Bleach Plant Materials Selection

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ABSTRACT

The paper is a practical review of materials selection in the bleach plant. It briefly covers oxygen delignification, and the alkaline extraction and chlorine dioxide stages along with acids used for pH control. The focus is on metals though for chlorine dioxide bleaching, non-metals are an important consideration. Bleach plant corrosivity varies by mill and even over time within a single mill depending on process changes. For any significant process change or new equipment installation it is advisable to carry out corrosion testing in advance to ensure materials with sufficient resistance are selected. As an example, results of several field coupon tests are presented and show that duplex stainless steels are a good choice for a variety of bleach plant environments including low residual chlorine dioxide solutions.

INTRODUCTION

This paper presents a summary of materials selection for the bleach plant. There are several good references that cover bleach plant materials and processes in more detail than is discussed here. The first is “Stainless steel and Specialty Alloys for Pulp, Paper and Biomass Conversion” [1]. The second is “Corrosion of Plastics and Rubber in Process Equipment – Experiences from the Pulp and Paper Industry” [2]. The latter provides extensive coverage of FRP applications. Additionally, the damage mechanisms described here are documented in WRC Bulletin 488 Damage Mechanisms Affecting Fixed Equipment in the Pulp and Paper Industry [3].

The nominal composition of the alloys mentioned in this paper is shown in Table I. Each one is assigned to an alloy type or family. Historically 316L, 317L and the super austenitic stainless steels were the primary iron-based alloys used in the bleach plant. Today the wide selection of duplex stainless steel increases the choices available for corrosion control.

Table I. Nominal composition of alloys mentioned in this paper.

Alloy	UNS number	%Cr	%Ni	%Mo	Other Elements	Alloy Type
316L	S31603	16.0	10.0	2.0	N	Austenitc stainless steel
317L	S31703	18.0	11.0	3.0	N	Austenitc stainless steel
Alloy 20	N08020	20.0	33.0	2.2	3.0 Cu, Nb	Austenitc stainless steel
254SMO	S31254	20.0	18.0	6.1	Cu, N	Super austenitic stainless steel
AL6XN	N08367	20.5	24.0	6.3	Cu, N	Super austenitic stainless steel
2101	S32101	21.5	1.5	0.3	5.0 Mn, N, Cu	Lean duplex stainless steel
2304	S32304	23.0	4.0	0.3	Mo, Cu, N	Lean duplex stainless steel
2003	S32003	21.0	3.5	1.6	N	Low Mo duplex stainless steel
2205	S32205	22.0	5.0	3.0	N	Duplex stainless steel
CD4MCU	J93370	25.0	5.0	2.0	3.0 Cu	Cast duplex stainless steel
Zeron 100	S32760	25.0	7.0	3.5	W, N	Super duplex stainless steel
2507	S32750	25.0	7.0	4.0	N	Super duplex stainless steel
2707	S32707	27.0	6.5	5.0	B	Hyper duplex stainless steel
G30	N06030	30	46	5.5	15.0 Fe, 2.5 W	Nickel chromium iron alloy
625	N06625	21.5	61.0	9.0	4.0 Fe	Nickel alloy
C276	N10275	15.5	57.0	15.5	5.5 Fe, 4.0 W	Nickel alloy
Ti Gr 2	R50400				Ti	Titanium

OXYGEN DELIGNIFICATION

Oxygen delignification can be considered the end of brownstock washing but material selection was initially driven by proximity to the bleach plant and the concept that the reactor may be exposed to highly oxidizing conditions and/or lower pHs. 316L rather than 304L was chosen and with the reality that the internal pH has remained high, it is very resistant on the process side. Externally though, 316L oxygen reactors experienced external under insulation stress corrosion cracking (SCC) which caused the vessels to leak. One example is covered in more detail in reference 4. After several similar incidents in the industry, suppliers switched the material of construction to 2205 duplex stainless steel. Given the mild internal environment and now that lean duplex is available in plate form, it has become the preferred choice.

Under insulation SCC occurs at temperatures between 60 and 150 ºC (140 and 300 ºF) and stainless steel is most susceptible to cracking around 95 ºC (200 ºF). It is possible that insulated stainless steel vessels elsewhere in the bleach plant could experience this type of damage, however, it has not been reported in the literature.

EXTRACTION STAGE

The alkaline extraction stage is a mild environment because of the high pH conditions. Historically 316L and 317L have been used and could still be selected today. As with oxygen delignification, for both internal and external corrosion resistance, this stage is well suited for duplex stainless steel, either lean or standard grade 2205.

