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MATERIAL SELECTION FOR DEFENSE WASTE PROCESSING FACILITY

by

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ABSTRACT

Construction has started on a facility to immobilize high-level radioactive waste in borosilicate glass at the Department of Energy's Savannah River Plant. Type 304L stainless steel is generally sufficient for supply tankage and service lines. It is used as the reference material in chemical reprocessing of reactor target and fuel tubes. Type 304L, however, has unacceptable stress corrosion cracking resistance in solutions containing formic acid and chloride. Scouting tests were performed on twelve commercial nickel-based alloys in simulated process solutions containing halides, sulfates, nitrates, mercury and formic acid. Mercuric ions and halides interact in acidic environments to increase pitting and crevice attack. Alloys with combined chromium plus molybdenum contents greater than 30%, that also contain greater than 9% molybdenum, were most resistant to pitting and crevice corrosion. Based on this testing, Alloy C-276 has been selected as the reference process equipment material, with Inconel 690 and ALLCORR selected for specialty areas.

A DEFENSE WASTE PROCESSING FACILITY (DWPF) is being constructed at the Department of Energy's Savannah River Plant. This facility will immobilize the radioactive nuclides contained in the approximately 30 million gallons of high-level radioactive waste generated during the 30 years following startup (1)*. The Savannah River Plant (SRP) is the nation's primary source of tritium, weapons plutonium, and several other radionuclides for defense, space, medical, and energy applications. SRP was built by E.I. du Pont de Nemours & Company, Inc., for the U.S. Atomic Energy Commission in the early 1950's. The plant is still operated by the Du Pont Company for the U.S. Department of Energy (DOE). The plant comprises a large, remote land area with extensive support facilities.

The radionuclides produced at SRP are generated in nuclear reactors by irradiating the appropriate target materials with neutrons from fusion of uranium fuels and then chemically separating the products of the irradiations in two onsite reprocessing plants. The high level radioactive waste from this chemical separation contains the radioactive fission products generated in the reactors, some unrecovered uranium, trace quantities of plutonium and other irradiation products, most of the reprocessing chemicals and non-radioactive target materials, and large amounts of sodium nitrate. The sodium nitrate is formed during neutralization of nitric acid based reprocessing solutions with sodium hydroxide. This alkaline waste is stored in carbon steel tanks on plant. About 10% of the waste is sludge formed from precipitates of the hydroxides of iron, manganese, and aluminum. The sludge contains most of the strontium-90 and small amounts of actinide elements not recovered in the reprocessing plant.

Storage of radioactive waste in tanks is temporary, and is not considered a method for permanent disposal of SRP waste. A long term solution to nuclear waste at SRP is to remove the waste from tanks and immobilize that waste in a high integrity solid form. Thus, high level waste will be incorporated into borosilicate glass, stored temporarily on site, and then transferred to a federal repository when one is available.

MATERIAL OF CONSTRUCTION SELECTION CRITERIA

The DWPF is designed with a minimum of hands-on maintenance in accord with existing separations facilities. Radioactive process cell operations are controlled from outside of the cell. All major equipment has to be removed from the cell for decontamination before repair, replacement, or disposal. Thus, equipment depends upon gasketed connections rather than field welds for mating between equipment and transfer lines. This makes equipment especially sensitive to pitting and crevice corrosion. Design life is 20 years for major equipment other than the Glass Melter, and 5 years for easily replaced equipment. The Melter is designed for a minimum 2 years of continuous operation.

An additional criterion is minimization of the number of

* Denotes reference found in back of text

alloys used, to maximize interchangability and to maintain quality control in installation and field repairs.

A third criterion has been to avoid the addition of chemical corrosion inhibitors, since their use tends to increase the amount of waste that eventually must be processed and stored.

