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NYO-1330

THE CORROSION RESISTANCE OF SEVERAL ALLOYS IN AN HF-H₂O
ATMOSPHERE AT HIGH TEMPERATURES

C. F. Ritchie and E. K. Teter

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Mallinckrodt Chemical Works
St. Louis, Missouri

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Author: C. F. Ritchie and E. K. Teter

A B S T R A C T

Numerous alloys have been tested for corrosion resistance when exposed to an HF-H₂O atmosphere at 1070°F.

The tests indicate that the Ni-Cr alloys, as a group, give the best corrosion resistance where strength at high temperatures is desired. Other alloys have given excellent results; however lack of strength at the higher temperatures, or cost, are against their use in production equipment.

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INTRODUCTION

This report covers macroscopic observations of various alloys which have been exposed to an HF-H₂O atmosphere at 1070°F. This report covers all the alloys not previously reported, a few alloys previously "rejected" then brought back for continued testing and the alloys still being tested as of the last report (NYO 1309).

PURPOSE

The purpose of the testing program is to find metals or alloys which could be used as a material of construction for UF₆ process equipment.

REFERENCE

Previous reports on the corrosion resistance of various alloys, tested in an HF-H₂O atmosphere at 1070°F, may be had by referring to MCW reports Nos. 150, 161, 178, 203, 249 and NYO Report No. 1309.

SUMMARY

The alloys tested and included in this report have been placed in groups, each group having similar principal constituents. These groups, and their respective alloys, are as follows:

GROUP A - The Cobalt Alloys

1. L-605
2. Multiment 95
3. N-155
4. S-590

GROUP B - The Nickel-Chromium Alloys

1. Inconel (Fluoride coated)
2. Electro-alloy H3
3. Illium G (cast)
4. Illium R (rolled)
5. N-238
6. Hastelloy C.
7. D-10 (cast)
8. Chromel P
9. Hundred Metal
10. Nichrome V
11. Hastelloy C - stabilized

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GROUP C - The Chromium-Nickel Alloys (Stainless Steels)

1. 310 S.S.
2. 16-25-6

GROUP D - The Copper-Nickel Alloys

1. Ambralloy 917
2. Monel (Fluoride coated)
3. Super Nickel (702)
4. 326 Monel
5. Advance Metal
6. Copel

GROUP E - Miscellaneous Alloys

1. Z-Nickel
2. Stellite #6
3. Maleable Cast Iron

GROUP F - The Precious Metal Alloys

1. 95 Pt - 5 Ir
2. 95 Pd - 5 Au on a Ni (2% Mn) base
3. 95.5 Pd - 4.5 Au
4. 60 Pd - 35 Ag - 5 Au
5. 60 Pd - 40 Cu
6. 80 Au - 20 Pd

For construction metals that must be able to withstand stress and high heat the Ni-Cr alloys are still the most promising. Illium R (and Illium G) are in use at the present time and have given very good service. However, the main objection to Illium is its lack of availability and its cost. Electro-alloy H-3, Hundred Metal, Chromel-P and Hastelloy C have given very good test results. Hastelloy C now seems to be a very promising alloy. It has high strength at elevated temperatures (see Appendix #1) as reported by Battelle Memorial Institute, and in the equipment tested has not contaminated the product any more or any less than Illium R. It has, however, shown to be brittle when cool (after "age-hardening") and this may be against its use for equipment that is subjected to sudden impact or shock.

Of the low creep strength alloys, 326 Monel and Copel (Advance Metal) could be used very well where structural strength is not needed, i.e. for rotary valves, valve seats or as a liner material.

Many precious alloys have given excellent results under the test conditions. The 95, Pt-5 Ir is by far the best of all alloys tested. However, the cost of these alloys is an important factor; they could not be used where structural strength is required.

Stellite #6 has given good results and could be used where wear plate or liner material is needed.

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METHOD OF TESTING

The method of testing was the same for all the alloys. Each alloy was stamped with some identifying symbol and placed in a small test box containing UO_2 . The test samples were then placed in the Plant 4 green salt reaction trays and subjected to a gradually increasing HF concentration (from 60% to 100% HF, water being the other constituent) at 1070°F for a 20 hour reaction period. The trays were then air cooled to room temperature and the test boxes removed. This procedure was repeated on an average of three times a week.

