IN-PLANT TESTS ON HIGH ALLOYED STAINLESS STEELS IN C, D AND C_D STAGE BLEACH WASHERS

G.Singh and A. K. Singh
University of Roorkee
Institute of Paper Technology
Saharanpur-247001, India

ABSTRACT

The present paper reports findings on the suitability of the materials for bleach washers and related process equipment on the basis of in-plant test performed in C, D and C_D bleach washers of two Indian mills who have recently introduced chlorine-dioxide bleaching. Considering the extent of pitting, crevice and weld related corrosion vis-a-vis the cost and strength of the tested materials, the more appropriate material among them has been proposed.

INTRODUCTION

Globally, the paper industry is undergoing major changes in their paper making process for several reasons - controlling water and air pollution within stipulated limits is the most important of them. Accordingly, as a part of pollution controlling exercise, the paper mills are renovating their bleach sequences by introducing chlorine-dioxide (D) bleach stage and/or substituting chlorine partly by chlorine-dioxide (C_D). Changes in the bleach chemicals affect the corrosivity of the liquid media which in turn may affect the credibility of metallurgy of bleach plants. To understand this aspect of corrosion, several laboratory and in-plant tests have been performed on stainless steels, Ni-based alloys and Titanium in D and C+D bleaching stage or in environment observed in these bleach plant conditions.

The first reported laboratory tests (1) were performed to study the effect of chemical composition on the corrosivity of the chlorine-dioxide solution and the corrosion resistance dependence of stainless steel on the Mo and Si content. In laboratory immersion test performed in D-washer media (2,3), Ni base alloys, unwelded and when welded autogeneously/with Ni-base fillers show general corrosion. Increasing the iron content in these alloys shifts the transition to transpassivity to higher potentials(3). Stainless steels having high resistance to localised corrosion perform satisfactorily (2) while lower alloyed stainless steels fail by pitting/crevice corrosion (3). Their welded area shows localised attack or no attack depending upon iron filler composition, while distinctive localised corrosion was observed when Ni-base fillers were used (2). Another test performed in near neutral D-bleach media (4), shows the importance of Mo content in resisting crevice attack both on stainless steels and Ni alloys. In a test conducted to observe the performance of super-austenitic stainless steels (5) under service conditions (Temp. 64, pH 2.4-3, ClO2 42-160ppm, Cl 312-483ppm), SS904L shows breakdown of passivation even for lowest Cl and 50C temperature while 254SMO shows passivation even at Cl level that is 12 times that found in service conditions and at 65C thus exhibiting the beneficial effect of Mo. Only at 80C, the pitting is observed in 254SMO.

Immersion and electrochemical test (6) performed on Ni alloys exposed to C+D mixer media with chlorine dioxide substituting chlorine from 0-100%, shows general and localised corrosion. Another test (7,8) shows that Ni-base alloys do not have adequate corrosion resistance in these media. However, C-276 Ni alloys perform better than stainless steels in chlorination (0% D-substitution), the latter material not acceptable in these applications. Titanium has been suggested ideal choice for C+D media provided efforts are made to exclude dry Cl_2 in mixer environment which attacks titanium.

In the earliest performed in-plant test related to D-stage washer (9), 6%Mo containing SS is observed to be best among the tested SS. While Ni base alloys and Ti performed even better. In case of coupons exposed to inlet box and hood of D-stage washers (10), extensive localised corrosion is observed even on 6% Mo-stainless steel while Ni alloys show acceptable performance. Another test (11) in C and D stage bleach plant shows Ti suitable for mixer-to-tower piping in case of D and solid or clad Ni base alloys in case of C bleaching, solid or clad Ni base alloys are suitable for tower to vat section piping and 6%Mo alloy with alloy 625 or higher Mo content filler metal are excellent candidate for washers and filtrate piping. In the latest reported (12) in-plant test, the materials were exposed in tanks for spent acid from chlorine dioxide production, bleach plant scrubbers, bleach tower inlets and bleach washer. Two highly alloyed materials 904L and 2205 proved to be unsuitable for any of these environments. 254SMO was resistant in all except bleach tower inlet where its welded area was attacked. Stainless steel 2750 (25% Cr, 7% Ni, 4% Mo) experienced pitting in weld area in spent acid tank while 654SMO and Ti was resistant in all cases. Ni alloys were also resistant except in inlet of bleach tower where it experienced extensive uniform corrosion.

