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A STUDY OF THE EFFECTS OF INTERLEAKAGE OF AMMONIA AND SEAWATER ON CORROSION AND SCALING OF CANDIDATE MATERIALS FOR OTEC HEAT EXCHANGERS

Final Report

W. D. Grimes, C. F. Schrieber,
and J. A. Manning

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9700 South Cass Avenue
Argonne, Illinois 60439

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July 1980

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EXECUTIVE SUMMARY

The purpose of this work was to assess the extent the interleakage of ammonia and seawater affect corrosion and scaling of candidate OTEC heat excanger materials -- Al-5052, Alclad 3003, copper alloys 706, 715 and 722, AL-6X stainless steel and CP titanium.

Systems were constructed to investigate four different possible OTEC environments. Projected leak site corrosion and scaling behavior was evaluated using 1.8 m/sec (6 fps), 30°C (86°F) seawater inside 6.1 m (20 ft) tubes, with 16°C (60°F) anhydrous ammonia injected into the tube at a predrilled artificial leak site. The effects of uniform ammonia concentrations in seawater were determined using 1.8 m/sec, 30°C recirculating refreshed seawater plus 5,10, and 20 ppm ammonia, with both tube and coupon specimens. Coupons only were exposed to recirculating 1.8 m/sec, 30°C anhydrous ammonia plus 0.0 and 1.0% seawater, and to 1.8 m/sec, ambient temperature, once-through seawater.

Corrosion resistance was determined by evaluation of localized and uniform surface attack. In these tests, CP titanium and AL-6X stainless steel showed no measureable corrosion. Alclad 3003, with 7 mils of Al-7072 cladding alloy, showed good performance in seawater and seawater plus ammonia. Al-5052 tubes showed unacceptable service in contact with seawater and ammonia interleakage. Copper alloys tested showed similar but more rapid failure with seawater inside the tubes and ammonia interleakage.

Leakage of ammonia into seawater can produce calcareous deposits on heat transfer surfaces. Given appropriate conditions, ammonia into seawater leaks may be self-healing, but the presence in seawater of ammonia concentrations above 20 ppm will require continuous cleaning to maintain good heat transfer rates.

ABSTRACT

Assessment has been made on the effect of small concentrations of ammonia in seawater and varying concentrations of seawater in anhydrous ammonia upon corrosion and scaling of candidate OTEC heat exchanger materials -- Al-5052, Alclad 3003, copper alloys 706, 715 and 722, AL-6X stainless steel and CP titanium.

AL-6X stainless steel and CP titanium showed exceptional corrosion resistance to all test environmets.

Alclad alloy 3003 showed satisfactory performance in seawater and seawater plus ammonia environments. Only minimal pitting was observed and this was limited to the sacrificial cladding in seawater plus ammonia only. Cladding alloy 7072 showed unacceptable corrosion resistance in anhydrous ammonia containing low seawater concentrations.

Al-5052 tubes showed unsatisfactory corrosion behavior in the presence of seawater flow with ammonia interleakage.

Copper alloys considered showed unacceptable corrosion resistance in all seawater environments containing ammonia.

Low pressure differentials between seawater and anhydrous ammonia in the tube testing unit resulted in scaling and moderately efficient plug seal formation at the artificial leak sites of the tubes.

It is recommended that Alclad 3003, CP titanium and AL-6X stainless steel tubes be assessed for suitability in the presence of probable OTEC cleaning systems.

I. INTRODUCTION

Ocean Thermal Energy Conversion (OTEC) systems employ the thermal difference available between warm surface waters and cold deep ocean waters to power an ammonia-cycle electric generator. Operation of a commercial-size plant would require the flow of large quantities of seawater through condensers and evaporators, where heat exchanger costs may represent a substantial fraction of the total plant investment.

The potential for tube leakage during operation of these heat exchangers and the resulting effects of seawater-ammonia mixtures on system materials is an important performance factor for consideration in system design. In recognition of the need for additional corrosion and fouling information, a study was sponsored by the Department of Energy, Division of Central Solar Technology, under the direction of the Argonne National Laboraory and conducted by The Dow Chemical Company at Freeport, Texas. Candidate OTEC heat exchanger materials included aluminum, copper, stainless steel and titanium alloys. Relevant information sought included degree and composition of scaling films and measurement of general and localized corrosion under the test conditions.

II. SUMMARY AND CONCLUSIONS

1. Superior performance was shown by AL-6X stainless steel and CP titanium in all environments tested. Corrosion weight losses for these materials were so low as to approach the limits of detection using conventional laboratory equipment and techniques. No significant localized attack was observed on any specimen, including those exposed to the most severe condition of anhydrous ammonia leakage directly into seawater.

2. Satisfactory performance was given by Alclad alloy 3003 in seawater and seawater plus ammonia environments. Tubes of this material showed minimal pitting which was limited to the depth (0.0065 inches) of the Al-7072 sacrificial cladding. In all cases, the Al-3003 base alloy was not penetrated. Test duration of the tubular material was 5.5 months.

3. Al-7072 clad alloy showed unacceptable corrosion resistance in anhydrous ammonia containing low concentrations of seawater.

4. Al-5052 tubes showed unacceptable service in the presence of seawater with ammonia interleakage. Four of six tubes tested under conditions of anhydrous ammonia leakage into seawater failed because of continued leak hole enlargement. The two remaining tubes experienced perforation upstream and downstream of the artificial leak sites after 6 and 8 months test duration.

5. Copper alloys tested showed unacceptable performance under conditions of ammonia interleakage into flowing seawater. Copper alloys 706, 715 and 722 were considered. Tube failure was most evident at the artificial leak sites in the form of smooth uniform attack. Less rapid, but equally uniform attack was observed on surfaces adjoining the leak areas.

6. Copper alloy 706 (CA* 706) showed favorable corrosion behavior in seawater and anhydrous ammonia 99.99%. It should be recognized however that this observed resistance might not extend to ammonia containing the concentration of water required to inhibit stress corrosion cracking of steel. Ammonia is commonly handled and shipped in steel.

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*Copper Alloy. Designation developed by The Copper Development Association.

7. Tube test unit operation at a 0.4 kg/cm^2 (5 psi) anhydrous ammonia-seawater pressure differential resulted in plugging and sealing of 100% of the initial artificial leak sites. Operations at a pressure differential of 0.7 kg/cm^2 (10 psi) were also highly effective at differentials of 1.4 kg/cm^2 (20 psi), scale formation was observed to plug the artificial leak.

   The observation of leak plugging by precipitated scale at the pressures investigated might be considered as a basis for limiting pressure differentials to the greatest practical extent.

   Calcite scale forms initially followed by aragonite and brucite as ammonia concentrations increase. At concentrations of over 1000 ppm ammonia in seawater, as have occurred at large ammonia-seawater heat exchanger leak sites, scales greater than 3 mm (0.12 in) thick have formed in less than 8 hours. These scales are reasonably adherent rather than loose and flocculant and have little tendency to be carried away in a water stream as fast as they are formed.

III. EXPERIMENTAL PROCEDURES

A. Program Methodology

Consultation between The Dow Chemical Company, Argonne National Laboratory, and Department of Energy officials on May 3, 1978, in Washington, D.C., established the general program objectives for the research effort at Freeport, Texas. Data generated later in the program resulted in the inclusion of aluminum Alclad alloy 3003 and copper alloys 715 and 722 into the research effort. The final program methodology for the test program is given in Table III-1.

B. Test Unit Description

Program requirements separated into four distinct tasks. Five test units were deemed necessary to meet the program objectives.

1. Tube Test Unit

The tube test unit was designed to assess the performance of candidate alloy tubes in the event of ammonia leakage into seawater. Schematics of the test apparatus are given in Figures III-1 and III-2, and a photograph of the test unit is given in Appendix B, Figure VIII-2. The system maintained a continuous average tube flow of 1.8 m/sec (6 fps), 30°C (86°F) seawater and and an ammonia pressure of 9.8 kg/cm^2 (140 psi) at 16°C (60°F). Specimens consisted of 25.4 mm (1 in) outside diameter tubes 6.1 m (20 ft) long. Within the tube unit, two independent anhydrous ammonia loops operated to isolate the contact between the copper and non-copper containing tubes. The unit was designed for automatic operation with failsafe system shutdowns in the event of pump failure, ammonia overpressure, or excessive ammonia leakage.

Interleakage of ammonia into seawater was accomplished by drilling 0.1 mm (0.004 in) and 0.2 mm (0.008 in) "leak" holes in the tube wall 1.5 m (5 ft) downstream of the tube end. Flow control for ammonia flowing into seawater consisted of regulating the seawater-anhydrous ammonia pressure differential.

TABLE III-1
PROGRAM METHODOLOGY

I. Tube Test Unit

Conditions			Materials	
Seawater	Ammonia	Exposure[1] (months)	Specimens	Alloys
Once-through 30°C, 1.8m/sec	Anhydrous 99.7% through 2 drilled "leaks" 1.5m downstream	4.5 9	25mm O.D., 6.1m long tubes	Alclad 3003 Al 5052-H32 Cu Alloy 706 Cu Alloy 715 Cu Alloy 722 Stainless AL-6X CP titanium

II. Seawater plus Ammonia Test Units
(non-copper and copper)

Conditions			Materials	
Seawater	Ammonia	Exposure (months)	Specimens	Alloys
Recirculating filtered, 30°C, 1.8m/sec with refreshment	Ammonium hydroxide		25mm O.D., 0.61m tubes and 150 x 19 x 1.5mm coupons	Al 5052-H32 Cu Alloy 706 Stainless AL-6X CP titanium
	5 ppm →	0.5,1.5,3		
	10 ppm →	0.5,1.5,3		
	20 ppm →	0.5,1.5,3		

III. Anhydrous Ammonia Test Unit

Conditions			Materials	
Ammonia	Seawater	Exposure (months) Non-Cu/Cu	Specimens	Alloys
Anhydrous, 99.99% 30°C, 1.8m/sec	Filtered		150 x 19 x 1.5mm coupons	Alclad 3003[2] Al 5052-H32 Cu Alloy 706 Stainless AL-6X CP titanium
	0% →	0.5,2/0.5,2		
	1% →	0.5,2/0.5,2		

IV. Once-Through Seawater Test Unit

Conditions		Materials	
Seawater	Exposure (months)	Specimens	Alloys
Filtered once-through 1.8m/sec	.5,1.5,3 6,12	150 x 19 x 1.5mm coupons	Alclad 3003 Al 5052-H32 Cu Alloy 706 Stainless AL-6X CP titanium

---

[1] Subject to alloy performance and insertion time.

[2] Alclad 3003 tested only in 1% seawater solution.

FIGURE III-1
GENERALIZED SEAWATER FLOWSHEET SCHEMATIC FOR TUBULAR TEST UNIT
TO DETERMINE THE EFFECTS OF AMMONIA LEAKAGE INTO SEAWATER

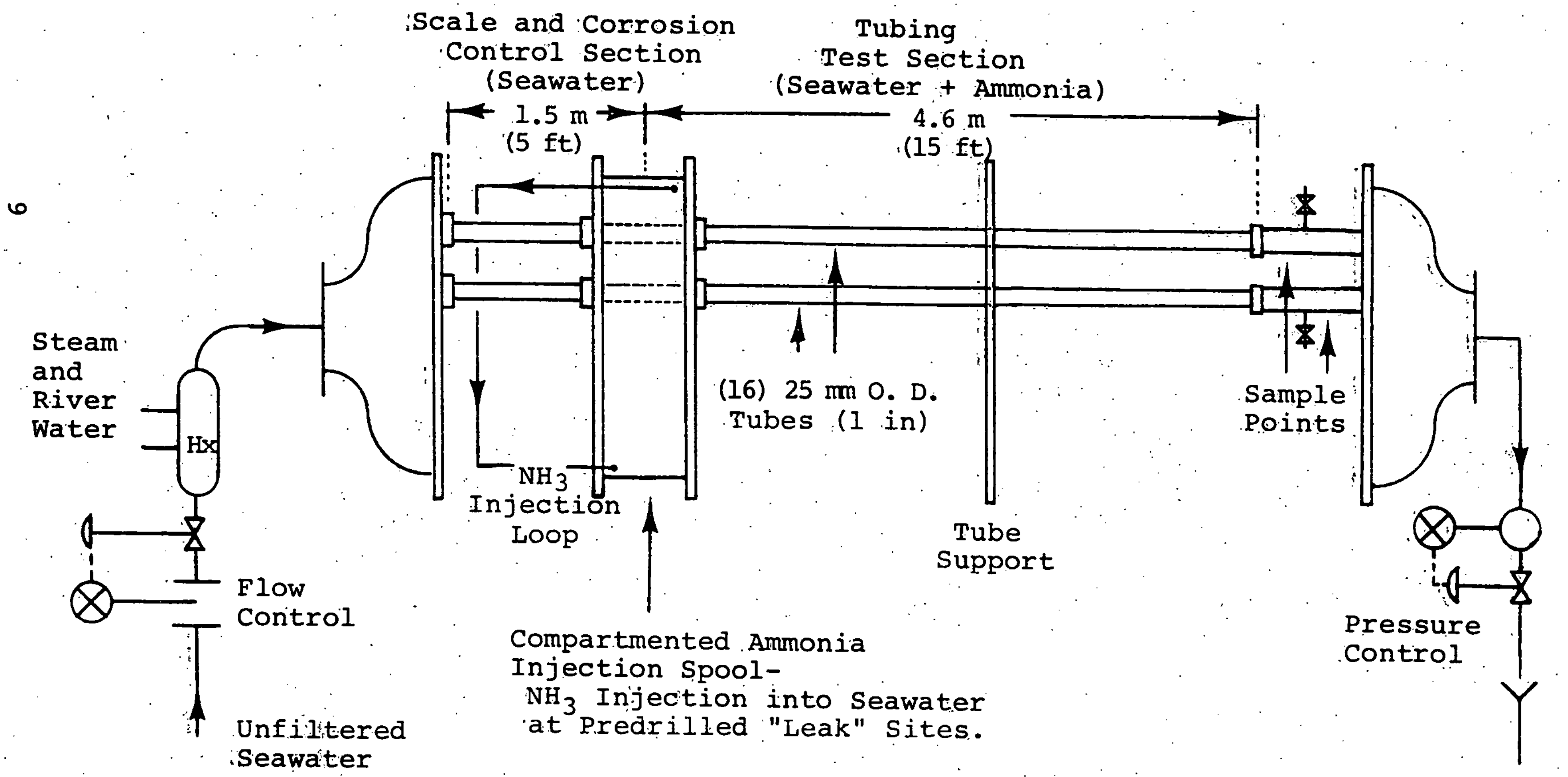
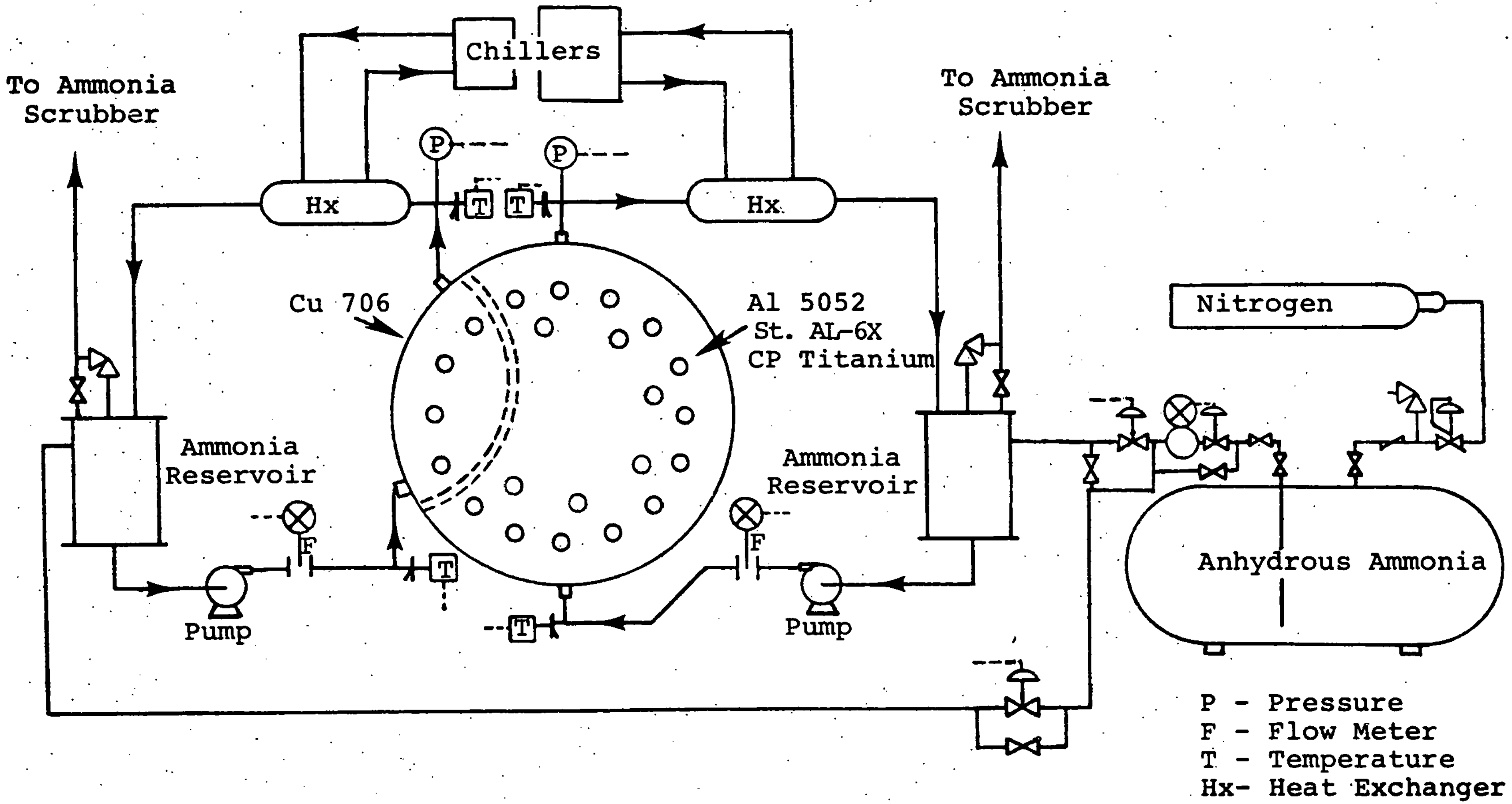


Figure III-2
SIMPLIFIED SCHEMATIC OF
AMMONIA INJECTION LOOP



Pressure of the anhydrous ammonia was fixed, control of the seawater at a lower absolute pressure. Factors having a serious impact on ammonia flows were corrosion and scale formation at the leak site.

Constructional materials for the unit were plastic-lined pipe (polypropylene, Saran®, and Teflon®), Alloy 20 stainless steel pumps, Hastelloy®C alloy seawater heat exchanger, type 316 stainless steel ammonia heat exchangers, and mild steel heat exchanger test spool and heads. Test specimens were afforded galvanic insulation by the use of nylon Swagelok® fittings at the tube test unit heads, and oversize mild steel Swagelok fittings with Teflon insulating sleeves at the ammonia test spool. The seawater sampling system placed at the downstream end of each tube was entirely of non-metallic (PVC) construction. Anhydrous ammonia was cooled using a chilled glycol-water solution. Seawater temperature was controlled using a mixture of steam and river water. Anhydrous ammonia was of technical (99.7%) purity. Seawater was as-received and unfiltered.