All "hours tested", as referred to in this report, are hours actually under HF at 1070°F .

At various times the samples were examined for hardness of scale by scratching with a knife, and for scale tenacity and embrittlement by bending the sample 90° and back.

In the instance of metals like Monel, Copel, Nickel etc., cracking during the bend test represented "intergranular" corrosion,---thought to be due to intergranular sulfide (and sometimes oxides) formation. Later, however, it was found that certain alloys exhibited a cold brittleness, due to "age hardening" because of being held at 1070°F for long periods. To prove that this was a metallurgical problem, rather than a corrosion problem, we instructed Battelle Memorial Institute to run "high-temperature" elongation and tensile tests on several alloys known to be relatively corrosion resistant, but subject to age hardening. Please see Appendix 1 for such results.

The above batch testing procedure ends with the writing of this report because of shutting down Plant 4. New continuous testing is being designed for use at Plant 7.

TESTING RESULTS

Please refer to the table at the end of this report for data on the approximate compositions of the alloys tested, and the manufacturer or supplier. The hours recorded after each alloy are hours actually under HF at 1070°F .

Group A - The Cobalt Alloys

1. L605, 4500 hours. No corrosion at previous bends. A medium hard, black scale which flakes slightly when bent. The sample broke with a 90° bend. The sample is still being tested. Worth a cautious plant trial; alloy is commercially available.
2. Multimet A-95, 4500 hours. Has a thick hard, black scale. Sample is badly pocked, indicating flaking in use. Scale adherent on bending. The sample is still being tested.
3. N-155, 4140 hours. Has a thick, medium hard scale. Sample is heavily pocked (see above). Scale flaked mildly when bent. The sample is still being tested.

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4. S-590, 4520 hours. Sample has a medium thick, black, fairly soft scale. Scale was adherent though samples broke on bending. The sample is still being tested. Temporarily "shelved", because of its Cb (niobium) and Ta contents.

Group B - The Ni-Cr Alloys.

1. Inconel (fluorine treated), 620 hours. This sample has a fluoride coating, applied with elemental fluorine. Treated with a 15 mol % F₂ for 3 1/2 hours at 350°F. The sample now has a thin, black, hard scale which flakes slightly when bent. The fluoride coating, at this writing, has shown no beneficial affect on the sample. The sample is still being tested. Inconel, per se, is a good corrosion-resistant alloy, though slightly weak at the higher temperatures.
 2. Illium R (fluorine treated)*, 620 hours. This sample was treated with fluorine, as above. The sample has a thin, black, hard scale which sheds slightly when bent. The sample is no better than untreated Illium R (which see, below) up to the presented time. The sample is still being tested.
 3. Electro-Alloy H-3, 3300 hours. This sample is very good. The scale is black, very hard and thin. The identification markings are still very clear. The sample was removed for exhibit and study, and a new H-3 sample tested for 1100 hours. The new sample has a medium thick, medium hard, black scale. The sample is cast and is too thick to bend. The sample is still being tested. See Appendix #2 for the story on H-3 vs Hastelloy C.
 4. Illium G (cast), 4560 hours. There are three Illium G cast samples on test. All three have a thick, black, tenacious, medium hard scale. This alloy is still, corrosion-wise, one of the better alloys tested. The samples are still being tested. Its metallurgical behaviour (strength and stability) at temperatures above 1100°F may, however, need further study.
 5. Illium R, 4560 hours. This sample has a thin, medium hard scale which is adherent on bending. Practical experience in Plant 7 is apparently confirming our rather crude method of examining and evaluating metals,--- by visual observation of scale thickness and tenacity. Illium R has given very good plant service in large scale operations. The sample is still being tested. The alloy has good strength at 1100°F and seems to be fairly stable.
- * The Inconel, Illium-R, and Monel samples were treated with fluorine by the courtesy of Mr. W. B. Humes and Dr. R. B. Korsemeyer, Carbide and Carbon Chemicals Co., Oak Ridge, Tennessee.