Alkaline peroxide used for bleaching is corrosive to titanium. This is mainly a concern when considering total chlorine free bleaching using existing titanium equipment. The damage is primarily in the form of general corrosion though pitting can occur. Welds are often preferentially attacked. The higher the pH and temperature the higher the corrosion rate. Process contaminants can act as inhibitors, so it is always essential to field test titanium before exposure.

CHLORINE DIOXIDE STAGE

Chlorine dioxide is one of the most aggressive environments in a pulp mill. However resistant materials are readily available so corrosion can be well controlled. Even so, suitable materials, particularly metals, are relatively high in cost so some care must be taken with D-stage material selection.

ClO_2 produced by the generator is about 10 g/l. Titanium is the only metal that is resistant at this concentration. Once injected into the bleach plant, reactions are so fast that the ClO_2 residuals drop to levels that allow for the use of highly alloyed stainless steel. Rough limits of residual chlorine dioxide are 25 mg/l for 2.6% molybdenum (European grade) 316L and 50 mg/l for 6% Mo stainless steel at 65 ºC (150 ºF) [5].

Metals

Pitting and crevice corrosion of stainless steel in acidic, chloride solutions containing bleaching oxidizers is the damage mechanism associated with chlorine dioxide. Visually the damage appears as pitting, rust may or may not be present. Crevices are highly susceptible to corrosion. If the residuals are high enough bare plate, absent of welds, deposits or crevices will pit as well. Figure 1 shows a 317L stainless steel E/O repulper that was using recycled D2 filtrate (50 – 80 mg/l residual ClO_2 at 71 ºC (160 ºF)). The rotor and trough are experiencing localized pitting and crevice corrosion under the D2 inlet pipe.

317L has been considered the minimum grade of stainless steel that can be used in ClO_2. It now has limited use due to the popularity of duplex stainless steels. As will be shown later in this paper, 2205 has superior corrosion resistance. The 6% molybdenum super austenitic stainless steels do very well in ClO_2 filtrate and all grades in this family have equivalent resistance. The two most used in North America are AL6XN and 254SMO. Tunnicliffe showed that for the given process conditions super duplex stainless steels have near equivalent or superior resistance to the super austenitics [6]. Based on his study the alloys were ranked 2205 > Zeron 100 > 254SMO > 2507 > 2707 in order of increasing resistance. With the exception of 2707 which is only produced in tube form, all the other materials have been used successfully in the bleach plant.

Figure 1. A 317L E/O repulper exposed to D2 filtrate (top) and detail of pitting on the bare trough metal below the inlet pipe (bottom).

Corrosion resistant alloys depend on oxygen to replenish the protective passive layer that gives the metal its resistance. If the solution is reducing or there is too little oxygen to maintain the passive layer, these alloys will corrode. This is known as active corrosion and a common example is dilute sulfuric acid corrosion of 316L stainless steel. In the range that these alloys are passive, pitting is possible in crevices and under deposits, anywhere where stagnant conditions deprive the passive layer of oxygen. If there is too much oxygen, the passive layer is dissolved and corrosion can occur on bare metal away from crevices. This is called transpassive corrosion and a good example is the corrosion of nickel alloys in chlorine dioxide when the pH is above 4. In this instance, it is also called near neutral ClO_2 corrosion of nickel alloys. When it occurs, the result is often uniform thinning. Figure 2 shows a corroded part from a D1 stage mixer. The flat machined face exposed to chlorine dioxide has roughened and turned blue.

For this reason, nickel alloys are not used to a great extent in bleach plants even though they are known for having good acid resistance in other applications. There are two situations however, in which nickel alloys are still considered. The first is with instrumentation. Material selection is often limited because they are assembled parts in which suppliers for their own efficiency stock the fewest materials that cover the most situations. As an example, the choice for a D stage level indicator may come down to 316L or C276. In this case the latter, a nickel alloy, is the best choice.

The second situation arises when choosing welding fillers for super austenitic stainless steel. Since there are no matching fillers, nickel alloys are used. The guidance in Reference 1 is to use C276 rather than 625 for pHs below 4 and G30, a nickel-chromium-iron alloy, when the pH is above 4.

Figure 2**.** C276 D1 mixer corrosion. The blue coloring is caused by corrosion.

