

Corrosion Testing in Flash Tanks

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As kraft pulp mills adopt modified cooking processes, an increasing amount of corrosion of carbon steel digester systems is being encountered. Many mills have had severe corrosion in the flash tanks, in particular, the first (#1) flash tank. The work described in this report was aimed at characterizing the corrosion. Coupons of carbon steel, several stainless steels and titanium were exposed at two mills. At mill A, identical sets of coupons were exposed in the #1 and #2 flash tank. At mill B, three identical sets of coupons were placed in flash tank #1. The results of the exposures showed that both carbon steel and titanium suffered high rates of general corrosion, while the stainless steels suffered varying degrees of localized attack. The ranking of the resistance of corrosion in the flash tank was the same ranking as would be expected in a reducing acid environment. In the light of the coupon results, organic acids is concluded to be the most likely cause of corrosion of the flash tanks.

INTRODUCTION

As mills move towards modified variations of kraft cooking in an effort to reduce the amount of bleaching chemical that is used to produce pulp of market brightness, an increasing amount of equipment corrosion is occurring, especially in the lower part of the kraft continuous digester and the #1 flash tank. The incidence of flash tank corrosion is widespread and has not yet been related to a specific cooking practice. Table 1 (page 2) shows the experience of some Canadian mills with different fibre furnishes, and different cooking schemes. As part of Paprican's on-going effort to determine the limits of existing pulp mill equipment and to determine the

optimum materials for new processes, some test work was undertaken at two of these mill-sites. Both mills have experienced flash tank corrosion, one of them at an unexpectedly high rate. Because the environment in the flash tank is a foam, and therefore no continuous electrolyte was present, electrochemical testing was not attempted. This report describes some corrosion testing that was undertaken in flash tanks at the two mills.

EXPERIMENTAL METHOD

All coupons were prepared from commercial alloys of known composition. All mill test certificates met ASTM specifications. All coupon faces were ground to a surface

roughness of no greater than 120-grit. All circular coupon edges were machined and square coupon edges were ground to a surface roughness no greater than 120-grit. The weight of each coupon was measured to the nearest 0.1 mg on an analytical balance. The coupons were installed on Teflon™ covered rods and separated by Teflon spacers. One face of each coupon was contacted with a segmented Teflon spacer to determine if the material was susceptible to crevice attack.

Coupons of Grade 2 titanium, 304L, 2304, 2205 stainless steels and carbon steel were exposed in the flash tanks at mill A. Coupons of Grade 2 titanium, 304L, 316L, 2304, 2205 (welded) and carbon steel were exposed in the #1 flash tank only at mill B. The composition of the alloys is given in Table 2 (page 2). In mill A, coupon racks were placed in the upper half of both #1 and #2 flash tank. In the exposure at mill B, coupon racks were placed at the top, middle and bottom of the #1 flash tank only. The exposure at mill A was 120 days and the exposure at mill B was 105 days. The temperatures in the #1 and #2 flash tanks at mill A were 134°C and 118°C respectively. The temperature in the #1 flash tank at mill B was 120°C. All coupons were cleaned before the corrosion rates were calculated. Carbon steel coupons were ultrasonically cleaned in inhibited acid (8 ml 35% 2-butene 1,4 diol, 6 ml 35% HCl, 100 ml distilled H₂O). Stainless steel coupons were ultrasonically cleaned in 1% EDTA.

RESULTS

All coupons were examined in the scanning electron microscope (SEM). Some of the results of these examinations are shown in Figures 1-7 (page 4-5). The results of the coupon exposures at mills A and B are shown in Tables 3 and 4 (page 3) respectively.

Installation of the racks at mill B resulted in deposit of weld spatter on the coupons. This meant that weight losses and the derived corrosion rates could not be determined from most of these coupons. However, where corrosion rates were high, the weld spatter was either consumed or fell off with the corrosion products from the coupons. This resulted in spatter-free coupons from which corrosion rates could be determined. In addition, two of the 304L coupons in this exposure had no weld spatter and so their weight losses were measured. Chemical analyses of the black liquors which enter the flash tanks (the extraction liquors) in mills A and B are shown in Table 5 (page 3).