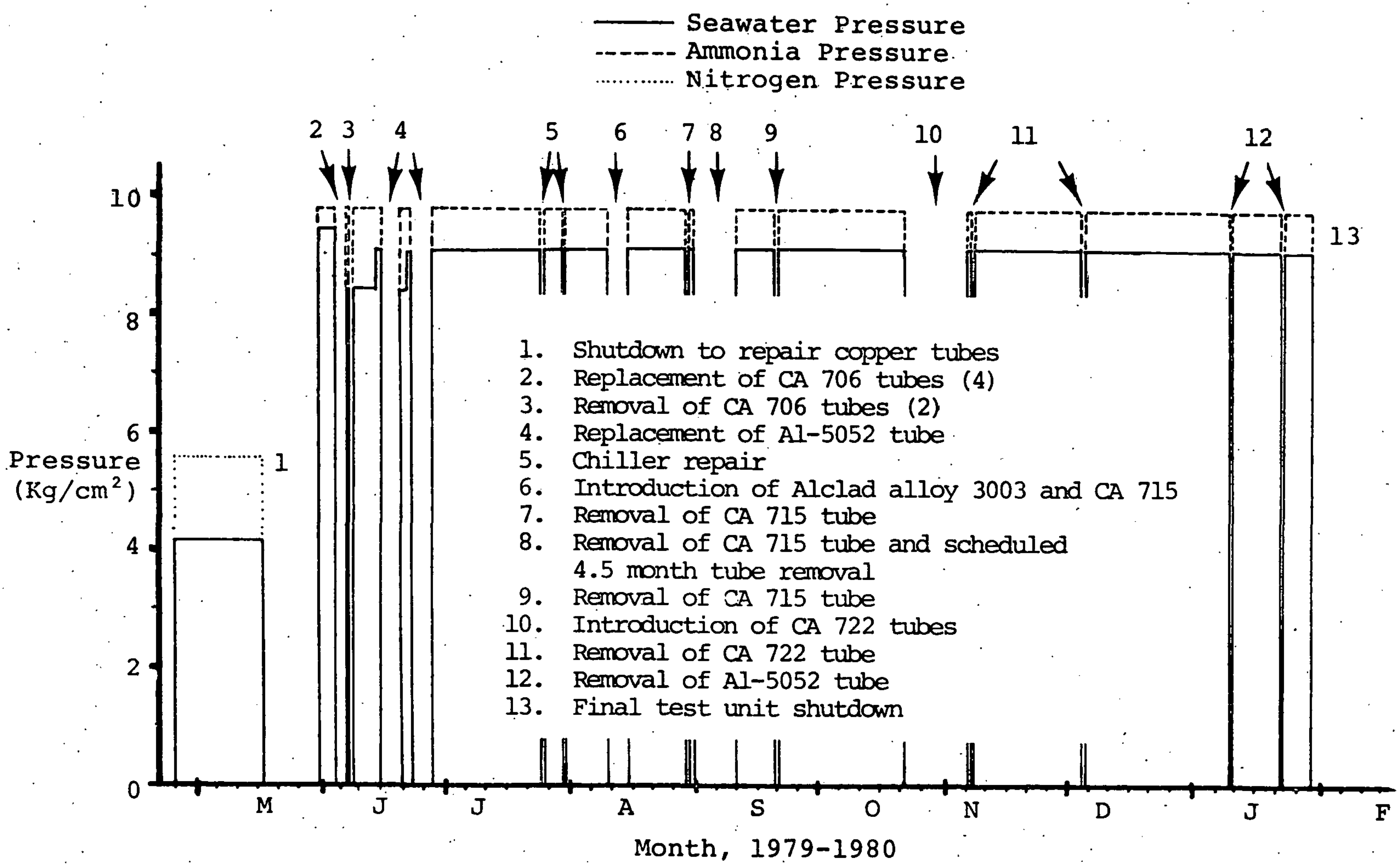
The tube test unit was scheduled for specimen removal after 4.5 and 9 months operation. A summary of operations is presented in Figure III-3.

2. Seawater Plus Ammonia Test Units

Two units were built for exposure of candidate alloys in seawater plus uniform concentrations of ammonia. A schematic of the test unit is presented in Figure III-4, and as photographs in Appendix B, as Figures VIII-3 and VIII-4. Exposure was to 5, 10, and 20 ppm ammonia in 1.8 m/sec (6 fps), 30°C (86°F) seawater. Specimens consisted of 0.61 m (2 ft) long, 25.4 mm (1 in) outside diameter tubes and 150 x 19 x 1.5 mm (6 x 0.75 x 0.06 in) flat coupons. One loop was used specifically for copper alloys. The other loop for all other alloys. Both units contained the instrumentation necessary for automatic control and failsafe shutdown to prevent specimens from experiencing excessive ammonia concentrations.

FIGURE III-3

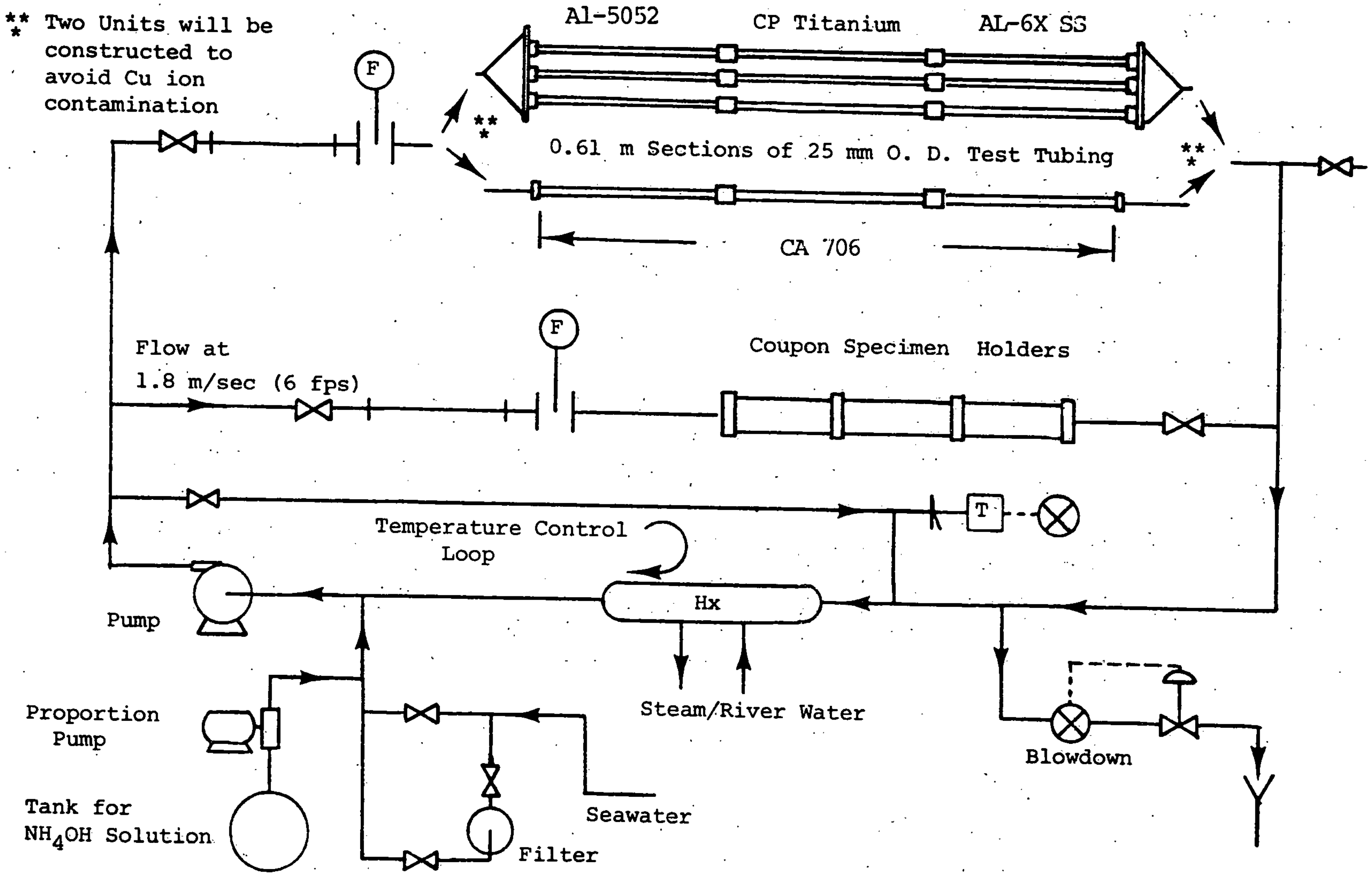
OPERATIONAL LOG FOR TUBE TEST UNIT



1. Shutdown to repair copper tubes
2. Replacement of CA 706 tubes (4)
3. Removal of CA 706 tubes (2)
4. Replacement of Al-5052 tube
5. Chiller repair
6. Introduction of Alclad alloy 3003 and CA 715
7. Removal of CA 715 tube
8. Removal of CA 715 tube and scheduled 4.5 month tube removal
9. Removal of CA 715 tube
10. Introduction of CA 722 tubes
11. Removal of CA 722 tube
12. Removal of Al-5052 tube
13. Final test unit shutdown

FIGURE III-4

SIMPLIFIED SCHEMATIC FOR CORROSION TEST UNITS TO DETERMINE THE EFFECTS OF SEAWATER PLUS LOW CONCENTRATIONS OF AMMONIA



** Two Units will be constructed to avoid Cu ion contamination

Aqueous ammonia was injected into the refreshment seawater downstream of the recirculating pump to assure adequate mixing of the test solutions. Concentrations of ammonia in seawater were routinely monitored by analysis of the system blowdown. Solution renewal was provided for by the use of a continuous system blowdown of 15 liter/min (4 gpm), for a total seawater replacement interval of 1.5 minutes for the 22 liter (6 gal) system.

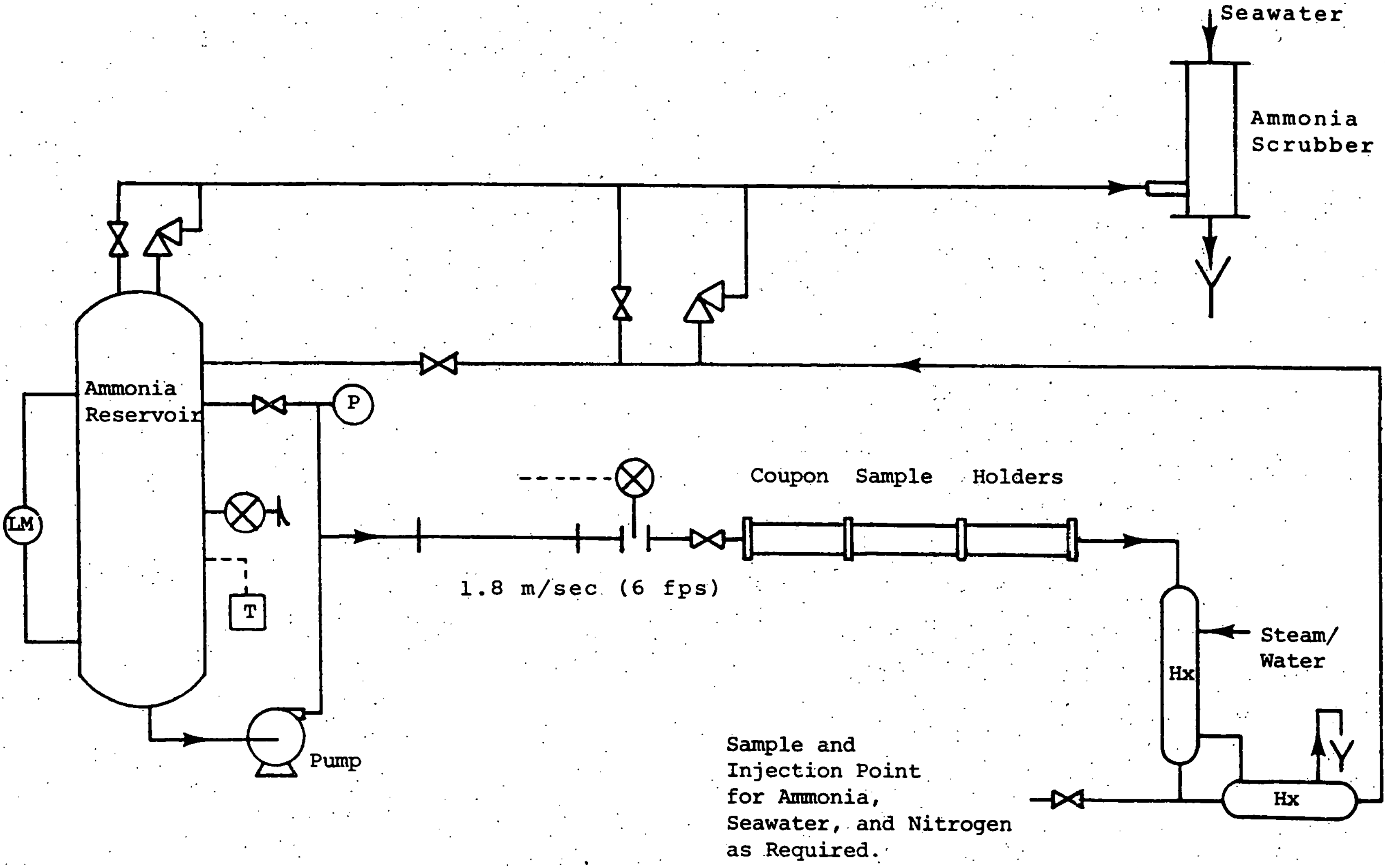
Construction materials consisted of plastic-lined steel (polypropylene, Saran, Teflon, and Kynar®) and Alloy 20 stainless steel. Temperature control was maintained by recirculating seawater through an Alloy 20 heat exchanger coupled to steam and river water inlets. Specimens were mounted using galvanically insulating nylon Swagelok fittings and Teflon plastic coupon holders. Seawater entering the units was filtered of all particles larger than 25 μm (0.001 in). Aqueous ammonia injected into the seawater was technical grade (29% NH_3) ammonia hydroxide.

Specimens were removed after 0.5, 1.5, and 3.0 months exposure to each level of ammonia concentration. A summary of the test unit operation is given in Appendix A, Tables VIII-5, VIII-8, and VIII-11.

3. Anhydrous Ammonia Test Unit

This unit was built for operation with high-pressure liquid anhydrous ammonia containing small fractions of water. A schematic of this test unit is given in Figure III-5, with a photograph shown in Appendix B, Figure VIII-5. Exposure conditions were to 1.8 m/sec (6 fps) anhydrous ammonia and anhydrous ammonia plus 1% seawater, and testing was performed using 150 x 19 x 1.5 mm (6 x 0.75 x 0.06 in) coupons. Testing was performed sequentially, non-copper materials first then copper, to prevent undesireable copper ion contamination. The test loop contained a suitable pump, temperature detectors, flow control and monitoring, as well as all automatic controls for failsafe operation.

FIGURE III-5
SIMPLIFIED SCHEMATIC OF ANHYDROUS AMMONIA TEST UNIT



The test loop was designed for total recycle of 45.4 kg (100 lb) of anhydrous ammonia. Seawater, when used, was injected into the system's heat exchanger upstream of the pump and reservoir to allow mixing to occur before specimen contact. Analysis performed on the anhydrous ammonia-seawater mixtures included water content, chloride ion concentration, and sodium ion concentration.

Materials of test unit construction were welded type 304 stainless steel, Teflon plastic-lined steel, and Alloy 20 stainless steel. Temperature of the test solution was maintained at 30°C (86°F) by a steam-fresh water mixture. Coupons were mounted in Teflon plastic holders to prevent galvanic interaction. Anhydrous ammonia was 99.99% pure, and seawater was 35% ASTM standard artificial seawater.

Specimens were removed after 0.5 and 2.0 months exposure to each environment. A summary of the test unit operation is given in Appendix A, Tables VIII-16 and VIII-18.

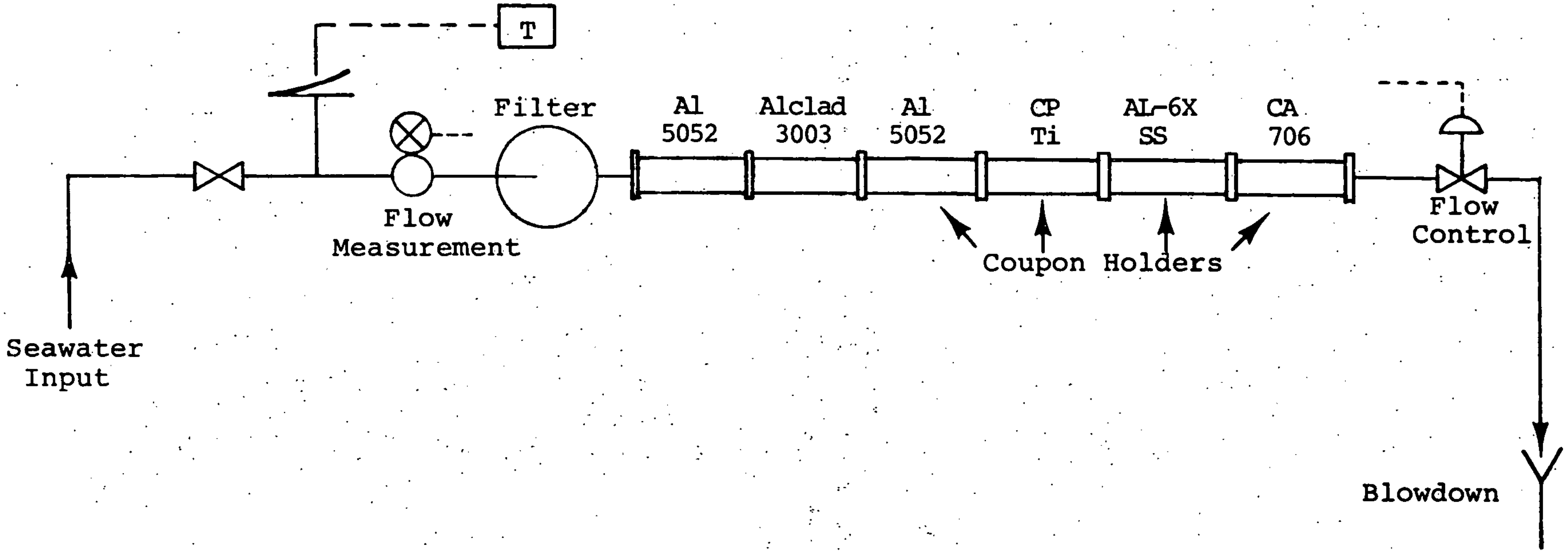
4. Once-Through Seawater Test Unit

This test unit was designed to expose 150 x 19 x 1.5 mm (6 x 0.75 x 0.06 in) coupons to once-through, 25 μm (0.001 in) filtered, 1.8 m/sec (6 fps), flow-controlled seawater. The unit is schematically depicted in Figure III-6, and a photograph is given in Appendix B, Figure VIII-6. Operation was such that CA 706 was downstream of the non-copper alloys, thus preventing copper ion contamination. Temperature control was not imposed on the as-received seawater, although temperature was monitored throughout all testing.