The chlorine dioxide bleaching process has not markedly changed in the last twenty years. Improvements have been made to make it more efficient with the result that it is not as corrosive as it once was [7]. Nevertheless, there is a benefit to conducting field coupon testing as it will allow mills to optimize material selection and make better use of duplex stainless steel for their specific process.

Non-Metals

Non-metals have wide applicability in the bleach plant because of their all-round resistance to corrosive chemicals, and chlorine dioxide in particular. Hinely mentions four classes of non-metals that are useful [8]:

1. Fiberglass Reinforced Plastics (FRP)
2. Thermoplastics
3. Monolithic Resin-based Linings
4. Chemical-Resistant Masonry (Tile/Brick)

The primary damage mechanisms affecting these materials are oxidative, mainly ClO_2, attack of organic/polymer resins and tile lining degradation.

FRP is an engineered material and on a basic level is made up of a chemically resistant thermosetting polymer resin reinforced with glass fibers for strength. FRP is the alternate to titanium as a structural material for handling chlorine dioxide. It is used for storage tanks and piping in the bleach plant. It can also be used as a liner itself or as a structural support for thermoplastic liners. Resin selection is based on the chemical it will be exposed to. As with other organic materials they are ultimately limited by temperature. Because these resins degrade over time, FRP equipment is designed to last for a specific number of years and 15 is not unusual before repairs are required.

Thermoplastics, unlike thermosetting resins used in FRP, are moldable upon heating. This class of materials includes fluoropolymers which according to TIP 0402-37 [9] "have unique properties among thermoplastic including outstanding chemical resistance to higher temperatures". Several types of fluoropolymers used in ClO_2 service in order of increasing temperature resistance are PVDF, ECTFE and PTFE. They have poor mechanical properties so are often used as a lining. PVDF lined pipe is commonly used for ClO_2 piping. ECTFE is used as a liner in dual laminate FRP tanks and piping. PTFE which is resistant to most chemicals is also the most expensive with the poorest mechanical properties. It is used for gasketing across the pulp mill and as a liner when its temperature resistance is needed. Other thermoplastics such as CPVC have lower temperature resistance than the flouoropolymers.

Monolithic resin-based linings use the same thermoset resins as for FRP. It is a relatively thick coating, trowelled on steel, concrete, brick and tile. As with any coating, the quality of the initial application determines its lifespan.

Acid-resistant masonry linings with resin mortar are used to protect steel and concrete substrates in towers, tanks, washer vats, etc. [1]. Brick and tile linings have been used for over one hundred years. However, in ClO_2 service they are maintenance intensive in terms of inspection and ongoing lining repair [10]. In recent years it has been reported that bleach towers have been successfully lined with super duplex 2507 metal sheeting along with the outcome of reduced maintenance [6].

ACID CORROSION

Acids are used for chemical cleaning/scale removal and pH control. For chemical cleaning, given a short duration, the correct concentration, temperature, inhibitor, and post cleaning water rinse, corrosion is usually not a problem. However, for pH control in the bleach plant there are several situations in which acid corrosion is a concern.

Sulfuric acid

Sulfuric acid resistance is defined by concentration, temperature and velocity [11]. Figure 3 shows a corroding alloy 20 pump casing. This alloy was designed for sulfuric acid and even though the acid concentration and temperature were within the alloy's tolerance, the velocity was too high. Carbon steel is used for concentrated sulfuric acid. Once it is diluted the acid becomes very corrosive to carbon and stainless steel. Upstream of dilution, piping material is often switched to PVDF line steel pipe. If the reaction temperature is too high, PTFE line pipe may have to be used instead.

Titanium has no resistance to sulfuric acid. However, the acid has been successfully injected into Ti Gr 2 pipe given that a PTFE quill is used to inject the acid into the center of the pipe and the process is controlled so it occurs only when solution is flowing through it. This is possible because chlorine dioxide is so oxidizing, and the reactions are quick enough to consume all the sulfuric acid. Injecting the acid into a standpipe may actually be more difficult in that the acid may run down the side of the tank. If the acid lingers on the wall without reacting fast enough the metal may pit. In this case even the more highly alloyed stainless steels, resistant to ClO_2, would not be immune from sulfuric acid attack.

Figure 3. Alloy 20 pump casing corrosion in the form of etching and pitting. The acid concentration was less than 10% acid and the temperature 71 ºC (160 °F). High velocity caused it to corrode.

