Field Testing of Heat Exchanger Tube Coatings

Prepared by:
Gawlik, K.; Sugama, T.; Webster, R.;
Reams, W

NREL/SR-550-26210

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September 20-23, 1998
Geothermal Resources Council
1998 Annual Meeting, San Diego,
California

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NOTICE

FIELD TESTING OF HEAT EXCHANGER TUBE COATINGS

Keith Gawlik
National Renewable Energy Laboratory
(303) 384-7515

Toshifumi Sugama, Ronald Webster, and Walter Reams
Brookhaven National Laboratory
(516) 344-4029

Abstract

In order to reduce the capital cost of geothermal power plant heat exchange equipment and the maintenance cost associated with fouling, the National Renewable Energy Laboratory has been conducting tests of polymer-based liner systems developed by Brookhaven National Laboratory. These liner systems protect low-cost carbon steel tubing from corrosion and are formulated to reduce the rate of fouling. Field test data of the latest liner systems show a fouling rate reduction of 15% to 19% compared to previous liner formulations. New substrates used between the polymer liner and tube are expected to improve the liner-to-tube bond and enhance durability during cleaning operations.

Introduction

Corrosion, erosion, and fouling by scale deposits are critical issues for brine-wetted heat exchanger tubes in geothermal power plants in the Salton Sea reservoir. Replacing these tubes is very costly and time consuming. At present, titanium alloys and stainless steels are commonly used in shell and tube heat exchangers for their corrosion resistance. These materials have no special anti-fouling properties. These metals are considerably more expensive and have much lower thermal conductivities than copper and carbon steel. The capital cost of these large heat exchangers would be considerably reduced if an inexpensive tube made of carbon steel could be coated with a thermally conductive material that provides corrosion resistance equal to high-grade alloy steels. Thus, the development of corrosion/erosion/fouling-resistant coating and lining systems for carbon steel-based tubes is the subject of ongoing investigations at Brookhaven National Laboratory (BNL) and the National Renewable Energy Laboratory (NREL).

In 1987, BNL developed a silicon carbide (SiC) grit-filled polymer liner that provided a corrosion protective barrier and excellent thermal conductivity for heat exchanger tubes used in geothermal environments at temperatures up to 150ºC (302°F) (Fontana et al., 1987). The overall heat transfer coefficient of the SiC/polymer-lined tubes was only 9% less than that of expensive stainless steel (AL-6XN) tubes commonly used in geothermal power plants (Hassani and Hoo, 1995). The polymer matrix that bound the thermally conductive SiC grit into a coherent mass was composed of a trimethylolpropane trimethacrylate-cross linked styrene/methyl methacrylate (ST-TMP) copolymer network.

However, a major drawback of this lining material was that the polymer surfaces suffered hot brine-induced oxidation, forming a functional carboxylate group (Sugama, 1997). This group preferentially reacted with Ba^{+2} ions present in the geothermal brines to form Ba-complexed carboxylate hydrolysates. The oxidation not only caused the disintegration of the highly cross-linked polymer binder, but also promoted the deposition of geothermal brine-induced scale on the surface of the SiC-filled polymer liner. The strongly bonded Ba-carboxylate hydrolysates formed at the interface between the liner and the scale produced a scale that was difficult to remove.

Accordingly, a primary criterion for polymeric materials used in the coatings and liners at temperatures ranging from 90ºC (194°F) to 110ºC (230°F) is resistance to the oxidation reaction with brine, thereby minimizing the rate of scale deposition. To achieve this goal, two polymeric materials—antioxidant (PDA)-modified ST-TMP and polyphenylenesulfide (PPS)—were evaluated for use as corrosion- and fouling-resistant liners and coatings.

1

Gawlik, et al.

In addition, it has been found that the surface modification of metals can improve the adherence of liners and coatings to the steel substrates and can reduce the rate of corrosion and wear of the steels (Sugama and Carciello, 1992). To this end, a zinc phosphate (ZP) ceramic precoating was deposited directly on the steel surfaces for use as an interfacial tailoring and modification material.

