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METAL CORROSION ASSOCIATED WITH
THERMAL CYCLING OF INHIBITED AND
UNINHIBITED PROPYLENE GLYCOL/WATER SOLUTION
IN SOLAR DHW SYSTEMS

by

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ABSTRACT

As part of the Solar Reliability and Materials Program at Argonne National Laboratory, metal corrosion associated with thermal cycling at 82°C "circulating" and 176°C "stagnating" temperatures of propylene glycol and ASIM corrosive water mixture (50% v/o) was investigated.

Preliminary data indicate that in a mixed metal system of copper, steel, and aluminum specimens stagnating together in a glycol solution, the copper randomly pits and the pitting stops when the pit depth extends to about 1-1/2 mil. The addition of 1% molybdate as an inhibitor to the glycol solution is slightly beneficial for steel, but the added expense of adding and maintaining the concentration of an inhibitor may not be warranted. Dissolved copper rapidly deposits on the aluminum surface and promotes severe galvanic corrosion.

1. INTRODUCTION

The assessment of corrosion of materials by solar fluids is a part of the overall Solar Reliability and Materials Program at Argonne National Laboratory (ANL), and the specific tasks were documented in the "ANL Solar Data Project Plan," ANL/SDP-TM-78-1 (Sept. 1978). Most typical solar domestic-hot-water (DHW) systems contain primary and secondary loops. In cold climatic regions (where temperature can drop below 0°C), a solution of propylene glycol/water with inhibitors is commonly used as the primary fluid to prevent freezing.

During normal operation, the primary fluid circulates at temperatures from 29 to 88°C, with only a slight increase (with time) in corrosivity for commercially available organically inhibited propylene glycol (PG)/water solutions. However, under abnormal conditions such as the cessation of circulation (stagnation), the fluid temperature can reach between 149 to 177°C; at these higher temperatures, the PG/water solution and its organic inhibitors can break down to form acids that can corrode the metals in the primary loop.

This report is a preliminary study of metal corrosion in the primary loop associated with an inorganically inhibited PG/water solution, subjected to simulated thermal cycling conditions in solar DHW systems. A temperature of 82°C was maintained with the fluid circulating, and then the temperature was raised to 176°C with the fluid stagnating. The study evaluates sodium molybdate as an effective and stable inorganic inhibitor at 176°C.

II. EXPERIMENTAL APPARATUS AND METHODS

A. Fluid Corrosion Loop

In early 1981, it was decided BY DOE and ANL to study the corrosive behavior of a fluid in a simulated thermal cycling environment in solar DHW systems. A corrosion loop was then designed and installed* in Building 362 at ANL. The loop is intended for the study of hydrodynamic effects (those associated with fluid shear rate, velocity, residence time and temperature) under the alternating conditions of circulating and stagnation; fluid with respect to metal corrosion and scale deposition. Figure 1 is a schematic diagram of the loop. It has four basic components and associated support hardware. The basic components are: (1) a 9.8-L Teflon lined storage tank, fabricated from 8-in. schedule 80 carbon steel pipe, (2) a Type 304 stainless steel centrifugal pump,[†] rated for use at 1.138 MN/m² (150 psig) with 260°C (500°F) fluid, (3) a liquid-to-liquid copper heat exchanger[‡], and (4) a 68-cm-overall-length test chamber (fabricated from 1/2 in. schedule-40 stainless steel pipe with the internal and external surfaces of internal components lined with Teflon [including a 1.6 cm O.D. by 17.8 cm long specimen holder, temperature sensor thermal weld, and cartridge heater]). Figures 2 and 3 are photographs of the loop and test chamber, respectively. The support hardware include copper tubing (1/2-in. main line and 3/8-in. connecting lines), brass control valves, glass rotometer, Type 304 stainless steel expansion tank (2.5 L) and relief valve, band and wrap-around heaters, and a temperature controller.

B. Loop Operation

The storage tank is first filled with solar fluid, and the fluid then is pumped throughout the loop system to fill the components. The volume of fluid desired (approximately 12.2 L) is controlled by the liquid-level sensor in the storage tank. The system is then nitrogen purged at room temperature. The thermal cycling at circulating and stagnating conditions is then begun. The cycling procedure is explained in detail in the Appendix to this report. Briefly, the circulating condition is obtained by heating the fluid in the storage tank to 82°C and maintaining that temperature in the heated test chamber, with the fluid flowing at the rate of 0.095 L/s (1.5 gal/min). The

*By ECA, Inc., Lisle, IL

[†]Gelber Pump Co. Model #DH-11

[‡]Bell & Gossett Co.