The above studies indicate that chlorine-dioxide is equally or in certain conditions more corrosive than chlorine, hence D and C_D stages are expected to be more aggressive than C stage. It is therefore of immense importance if experimental data is generated on the corrosion performance of the presently used material and the possible candidate materials in the bleach plants using chlorine dioxide. Since the process parameters for bleaching depend on the fibrous raw material and the pulping conditions, it was considered necessary to generate data on corrosion performance in the local mills which have only recently started using chlorine dioxide and have reported corrosion failures apprehended because of this change. Consequently, a project was undertaken to suggest suitable material of construction for bleach washers and related machinery of C, D and C_D stages. To achieve this objective, coupons of five stainless steels were exposed for about six months in bleach plants of two Indian mills. These coupons were analysed for uniform, localised and weld related corrosion.

EXPERIMENTAL

Material

Corrosion test coupons, size 2”x $^{3}/_{4}$”x $^{1}/_{8}$” with a hole of diameter 0.375” at its centre, of austenitic and one of duplex stainless steels were selected for the present tests. The composition of these stainless steels is given in **Table 1**. All the samples were solution annealed as per ASTM spec A240. They were welded on one side autogenously using GTAW (TIG) and then ground smooth using 120 grit. Before exposure, the coupons were degreased using acetone solution and weighed. Duplicate coupons of all the five stainless steels were fixed in a rack made from SS-316 stainless steel plates, rods and nuts. The coupons were insulated from each other and from the stainless steel rod with the help of teflon shoulder washers, spacers and tape. The covering of metal by shoulder washer provided the sites for initiating crevice corrosion. The coupons in the racks were tightened to a fixed torque using torque wrench so that position of coupons is not disturbed during the exposure so as to avoid any mechanical wearing. These racks were welded at different locations in bleach washers for corrosion exposure as detailed below.

Alloy	C	Cr	Ni	Mo	Cu	Mn	P	S	Si	N
316L	0.016	16.45	10.22	2.11	0.25	1.75	0.026	0.008	0.44	0.03
317L	0.019	18.19	13.23	3.11	0.42	1.78	0.031	0.001	0.55	0.047
904L	0.009	19.7	24.1	4.26	1.40	1.52	0.02	0.001	0.28	0.045
2205	0.025	22.00	5.700	3.01	0.29	1.44	0.022	0.001	0.48	0.154
254SMO	0.011	19.9	17.9	6.07	0.67	0.43	0.024	0.001	0.31	0.203

Table 1. Composition of stainless steels.

Test - sites and Environment

In-plant tests were conducted in two kraft pulping mills (named as mill A and B). Mill A uses 10 - 20% bamboo and rest hardwood for making pulp. Their bleaching sequence is $CE_1D_1E_2D_2$ type which help them achieve 84% pulp brightness. Mill B uses 67% bamboo and rest hardwood as fibrous raw material. The bleaching sequence is C_DE_OHED for achieving 86 - 87% pulp brightness. Both mills recycle their filterates and use SO_2 as antichlore in D washer.

The racks of test coupons were exposed in washers of C and D_1 stage of Mill A and of C_D and D stage of Mill B. The process conditions in these washers are given in **Table 2.** The coupons were exposed by welding the racks in vat and on the hood (inside washer) in both the mills while, in addition, over the rotating drum in Mill A. Thus the metals corroded due to liquid, gaseous and wet/dry phases of the media respectively. Duration of exposure of the coupons was 202 to 206 days. After the exposure, the corroded coupons were cleaned by light brushing and by treating with cold solution of concentrated HCl with 50 gpl $SnCl_2$ and 20 gpl $SbCl_3$ (13).

Mill	Bleach Stage	Temperature (°C)	pH	Residual Oxidant (ppm)	Chloride (ppm)
Mill A	C	39±1.5	2.1 ± 0.4	88.5 ± 53.5	951±150
	D_1	53±3.0	2.4 ± 0.3	136.6 ± 90.4	490±40
Mill B	C_D	35.5 ± 2.5	1.8 ± 0.1	57 ± 14	3000 ± 500
	D	55 ± 5	2.9 ± 0.1	33 ± 23	550 ± 100

Table 2. Process condition in bleach washers.