The coatings systems were evaluated in the laboratory and in the field. In the laboratory, the coatings were tested on carbon steel coupons in a low pH, hypersaline brine and found to have improved oxidation resistance compared to previously used coatings. After the laboratory evaluation, the coatings were put into service as carbon steel tube liners in brine-wetted heat exchangers at the Hoch power plant in the Salton Sea Geothermal Area. They were installed in an apparatus that was originally built by Idaho National Engineering Laboratory (INEL) and used for testing BNL's liner systems in the early 1990s. More information on this apparatus is at a later point in this paper. The field evaluation consisted of testing the tubes for 45 days in a highly aggressive brine environment. The results from the test apparatus show that the antioxidant has been effective in reducing the fouling rate of the tubes by up to 19%.

Properties of polymers and coatings

Information on the thermal characteristics of ST-TMP, PDA-modified ST-TMP, and PPS polymers was obtained using the combined techniques of thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) in air. The results of these tests are given in Table 1.

Table 1. *Thermal Properties of Polymers*

System	Type	Melting Point, $^{\circ}$C	Crystalliza-tion point on cooling, $^{\circ}$C	Onset temperature of decomposition, $^{\circ}$C
ST-TMP	Thermo-set	_	_	270
PDA-mod. ST-TMP	Thermo-set	_	_	230
PPS	Thermo-plastic	280	202	380

Two of the polymer systems, ST-TMP and PDA-modified ST-TMP, are thermoset materials composed of a cross-linked polymer network structure, so that they do not have melt characteristics. In contrast, the PPS polymer is a thermoplastic; i.e., it can be remelted many times as a recyclable polymer. The major characteristic of PPS is the molecular orientation caused by chain extension at its melting point of 280°C (536°F). This orientation causes crystallization of the molten polymer during cooling when the crystalline point is reached at 202°C (396°F). The semicrystalline PPS polymer has excellent thermal stability, as indicated by the fact that the onset of thermal decomposition begins around 380°C (716°F). In contrast, the thermal decomposition of the ST-TMP and PDA-modified ST-TMP polymers start near 270°C (518°F) and 230°C (446°F), respectively.

The overall appearance of the coated steel panels (7 by 7 cm, or 18 by 18 in.) after exposure for up to 21 days in low pH, hypersaline brine (1 wt% H_2SO_4, 13 wt% NaCl, and 86 wt% water) at 200°C (392°F) was examined to determine the hydrothermal stability and ability of the coating films to protect the underlying steel against corrosion (Table 2).

Table 2. *Visual Observation of Coating Films as a Function of Exposure Time at 200°C*

Coating System	Exposure time, day			
	1	3	14	21
ST-TMP/ZP/steel	good	good	good	good
PDA-ST-TMP/ZP/steel	good	good	good	good
PPS/ steel	good	blister	peels	--
PPS/ZP/steel	good	good	good	good

All of the coatings, except the PPS/steel system, exhibited no visual signs of delamination and damage after 21 days of exposure. The underlying steel in the "good" cases showed no corrosive attack at all. When the PPS coating was deposited directly on the steel substrate, in the absence of the zinc phosphate precoating, blistering was observed after only 3 days of exposure. Extending the exposure time to 14 days caused the film to peel from the steel substrate. These results indicated that the zinc phosphate precoating contributed significantly to improving the durability of the bond at the interface

between the coating and the steel substrate, and in providing increased protection of the steel against corrosion during exposure to a wet, harsh environment. X-ray photoelectron spectroscopy (XPS) analysis for 0-, 3-, 14-, and 21-day exposures was conducted to determine the degree of the oxidation of the coatings. Figure 1 illustrates the changes in the atomic ratio of oxygen/carbon (O/C) for the coating surfaces before and after exposure in an autoclave to low pH, hyper saline brine at 200ºC (392°F).
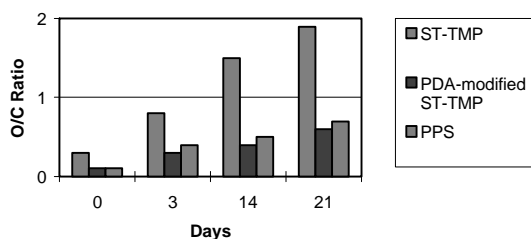