DISCUSSION

The results of the two sets of coupon exposures show that the carbon steel and titanium experienced general corrosion, the austenitic stainless steels (304L and 316L) pitted and the duplex stainless steels (2304 and 2205) showed very little or no corrosion at all. To interpret the results from the coupon exposures, it is necessary to understand the corrosion resistance that the different alloys have to various environments. The following is a literature survey of the corrosion resistance of the materials that were used in this work to various environments.

Table 1.

Corrosion experience in the #1 flash tank at six Canadian kraft mills, all mills except mill D have carbon steel vessels.

Mill	Startup	Species	Cook	Corrosion Experience
A	1994	Softwood	Lo-Solids	The # 1 flash tank at this mill is severely corroded. Carbon steel weld overlay was applied after two years but was consumed during the subsequent year of operation. This vessel was replaced in 1998. The new vessel is made of 2205.
B	1980	Softwood	MCC	The bottom cone of the flash tank was supplied 19 mm (0.75 in.) thick, 9.5 mm (0.375 in.) pressure envelope plus 9.5 mm (0.375 in.) corrosion allowance. The bottom cone of this vessel had to be replaced after 10 years because the corrosion allowance had been consumed. This represents an average corrosion rate of 0.95 mm/y (37.5 mpy). The mill also has continual problems with the support bars for the umbrella in the center of the flash tank.
C	1991	Softwood	Lo Solids	In 1996 the lower cone was severely attacked. The vertical walls were also significantly attacked. Pits up to 10 mm deep were common. Many of the vertical welds on the cone were even more severely attacked.
D	1989	Softwood	EMCC	This mill has a 304L stainless steel flash tank. To date, there have been no corrosion problems in this vessel.
E	1964	Softwood	Standard	The #1 flash tank was replaced due to corrosion of the shell and internals in 1995.
F	1993	Hardwood	EMCC/Lo Solids	The #1 flash tank has shown some corrosion on the shell. The umbrella supports (carbon steel) are a constant corrosion problem and have to be repaired each shutdown.

Table 2.

Compositions of the materials used in the coupon exposures in the flash tanks. Data copied from mill test certificates.

Alloy	Element (wt%)													
	C	Si	Mn	P	S	Cr	Ni	Mo	Cu	N	Fe	O	H*	Co
A 516 Gr.70 steel	0.14	0.025	0.78	0.01	0.014	-	-	-	-	-	Bal.	-	-	-
304L	0.022	0.48	1.78	0.027	0.006	18.3	9.97	0.3	0.2	0.017	Bal.	-	-	0.28
316L	0.022	0.49	1.57	0.026	0.001	22.6	4.8	0.36	0.28	0.101	Bal.	-	-	-
2304	0.13	0.4	1.52	0.026	0.001	22.1	5.7	2.98	-	0.17	Bal.	-	-	-
2205	0.016	0.46	1.42	0.021	0.001	22.1	5.7	3.01	-	0.15	Bal.	-	-	-
2205 (W)**	0.03	0.48	1.44	0.2	0.001	22	5.7	3.01	-	0.15	Bal.	-	-	-
Titanium	0.02	-	-	-	-	-	-	-	-	0.01	0.06	0.15	0.19	-

** (W) represents welded coupon.

* (ppm)

Table 3.

The corrosion rates of the coupon exposures in the two flash tanks at mill.

Alloy	Flash Tank	Corrosion Rate mm/y	Notes
A516 Gr. 70 Steel	#1	0.345	General corrosion
	#2	0.216	General corrosion
304L	#1	0.0002	Slight pitting and crevice attack
	#2	0.0001	Slight pitting and crevice attack
2205	#1	0	No localized attack
	#2	0	No localized attack
2304	#1	0.0001	Slight pitting and crevice attack
	#2	0.0001	Slight pitting and crevice attack
Grade 2 Titanium	#1	2.25	General corrosion
	#2	0.89	General corrosion

Table 4.

The corrosion rates of the coupon exposures in the two flash tanks at mill.