RESULTS AND DISCUSSION

The cleaned coupons were weighed to calculate the corrosion rates (**Table 3)**. They were also analysed for the extent of pitting, crevice corrosion and weld related corrosion. For knowing these parameters, depths of the pits formed on (i) open surface, (ii) under crevices (part of coupons covered under shoulder washers) and (iii) on welded area of the coupons respectively were measured under the microscope. Depth of the deepest pit (maximum pit depth), formed under different conditions, has been considered as a parameter representing the degree of attack. On this basis and the criteria adopted earlier (10), the materials have been classified in three categories namely unacceptable, satisfactory and superior. This categorisation is shown in Tables 4, 5 and 6.

	Mill A						**Mill B**			
Materials	***C***			***D***			C_D		**D**	
	vat	hood	drum	vat	hood	drum	vat	hood	vat	hood
316L	0.07	*	0.02	1.52	0.06	1.09	0.04	1.25	0.004	N
317L	0.07	13.47	0.02	0.65	0.11	0.33	0.05	0.35	0.003	0.004
904L	0.05	4.27	.02	0.40	0.06	0.21	0.01	0.10	0.009	N
2205	0.06	4.00	0.09	0.43	0.03	0.20	0.02	0.07	0.001	N
254SMO	0.04	0.07	0.04	0.30	N	0.11	N	N	N	N

* samples were corroded completely to brittle pieces.
N - No measurable/visible attack.

Table 3. Corrosion rates of stainless steels.

Corrosivity of Washers

The coupons exposed to gaseous phase in C-washer have been attacked most, predictably due to presence of gaseous Cl_2 along with moisture which together create highly corrosive media of pH ~ 1-2 with dissolved Cl_2 and Cl^- (14).The next corrosive stage is D-vat. This is probably due to higher oxidising power of ClO_2 as compared to Cl_2, higher concentration of residual oxidant (15) as compared to C-vat although liquor in D-vat is less acidic. Pitting attack on coupons exposed to gaseous phase of D-washer is of the same order as in D-vat. The attack is lesser as compared to C-hood due to lesser concentration of ClO_2 (in gaseous phase of D-washer) as its solubility, in water, is more than Cl_2.

Coupons exposed on washer drum experience lesser corrosion as they undergo immersion in D-vat in cyclic manner thus alternating between liquid and gaseous media. Liquor in C-vat poses similar degree of corrosivity under crevices as D-drum. The corrosivity of liquor in C-vat is comparatively less, although its pH is more acidic than that of D-vat's liquor. Crevice attack on coupons exposed on C-drum is slightly less. The media in C_D-washer (Mill B) are more corrosive as compared to D-washer. This may due to (i) lower pH (ii) more oxidising nature of the liquors being mixture of Cl_2 + ClO_2. Under some conditions, this may be equally corrosive as the liquor of C-washer.

Overall, the conditions in Mill A appear more corrosive than in Mill-B. This could be attributed to higher concentration of residual oxidants and lower pH in some cases in Mill A.

Performance of Materials

Corrosion rates of various stainless steels are not high except for the coupons exposed in C-stage gaseous phase (barring 254SMO) for instance 316L coupon was overwhelmingly 'eaten' and 317L showed unacceptably higher attack. The different stainless steels may be put in following order of increasing degree of resistance against uniform corrosion :

316L < 317L < 904L and 2205 < 254SMO

However, in the Cl^- containing acidic oxidising media, more cases of materials' failure have been reported due to localised corrosion, as such it will be better to assess the extent of localised corrosion on the tested materials for estimating their suitability. The results (**Table 4**) show pitting on 316L as maximum followed by 317L, 904L, 2205 and 254SMO. In some cases, 317L and 904L show equivalent performance. The present results are in accordance with the latest reported in-plant test (12) according to which 904L experiences high degree of localised attack while 254SMO was resistant in D-washer having less than 100 ppm of residuals. In present case, 904L exposed in D-washer of mill-A experiences high degree of pitting where amount of residuals are quite high in comparison to the observations in Mill-B. In an earlier reported in-plant test results (10), extensive localised corrosion is observed even on 6% Mo-stainless steel exposed in hood of D-washer which is an attribute of high temperature and residual oxidant. In both the mills (A, B), where presently reported tests were conducted, the temperatures and amount of residual oxidant were comparatively low and the localised attack on 254SMO was also negligible. This significantly different observation on corrosion performance all the more justifies performing in-plant tests in different mills because of substantial differences in process conditions.