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6. N-238 (Hastelloy E), 4500 hours. Sample has a black, hard, thick, lightly surface-pocked scale. When scraped both metal and scale are removed for a considerable depth. The scale flakes slightly when bent. The sample is still being tested (also a Cb-Ta bearer).
7. D-10, 4300 hours. This sample is similar to Illium G. The scale is very hard, black and thin. A previous bend became badly corroded and finally broke. The sample was cast. The metal is much softer than the scale. The sample is still being tested.
8. Chromel-P, 6780 hours. This sample has given very good results. The greyish-black scale is soft and thin. The scale flakes slightly when bent. No embrittlement noticed. The sample is still being tested. (No strength data are available for this alloy; we have requested the International Nickel Co. to consider a study of the properties of this "electrical-resistance" alloy.)
9. Hundred Metal, 1300 hours. This alloy has a very hard, thick, black scale. The scale is harder than the metal. The sample is cast so was not subjected to a bend test. The alloy shows great promise and is still being tested. (Note similarities in composition to Hastelloy C, D-10, Illium G and H-3).
10. Nichrome V, 6320 hours. The sample has an enamel-like scale which is rather thick. On bending considerable scale flakes off. No evidence of embrittlement. Alloy has few pock marks. The sample was removed from service.
11. Hastelloy C, 2400 hours. This sample was previously reported "rejected" but was brought back into testing service for comparison with stabilized Hastelloy C. (See detailed story in Appendix #2.) The sample has a thin, hard, black scale which flakes slightly when bent. The sample broke with one 90° bend, (age hardening). The sample has two noticeable scale layers; a top layer as above, then a brown-tan layer which is fairly soft. The sample is still being tested.
12. Hastelloy C (stabilized), 1100 hours. The alloy was fully annealed then stabilized at 2025°F $\pm$ 25°F. Two such samples are being tested, one has been sand-blasted and the other polished. There is little difference between the two surfaces, other than surface smoothness. Both have a hard, black, thin scale. The polished sample broke with a 30° bend, apparently due to "age hardening" shortness at room temperature. Both have two scale layers, the polished sample having a slightly thinner intermediate layer than the sand-blasted sample. Other than the cold brittleness, mentioned above, these samples have shown promising results. The samples are still being tested. See Appendix #1 for further data on strength tests.

A new cast sample of Hastelloy C has the above scale characteristics except that it is too heavy to subject to a bend test and the scale is harder. This sample has been tested for 1100 hours and is still being tested.

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Group C - The Cr-Ni Alloys.

1. 310 Stainless Steel, 60 hours. The alloy was tested only to reaffirm our previous tests on stainless steel. The scale formed was very heavy and soft and flaked easily. The sample was rejected.
2. 16-25-6, 3520 hours. This sample up to 2420 hours gave excellent results - no corrosion of any kind was observed. However, at this time the sample was washed with an ammonium oxalate solution to remove the UF_6 (this is done occasionally with all the samples, without harm, to reveal hidden pits or cracks). As 16-25-6 is essentially a stainless steel, with an abnormally high Mo content, its final behaviour was "as expected". Why it stood up so well prior to the ammonium oxalate wash, we are at a loss to explain. The answer might lay in some peculiarity of "surface resistance" which was disrupted because of the aforesaid wash. This piece (2" x 1" x 1/8") was "milled" from a thicker piece of a forging (or possibly a casting) in order to prepare a "manageable" specimen. Possibly the milling action affected a "skin condition", which was later destroyed. A good subject for metallurgical research.

Group D - The Copper-Nickel Alloys.