FIG. 1 Schematic Diagram of Corrosion Loop. Value Numbers are Referred to in the Appendix.

flow is regulated by the pump pressure and throttling valves. The stagnating condition is obtained by shutting off the inlet and outlet valves connected to the test chamber, stopping the pump, and adjusting the temperature controller to 176°C. This simulates the conditions in the flat-plate collector of a solar DHW primary system when a pump fails and flow stops; the only circulation is by convection. To return to the circulating condition, the temperature in the test chamber is lowered back to 82°C before opening the inlet and outlet valves and turning on the pump to recirculate the fluid throughout the loop.

The specimen holder in the upper test chamber is Teflon lined and holds nine metal specimens (1.715 cm O.D. x 1.588 cm I.D. x 1.270 cm long), which are separated by Teflon washers.

FIG. 2 Closeup of Fluid Corrosion Loop--Before Insulation.

FIG. 3 Closeup of Test Chamber--Before Insulation.

C. Corrosion Tests

Three commonly used metals in primary solar DHW systems in contact with PG/water solutions were tested: copper (CA 122), aluminum (6061) and steel (black iron--carbon steel 1008). Four solutions were proposed for testing; they were 50% by volume PG/water mixtures containing the following:

- a. no inhibitor
- b. organic inhibitor (GM-6038 formulation)
- c. inorganic inhibitor (Dowfrost--phosphate addition), and
- d. Inorganic/organic inhibitor (Molybdenum Corp. formulation--molybdate and tolytriazole addition).

This report evaluated two solutions containing 50% by volume of ASTM corrosive water [ASTM B-2570-73--100 ppm each of chloride, sulfate, and bicarbonate addition to distilled water] and 50% by volume of

- a. uninhibited PG; or
- b. PG inorganically inhibited with molybdate addition (Molybdenum Corp. formulation minus the organics).

The latter was chosen because without organic inhibitors (tolyltriazole and Pluronic L-61) the solution is nontoxic. Thus, it might be approved as a solar fluid suitable for a single-wall heat exchanger in solar DHW systems, as in the case of distilled water.

1. Uninhibited Propylene Glycol/Water Solution: The simulated thermal-cycling test on metal ring specimens in uninhibited (PG)/water solution lasted 19 cycles. Each cycle consisted of alternate 50-h periods at circulating (82°C) and stagnating (176°C) conditions. A set of specimens (copper, aluminum, and steel) was removed at the end of the 5th, 9th, and 19th cycles.

2. Inorganically Inhibited Propylene Glycol/Water Solution: The thermal-cycling test on metal specimens in inorganically inhibited PG/water solution consisted of a total of 886 at circulating (82°C) and 1275 at stagnating(176°C) conditions in 15 cycles. In the first seven cycles, each cycle had around 65 circulating and 96 stagnating conditions, alternately. During this period, the system flow gradually decreased and finally stopped circulating due to clogging of flow passage by residues and corrosion products. The system was cleaned and restarted. The next six cycles varied between 56 to 96 circulating and 72 to 96 stagnating, alternately. Finally, in the 15th cycle, the pump broke down after 24 circulating, and the test was terminated after an additional 110 stagnating. Test specimens were removed and or reinserted at various intervals.

III. RESULTS AND ANALYSIS

Chemical analyses of the test solutions (taken at different intervals) are summarized in Table 1, and the corrosion pitting evaluations are shown in Table 2. Metal-loss data are excluded because excessive descaling was necessary to conduct pitting measurements. In general, the metal-loss data corresponded with the average pitting data.

The analyses of the chemical compositions of the initial uninhibited (solution 1) and inhibited (solution 3) PG/water mixtures in Table 1 agree with the original compositions. The formation of glycolic acid during stagnating at 176°C is observed in solutions 2, 4, and 5. This resulted in a drop in pH from the original solution. The increase in chloride content in solutions 1 and 3 from the original make-up could be from the propylene glycol or from the loop system.