Urea Hydrochloride

There is almost no corrosion information on this acid. Supplier product brochures claim that it behaves like inhibited hydrochloric acid so is not a corrosive. It has been used for scale removal without issue. However, limitations have been noted that if the temperature is too high or the solution is too dilute, it can behave as hydrochloric acid. Figure 4 shows a pitted 316L line used to deliver urea HCl for pH control to a standpipe. It is possible that the feed was not continuous and stagnant conditions as a result of residual urea HCl standing in the pipe caused the metal to pit. The line was replaced with C276.

Figure 4. Urea hydrochloride used for pH control pitted 316L piping.

CORROSION TESTING

This section covers coupon testing conducted at two separate mills. 2.54 x 5.08 cm (1 x 2 inch) metal coupons were polished to 120 grit except for the 2101 and 2003 samples which were used as-received. The coupons were mounted in racks made up of 316L rods, plates and fasteners. The coupons were separated by serrated PTFE crevice washers. Since the racks could only be installed and retrieved during outages, the test duration was at least one year. They were weighed in the lab before and after immersion. Weight loss was converted to a corrosion rate and coupons were visually examined for pitting. A typical arrangement is shown in Figure 5.

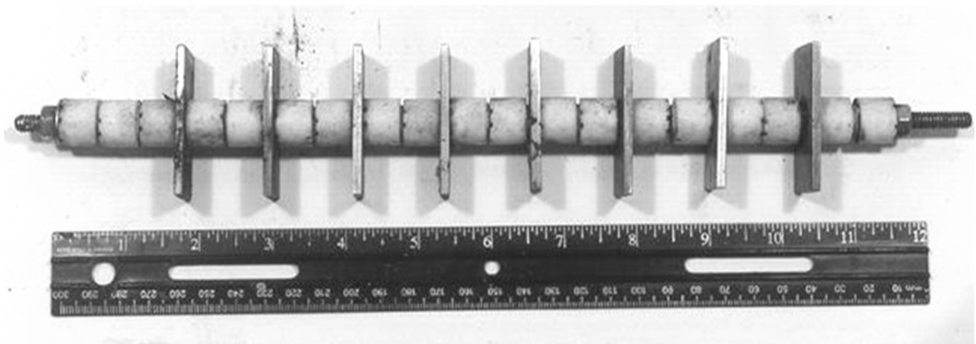

Figure 5. Example coupon rack consisting of metal samples separated by serrated PTFE crevice washers.

Mill 1 Material Selection for D2 Piping

The purpose of this test was to find an alternate material for 317L D2 filtrate piping. The test conditions were:

- Temperature: 160 – 200 °F
- pH: 3.3 – 4.0
- ClO_2 concentration: 0.08 g/L

Four coupons each of 317L, 2205, 254SMO and Titanium Gr. 2 were mounted on two racks and installed in the D2 filtrate tank. The test duration was approximately 345 days. Visual results of selected coupons are shown in Figure 6. For titanium, there was some staining around the central hole but otherwise there was no apparent corrosion nor was any corrosion rate measured. For 254SMO there was some staining and small pits around the hole. For 2205, not only was there staining and pitting around the hole, but also some pitting on the open portion of the surface. Even so the corrosion rate was slightly lower than 254SMO. The 317L coupon showed the most damage. It had the highest corrosion rate and much more severe pitting around the hole and bare surface. The results are summarized in Table II. The corrosion rate in mils per year (mpy) is an average of four coupons.

Table II. Mill 1 test results.

Alloy	Corrosion Rate (mpy)	Crevice Pitting	Open Pitting
Titanium Gr. 2	0	No	No
254SMO	0.26	Yes	No
2205	0.20	Yes	Yes
317L	1.95	Yes	Yes

Because the primary damage mechanism in D stage service is pitting and crevice corrosion, 254SMO had the edge in corrosion resistance over 2205. Even so, 2205 outperformed 317L and was chosen as the replacement material.

Mill 2 Material Selection for Filtrate Piping and Tanks

D1 and D2 stage filtrate piping and tanks had been made from FRP. Some of this equipment was coming to the end of its life and the mill preferred to rebuild it with metal. Some lines had already been replaced with 316L which were holding up well. Over seven years, coupon testing was carried out to determine which metals were suitable. The test conditions and results are given in Table III. Note that the mill did not provide a precise ClO_2 residual. All they could say was that it was low. These process conditions were considered applicable to all the testing carried out at this mill.