Alloy	Location in Flash Tank #1	Corrosion Rate mm/y	Notes
A 516 gr. 70 Steel	Top	0.066	General corrosion
	Middle	0.0209	General corrosion
	Bottom	0.226	General corrosion
304L	Top	0.0003	Weld spatter
	Middle	0.0003	
	Bottom	–	
2205	Top	–	Weld spatter
	Middle	–	Weld spatter
	Bottom	–	Weld spatter
Grade 2 Titanium	Top	0.629	General corrosion
	Middle	1.34	General corrosion
	Bottom	1.42	General corrosion
316L	Top	–	Weld spatter
	Middle	–	Weld spatter
	Bottom	–	Weld spatter

Table 5.

Analysis of the black liquor (extraction) from mills A and B, all anions are expressed in terms of their sodium salts.

	Concentration g/l	
	A	B
Na ₂ S	14.2	10.1
Na ₂ S ₂ O ₂	5.75	2.43
NaOH	6.3	4
NaCl	1.02	1.27
pH (1/10 dilution)	12.3	12.3

CORROSION IN CAUSTIC ENVIRONMENTS

Titanium

The corrosion resistance of titanium in alkaline environments is affected both by temperature and pH of the solution. Corrosion rates are acceptable in most caustic environments. For example, in boiling 50% NaOH, commercially pure titanium has a corrosion rate of 0.05

mm/y. Similarly, in boiling 10% NaOH the corrosion rate of titanium is 0.02 mm/y [1]. In 40% NaOH at 121°C, the corrosion rate is 0.13 mm/y, which is generally considered as being on the upper limit of acceptable for a corrosion rate [2]. Corrosion of titanium is influenced by the degree of aeration of the environment, therefore in boiling environments, corrosion rates will be maximized because they are free of oxygen. Aeration tends to reduce corrosion rates and can reduce them significantly due to the formation of a tenacious passive layer on titanium, conversely de-aerated environments (which are also free of other oxidants) allow high corrosion rates because the passive layer cannot form [3]. Titanium can also be rapidly corroded in the presence of hydrogen peroxide at elevated pH. Experience with alkaline peroxide bleaching media show that titanium has very poor corrosion resistance when the pH exceeds about 11, even in the presence of small concentrations of hydrogen peroxide [4].

Carbon steel

Carbon steel can exhibit passive behaviour in a wide range of caustic environments, and is used industrially for handling caustic solutions, including 50% caustic soda, as well as black, white and green liquor. Corrosion rates in these environments depend upon the electrochemical state of the carbon steel. Active corrosion can result in general corrosion rates of several millimetres per year, or in the absence of oxidants and high flow rates, corrosion rates can be acceptable. Passive behaviour of carbon steel in kraft liquors typically results in corrosion rates of between 0.08 and 0.13 mm/y [5]. Because of the ability to form a passive layer in caustic environments, carbon steel was used extensively in the kraft process for several decades. Carbon steel is susceptible to stress corrosion cracking in caustic media, especially at elevated concentrations, temperatures and at stresses approaching the yield stress of the material [6,7]. Carbon steel has been the traditional material of

construction for digester flash tanks since the introduction of the continuous digester. Until recently, this material has been acceptable.

Stainless steels

Austenitic stainless steels generally have excellent corrosion resistance to dilute or cool concentrated caustic media. The 300 series of stainless steels corrode at less than 0.025 mm/y at concentrations up to about 60% and temperatures up to about 120°C. At lower concentrations, higher

temperatures can be tolerated. The apparent stress corrosion cracking (SCC) boundary for caustic cracking of the 300 series stainless steels in 10% caustic (the same nominal concentration as white liquor) is about 160°C [8] (Figure 8, page 5). Both 304L and 316L perform very well when immersed in white, green and weak-wash liquors at 90°C, although 304L performs marginally better than 316L [5]. Both alloys have been used successfully in the kraft process for many years. Chloride ion concentration has little

effect on the general corrosion rate or caustic cracking susceptibility of stainless steel in caustic media and would not be expected to cause pitting of either of the 300 series stainless steels that were used in this set of exposures. The corrosion resistance of duplex stainless steels to de-aerated caustic media is, if anything, better than the austenitic stainless steels. Table 6 (page 6) gives some typical corrosion rates for stainless steels used in this study in a range of caustic environments.