The corrosion-pitting measurements (according to ASTM G46-76) in Table 2 are reported at intervals of stagnating hours because corrosion attack occurs during the stagnating part of a cycle, when the PG/or inhibitors or both break down to form glycolic acid or a reduction in inhibitor concentration or both. At the lower temperature (82°C) of the circulating part of a cycle, corrosion pitting is not expected to occur, as witnessed in previous unpublished experiments and by service experience, is well known. Comparisons of pitting corrosion of metal specimens exposed to uninhibited and inhibited PG/water mixtures for copper (CA 122), steel (Black Iron--1008) and aluminum (6061) are discussed separately below.

Copper randomly pits in either uninhibited or inhibited (molybdate) solution, and the pitting seems to stop propagating when the pit depth reaches 1.5 mil. The pitting initiation can occur any time in the first few thermal cycles. However, such shallow pits of 1.5 mil are not detrimental to the reliability of the solar DHW systems.

In the case of steel, the addition of an inhibitor results in increased resistance to pitting corrosion, as shown in Fig. 4 which relates the pit depth P to time of exposure T . However, the pitting data in Table 2 for the 15th cycle are excluded in Fig. 4. The reason is that during this last 110 stagnating hours, the molybdate concentration is below 0.05%; therefore, the molybdate loses its effectiveness as an inhibitor. Molybdenum Corporation recommends a molybdate concentration between 0.05% to 1.0% by weight to provide adequate inhibition in PG/water systems. This loss of inhibition also explains the high pit depth of 3 mils maximum and 2 mil average for only 110 stagnating hours (starting with a new specimen) in the 15th cycle. In fact this pitting data is even higher than the 228-h stagnating data in the uninhibited solution. The latter solution probably has less than 0.5% glycolic acid from the gradual breakdown of PG, whereas the 15th-cycle inhibited (less than 0.05% molybdate) solution starts with 1.45% glycolic acid to promote pitting.

Table 1. Chemical Analyses of PG/Water Mixtures

Type:	Uninhibited Mixture		Inhibited Mixture		
Solution:	1	2	3	4	5
Description:	Cycle 1 - 48 h Circulating	Cycle 19 - 912 h Stagnating	Cycle 1 - 48 h Circulating	Cycle 7 - 600 h Stagnating	Cycle 14 - 1165 h Stagnating
PG, %	49	48	46	45	40
Density, gm/cc	1.04	1.04	1.06	1.05	1.06
B.P., °C	194	194	195	195	191
pH	6.5	4.8	8.1	6.7	7.0
Glycolic Acid, %	0	1.47	0	1.06	1.45
Total Alk., mg/l	—	—	850	450	600
Molybdate, %	—	—	0.11	0.07	<0.05
Borate, %	—	—	0.13	0.16	0.30
Silicate, %	—	—	0.17	0.16	0.18
Nitrate, %	—	—	0.07	0.06	0.05
Sulfate, ppm	48	50	46	50	54
Bicarbonate, ppm	52	60	56	64	62
Chloride, ppm	115	110	116	103	114

Original Composition Makeup:

Uninhibited Mixture — 50% by volume of Dow Chemical Company's uninhibited PG and 50% by volume of ASTM corrosive water (containing 100 ppm each of sodium chloride, sodium sulfate and sodium bicarbonate).

Inhibited Mixture — Composition in weight percent - 50.29% PG, 0.12% NaNO_3 , 0.52% $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5 \text{H}_2\text{O}$, 0.084% $\text{Na}_2\text{SiO}_3 \cdot 5 \text{H}_2\text{O}$, 0.25% $\text{Na}_2\text{MoO}_4 \cdot 2 \text{H}_2\text{O}$, 0.084% NaOH and 50% ASTM corrosive water.