Figure 1. *Changes in O/C ratio for ST-TMP, PDA-modified ST-TMP, and PPS polymer surfaces as a function of exposure time in low pH, hypersaline brine at 200ºC.*

As is illustrated, the O/C ratio for the ST-TMP coating increased with exposure time. The O/C ratio for the ST-TMP system after 21 days of exposure was determined to be 1.8, indicating that the polymer suffered oxidation damage in the hot brine. An O/C ratio under 1.0 is considered acceptable. In contrast, the addition of the PDA antioxidant to the ST-TMP system acted to greatly inhibit the degree of oxidation, as illustrated by the O/C ratio of 0.6 measured after 21 days of exposure. As expected, the PPS surfaces also displayed excellent resistance to oxidation.

Fabrication of HX tubes for field evaluation

Eight heat exchanger tubes were fabricated using 6.0 m (19 ft. 8 in.) lengths of 2.54 cm (1 in.) outside diameter by 1.24 mm (0.049 in.) wall carbon steel tubing. A 5.1 cm (2 in.) long AL-6XN stub was welded to each end of the tube to provide the safe end connection to the tubesheet. This produced a tube with an overall length of 6.09 m (20 ft.), 6.04 m (19 ft. 10 in.) of which was lined or coated. The first 2.54 cm (1 in.) of each stub was left unlined in order to provide a location for roller expanding the

safe end to the tubesheet. Presented in Table 3 is a summary of the tubes fabricated for field evaluation and the locations of the tubes within the test skids.

Table 3. *HX tube liners and coatings selected for field evaluation at the Hoch power plant.*

Leg	Test Skid A
1	PDA-modified ST-TMP/SiC
2	PDA-modified ST-TMP/SiC
3	ZP/PDA-modified ST-TMP/SiC
4	ZP/PPS

Leg	Test Skid B
1	ST-TMP/SiC
2	ST-TMP/SiC
3	ZP/ST-TMP/SiC
4	ZP/PPS/SiC

A combination of techniques was used to apply the various coatings. Common to all applications was careful preparation of the tube interior through degreasing and sandblasting. The zinc phosphate coating is applied using a "fill and drain" technique. The tube is coated by attaching a valve to one end of the tube and then inserting it into a vertically oriented furnace. The coating solution is poured into the tube from the top and then drained from the bottom once the coating process has been completed. The PPS and PPS/SiC coatings are also applied using the "fill and drain" technique. The PPS system consists of two layers of PPS while the PPS/SiC system consists of one layer of PPS and a top layer of SiC-filled PPS. The ST-TMP/SiC and PDA-modified ST-TMP/SiC liners are centrifugally cast inside the tubing. Details of the application techniques are in the Appendix.

The formulations of PPS/SiC and ST-TMP are detailed in Table 4. The ST-TMP/SiC and PDA-modified ST-TMP/SiC systems are polymer composite systems consisting of 18 wt% monomer and 82 wt% SiC grit. The formulation of the PDA-modified ST-TMP monomer system is identical to that of the ST-TMP monomer system with the exception that 0.1 wt% antioxidant is added to the system.

Table 4. *Compositions of PPS/SiC and ST-TMP.*

Composition of the PPS/SiC slurry
45 wt% isopropyl alcohol
36 wt% PPS
18 wt% SiC
1 wt% surfactant

Composition of the ST-TMP monomer system
53 wt% styrene-polystyrene mixture
35.4 wt% TMP
4.8 wt% poly(methyl methacrylate)
4.8 wt% ambient and high temperature initiators
1 wt% silane coupling agent
1 wt% promoter

Field Test Results

The eight tubes were put into service at the Heat Exchanger Test Skid, located at CalEnergy Operating Company's Hoch plant in the Salton Sea Geothermal Area. This area is known for its aggressive brines with high suspended and dissolved solids content. The test skid consisted of two parallel sets of four 6.1 m (20 ft.) long counterflow heat exchangers. Each heat exchanger was made up of one lined, 2.54 cm (1 in.) outside diameter tube surrounded by a shell made of 3.81 cm (1.5 in.) schedule 80 pipe. Brine from the clarifier tank flowed through the tubes, which were cooled on the shell side by water in a closed loop system. The heat exchangers were plumbed so that brine and cooling water flowed in countercurrent directions from one heat exchanger to the next. Figure 2 is a depiction of this apparatus.