1. Ambralloy 917, 6980 hours. This sample has a very thick, soft scale which flakes badly when bent. There was considerable corrosion under the scale. The sample was removed from service. See previous reports, and note that while the sample was "rejected" after 7000 hours, its resistance was "reasonable".
2. Super Nickel #702, 5900 hours. Scale is very soft and flakes easily when bent. A previous bend is showing corrosion. No embrittlement observed. The alloy is still being tested.
3. 326 Monel (non-magnetic Monel), 4660 hours. This sample is still clean looking. The grey scale is thin and soft. The scale flakes slightly when bent. This alloy would give very good service where strength is not needed. The sample is still being tested.
4. Copel (Advance Metal), 6740 hours. This alloy is very similar to 326 Monel. The scale is soft and thin and flakes slightly when bent. No embrittlement observed. The alloy is still being tested.
5. Copel (International Nickel), 5760 hours. This sample is not as good as 326 Monel or Advance Metal. Some corrosion has occurred at previous bends. The scale is soft, but thicker than 326 Monel. The scale flakes slightly when bent. No embrittlement noticed. The alloy is still being tested.
6. Monel (fluoride treated), 620 hours. This sample was treated with fluorine as explained above. The sample is still relatively "clean" looking. The scale is light grey, thin and very soft. The scale flakes readily when bent. The sample shows no benefit, up to this writing, on having a fluoride coat. The sample is still being tested.

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Group E - Miscellaneous Alloys.

1. Z - Nickel, 2100 hours. This alloy has a black, thin, soft scale which flakes mildly when bent. Some intergranular corrosion noticed at previous bends. The sample broke with a 90° bend. The sample is still being tested.
2. Stellite #6, 1100 hours. The alloy has a fairly thin, very hard, black scale. The alloy is purposely a very brittle one, so a bend test cannot be made. There is no noticeable corrosion. The sample is still being tested.
3. Malleable Cast Iron, 240 hours. This sample was tested because of its high temperature strength and "newness" to the alloy field. The sample scaled very badly and was removed from service.

Group F - The Precious Metal Alloys.

1. 95 Pt-5 Ir., 7100 hours. This sample is very good. There is a very thin, adherent, shiny, soft scale on the alloy. No corrosion of any kind observed. This alloy withstands the test conditions best of any of the alloys tested. The sample is soft and did warp slightly. The alloy was removed from service for it was felt additional testing would give no more information.
2. 95 Pd-5 Au, on a Ni (2% Mn) base, 7680 hours. This sample has changed very little since last reported. The alloy shows very little corrosion. The scale is thin, dull and soft. The welded portion of the sample has held up very well. The backing shows considerable corrosion. The sample is still being tested.
3. 95.5 Pd - 4.5 Au, 6580 hours. This sample was cracking where welded. The scale was soft, thin and tenacious. Some evidence of thinning at the weld area. The sample was removed from service.
4. 60 Pd - 35 Ag - 5 Au, 7440 hours. Sample has a thin, dull, soft, tenacious scale. No corrosion or embrittlement observed. The sample is still being tested.
5. 60 Pd - 40 Cu, 5900 hours. This sample has a dull, thin, soft, adherent film. The sample was gradually splitting along the length from both ends. No embrittlement observed, however, upon bending. The sample was removed from service.
6. 80 Au - 20 Pd, (Palau), 6100 hours. This small sample was lost, but on the last examination it had a soft, dull tenacious film. No corrosion or embrittlement observed.