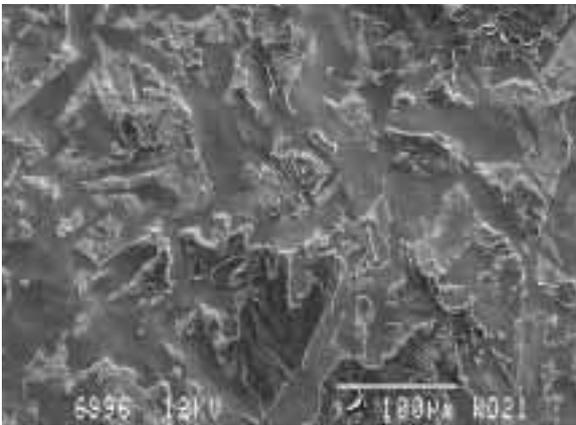


Figure 1.

The carbon steel coupon after 120 days exposure in the #1 flash tank at Mill A. Macroscopically, the sample appeared to be generally corroded. At high magnification, it can be seen that the sample has been etched by the process. Iron carbide is less attacked than the ferrite matrix.

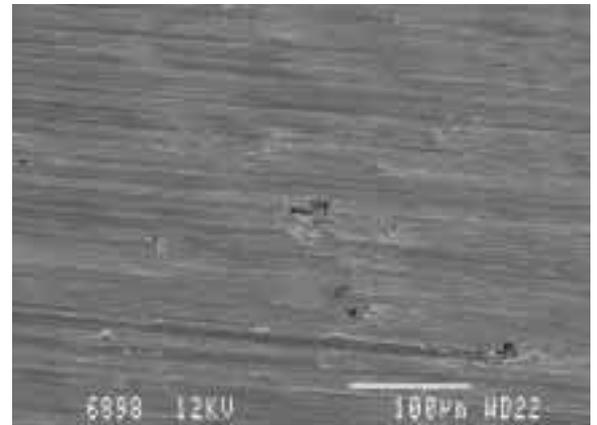


Figure 2.

The 304L steel coupon after 120 days exposure in the #1 flash tank at Mill A. The sample shows many small pits.

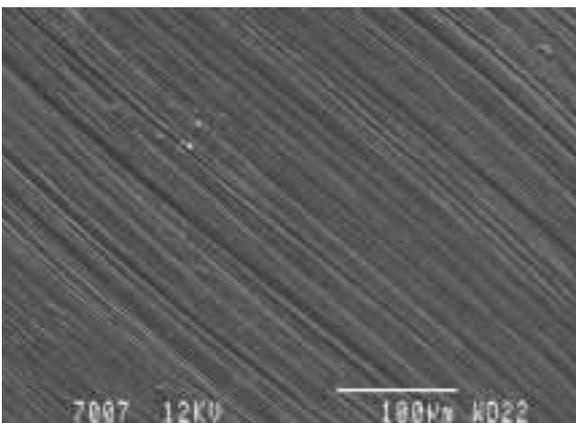


Figure 3.

The 2205 stainless steel coupon after 120 days exposure in the #1 flash tank at Mill A. There is very minor corrosion on this sample. The original grinding marks from when the coupon was prepared are still clearly evident.

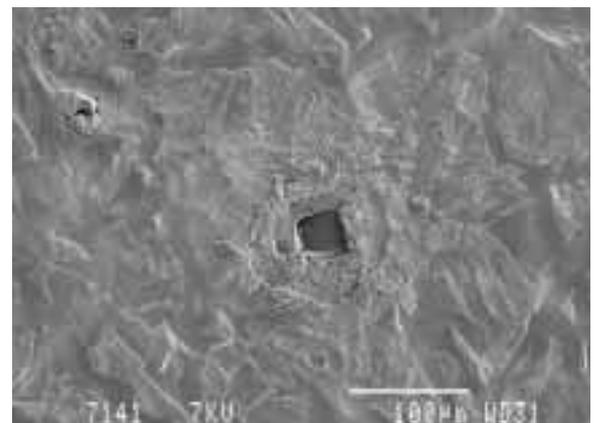


Figure 4.

