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FOSSIL ENERGY

EVALUATION OF HIGH CHROMIUM OVERLAYS TO PROTECT LESS ALLOYED SUBSTRATES FROM CORROSION IN A COAL GASIFICATION ATMOSPHERE

Quarterly Report for December 1, 1978—February 28, 1979

By
Edward P. Sadowski

MASTER

Work Performed Under Contract No. EF-77-C-01-2621

The International Nickel Company, Inc.
Inco Research and Development Center
Sterling Forest
Suffern, New York

U. S. DEPARTMENT OF ENERGY

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EVALUATION OF HIGH CHROMIUM OVERLAYS TO PROTECT LESS ALLOYED SUBSTRATES FROM CORROSION IN A COAL GASIFICATION ATMOSPHERE

QUARTERLY REPORT

FOR PERIOD DECEMBER 1, 1978 - FEBRUARY 28, 1979

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Edward P. Sadowski

THE INTERNATIONAL NICKEL COMPANY, INC.
INCO RESEARCH AND DEVELOPMENT CENTER
STERLING FOREST
SUFFERN, NY 10901

PREPARED FOR DEPARTMENT OF ENERGY
MER/MATERIALS BRANCH
UNDER CONTRACT No. EF-77-C-01-2621

FOREWARD

This report covers work performed under Contract No. EF-77-C-01-2621 for the period December 1, 1978 through February 28, 1979. The work was administered by the Fossil Energy Division of the DOE with Mr. S. Dapkunas as project manager. The report was prepared by E. P. Sadowski of The International Nickel Company, Inco Research and Development Center, at Suffern, NY.

The work was performed under the direction of E. P. Sadowski as principal investigator. The various functions were carried out by the following personnel: L. R. Meisch - Welding; C. M. Wessner - Dy Chek; F. J. Veltry - Electron Microprobe Analysis; L. R. Sephton - Metallographic and Photography; R. G. Laggett - Mechanical Testing; M. M. Yanak and R. Liu - Chemical Analyses and R. H. Kane - Corrosion. The exposures in the coal gasification atmosphere were made at Martin-Marietta, Denver Colorado, under sub-contract. The program manager at Martin-Marietta was M. E. Wakefield.

ABSTRACT

The 1000 hour exposure at 1800°F in the coal gasification atmosphere was completed in this quarter. The gas composition consisted of 12%CO_2, 18%CO, 24%H_2, 5%CH_4, 1%NH_3, 1%H_2S and 39%H_2O. The gas was exchanged hourly. A detailed description of the test procedure and operation is appended to this report.

Visual observation of unwelded samples of the substrate after exposure indicate that the corrosive environment was quite severe. The 304L coupons were completely disintegrated and the INCONEL alloy 800H was scaled severely. The 310 SS appeared to be reasonably corrosion resistant.

The overlays of INCONEL Filler Metals 72, and R139 appeared corrosion resistant whereas the 309 overlay formed a scale which spalled.

Stress rupture testing of composite specimens is essentially complete. The composition of the substrate had most effects on the stress rupture stength and the overlaid INCOLOY alloy 800H had the highest stress rupture strength. Generally, the gas metal arc processes gave higher stress rupture strength than the submerged-arc process.

Microprobe analysis for elemental distribution showed that a higher Cr content was maintained for a greater depth in the overlay with the submerged-arc process than with the inert gas processes. Conversely, the Fe content of the overlays was the lowest with the submerged-arc process. The amount of Cr of the overlays deposited by the INCONEL Filler Metal 72 was not affected appreciably by the composition of the substrate except in the fusion area.

1.0 OBJECTIVE

The objective of this program is the development and evaluation of weld deposited overlays to provide resistance to corrosion in coal gasification atmospheres (CGA).

44%Cr and 31%Cr-15%Fe-3.5%Al nickel-base alloys will be overlain onto lower alloyed materials in order to provide protection from the corrosive atmosphere. These materials will be compared to a conventional stainless overlay material, AWS-ER309.

After the welding procedures are established for defect-free overlays, the effects of a 1000-hour exposure on weldments in a CGA at 1800°F on corrosion, metallurgical stability, mechanical properties, and the diffusion of major alloying elements will be determined.

2.0 SCOPE

The program is divided into four tasks as follows:

2.1 Task I - Development of Weld Deposition Techniques and Procedures

Weld deposition techniques and procedures for three weld processes will be established. The weld processes will be gas tungsten arc with a hot wire addition, gas metal-arc and submerged-arc. Three substrates will be overlain with INCONEL Filler Metal 72 (44%Cr), R139 (31.5%Cr-3.5%Al) and AWS-ER 309. The substrates will be AISI 304L and 310 and INCOLOY* alloy 800H. Single and double layer overlays will be deposited on both sides of the substrate.

2.2 Task II - Exposure to a Coal Gasification Atmosphere

A test cell will be constructed by Martin-Marietta, Denver and the weldments will be exposed for 1000 hours to a coal gasification atmosphere at 982°C (1800°F). The composition of the gas will be CO_2-12%, CO-18%, H_2-24%, NH_3-1%, H_2S-1% and H_2O balance. The weldments exposed will be sufficiently large for the machinery of tensile, hardness and corrosion specimens after exposure.

2.3 Task III - Testing

Specimens in both the pre- and post-exposure conditions will be tested. Data will be obtained on the extent of corroison, change in elemental distribution, microstructure, and mechanical properties as a result of exposure to a CGA.

Stress-rupture properties will be determined in air at 1800°F for time periods up to 1000 hours on "as-welded" specimens consisting of both overlay and substrate.

2.4 Task IV - Collate and Analyze Data and Final Report

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3.0 PROGRESS SUMMARY

3.1 Task I

Welding, Dy-chek and visual inspection at 10X of the overlay surfaces is complete. All welding was completed without difficulty.

3.2 Task II

The 1000 hour exposure of the weldments in a coal gasification atmosphere at 982°C (1800°F) is complete.

3.3 Task III

All pre-exposure bend, tensile and hardness traverse tests have been completed. Stress-rupture tests are nearing completion. Bulk chemical analysis are complete for all overlays. Elemental distribution by microprobe analysis has been completed on 37 of the 42 weldments in the "as-welded" condition. Metallographic examinations of specimens in the "pre-exposed" condition are partially complete. Preparation of samples for evaluation for corrosion is beginning.

4.0 WORK ACCOMPLISHED

4.1 Task II - Exposure to a Coal Gasification Atmosphere

The 1000 hour exposure of the weldments to a coal gasification atmosphere at 982°C (1800°F) was completed late in the quarter. The gas composition was similar to the gas used at ITTRI at the high H_2S level. The gas composition was: 12%Co_2, 18%CO, 24%H_2, 5%CH_4, 1%NH_3, 1%H_2S and 39%H_2O. A fresh supply of gas equivalent to 130% of the test cell volume was injected hourly. Initially, the gas injection was made on an hourly basis but after 168 hours of testing the injection cycle was changed to inject 1/4 of the amount required hourly at 15 minute intervals. The exposure was interrupted after 700 hours of test time when the temperature dropped 149°C (300°F) because of a power outage. The tests cell was purged with argon, the system temperatures increased to 982°C (1800°F) and the exposure started after injection of the CGA. A detailed description of the test (M. E. Wafefield, Martin-Marietta) is given in the attached appendix.

4.1.1 Corrosion Test Results

The appearance of the unwelded plate materials after exposure in the CGA are shown in Figures 1, 2 and 3. These samples were to serve as standards in order to judge the severity of the test environment. Based upon the results, the test conditions must be considered severe. The 304L was completely disintegrated (Figure 1), the INCOLOY alloy 800H appears to be scaled severely (Figure 2) and the type 310 SS attacked only modestly (Figure 3). Quantitative values on the depth of sulfide penetration will be obtained on the 310 SS and 800H.

Some examples of the protection obtained by the various overlay materials are shown in Figure 4. Based upon visual inspection, the INCONEL Filler Metal 72 overlays appeared to resist corrosion, the R139 overlays formed an adherent scale and the 309 overlays formed a scale that spalled. Quantitative values for depth of penetration will be obtained and photographs of all weldments will be given in a future report.

The manner in which small differences in corrosive conditions may affect the corrosion resistance of a material in CGA environments was observed on the 310 SS tubing used to transport the corrodents into the test cell. By visual examination, the tubing inside the test cell (982°C) was corroded, but not severely, on both the inside and outside and the passage was clear. However, a position of the tubing located about 5-6 inches above the test cell entrance was severely corroded, embrittled and the passage almost completely blocked with a deposit believed to be a sulfide.

The embrittled portion of the tubing was located above the heater in vermiculite and the temperature of the tubing was estimated to be about 810°C (1400°F) at that point.

4.2 Task III - Testing

Stress-rupture testing at 982°C (1800°F) and microprobe analyses has continued. All other pre-exposure testing is complete.

4.2.1 Stress-Rupture

The results of the stress-rupture tests completed in the last quarter are given in Table I. The stresses to produce rupture in 100 and 1000 hours are given in Tables II and III. The effects of substrate compositions, weld process and filler metal composition on the 1000 hour rupture stress of overlaid specimens are shown in Figures 5, 6, 7 and 8. The data indicates: (1) the highest stress-rupture strength was obtained when INCOLOY alloy 800H was used as the substrate material (Figure 5), (2) generally, the inert gas welding processes give higher stress-rupture strength than the submerged-arc process (Figure 6), (3) R139 and INCONEL Filler Metal 72 overlays give higher rupture strength than 309 on 304L except with the SAW process (Figure 7), and (4) R139 gives slightly higher rupture strength than INCONEL Filler Metal 72 (Figure 8).

4.2.2 Elemental Distribution

The distribution of Cr, Ni, Fe, Mn, Ti and Al from the surface of the overlay to a location in the substrate 5 mm (.20 inch) below the fusion line was determined on 10 weldments. In addition the distribution of Si was obtained on two SAW weldments since Si was picked up from the flux in earlier work. The results are given in Tables IV through IX.

The effect of weld process on the distribution of chromium in INCONEL Filler Metal 72 overlays deposited on INCOLOY alloy 800H, 304L and 310 SS is given in Figures 9, 10 and 11. The SAW process maintained a relatively high Cr content for a greater distance from the surface than either of the inert gas weld processes. The effect of the substrate compositon on INCONEL Filler Metal 72 overlays deposited by the SAW process is shown in Figure 12. The composition of the substrate did not have a large effect on the Cr composition of the overlay except in the area adjacent to the interface of the overlay and substrate. The effect of weld process on the distribution of Fe in INCONEL Filler Metal 72 overlays deposited on INCOLOY alloy 800H is shown in Figure 13. The Fe content of the overlay is the result of dilution of the filler metal (originally .20%Fe) by the substrate. The lowest Fe contents were obtained in the overlays for a distance of 6 mm (.25 inch) with the SAW process.

5.0 FUTURE WORK

5.1 Task I

None. Work is completed.

5.2 Task II

None. Complete.

5.3 Task III

Post-exposure evaluation including hardness and tensile tests, microprobe analysis for elemental distribution and corrosion evaluation will be made. Microprobe analyses on "as-welded" specimens will be completed.

6.0 OPEN ITEMS

There are no open items.

7.0 OVERALL STATUS

The overall program is progressing satisfactorily.

E. P. Sadowski
Project Manager

EPS:dp

TABLE I

STRESS-RUPTURE TESTS AT 982°C (1800°F) COMPLETED DURING QUARTER

Weld No. (a)	Process	Substrate	Filler Metal	Stress MPa	Stress psi	Life in Hours	% Elongation in 38.1 mm (1.5 in)
6192	GMAW	310	INCONEL FM72	13.8	2000	256	30
				9.0	1300	938	10
6193	GMAW	310	INCONEL FM72	10.3	1500	835	18
6197	GTAW-HW	310	INCONEL FM72	9.4	1350	807	17
6198	GMAW	INCOLOY alloy 800H	INCONEL FM72	13.8	2000	1408	10
6199	GMAW	INCOLOY alloy 800H	INCONEL FM72	15.2	2200	1345	9
6208	GTAW-HW	304L	R139	10.3	1500	1106	13
6209	GTAW-HW	304L	R139	10.3	1500	437	16
				8.3	1200	831	13
6210	GMAW	310	R139	9.0	1300	969	15
6211	GMAW	310	R139	9.0	1300	1919	31
6212	SAW	310	R139	24.1	3500	73	13
				17.2	2500	263	15
				12.1	1750	753	15
6213	SAW	310	R139	20.7	3000	108	21
				17.2	2500	152	18
				12.1	1750	675	23
				10.3	1500	906	23
6216	GMAW	INCOLOY alloy 800H	R139	31.0	4500	100	14
				24.1	3500	242	9
6217	GMAW	INCOLOY alloy 800H	R139	31.0	4500	102	9
				24.1	3500	247	7
6218	SAW	INCOLOY alloy 800H	R139	31.0	4500	81	9
				24.1	3500	203	8
				16.2	2350	813	15
6219	SAW	INCOLOY alloy 800H	R139	31.0	4500	73	13
				24.1	3500	202	8
				16.2	2350	764	7
6220	GTAW-HW	INCOLOY alloy 800H	R139	20.7	3000	403	9
				15.9	2300	1191	15
6321	GTAW-HW	INCOLOY alloy 800H	R139	24.1	3500	253	9
				20.7	3000	403	9
				16.9	2450	852	12

(a) Even numbered welds are double layer overlay; odd numbered welds are single layer overlays.

TABLE II

STRESS IN MPa (psi) TO PRODUCE RUPTURE IN 100 HOURS AT 982°C (1800°F)

Process	SAW - Double Layer			SAW - Single Layer		
Substrate	304	310	800H	304	310	800H
FM 309	11.6 (1675)	-	-	10.3 (1500)	-	-
FM R139	15.2 (2200)	22.4 (3250)	30.0 (4300)	13.8 (2000)	21.0 (3050)	29.0 (4200)
FM 72	11.0 (1600)	15.9 (2300)	24.7 (3575)	11.0 (1600)	15.4 (2225)	29.3 (4250)

Process	GMAW - Double Layer			GMAW - Single Layer		
Substrate	304	310	800H	304	310	800H
FM 309	13.1 (1900)	-	-	11.7 (1700)	-	-
FM R139	15.9 (2300)	19.3 (2800)	31.0 (4500)	15.2 (2200)	20.0 (2900)	31.0 (4500)
FM 72	17.2 (2500)	18.6 (2700)	31.0 (4500)	13.6 (1975)	17.9 (2600)	29.7 (4300)

Process	GTAW-HW Double Layer			GTAW-HW Single Layer		
Substrate	304	310	800H	304	310	800H
FM 309	13.1 (1900)	-	-	10.3 (1500)	-	-
FM R139	17.2 (2500)	22.4 (3250)	29.3 (4250)	15.7 (2275)	20.7 (3000)	31.0 (4500)
FM 72	14.0 (2025)	20.7 (3000)	31.0 (4500)	13.3 (1925)	17.7 (2575)	32.1 (4650)

TABLE III

STRESS IN MPa (psi) TO PRODUCE RUPTURE IN 1000 HOURS AT 982°C (1800°F)

Process	SAW - Double Layer			SAW - Single Layer		
Substrate	304	310	800H	304	310	800H
FM 309	6.9 (1000)	-	-	5.9 (860)	-	-
FM R139	6.7 (970)	(a) 9.7 (1400)	15.2 (2200)	6.6 (960)	(a) 9.7 (1400)	(a)15.0 (2175)
FM 72	6.2 (900)	8.1 (1175)	10.3 (1500)	6.4 (925)	8.6 (1250)	10.3 (1500)

Process	GMAW - Double Layer			GMAW - Single Layer		
Substrate	304	310	800H	304	310	800H
FM 309	7.4 (1075)	-	-	7.0 (1010)	-	-
FM R139	9.7 (1400)	8.8 (1275)	(a)17.6 (2550)	9.3 (1350)	10.5 (1525)	17.6 (2550)
FM 72	10.2 (1475)	8.8 (1275)	15.2 (2200)	9.1 (1325)	9.7 (1400)	16.2 (2350)

Process	GTAW-HW Double Layer			GTAW-HW Single Layer		
Substrate	304	310	800H	304	310	800H
FM 309	7.4 (1075)	-	-	7.9 (1150)	-	-
FM R139	10.5 (1525)	12.1 (1750)	16.6 (2400)	7.8 (1125)	12.1 (1750)	16.2 (2350)
FM 72	7.6 (1100)	9.8 (1425)	14.8 (2150)	8.6 (1250)	9.0 (1300)	15.9 (2300)

(a) Extrapolated - still in test.

TABLE IV

DISTRIBUTION OF ELEMENTS - INCONEL FM72 OVERLAY ON 310 SS - GMAW PROCESS

Distance from Surface (mm)	Weld 6192 - Double Layer Ni	Cr	Fe	Mn	Ti	Al
0	44.75	40.46	12.73	0.43	.17	.28
1	44.52	41.79	11.10	0.63	.21	.34
2	45.45	41.57	11.11	0.62	.23	.41
3	45.90	40.15	12.35	0.43	.20	.18
4	45.36	41.16	13.00	0.48	.21	.47
5	45.26	41.63	11.11	0.62	.33	.27
6	37.78	36.11	24.41	0.73	.10	.25
7	35.49	38.48	24.17	0.88	.18	.32
7.5	37.08	35.88	25.33	0.79	.21	.50
7.6	39.22	37.50	21.33	0.86	.28	.25
7.7	37.76	36.68	21.67	0.69	.20	.35
7.8	39.08	38.68	20.99	0.92	.14	.00
7.9	39.87	37.79	21.01	0.84	.15	.31
Interface						
8.0	18.88	26.12	52.54	1.60	.00	.00
8.1	18.78	26.28	52.32	1.70	.00	.00
8.2	18.61	26.38	52.02	1.70	.03	.00
12.9	18.71	25.55	49.62	1.90	.00	.00

Distance from Surface (mm)	Weld 6193 - Single Layer Ni	Cr	Fe	Mn	Ti	Al
0	39.08	36.15	23.42	.66	.11	.38
1	38.93	36.06	21.19	.93	.22	.19
2	38.51	38.92	20.93	.88	.13	.15
3	39.14	36.34	24.42	.78	.17	.09
4	39.83	36.40	23.15	.74	.12	.17
5	38.77	35.97	20.67	.89	.17	.18
5.5	38.53	36.61	22.59	.98	.22	.36
5.6	32.38	42.04	20.51	1.01	.16	.12
5.7	36.51	38.71	24.51	.70	.14	.24
5.8	34.25	37.94	21.49	.80	.15	.31
Interface						
5.9	18.82	25.97	50.52	1.67	.00	.00
6.0	19.21	25.94	51.03	1.69	.01	.00
6.1	18.85	25.54	52.21	1.74	.00	.00
10.8	19.04	26.21	49.70	1.72	.00	.00

TABLE V

DISTRIBUTION OF ELEMENTS IN R139 OVERLAYS ON 304L SUBSTRATE - GMAW PROCESS

Distance from Surface (mm)	Weld 6204 - Double Layer Ni	Cr	Fe	Mn	Al	Ti
0	42.49	34.88	18.99	.50	2.21	.25
1	42.95	34.05	17.36	.61	2.69	.38
2	42.38	33.95	18.32	.56	2.51	.20
3	43.16	35.14	16.10	.57	2.77	.46
4	43.07	34.75	17.76	.53	2.88	.47
5	37.84	32.43	23.15	.97	2.91	1.41
6	36.84	30.84	26.89	.86	3.30	.85
6.5	36.25	31.01	28.75	.71	2.31	.26
6.6	36.80	32.01	27.52	.82	2.34	.39
6.7	37.01	32.50	26.12	.71	2.19	.29
6.8	36.51	31.28	28.92	.71	2.27	.32
6.9	37.08	31.90	26.43	.87	2.21	.30
7.0	36.27	31.24	29.17	.83	2.05	.27
Interface						
7.1	9.89	21.20	67.99	1.79	.00	.03
7.2	10.52	19.45	67.92	1.79	.00	.00
7.3	10.26	19.02	67.60	1.79	.00	.00
12.0	9.28	18.46	65.51	1.73	.00	.00

Distance from Surface (mm)	Weld 6205 - Single Layer Ni	Cr	Fe	Mn	Al	Ti
0	39.23	33.43	20.89	.76	2.78	.24
1	39.73	34.08	20.85	.84	2.82	.49
2	38.56	32.60	23.76	.63	2.69	.51
3	36.93	32.21	26.44	.85	2.39	.28
3.1	37.41	32.24	24.57	.71	2.69	.31
3.2	36.81	32.55	25.09	.92	2.18	.23
3.3	35.56	31.48	26.79	.90	2.91	.53
3.4	33.42	30.84	31.32	.92	1.91	.22
3.5	36.49	32.09	28.05	.72	2.75	.30
3.6	31.49	31.75	32.74	.86	1.61	.15
Interface						
3.7	9.85	18.80	68.11	1.81	.00	.00
3.8	10.00	19.17	68.96	1.69	.00	.00
3.9	9.97	19.09	67.07	1.85	.00	.00
8.6	9.67	19.37	68.55	1.66	.00	.00

TABLE VI

DISTRIBUTION OF ELEMENTS - R139 OVERLAY ON 310 SS - SAW PROCESS

Distance from Surface (mm)	Weld 6212 - Double Layer						
	Ni	Cr	Fe	Si	Mn	Ti	Al
0	44.63	33.91	17.70	.44	.90	.32	2.56
1	43.90	33.15	17.59	.61	1.12	.25	2.59
2	44.24	33.97	19.86	.33	.92	.39	2.48
3	42.66	31.68	18.65	.47	.92	.47	2.69
4	44.54	33.74	18.87	.49	.94	.33	2.54
5	44.26	33.31	18.93	.35	.92	.35	2.79
6	37.70	30.95	25.93	.44	1.14	.32	2.11
7	38.32	31.71	26.55	.23	.92	.41	1.96
8	38.83	30.99	24.68	.38	1.10	.49	2.22
9	38.23	31.83	26.44	.62	1.30	.16	2.18
10	37.75	30.28	25.19	.52	1.22	.30	2.39
10.5	37.42	31.79	24.85	.33	1.05	.37	2.20
10.6	35.61	31.04	25.05	.46	1.30	.37	2.24
10.7	28.61	30.25	38.07	.38	1.21	.08	1.42
Interface							
10.8	21.01	26.78	47.75	.30	1.88	.00	.83
10.9	20.43	26.10	47.10	.20	1.71	.00	.74
11.0	20.33	26.34	46.34	.20	1.59	.01	.84
(a)15.7	19.36	25.66	50.18	.18	1.63	.00	.67

Distance from Surface (mm)	Weld 6213 - Single Layer						
	Ni	Cr	Fe	Si	Mn	Ti	Al
0	34.25	29.64	30.98	.37	1.04	.15	1.80
1	32.99	29.87	30.49	.30	1.03	.15	1.92
2	34.36	31.39	26.75	.42	1.27	.62	2.17
3	33.99	31.01	29.16	.43	1.25	.32	2.08
4	34.47	31.34	27.83	.09	1.06	.30	2.07
5	34.42	30.91	24.22	.33	.96	.63	2.42
5.5	35.56	31.07	27.61	.21	1.21	.34	2.33
5.6	35.72	31.58	27.84	.14	1.27	.17	2.15
5.7	34.82	30.80	27.23	.17	1.37	.25	1.92
5.8	34.93	30.37	28.47	.42	1.24	.22	2.18
5.9	33.95	31.34	27.80	.41	1.29	.23	2.43
Interface							
6.0	18.53	25.77	48.01	.14	1.58	.00	.81
6.1	18.89	25.69	47.88	.25	1.73	.00	.72
6.2	18.50	25.80	46.62	.24	1.72	.00	.76
(a)10.9	18.69	26.37	52.16	.15	1.52	.00	.67

(a) 5 mm from interface (fusion line).

TABLE VII

DISTRIBUTION OF ELEMENTS - R139 OVERLAY ON 310 SS - GTAW-HW PROCESS

Distance from Surface (mm)	Weld 6214 - Double Layer					
	Ni	Cr	Fe	Mn	Ti	Al
0	40.10	33.24	21.42	.80	.28	1.87
1	40.09	32.16	24.16	.65	.34	2.50
2	40.65	32.63	22.23	.63	.40	2.23
3	41.44	32.96	20.02	.98	.25	2.00
4	40.81	33.14	23.59	.67	.65	2.80
5	39.30	31.84	24.77	.75	.15	1.88
6	34.57	31.45	30.74	.97	.38	1.63
6.5	34.21	30.31	30.65	1.07	.11	.94
6.6	33.60	31.06	30.11	1.07	.18	1.04
6.7	34.32	30.97	29.96	1.10	.20	1.25
6.8	34.76	31.14	29.93	1.11	.21	1.12
6.9	34.46	30.17	30.59	1.10	.48	1.60
7.0	34.79	30.97	31.92	.91	.21	1.22
7.1	27.26	30.02	39.47	1.12	.23	1.23
Interface						
7.2	18.78	25.91	49.28	1.32	.02	.00
7.3	18.92	25.80	52.66	1.66	.03	.00
7.4	18.63	26.62	51.89	1.58	.03	.00
7.5	18.53	26.10	52.26	1.69	.02	.00
(a)12.1	18.73	26.24	50.67	1.77	.01	.00

Distance from Surface (mm)	Weld 6215 - Single Layer					
	Ni	Cr	Fe	Mn	Ti	Al
0	33.83	29.87	31.96	.90	.12	1.15
1	34.53	29.27	31.36	1.11	.63	1.23
2	34.08	30.15	32.32	.91	.16	1.03
3	34.23	31.05	31.78	.97	.16	.91
4	33.69	30.19	33.15	.89	.12	.97
4.1	33.77	30.66	31.19	1.08	.25	1.30
4.2	33.31	29.98	28.79	1.30	.16	.87
4.3	32.90	30.28	29.64	1.27	.20	1.13
4.4	33.85	31.12	31.59	1.05	.27	1.25
4.5	31.96	30.43	33.26	1.16	.20	1.00
4.6	31.30	29.75	34.83	1.03	.21	1.02
Interface						
4.7	23.81	27.34	44.69	1.37	.04	.00
4.8	19.25	28.34	50.90	1.66	.02	.00
4.9	18.89	26.39	53.18	1.62	.02	.00
5.0	19.18	26.18	52.14	1.68	.02	.00
(a)9.6	18.99	26.63	51.76	1.74	.02	.00

(a) 5 mm from interface (fusion line).

TABLE VIII

DISTRIBUTION OF ELEMENTS - R139 OVERLAY ON INCOLOY ALLOY 800H - GMAW PROCESS

Distance from Surface (mm)	Weld 6216 - Double Layer Ni	Cr	Fe	Mn	Ti	Al
0	44.76	34.31	18.20	.57	.33	3.76
1	43.87	34.22	17.76	1.17	.31	3.56
2	44.69	33.28	16.99	.79	.40	4.08
3	44.82	33.51	16.59	.62	.50	3.41
4	43.36	33.32	17.31	.59	.69	3.43
5	41.98	31.80	24.56	.65	.26	3.61
6	42.07	32.92	21.66	.65	.63	3.48
7	41.76	31.59	22.49	.63	.51	2.92
7.1	40.40	31.28	22.35	.51	.59	2.74
7.2	41.52	31.06	25.10	.62	.31	3.14
7.3	40.78	31.57	25.22	.80	.26	3.25
7.4	41.10	32.13	25.32	.76	.31	3.97
7.5	40.42	30.60	22.88	.50	.50	3.18
7.6	49.90	30.19	25.35	.57	.41	2.56
Interface						
7.7	31.59	20.12	44.53	.69	.34	.56
7.8	30.33	20.81	44.54	.73	.36	.75
7.9	30.84	20.65	45.19	.64	.34	.60
8.0	31.67	20.79	44.90	.66	.34	.54
(a)12.6	32.60	20.42	44.15	.69	.34	.63

Distance from Surface (mm)	Weld 6217 - Single Layer Ni	Cr	Fe	Mn	Ti	Al
0	41.83	30.38	22.72	.60	.29	2.98
1	41.36	30.37	24.76	.64	.25	3.52
2	41.88	31.40	24.48	.70	.24	3.32
3	41.97	32.01	22.70	.51	.24	3.12
3.5	42.77	30.57	24.20	.51	.19	3.10
3.6	40.78	31.95	22.23	.77	.38	3.53
3.7	42.05	31.85	22.28	.67	.34	3.58
3.8	42.28	31.31	22.32	.68	.25	3.84
3.9	42.01	31.22	21.80	.59	.35	2.90
4.0	42.32	31.36	22.08	.67	.40	3.29
4.1	39.64	29.28	27.95	.67	.26	3.26
4.2	37.32	27.30	31.17	.78	.29	2.30
Interface						
4.3	32.10	20.28	44.36	.74	.37	.52
4.4	32.20	20.95	43.96	.81	.40	.69
4.5	31.24	21.36	44.62	.75	.31	.61
4.6	31.91	20.62	44.54	.70	.31	.68
(a)9.2	31.92	21.27	44.68	.68	.35	.59

(a) 5 mm from interface (fusion line).

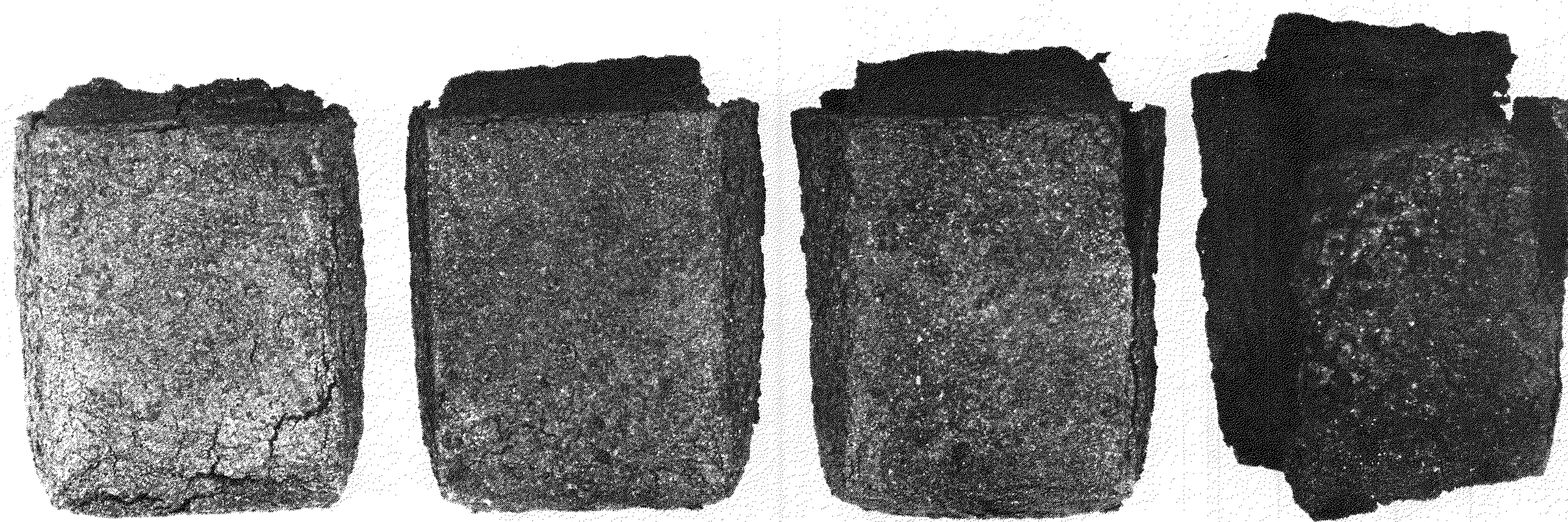


FIGURE 1 - Appearance of unwelded 304L after exposure.

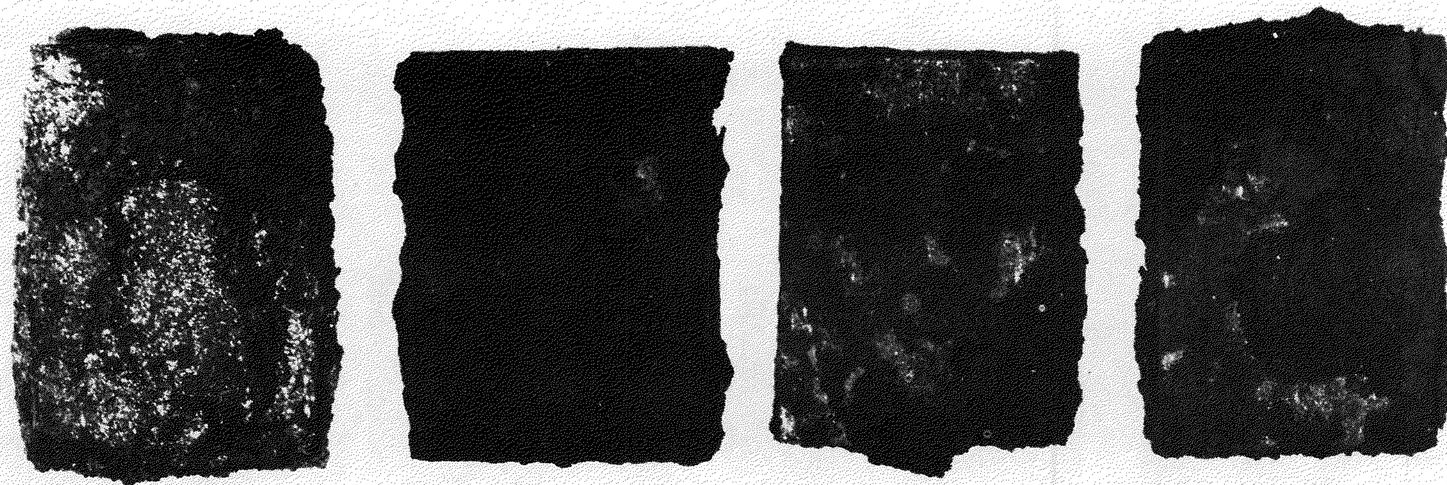
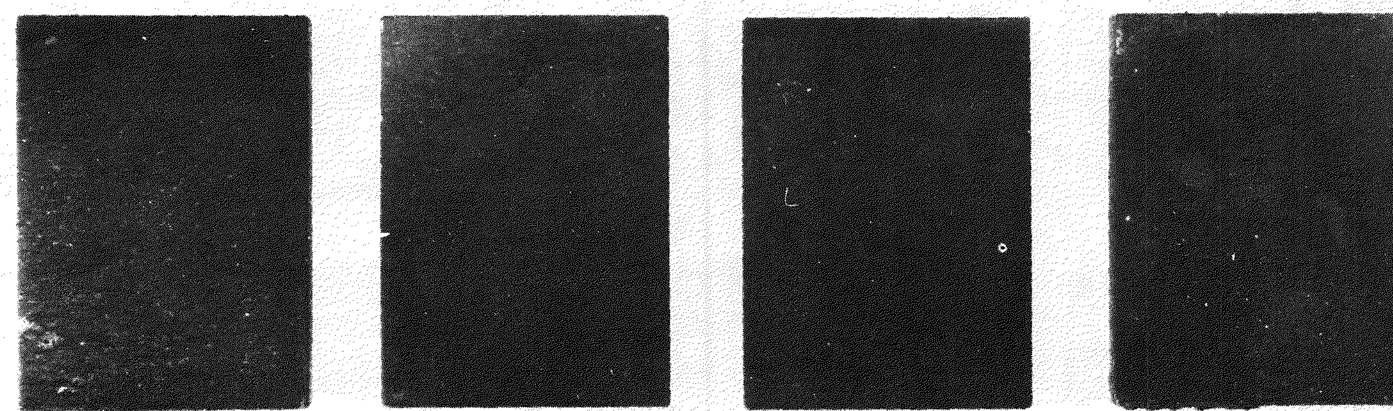


FIGURE 2 - Appearance of INCOLOY alloy 800H after exposure.



<u>FIGURE 3</u> - Appearance of unwelded 310 SS after exposure.

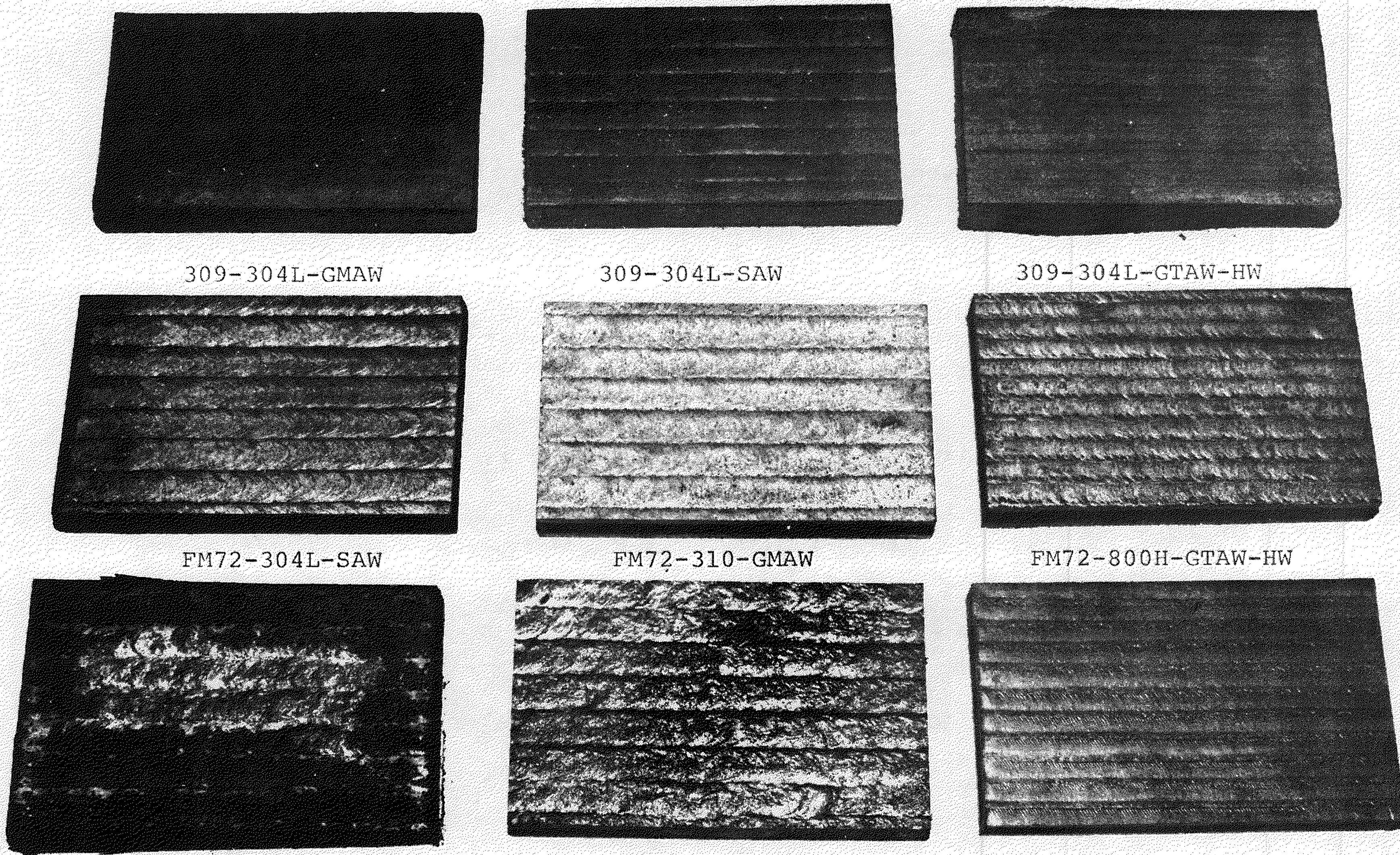


FIGURE 4 - Representative appearance of overlays after exposure.

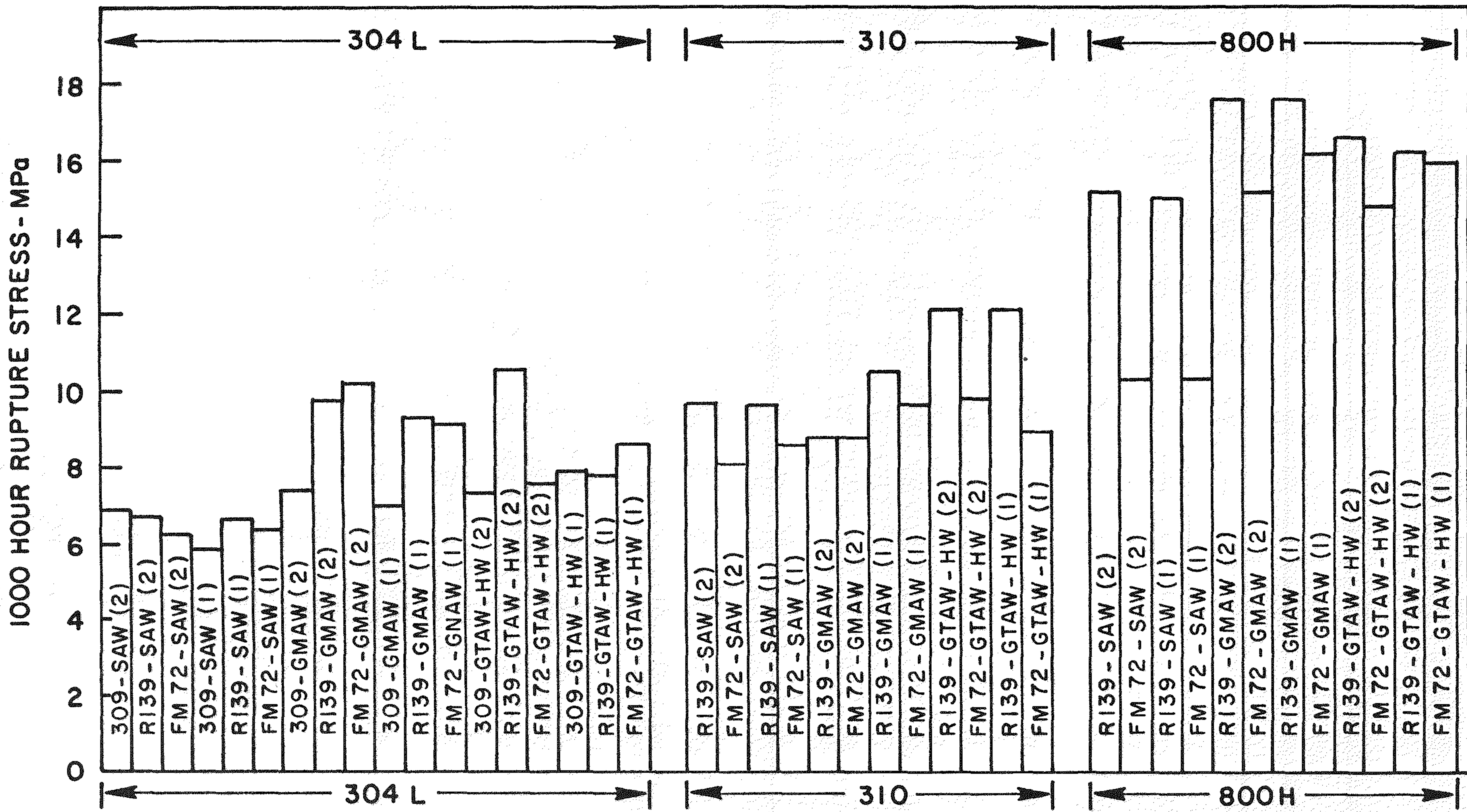


FIGURE 5 - EFFECT OF SUBSTRATE ON 1000 HOUR RUPTURE STRESS AT 982 °C (1800 °F)

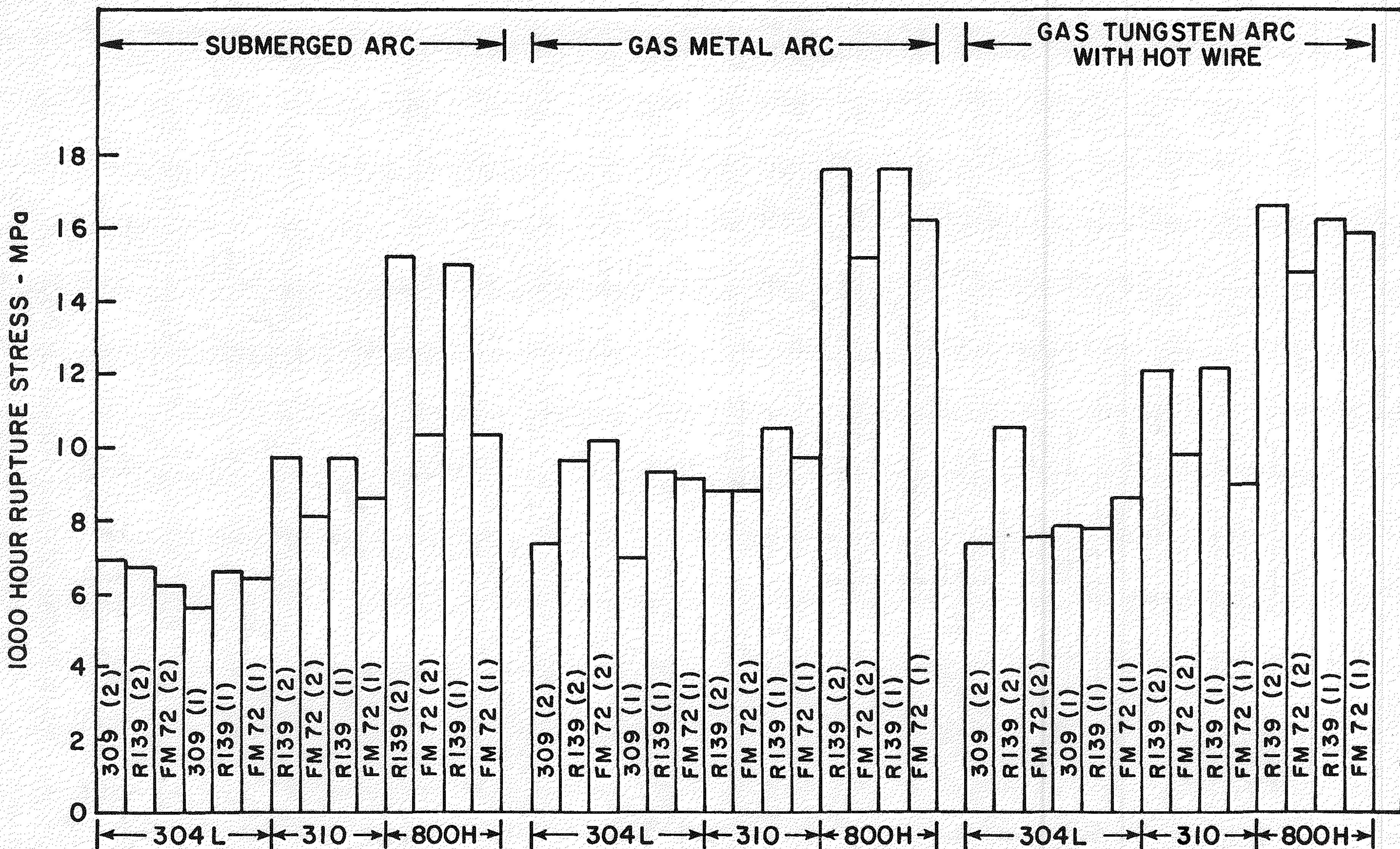


FIGURE 6 - EFFECT OF WELDING PROCESS ON 1000 HOUR RUPTURE STRESS AT 982°C (1800°F).

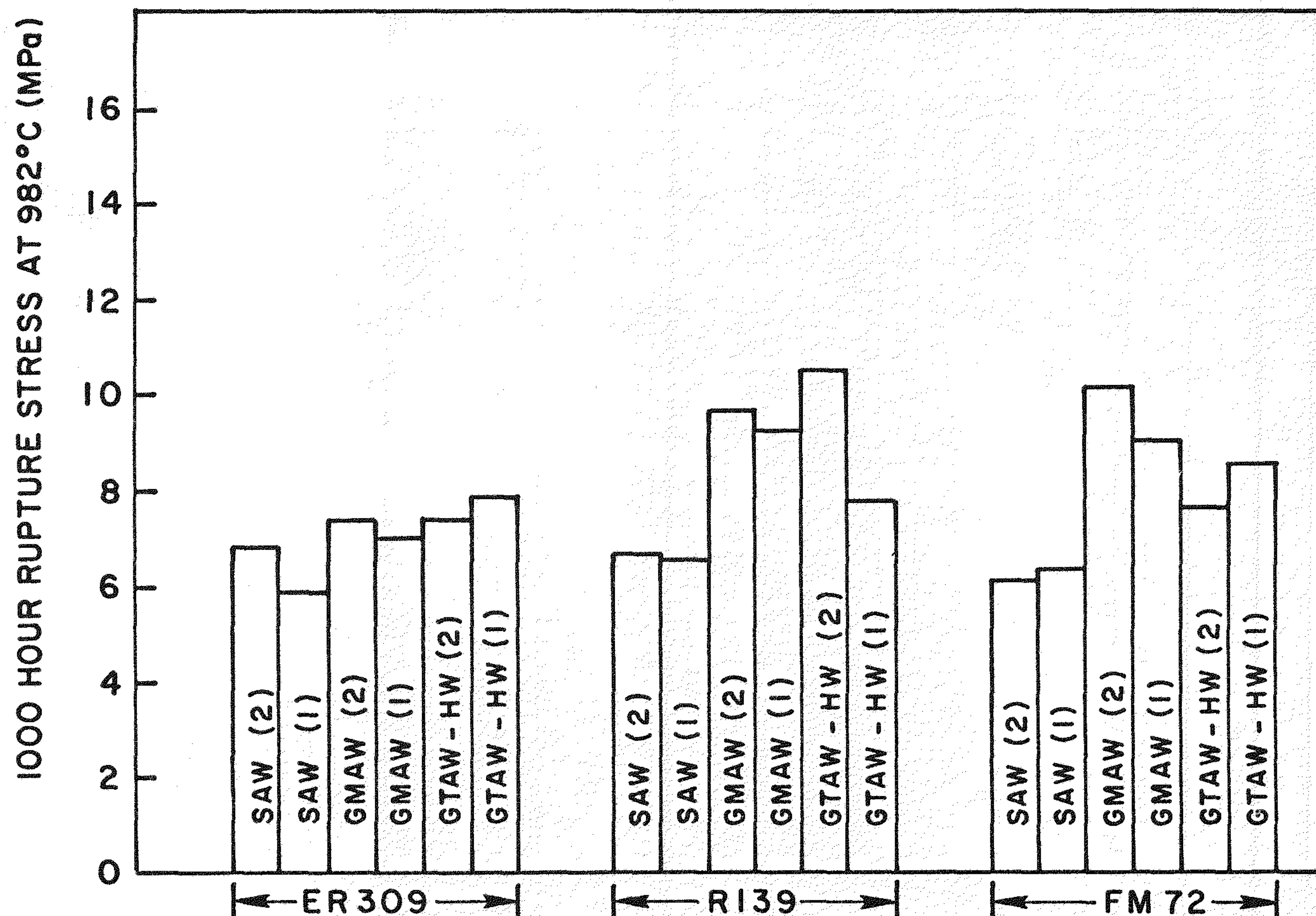


FIGURE 7 - EFFECT OF OVERLAY COMPOSITION ON THE 1000 HOUR STRESS RUPTURE STRENGTH AT 982°C (1800°F) OF 304L SUBSTRATE.

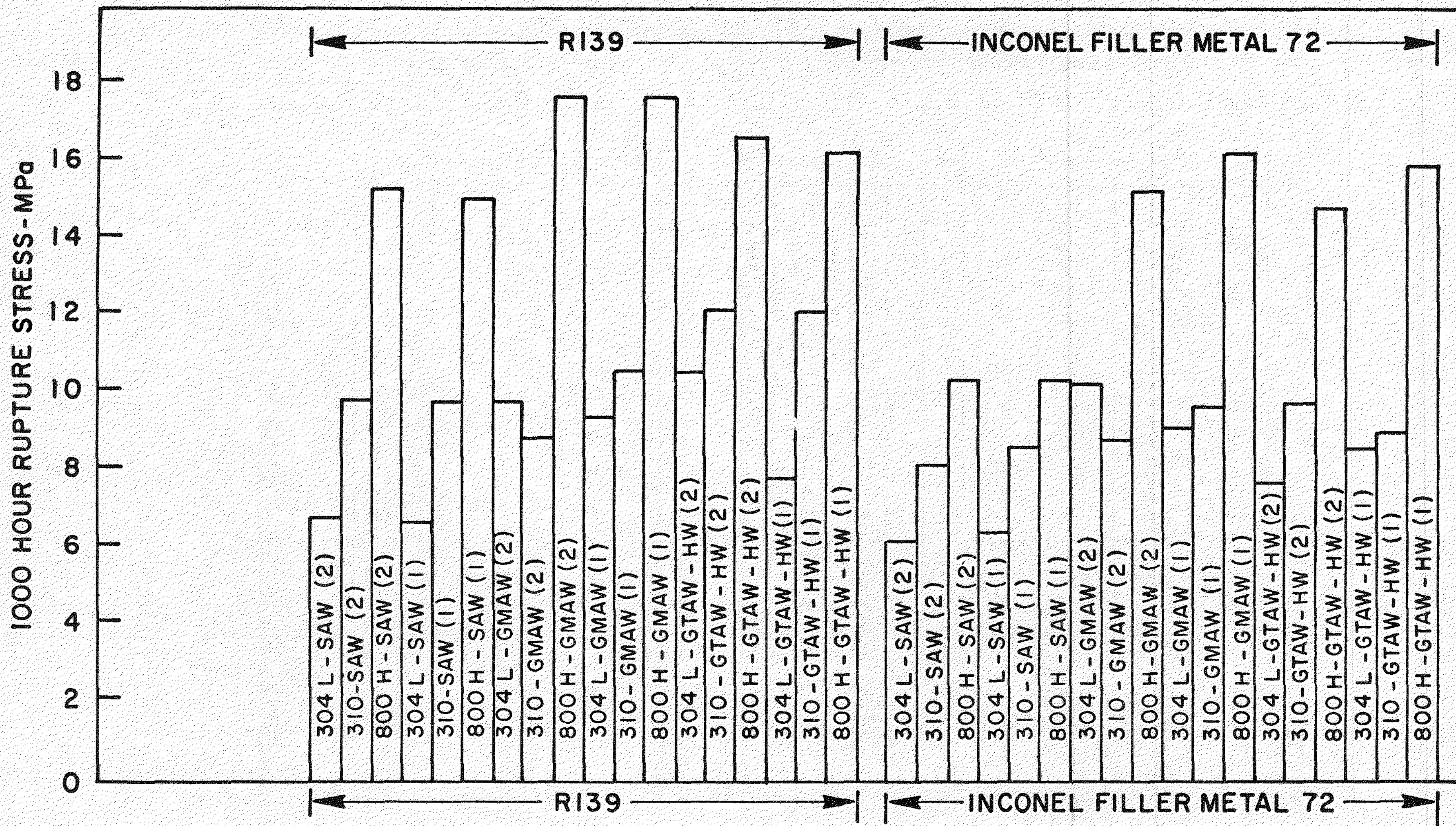


FIGURE 8 - EFFECT OF FILLER METAL COMPOSITION ON 1000 HOUR RUPTURE STRESS AT 982°C (1800°F) - ALL SUBSTRATES AND WELD PROCESSES.

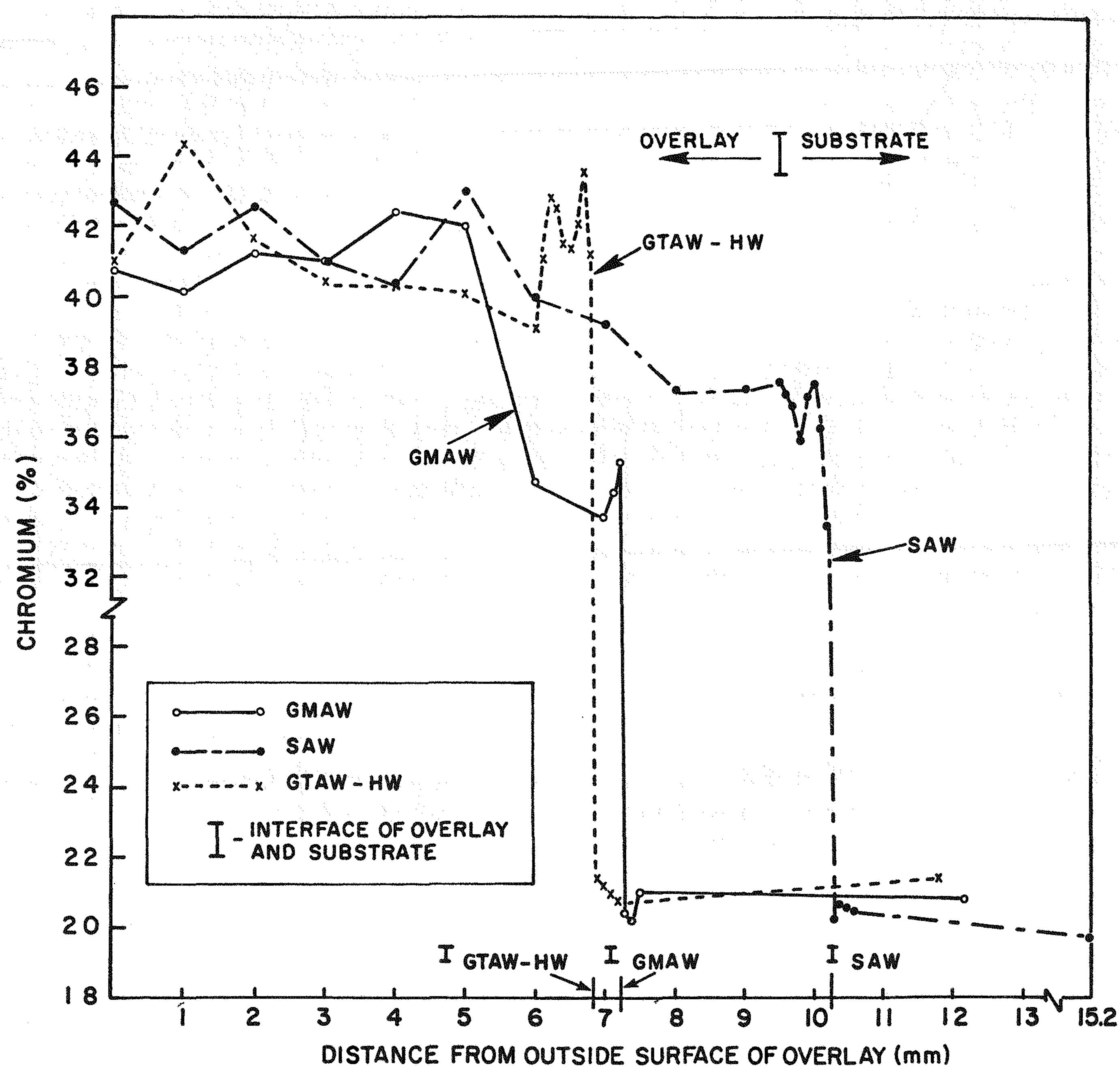


FIGURE 9 - EFFECT OF WELD PROCESS ON CHROMIUM DISTRIBUTION - INCONEL FM 72 DEPOSITED ON INCOLOY ALLOY 800H (DOUBLE LAYER OVERLAYS).

