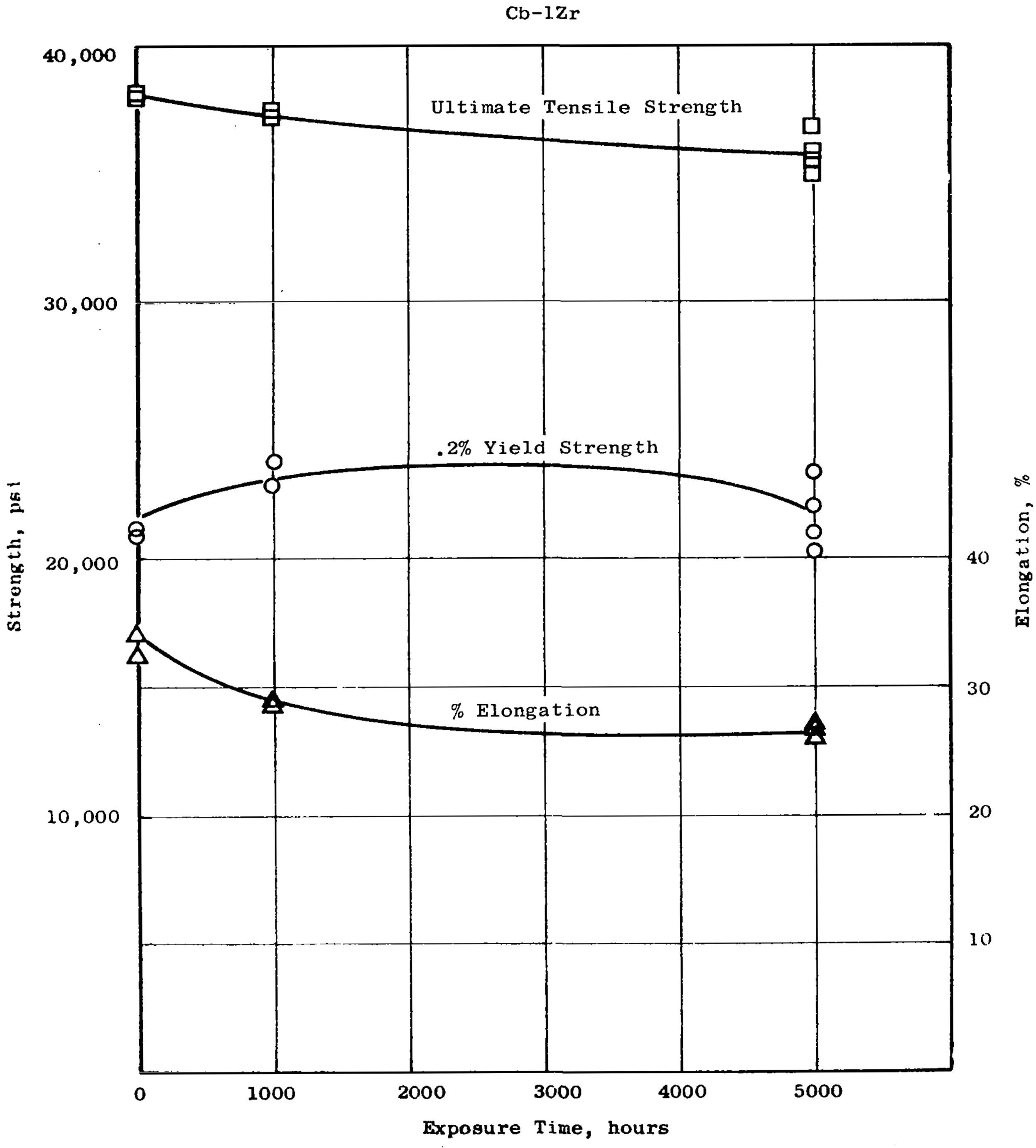


Figure 26. Tensile Properties of Cb-1Zr in Relation to Time of Exposure to Lithium Fluoride in 40° Ring Sector Capsules Cycled Between 1500°F and 1900°F.