The brine was supplied from the clarifier tank at $110^{\circ}C$ ($230^{\circ}F$) and pumped to the test skid with a single pump. Flow rates through the two sets of heat exchangers were sensed with magnetic flowmeters and controlled by globe valves at the
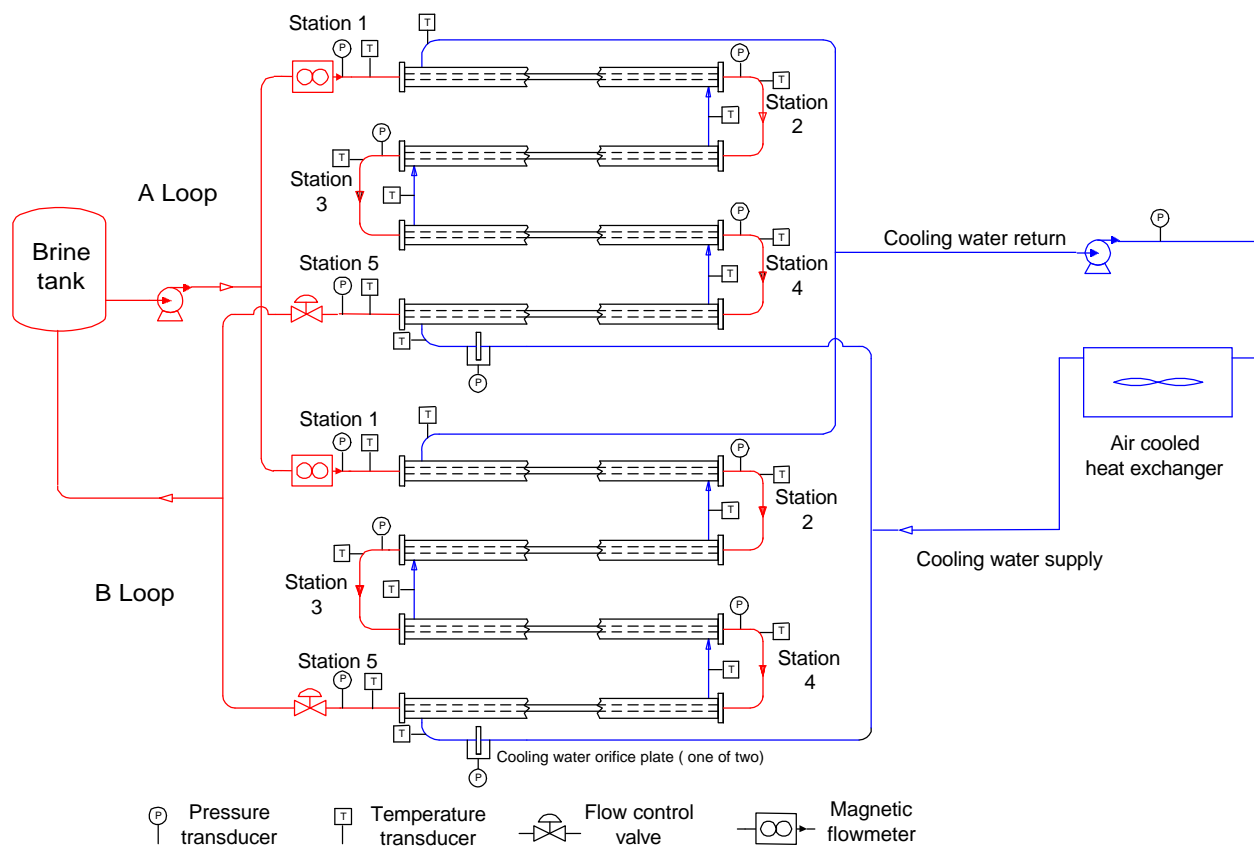
Figure 2. *Schematic of the Heat Exchanger Test Skid.*

exit of each loop through individual proportional-integral-derivative control systems. Total dissolved solids levels were approximately 140,000 ppm entering the skid.