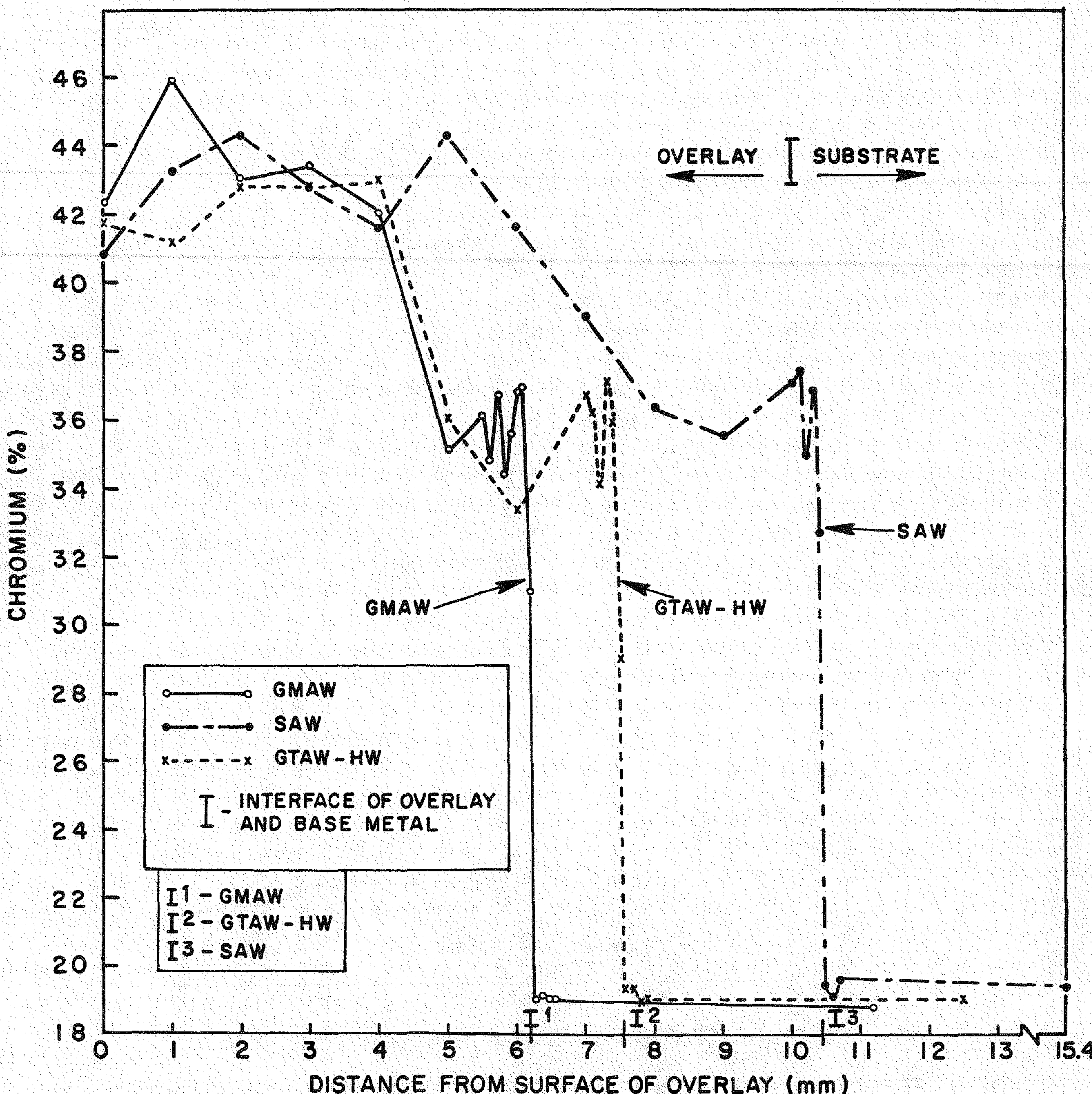


FIGURE 10 - EFFECT OF WELD PROCESS ON CHROMIUM DISTRIBUTION - INCONEL FM 72 DEPOSITED ON 304 L (TWO LAYER OVERLAY).

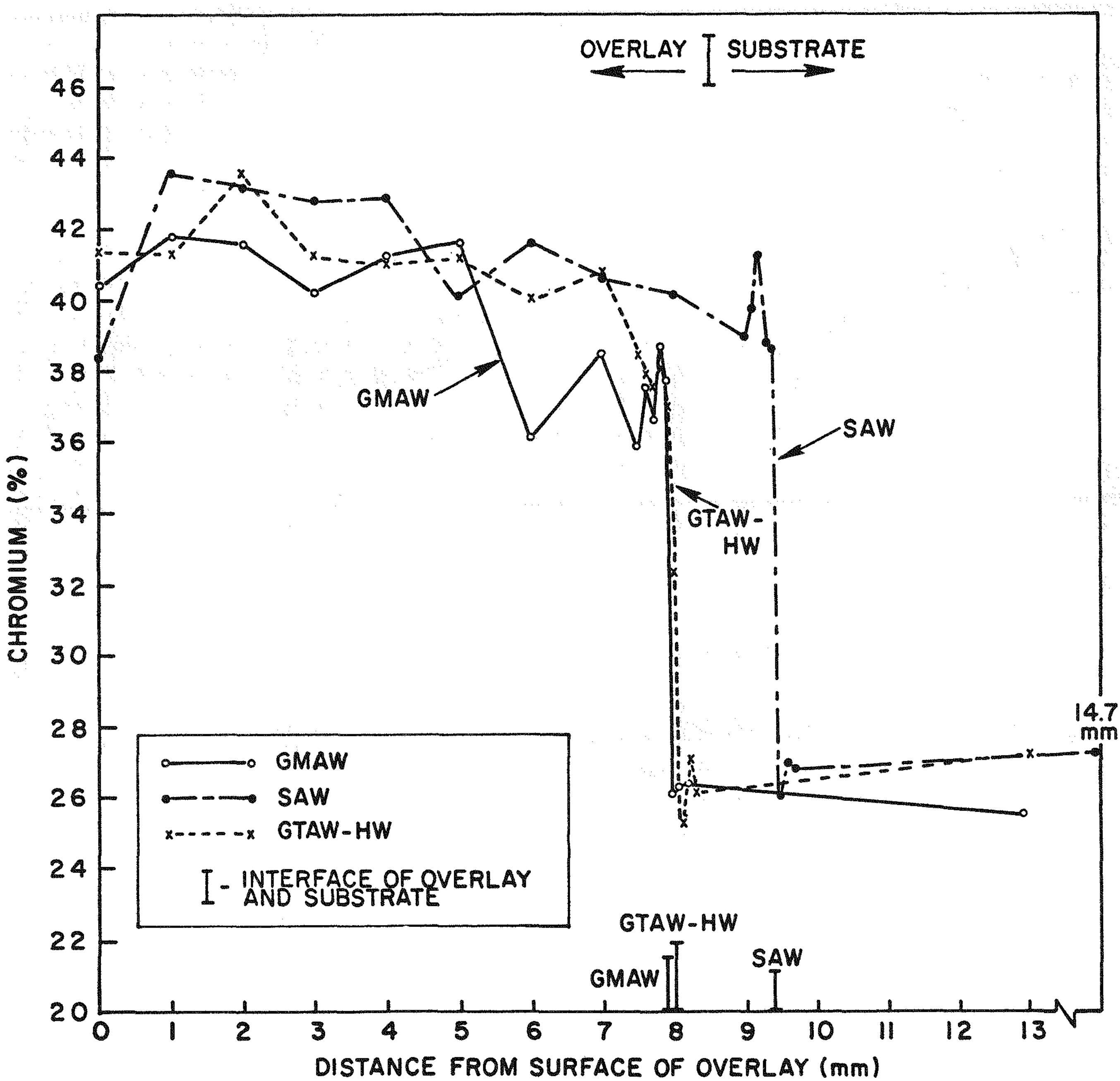


FIGURE 11 - EFFECT OF WELD PROCESS ON CHROMIUM DISTRIBUTION - INCONEL FM 72 ON 310 SS (DOUBLE LAYER).

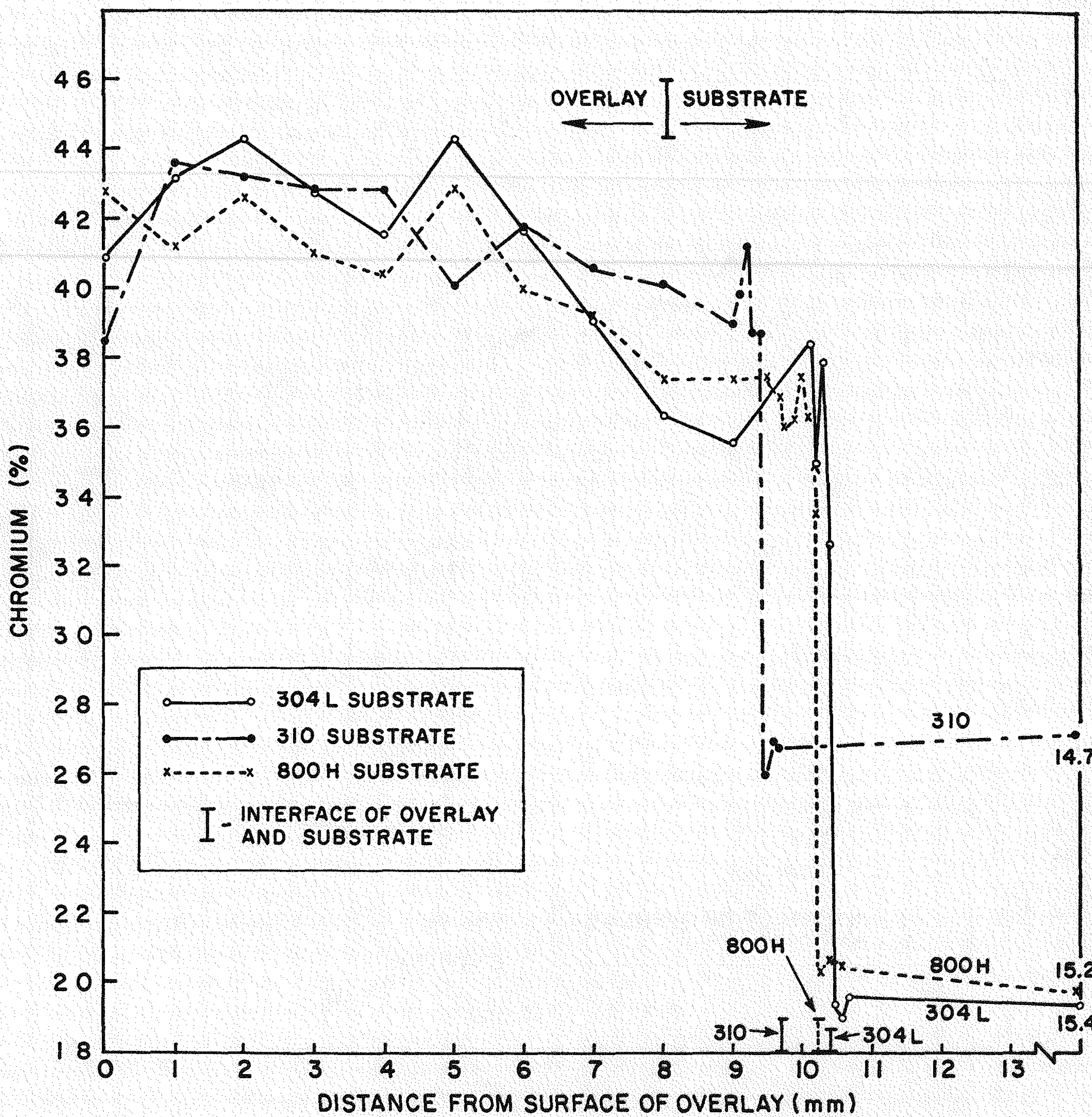


FIGURE 12 - EFFECT OF SUBSTRATE ON CHROMIUM DISTRIBUTION OF INCONEL FM 72 OVERLAYS DEPOSITED BY THE SAW PROCESS (DOUBLE LAYERS).

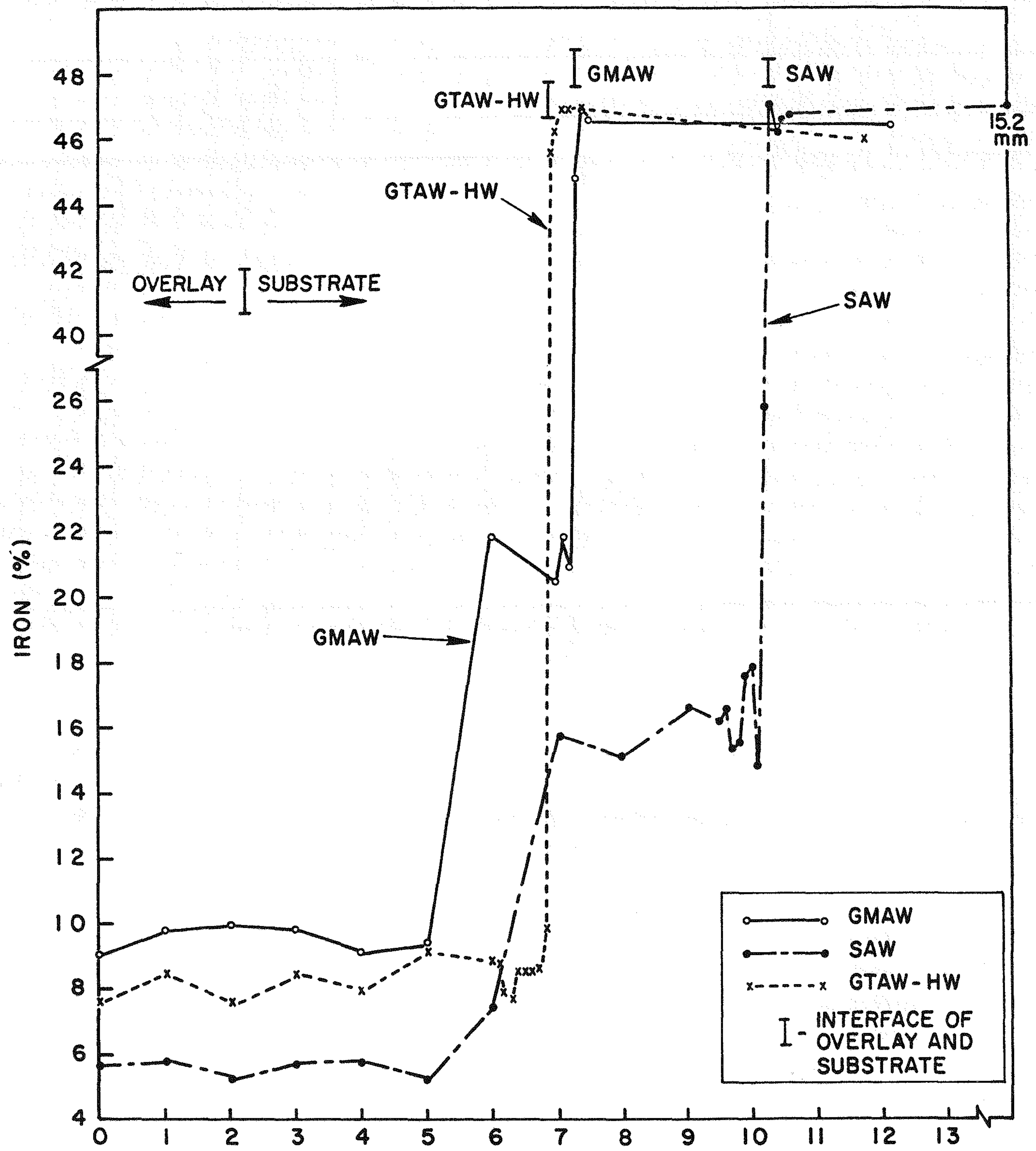


FIGURE 13 - EFFECT OF WELD PROCESS ON DISTRIBUTION OF IRON IN INCONEL FM 72 DEPOSITED ON INCOLOY ALLOY 800 H (DOUBLE LAYERS).

APPENDIX

DETAILS OF 1000 HOUR TEST IN A COAL GASIFICATION ENVIRONMENT

1.0 SUMMARY

A variety of weld overlay materials were exposed to a coal gasification mixture at 1800°F for a period of 1000 hours. The corrosion observed ranged from minor to severe, depending on the material. The facility description, test operation, and preliminary results are discussed in this report.

This exposure was conducted for the International Nickel Company, Inc., Suffern, New York, who also provided the test specimens and criteria for test conditions and who will perform the post-test metallurgical analyses.

1.1 Objective

The objective was to provide a controlled thermal/gaseous environment on a number of test specimens to allow metallurgical evaluation of corrosion to be made.

1.2 Test Conditions

Test Temperature:	1800°F
Pressure:	Local Atmospheric
Gas Environment:	12% CO_2
	18% CO
	24% H_2
	5% CH_4
	1% NH_3
	1% H_2S
	39% H_2O
Gas Exchange Rate:	Hourly
Duration:	1000 hours

2.0 TEST SETUP

2.1 Oven

The overall test facility is shown in Figure 1. The oven was housed within the large central box, and surrounded with vermiculite insulation. The controls for temperature and for commodity injection are on the left side. The commodity injection system was mounted on the face of the box, with the gas supply bottles on the right side.

A schematic of the oven configuration appears as Figure 2. The fiberblock insulation was stacked in place directly on the concrete floor. The lower section of the lightweight castable refractory was placed on the fiberblock as a support for the remaining oven assembly. The heater elements were cast directly into the refractory material. Redundant heaters were installed, since the heaters would not be serviceable with the oven at temperature. All connection wires were passed through the backside of the refractory into the vermiculite insulation area. The connecting wires were stranded nickel, and were attached using tubular stainless steel crimps.

The specimen holding box was constructed of 310 SS, and all closures were completely welded. The inlet pipe was vertical to allow the gas/liquid mixture to make its way quickly to the 1800^{o}F area. The outlet pipe was on the side of the box, and a baffle forced the exhaust to be from the bottom or coolest area of the box. Extra pipes were included on the box as spares, in the event of any blockage during operation.

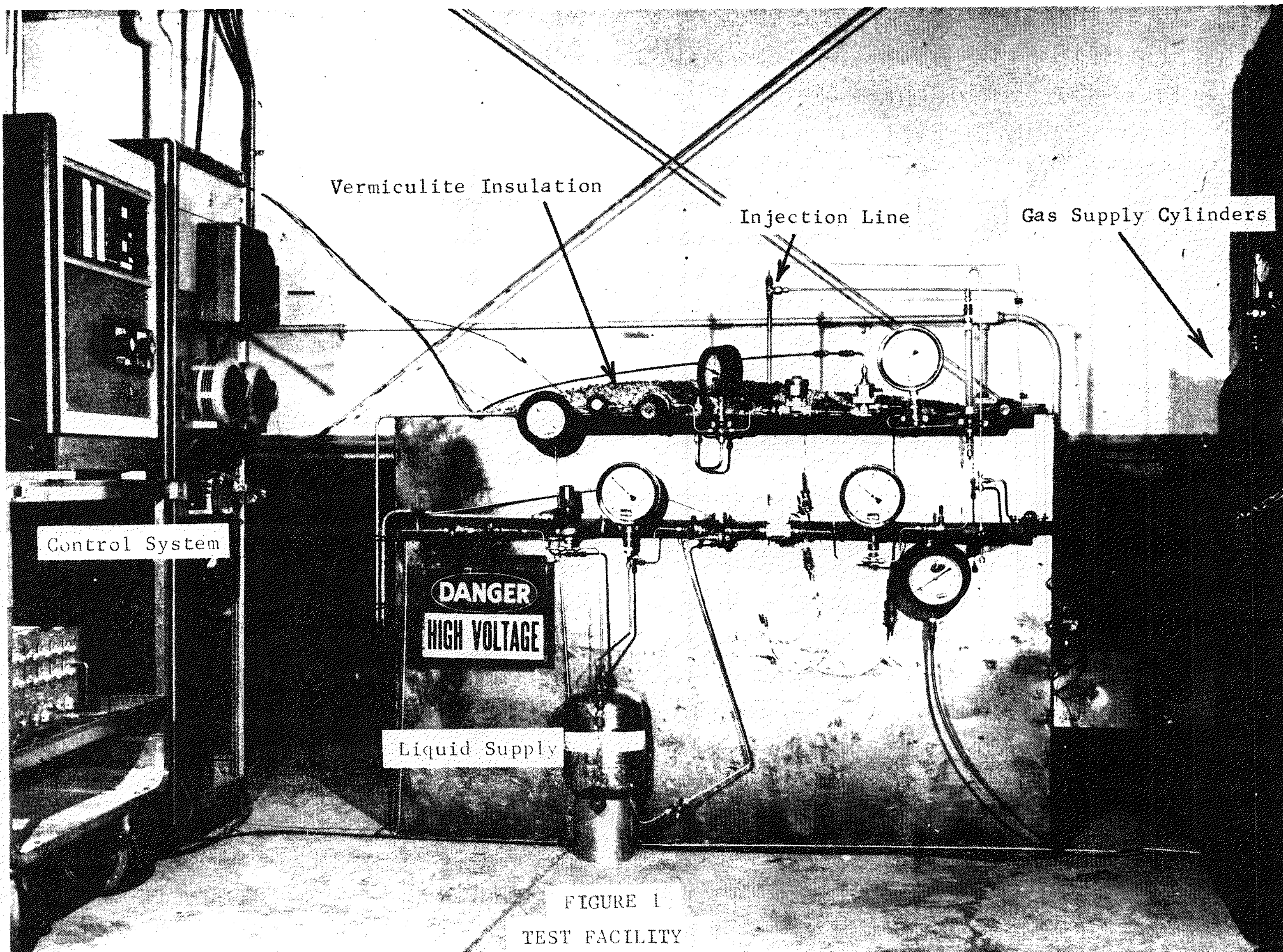


FIGURE 1
TEST FACILITY

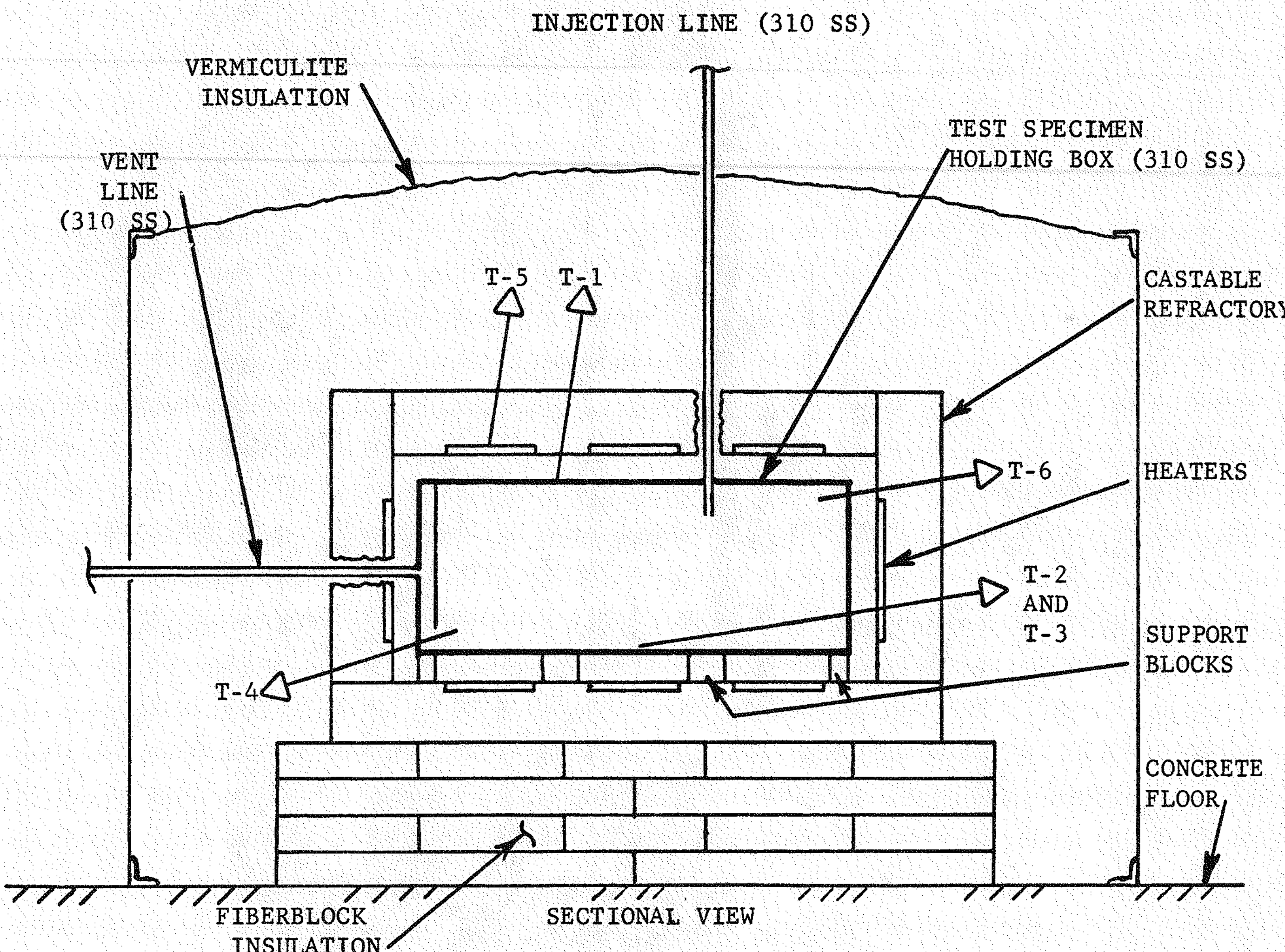


FIGURE 2

OVEN CONFIGURATION

2.2 Injection System

The desired gas composition in the specimen box was:

Carbon Dioxide (CO_2)	12%
Carbon Monoxide (CO)	18%
Hydrogen (H_2)	24%
Methane (CH_4)	5%
Ammonia (NH_3)	1%
Hydrogen Sulfide (H_2S)	1%
Water (H_2O)	39%
	100%

This was achieved by mixing a liquid consisting of H_2O, H_2S and NH_3 with a gas mixture of CO, CO_2, CH_4 and H_2. The injection system is shown schematically in Figure 3.

The liquid supply was a vessel of the mixed fluid, pressurized to 20 psig. The vessel outlet was controlled by tandem solenoid valves leading to a needle valve and flowmeter, then into the specimen box. HV-7 was provided to allow the initial setting of the HV-6 needle valve. The injection line into the specimen box was 1/8 inch diameter tubing with a small I.D. All materials in the liquid system were stainless steel, Teflon or glass.

The gas supply system used high pressure storage bottles of the gas mixture. This was regulated down to 20 psig, then routed through tandem solenoid valves into a control orifice and flowmeter into the specimen box. Most of this system was stainless steel, although corrosion was not anticipated to be a problem.

The injections of gas and liquid were to occur simultaneously one time each hour. The quantity injected was calculated to totally exchange the gas volume in the specimen box. An electronic timer was adjusted to provide the proper actuation duration for the four solenoid valves.

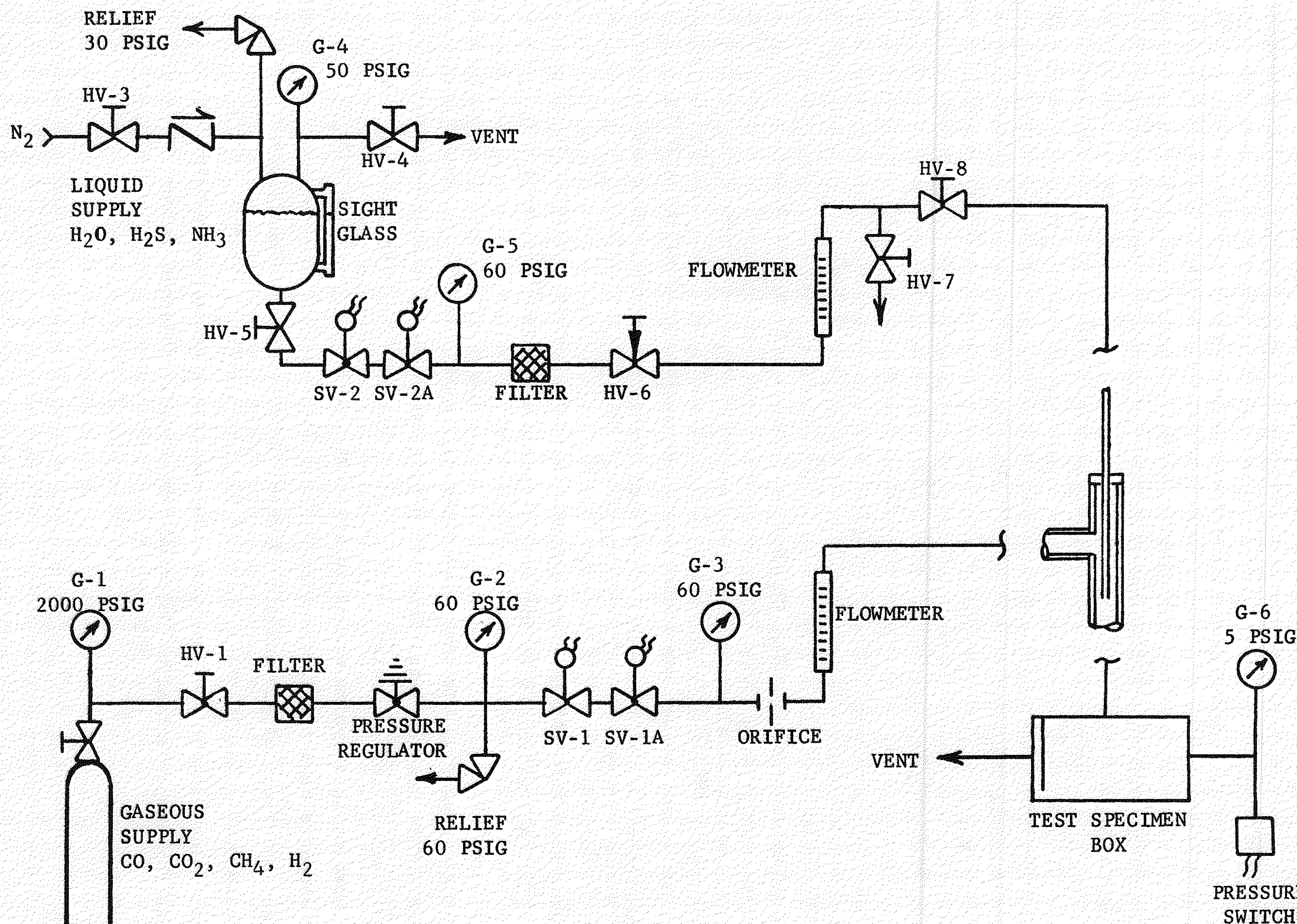


FIGURE 3

INJECTION SYSTEM SCHEMATIC

2.2.1 Test Volume

The free volume of the test chamber was calculated to be 4.9 ft^3. The volume of the test specimens was calculated to be 2.6 ft^3. The effective volume of the test chamber was then 2.3 ft^3. All of the injection rate calculations assumed a 3.0 ft^3 volume, or a 30% margin.

2.2.2 Gas Injection Calculations

The criteria required 59% of the injected commodity to be provided from the mixed gas supply, that is;

$$12\%\ CO_2 + 18\%\ CO + 24\%\ H_2 + 5\%\ CH_4 = 59\%.$$

$$(59\%)(3.0\ ft^3) = 1.77\ ft^3 \text{ replenished from gas supply}$$

Determine weight of gas to be injected:

Gas;	*Density (lb/ft^3)	x Concentration	x Volume (ft^3)	= Wt (lb)
CO_2;	$\frac{0.765}{35.13} = 0.0218$	x 0.203	x 1.77	= 0.00783
CO;	$\frac{0.765}{55.19} = 0.0139$	x 0.305	x 1.77	= 0.00750
H_2;	$\frac{0.765}{766.8} = 0.000997$	x 0.407	x 1.77	= 0.000718
CH_4;	$\frac{0.765}{96.35} = 0.00794$	x 0.085	x 1.77	= 0.00119
		1.000		0.0172 lb

$$* \text{ Density} = \frac{P(144)}{RT} = \frac{(12)(144)}{R(2260)} = \frac{0.765}{R}$$

Where P = pressure, lb/in^2 absolute

R = gas constant, ft lb/lbm $^{\circ}$Rankine

T = temperature, $^{\circ}$Rankine

For an injection duration of 8.4 seconds each hour, the gas flowrate required was:

$$\frac{0.0172}{8.4 \text{ sec}} = 0.00205 \text{ lb/sec}$$

This was calculated to require a control orifice of 0.082 inch I.D. with an inlet pressure of 20 psig.

2.2.3 Liquid Injection Calculations

The criteria required 41% of the injected commodity to be provided from the mixed liquid supply, that is: 39% H_2O + 1% H_2S + 1% NH_3 = 41%.

$$(41\%)(3.0 \text{ ft}^3) = 1.23 \text{ ft}^3 \text{ replenished from gas supply}$$

Determine weight of the liquid to be injected:

Liquid; Density (lb/ft^3) x Concentration x Volume (ft^3) = Wt (lb)

H_2O;	$\frac{0.765}{85.9}$ = 0.00891	x	.9512	x	1.23	=	0.0104
NH_3;	$\frac{0.765}{90.9}$ = 0.00842	x	.0244	x	1.23	=	0.0003
H_2S;	$\frac{0.765}{45.5}$ = 0.0168	x	.0244	x	1.23	=	0.0005
			1.000				0.0112 lb

For an injection duration of 8.4 seconds each hour, the liquid flowrate required was:

$$\frac{0.0112 \text{ lb}}{8.4 \text{ sec}} = 0.00133 \text{ lb/sec}$$

2.3 Power System

The heaters were operated from a three phase variable voltage control. A parallel wiring arrangement was used, to minimize the system voltage. The top, middle and bottom oven heaters were connected to separate phases, to allow individual control if excessive temperature differences were noted.

2.4 Instrumentation

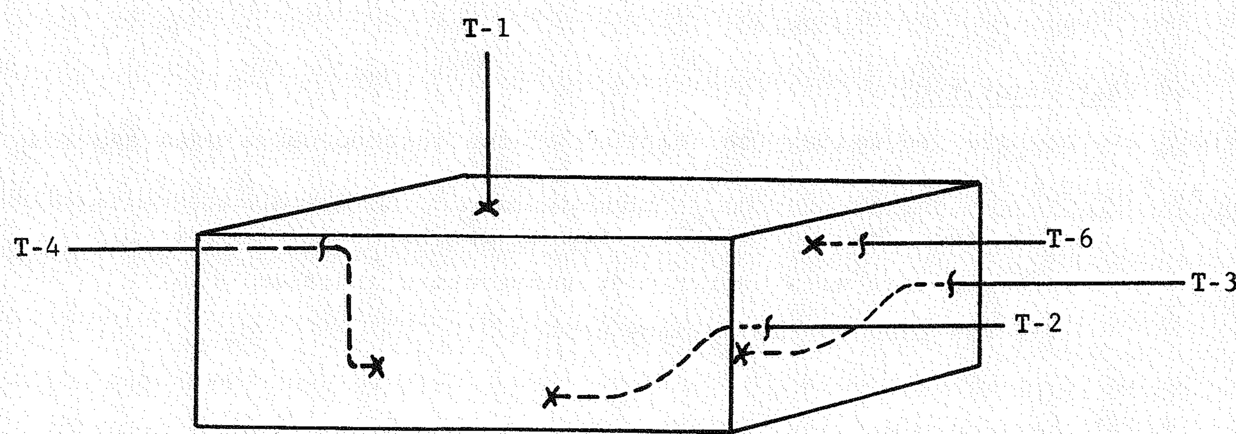
A schematic indicating the location of the thermocouples used appears as Figure 4.

T-1, T-2, T-3 and T-4 were used to monitor oven temperature
T-5 was used as a heater over-temperature shutdown
T-6 was used as the primary control

All thermocouples were chromel-alumel, with 310SS, 1/8 inch diameter sheath material.

2.5 Test Specimens

Each of the test specimens consisted of a substrate material clad with a filler metal. Fourty-two of these were exposed to the coal gasification environment. The size of each specimen was approximately 11 x 6 5/8 x 1 1/2 inches, and each specimen weighed approximately 33 lbs. The various substrate materials, filler metal materials, and welding processes are listed on Table 1.



T-5 is located on
a heater element

FIGURE 4

THERMOCOUPLE LOCATIONS

10

TABLE 1
TEST SPECIMEN MATERIALS

Specimen No.	Process(a)	Substrate	Filler Metal
6180	GMAW	304L	309
6181	GMAW	304L	309
6182	SAW	304L	309
6183	SAW	304L	309
6184	GTAW-HW	304L	309
6185	GTAW-HW	304L	309
6186	GMAW	304L	72(b)
6187	GMAW	304L	72
6188	SAW	304L	72
6189	SAW	304L	72
6190	GTAW-HW	304L	72
6191	GTAW-HW	304L	72
6192	GMAW	310	72
6193	GMAW	310	72
6194	SAW	310	72
6195	SAW	310	72
6196	GTAW-HW	310	72
6197	GTAW-HW	310	72
6198	GMAW	800H	72
6199	GMAW	800H	72
6200	SAW	800H	72
6201	SAW	800H	72
6202	GTAW-HW	800H	72
6203	GTAW-HW	800H	72
6204	GMAW	304L	R139
6205	GMAW	304L	R139
6206	SAW	304L	R139
6207	SAW	304L	R139
6208	GTAW-HW	304L	R139
6209	GTAW-HW	304L	R139
6210	GMAW	310	R139
6211	GMAW	310	R139
6212	SAW	310	R139
6213	SAW	310	R139
6214	GTAW-HW	310	R139
6215	GTAW-HW	310	R139
6216	GMAW	800H	R139
6217	GMAW	800H	R139
6218	SAW	800H	R139
6219	SAW	800H	R139
6220	GTAW-HW	800H	R139
6221	GTAW-HW	800H	R139

(a) GMAW = Gas Metal Arc
SAW = Submerged Arc
GTAW-HW = Gas Tungsten Arc with Hot Wire

(b) INCONEL Filler Metal 72

In addition to these clad specimens, small samples of various metals were also placed in the specimen box. Each of these measured approximately 1/4 in. x 1.0 in. x 1.0 in. Four each of 304L stainless steel, 310 stainless steel, and Incoloy 800H were tested.

The arrangement of the test specimens during their exposure was random, in order to balance any effects due to possible varying conditions throughout the specimen box. This arrangement is shown in Figure 5.

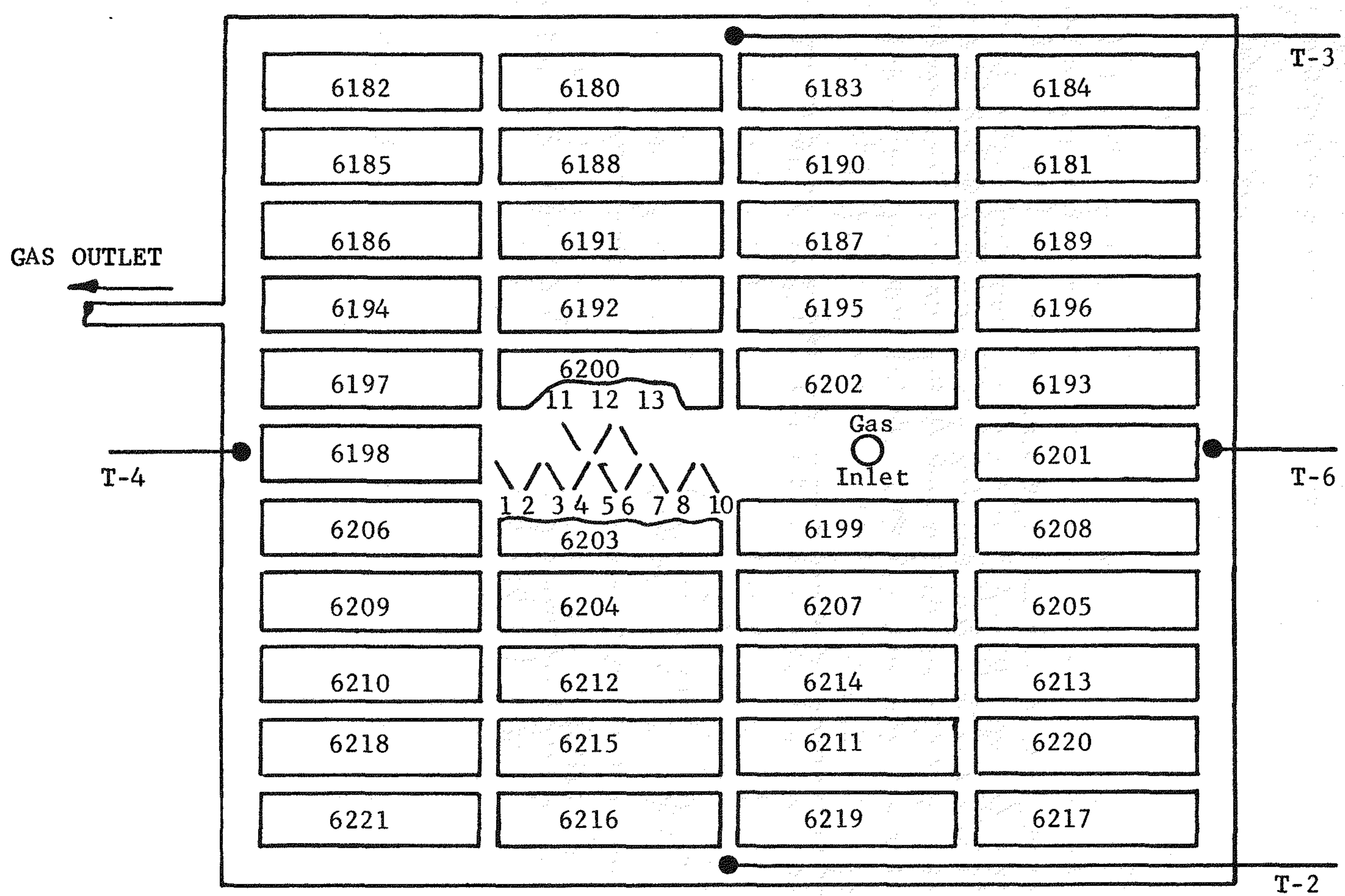


FIGURE 5

ARRANGEMENT OF SPECIMENS
(TOP VIEW)

3.0 TEST METHOD AND PROCEDURE

The test specimens were oriented in the oven with the 11 inch dimension vertical. As an added precaution against loss of identification, the identifying number was stamped into the lower surface substrate material of each specimen. The oven lid was then welded in place using a 310 stainless steel shielded metal arc weld, and the heaters and insulation were placed to complete the assembly.

A step-by-step startup, monitoring and shutdown procedure was used to ensure safety and test fidelity. All procedural aspects effecting specimen environment were discussed with the International Nickel Co. Inc. representative to ensure proper exposure.

The exposure was started by first purging the specimen box with gaseous argon to remove all normal atmospheric gases. The temperature was then slowly increased to 1800°F, while maintaining the argon environment. The automatic cycling system controlling the liquid and gas injection was then started, which began the 1000 hour exposure duration. Initially, the injection duration was increased to exchange the volume several times to remove the argon and replace it with the test environment. The automatic cycling continued throughout the 1000 hour exposure, after which argon was again used to purge out all test gases.

The system was allowed to cool to room temperature, and, after removal of the insulation and heaters, the lid of the specimen chamber was removed by grinding away the weld material at its circumference.

The test specimens were removed and inspected, their identification was verified, they were photographed, and then prepared for shipment to the International Nickel Co., Inc. for evaluation.

4.0 TEST OPERATIONS HISTORY

A trial operation of the oven system was conducted from 11-8-78 through 11-13-78. This assured the capability of the system to achieve and maintain 1800^{o}F. The lid of the specimen box was set into position, but was not welded for this checkout. At this time the liquid and gas injection systems were checked out; although no injection was directed into the specimen box.

The gas system was operated to verify the size of the control orifice by comparison to the flowmeter F-1. After satisfactory calibration of the orifice, the flowmeter was removed from the system.

The liquid system was operated to verify adjustment of the micrometer valve HV-6 by comparison to the flowmeter F-2. The flowmeter was left in the system throughout the test to verify that the HV-6 adjustment did not shift.

Following successful checkout of the oven and injection systems, the specimens were loaded, the lid was welded on, the heaters and insulation reassembled, and system heating was begun. The heaters were energized on 12-1-78, with an argon atmosphere maintained until 12-7-78. At this time the oven was at temperature and the injection system was activated. On 12-13-78, the injection logic was changed to inject a smaller quantity at 15 minute intervals. This was done to reduce the back pressure noted in the specimen box during the injection period. The total quantity of gas injected every hour was not changed.

On 12-17-78, the current readout on the system supplying power to the heaters in the oven walls was found to be zero. The other two systems, supplying power to the oven top and bottom, were increased slightly to compensate for the loss.

On 1-5-79, the temperature was found to be 300^{o}F low, indicating a power outage the previous night. The specimen box was purged with argon, and the system temperature was again increased. On 1-6-79, the injection system was restarted. Also on 1-6-79, the upper heater bank became inoperative, leaving only the lower heaters. Again, the voltage was increased.

On 1-9-79, a portion of the lower heater bank also failed; so the voltage was increased on the remaining heaters. Although the differential temperature from the top to the bottom of the specimen box increased to nearly 60°F, the necessary voltage to the remaining heaters was still only 182 VAC compared to their 230 VAC rating. This proved to be the last heater problem, and the system continued operating in this mode until 1-20-79, when the injection and heating were terminated and the specimen box was purged with argon to terminate the 1000 hour exposure.

During the entire test period, the rate of usage of the liquid and gaseous supplies was monitored to assure a proper overall rate. The liquid usage was monitored by observation of the sight glass on the liquid supply vessel while the gas usage was calculated from pressure decay of the supply pressure cylinder.

The temperature history of the exposure is presented in Table 2.

TABLE 2

TEMPERATURE HISTORY

Date	T-1	T-2	T-3	T-4	
12-1-78	59	60	60	58	Purged with argon.
12-2-78	759	755	751	730	
12-3-78	1100	1096	1094	1086	
12-4-78	1350	1342	1345	1336	
12-5-78	1788	1783	1786	1778	
12-7-78	1804	1793	1792	1772	10 am; started injection, 1 hour frequency.
12-8-78	1807	1794	1791	1791	
12-9-78	1806	1778	1771	1797	
12-10-78	1808	1756	1751	1799	
12-11-78	1809	1742	1736	1801	
12-12-78	1789			1781	
12-13-78	1808	Note: T-2 and T-3 continued		1798	Changed to 15 min. injection frequency.
12-14-78	1806	to decrease		1796	
12-15-78	1811	until the end		1803	
12-16-78	1802	of the exposure,		1795	
12-17-78	1808	due to plug-in connectors		1799	Middle heater bank failed.
12-18-78	1789	becoming too		not read	
12-19-78	1814	hot.		1807	
12-20-78	1814			not read	
12-21-78	1816			1809	
12-22-78	1816			1808	
12-23-78	1811	-	-	1804	
12-24-78	1808			1802	
12-25-78	1809	-	-	not read	
12-26-78	1806			1800	
12-27-78	1809	-	-	1803	
12-28-78	1806			not read	
12-29-78	1809	-	-	not read	
12-30-78	1805			1800	
12-31-78	1810	-	-	1806	
1-1-79	1806			not read	
1-2-79	1805	-	-	1801	
1-3-79	1807			not read	
1-4-79	1809	-	-	not read	4 pm
1-5-79	1497			1483	7 am; power outage last night. Now on. Purged with argon. Lost upper heater bank.

1-6-79	1729	-	-	1776	1 pm; restarted injection.
1-7-79	1810	-	-	1829	
1-8-79	1802	-	-	1839	
1-9-79	1793			1835	Lost part of lower heaters.
1-10-79	1774	-	-	1826	
1-11-79	1778			1835	
1-12-79	1771	-	-	1830	
1-13-79	1761			1820	
1-14-79	1763	-	-	1821	
1-15-79	1761			1821	
1-16-79	1754			1814	
1-17-79	1754	-	-	1813	
1-18-79	1757			1816	
1-19-79	1759	-	-	1818	
1-20-79	1761	1417	1452	1821	8 am; purged with argon and turned power off.

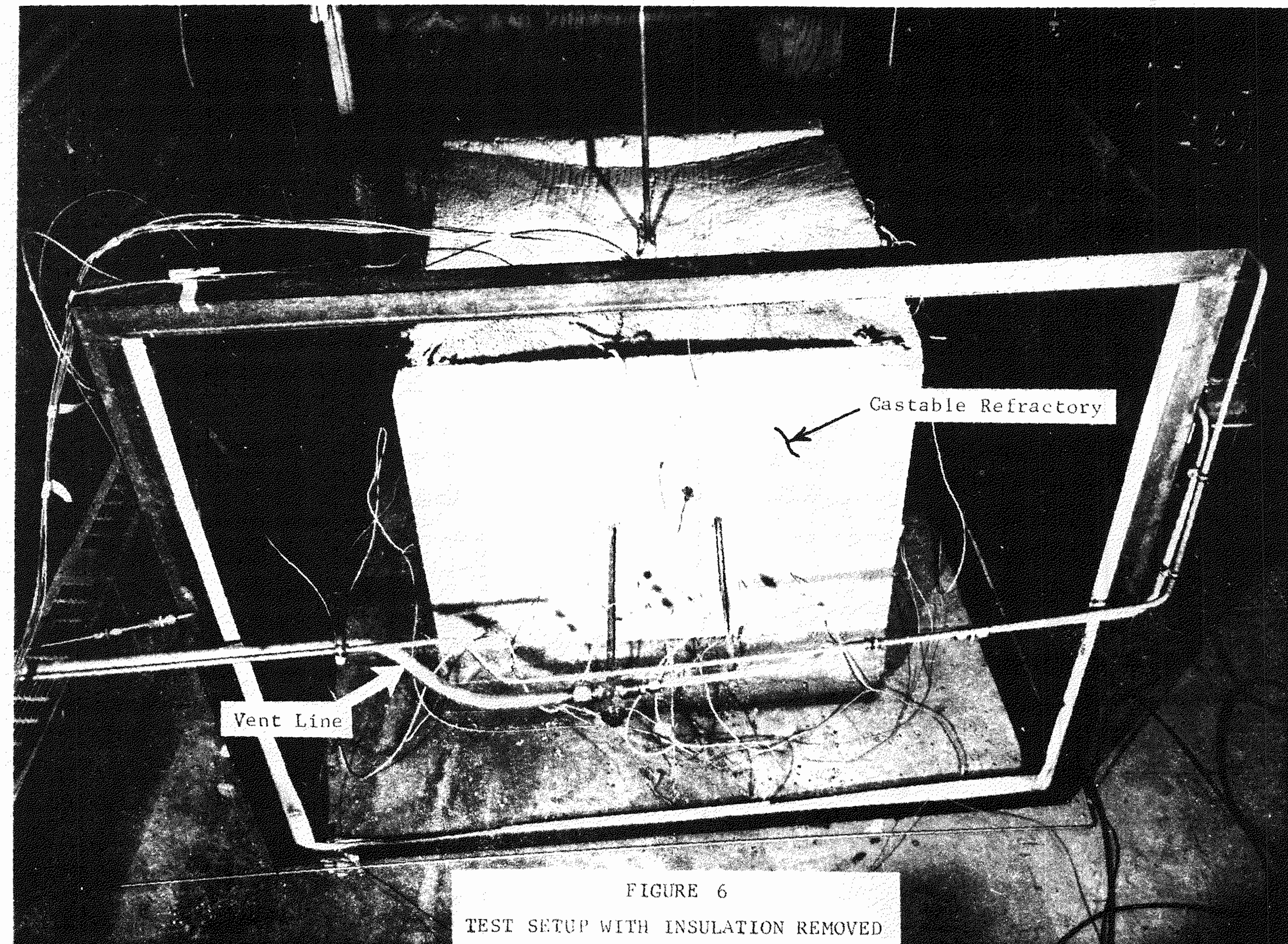


FIGURE 6

TEST SETUP WITH INSULATION REMOVED

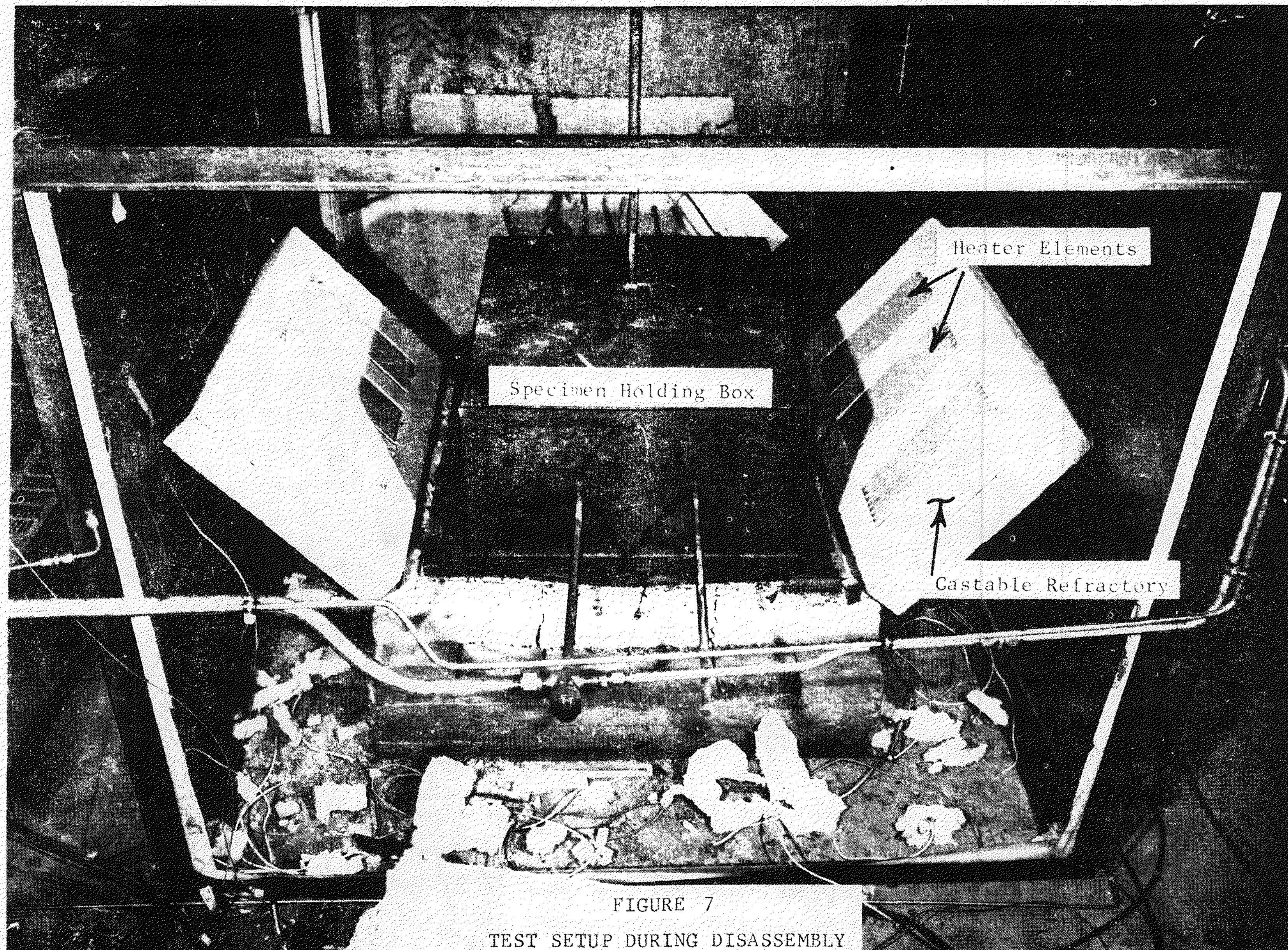


FIGURE 7

TEST SETUP DURING DISASSEMBLY

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