Table III Process conditions for the duration of mill 2 testing

Process Condition	D1 Filtrate	D2 Filtrate
Temperature	140 ºF	160 ºF
pH	2 – 3	5 – 6
ClO_2 residual	Low	Low

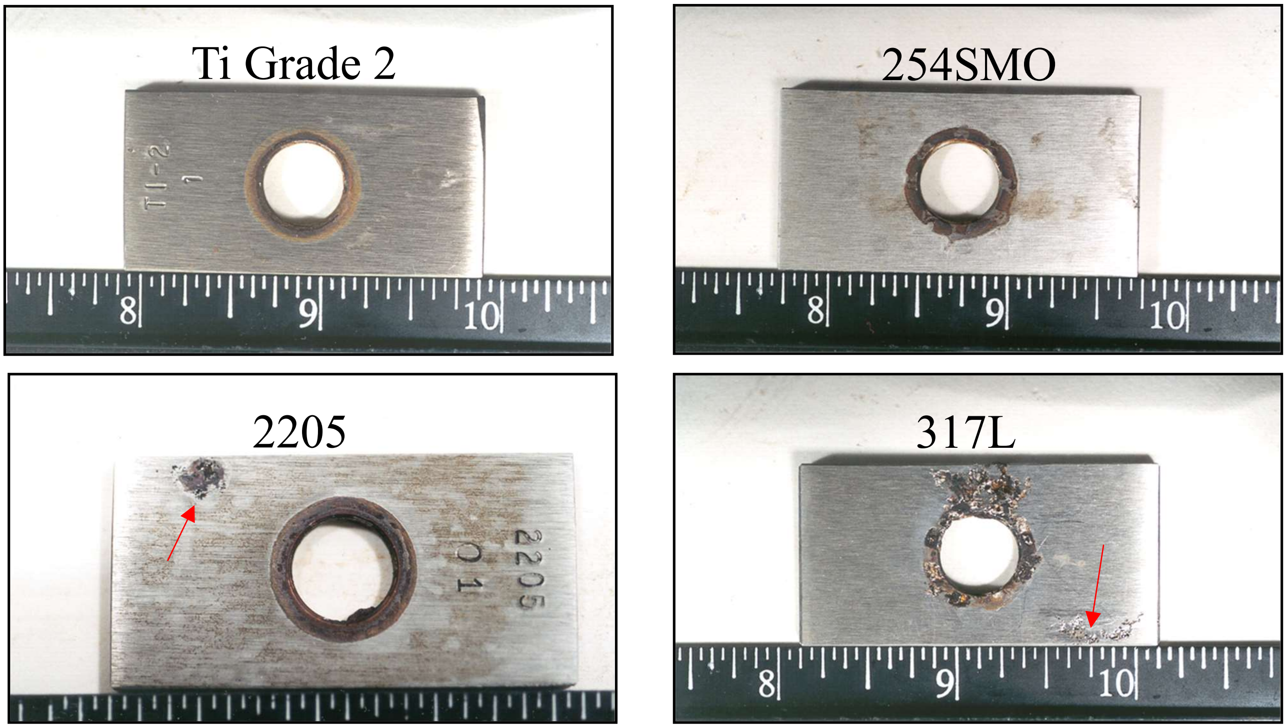

Figure 6. Mill 1 visual tests results. 2205 and 317L are pitting on the bare face. 254SMO pitted in the crevice area. Titanium did not corrode.

Round 1 – Immersion for one year.

A coupon rack was installed below the liquid level in each of the D1 and D2 filtrate tanks. When they were returned to the lab after one year, the D1 coupons were coated with a yellowish-white deposit which was easily removed. After cleaning there were no signs of pitting or corrosion. The D2 coupons were generally free of deposits and needed little cleaning. The 625 coupon had changed color. Figure 7 shows the post immersion coupons.

As shown in Table IV, the corrosion rates are all well under 1 mil/yr which indicates they are all resistant to uniform corrosion. This means that even 316L is resistant and shows that these ClO_2 filtrates are not aggressive. Even so some differences can be seen in corrosion performance. The D2 solution is slightly more corrosive than the D1 solution. The nickel alloys, 625 and C276 have the highest rates. This is consistent with the discussion above regarding high nickel alloys corroding in chlorine dioxide solutions.

Table IV Round 1 test results. Corrosion rates are low.