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SUPPLIER AND COMPOSITION OF THE TESTED ALLOYS

Group	Alloy	Supplier	Ni	Cr	Fe	Cu	Al	Mo	W	Cb	Mn	Si	Co	Mg	Ti	C	Ta	Comments
A	L605	Haynes Stellite	10.0	20.0	9.0				15.0				46.0					
A	Multimet 95 (N155)	" "	20.0	20.0	34.0				2.0	1.0	3.0		20.0					
A	N155	Allegheny-Ludlum	20.0	20.0	31.1			3.0		1.0	1.5	0.7	20.0		2.2			
A	S590	" "	19.4	20.8	26.5			4.3	3.9	3.9	1.7	0.7	19.3				0.4	
B	Inconel (F. coated)	International Nickel*	77.0	15.0	7.0	0.2					0.3	0.3				0.2		
B	Electro-Alloy H-3	American Brake Shoe Co.	59.0	14.0	6.0			16.0	4.0									
B	Illium G (Cast)	Illium Corporation	62.0	21.0	6.0	4.0		6.0										
B	Illium R	" "	62.0	21.0	8.0	3.0		5.0										Also an Illium R Fluoride coated sample.
B	N238	Haynes Stellite	45.0	22.0	22.0			6.0	2.0	2.0								
B	D-10 (Cast)	Duriron Co.	66.6	23.3	2.7	3.0		2.4	0.2		0.8	0.8						
B	Chromel-P	Hoskins	90.0	10.0														
B	Hundred Metal	Midwest Alloy	58.0	22.0	--			15.0	4.0			1.0						
B	Nichrome V	Driver-Harris	80.0	20.0														Rejected
B	Hastelloy C	Haynes Stellite	57.0	16.0	6.0			17.0	4.0									
B	Hastelloy C-Stabilized	" "	57.0	16.0	6.0			17.0	4.0									
C	16-25-6	Timken	25.0	16.2	52.0			5.9			1.7							Rejected
C	310 S.S.	?	20.0	25.0	54.5													Rejected
D	Ambralloy 917	American Brass Co.	5.0		2.5	82.0	9.5				1.0							Rejected
D	Monel (F. coated)	International Nickel*	67.0		1.4	30.0					1.0	0.1						
D	Super Nickel (702)	American Brass Co.	30.0		0.4	69.6												
D	326 Monel	Steel Sales	40.0			60.0												
D	Copel (Advance)	Hoskins Mfg. Co.	45.0			55.0												
D	Copel	International Nickel	45.5		0.1	53.2					1.0							
E	Z-Nickel	International Nickel	93.7		0.4		4.4				0.3	0.5				0.2		
E	Stellite #6	Haynes Stellite	3.0	30.0	3.0			5.0					56.0			1.3		
E	Malleable Cast Iron	International Nickel	0.9		92.2						0.6	2.5		0.1		3.7		Rejected

* Fluoride coating was done through the courtesy of Mr. W. B. Humes and Dr. R. B. Korsemeyer, Carbide and Carbon Chemicals Co., Oak Ridge, Tenn.

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APPENDIX # 1

Attached to this appendix is data supplied by the Battelle Memorial Institute through the courtesy of Dr. H. Cross. The Illium R samples for testing were supplied, by Mallinckrodt, from stock; Hastelloy C samples were prepared and donated by the Haynes Stellite Co. Six test specimen of each group were cut by Battelle and then three specimen of each group were treated at Plant 4 under the test conditions listed earlier in this report for a total of 160 hours. All samples (HF treated and the non-HF treated) were heated to 1200°F at Battelle and tested for offset and tensile strengths. Rate of pull is given on the data sheet.

APPENDIX # 2

The original Hastelloy C (plate) sample tested under HF at 1070°F gave a very hard, black, smooth scale which was beginning to show signs of scale flaking off in a few places (edges especially). A 10° bend resulted in dislodging scale from about 1/2 of the sample. A cast Hastelloy C sample, tested at the same time; gave similar results,---though the sample was too thick to bend. Please refer to MCW report #150 and #203 for reference.

About the same time, H-3 was being tested. Being cast, little enthusiasm was developed about it until we were assured by American Brake Shoe Co. that their H-3 was nothing more than Hastelloy C in a cast form. H-3 had given excellent results,---a thin scale with no edge flaking, a good tenacious, hard scale. The American Brake Shoe Co. assured us that H-3 could be rolled, if Hastelloy C could be rolled.

Both Hastelloy C (cast and sheet) samples and the H-3 sample were thereupon sent to Haynes Stellite (through Mr. E. Duce) for an explanation of this anomalous behaviour. While H-3 was found, upon analysis, to contain less Fe, Haynes Stellite Metallurgists refused to ascribe corrosion differences to this constituent. They felt that the thermal histories of the two alloys would be the most significant reason for the difference in the corrosion resistance. They claimed that the Hastelloy C samples (both cast and sheet) had been fully annealed, whereas microscopic examination showed the H-3 casting to lack such a heat treatment.

Accordingly, Haynes Stellite forwarded for testing plate Hastelloy C in a "stabilized-annealed" condition, and also a piece of cast Hastelloy C which was ground smooth but not heat treated (supposedly to duplicate the H-3).

All these samples showed excellent resistance (see this report). They were like H-3, also like Illium R - our previous "practical" first choice for plant use (see report MCW #249).

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To complicate the picture even more, after testing and rejecting the early Hastelloy C samples, another sample of Hastelloy C plate was submitted. This sample was listed as being rolled at the new Haynes Stellite mill. This "new" sample was "fully annealed", like the early samples which had failed. This sample, of "new and improved" Hastelloy C plate, stood up as well as the stabilized annealed samples and as well as the old H-3 sample. We can get no firm data from Haynes Stellite as to any great improvement in composition or rolling of this "new" Hastelloy C, except as to claims of "quality control" now being practiced at their own mill.*

One factor that could enter into the behaviour differences between the "new" and "old" Hastelloy C is the purity of the AHF (anhydrous hydrofluoric acid) used in our plant process. Between the testing of the new and the old Hastelloy C, the AHF improved in purity; an excellent product is now being received from Penn Salt at Calvert City. The higher-purity AHF contains less SO_2 than the previous acid. Since sulfur is known to be hard on nickel alloys, this change might explain the corrosion difference. Except: that the H-3 and the "old" Hastelloy C pieces were both tested with the old, higher- SO_2 , hydrofluoric acid! The H-3 gave good results and the "old" Hastelloy C (plate and cast) gave very bad results. The better HF, with possibly some other factors, might be giving the better results; maybe the lower Fe content of the H-3 explains its resistance to the old, high- SO_2 acid.

This leaves a lot of unsolved problems. Practically, it now seems worthwhile to build a screw of Hastelloy C to give it a plant test. Being a commercial material its use should eliminate some of the headaches previously encountered with the procurement of Illium. Personnel at Haynes Stellite are giving us excellent cooperation, both in this matter of corrosion-vs-composition, and with respect to the high temperature properties of Hastelloy C. With respect to the latter, apparently their new "quality control" of Hastelloy C has resulted in improved physical properties of the alloy---and these "new" properties are sufficiently different to warrant a complete re-evaluation of them.

* Previously, Hastelloy C had been rolled for Haynes by outside mills. Apparently, when working with the alloy, Haynes found it advantageous to set closer control limits on several elements.

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TENSILE PROPERTIES AT 1200°F.

(Rate of pull: 0.02 inch per minute to the yield strength,
then 0.06 inch per minute to rupture.)

Material	Specimen Number	Area, Sq. In.	Yield Strength, psi		Tensile Strength, psi	Elongation, %
			0.1% Offset	0.2% Offset		
Hastelloy C, fully annealed	H-1*	0.1294	43,900	44,200	90,800	40.5
	H-2*	0.1294	52,800	53,500	92,400	39.0
	Average		48,400	48,900	91,600	39.7
Hastelloy C, fully annealed	H-4	0.1288	40,400	40,700	88,600	37.0
	H-5	0.1290	41,600	43,000	84,500	31.0
	Average		41,000	41,800	86,500	34.0
Hastelloy C, stabilized annealed	H-7*	0.1295	42,900	44,600	95,100	32.5
	H-8*	0.1305	44,400	46,200	92,500	21.0
	Average		43,600	45,400	93,800	26.7
Hastelloy C, stabilized annealed	H-10	0.1299	39,200	42,500	97,500	28.0
	H-11	0.1297	38,500	39,900	92,600	43.0
	Average		38,800	41,200	95,000	35.5
Illium R	I-1*	0.1279	39,300	40,500	66,900	11.5
	I-2*	0.1281	41,200	42,100	68,700	14.0
	Average		40,200	41,300	67,800	12.7
Illium R	I-4	0.1285	40,800	42,600	64,200	11.1
	I-5	0.1300	40,400	41,700	63,900	11.2
	Average		40,600	42,100	64,000	11.2

* Exposed two or three weeks at 1000-1100°F under operating conditions.

Data by Battelle Memorial Institute, December-1951.