PRELIMINARY EVALUATION AND TESTING

Accumulation and concentration of sludge formed during caustic neutralization presents unique problems in the selection of materials for construction of vitrification process equipment. Halides, nitrates, nitrites, sulfates and phosphates which are concentrated during waste storage remain relatively unreactive in the alkaline storage environment. The gelatinous hydroxide sludge includes iron, copper and mercury which can contribute to crevice corrosion and pitting. The vitrification process requires reacidification of the concentrated sludge, which results typically in a pH of 3 with several thousand parts per million (ppm) chloride and fluoride. Temperatures range from ambient to 1150 C, and numerous reactions occur between the chemicals, which include organics, oxidizing, reducing and inert species.

Alloy selection began by summarizing waste types to be processsed, and definition of maximum anticipated concentrations of chemical species. Computerized chemical process evaluation programs were used to determine recycle and processing effects on concentrations. The levels of 135 chemical species in 177 process streams were computed. Test solutions were formulated based upon major chemical components, acidic species, and ionized transition metals that are known to enhance general or localized attack (e.g. cupric and ferric ions).

General conditions for preliminary tests are shown in Table 1. Time, temperature, and pH for major operations were determined from pilot scale operations, which have generally been conducted in 304L vessels, without halides, and without hazardous species such as mercury (2,3). Equipment with similar corrosive species and conditions were grouped together resulting in the definition of four major "Corrosion Control Zones": Sludge Receipt and Adjustment (SRAT), Slurry Mix and Evaporation (SME), Glass Melter, and Melter Offgas. The SRAT and SME processes require boiling the sludge portion of the waste with formic acid to break down the gelatinous hydroxides, and to reduce mercury contained in the sludge to a metal which can be stripped from the melter feed.

The vitrification process occurs between 600 and 1150°C, and results in various amounts of partitioning, decomposition, vaporization, and combustion of the chemical species. Compounds and concentrations of species in the melter offgas were calculated based on experimentally determined partitioning coefficients, from engineering melter operations with simulated nuclear wastes (2,3).

Alloy 20 (i.e. Carpenter 20Cb-3) was selected as the reference material for all process vessels operating below 300°C in the Melter Feed Preparation and Melter Offgas areas. This selection was primarily based on this alloy being the least expensive, and most readilly available alloy with virtual immunity to chloride stress corrosion cracking. Additional materials were selected for testing which offered superior corrosion resistance or cost savings relative to Alloy 20. One reason that backup materials were necessary, was concern that the relatively high copper content of Alloy 20 (3.5 %) might cause accelerated attack by the high mercury content of the radioactive waste. Inconel 690 was selected as the reference Melter alloy based upon demonstrated oxidation and sulfidation resistance in engineering melter tests (4,5,6).

The greatest uncertainties remaining after preliminary evaluation involved the interactions between formic acid, halides, mercury and abrasion in the feed preparation areas (SRAT & SME), and corrosion effects of mercury in the Melter Offgas. To address these concerns, a small scale melter test was contracted to Battelle Pacific Northwest Laboratory (BNWL), to expose metal coupons to mercury, and higher halide concentrations than possible in engineering scale equipment. The melter feed was produced at the Savannah River Laboratory (SRL) using normal processing cycles and included all the anticipated chemical species. Non-radioactive nuclides or elements with similar chemical characteristics were substituted for radioactive species. BNWL specially constructed a virtually leak-free melter for the tests, allowing full control of offgas humidity and oxidation/reduction state.

Selection of reference materials was followed by short tests to provide a relative ranking of the alloys, and define the modes of attack. Tests were run for 1 to 2 weeks at higher than anticipated concentrations, under submerged, vapor space and condensate conditions. Two-liter flasks with attached condensers (Demo flasks) (7) were run under total reflux conditions. Parameters evaluated included alloy composition, temperature, pH, halide concentration, crevices, stresses, welds, and form of mercury.

RESULTS OF PRELIMINARY TESTING

Preliminary tests were run to determine if Type 304L was acceptable at low formic acid concentrations in the melter feed preparation areas (SRAT & SME). This data was not available in the literature. Table 2 shows acceptable general corrosion rates for Types 304 and 304L in formic acid concentrations at or below 3.5%. Alloys 316L, 20, and C-276 have acceptable general corrosion rates at higher formic acid concentrations.

Although Types 304L and 316L had acceptable corrosion rates in formic acid, the stainless steels had questionable resistance to chloride stress corrosion cracking in SRAT and SME solutions. Table 3 shows cracking in dilute formic acid solutions, with chloride contents as low as 100 ppm. This chloride level is far below the minimum level expected in DWPF sludge. Therefore, the earlier elimination of Type 304L as a reference material for DWPF process tankage and process lines was confirmed. To minimize costs, Type 304L was retained for most radioactively contaminated service piping and chemical feed tankage not in contact with sludge. Alloy Type 304L was also retained in laboratory testing as a reference for general corrosion rates.

Four alloys were tested in simulated SRAT solutions. This testing provided verification of anticipated low corrosion rates, Table 4. Less expected were exceptionally severe pitting and crevice attack of Alloy 20. This testing indicated that Alloy 20 was not sufficiently alloyed for the SRAT area. Only the highly alloyed C-276 resisted localized attack and had a very low general corrosion rate.

Simultaneously, tests were run in simulated SME solutions, using the same alloys tested for SRAT service, Table 5. Based on these results, Alloy 20 was not suitable, but Alloy C-276 was satisfactory.

Critical operating conditions for further offgas corrosion tests were based upon results of the SRL/BNWL melter tests: Offgas quencher solution pH is 2.2, or higher, with a major fraction of the mercury as mercuric chloride. The melter tests also produced realistic offgas condensate that has been used in laboratory corrosion tests. Examination of the melter after testing revealed that Inconel 690 is not locally attacked where temperatures remain above the dew point. However, severe pitting was seen after one month's operation of the Inconel 690 offgas Quencher. Therefore, the offgas Quencher became an area of special study. The melter's Monofrax K-3 refractory experienced no unusual attack. This gives confidence that Inconel 690, and Monofrax K-3 refractory are suitable for melter construction. Based upon this test, Inconel 690 was retained as the Melter reference material. Metal samples exposed in the melter offgas condensate tank indicated that highly alloyed nickel-based alloys are required to restrict crevice corrosion and pitting, Figure 1. Directional pitting (9) occurred in Alloy 20. However, general corrosion rates were low in all alloys.

Additional alloys were investigated during laboratory testing to approximate offgas quencher conditions. Results are summarized in Table 6. Of the alloys tested, again Alloy C-276 was the only alloy with a high resistance to both general and localized attack. Localized attack was especially severe in Alloy 20 exposed to melter offgas condensate. Directional pitting produced holes completely through 1/4 inch samples in 72 hours.

Since Alloy 20 suffered from localized attack in all the simulation tests, it was removed as the reference material for SRAT, SME, and Melter Offgas Systems. Alloy C-276 was selected as the new reference material for these process zones.

SCOUTING TESTS AND RE-EVALUATION OF OFFGAS REFERENCE MATERIALS

In the preliminary tests, Alloy 20 experienced unusual pitting attack. Similar attack had been observed previously by Streicher in ferric chloride solutions (9). Based on these observations, Warren (10) recommended testing other alloys with combined chromium and molybdenum contents above 30%. Table 7 lists some of the alloys that fit this criterion. These alloys were tested in simulated offgas solutions at different pH's and temperatures. Figure 2 shows that general corrosion tends to decrease as chromium plus molybdenum content of the alloy increases. As expected, the lower the pH and the higher the temperature, the more severe the general attack. Table 8 shows that localized attack follows the same general trends. When severity of localized attack (the maximum depth of pitting or crevice attack) was plotted as a function of pH and alloy composition (combined chromium and molybdenum content) it was apparent that increases in pH reduced the quantity of alloying agent required to suppress localized attack. Figures 3 and 4. Several alternative ways of combining the alloying elements were plotted, and the best overall fit occurred when composition was described as chromium content plus two times molybdenum content minus four times the copper content. Figures 5 and 6. Thus, nickel-based alloys required at least 9% molybdenum to resist pitting and crevice corrosion at pH 1.6. Copper was very deleterious as an alloying agent in this system. Alloy C-276 was locally attacked at low pH and high temperature. The only alloy without localized attack was ALLCORR, which is a new alloy without operating experience in a chemical processing plant.

Tests with several alloys containing copper showed that their pitting rates were proportional to copper content, suggesting an undesireable interaction between the copper in Alloy 20 and the mercury in the test solutions.

Testing with mercury as the reduced metal, as mercurous chloride (Hg_2Cl_2), and as mercuric chloride ($HgCl_2$) revealed that mercuric ion (Hg^{++}) acts as a pitting and crevice corrosion catalyst at concentrations as low as 100 ppm. The other forms of mercury did not significantly affect corrosion in the tests. Dissolved mercuric chloride was reacted with sodium hydroxide to determine what level of caustic was necessary to convert corrosive species to unreactive forms. Based upon X-ray diffraction of the resulting precipitate, a minimum of 0.5 molar free caustic was necessary.

VERIFICATION TESTING

After scouting tests had determined the relative ranking of alloys under nominal conditions, it was necessary to verify the material selection using prolonged exposure, realistic process cycles, and equipment with construction methods and details similar to actual vessels.

SRAT and SME verification was performed in a specially constructed Alloy C-276 vessel with 35-gallon capacity. The vessel has a flanged top, and flanged condenser, permitting disassembly for inspection of steam and cooling coils, welds, agitator and agitator baffles. Racks in the vessel and the condenser permitted quantitative testing of Alloy C-276 and other materials. Full process cycles were conducted, with the maximum anticipated levels of halides, nitrates, sulfates, and mercury. Extended tests in Demo flasks have simulated the extremes of pH and temperature for times equivalent to one year operation with negligible corrosion of Alloy C-276.

Volatile gasses off the melter at 650°C are quenched to 60°C with recycled offgas condensate in the Melter Offgas Quencher. SRL/BNWL melter test indicated that Inconel 690 is satisfactory for offgas system components that are maintained above the dew point, but the Quencher operates in wetted conditions at temperatures where pitting was seen in Alloy C-276 during preliminary testing. Table 9 summarizes laboratory tests using SRL/BNWL and simulated offgas solutions. Again, general corrosion was minimal. ALLCORR has been selected as the reference quencher material based on these and preliminary tests. A spare quencher will be built from Alloy C-276. The economic and performance trade offs of these two alloys will be evaluated when the quenchers are routinely inspected during melter changeout.

Melter offgas materials have been exposed to maximum anticipated concentrations of halides and mercury for long term verification tests. Table 10. Although the ALLCORR samples had superior general corrosion rates, the corrosion rates for Alloy C-276 are acceptable.

A pilot scale Melter, with a Melter Offgas System constructed of Alloy C-276 with an ALLCORR Quencher, is being operated to verify materials during long exposures to operating cycles with maximum anticipated concentrations of mercury and halides.

SUMMARY AND CONCLUSIONS

Type 304 stainless steel is satisfactory for general service piping and tankage in the DWPF. The combined effects of elevated temperatures, reacidification, and concentration of corrosive agents, however, make Type 304L unacceptable for use with process solutions derived from sludge.

Mercuric ion catalyzes halide pitting and crevice attack in melter off gas solutions. Attack is most severe at high temperatures and low pH. Alkalinity exceeding 0.5 molar free caustic is necessary to deactivate the mercuric ion.

Alloys containing more than 0.5% copper are subject to accelerated corrosion or directional pitting due to interaction with mercury in the process solutions.

All alloys with a total chromium plus molybdenum content exceeding 30% were able to resist crevice corrosion in simulated process solution at pH 6 and 40°C. Only nickel based alloys with a minimum of 9% molybdenum were able to resist crevice attack at pH 1.6 and 40°C. Only Alloy C-276 and ALLCORR resisted localized attack at pH 6 and 90°C. ALLCORR was the only alloy tested that could resist pitting and crevice corrosion at pH 1.6 and 90°C.

Inconel 690 is satisfactory for melter construction, provided that temperatures remain above the dew point. If condensation occurs, then Inconel 690 is subject to pitting and crevice corrosion. Monofrax K-3 refractory was satisfactory in contact with glass containing halides and sulfates.

Alloy C-276 has been selected as the reference material for process equipment. Inconel 690 is the reference alloy for Melter fabrication. ALLCORR has the best corrosion resistance of materials tested for the melter off gas Quencher. Alloy C-276 or Hastelloy Alloy C-22 may also be satisfactory for the Quencher.

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Table 1 - Conditions for Preliminary and Verification Tests

	SRAT*		SME*		Offgas**	
	Pre.	Ver.	Pre.	Ver.	Pre.	Ver.
Cl,ppm	20000	3800	20000	3800	20000	2500
F,ppm	2300	400	2300	400	2300	300
SO_4,ppm	300	60	300	60	1400	800
NO_3,ppm	--	--	--	--	0	6000 & 0
Hg,ppm	28000	10600	10000	10600	4500	1000
pH	4,6	3.2,6	4,6	3.2,6	1.6,6	2.2,6,9,12
Temp,°C	BOIL	BOIL	95	95	40,90	40,60,90

* pH adjusted with formic acid
** pH adjusted with sulfuric acid

Table 2 - General Corrosion Rates in Formic Acid Solutions
1000 Hours at 98°C, mils/year

Alloy	0.0004% (pH4)	0.04% (pH3)	3.5% (pH2)	5%(8)	90%(8)
304L	NIL	NIL	2	59	26
304 (Cast)	--	--	9	--	--
316L	--	--	2	2	1
316 (Cast)	--	--	0.5	--	--
20Cb-3	--	--	--	8	1
C-276	--	--	--	1	1

Table 3 - Stress Corrosion Cracking of 304L in Formic Acid
Welded U-Bend Samples, pH4 at 85°C

Chloride, ppm	250 Hours	500 Hours
1000	2 of 3 Cracked	2 of 3 Cracked
500	0 of 3 Cracked	2 of 3 Cracked
100	1 of 3 Cracked	1 of 3 Cracked

Table 4 - Preliminary Testing of Sludge Receipt/Adjustment Tank Crevice Coupons Submerged in Boiling Formated Sludge 340 Hours

Material	General Corrosion (mils/year)	Localized Attack
304L	4	Crevice & Pitting to 19 mils
316L	1.4	Crevice & Pitting to 3 mils
20Cb-3	0.2	Crevice & Pitting to 1 mil
C-276	0.1	No Visible Attack

Table 5 - Preliminary Testing of Slurry Mix/Evaporator Crevice Coupons Submerged in Formated Sludge and Frit - 340 Hours at 95°C

Material	General Corrosion (mils/year)	Localized Attack
304L	1.2	Crevice & Pitting to 29 mils
316L	0.9	Crevice & Pitting to 10 mils
20Cb-3	0.2	Crevice to 5 mils
C-276	0.1	No Visible Attack

Table 6 - Preliminary Testing Of Melter Offgas System Crevice Coupons Submerged in Concentrated Synthetic Solution - 340 Hours at 90°C

Material	General Corrosion (mils/year)	Localized Attack
304L	38	Crevice & Pitting to 27 mils
316L	21	Crevice & Pitting to 33 mils
20Cb-3	14	Crevice & Pitting to 5 mils
600	22	Crevice & End Grain & Weld Attack
690	8	Crevice & Weld Attack
800	22	Crevice & Pitting & IGA
C-276	1	No Visible Attack

Table 7 - General Corrosion in Preliminary Melter Offgas Solution in mils/year

Alloy	pH1.6 @ 40°C	pH1.6 @ 90°C	pH6 @ 40°C	pH6 @ 90°C
690	1.9	22	0.1	2.3
617	0.2	20	0.2	0.6
20Mo-4	4.2	11	0.1	0.5
HR13	0.4	10	0.2	0.9
X	1.1	8.7	0.1	0.5
C-276	0.3	8.7	0.1	0.1
SAN28	2.8	6.3	0.1	0.2
625	0.2	6.4	0.1	0.4
C-22	0.2	2.0	0.2	0.5
G-30	0.3	0.4	0.1	0.2
ALLCORR	0.1	0.2	0.1	0.1

Table 8 - Localized Attack in Preliminary Melter Offgas Solution Submerged Crevice Coupons

Alloy	pH1.6 @ 40°C	pH1.6 @ 90°C	pH6 @ 40°C	pH6 @ 90°C	%Cr	%Mo
SAN28	CC:45	GC,CC:8,P:19	CC:7	CC:5	3.5	3.5
20Mo4	CC:54	GC,CC:18	NVA	CC:	23.5	4
690	CC:7	GC,CC:1	NVA	CC:11	29	-
HR13	CC:3	GC,CC:1	NVA	CC:1	25.5	5.7
617	IGA,CC:1	IGA,GD	NVA	IGA,CC	22	9
G-30	CC:3	CC	NVA	CC	29.5	5.5
X	CC	GC,CC	NVA	CC	22	9
C-22	NVA	CC:2	NVA	CC:2	21	13
625	NVA	GC,CC:1	NVA	CC:1	21.5	9
C-276	NVA	GC,CC:1	NVA	NVA	15.5	16
ALLCORR	NVA	NVA	NVA	NVA	31	10

40°C samples exposed 187 hours, 90°C samples exposed 329 hours.
Numerals = depth in mils during length of test.
No numeral indicates less than 1 mil localized attack.

CC = crevice corrosion
IGA = intergranular attack
P = pitting
GC = general corrosion evident
GD = grain dropping
NVA = no visible attack

Table 9 - Simulations of Melter Offgas Quencher

Material	General Corrosion (mils/year)	Localized Attack
o Submerged in Boiling SRL/BPNL Quench Tank Solution, 65 Days		
ALLCORR	0.03	Crevice Corrosion
C-4	0.28	Crevice Corrosion, Weld Etched
C-22	0.06	Crevice Etched
C-276	0.42	Weld Etched
o Wet/Dry Cycles with SRL/BPNL Quench Tank Solution, 96 Cycles		
ALLCORR	0.49	Discolored, No Visible Attack
C-4	0.92	Discolored, No Visible Attack
C-22	0.47	Discolored, No Visible Attack
C-276	0.65	Discolored, No Visible Attack
o Verification Solution Sprayed on 375°C Crevice Coupons, 55 Cycles		
ALLCORR	2.9	Intergranular Attack Pitting less than 1 mil
C-276	4.2	Pitting to 1 mil
o Verification Solution With 0.1% NO_3, Sprayed on 375°C Coupons, 55 Cycles		
ALLCORR	0.1	No Visible Attack
C-22	0.7	No Visible Attack
C-276	0.3	No Visible Attack

Table 10 - Verification Testing in Offgas Solution
Welded Crevice Coupons Submerged in pH2.2 at 100°C

Alloy	Hours	General Corrosion (mils/year)	Observations
Verification Solution			
ALLCORR	744	0.08	No Visible Attack
ALLCORR	2539	0.03	No Visible Attack
C-276	744	3.6	Uniform Gen. Corr.
C-276	2061	1.3	Uniform Gen. Corr.
Verification Solution with 0.10% NO_3^-			
ALLCORR	744	0.08	No Visible Attack
ALLCORR	2539	0.04	No Visible Attack
C-276	744	3.6	Uniform Gen. Corr.
C-276	2061	1.3	Uniform Gen. Corr.