The objective of the test program was to obtain 45 days of good data. From the results of previous tests, this amount of data was considered sufficient to evaluate liner performance. The data collection was not continuous, though, because of a number of problems that interrupted the test, which lasted from September 1997 to April 1998. Also, the tubes in one loop have about 100 more hours of total test time than the other loop. The difference is due to temporary loss of brine flow in one loop when a tube on that side of the rig completely plugged near one of its ends.

The brine flowrates were not constant during the test. Initially, the flowrates through each loop were 0.63 liters/sec (10 gpm), but as the tubes scaled, it became increasingly difficult to maintain these flowrates. When the flows dropped below 0.06 liters/sec (1 gpm) on each side, the rig was shut down to remove scale, which often accumulated at the entrances and exits of the tubes.

The apparatus was periodically shut down for other maintenance as well. The brine pump impeller and the brine piping upstream and downstream of the heat exchangers were hydroblasted and mechanically cleaned numerous times. The welds between the AL-6XN safe ends and the carbon steel tubing on two tube assemblies cracked and developed leaks, which led to failure of the cooling system expansion tank. Rough handling during installation and weakening of the welds by roller expansion performed near them presumably caused the cracking. The tubes were repaired and returned to service. Failure of the phone line to the data acquisition system and scheduled plant shutdowns also delayed completion of the test. In response to problems with sticking of the brine valves in earlier tests, a scheme was developed to stroke the brine valves through their complete range of motion every three hours. This was effective in largely preventing valve sticking.

The following figure shows the brine flowrates through the two loops, designated A and B. These data are for periods in which both the brine and

coolant systems were operating normally. The data do not show a smooth transition from a nominal 0.63 liters/sec (10 gpm) flowrate to a reduced flow because there was a period of operation when the brine was flowing, but no cooling water was available. The data from this period were not used.
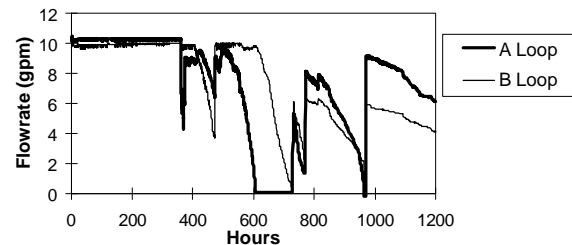

Figure 3. *Brine flow history for both loops.*

As seen in the plot, the A side experienced a prolonged down period when one of the tubes on that side plugged completely. The B side remained open, however, and brine continued to flow through the apparatus. The jumps from a low to high brine flow represent occasions when the apparatus was taken out of service to clean the pump impeller and remove restrictions in the tubes or brine piping.

The heat exchangers were instrumented to provide data on the pressure drop through the tubes, temperature change of the brine and cooling water through each heat exchanger, and brine and cooling water flowrates. With this information, one could calculate the increase in resistance to heat flow through the lined tubes as scale formed in the interior. This additional resistance to heat flow is defined as the fouling coefficient, or

$$R_f = \frac{r_2}{r_4}\left[\frac{1}{U_0} - \frac{1}{h_0} - \frac{r_4}{r_1}\frac{1}{h_i}\right] - \frac{r_2}{k_{steel}}\ln\left(\frac{r_4}{r_3}\right) - \frac{r_2}{k_{liner}}\ln\left(\frac{r_3}{r_2}\right)$$

where r_1 is the inner radius of scale buildup; r_2, inner radius of the liner; r_3, the inner radius of the tube; r_4, the outer radius of the tube; h_i, the tube interior heat transfer coefficient; h_o, the heat transfer coefficient on the tube exterior; U_0, the overall heat transfer coefficient from brine to water; k_{steel}, the thermal conductivity of the carbon steel tube; and k_{liner}, the thermal conductivity of the liner system. The geometry is illustrated in Figure 4.
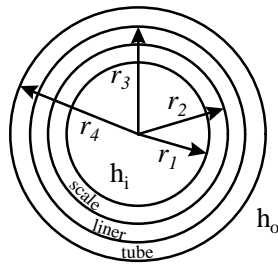
5

Gawlik, et al.