Initial alloys placed on test on February 13, 1979, were Al-5052, CA 706, AL-6X SS, and CP titanium. On April 27, 1979, Alclad alloy 3003 and added Al-5052 were placed upstream of the existing test. Specimens were removed after 0.5, 1.5, 3.0, 6.0, and 12.0 month seawater exposures.

FIGURE III-6

FLOWSHEET OF UNIT FOR CONTROL TESTING OF FLAT SPECIMENS IN AMBIENT TEMPERATURE FILTERED SEAWATER



5. Seawater Quality

Natural seawater utilized in these tests was from the Gulf of Mexico intake basin located at the Dow Chemical Texas Division industrial site "A" at Freeport, Texas. After large incoming debris was screened out at the Dow intake basin, the seawater flowed through flumes (canals) to a second smaller sedimentation basin near the OTEC test facility. The seawater was then transferred by an Alloy 20 stainless steel pump through a system of copper-free piping to the test units. The composition and range of the seawater used in these tests appears in Table III-2.

The variation of seawater temperature for selected years is shown in Figure III-7. Included on this figure are temperatures for the 1979-1980 test program.

C. Specimen Procedure

In the course of tests, specimens of seven alloys were exposed to determine their corrosion and fouling behavior.

1. Test Alloys

Alloys specified for use in these tests included aluminum alloys 5052 and Alclad 3003, copper alloys 706, 715, and 722, AL-6X stainless steel, and CP titanium. Table III-3 presents the composition of the test alloys.

Of the alloys tested, the only bimetallic material placed on test was aluminum Alclad alloy 3003. Base metal for this material was Al-3003, and the sacrificial cladding consisted of a thin layer of Al-7072. In tube form, cladding was internal only and was 13% of the tube wall thickness. Coupons of this material were clad on both sides and each cladding layer represented 6% of the total thickness. Figures VIII-10 and VIII-14 in Appendix A are metallographic cross-sections of tube and coupon specimens etched with Keller's reagent to optically separate the two aluminum alloys.

TABLE III-2
SEAWATER COMPOSITION AND RANGE GULF OF MEXICO

Annual Average at Freeport, Texas

Measured Parameters	Range, mg/l	Typical Value, Annual Average, mg/l
Alkalinity as $CaCO_3$	75 - 125	100
Aluminum as Al	--	0.5
Ammonium ion as NH_4^+	0.01 - 0.1	00.05
Calcium as Ca	240 - 400	340
Carbon Dioxide (free) as CO_2	2 - 5	3
Chloride as Cl	11,700 - 19,400	16,500
Copper as Cu (total)	0.01 - 0.1*	0.01
Hydrogen Sulfide as H_2S	--	<0.01
Iron as Fe (total)	0.05 - 5.0*	2
Magnesium as Mg	780 - 1,300	1,100
Mercury as Hg	--	<0.0001
Oxygen, dissolved, as O_2	1.5 - 6.0	5
pH	7.5 - 8.6	8.4
Potassium as K	230 - 380	325
Salinity, % "Normal"	60 - 100%	85%
Sodium as Na	6,340 - 10,560	9,000
Sulfate as SO_4	1,800 - 3,000	2,600
Suspended solids	5 - 50	20
Temperature, °F	60 - 80	70 - 75

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* Varies with suspended solids present.

NOTE: When specific gravity = 1, mg/l = ppm.

FIGURE III-7

INCOMING SEAWATER TEMPERATURE AT
DOW CHEMICAL COMPANY, FREEPORT, TEXAS PLANT

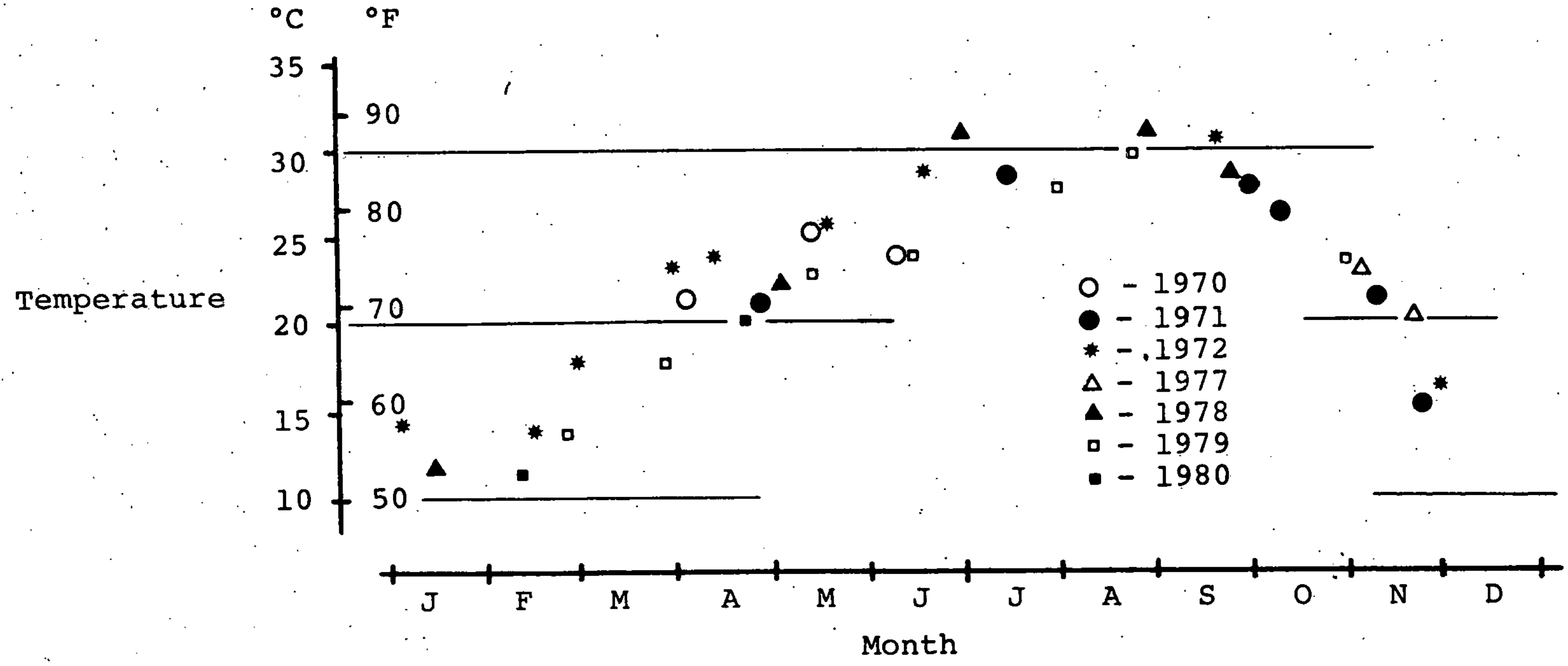


TABLE III-3

NOMINAL ALLOY COMPOSITION

	B.W.G., Table Guage	Thickness mm
Aluminum Alloy		
3003: Al, 1.2% Mn	16	1.7
5052: Al, 0.25% Cr, 2.5% Mg	18	1.2
7072: Al, 1% Zn	-	
Copper Alloy		
706: Cu, 1.4% Fe, 10% Ni	18	1.2
715: Cu, 0.5% Fe, 31% Ni	18	1.2
722: Cu, 0.5% Cr, 0.7% Fe, 16.5% Ni	18	1.2
Stainless		
Allegheny Ludlum-6X: Fe, 20.1% Cr, 24.6% Ni, 1.4% Mn, 6.3% Mo	22	0.7
Titanium		
CP: Commercially Pure ASTM Grade 2 Spec/ B265	20	0.9

2. Specimen Preparation

Specimens placed on test consisted of 25.4 mm (1 in) outside diameter tubes and 150 x 19 x 1.5 mm (6 x 0.75 x 0.06 in) flat coupons.

Tubes placed on test were first cut to length using a wheel-type pipe cutter. Tubes for test were then prepared by drilling 0.1 mm (0.004 in) and 0.2 mm (0.008 in) "leak" sites in the wall 1.5 m (5 ft) from a tube end. Drilling of the artificial leaks required that a larger drill be used first because of the short length available with the small sized drills. Examples of the leak site cross-section are presented in Figure III-8. Tubes were then degreased using Chlorothene®NU solvent prior to exposure. The shorter 0.61 m specimens exposed to renewed recirculating seawater plus small concentrations of ammonia were identified on the exterior center using an electric scribe. Longer tube specimens were marked with permanent black ink.

Coupons were also employed in the test program in order to get a second estimate on general corrosion rates in the test environment. The coupons, as prepared by Corrosion Test Supplies Company, all had a buffed surface finish with all edges rounded and free of burrs. For identification, a two-digit number was stamped into one side of the center face of each coupon. All coupons were degreased using Chlorothene NU solvent, dryed, weighed and stored in a desiccator until used. A photograph of the holder used to expose flat metal specimens is shown in Figure III-9.

Coupons of Alclad alloy 3003 posed a special problem since exposure of the Al-3003 base metal at the coupon edge could accelerate edge attack of the Al-7072 cladding and produce misleading weight losses and corrosion attack. All Alclad coupons (in the seawater exposure) therefore received a thin edge coating of insulating Microshield® stop-off lacquer after weighing. Numbering was accomplished by putting indentations in the coupon edge prior to coating with the lacquer.

Arrangement of specimens varied with test unit and type of exposure. Tubes in the long tube unit were arranged in an alternating, random-like pattern, with non-copper and copper tubes on respective sides of the ammonia test spool. Specimens placed on exposure in sequentially used test units, as the seawater plus ammonia and anhydrous ammonia test units, were inserted by groups according to time of exposure with upstream specimens scheduled for earlier removal. For exposures to once-through, ambient temperature seawater, the need to place copper alloys downstream in the test loop resulted in separation of the test specimens by alloy.

Upon removal from the test units, specimens were first rinsed and then dried for 24 hours. Long tubes were then split, and half of each tube then acid cleaned for corrosion analysis. Tubes and coupons from the remaining seawater-ammonia test units were first weighed with the fouling films, acid cleaned, solvent degreased, and then reweighed clean. Chlorothene NU was used as a degreasing agent for all except coupons of Alclad alloy 3003, for which acetone was employed to remove the Microshield lacquer as well as water and hand-grease films. These shorter tubes were then split, and observations were made of all surfaces. Additionally, fouling control coupons receiving identical exposure as the subject test specimens were withdrawn and subjected to chemical and biological film analysis. Acid cleaning solutions employed and exposure times are shown below.

Alloy	Solution	Time
Aluminum alloys	2% chromic acid plus 5% phosphoric acid 80°C (176°F)	30 min
Copper alloys	1:1 hydrochloric acid plus 0.1% Dowell®A-120 inhibitor, room temp.	1 min
AL-6X SS and CP titanium	10% nitric acid 60°C (140°F)	10 min

FIGURE III-8

Cross-Section of Artificial Leak Sites
Before Exposure

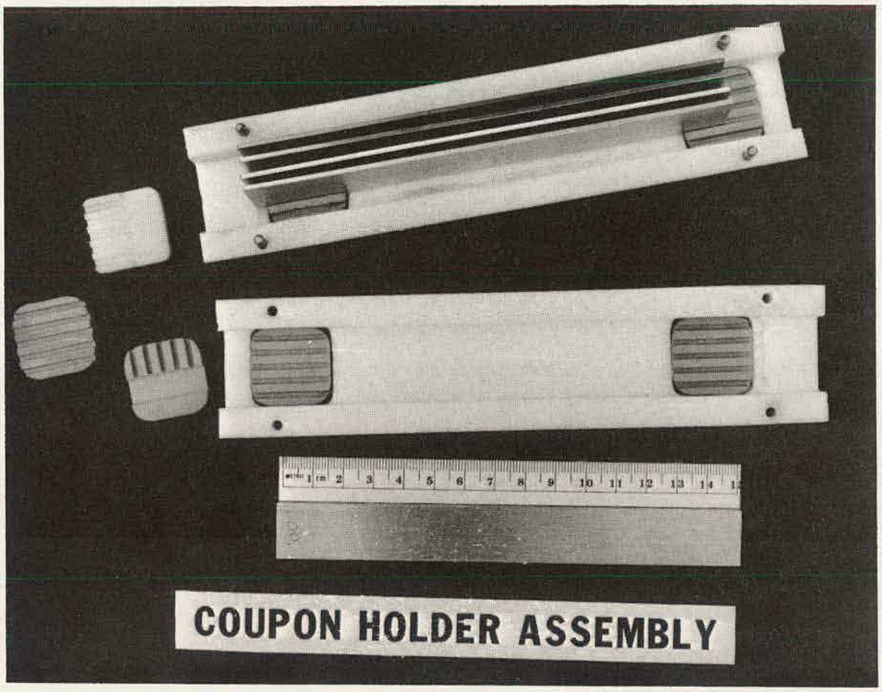


FIGURE III-9

Teflon Coupon Specimen Holder
(Scale in cm)

3. Data Resolution

Weight loss and observations were resolved into penetration, maximum pit depth, and approximate pit density.

In order to facilitate data usage by the OTEC community, corrosion was expressed and plotted as corrosion penetration for the specific exposure. This was calculated using the following equation:

$$(\text{Metal Penetration}) = \frac{(\text{Specimen Weight Loss})}{(\text{Specimen Area})(\text{Metal Density})}$$

Data are commonly expressed in terms of mm (mils) penetration.

In order to assist the reader, a Metric/English Corrosion Nomograph is presented in Figure III-10. Average areas of specimens used in these tests were 447 cm^2 (69 in^2) for the 0.61 m long tubes, 64.5 cm^2 (10 in^2) for bare coupons, and 49 cm^2 (7.6 in^2) for the lacquered Alclad alloy 3003 coupons. Densities used in performing the calcuations were as follows:

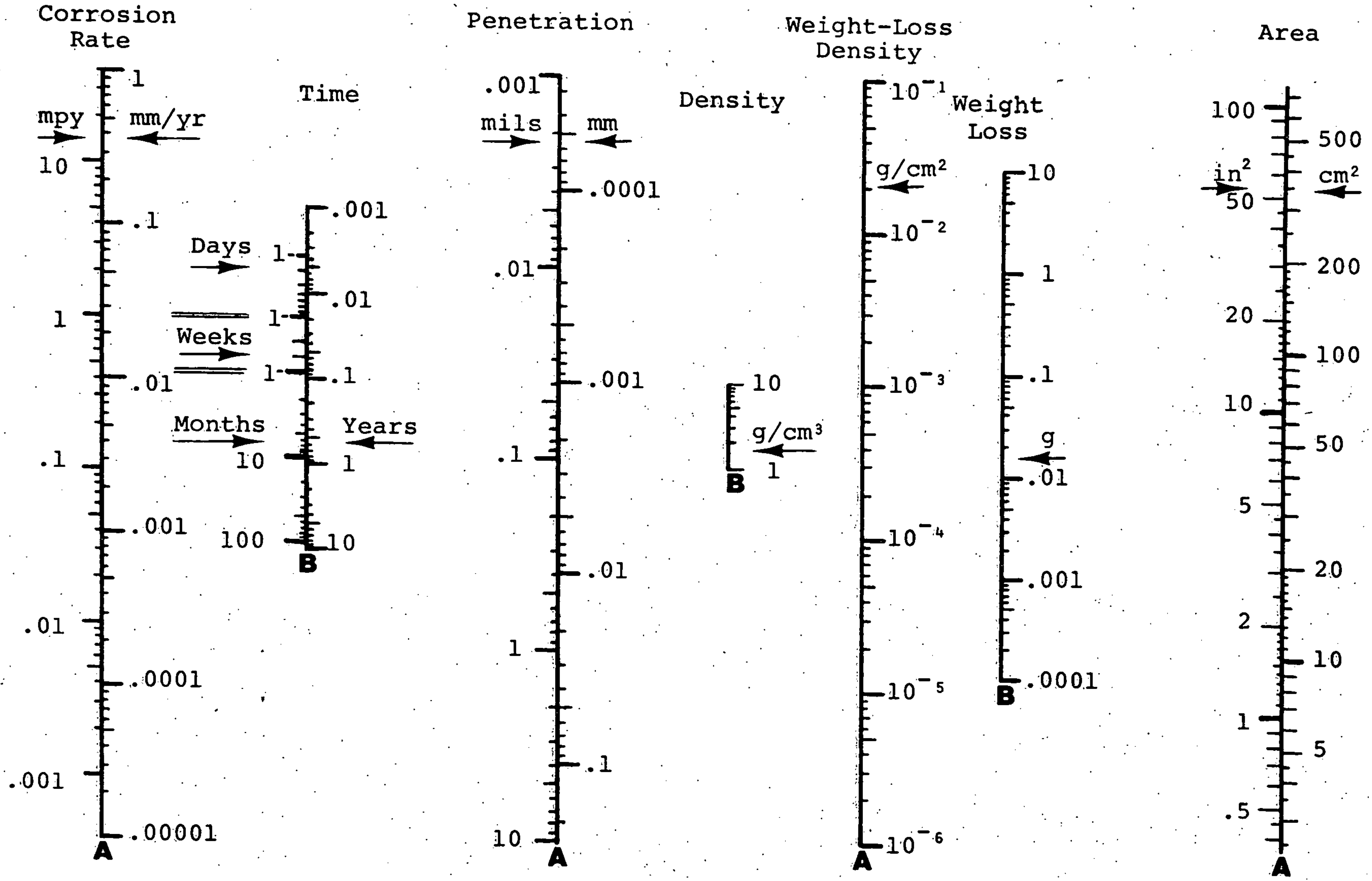
Material	Density (g/cm^3)
Al-5052	2.70
Al-7072	2.73
CA-706	8.93
AL-6X SS	8.02
CP Titanium	4.54

Localized corrosion was assessed using the following general format:

Small pits:	Pits 0.025-0.01 mm deep, discernable as pit under 10X magnification and visible to naked eye.
Pits:	Pits 0.025-0.05 mm deep (unless otherwise indicated), readily visible as pits to naked eye.
Very few:	Less than 1/sq cm
Few:	1-2/sq cm
Several:	2-3/sq cm
Numerous:	More than 3/sq cm

FIGURE III-10

METRIC/ENGLISH CORROSION NOMOGRAPH



Perform calculations by drawing a straight line through values on adjacent lines A-B-A

This format was developed to assess observations from the limited surface area exposed. When feasible, each specimen was examined under 10x magnification to generate the required data. For those pits greater than 0.05 mm (2 mils) deep, an Ames depth gauge (No. 11B-202P) was employed to measure pit depth.