Table 2. Corrosion Pitting Evaluation

Solution Type	Time at Stagnating Temp. (176°C), h	Pitting, mils		Remarks on Appearance
		Max	Avg	
<u>I. Copper (CA 122)</u>				
(a) Uninhibited	228	1.5	1.3	--
PG/Water	456	0.8	0.5	--
Mixture	912	1.2	1.0	tarnished
(b) Inhibited	384	0.6	0.5	--
PG/Water	781	1.4	1.1	--
Mixture	1165	1.3	1.1	tarnished
(Molybdate)	1165 + 110 ^a	1.4	1.1	tarnished
	110 ^a	1.3	1.2	tarnished
<u>II. Steel (Black Iron--1008)</u>				
(a) Uninhibited	228	2.5	1.4	gray
PG/Water	456	3.9	2.0	black
Mixture	1165	3.2	2.5	black
(Molybdate)	1165 + 110 ^a	4.4	3.2	black/tarnished
	110 ^a	3.0	2.0	black/tarnished
<u>III. Aluminum (6061)</u>				
(a) Uninhibited	228	1.0	0.8	tarnished Cu
PG/Water	456	1.6	1.1	tarnished Cu
Mixture	912	1.4	1.0	tarnished Cu
(b) Inhibited	384	5.7	3.8	Cu plated (heavy)
PG/Water	600	(Perforated		Cu plated (heavy)
Mixture	600	88 mil Wall)		Cu plated (light) ^b
(Molybdate)	565	2.8	2.0	Cu plated (light) ^b
	565 + 110 ^a	3.2	2.2	Cu plated (light) ^b
	110	2.0	1.4	Cu plated (light) ^b

^a15th cycle--only 24 h circulating prior to stagnating and molybdate below 0.05%.

^bLoop cleaned before restart of 8th cycle.

A conservative estimate* of the number of years needed to perforate and/or to degrade the structural integrity of a typical 20-mil wall steel flat-plate collector can be extrapolated from Fig. 4. Assuming eight stagnation weekends of 50 hours each per year (400 stagnating hours per year), it will take about 15 and 18 years to perforate the 20-mil wall in uninhibited (calculated from $P = 3.00 \times 10^{-3} T + 2.45$) and inhibited (calculated from $P = 2.80 \times 10^{-3} T$) 50 vol % PG/water solutions, respectively. Since the metal loss corresponds to the average pit depth, we can similarly estimate the number of years before the collector loses half of its wall thickness (i.e., 10 mils) and thereby its structural integrity. The estimation is about 8 years (calculated from $P = 2.95 \times 10^{-3} T + 0.70$) in uninhibited solution and 11 years (calculated from $P = 2.25 \times 10^{-3} T$) in inhibited solution.

FIG. 4 Pitting Corrosion of Steel (Black Iron -- 1008) in 50% PG/Water Mixture.

With regard to aluminum, in a mixed-metal system containing both copper and aluminum under the stagnating condition in the same test chamber, the copper oxide or copper metal will deposit on the aluminum specimen surface to initiate localized galvanic corrosion in both the uninhibited and inhibited PG/water solutions. The severity of galvanic corrosion attack depends on the concentration of copper and the movement of the test solution in the test chamber. Thus the corrosion pitting attack is less severe when the concentration of copper is lower in (1) the first run in uninhibited solution and (2) the last part of the second run in the inhibited solution after the loop cleaning.

*Pitting propagation in steel normally follows a fraction power law rather than linear law.

It would be interesting to make a run without a copper specimen in the test chamber to simulate the condition of an aluminum flat-plate collector with a copper heat exchanger and condenser.

IV. SUMMARY AND RECOMMENDATIONS

Based on the limited preliminary study on mixed metal corrosion in 50 vol % PG/water solutions under thermal cycling at alternate circulating (82°C) and stagnating (176°C) conditions, the following conclusions and recommendations are made.

A. SUMMARY

1. An accelerated corrosion test has been developed which simulates corrosion of metals in PG/water solutions with thermal cycling at circulating 82°C and stagnating (176°C) conditions, alternately.

2. Metal specimens of copper, steel and aluminum (separated by Teflon washers) were simultaneously evaluated in an inert test chamber which contained 50 vol % of PG and ASTM corrosive water with and without molybdate inhibitor. The results indicate the following:

- a. Copper pits randomly, and the propagation of pitting appears to stop at a pit depth of 1-1/2 mils in uninhibited or inhibited solution.
- b. Based on 400 stagnating hours per year, it is estimated that 20-mil wall steel flat-plate collector probably will exhibit perforation from pitting in about 15 years and 18 years in uninhibited and inhibited (1% molybdate) solutions, respectively. Similarly, the collector can lose half of its wall thickness (and thereby its structural integrity) in 8 and 11 years in the glycol solutions without and with molybdate inhibition, respectively.
- c. Under stagnating conditions, the dissolved copper in the test chamber rapidly deposits on the aluminum specimen surface to initiate and promote severe galvanic corrosion.

B. Recommendations

1. The beneficial effect of molybdate addition to the glycol solution to extend the service life of the steel collector during stagnating temperature conditions does not appear to be sufficient to warrant the added expense of paying for the inhibitor and maintaining the inhibitor concentration.

2. Preliminary data reveal that both copper and steel are satisfactory in uninhibited glycol solution. Additional tests should be conducted to confirm the preliminary data.

3. Thermal-cycling tests, similar to the ones described here, are recommended with only aluminum specimens in the test chamber in uninhibited and inhibited glycol solutions, to simulate a solar DHW system containing aluminum flat-plate collector and copper heat exchanger and/or condenser.

APPENDIX
LOOP OPERATING PROCEDURE
(Refer to Fig. 1)

FILL PROCEDURE

1. Open all valves.
2. Close V4, V12, V13, V14.
3. Connect polyvinyl tubing to V6 (use as vent).
4. Connect polyvinyl tubing to V3 (use as sight gauge).
5. Remove pressure gauge from V1.
6. Place funnel into V1 opening.
7. Add test fluid through funnel in V1, until storage is full as indicated by sight glass attached to V3 (7.49 L).
8. Close V1, V2, V3, V6, V5.
9. Open V12.
10. Connect polyvinyl tubing to V13 and place opposite end into container (leave V13 closed).
11. Remove polyvinyl tubing from V6.
12. Connect regulated nitrogen supply to V6 and adjust pressure to 10 psig on regulator for 10 min to remove air.
13. Slowly open V6 until pressure gauge on storage tank indicates approximately same as pressure regulator (30 psi).
14. Slowly open V13 until steady stream of fluid is indicated, then close V13.
15. Open V2, V10, V11.
16. Start pump.
17. Adjust flow for approximately 1.5 gpm, using valve on flow meter.
18. Very slowly, crack open V9. Pump may become noisy due to air in V9 line. When pump noise level is normal, open flow meter valve fully, and adjust flow for 1.5 gpm using V9.

19. Check level indicator probe on storage tank (RX1000). If no indication, more fluid will need to be added. If indicating, skip to step 26, if not, continue to step 20.
20. Stop pump.
21. Close V6 and nitrogen pressure bottle valve.
22. Remove nitrogen fill hose from V6, and relieve pressure from storage tank. Leave V6 open.
23. Open V1 and add fluid through funnel until probe is satisfied.
24. Close V1, V6.
25. Start pump and return to step 19.
26. Replace pressure gauge on V1 and open V1, remove polyvinyl tubing from V13.
27. Stop pump.
28. Relieve pressure from storage tank by opening V6.
29. Open V3 sight glass. If necessary lower storage tank level by draining through sight gauge until level is at least 3" from full.
30. Close V3, remove sight gauge. End of procedure.

CIRCULATION OPERATING PROCEDURE

- a. Assure that proper valves are open for circulation.
 - b. Assure that level probe on storage tank is indicating level.
 - c. Pressurize storage tank to 5 psig with nitrogen.
1. Start circulation pump.
 2. Adjust flow to 1.5 gpm using V9. Valve on flowmeter should be fully open, then closed 1 turn.
 3. Set both column heater and band-heater Variacs to 100%.
 4. Set auto controller at 82°C.
 5. Turn on column heater, band heater and storage tank emersion heater. Emersion heater is thermostatically controlled and preset for operating temperature.

6. When temperature indicated on T-1 approaches operating temperature, turn off storage-tank immersion heater.
7. Initial fine adjustment of auto controller may be necessary to maintain temperature at T-1.
8. Maintain flow for an accumulated 50.
9. Stop pump, turn off column heater and band heater. End of procedure.

STAGNATION OPERATING PROCEDURE

1. Circulating procedure through step 6 should be followed.
2.
 - (a) Close valve V2.
 - (b) Close valve V11.
 - (c) Stop pump.
 - (d) Turn off band heater.
3. Reduce pressure to 5 psig on V1 gauge by opening and closing V2.
4. Observe V1 gauge and digital temperature while performing step 5. Do not allow pressure to rise above 160 psig on V1 gauge. If pressure rises above 160 psig, reduce temperature and review procedure.
5. Increase auto controller in 10°C increments until 176°C is reached on digital temperature indicator. Pressure on V1 gauge should be equalized before increasing to the next 10°C increment. Auto controller may need fine temperature adjustments, from 176°C, until 176°C is obtained on digital temperature indicator.
6. Maintain stagnation at 176°C for 50.
7. Turn off column heater. End of procedure.

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