Figure 4. *Definition of system used in R_f eqn.*

The values of the heat transfer coefficients were determined from well-known correlations. The overall heat transfer coefficient was calculated from the data at the beginning of the test. The conductivity of the carbon steel was taken from handbook data and of the liner, from the test data at the beginning of the run when the tubes were clean. The thicknesses of the liners were determined from x-ray photographs of the tubes before they were installed in the apparatus.

The results show a reduction in fouling due to the presence of the antioxidant. The fouling coefficients for one of the PDA-modified ST-TMP/SiC-lined tubes and a plain ST-TMP/SiC-lined tube are shown in Figure 5. For these tubes, the reduction in fouling due to the presence of the antioxidant is 15%; this calculation was made at the 1080 hour point (the end of the 45-day test period) for both tubes. It should be noted that this plot does not represent continuous data collection. There were many periods when the brine flow was lost or became so low that the calculation of R_f became highly inaccurate.
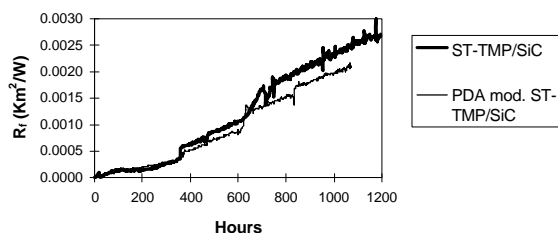

Figure 5. *Performance of the ST-TMP and PDA-modified ST-TMP liner systems.*

The jump in R_f at the 630 hour point is due to a period when brine was stagnant in the tube after that side of the apparatus plugged. Note that there are about 100 more hours of operating time on the ST-TMP/SiC tube because it was installed on the side of the apparatus that maintained brine flow after the A side plugged.

There is also available a side-by-side comparison of PDA-modified and plain ST-TMP/SiC liners applied over the zinc phosphate coating. The purpose of the zinc phosphate coating is to increase the liner-to-tube bond strength, and its effectiveness will be tested when the tube interiors are hydroblasted. The coating has no effect on the fouling rate. But comparison of the results for these two liner systems (Figure 6) confirms the effectiveness of the PDA addition. As for the liner systems that do not include the zinc phosphate coating, there is a reduction in fouling when the antioxidant is used. In this case, the reduction is 19%.
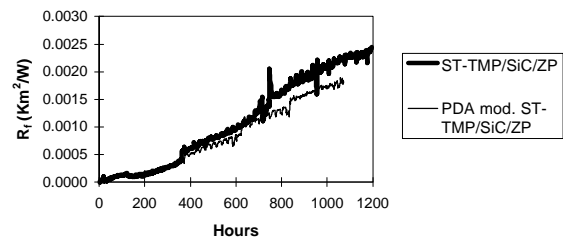

Figure 6. *Performance of the ST-TMP and PDA-modified ST-TMP liners applied over zinc phosphate.*

A reduction in fouling can be used to extend the time between cleanings, so fewer cleanings per year will be required to maintain a heat exchanger made using these materials. A fouling reduction of 19% translates to an equivalent reduction in annual cleaning costs.