Alloy	D1 Corrosion Rate (mpy)	D2 Corrosion Rate (mpy)
316L	0.000	0.001
317L	0.001	0.002
2205	0.001	0.000
CD4MCU	0.001	0.001
254SMO	0.000	0.000
AL6XN	0.001	0.003
625	0.001	0.056
C276	0.010	0.044

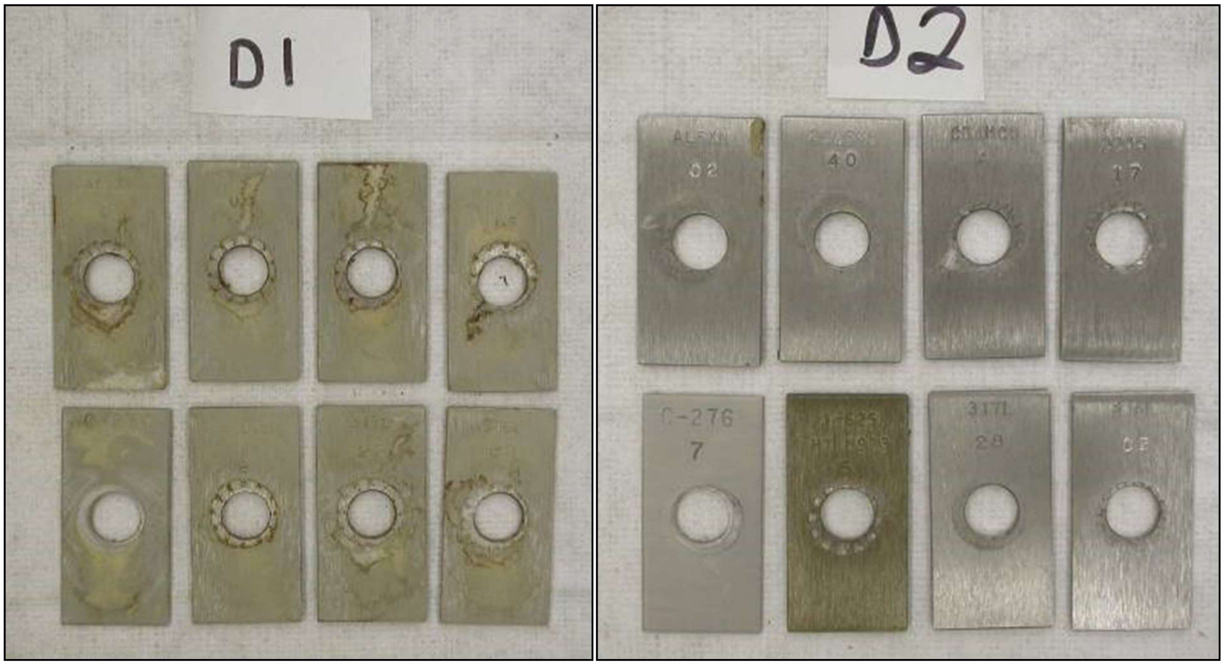

Figure 7. Round 1 coupons after one year of immersion in the D1 and D2 filtrate tanks. The D1 coupons were easily cleaned. No pitting was observed on any of the surfaces.

Round 2 – Immersion for one year.
A second, yearlong test was conducted in the same filtrate tanks. Three duplex stainless steels were substituted for higher alloy metals that would not be needed for this service. In neutral to acid conditions, 2003 and 2304 are comparable to 316L whereas 2101 is not expected to have equivalent resistance. 2003 is a low molybdenum duplex stainless steel and this should give it an edge over 2304 in acidic solutions. 317L, 2205, AL6XN and C276 were carried on from the first test. The D1 set of coupons did not display any pitting at all. Figure 8 shows the bottom side of the D2 coupons. There were small crevice pits on the 316L, 317L and 2101 coupons. There was a crevice pit on the top side of the 2101 coupon. Figure 9 shows pitting on the bottom side of the 316L and 317L coupons and Figure 10 shows pitting on both sides of the 2101 coupon. Note that for both filtrates, the 2101, 2003 and C276 coupons were darker after testing.

Table V shows that the filtrates are mild though somewhat more oxidizing than the first round which had been completed a year earlier. There was no pitting of the D1 coupons and again the corrosion rates were all under 1 mil/yr. Even though the rate is very low, C276 has the highest rate similar to the first round. The staining of the coupon may reflect this somewhat higher rate. Again, the D2 filtrate is more corrosive than the D1 filtrate but this time 316L, 317L and 2101 were pitted in the crevice area round the central hole. The approximate percentage of surface area covered by the pitting is included in the table. Though it couldn't be seen without magnification, there was fine pitting on the bare surface of the 2101 coupons.