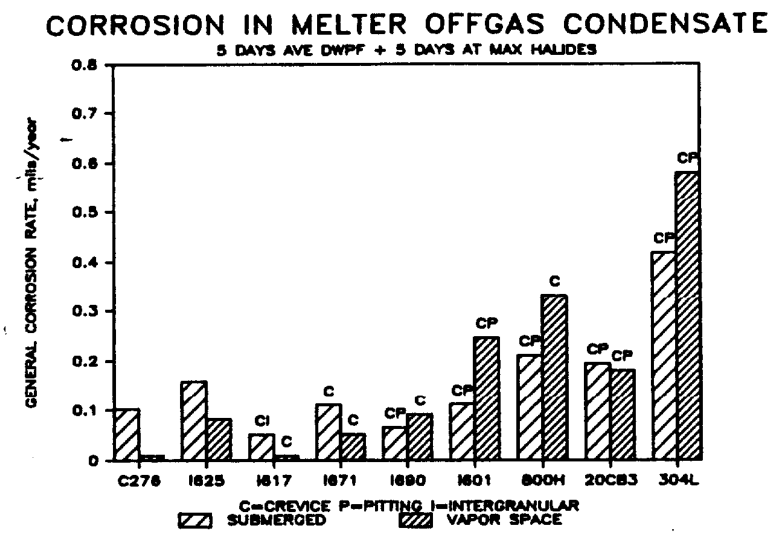

Figure 1 - Alloy Samples Exposed 10 Days to Melter Condensate

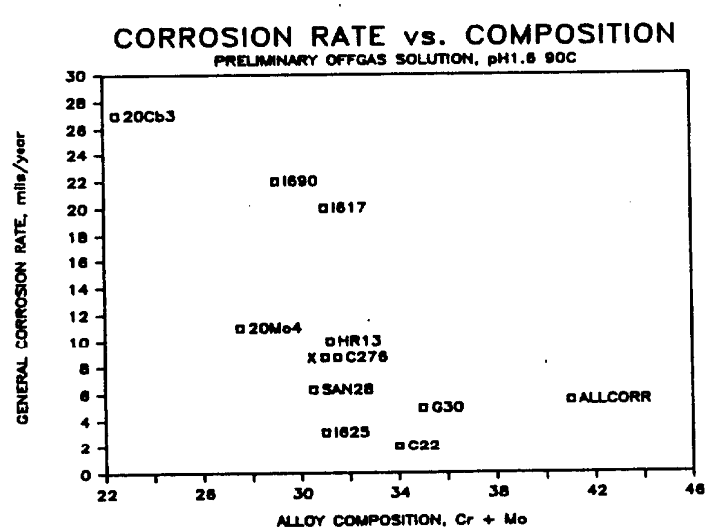

Figure 2 - Alloy Cr & Mo Decrease General Corrosion Rate

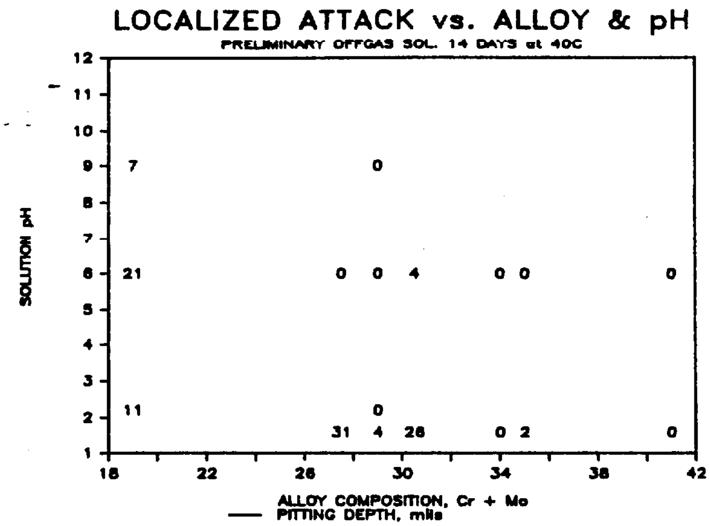

Figure 3 - 40C Localized Attack is Decreased by pH. and Cr + Mo