The fouling rates of the PPS-based systems were not significantly affected by the addition of SiC. Shown in Figure 7 are the fouling rates for the PPS and PPS/SiC liners, both applied over zinc phosphate. While for a period near the 800 hour point the PPS liner had a fouling coefficient approximately 13% less than the PPS/SiC liner, for the remainder of the test before and after this point the amount of fouling was the same for the two liners.
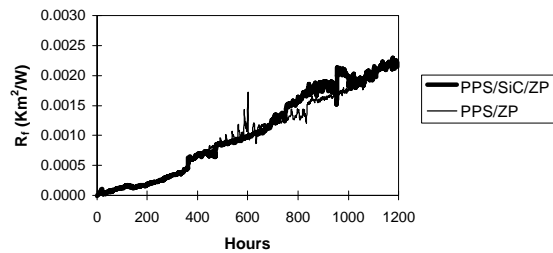
Figure 7. *Performance of the PPS liner systems.*

The use of SiC in PPS greatly enhanced heat transfer through the coating. Initially, when the tubes were clean, the overall heat transfer coefficient for the PPS/SiC-lined tube was 55% greater than the PPS-lined tube. This was due to a 92% greater thermal conductivity of PPS/SiC over PPS.

The PPS liner fouled to the same level as the PDA-modified ST-TMP/SiC liner. In Figure 8 are shown the results for PDA-modified ST-TMP/SiC and PPS, both applied over a zinc phosphate coating. The fouling rates of the liners at the beginning and end of the test period are similar. There is some difference between the two in the period between the time the skid was temporarily shut down due to the failure of the expansion tank and the point when the flow stopped in this loop. The similarity in amount of scaling supports the link between surface ox-idation characteristics and fouling rate. As shown in the laboratory coupon tests, these two materials demonstrated good surface oxidation characteristics, and they have also been found to have similar low fouling rates.
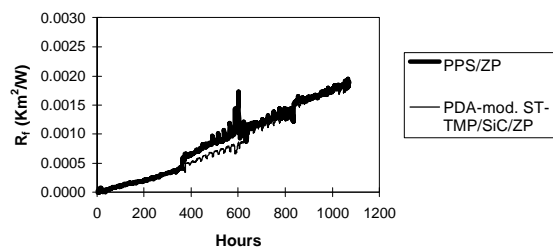

Figure 8. *Comparisons between two liners with antioxidant properties.*

In previous field tests (Hassani and Hoo, 1995), it was shown that plain ST-TMP-lined tubes scaled at the same rate as unlined AL-6XN tubes. Therefore, the advantages of using PDA-modified ST-TMP or

PPS-lined tubes can be assumed to apply relative to unlined stainless steel tubes, too.

Post-field-test evaluations include determing if the tubes may be cleaned without damage to the liners and examining the scale composition and the nature of the scale-to-liner bonds. The tubes will undergo a thorough evaluation of their ability to be effectively cleaned without liner removal by hydroblasting the interiors using a variety of pressures and nozzle styles. BNL will analyze scaled tubes to determine the composition of the scale and the strength of the adhesion between scale and liner and between liner and tube wall.

In addition, new liner systems will be tested during the summer of 1998 with test completion hopefully by fall.

Conclusions

This study's results show that the addition of PDA to the ST-TMP/SiC liner system has reduced that system's rate of fouling in a highly aggressive brine environment 15% to 19%. The PPS and PDA-modified ST-TMP/SiC liners, two systems with demonstrated oxidation-resistant properties in the coupon tests, fouled to the same level, which is low relative to the plain ST-TMP/SiC. The effective-ness of the zinc phosphate substrate will be determined when the liner-to-tube bond strength is evaluated by hydroblasting. The PPS-based systems' results show that the addition of SiC has no effect on the fouling rate but a large effect on improving thermal conductivity.

The fouling reduction results show promise in lowering plant maintenance costs if a brine heat exchanger is made of carbon steel tubes lined with an oxidation-resistant polymer coating. Annual tube cleaning costs may be reduced up to 19% by use of materials tested in this study.

In addition, the use of coated carbon steel can reduce capital costs of brine heat exchangers. As shown in an NREL study (Scholl, 1997), a heat exchanger made of ST-TMP/SiC-lined carbon steel tubes, and using carbon steel tubesheets and channels coated with flame-sprayed metal liner, is 34% of the cost of a heat exchanger made with titanium tubes and monel- and titanium-clad channels and tubesheets.