The carbon steel coupon after 105 days exposure at Mill B. The appearance is very similar to that observed on the carbon steel coupons which were exposed in Mill A.

INORGANIC ACIDS

Titanium

Titanium has variable resistance to inorganic acids. The degree of resistance is largely dependant upon whether the acid is reducing or oxidizing in nature. For instance, titanium is readily attacked in (reducing) hydrochloric and sulphuric acids in de-aerated environments, but has very good resistance to (oxidizing) nitric acid (exposure of titanium to red fuming nitric acid is extremely

hazardous) [2,13]. Attack by reducing acids results in general corrosion. Corrosion rates in dilute reducing acids can be of the order of tens of mm/y and corrosion rates increase with concentration and temperature [13].

Carbon steel

Carbon steel is readily attacked by a wide range of dilute inorganic acids and may passivate in concentrated acid. This however, depends on the type of acid used. For example, carbon steel is rapidly corroded in hydrochloric and

nitric acids but passivates in concentrated sulphuric acid, and can be used to handle ambient temperature, static-or slow moving, concentrated sulphuric acid [14].

Stainless steels

The stainless steels that were used in this study can be attacked by inorganic acids. The factors which affect the nature of the attack are: acid anion, temperature, concentration (pH), velocity, chloride ion concentration, the extent of aeration of the acid and how

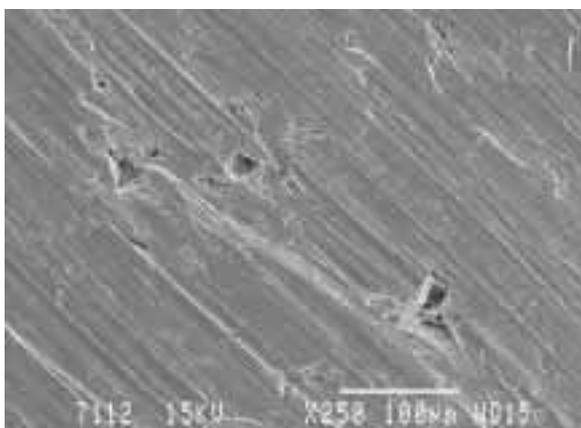


Figure 5. 304L coupon after 105 days exposure at Mill B. Small, established pits can be seen. This type of pitting was seen over the entire surface of the coupon.

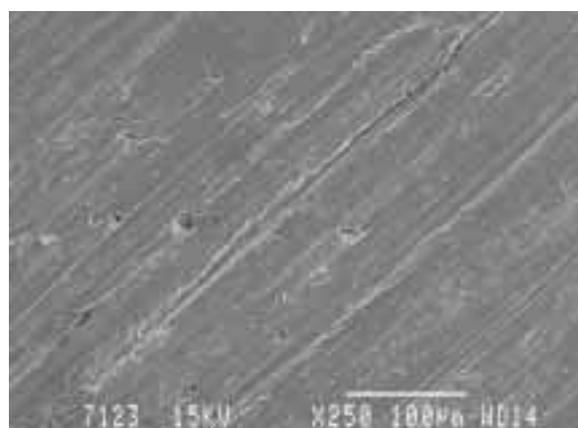


Figure 6. 316L coupon after 105 days exposure at Mill B. Pitting is evident although it is less severe than on the 304 coupon which was exposed to the same conditions.

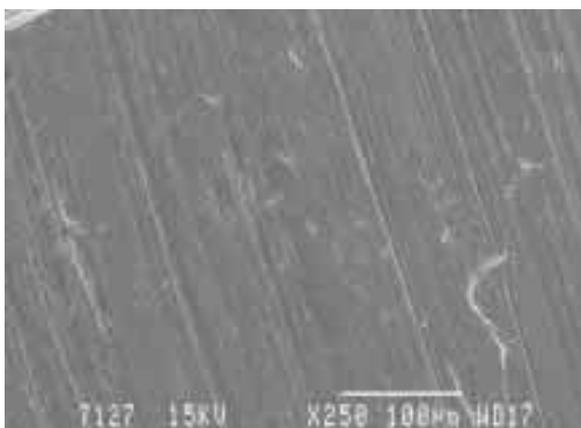


Figure 7. The 2205 coupon after 105 days exposure at Mill B. There is very little attack on the surface. No pits have become established. The resistance of the material to this environment is good.

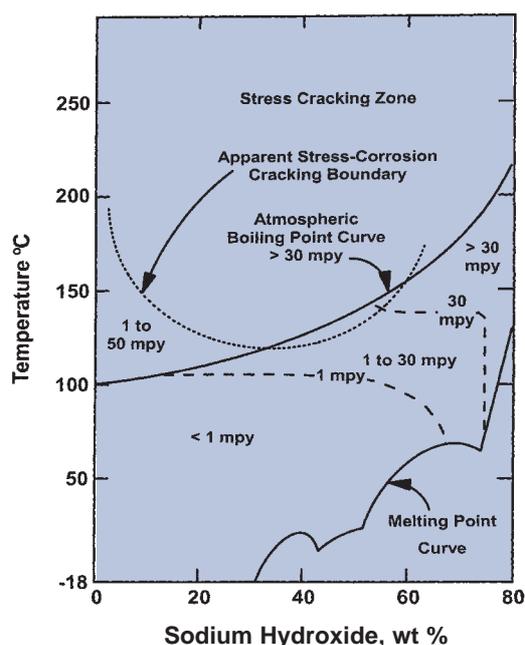


Figure 8. Isocorrosion diagram for 300 series stainless steels (304L/316L) in pure caustic solution [8].

highly alloyed (with Mo, Cr and N) the stainless steel is. For this reason, 304L is the least resistant to corrosion by acids, 316L has better resistance to acids due to its molybdenum content. Similarly, the duplex stainless steels, while having better resistance than comparable austenitic stainless steels, have increased corrosion resistance with increasing Mo, Cr and N contents. Hydrochloric acid is one of the more aggressive inorganic acids. All stainless steels have limits of concentration and temperature in this acid. Similarly, acidic environments (such as sulphuric acid) which are contaminated with chloride are notably more corrosive than the uncontaminated acid [11].

Stainless steels in chloride-bearing acidic environments are likely to undergo pitting corrosion, or crevice corrosion may occur under deposits where the local environment is more severe than that of the bulk. The ranking of resistance to acidic environments for the stainless steel used in this study is; 304L, 316L/2304, 2205.

ORGANIC ACIDS

Titanium

The resistance of titanium to organic acids depends largely on which acid is being considered. Titanium has good resistance to many organic acids and is

used industrially in the production of acetic, adipic and terephthalic acids. Oxalic acid is a good example of an organic acid which is corrosive toward titanium. Titanium is corroded by oxalic acid over a wide range of concentrations and temperatures (Table 7). Most other organic acids require high concentrations to cause corrosion of titanium. For example, boiling 50% citric acid produces a corrosion rate of 0.35 mm/y whereas 25% formic acid at 100°C produces negligible corrosion of titanium [12]. Compared to these, oxalic acid is extremely corrosive toward commercially pure titanium. Propionic acid vapours at 190°C are extremely corrosive toward titanium [1].

Table 7.

Corrosion of titanium in oxalic acid solutions.

Oxalic Acid Concentration (wt%)	Corrosion Rate (mm/y)		
	35°C	60°C	100°C
0.5	0.14	2.4	2.1
1.0	0.15	4.5	21
5.0	0.13	9.3	30.5
10	0.01	11.43	31.5
25	–	11.9	49.4

Carbon steel

Carbon steel has little resistance to acetic or formic acid over a wide range of concentrations and temperatures. Similarly, oxalic acid is corrosive toward carbon steel unless it is aerated, when the carbon steel may passivate under some conditions [15]. Otte and Skinner have shown that the corrosion of carbon steel in fatty acids varies with concentration, chain length and temperature [16]. In fatty organic acids with chain lengths ranging from two carbon atoms (acetic acid) to sixteen carbon atoms (palmitic acid) at 90°C, corrosion rates were highest for acetic acid, when the corrosion rate was 8.1 mm/y. At 250°C, the highest corrosion rate was measured in decanoic acid (ten carbons). The corrosion rate in this case was 90 mm/y. The data shows that at flash tank temperatures of about 135°C, the most corrosive acid is likely to be in the chain length range C3 to C6 (propionic, butyric or valeric acid).