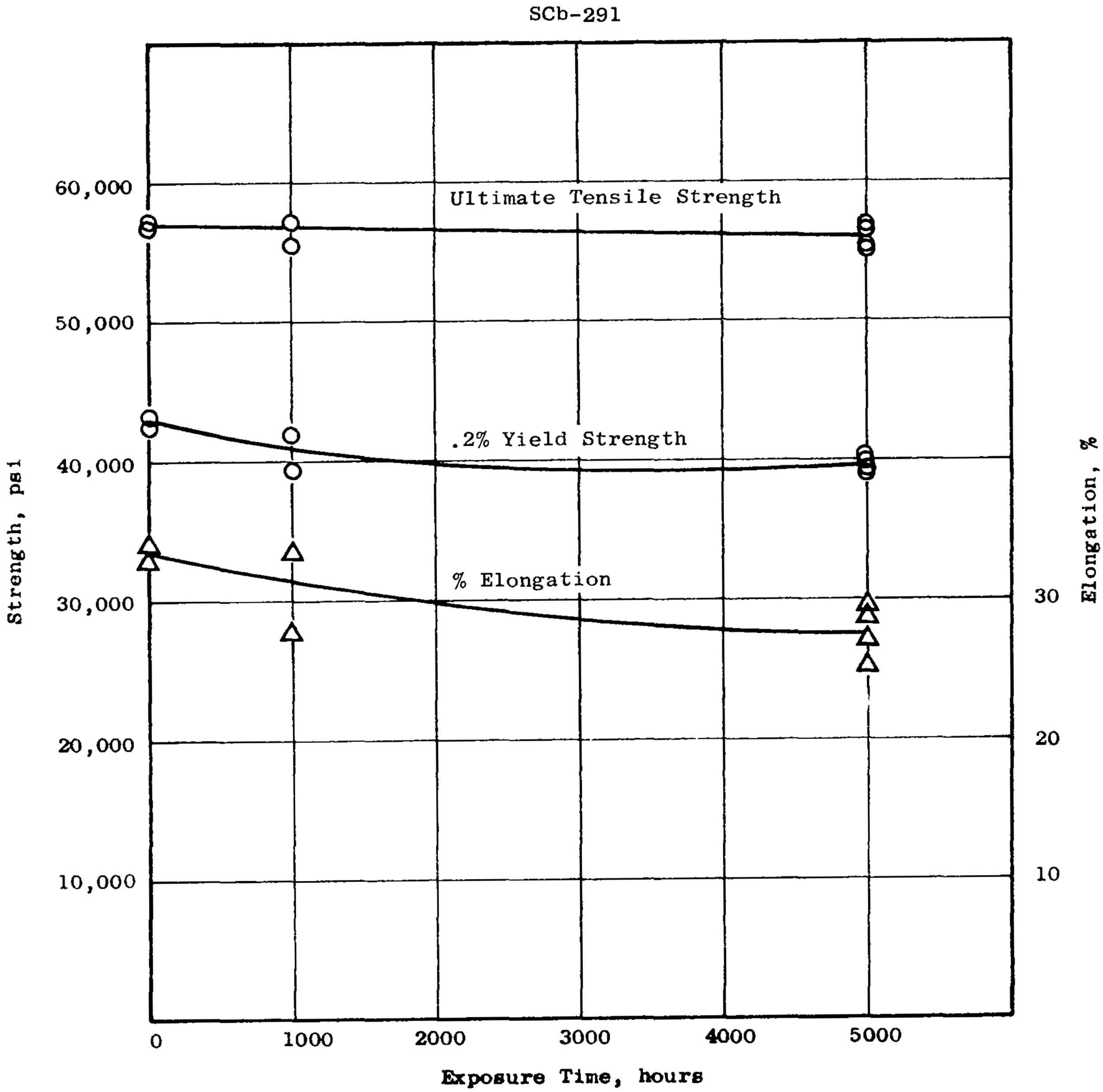


Figure 27. Room Temperature Tensile Properties of SCb-291 in Relation to Time of Exposure to Lithium Fluoride in 40° Ring Sector Capsules Cycled Between 1500°F and 1900°F.

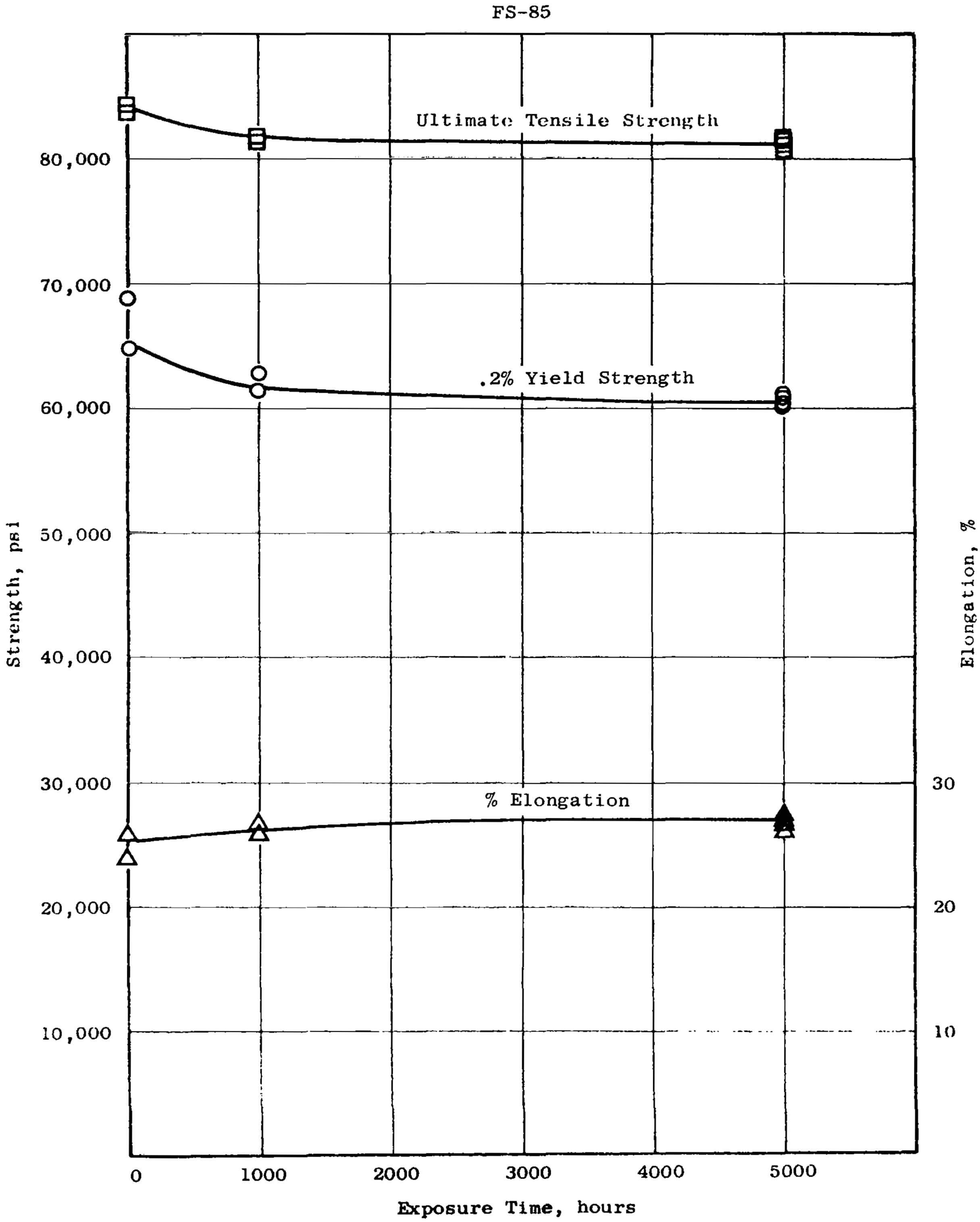


Figure 28. Room Temperature Tensile Properties of FS-85 Alloy in Relation to Time of Exposure to Lithium Fluoride in 40° Ring Sector Capsules Cycled Between 1500°F and 1900°F.