Gawlik, et al.

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Appendix

In general, the steps in the coating process were as follows: (1) degrease the interior surface of the tube, (2) sandblast the interior surface of the tube, (3) apply the zinc phosphate ceramic precoating, if required, and (4) apply the ST-TMP/SiC, PPS, or PPS/SiC coating system.

The application of the zinc phosphate coating is performed in the following manner: (1) insert the tube into the vertical furnace, (2) preheat the tube to 80°C (176°F), (3) preheat the zinc phosphate solution to 80°C (176°F), (4) fill the tube with the zinc phosphate solution and maintain the filled tube at a temperature of 80°±5°C (176°±9°F) for 30 min., (5) drain the solution from the tube and wash the interior with water which has been preheated to 80°C (176°F), (6) bake the zinc phosphate-treated tube at 125°C (257°F) for 1 hr to prepare the anhydrous zinc phosphate coating.

The application process for the PPS-based liners is as follows: (1) insert the tube into the vertical furnace, (2) coat the interior of the tube by filling it with a slurry of 50 wt% PPS, 49 wt% isopropyl alcohol, and 1 wt% surfactant, (3) drain the slurry from the tube, (4) allow the slurry-coated tube to stand for 1 hr to allow the alcohol to evaporate, (5) preheat the tube to 125°C (257°F) to drive off any remaining alcohol then raise the temperature of the tube to 320°C (608°F) and maintain it at this level for 3 hr to cure and crosslink the PPS, (6) allow the tube to cool to room temperature and then repeat the process to apply a second coat of PPS or a top coat of the SiC-filled PPS.

The application procedure for the ST-TMP/SiC liners is as follows: (1) The tube is inserted into the spinning assembly and locked into position. The monomer/SiC mixture is then poured into the tube and distributed along its length using a screed designed to uniformly distribute enough material along the length of the tube to produce a 0.76 mm (0.030-in.) thick liner. (2) The tube is slowly rotated to allow the mix to fully coat the interior surface. The drive motor speed is then gradually increased to 600 rpm and the tube is spun for 4 hr to compact the liner against the tubing and to allow the liner mixture to take its initial set. (3) Once the spinning has been completed the tube is post-cured using a two-step curing process. The first step involves spinning the tube at 80°C (176°F) for 2.5 hr in order to complete the initial curing of the liner. This is accomplished by placing an enclosure over the spinning assembly and heating the air space within the enclosure. The tube is then removed from the spinning assembly and placed inside a curing chamber where it is cured a second time at a temperature of 175°C (347°F) for 4 hr.

REPORT DOCUMENTATION PAGE

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1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE November 1998	3. REPORT TYPE AND DATES COVERED NREL Conference Paper

4. TITLE AND SUBTITLE

Field Testing of Heat Exchanger Tube Coatings

5. FUNDING NUMBERS

Task #: 55004000

6. AUTHOR(S)
Gawlik, K.; Sugama, T.; Webster, R.; Reams, W

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

National Renewable Energy Laboratory
1617 Cole Boulevard
Golden, CO 80401-3393

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

NREL/CP-550-26210

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT

National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road
Springfield, VA 22161

12b. DISTRIBUTION CODE

UC-600

13. ABSTRACT *(Maximum 200 words)* In order to reduce the capital cost of geothermal power plant heat exchange equipment and the maintenance cost associated with fouling, the National Renewable Energy Laboratory has been conducting test of polymer-base liner systems developed by Brookhaven National Laboratory. These line systems protect low-cost carbon steel tubing from corrosion and are formulated to reduce the rate of fouling. Field test data of the latest liner systems show a fouling rate reduction of 15% to 19% compared to previous liner formulations. New Substrates used between polymer liner and tube are expected to improve the liner-to-tube bond and enhance durability during the cleaning process.

14. SUBJECT TERMS geothermal, polymer-based, liner, fouling, corrosion heat exchanger tubing.

15. NUMBER OF PAGES
8

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT