MASTER

CORROSION TESTS IN MOLTEN LEAD-LEAD CHLORIDE

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Chemical Development Branch

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A B S T R A C T

Corrosion tests were run on some commercial grade metals, an alloy steel, stainless steels, chromium-nickel-iron alloys, nickel base alloys, cobalt base alloys, and a chromium-nickel-cobalt-iron alloy in the system: lead-lead chloride-lead chloride vapor at 528°C under an argon atmosphere. The following metals and alloys showed a corrosion rate of nine mils per month or less and did not suffer intergranular or other localized attack: tantalum, Incoloy 804, Hastelloy F, Carpenter-20 (Cb), stainless steels 316L, 318 Cb, Haynes Alloy 25, and Haynes Multimet.

CORROSION TESTS IN MOLTEN LEAD-LEAD CHLORIDE

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CORROSION TESTS IN MOLTEN LEAD-LEAD CHLORIDE

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I. SUMMARY

Specimens of molybdenum, niobium, tantalum, the alloy steel Croloy 2 1/4, the stainless steels 347, 309 Cb, 316L, 304, 304L, 318 Cb, the chromium-nickel-iron alloys Incoloy 804, Carpenter-20 (Cb), Hastelloy F, the nickel base alloys Inconel, Inconel X, Hastelloy C, Hastelloy B, INOR-8, Monel, the cobalt base alloys Haynes Alloy 25, Haynes Alloy 36, Haynes Stellite Alloy 21, and the chromium-nickel-cobalt-iron alloy Haynes Multimet were exposed to mixtures of lead-lead chloride at 528°C. The test periods were 5, 10, and 18 days and the tests were conducted under an argon atmosphere.

Corrosion rates calculated from weight losses of the specimens ranged from 2 to 167 mils per month. Some specimens suffered pitting and some were attacked at the interface. A metallurgical examination of Hastelloy F (welded), Incoloy 804, Inconel X and Carpenter-20 Cb specimens was made. Some intergranular corrosion was found to be present in the case of the Inconel X specimens.

The results obtained indicate that tantalum, Incoloy 804, Hastelloy F, Carpenter-20 Cb, SS 316L, and 318 Cb, Haynes Alloy 25, and Haynes Multimet would be recommended for further evaluation in the lead-lead chloride process media.

II. INTRODUCTION

The dissolution of zirconium-based fuel elements by molten lead chloride in the Arco (Alloy Reguline Chloride Oxidation)⁽¹⁾⁽²⁾ process has been proposed at the Idaho Chemical Processing Plant. Zirconium or zirconium-uranium alloys react rapidly with molten lead chloride to produce volatile zirconium tetrachloride and a residue of molten lead and of uranium chloride dissolved in excess lead chloride. This process presents the problem of finding container materials that can resist corrosion by the lead-lead chloride system above the melting point of lead chloride (500°C). Since no information concerning the evaluation of metals in the lead-lead chloride system was found in the literature, scoping experiments were initiated with some metals and alloys which were potentially resistant to lead-lead chloride at 528°C.

III. MATERIALS AND EXPERIMENTAL PROCEDURES

A. Materials

The following metals and alloys were evaluated: the commercial grades of the metals molybdenum, niobium, tantalum, the alloy steel Croloy 2 1/4, the stainless steels type 347, 309 Cb stabilized, 316L (low carbon), 304, 304L, 318 Cb stabilized, the chromium-nickel-iron alloys Incoloy 804, Carpenter-20 Cb stabilized, Hastelloy F, the nickel base alloys Inconel, Inconel X, Hastelloy C, Hastelloy B, INOR-8, Monel, the cobalt base alloys Haynes Alloy 25, Haynes Alloy 36 and Haynes Stellite Alloy 21, and the chromium-nickel-cobalt-iron alloy Haynes Multimet. The nominal chemical compositions of these alloys are given in Tables 8, 9, 10, 11, 12, and 13 in the Appendix.

Six specimens 1 1/2" x 1" x (thickness of metal stock) of each alloy were tested. The specimens were milled smooth on all sides.

B. Procedures

Duplicate specimens were cleaned and weighed and immersed in Berzelius beakers containing approximately equal volumes of lead and lead chloride. The specimens were so arranged to provide exposure to the lead, lead chloride and lead chloride vapor phases. A watchglass was used as a cover for the individual beakers. Six specimens of each alloy, duplicates for 3 exposure periods, were used. A set of 15 beakers was used per test: these were removed in sets of five after 5, 10, and 18 days exposure. The beakers were kept at $528^{\circ}\text{C} \pm 10^{\circ}$ in a furnace under an argon atmosphere. The vapor pressure of lead chloride is about 0.2 mm mercury at 528°C .