Stainless steels

Stainless steels have variable resistance to organic acids. Stainless steels are attacked by oxalic acid. Boiling 5% oxalic acid corrodes both 304L and 316L at between 1.1 and 11 mm/y. Raising either the concentration or the temperature results in higher corrosion rates on these alloys [17]. The corrosion of stainless steel in acetic acid depends

Table 6.

Corrosion of stainless steels in a range of caustic environments.

Alloy	Concentration NaOH (vol%)	Temp.°C	Corrosion Rate (mm/y)	Comments	Ref.
304L	5	Room	0.0025	–	9
304L	10	87	0.005	+12% NaCl, aerated	10
304L	14	87	0.003	No aeration, static	10
304L	18–22	37–104	0.003	Aerated, rapid agitation + mercaptans and cresolates	10
316L	20	152	0.003 max.	Plastic distillation service; no aeration; rapid agitation. Plus 2–7% KOH, still pot in cracking column	10
2304	70	130	0.1–1	–	11
2304	10	108	<0.1	20–25% NaCl	11
2205	10	108	<0.1	20–25% NaCl	11

on the degree of aeration. Aeration increases the corrosion rate of stainless steels. Molybdenum-bearing grades are superior to the molybdenum-free grades. 316 stainless steel corrodes at about 0.1 mm/y in boiling 80% acetic acid, whereas the corrosion rate of 304 in a similar environment approaches 1 mm/y. Pitting is more likely in formic acid than in acetic acid, especially at higher temperatures. Molybdenum bearing grades have superior resistance to these acids [18]. Both 304L and 316L are susceptible to corrosion in propionic acid. For example, in 100 % propionic acid at 145°C, 304L corrodes at 1 mm/y while 316L corrodes at 0.3 mm/y [10].

SUMMARY

While corrosion of the materials used in the coupon exposures is possible in caustic environments, high caustic concentrations (greater than 40%) and temperatures greater than 120°C would be required to produce significant corrosion on either carbon steel or titanium. The analysis of the extraction liquor from mill A shows that the total caustic concentration was 6.3 g/l as NaOH, which is too low to cause significant corrosion on either carbon steel or titanium at the flash tank temperature of about 120°C. Corrosion of stainless steels in caustic environments at temperatures below about 160°C usually occurs as general thickness loss. The stainless steels coupons in the flash tanks have suffered pitting, which is inconsistent with corrosion in caustic media.

Corrosion in an acidic environment is a more logical explanation for the corrosion that was observed in both flash tanks because there was general corrosion on both the carbon steel and titanium and pitting of the stainless steels. The stainless steels with the greater pitting resistance in acidic environments (eg 2205) had the best resistance to the flash tank environments at both mills.

From the data we have collected so far, it appears that the corrosion mechanism in the flash tank is caused by an acidic environment, with organic acids being the most likely corrodents or constituents of the corrosive agent. It also appears that the caustic concentration in the flash tank is too low to maintain a stable passive film on the carbon steel. Once passivity is lost, corrosion by the organic components in the flash tank environment proceeds. Pitting of the low grade austenitic stainless steels can be attributed to the measurable chloride content in the black liquor that is fed to the flash tanks.

CONCLUSIONS

- Carbon steel is unsuitable for fabrication of kraft digester flash tanks in digesters, which operate modified cooks.
- Low grade austenitic stainless steels such as 304L or 316L are marginal for flash tank service.
- Duplex stainless steels, which contain 22% Cr (UNS S31803/UNS S32205) performed well in the two exposures described in this report.
- The corrosion mechanism in the flash tanks is likely due to the presence of organic acids in the flash tank. Lower temperature, lower flow rate and less volatile organic compounds in the #2 flash tank are likely reasons why the corrosion in this vessel is usually less than in the #1 flash tank.

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