IV. CORROSION

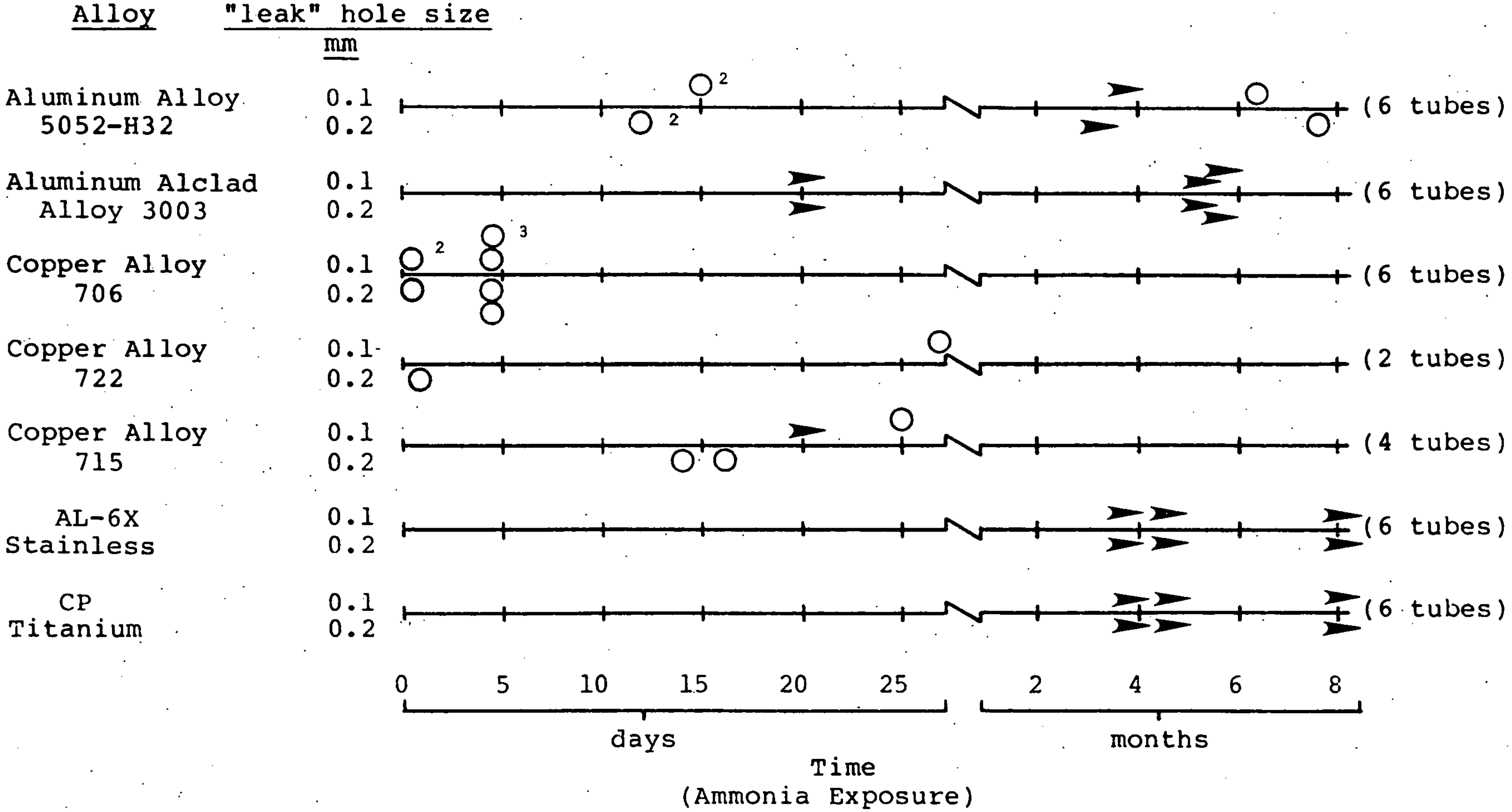
The heat exchangers of proposed OTEC systems may involve formation of ammonia-seawater environments for which materials performance data is limited. Research on those candidate materials believed to have specific performance or economic advantage in OTEC applications was desireable. Results of corrosion tests were separated into four basic environmental exposures as follows:

A. Leak Test

Candidate alloy tubes 6.1 m (20 ft) long were exposed in the tube test unit to 1.8 m/sec (6 fps), once-through, unfiltered 30°C (86°F) seawater to which anhydrous ammonia was added through 0.1 mm (0.004 in) and 0.2 mm (0.008 in) drilled "leak" holes, 1.5 m (5 ft) downstream of the tube end. The tube test unit commenced operation April 25, 1979, with four tubes each of alloys Al-5052, CA 706, AL-6X stainless steel, and CP titanium. On August 9, 1979, aluminum Alclad alloy 3003 and CA 715 arrived and were introduced into the tube test unit; and on November 6, 1979, CA 722 was placed on test. Tube corrosion performance summaries are given by Figure IV-1 and Tables VIII-1 and VIII-2, in Appendix A.

The initial four candidate alloy tubes were exposed to 30°C (86°F) seawater at 1.8 m/sec (6 fps) and a positive nitrogen pressure to permit tube surfaces to recieve a seawater contact history. After over three weeks exposure, severe leaks were detected in CA 706 tubes in which the 0.1 and 0.2 mm (0.004 and 0.008 in) diameter holes had enlarged to as much as 3.0 mm (0.12 in) in diameter. These enlarged holes in CA 706 were sealed, and new leak sites were drilled prior to ammonia exposure.

FIGURE IV-1
TUBE TEST UNIT: TUBE PERFORMANCE [1]



○: Tube removed due to unacceptable leak hole enlargement.

➤: No failure. Scheduled tube removal.

[1] Ammonia pressure was 9.8 kg/cm^2. Average seawater pressure was 9.1 kg/cm^2 unless otherwise stated.

[2] Seawater pressure was 8.4 kg/cm^2.

[3] Seawater pressure was 9.5 kg/cm^2.

Upon reinsertion of CA 706 tubes, testing was begun using anhydrous ammonia at 9.8 kg/cm^2 (140 psi) and seawater at 9.4 kg/cm^2 (135 psi). The system was in operation for 4.5 days before very high levels of ammonia were detected in the copper tube seawater flow streams. Inspection revealed that all four CA 706 tubes on test had experienced severe leak site corrosion. These four tubes were thus removed, and the remaining two CA 706 tubes, without seawater conditioning, were placed on test. Since previous operation with a 0.4 kg/cm^2 (5 psi) tube wall pressure differential resulted in scale plugging of all the artificial leak sites, restart occurred at a seawater pressure of 8.4 kg/cm^2 (120 psi), or a 1.4 kg/cm^2 (20 psi) differential pressure. Within eight hours of startup at the increased pressure differential, the remaining CA 706 tubes failed because of corrosion at the artificial leak site. Further testing of CA 706 in this test unit was stopped. Photomicrographs of cross-sections of two of the CA 706 tube failure leak sites appear in Appendix B, Figures VIII-7 and VIII-8.

After failure and removal of the final CA 706 tubes, the test was continued using the three non-copper alloys. The system was in operation 6 more days at a seawater pressure of 8.4 kg/cm^2 (120 psi) when analysis of the ammonia concentration in the once-through seawater indicated imminent failure on the part of an Al-5052 tube (0.2 mm, 0.008 in leak). The tube wall pressure differential was reduced from 1.4 kg/cm^2 (20 psi) to 0.7 kg/cm^2 (10 psi) in an attempt to encourage scaling and plugging of the enlarging leak; however, corrosion continued and failure occurred within 24 hours. A photomicrograph of the cross-sections of this leak site appears in Appendix B, Figure VIII-9. After the aluminum alloy 5052 tube was removed and replaced, the tube test unit was returned to operation. Four days later, a second Al-5052 tube (0.1 mm, 0.004 in leak) failed after a similar attempt to plug the leak site. After replacement of this Al-5052 tube, the system returned to operational status at a seawater pressure of 9.1 kg/cm^2 (130 psi).

Test unit operation continued until August 9, 1979, when Alclad alloy 3003 and CA 715 were introduced to the test unit. Operation resumed for 14 days until failure of CA 715 tube (0.2 mm, 0.008 in leak). Two days after operation again resumed, a second CA 715 tube, also possessing the larger leak drilling, failed because of unacceptable leak hole enlargement. No further failures of this alloy were observed until the 4.5 month pull was completed September 10, 1979. At the scheduled tube removal, half the candidate alloy tubes remaining on test were removed for inspection, including one of the two remaining CA 715 tubes. A photomicrograph of the leak site cross-section of one of the removed Alclad alloy 3003 tubes is shown in Appendix B, Figure VIII-10. The remaining (0.1 mm, 0.004 in leak site) CA 715 tube continued on test until September 21, 1979, when it too experienced unacceptable leak enlargement.

The last copper alloy tested, CA 722, was placed on test November 6, 1979. Of the two tubes placed on test, one experienced failure after 20 hours of exposure (0.2 mm, 0.008 in leak), and the second tube (0.1 mm, 0.004 in leak) performed for 27 days before failure because of unacceptable leak enlargement. This concluded testing of copper alloys in the tube test unit. The alloys remaining on test, each having survived 4 months exposure, included two tubes of Al-5052 and four tubes each of Alclad alloy 3003, AL-6X stainless steel, and CP titanium.

Test unit operation continued as routine until just before final test unit shutdown on January 22, 1980 when the remaining two Al-5052 tubes on test experienced leak site corrosion failure. Previously, both of these tubes had experienced perforation; however, the resulting seawater leakage could be sealed, and this type of failure did not result in shutdown of the tube unit. Of the alloys on test, Al-5052 tubes were the only tubes to experience perforation at other than the artificial leak site. Tube materials which survived until the final tube unit shutdown included Alclad alloy 3003, AL-6X stainless steel, and CP titanium.

B. Seawater Plus Ammonia

Both tube and coupon specimens of the candidate alloys were exposed in the seawater plus ammonia test units. This environment consisted of 30°C (86°F), 1.8 m/sec (6 fps), filtered, refreshed, recirculating seawater plus 5, 10, and 20 ppm ammonia. Corrosion data for the alloys are given in Figures IV-2, IV-3, and IV-4, and in Appendix A, Tables VIII-3, VIII-4, VIII-6, VIII-7, VIII-9, and VIII-10. A summary of the observations is given by each level of ammonia concentration.

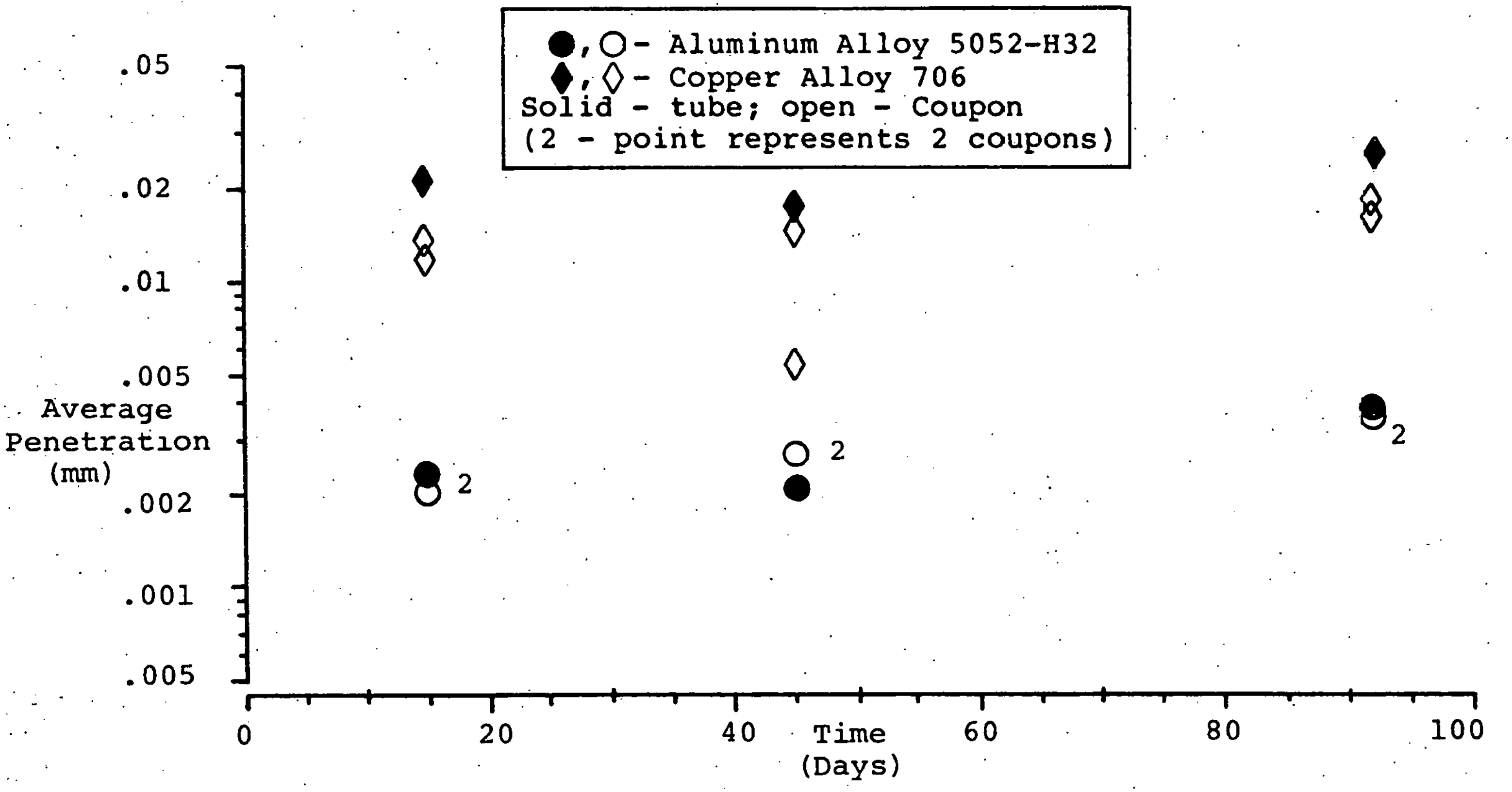
Tests employing 5 ppm ammonia additions to recirculating seawater commenced March 8, 1979, and were completed after 15, 45, and 92 day specimen removals. All alloys exhibited corrosion penetrations comparable to those exposed in seawater. However, Al-5052 tubes pitted severely at this low ammonia concentration and CA 706 coupons experienced severe crevice corrosion. A photomicrograph of a pit cross-section on the Al-5052 tube and a photograph of CA 706 coupon crevice corrosion appear in Appendix B, Figures VIII-12 and VIII-13. Performance of AL-6X stainless steel and CP titanium were excellent in this environment.

Testing at a concentration of 10 ppm ammonia in seawater was carried through exposures of 16, 48 and 98 days. As observed at the 5 ppm ammonia addition, all alloys exhibited average corrosion penetrations at or below comparable seawater exposures. During this exposure, however, both tubes and coupons of Al-5052 experienced significant pit attack. CA 706 specimens experienced mild pit and crevice attack at approximately half the severity encountered at the 5 ppm ammonia concentration. AL-6X stainless steel and CP titanium exhibited overall superior performance.

Specimen examinations for the 20 ppm ammonia concentration in seawater occurred after 21, 48, and 98 days of exposure. Heavy scaling occurred on all specimens at this exposure which may account for most of the average corrosion penetrations being below that of comparative seawater tests. Localized pit attack was minimal on Al-5052. CA 706 experienced crevice attack comparable to the 10 ppm ammonia plus seawater exposure. AL-6X stainless steel and CP titanium continued to exhibit excellent performance.

FIGURE IV-2

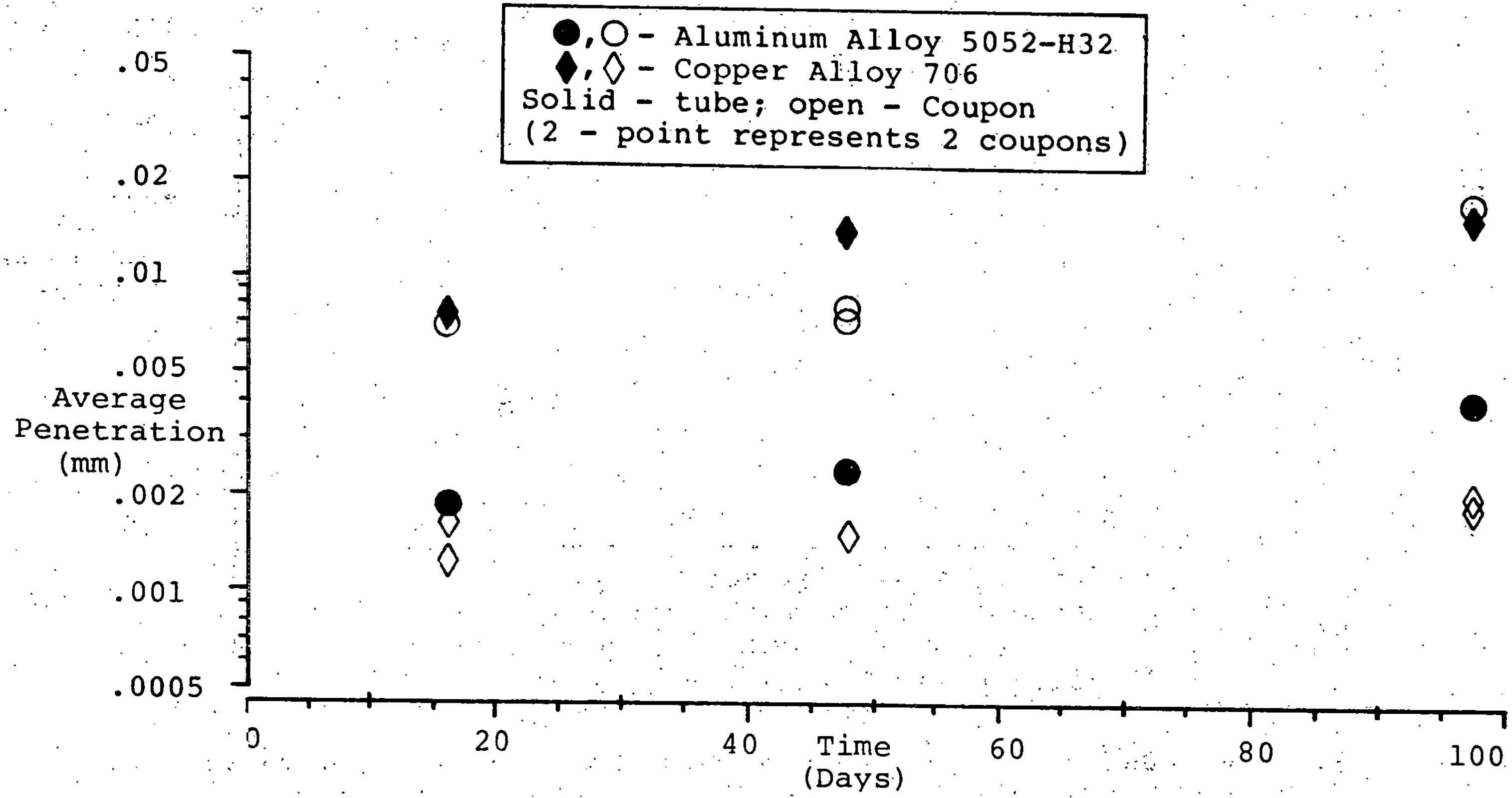
CORROSION PERFORMANCE OF OTEC CANDIDATE MATERIALS
EXPOSED TO RECIRCULATING SEAWATER PLUS NOMINAL 5 PPM AMMONIA*



* Corrosion penetration for AL-6X stainless steel and CP titaniun was less than 0.0005 mm.