After withdrawal, the specimens were immersed in distilled water and boiled until all scale from the lead and lead chloride was removed. After de-scaling, the specimens were rinsed with acetone prior to weighing. Corrosion penetration was calculated from the weight loss of the specimens. Some specimens were also subjected to metallurgical examinations.

IV. RESULTS

The corrosion rates are given in Tables 1, 2, 3, 4, 5, 6, and 7. In Table 1 which lists rates for unalloyed metals one sees that molybdenum suffered a severe localized attack in the form of a knifeline at the lead-lead chloride interface. See Figure 1. Therefore, molybdenum is unsuitable for Arco dissolver container material although its overall rate of corrosion is less than 9 mils per month. (At the present time, nine mils per month is considered a satisfactory corrosion rate for the Arco process). The rate for niobium is too high, being about 14 mils per month, and the rate for tantalum is about 7 mils per month. In Table 2 the corrosion rate for the alloy steel Croloy 2 1/4 is given at about 40 •

Table 1

CORROSION OF UNALLOYED METALS IN LEAD-LEAD CHLORIDE AT 528°C

<u>Specimen</u>	<u>Exposure Time Hours</u>	<u>Corrosion Rate mils/mo</u>	<u>Remarks</u>
Molybdenum	432	3.75 ± 2.65	Severe knifeline attack at lead-lead chloride interface
	240	4.7 ± 0.1	Severe knifeline attack at lead-lead chloride interface
	120	5.4 ± 0.3	Severe knifeline attack at lead-lead chloride interface
Niobium	432	13.9 ± 0.6	
	240	16.3 ± 0.1	
	120	7.8 ± 0.1	
Tantalum	432	7.6 ± 0.0	
	240	4.8 ± 2.4	
	120	6.2 ± 0.2	

$\frac{\text{PbCl}_2}{\text{Pb}}$

Fig. 1 - Interfacial Attack on Molybdenum in Pb-PbCl₂ at 528°C (2X)

mils per month based on a 432-hour exposure. This alloy is therefore unsuitable for use in the Arco process. There is no apparent attack between 240 and 432 hours since the rate for the 432-hour exposure is about half that for the 240-hour exposure.

Table 2

CORROSION OF ALLOY STEEL IN LEAD-LEAD CHLORIDE AT 528°C

<u>Specimen</u>	<u>Exposure Time Hours</u>	<u>Corrosion Rate mils/mo</u>
Croloy 2 1/4 Pipe Sections (Welded and Unwelded)	432	38.3 $\pm$ 1.3
	240	77.8 $\pm$ 3.6
	120	70.6 $\pm$ 3.0

The data for the corrosion of stainless steels given in Table 3 show that SS 347 and 304L suffered intergranular attack and are therefore not suitable as Arco container materials. The stainless steel type 347 seems to suffer no weight loss after 120 hours of exposure. The stainless steels 316L and 318 Cb have corrosion rates less than 9 mils per month. It is interesting to note that the long-term corrosion rate for 304 is about twice that for 304L and the initial rate is about 5 times that for 304L. Since the 304L suffers much intergranular attack, the mechanism for this attack in this case does not involve the presence of carbon at grain boundaries. The corrosion rate for SS 309 Cb was still increasing rapidly after 432 hours. Of this group only SS 316L and 318 Cb should be considered for further study.

Table 3

CORROSION OF STAINLESS STEELS IN LEAD-LEAD CHLORIDE AT 528°C

<u>Specimen</u>	<u>Exposure Time Hours</u>	<u>Corrosion Rate mils/mo</u>	<u>Remarks</u>
347	432	13.3 ± 0.7	Some intergranular attack
	240	31.8 ± 3.0	Some intergranular attack
	120	61.2 ± 1.8	Some intergranular attack
309 Cb stabilized	432	61.9 ± 14.3	Attacked in lead chloride and in vapor phase
	240	11.5 ± 0.8	Attacked in lead chloride and in vapor phase
	120	12.0 ± 3.5	Attacked in lead chloride and in vapor phase
316L (low carbon)	432	5.2 ± 0.2	Attacked in lead chloride and in vapor phase
	240	4.5 ± 0.1	
	120	5.7 ± 0.3	Attacked in lead chloride and in vapor phase
304	432	45.5 ± 0.5	
	240	82.0 ± 0.4	
	120	165.8 ± 1.5	
304L (low carbon)	432	16.9 ± 3.9	Severe intergranular corrosion in vapor phase
	240	27.5 ± 3.3	Intergranular attack in lead chloride and in vapor phase
	120	31.5 ± 5.1	Severe intergranular corrosion in lead chloride and in vapor phase