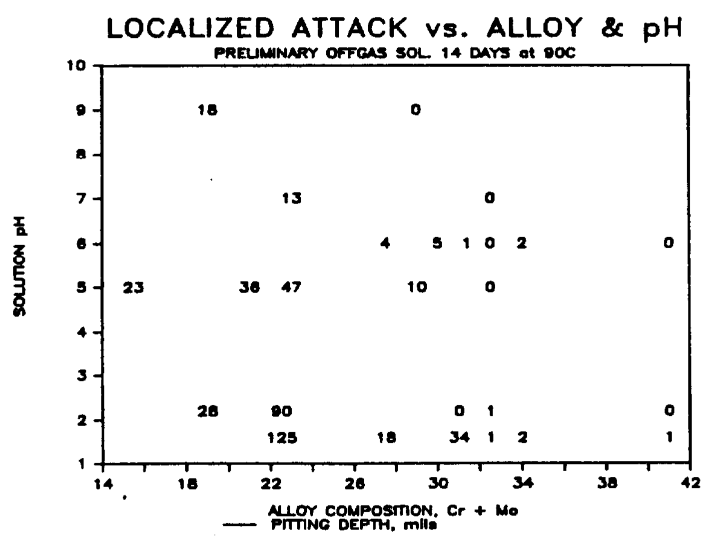

Figure 4 - 90C Localized Attack is Decreased by pH, and Cr + Mo

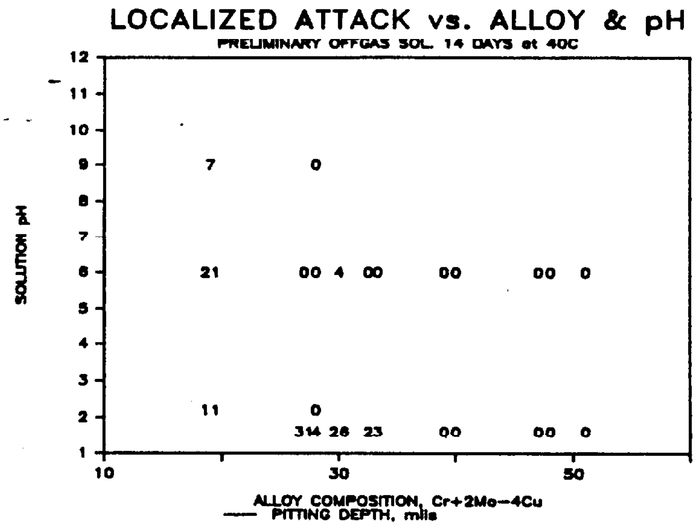

Figure 5 - Maximizing Cr + 2Mo - 4Cu Minimizes 40C Localized Attack

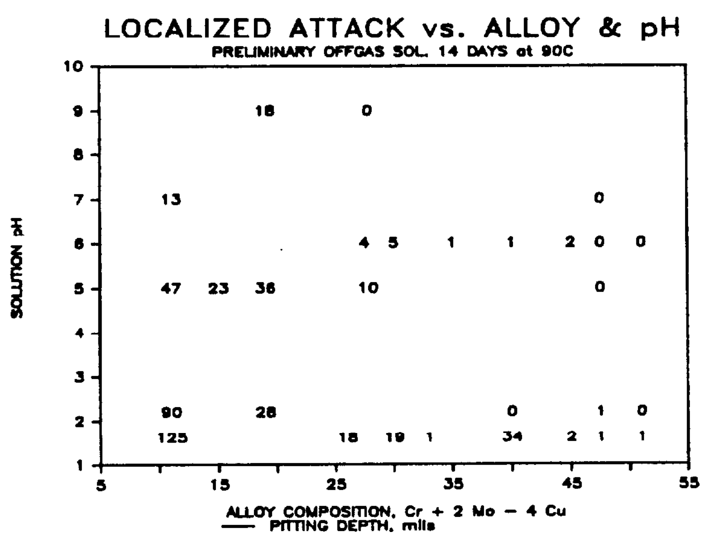

Figure 6 - Maximizing Cr + 2 Mo - 4Cu Minimizes 90C Localized Attack