FIGURE IV-3

CORROSION PERFORMANCE OF OTEC CANDIDATE MATERIALS
EXPOSED TO RECIRCULATING SEAWATER PLUS NOMINAL 10 PPM AMMONIA*



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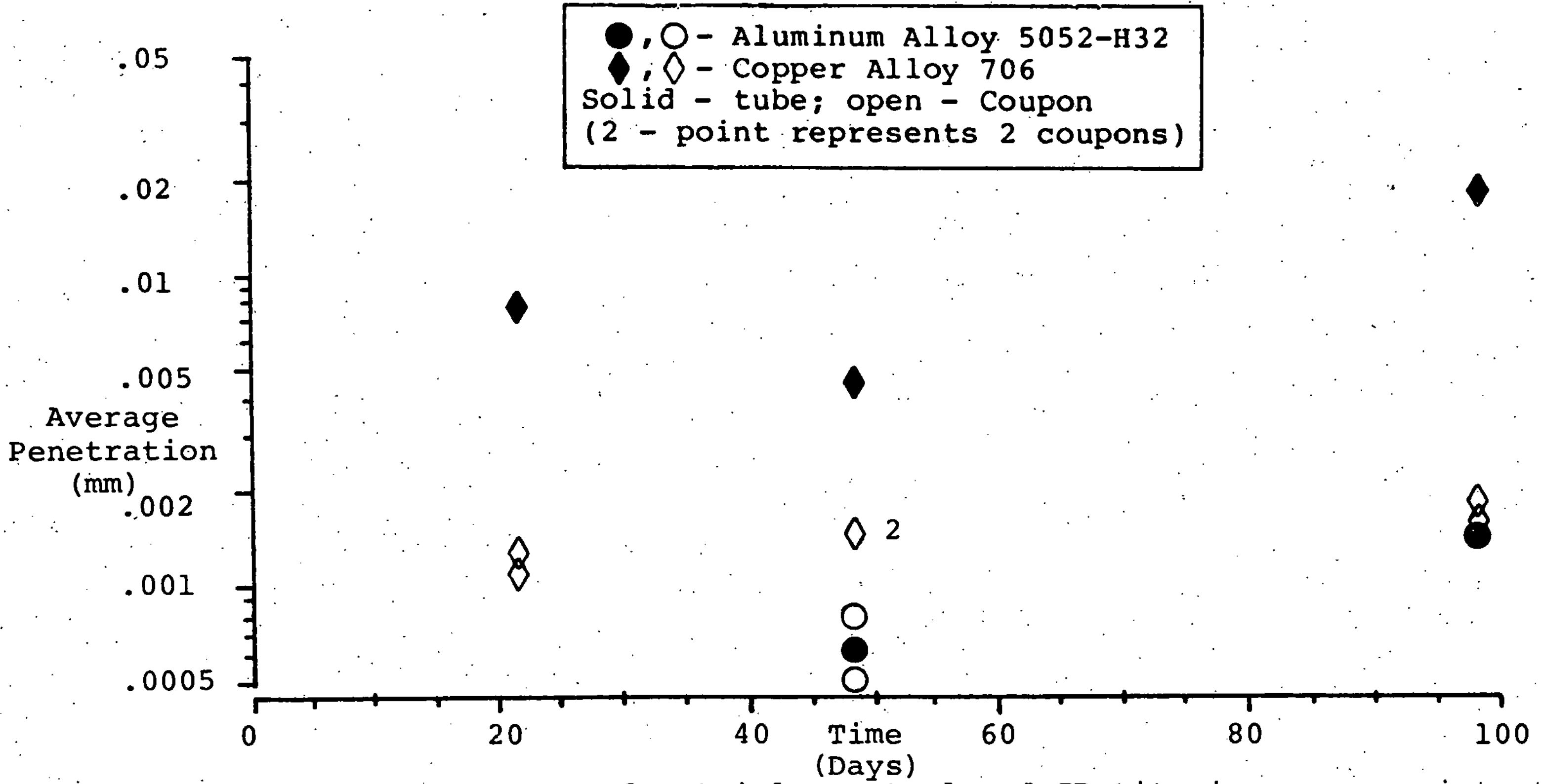
* Corrosion penetration for AL-6X stainless steel and CP titanium was less than 0.0005 mm.

FIGURE IV-4

CORROSION PERFORMANCE OF OTEC CANDIDATE MATERIALS
EXPOSED TO RECIRCULATING SEAWATER PLUS NOMINAL 20 PPM AMMONIA*



* Corrosion penetration for AL-6X stainless steel and CP titanium was consistently less than 0.0005 mm. Corrosion penetration of Al-5052 was below 0.0005 mm intermittently.

C. Anhydrous Ammonia

The anhydrous ammonia unit was designed to evaluate flat specimens in 30°C (86°F), 1.8 m/sec (6 fps) recirculating anhydrous ammonia plus 0.0 and 1% seawater additions. Corrosion data for these exposures are summarized in Figure IV-5 and in Appendix A, Tables VIII-15 and VIII-17.

Tests conducted using anhydrous ammonia were completed after 17 and 61 days exposure for the non-copper alloys and after 15 and 69 days exposure for CA 706. Average corrosion penetrations were acceptable for the four alloys tested with no significant localized attack.

Investigations using 1% seawater in anhydrous ammonia, employed specimen pulls at 14 and 60 days for the non-copper test and at 14 and 65 days exposure for CA 706. As shown in the case above, no significant localized attack occurred although average corrosion penetrations on Alclad alloy 3003 were severe. The cladding alloy, Al-7072 evidently has a very low resistance to this environment as attested to by the fact that 88% of the 0.1 mm (0.004 in) layer was depleted within 60 days. The base alloy did not experience severe attack. This is in agreement with previous tests using Al-3003 which showed satisfactory performance in comparable exposures. All remaining alloys tested experienced acceptable average corrosion penetrations.

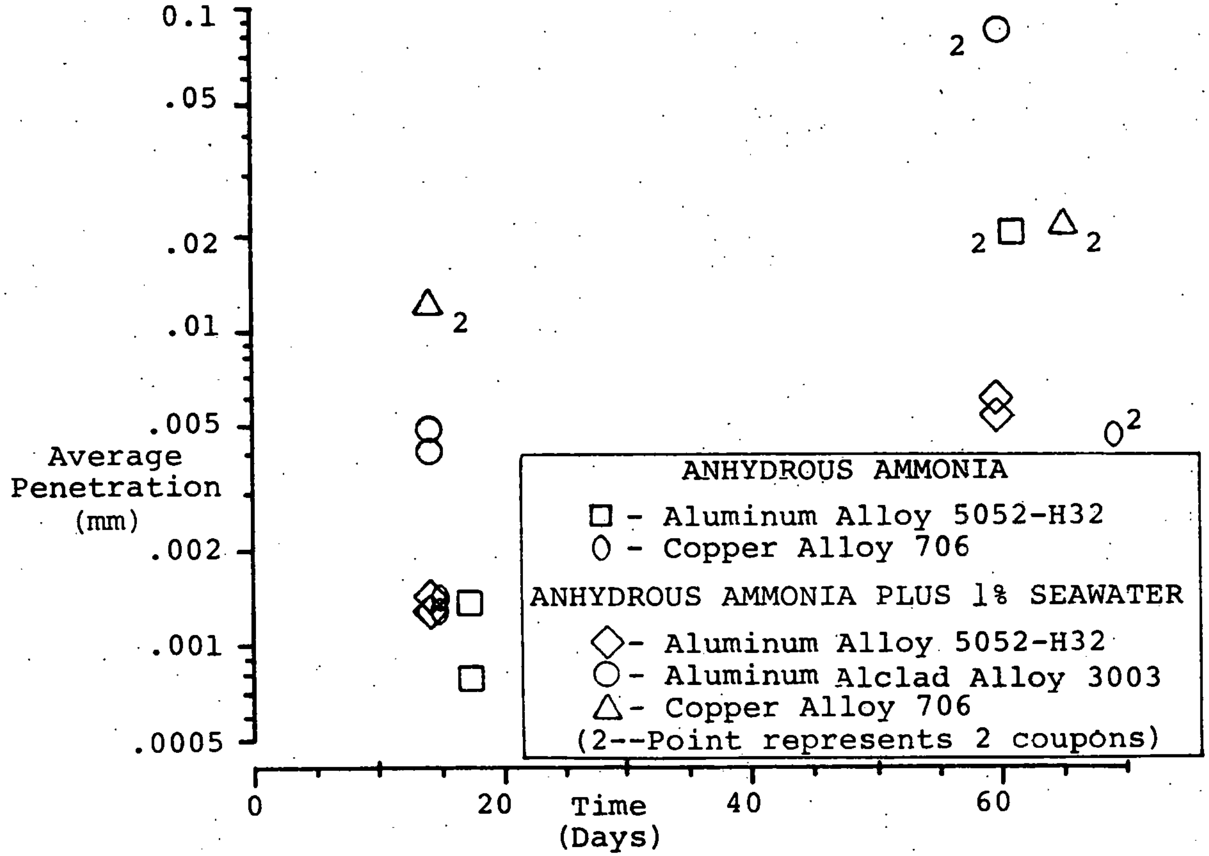
D. Seawater Control Specimens

Comparative data denoted as the seawater controls is based on coupon exposures to 1.8 m/sec (6 fps), ambient temperature, filtered, once-through seawater. Results of these tests are summarized on Figure IV-6, IV-7, and in Appendix A, Tables VIII-21 and VIII-22.

Observations of all specimens failed to find any significant localized corrosion attack on the subject alloys. Corrosion observed was consistently uniform, with average corrosion rates decreasing with time for the aluminum and copper alloys. AL-6X stainless steel and CP titanium showed no discernable corrosion, and for these two alloys, there was no readily observable difference between exposed coupons and coupons not placed on test.

FIGURE IV-5

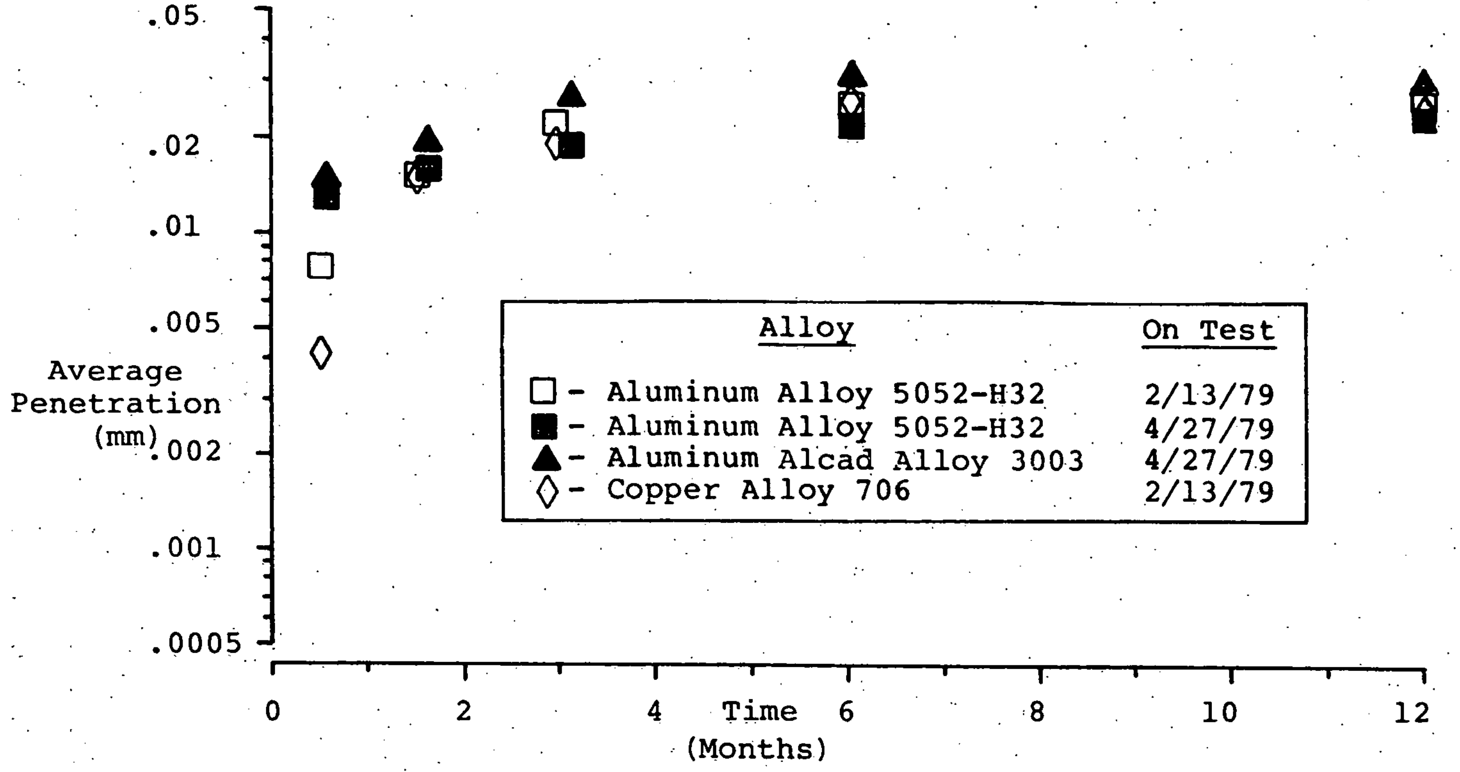
CORROSION PERFORMANCE OF OTEC CANDIDATE MATERIALS EXPOSED TO ANHYDROUS AMMONIA AND ANHYDROUS AMMONIA PLUS 1% SEAWATER*



* Corrosion penetration for AL-6X stainless steel and CP titanium was less than 0.0005 mm.

FIGURE IV-6

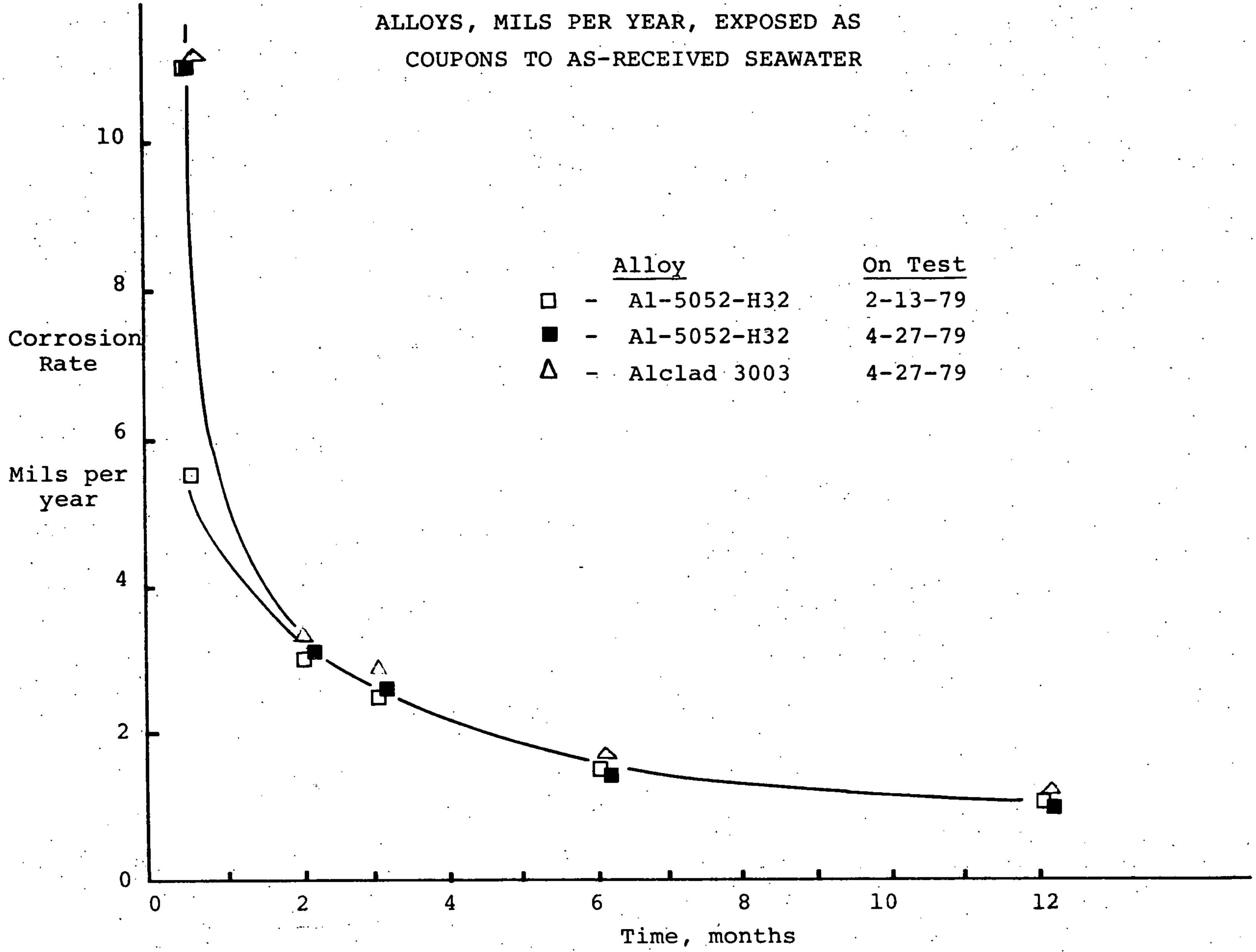
CORROSION PERFORMANCE OF OTEC CANDIDATE MATERIALS
EXPOSED AS COUPONS TO AS-RECEIVED SEAWATER*



* Corrosion penetration for AL-6X stainless steel and CP titanium was less than 0.005 mm.

FIGURE IV-7

CORROSION RATE OF CANDIDATE ALUMINUM ALLOYS, MILS PER YEAR, EXPOSED AS COUPONS TO AS-RECEIVED SEAWATER



V. FOULING

Heat transfer may be seriously impaired by the formation of heavy fouling films on heat exchanger surfaces. Normally, these films include corrosion products, sediments, and fouling organisms. Interleakage of seawater and ammonia can precipitate additional scaling films onto the heat transfer surface.

A. Seawater Plus Ammonia

Seawater fouling consists of both inorganic sediments and scales and biofouling organisms. Investigations were made into the effects of ammonia leakage into seawater on both of these fouling mechanisms.

1. Inorganic Deposits

Mixing of ammonia and water will result in hydrolysis of the ammonia to form ammonia and hydroxyl ions. The resulting solution is thus more alkaline, and in seawater, an increase in alkalinity enhances calcium carbonate and magnesium hydroxide precipitate formation. Precipitates so formed may then nucleate and form scales on heat exchanger surfaces in a manner which will seriously impair heat transfer.