316L, 317L, 2101 and C276 all had higher corrosion rates in round 2. The duplex stainless steels and AL6XN were the best performers. The rates did not change for 2205 and AL6XN. 2304 and 2003 were new to the test. The 2101 and 2003 coupons were darker after testing. This may be caused by the rougher surface however, it did not seem to be the result of excessive corrosion, especially 2003 which didn't corrode at all.

For immersion service 316L is adequate for D1 filtrate. Duplex stainless would add more protection in the event there were any process upsets, particularly with the filtrate tank; piping is more easily replaced. For the D2 filtrates, austenitic stainless steels and 2101 don't have sufficient pitting resistance. The remaining choices are 2003, 2304, 2205 or 6% Mo stainless steel. With lower molybdenum and nickel concentrations, 2003, should offer a cost advantage over 2205, however it is not readily available. 2304 is not made as piping so the best choice for duplex stainless steel is 2205.

Table V Round 2 test results. Corrosion rates are low though some of the alloys pitted in the D2 filtrate.

Alloy	D1 Corrosion Rate (mpy)	D2 Corrosion Rate (mpy)	D2 Pitting, % Crevice Area
316L	0.001	0.022	55
317L	0.000	0.002	10
2101	0.000	0.008	6
2003	0.000	0.000	
2304	0.000	0.000	
2205	0.000	0.000	
AL6XN	0.000	0.004	
C276	0.007	0.075	

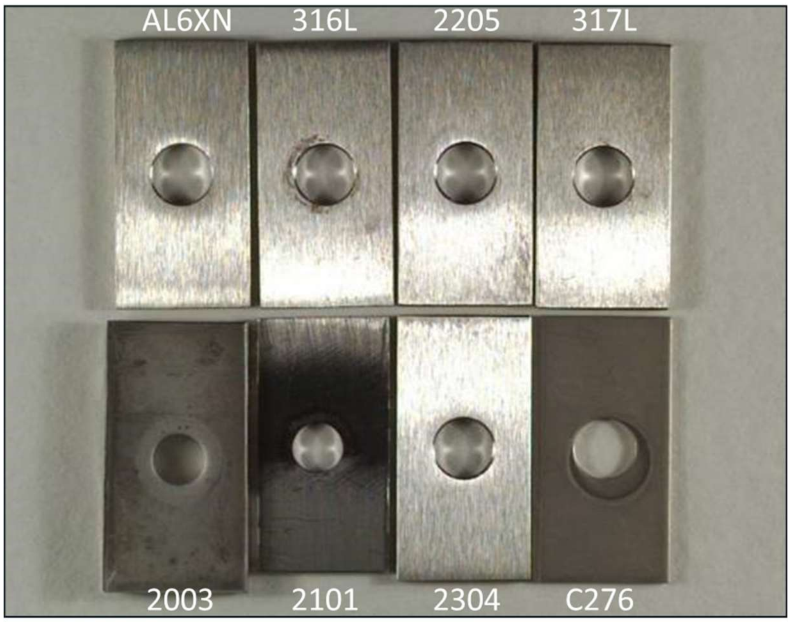

Figure 8. The bottom side of the Round 2 coupons after one year of immersion in the D2 filtrate tank. Pitting around the hole of the 316L coupon is visible. Crevice pitting of the 317L and 2101 is present but cannot be seen in this view.

Figure 9. Pitting on the bottom side of the crevice area on the 316L (left) and 317L (right) coupons.