Table 3 (Cont'd)

<u>Specimen</u>	<u>Exposure Time Hours</u>	<u>Corrosion Rate mils/mo</u>	<u>Remarks</u>
318 Cb stabilized	432	7.7 ± 2.0	Slight corrosion in lead chloride phase on one sample. Considerable corrosion in lead chloride phase on other sample
	240	8.5 ± 0.3	Corrosion in lead chloride and in vapor phase
	120	7.2 ± 1.8	One sample had considerable corrosion in vapor phase

In Table 4 which gives the corrosion data for chromium-nickel-iron alloys, one sees that all of them, Incoloy 804, Carpenter-20 Cb and Hastelloy F have corrosion rates less than 9 mils per month and suffer no localized attack. They can therefore be considered promising for the Arco process. Welded specimens gave about the same corrosion results as unwelded samples.

The corrosion data for the nickel base alloys are given in Table 5. It is seen that Inconel and Inconel X suffer localized attack at the interface. Welded Inconel X showed lower rates for the 120 and 240-hr exposure but a higher rate for the 432-hr exposure than did unwelded Inconel. Hastelloy B, INOR-8 and Monel all suffered intergranular attack. These five alloys are therefore unsuitable for Arco service. Hastelloy C does not suffer localized attack and the corrosion rate after 432-hr is about 9 mil/month average, and after 120-hr, 7.6 mil/month. However, the average corrosion rate after 240 hours exposure is 13.0 mils/mo so Hastelloy C is not recommended for further study. This high rate makes Hastelloy C unsuitable for runs from 120 to 240 hours.

Table 4

CORROSION OF CHROMIUM-NICKEL-IRON ALLOYS IN
LEAD-LEAD CHLORIDE AT 528°C

<u>Specimen</u>	<u>Exposure Time Hours</u>	<u>Corrosion Rate mil/mo</u>	<u>Remarks</u>
Incoloy 804	432	6.3 ± 3.0	Two runs made. Attack in vapor phase
	240	5.4 ± 3.6	Two runs made. Attack in lead chloride and in vapor phase
	120	9.1 ± 5.6	Two runs made. Attack in vapor phase
Incoloy 804 (Welded)	384	6.6 ± 0.7	Attack in vapor phase
	240	8.5 ± 1.4	Attack in vapor phase
	120	5.9 ± 0.6	Attack in vapor phase
Carpenter-20 Cb stabilized	432	7.4 ± 3.2	Two runs made. Attack in lead chloride and in vapor phase
	240	10.7 ± 5.2	Two runs made
	120	8.8 ± 3.6	Two runs made. Attack in lead chloride and in vapor phase
Carpenter-20 Cb stabilized (Welded)	432	5.9 ± 0.6	Attack in lead chloride and in vapor phase
	240	14.1 ± 2.0	Attack in lead chloride and in vapor phase
	120	6.1 ± 0.2	Attack in lead phase
Hastelloy F	432	4.5 ± 1.2	Two runs made. Corrosion in lead chloride and in vapor phase
	240	4.3 ± 2.4	Two runs made. Corrosion in lead chloride phase
	120	8.6 ± 1.5	Two runs made
Hastelloy F (Welded)	432	4.5 ± 0.7	Attack in lead chloride phase
	240	5.8 ± 0.7	
	120	8.8 ± 1.2	Attack in lead chloride phase

Table 5

CORROSION OF NICKEL BASE ALLOYS IN LEAD-LEAD CHLORIDE AT 528°C

<u>Specimen</u>	<u>Exposure Time Hours</u>	<u>Corrosion Rate mils/mo</u>	<u>Remarks</u>
Inconel	432	5.3 ± 0.4	Small amount of attack at vapor interface
	240	9.0 ± 0.0	Small amount of attack at vapor interface
	120	6.6 ± 1.9	Small amount of attack at vapor interface
Inconel X	432	3.6 ± 0.8	Two runs made. Small amount of etching and attack at both interfaces
	240	22.4 ± 8.4	Two runs made. Small amount of attack on one sample at vapor interface
	120	24.0 ± 19.9	Two runs made. Small amount of corrosion at vapor interface
Inconel X (Welded)	432	6.8 ± 0.2	
	240	9.7 ± 3.4	Attack at lead-lead chloride interface
	120	17.7 ± 4.3	Attack in lead chloride and in vapor phase
Hastelloy C	432	8.9 ± 1.7	Three runs made. Attack in vapor phase
	240	12.9 ± 3.8	Three runs made
	120	7.5 ± 1.5	Three runs made. Attack in lead chloride and in vapor phase
Hastelloy B	336	2.8 ± 0.2	Intergranular attack in lead phase
INOR-8	336	3.8 ± 0.3	Intergranular attack
	120	3.6 ± 0.0	

Table 5 (Cont'd)

<u>Specimen</u>	<u>Exposure Time Hours</u>	<u>Corrosion Rate mils/mo</u>	<u>Remarks</u>
Monel	432	14.0 ± 7.4	Two runs made. Intergranular attack in lead chloride and in vapor phase
	240	15.9 ± 3.6	Two runs made. Severe etching and intergranular attack
	120	11.1 ± 1.6	Two runs made. Attacked principally in vapor phase

Corrosion data for three cobalt base alloys are given in Table 6. The corrosion rates for the three alloys, Haynes Alloy 25, Haynes Alloy 36, and Haynes Alloy 21 were very low, ranging from 1.1 to 2.7. However, only Haynes Alloy 25 deserves further consideration since the other two suffered pitting and Haynes Stellite Alloy 21 suffered attack at both interfaces.

Table 6

CORROSION OF COBALT BASE ALLOYS IN LEAD-LEAD CHLORIDE AT 528°C

<u>Specimen</u>	<u>Exposure Time Hours</u>	<u>Corrosion Rate mils/mo</u>	<u>Remarks</u>
Haynes Alloy 25	336	2.2 ± 0.1	Attacked principally in vapor phase
Haynes Alloy 36	336	2.0 ± 0.7	Localized attack in lead chloride phase
Haynes Stellite Alloy 21	336	1.2 ± 0.2	Some pitting and attack at both interfaces

Data for Haynes Multimet, a chromium-nickel-cobalt-iron alloy, is given in Table 7. This alloy suffers a corrosion rate of 6 mils per month with no localized attack so it can be considered suitable for further studies.

Table 7

CORROSION OF CHROMIUM-NICKEL-COBALT-IRON ALLOY
IN LEAD-LEAD CHLORIDE AT 528°C

<u>Specimen</u>	<u>Exposure Time Hours</u>	<u>Corrosion Rate mils/mo</u>	<u>Remarks</u>
Haynes Multimet	336	5.5 ± 0.2	Attacked principally in liquid lead chloride phase
	120	4.6 ± 0.0	

A metallurgical examination of Hastelloy F (welded), Incoloy 804, Inconel X and Carpenter-20 Cb specimens was made. These are alloys which suffered low corrosion rates. In all cases except Inconel X there was no grain boundary attack. In the case of Inconel X there were some spots where grain boundaries had been attacked at the edge of the weld. The depth of this condition was about ten mils in the instances observed. See Figure 2.

Figure 2. Intergranular Attack on Inconel X in Pb-PbCl₂ at 528°C. (60X)

V. REFERENCES

- (1) Hahn, H. T., and E. M. Vander Wall, Salt-Phase Chlorination of Reactor Fuels, I. Dissolution of Zirconium Alloys in Lead Chloride, IDO-14478 (1959)
- (2) Vander Wall, E. M., H. T. Hahn, and D. L. Bauer, Salt-Phase Chlorination of Reactor Fuels, II. Arco Process Definition and Scoping Studies, IDO-14525 (1960)

A P P E N D I X

Table 8

ALLOY STEEL CHEMICAL COMPOSITION, PERCENT

<u>Steel</u>	<u>C</u>	<u>Mn</u>	<u>Cr</u>	<u>Mo</u>	<u>Fe</u>
Croloy 2 1/4	0.15	0.3-0.6	2.0-2.5	0.9-1.1	95.7-96.7