This set of problems is due to the nature of the hydrolysis of ammonia and to the formation of hydroxyl ion:

$$NH_3 + HOH \rightarrow NH_4^+ + OH^-$$

As a result of the hydroxyl ion formation, solutions of ammonia in water are very basic. Then, the hydroxyl ion in seawater reacts with bicarbonate (the predominant ionic form of carbon dioxide) to produce carbonate ion:

$$HCO_3^- + OH^- \rightarrow CO_3^= + HOH$$

which in turn will react with calcium present to precipitate calcium carbonate:

$$Ca^{++} + CO_3^= \rightarrow CaCO_3$$

Or, alternately, the hydroxyl ion produced by the hydrolysis of ammonia can react directly with the magnesium present in seawater to precipitate magnesium hydroxide:

$$Mg^{++} + 2OH^- \rightarrow Mg(OH)_2$$

In general, if sufficient ammonia is present, the sequence of chemical events would be:

1. Conversion of HCO_3^- to $CO_3^=$
2. Precipitation of $CaCO_3$
3. Precipitation of $Mg(OH)_2$
4. Elevation of solution pH

Ammonia additions to seawater occurred in the tube test unit and seawater plus ammonia test units. Data from the tube test unit is qualitative and presents a more accurate portrayal of actual leak site behavior. Data derived from the seawater plus ammonia test loops is quantitative and represents scale formation at specific ammonia concentrations.

The scale formation in the tube test unit was adjacent to and downstream of the artificial leak. Typical scale formation is shown on the Alclad alloy 3003 photomicrograph in Appendix B, Figure VIII-10 and the scale formation photograph in Appendix B, Figure VIII-11. On the photomicrograph, note the scale formed directly over the artificial leak effectively plugging the site to significant flows of ammonia. The photograph shows a typical scale "plume" on the tubes as formed around and downstream of the leak sites. These plumes of scale ranged in length from 2 to 75 mm (0.08-2.95 in) for non-failing leaks and had an average length of approximately 25 mm (1 in). Thickness of the scale plumes averaged less than 0.5 mm (0.02 in).

Leak site closing was a common occurrence in the tube test unit. Tube test unit operation at a 0.4 kg/cm^2 (5 psi) anhydrous ammonia-seawater pressure differential resulted in plugging and scaling of all of the initial artificial leak sites. These scales were removed, but subsequent operations at a pressure differential of 0.7 kg/cm^2 (10 psi) were also highly effective at scaling the artificial leaks, and even at pressure differentials of 1.4 kg/cm^2 (20 psi), plugging of the artificial leak continued to occur. Corrosion failure at the leak sites resulted in massive introduction of anhydrous ammonia into the seawater stream and very extensive scaling immediately downstream of the leak site. It may be projected that continued operation of a heat exchanger under such conditions would eventually result in complete plugging of the affected tube, creating a plug seal over the much larger leak.

Tests performed in the seawater plus ammonia test loops were at the controlled concentrations of 5, 10, and 20 ppm ammonia. Data for these tests is given in Appendix A, Tables VIII-12, VIII-13, and VIII-14.

As a general observation, particularly with the non-copper alloys, fouling weight density tended to increase with test duration and increased ammonia concentration. Changes were also observed in intake water sediment load. Coupon data represents identical exposure conditions per duration of exposure. Tube data for CA 706 is not identical as upstream tubes more sensitive to sediment deposition were removed earlier. Scale formation followed the expected pattern of development with amorphous sediment deposits forming the majority of the fouling deposit at low concentrations of ammonia and calcite ($CaCO_3$) followed by aragonite ($CaCO_3$) scale formation as ammonia levels increased.

2. Biological Activity

A microbiological analysis was performed on samples removed from the seawater plus ammonia test loop. Samples consisted of filtered seawater and surface deposits from the test coupons.

On completion of the final test, at a concentration of 20 ppm ammonia in seawater, a dilution procedure was used to estimate the concentration of micro-organisms within the system. Up to approximately 10^3 terrestrial bacteria per milliliter and 8×10^4 marine bacteria per milliliter of filtered seawater were observed in the recirculation system. Observations on populations at a lower nitrogen (ammonia) concentration showed lower viabilities. At only the lowest ammonia concentration were differences noted between coupon bacterial concentrations when CA 706 bacterial populations were noted to be slightly less active than their counterparts on other alloys.

Isolation of species was performed by innoculating different nutrient media and incubating the culture dishes for 1-21 days. Using this technique, up to 9 terrestial slime bacteria, up to 7 terrestial colony bacterial, and one terrestial iron-reducing bacteria were isolated. Slime bacteria can grow in layers which would increase film resistance to heat transfer. Colony bacteria tend to grow in discreet colonies capable of forming localized sediment traps important to initiation of localized corrosion. The iron reducing bacteria, isolated from AL-6X stainless steel, is capable, over a long period, of causing pitting on some metal surfaces. Two fungi, facultative anaerobles, were also isolated from the system where their growth could have increased film resistance to heat transfer. The three marine bacteria isolated were found only in the filtered seawater and exhibited no affinity for any of the metal coupons. At no time were marine macrofouling organisms, barnacles, bryozoans, hydroids, or serpulids, observed in the filtered seawater system.

B. Ammonia Plus Seawater

Fouling and scaling, as they occurred in anhydrous ammonia and anhydrous ammonia plus seawater, were limited to inorganic film deposits. Investigations into this phenomenon were limited to tests in the anhydrous ammonia test unit since higher anhydrous ammonia pressures in the tube test unit prevented significant seawater into ammonia scale formation.

On entry of seawater into anhydrous ammonia, hydrolysis of ammonia can occur, with immediate precipitation from the seawater of calcium carbonate and magnesium hydroxide, just as in the case of ammonia injection into seawater. As the remaining salt water is abstracted into ammonia during the hydrolysis reaction, not only will the weak base, ammonium hydroxide form, but a much smaller quantity of acidic ammonium chloride may also form and ionize in the ammonia solution. Resulting pH of the solution will be a function of the interactions between these ions. Solution conductivity will also be significantly greater in the seawater-ammonia mixture.

Scale deposits found within the scope of these tests are listed and described in Appendix A, Tables VIII-13 and VIII-14. Deposit formation increased with duration of exposure and, generally, with addition of seawater to the ammonia. Fouling consisted primarily of corrosion products, chiefly of the metal exposed, although corrosion products from the coupons and test loop entered the test solution and redeposited on coupon surfaces. Thus AL-6X stainless steel and CP titanium which had excellent corrosion resistance still received a fouling deposit consisting primarily of aluminum and type 304 stainless steel corrosion products. The fouling deposits on these two uncorroded alloys were, however, significantly thinner than those deposits on aluminum and copper alloy coupons which experienced corrosion attack.

Scale deposits resulting from the seawater addition were not significant in the scope of these tests. Calculations of the maximum total solids injected into the system indicate that only 15.9 grams were introduced, of which only 1.5 g were calcium carbonate and magnesium hydroxide. Since the coupons placed on test represented only a small percentage of the total surface area in the test loop, deposition of significant seawater-ammonia scales was unlikely, and, in fact, did not occur to a significant level.

VI. RECOMMENDATIONS

1. Assess the suitability of Alclad alloy 3003, CP titanium and AL-6X stainless steel to probable OTEC cleaning systems.

2. Determine the corrosion behavior of Alclad alloy 3003, Al-5052, AL-6X stainless steel and CP titanium in gaseous (condensing) ammonia with and without inhibitors and seawater chlorides present.

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A special debt of gratitude is extended to Joe Darby for his special assistance in acquiring specific tube alloys required of this study.

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APPENDIX A

Detailed Tabular Data

TABLE VIII-1
TUBE TEST UNIT
INITIAL TUBE PERFORMANCE*

Alloy	Maximum Pit Depth (mm)	Performance
Aluminum Alloy 5052	1.24 (perforation)	Severe pitting occurred on all removed tubes (4) with up to approximately 20-40 pits per meter greater than 1 mm in diameter. Perforation of the tubes on test has occurred both upstream and downstream of the artificial leak site.
Aluminum Alclad Alloy 3003	0.18	Mild pitting occurred on all removed tubes (2), with up to approximately 5-15 pits per meter greater than 1 mm in diameter. Maximum pit depth was the approximate depth of the Al-7072 cladding.
Copper Alloy 706	<0.01	Tubes on test were free of localized attack (except at the artificial leak) and experienced general uniform corrosion.
Copper Alloy 715	<0.01	Tubes experienced only general uniform corrosion attack (except at the artificial leak) at a level below that experienced by CA 706.
Copper Alloy 722	<0.01	Tubes experienced only general uniform corrosion attack (except at the artificial leak) at a level below that experienced by CA 706.
AL-6X Stainless	<0.01	No observed corrosion.
CP Titanium	<0.01	No observed corrosion.

---

* Tubes removed during first 3.5 months ammonia exposure.

TABLE VIII-2
TUBE TEST UNIT
FINAL TUBE PERFORMANCE*

Alloy	Maximum Pit Depth (mm)	Performance
Aluminum Alloy 5052	1.24 (perforation)	Perforations appeared in these tubes at the rate of 1-2 per meter. Extensive pitting occurred with 40-50 pits per meter length greater than 1 mm in diameter.
Aluminum Alclad Alloy 3003	0.20	Extensive pitting occurred with 30-50 pits per meter greater than 1 mm in diameter. However, pit depth has been limited to the thickness of the Al-7072 cladding
AL-6X Stainless	<0.01	No observed corrosion.
CP Titanium	<0.01	No observed corrosion.

---

* Tubes removed after first 4-8 months ammonia exposure.

TABLE VIII-3
CORROSION PERFORMANCE OF CANDIDATE ALLOYS AS COUPONS IN 30°C, 1.8 M/SEC SEAWATER PLUS 5 PPM AMMONIA

	Average Penetration, mm (Maximum Pit Depth, mm)			
Exposure (months)	0.5	1.5	3.0	Pitting Observations
Copper Alloy 706	0.0124 (0.254)	0.0098 (0.64)	0.0164 (0.89)	After 0.5 months, severe surface attack in the form of numerous broad, shallow pits with up to 0.254 mm local penetration. Crevice attack at the specimen edge. After 1.5 months, severe surface attack in the form of broad, shallow pits with up to 0.64 mm local penetration. Attack was preferentially located at crevice contact areas and at the upstream coupon end. After 3.0 months, severe surface attack in the form of broad, smooth pits with up to 0.89 mm local penetration. Severe attack was preferentially located at the coupon end.

Average penetration corrected for losses occurring during post exposure cleaning.

Because of the low surface area and the short period of exposure, pitting data were based on the following key:

Small pits: Pits 0.025-0.01 mm deep, discernable as pit under 10X magnification and visible to naked eye.
Pits: Pits 0.025-0.05 mm deep (unless otherwise indicated), readily visible as pits to naked eye.
Very few: Less than 1/sq cm
Few: 1-2/sq cm
Several: 2-3/sq cm
Numerous: More than 3/sq cm

Table VIII-3 (continued)

	Average Penetration, mm (Maximum Pit Depth, mm)			
Exposure (months)	0.5	1.5	3.0	Pitting Observations
Aluminum Alloy 5052-H32	0.0021 (0.025)	0.0028 (0.05)	0.0036 (0.025)	After 0.5 months, 1 small pit was observed. After 1.5 months, 1 pit and 5 small pits were observed. After 3.0 months, 3 small pits were observed.
AL-6X Stainless	0.00001 (<0.01)	0.000003 (<0.01)	W.G. (<0.01)	No significant pitting.
CP Titanium	W.G. (<0.01)	W.G. (<0.01)	W.G. (<0.01)	No significant pitting.

Weight Gain

TABLE VIII-4
CORROSION PERFORMANCE OF CANDIDATE ALLOYS AS TUBES
IN 30°C, 1.8 M/SEC SEAWATER PLUS 5 PPM AMMONIA

	Average Penetration, mm[1] (Maximum Pit Depth, mm)			
Exposure (months)	0.5	1.5	3.0	Pitting Observations[2]
Aluminum Alloy 5052-H32	0.0024 (0.41)	0.0021 (0.05)	0.0037 (0.05)	After 0.5 months, 15 large pits, averaging 1.4 mm major axis dia. and up to 0.41 mm pit depth were observed. Each large pit was accompanied by numerous small pits in the immediate vicinity. Crevice attack at tube ends. Specimen removed at 1.5 months showed 1 pit and 2 small pits. Mild crevice attack at tube end. Specimen removed at 3.0 months showed 14 pits and 28 small pits.

---

[1] Average penetration corrected for losses occurring during post exposure cleaning.

[2] Because of the low surface area and the short period of exposure, pitting data were based on the following key:

Small pits: Pits 0.025-0.01 mm deep, discernable as pit under 10X magnification and visible to naked eye.
Pits: Pits 0.025-0.05 mm deep (unless otherwise indicated), readily visible as pits to naked eye.
Very few: Less than 1/sq cm
Few: 1-2/sq cm
Several: 2-3/sq cm
Numerous: More than 3/sq cm

Table VIII-4 (continued)

	Average Penetration, mm[1] (Maximum Pit Depth, mm)			
Exposure (months)	0.5	1.5	3.0	Pitting Observations[2]
Copper Alloy 706	0.0208 (0.025)	0.0175 (0.05)	0.0251 (0.076)	After 0.5 months, severe surface attack to 0.025 mm local penetration. Selective attack at tube ends. After 1.5 months, very uniform surface attack to 0.05 mm local penetration. Selective attack at tube ends. After 3.0 months, severe surface attack to 0.076 mm local penetration. Selective attack at tube ends and crevice attack at location of Swagelok fitting.
Stainless AL-6X	0.000044 (<0.01)	0.000025 (<0.01)	0.000038 (<0.01)	No significant pitting.
CP Titanium	0.000053 (<0.01)	0.000074 (<0.01)	0.000069 (<0.01)	No significant pitting.

TABLE VIII-5

SUMMARY OF EXPOSURE CONDITIONS
NOMINAL 5 PPM AMMONIA IN SEAWATER

Dates	Temperature °C	Blowdown l/min	Salinity o/oo	Blowdown pH
3/8/79- 3/23/79	30°C	15	17-30	--
3/23/79- 4/23/79	30°C	15	16-30	8.45-8.65
4/23/79- 6/8/79	30°C	15	15-24	8.4-8.8

TABLE VIII-6

CORROSION PERFORMANCE OF CANDIDATE ALLOYS AS COUPONS IN 30°C, 1.8 M/SEC SEAWATER PLUS 10 PPM AMMONIA

Exposure (months)	Average Penetration, mm (Maximum Pit Depth, mm) 0.5	1.5	3.0	Pitting Observations
Aluminum Alloy 5052-H32	0.00686 (0.05)	0.00752 (0.05)	0.0168 (0.86)	After 0.5 months, 1 pit and 1 small pit were present. After 1.5 months, 1 pit and 1 small pit were present. Crevice attack to 4 mils penetration. After 3 months, 10 large pits were observed, with local penetration to 0.86 mm and major axis diameter to 6 mm. Attack is predominately edge and crevice initiated with a few significant exceptions.
Copper Alloy 706	0.00142 (<0.01)	0.00152 (<0.01)	0.00197 (<0.01)	No significant pitting at 0.5 and 1.5 months. After 3.0 months, 15 small pits, smooth and rounded were present.

Average penetration corrected for losses occurring during post exposure cleaning.

Because of the low surface area and the short period of exposure, pitting data were based on the following key:

Small pits:	Pits 0.025-0.01 mm deep, discernable as pit under 10X magnification and visible to naked eye.
Pits:	Pits 0.025-0.05 mm deep (unless otherwise indicated), readily visible as pits to naked eye.
Very few:	Less than 1/sq cm
Few:	1-2/sq cm
Several:	2-3/sq cm
Numerous:	More than 3/sq cm

Table VIII-6 (continued)

	Average Penetration, mm (Maximum Pit Depth, mm)			
Exposure (months)	0.5	1.5	3.0	Pitting Observations
AL-6X Stainless	W.G. (<0.01)	0.000002 (<0.01)	0.000054 (<0.01)	No significant pitting.
CP Titanium	W.G. (<0.01)	0.000013 (<0.01)	W.G. (<0.01)	No significant pitting.

Weight Gain

TABLE VIII-7

CORROSION PERFORMANCE OF CANDIDATE ALLOYS AS TUBES IN 30°C, 1.8 M/SEC SEAWATER PLUS 10 PPM AMMONIA

	Average Penetration, mm[1] (Maximum Pit Depth, mm)			
Exposure (months)	0.5	1.5	3.0	Pitting Observations[2]
Aluminum Alloy 5052-H32	0.00191 (0.28)	0.00241 (0.33)	0.00406 (0.61)	After 0.5 months, 21 large pits averaging 0.76 mm in diameter and up to 0.28 mm deep. A few small pits were distributed over the tube interior. After 1.5 months, 6 large pits were observed with penetration to 0.33 mm and an approximate diameter of up to 1 mm. Crevice and end attack has occurred at the tube ends. After 3.0 months, 20 large pits were observed, accompanied by crevice and end attack on the tube specimen. Penetration was up to 0.61 mm and pit diameter up to approximately 5.3 mm.

---

[1] Average penetration corrected for losses occurring during post exposure cleaning.

[2] Because of the low surface area and the short period of exposure, pitting data were based on the following key:

Small pits:	Pits 0.025-0.01 mm deep, discernable as pit under 10X magnification and visible to naked eye.
Pits:	Pits 0.025-0.05 mm deep (unless otherwise indicated), readily visible as pits to naked eye.
Very few:	Less than 1/sq cm
Few:	1-2/sq cm
Several:	2-3/sq cm
Numerous:	More than 3/sq cm

Table VIII-7 (continued)

	Average Penetration, mm[1] (Maximum Pit Depth, mm)			
Exposure (months)	0.5	1.5	3.0	Pitting Observations[2]
Copper Alloy 706.	0.00744 (<0.01)	0.0138 (<0.01)	0.0152 (<0.01)	No significant pitting at 0.5 months. At 1.5 months, a very minor etch attack is apparent on the tube surface and crevice attack to 0.15 mm penetration is present at the tube ends. At 3.0 months, an etch is present on the tube surface and crevice attack to 0.38 mm penetration has occurred.
Stainless AL-6X	0.000028 (<0.01)	0.000038 (<0.01)	0.000041 (<0.01)	No significant pitting.
CP Titanium	0.000069 (<0.01)	0.000051 (<0.01)	0.000041 (<0.01)	No significant pitting.

TABLE VIII-8

SUMMARY OF EXPOSURE CONDITIONS
NOMINAL 10 PPM AMMONIA IN SEAWATER

Dates	Temperature °C	Blowdown l/min	Salinity o/oo	Blowdown pH
6/19/79- 7/5/79	30	15	18-32	8.45-8.8
7/5/79- 8/6/79	30	15	10-30	8.55-8.8
8/6/79- 9/24/79	30	15	8-25	8.55-8.75

TABLE VIII-9

CORROSION PERFORMANCE OF CANDIDATE ALLOYS AS COUPONS IN 30°C, 1.8 M/SEC SEAWATER PLUS 20 PPM AMMONIA

	Average Penetration, mm (Maximum Pit Depth, mm)			
Exposure (months)	0.5	1.5	3.0	Pitting Observations
Aluminum Alloy 5052-H32	0.00023 (0.025)	0.00064 (<0.01)	0.00033 (0.025)	After 0.5 months and 3.0 months, 1 small pit was observed.
Copper Alloy 706	0.00117 (0.05)	0.00144 (<0.01)	0.00170 (0.025)	At 0.5 months, one pit was observed. At 3.0 months, one small pit was observed.
AL-6X Stainless	0.000004 (<0.01)	0.000006 (<0.01)	0.000005 (<0.01)	No significant pitting.
CP Titanium	W.G. (<0.01)	0.000015 (<0.01)	W.G. (<0.01)	No significant pitting.