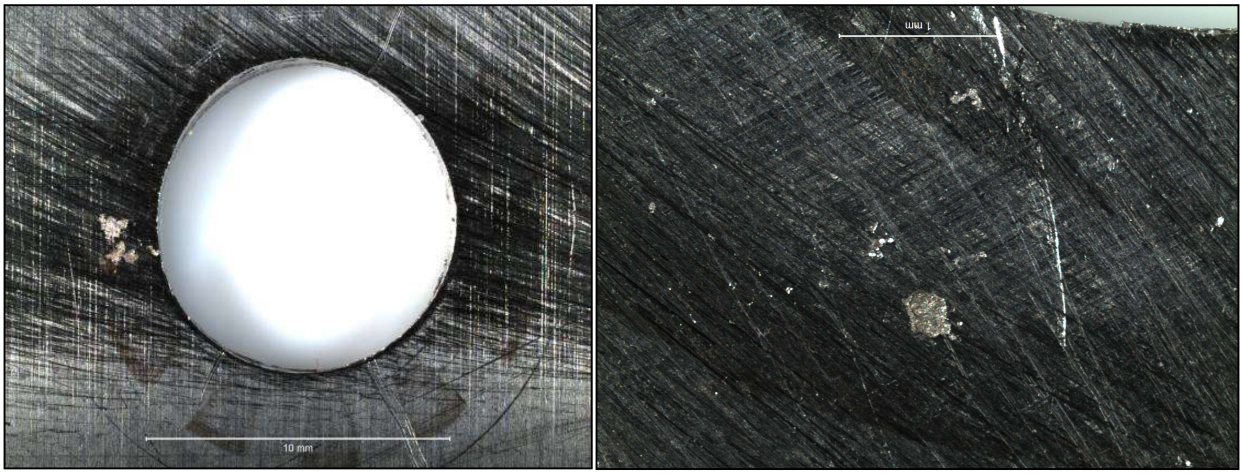

Figure 10. Pitting on the top and bottom side of the 2101 coupon. The bottom side view is enlarged because the pits are difficult to see by eye.

D1 vapor space exposure for two years.
The racks for this test were mounted in the vapor space above the liquid level. The D1 filtrate tank was not accessed for two years which accounts for the test duration. The coupons were covered with thicker deposits, Figure 11, than those that were immersed in the filtrate solution but cleaned up easily. None of the coupons were pitted and the corrosion rates were very low as seen in Table VI.


Figure 11. D1 coupons after exposure to the filtrate tank vapor space for two years.

Table VI D1 vapor space test results

Alloy	Corrosion Rate (mil/yr)
316L	0.001
317L	0.001
2101	0.001
2003	0.001
2304	0.000
2205	0.001
AL6XN	0.001

D2 vapor space exposure for five years

The coupon racks were installed at the same time as the D1 vapor space coupons however they were retrieved after five years. The results listed in Table VII show that of all the environments tested, this was the most corrosive. All the coupons pitted except for AL6XN. 2003 and 2205 did not uniformly corrode but experienced some pitting. The austenitic and lean duplex stainless steels all had measurable corrosion rates which were higher than those seen in the other tests. Visually the corrosion was more obvious with pitting occurring under the crevice washer and on the bare metal surface. Photographs of the coupons are included in the presentation.

Table VII D2 vapor space test results

Alloy	Corrosion Rate (mil/yr)	Damage Description
2101	0.054	Rough crevice and open area pitting
316L	0.035	Rough crevice and open area pitting
2304	0.033	Rough crevice and open area pitting
317L	0.012	Not as rough crevice and open area pitting
2003	0.000	Clean, few small crevice pits
2205	0.000	Clean, small crevice and open area pits
AL6XN	0.000	Clean, no pitting

Material selection recommendation for the piping and filtrate tanks.

After approximately seven years of insitu corrosion testing the following recommendations were arrived at based mainly on the variability in corrosivity from year to year and the D2 filtrate tank vapor space results.

- D1 piping: 316L, consider 2205 (lean duplex is not available as piping)
- D1 filtrate tank: though 316L did well, 2205 is preferred as a minimum alloy
- D2 piping: 2205
- D2 filtrate tank: 6% Mo stainless steel, consider 2507

SUMMARY

The main damage mechanisms in the bleach plant are:

- Under insulation stress corrosion cracking of stainless steel
- Peroxide corrosion of titanium
- Pitting and crevice corrosion of stainless steel in acidic, chloride containing bleaching oxidizers
- Transpassive corrosion of high nickel alloys in ClO_2
- Oxidative attack of organic resins
- Tile lining degradation
- Acid corrosion of metals and non-metals

References from the literature and corrosion testing indicate the bleach plant is not as corrosive as it once was. This is a result of improved operations which has led to lower chlorine dioxide residuals. It means that metals with lower alloying than those used in the past may be suitable. In order to do this, it is very important to know the chlorine dioxide residual, pH and temperature. Testing and experience has shown that duplex stainless steels have a place in the bleach plant. As an example, Figure 12 is a photograph of a 2205 pre-bleach storage tank. Duplex stainless steel was chosen because of the low chlorine dioxide residual. In the past it most likely would have been made as a tile lined carbon steel tank. In all instances 2205 should be substituted for 317L when new material is being installed. Given the popularity of 2205, 317L is becoming less available and more expensive as a result. Super duplex stainless steels should be considered as an alternative to the 6% Mo stainless steels. Finally, for optimized material selection test alloys in the actual conditions they will be exposed to.

Figure 12. 2205 duplex stainless steel pre-breach storage tank.

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