Table 9

STAINLESS STEELS CHEMICAL COMPOSITION, PERCENT

<u>Type</u>	<u>C</u> <u>Max</u>	<u>Mn</u> <u>Max</u>	<u>P</u> <u>Max</u>	<u>S</u> <u>Max</u>	<u>Si</u> <u>Max</u>	<u>Cr</u>	<u>Ni</u>	<u>Fe</u>	<u>Other</u>
347	0.08	2.00	0.045	0.03	1.00	17.00-19.00	9.00-13.00	64.0-70.0	<u>Cb</u> 10 x C Min
309 Cb	0.08	2.00	0.045	0.03	1.00	22.00-24.00	12.00-15.00	57.0-62.0	<u>Cb</u> 10 x C Min
316L	0.03	2.00	0.045	0.03	1.00	16.00-18.00	10.00-14.00	61.9-68.9	<u>Mo</u> 2.00-3.00
304	0.08	2.00	0.045	0.03	1.00	18.00-20.00	8.00-12.00	64.8-70.8	
304L	0.03	2.00	0.045	0.03	1.00	18.00-20.00	8.00-12.00	64.9-70.9	
318 Cb	0.08	2.50	0.045	0.03	1.00	17.00-19.00	13.00-15.00	58.5-63.5	<u>Mo</u> 2.00-3.00

Table 10

CHROMIUM-NICKEL-IRON ALLOYS CHEMICAL COMPOSITION, PERCENT

Alloy	C	Mn	Si	S	Fe	Ni	Cr	Cu	P	Mo	Other
Incoloy 804	0.07	0.81	0.66	0.007	25.36	41.54	30.53	0.45		None	Al 0.23 Ti 0.32
Carpenter-20 (Cb)	0.06	0.80	0.56	0.003	44.01	28.28	19.93	3.31	0.007	2.45	Cb 0.59
Hastelloy F	0.08 Max	1.0- 2.0	1.0 Max	0.03 Max	12.7- 21.5	44.0- 47.0	21.00- 23.00	0.15 Max	0.04 Max	5.5- 7.5	Cb + Ta 1.75-2.5 Ta 0.50 Min W 1.0 Max Co 2.50 Max

Table 11

NICKEL BASE ALLOYS CHEMICAL COMPOSITION, PERCENT

Alloy	Ni	Cu	Fe	Cr	Si	Mn	C	Mo	Other
Inconel	77.	0.2	7.	15.	0.25	0.25	0.06	None	S 0.007
Inconel X	73.	0.05	7.	15.	0.4	0.5	0.04	None	Cb 1. Al 0.9 Ti 2.5 S 0.007
Hastelloy C	52.1-60.1	None	4.5-7.0	15.5-17.5			0.15 Max	16.0-18.0	W 3.75-5.25
Hastelloy B	62.9-69.9	None	4.0-7.0	None			0.12 Max	26.0-30.0	
INOR-8	65 - 71	0.50 Max	5. Max	6 - 8	0.55 Max	0.8 Max	0.04-0.08	15 - 19	W 0.50 Max Co 0.2 Max Ti/Al 0.50 S 0.01 P 0.01 B 0.01
Monel	65.57	31.77	1.52	None	0.15	0.90	0.06	None	S 0.005

Table 12

COBALT BASE ALLOYS CHEMICAL COMPOSITION, PERCENT

<u>Alloy</u>	<u>C</u>	<u>Si</u>	<u>Mn</u>	<u>W</u>	<u>Cr</u>	<u>Ni</u>	<u>Fe</u>	<u>Co</u>	<u>Other</u>
Haynes Alloy 25	0.15 Max	1.0 Max	1.0- 2.0	14.0- 16.0	19.0- 21.0	9.0- 11.0	2.0 Max	46.9- 53.9	
Haynes Alloy 36	0.35- 0.45	0.35- 0.65	1.0- 1.5	14.0- 15.0	17.5- 19.5	9.0- 11.0	2.0 Max	49.9- 55.8	B 0.01- 0.05
Haynes Stellite Alloy 21	0.20- 0.35			None	25.0- 30.0	1.5- 3.50	2.0 Max	57.7- 66.8	Mo 4.5- 6.5

Table 13

CHROMIUM-NICKEL-COBALT-IRON ALLOY

<u>Alloy</u>	<u>C</u>	<u>Cr</u>	<u>Ni</u>	<u>Mo</u>	<u>Fe</u>	<u>Cb + Ta</u>	<u>N₂</u>	<u>W</u>	<u>Co</u>
Haynes Multimet	0.08- 0.16	20.0- 22.5	19.0- 21.0	2.5- 3.5	27.4- 37.1	0.75- 1.25	0.10- 0.20	2.0- 3.0	18.5- 21.0

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