a. Pretest.

b. Specimens removed from the side wall of 40° ring sector capsules tested for 1000 hours.

c. Specimens exposed to vacuum along with 40° ring sector capsules tested for 1000 hours.

d. Specimens removed from the side wall of 40° ring sector capsules tested for 5000 hours.

e. Specimens exposed to vacuum along with 40° ring sector capsules tested for 5000 hours.

Bend tests were performed according to Material Advisory Board recommended test parameters(1) with an Instron Universal Testing Machine and a crosshead travel speed of 0.2 in/min. All bend tests were conducted at 1t (radius of anvil equal to the specimen thickness). Specimens were tested with the weld face in tension and bending axis perpendicular to the weld axis. The bend test temperatures were controlled using liquid nitrogen for tests at -320°F, and 2-methyl-butane for temperatures of -100°F to -250°F. The tests were conducted with specimen, anvil, and stirrup all immersed in the respective liquids. Tests conducted in the 2-methyl-butane were cooled to the desired temperature by pumping regulated quantities of liquid nitrogen through a copper cooling coil placed in the bath. A copper/constantan thermocouple attached to the bend test stirrup was used to monitor the temperature.

The bend test results are as follows:

Cb-1Zr The ductile-to-brittle bend transition temperature for all Cb-1Zr specimens was found to be below -320°F. Specimens for each condition bent a full 105° without failure at -320°F.

SCb-291 Pretest specimens of SCb-291 bent 105° without failure at -250°F and failed at 60° bend at -320°F indicating

(1) "Evaluation Test Methods for Refractory Metal Sheet,Materials," AD 625606, National Academy of Sciences - National Research Council, Washington, D. C., November 1965.

the bend transition temperature to be approximately -300°F. Specimens for all other conditions bent 105° without failure at -320°F.

FS-85 The FS-85 bend test data is shown in Figure 29. The ductile-to-brittle bend transition temperatures determined from the plotted data are presented in Table XII. A comparison of the data obtained on the specimens which were exposed to vacuum and specimens removed from the capsule indicated the lithium fluoride exposure has a definite effect on raising the ductile-to-brittle transition temperature of FS-85. Both types of specimens had similar thermal history.

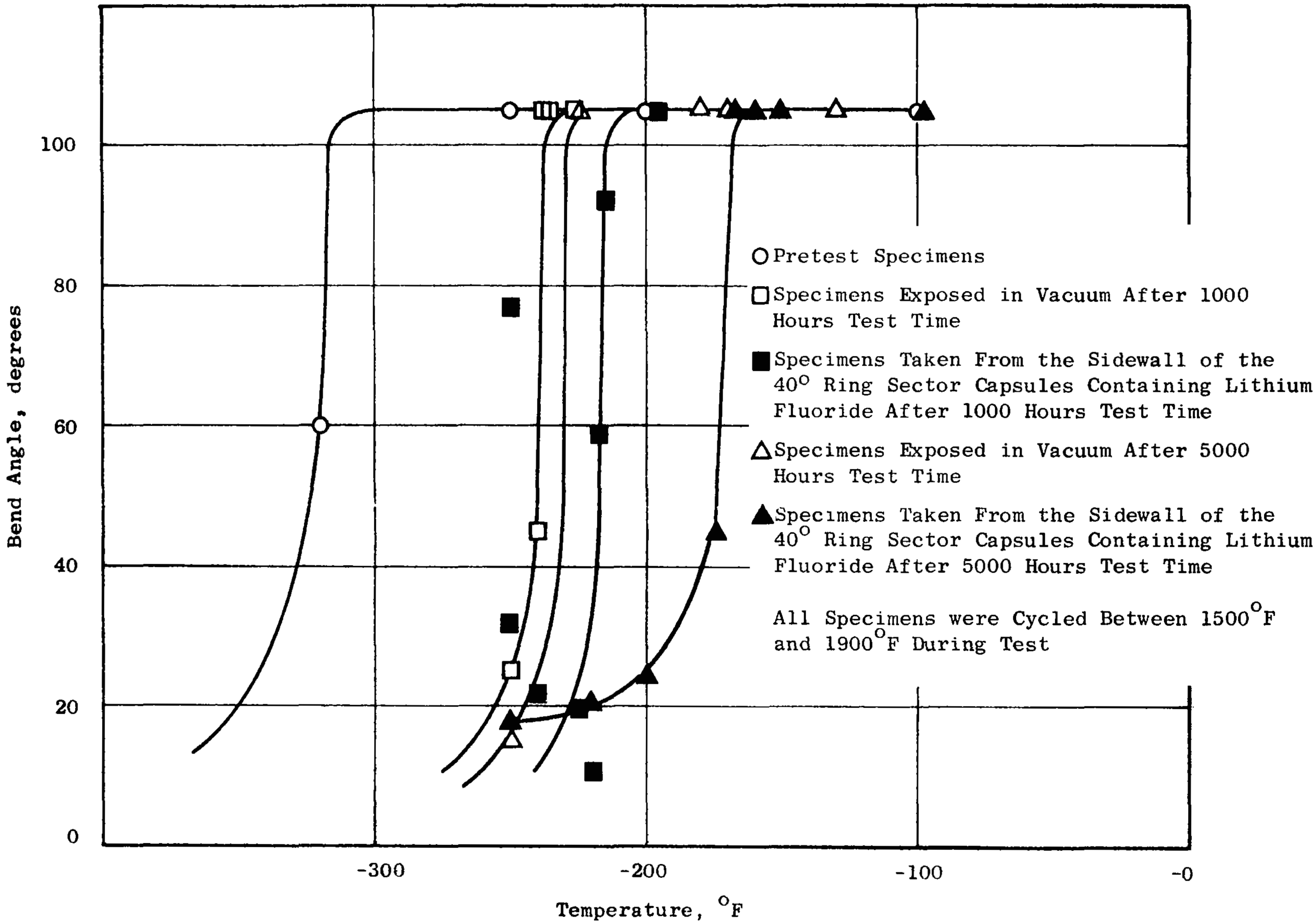


Figure 29. **Ductile-to-Brittle Bend Transition Temperatures of FS-85 Weldments.**

TABLE XII. DUCTILE-TO-BRITTLE BEND TRANSITION TEMPERATURES OF FS-85 LITHIUM FLUORIDE 40° RING SECTOR CAPSULE TESTS

Specimen	Exposure	Time, Hours	Ductile-to-Brittle Bend Transition Temperature, °F
Pretest	-	-	- 320
Weld Specimen[(a)]	Vacuum	1000	- 240
Weld Specimen	Vacuum	5000	- 230
Capsule Sidewall[(b)] Weld	Vacuum/LiF	1000	- 215
Capsule Sidewall Weld	Vacuum/LiF	5000	- 170

(a) Specimens exposed along with the 40° ring sector capsules and cycled from 1500°F to 1900°F.

(b) Specimens removed from sidewall weld of 40° ring sector capsules which were cycled from 1500°F to 1900°F.

VI. CONCLUSIONS AND RECOMMENDATIONS

The columbium alloys Cb-1Zr, FS-85 (28Ta-10.5W-0.9Zr-Bal Cb) and SCb-291 (10Ta-10W-Bal Cb) have demonstrated compatibility with lithium fluoride under thermal cyclic conditions between 1500°F and 1900°F for extended periods of time.

Although no corrosion effects of lithium fluoride on Cb-1Zr were observed this alloy lacks sufficient strength for lithium fluoride containment under thermal cyclic conditions at a maximum temperature of 1900°F. Under these high temperature conditions FS-85 containment is recommended.

NASA-Langley, 1970 —— 17 E-5385