Average penetration corrected for losses occurring during post exposure cleaning.

Because of the low surface area and the short period of exposure, pitting data were based on the following key:

Small pits:	Pits 0.025-0.01 mm deep, discernable as pit under 10X magnification and visible to naked eye.
Pits:	Pits 0.025-0.05 mm deep (unless otherwise indicated), readily visible as pits to naked eye.
Very few:	Less than 1/sq cm
Few:	1-2/sq cm
Several:	2-3/sq cm
Numerous:	More than 3/sq cm
Weight Gain	

TABLE VIII-10

CORROSION PERFORMANCE OF CANDIDATE ALLOYS AS TUBES IN 30°C, 1.8 M/SEC SEAWATER PLUS 20 PPM AMMONIA

	Average Penetration, mm (Maximum Pit Depth, mm)			
Exposure (months)	0.5	1.5	3.0	Pitting Observations
Aluminum Alloy 5052-H32	0.00041 (<0.01)	0.00061 (<0.01)	0.00142 (<0.01)	No significant pitting.
Copper Alloy 706	0.00757 (<0.01)	0.0043 (<0.01)	0.0184 (<0.01)	At 3.0 months, crevice attack to 0.33 mm at the tube ends.
AL-6X Stainless	0.000025 (<0.01)	0.000033 (<0.01)	0.000025 (<0.01)	No significant pitting.
CP Titanium	0.000074 (<0.01)	0.0000095 (<0.01)	0.00011 (<0.01)	No significant pitting.

Average penetration corrected for losses occurring during post exposure cleaning.

Because of the low surface area and the short period of exposure, pitting data were based on the following key:

Small pits: Pits 0.025-0.01 mm deep, discernable as pit under 10X magnification and visible to naked eye.
Pits: Pits 0.025-0.05 mm deep (unless otherwise indicated), readily visible as pits to naked eye.
Very few: Less than 1/sq cm
Few: 1-2/sq cm
Several: 2-3/sq cm
Numerous: More than 3/sq cm

TABLE VIII-11

SUMMARY OF EXPOSURE CONDITIONS NOMINAL 20 PPM AMMONIA IN SEAWATER

Dates	Temperature °C	Blowdown l/min	Salinity o/oo	Blowdown pH
9/26/79- 10/17/79	30°C	15	10-15	--
10/17/79- 11/13/79	30°C	15	15-22	9.04-9.20
11/13/79- 1/2/80	30°C	15	22-26	8.94-9.36

TABLE VIII-12
FOULING ON COUPON SPECIMENS
SEAWATER PLUS AMMONIA

Exposure	Period (Months)	Average Fouling Weight Density (mg/cm^2) Aluminum Alloy 5052-H32	Copper Alloy 706	AL-6X Stainless Steel	CP Titanium Alloy
Seawater plus Nominal 5 ppm Ammonia	0.5	1.16	2.21	0.51	0.55
	1.5	3.82	2.45	2.33	2.28
	3.0	1.87	4.13	1.51	1.32
Seawater plus Nominal 10 ppm Ammonia	0.5	2.41	0.59	0.64	0.76
	1.5	4.62	2.01	1.76	2.09
	3.0	8.16	2.23	4.09	4.08
Seawater plus Nominal 20 ppm Ammonia	0.5	17.2	1.27	9.08	12.5
	1.5	45.0	1.49	43.6	45.2
	3.0	94.6	9.38	40.9	43.2

TABLE VIII-13
FOULING ON TUBE SPECIMENS
SEAWATER PLUS AMMONIA

		Fouling Weight Density (mg/cm^2)			
Exposure	Period (Months)	Aluminum Alloy 5052	Copper Alloy 706	AL-6X Stainless Steel	CP Titanium Alloy
Seawater plus Nominal 5 ppm Ammonia	0.5	1.57	22.3	1.01	1.01
	1.5	3.96	9.38	3.88	3.68
	3.0	4.50	4.43	4.06	3.90
Seawater plus Nominal 10 ppm Ammonia	0.5	1.28	2.75	0.44	0.46
	1.5	4.27	-	-	2.24
	3.0	9.14	10.5	5.65	5.88
Seawater plus Nominal 20 ppm Ammonia	0.5	13.3	5.82	11.7	12.3
	1.5	61.4	1.64	58.2	60.2
	3.0	153.1	22.5	160.4	162.6

TABLE VIII-14
FOULING ANALYSIS*

Seawater Plus Ammonia

Exposure	Analysis
Seawater Plus 5 ppm Ammonia	Analysis of surface deposits indicated that amorphous phases, clay or amorphous quartz (SiO_2), were the major constituents of the fouling layer. Crystalline components of the fouling layer included kaolinite ($Al_2Si_2O_4(OH)_4$), quartz (SiO_2), and small amounts of corrosion products and calcite ($CaCO_3$).
Seawater Plus 10 ppm Ammonia	The major constituents of the fouling deposits were amorphous clays or quartz (SiO_2). Crystalline components included illite (K-Na-Mg-Fe-Al-SiO-H_2O), basic magnesium silicate ($Mg_9Al_3(Si_5Al_3)O_{20}(OH)_{16}$), magnetite ($Fe_3O_4$), calcite ($CaCO_3$), and small amounts of corrosion products.
Seawater Plus 20 ppm Ammonia	After 0.5 months of exposure, the major portion of the deposits were amorphous, with the crystalline components present consisting of quartz (SiO_2), muscovite ($KAl_2Si_3AlO_{10}(OH)_2$), and calcite ($CaCO_3$). After this initiation period, at 1.5 and 3.0 months, the major components of the deposit are crystalline, consisting chiefly of aragonite (CaCO) and small quantities of calcite ($CaCO_3$).

* Analysis were performed using X-ray diffraction, energy dispersive X-ray and infrared specteral methods.

TABLE VIII-15

CORROSION PERFORMANCE OF CANDIDATE ALLOYS AS COUPONS IN 30°C, 1.8 M/SEC ANHYDROUS AMMONIA

	Average Penetration, mm (Maximum Pit Depth, mm)		
Exposure (months)	0.5	2.0	Pitting Observations
Aluminum Alloy 5052-H32	0.0011 (0.025)	0.0206 (<0.01)	After 0.5 months, mild surface attack (etching) and 1 small pit were observed. After 2.0 months, a more severe surface attack (etching) was observed.
Copper Alloy 706	0.0016 (0.025)	0.0045 (0.025)	After 0.5 months, 2 small pits were observed. After 2.0 months, 3 small pits were observed.
AL-6X Stainless	0.000009 (<0.01)	W.G. (<0.01)	No significant pitting.
CP Titanium	W.G. (<0.01)	W.G. (<0.01)	No significant pitting.

Average penetration corrected for losses occurring during post exposure cleaning.

Because of the low surface area and the short period of exposure, pitting data were based on the following key:

Small pits: Pits 0.025-0.01 mm deep, discernable as pit under 10X magnification and visible to naked eye.
Pits: Pits 0.025-0.05 mm deep (unless otherwise indicated), readily visible as pits to naked eye.
Very few: Less than 1/sq cm
Few: 1-2/sq cm
Several: 2-3/sq cm
Numerous: More than 3/sq cm
Weight Gain

TABLE VIII-16

SUMMARY OF EXPOSURE CONDITIONS
ANHYDROUS AMMONIA

A. Non-Copper Alloy Testing

Date	Period Days	Temperature °C	Water ppm
4/6/79- 4/23/79	0 through 17	30°C	68
4/23/79- 6/6/79	17 through 61	30°C	78

B. Copper Alloy Testing

Date	Period Days	Temperature °C	Water ppm
9/5/79- 9/20/79	0 through 15	30°C	--
9/20/79- 11/13/79	15 through 69	30°C	424

TABLE VIII-17

CORROSION PERFORMANCE OF CANDIDATE ALLOYS AS COUPONS IN 30°C, 1.8 M/SEC ANHYDROUS AMMONIA PLUS 1% SEAWATER

	Average Penetration, mm (Maximum Pit Depth, mm)		
Exposure (months)	0.5	2.0	Pitting Observations
Aluminum Alloy 5052-H32	0.0015 (<0.01)	0.0060 (<0.01)	No significant pitting at 0.5 month. Surface etch of estimated 0.01 mm local penetration at 2.0 months.
Aluminum Alloy Alclad 3003	0.0047 (<0.01)	0.0876 (<0.01)	Surface etch of estimated 0.01 mm local penetration.
Copper Alloy 706	0.012 (<0.01)	0.022 (<0.01)	No significant pitting.
AL-6X Stainless	0 (<0.01)	0.000006 (<0.01)	No significant pitting.

Average penetration corrected for losses occurring during post exposure cleaning.

Because of the low surface area and the short period of exposure, pitting data were based on the following key:

Small pits:	Pits 0.025-0.01 mm deep, discernable as pit under 10X magnification and visible to naked eye.
Pits:	Pits 0.025-0.05 mm deep (unless otherwise indicated), readily visible as pits to naked eye.
Very few:	Less than 1/sq cm
Few:	1-2/sq cm
Several:	2-3/sq cm
Numerous:	More than 3/sq cm

Table VIII-17 continued

	Average Penetration, mm[1] (Maximum Pit Depth, mm)		
Exposure (months)	0.5	2.0	Pitting Observations[2]
CP Titanium	W.G.[3] (<0.01)	W.G. (<0.01)	No significant pitting.

---

[3] Weight Gain

TABLE VIII-18

SUMMARY OF EXPOSURE CONDITIONS ANHYDROUS AMMONIA PLUS NOMINAL 1% SEAWATER

A. NON-COPPER ALLOY TESTING

Date	Period, Days	Temp., °C	Water %	Cl^-, ppm	Na^+ ppm
6/7/79 - 6/21/79	0 through 14	30°C	1.04	130	84
6/21/79-	14 through 60	30°C	--	50	25.8

B. COPPER ALLOY TESTING

Date	Period, Days	Temp., °C	Water %	Cl^-, ppm	Na^+ ppm
11/14/79-11/28/79	0 through 14	30°C	--	278	106
11/28/79-1/17/80	14 through 65	30°C	1.10	228	93

TABLE VIII-19
FOULING ON COUPON SPECIMENS
ANHYDROUS AMMONIA TESTS

Exposure	Period (Months)	Fouling Weight Density (mg/cm^2)				
		Aluminum Alloy 5052-H32	Aluminum Alloy Alclad 3003	Copper Alloy 706	Stainless Alloy AL-6X	CP Titanium Alloy
Anhydrous Ammonia	0.5	0.23	-	-	0.026	0.021
	2.0	2.35	-	0.61	0.016	0.014
Anhydrous Ammonia plus 1% Seawater	0.5	0.41	0.80	3.11	0.070	0.064
	2.0	0.73	0.90	5.45	0.15	0.20

TABLE VIII-20
FOULING ANALYSIS*

Ammonia Plus Seawater

Exposure		Analysis
Non-Copper Alloy Exposures	Anhydrous Ammonia	Primary components of the deposits were corrosion products. Elemental analysis revealed the presence of copper and iron as common to all deposits
	Ammonia Plus 1% Seawater	Primary components of the deposits were corrosion products. Elemental analysis gave major peaks for Mg, Al, Si, and S, and minor peaks for Na, Fe, Cu, and Cl.
Copper Alloy 706 Exposures	Anhydrous Ammonia	Copper corrosion products $(Cu(OH)_2)$ were the major constituents of the deposit.
	Ammonia Plus 1% Seawater	Copper corrosion products $(Cu(OH)_2)$were the major crystalline and amorphous phases present.

* Analysis were performed using X-ray diffraction, energy dispersive X-ray, and infrared specteral methods.

TABLE VIII-21

CANDIDATE ALLOY PERFORMANCE AFTER EXPOSURE TO AS-RECEIVED FILTERED SEAWATER

	Average Cumulative Penetration (mm)				
Exposure Period (months)	0.5	1.5	3.0	6.0	12.0
Seawater Temperature (°C)	14°	18°	23°	30°	12°C
Aluminum Alloy 5052-H32	0.0076	0.0147	0.0211	0.0236	0.0259
Copper Alloy 706	0.0041	0.0145	0.0191	0.0257	0.0244
AL-6X Stainless	0.000004	0.000003	Nil	0.000004	0.000011
CP Titanium	Nil	Nil	Nil	Nil	Nil

Nil - Specimen weight loss too low to allow accurate estimate of corrosion behavior.

TABLE VIII-22

CANDIDATE ALLOY PERFORMANCE AFTER EXPOSURE TO AS-RECEIVED FILTERED SEAWATER

	Average Cumulative Penetration (mm)				
Exposure Period (months)	0.5	1.5	3.0	6.0	12.0
Seawater Temperature (°C)	14°	18°	23°	30°	2.0°
Aluminum Alloy 5052-H32	0.012	0.015	0.018	0.021	0.022
Aluminum Alclad Alloy 3003	0.014	0.019	0.026	0.030	0.031

APPENDIX B

Supplementary Photographs

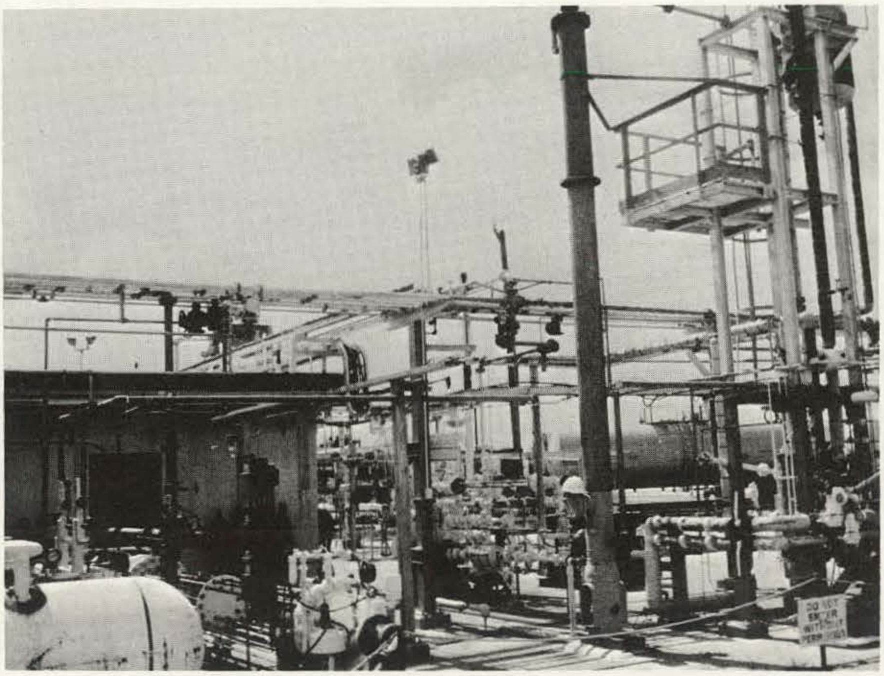

FIGURE VIII-1

Freeport, Texas, Test Bed Area
Dow Chemical Company U.S.A.

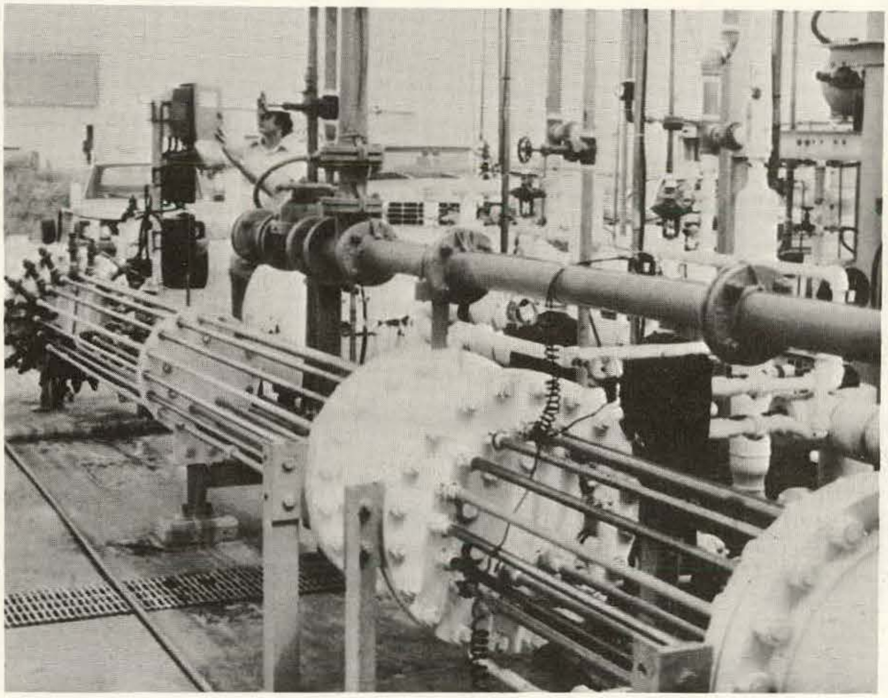

FIGURE VIII-2

Tube Test Unit

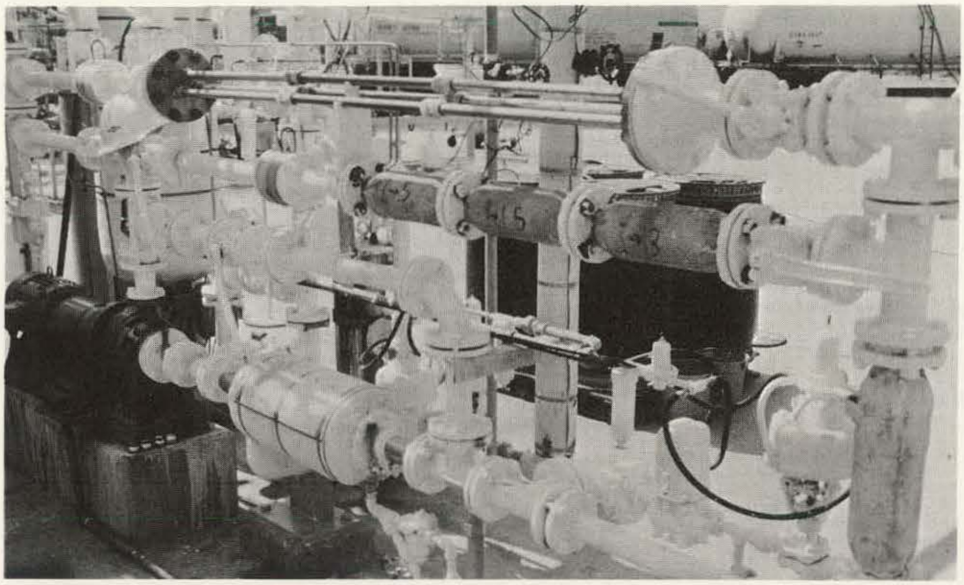

FIGURE VIII-3

Seawater Plus Ammonia Test Loop,
Non-Copper Alloy Testing

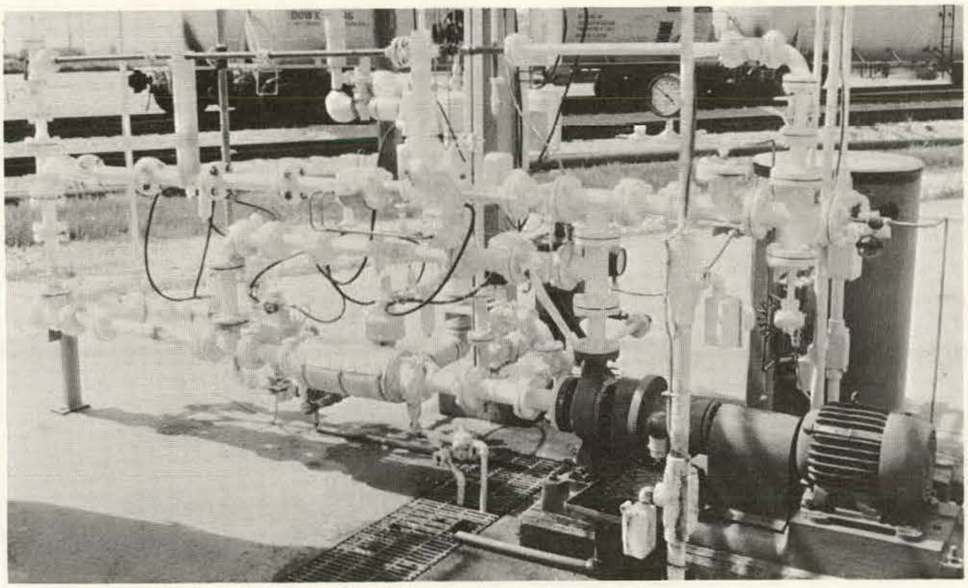

FIGURE VIII-4

Seawater Plus Ammonia Test Loop
Copper Alloy Testing

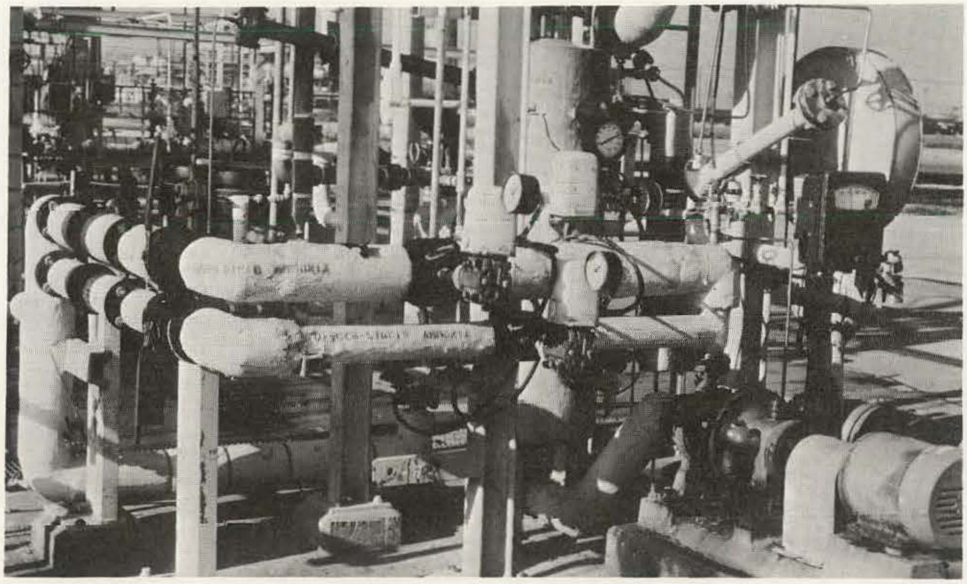

FIGURE VIII-5

Anhydrous Ammonia Test Unit

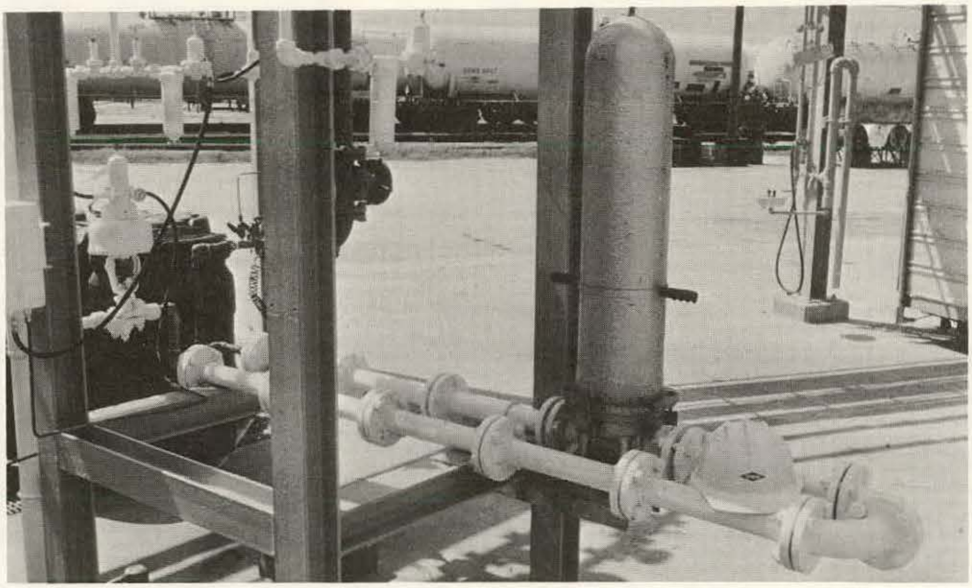

FIGURE VIII-6

Once-Through Seawater Test Loop

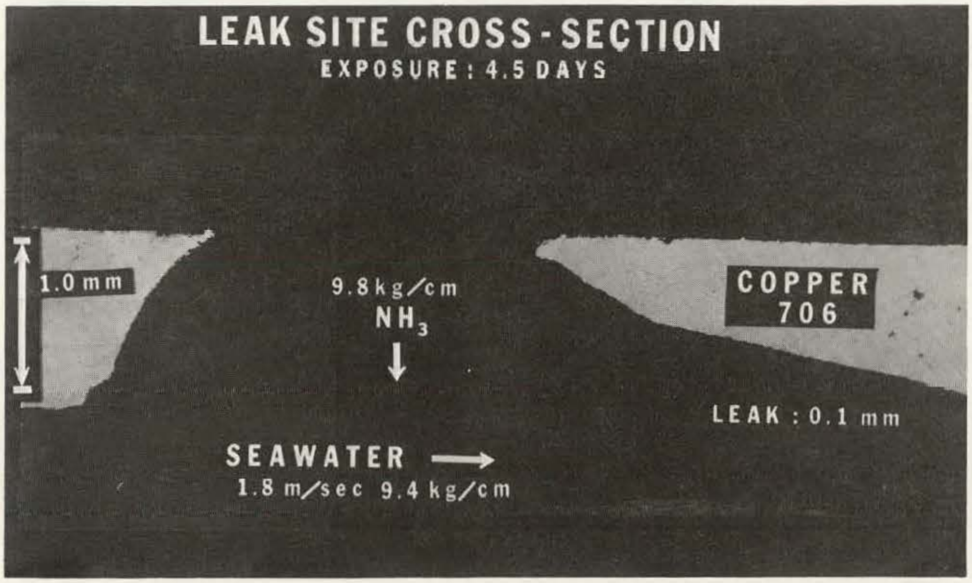

FIGURE VIII-7

Leak Site in Copper Alloy 706 Tube Exposed 4.5 Days in the Tube Test Unit at a 0.4 kg/cm^2 Ammonia/Seawater Pressure Differential

Premature Withdrawal

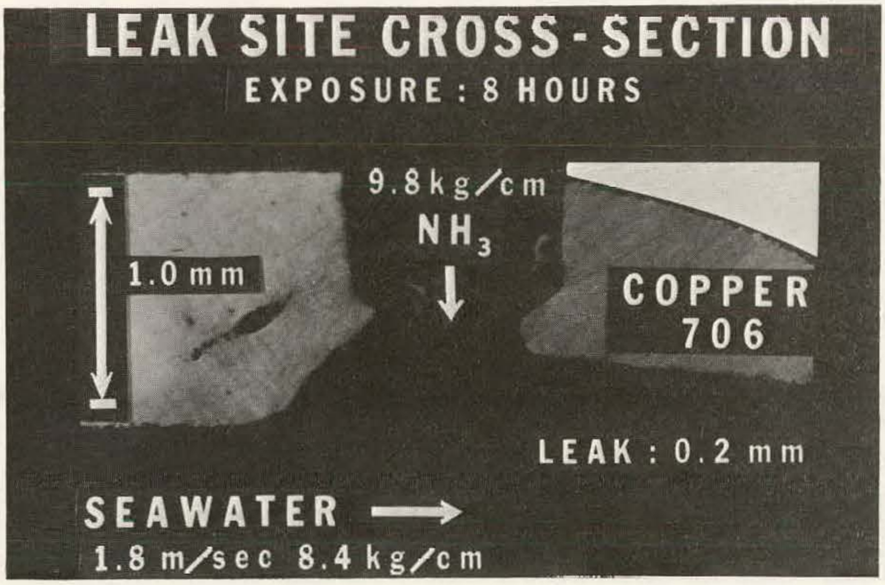

FIGURE VIII-8

Leak Site in Copper Alloy 706 Tube Exposed 8 Hours in the Tube Test Unit at a 1.4 kg/cm^2 Ammonia/Seawater Pressure Differential

Premature Withdrawal

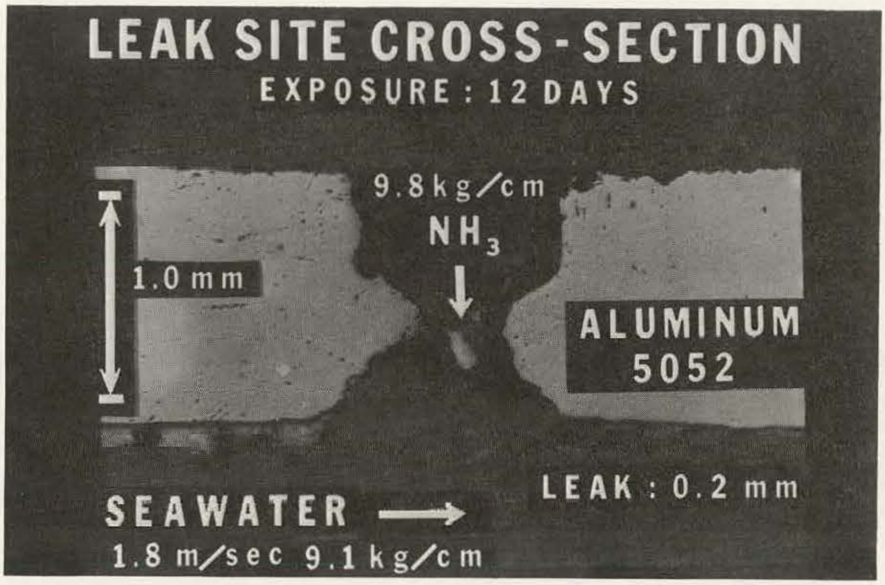

FIGURE VIII-9

Leak Site in Aluminum Alloy 5052 Tube Exposed 12 Days in the Tube Test Unit at a 0.7 kg/cm^2 Ammonia/Seawater Pressure Differential

Premature Withdrawal

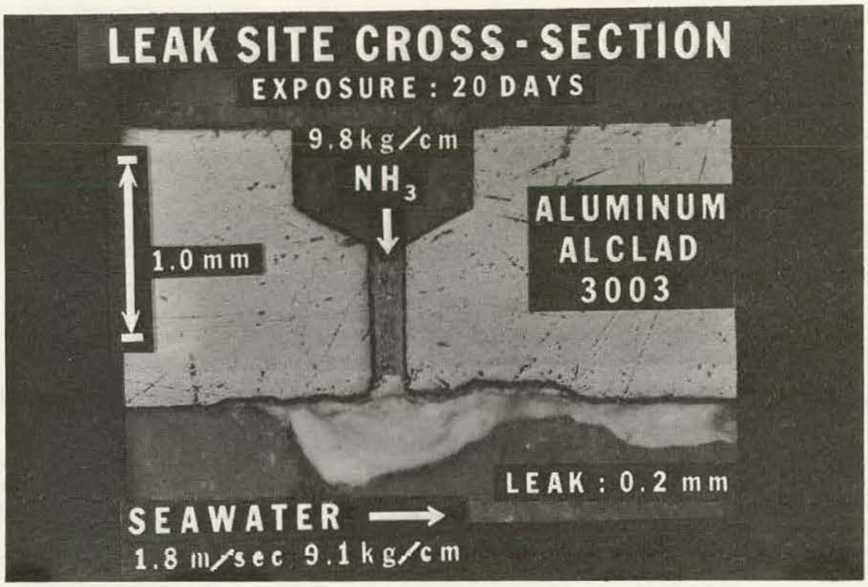

FIGURE VIII-10

Leak Site in Aluminum Alclad Alloy 3003
Exposed 20 Days in the Tube Test Unit
at a 0.7 kg/cm^2 Ammonia/Seawater
Pressure Differential

Scheduled Withdrawal

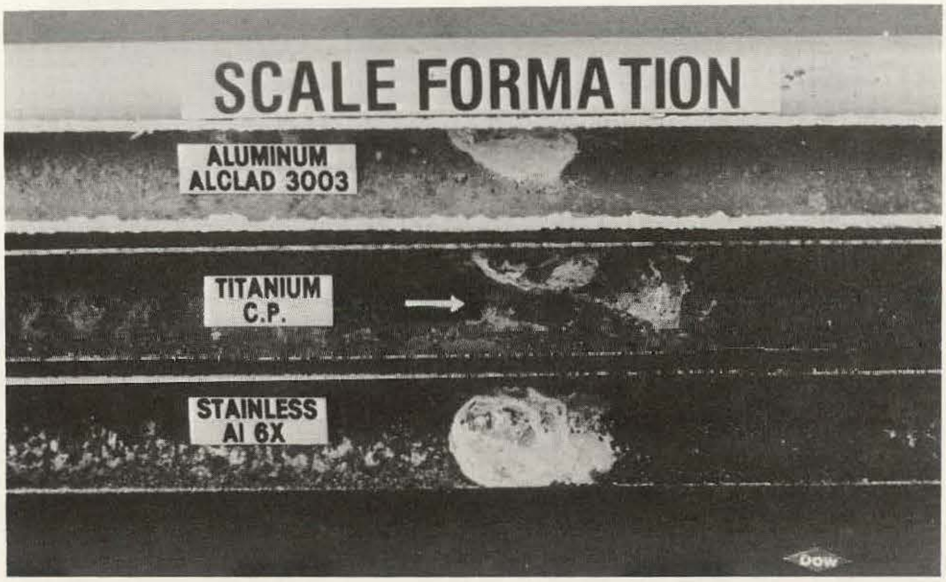

FIGURE VIII-11

Scale Formation at Leak Site
of Tubes Withdrawn from the Tube Test Unit

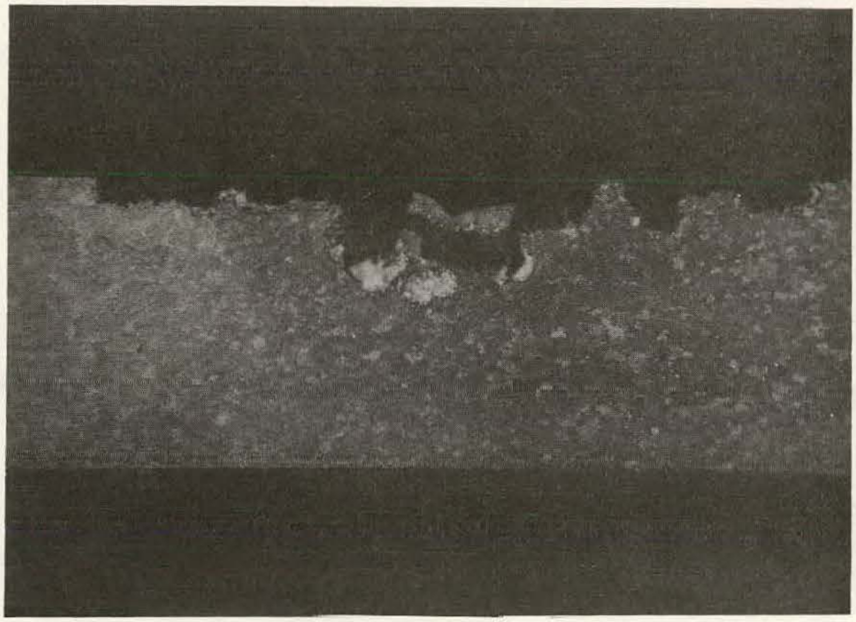

FIGURE VIII-12

Pit Cross-Section of Aluminum Alloy 5052 Tube
Exposed 0.5 Months to 30°C Seawater
Plus 5 ppm Ammonia
(Tube is 1.2 mm, 49 mils thick)

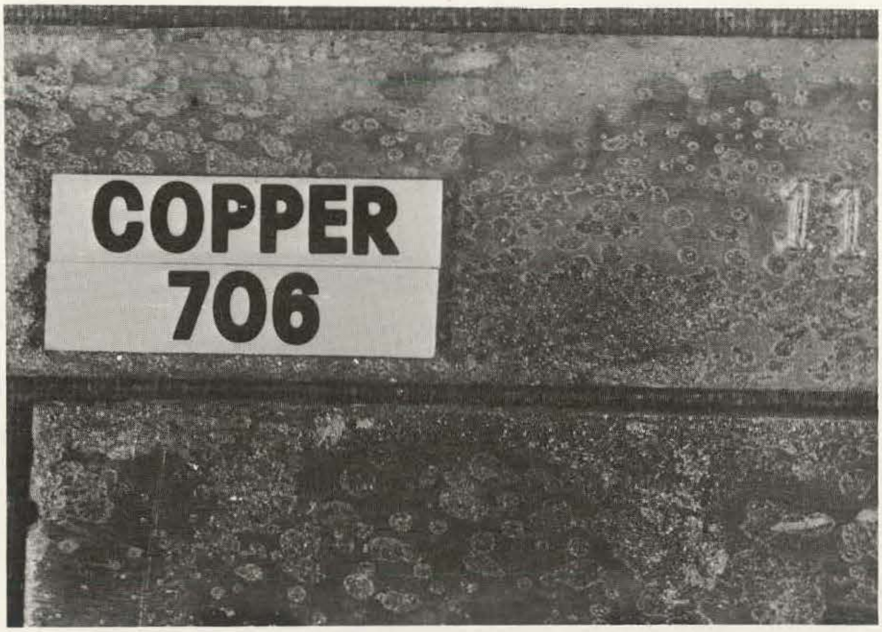

FIGURE VIII-13

Localized Corrosion on Copper Alloy 706 Coupon
Exposed 0.5 Months to 30°C Seawater
Plus 5 ppm Ammonia
(Coupon is 19 mm [0.75 in] wide)

FIGURE VIII-14

Cross-Section of Aluminum Alclad Alloy 3003 Coupon
No Exposure
(Coupon is 1.6 mm,63 mils, thick)

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