On comparing crevice corrosion (**Table 5**), in most cases 316L shows least resistance, followed by 317L, 904L, 2205, 254SMO. Some exceptions are observed however. Thus in case of coupons exposed in C-hood (Mill A)- attack on 904L is much more than at 317L (may be due to inconsistent or unequal flow of Cl_2 gas) (904L coupons were at the middle of rack so Cl_2 gas may easily pass around these coupons as compared to 317L). On the basis of pit density on the surface under the crevice, one observes that, in D-washer of mill-A, 904L and 2205 are attacked more than 317L and 316L in liquid media while 317L, 904L and 2205 are attacked to same degree and they show lesser resistance than 316L when exposed to wet/dry media of this washer. It seems, in D washer of mill A, conditions are such which makes SS with higher Mo more vulnerable to attack than SS-316L. However, presence of higher N (in 254SMO) seems to nullify this.

	Mill A						**Mill B**			
Materials	***C***			***D***			***C_D***		***D***	
	vat	hood	drum	vat	hood	drum	vat	hood	vat	hood
316L	*	***	*	***	*	***	*	***	*	*
317L	*	***	*	*	*	*	*	***	*	*
904L	*	***	*	*	***	*	*	*	*	*
2205	*	***	*	*	*	*	*	*	*	*
254SMO	*	*	*	*	*	*	*	*	*	*

Table 4. Material evaluation for pitting resistance.

	Mill A						**Mill B**			
Materials	***C***			***D***			***C_D***		***D***	
	vat	hood	drum	vat	hood	drum	vat	hood	vat	hood
316L	**	***	**	*	*	**	***	**	**	*
317L	**	***	*	**	*	*	**	**	**	*
904L	**	***	*	**	*	*	*	**	*	*
2205	**	***	*	**	*	*	**	*	*	**
254SMO	*	**	*	*	*	*	*	*	*	*

Table 5. Material evaluation for crevice corrosion resistance.

Higher degree of weld area attack as compared to pitting is observed in more corrosive media (test results of Mill A, **Table 6**). Thus coupons exposed in C-hood of Mill A have become totally porous in their weld areas. Mill B does not show these type of attack. In almost all cases, 316L shows maximum attack and 254SMO shows minimum. In majority of the cases, the attack on 2205 appears more than on 317L followed by 904L. It is known that welding practices result into microsegregation and coring. The materials showing higher degree of attack may have larger fraction of dendritic phase (Cr~14%, Mo~1.8%), which in turn may be the result of the composition (16). It is also expected that more richly alloyed stainless steels (Mo > 4%) may show higher degree of attack on their welded areas due to (i) alloy depleted region (ii) precipitation of intermetallics like sigma (σ) phase (17). Although all the coupons were autogeneously TIG welded, the strange behaviour of weld area attack on 317L/904L/2205 coupons could be attributed to the variation in their composition particularly Ni, Mo and N.

Materials	Mill A						Mill B			
	C			***D***			C_D		***D***	
	vat	hood	drum	vat	hood	drum	vat	hood	vat	hood
316L	*	***	*	***	*	*	*	**	*	*
317L	*	***	*	**	*	**	*	**	*	*
904L	*	***	*	*	**	*	*	*	*	*
2205	*	***	*	**	*	**	*	*	*	*
254SMO	*	***	*	*	*	**	*	*	*	*

***- unacceptable (high degree of attack), **- satisfactory (medium degree of attack),
*-superior (negligible/no visible attack)

Table 6. Material evaluation for resistance against weld area attack.

On the basis of all the above type of corrosion attacks, following can be suggested (i) In general, 316L is least while 254SMO is most resistant material. In one of the earliest performed in-plant test related to D-stage washer (9), 6% Mo containing SS is observed to be best among the tested SS.(ii) corrosion resistance is in the order : 316L<317L<904L ≤ 2205<254SMO.

The observations on pitting and crevice corrosion can also be checked with the help of pitting resistance equivalent (PRE) no. (16,17), critical pitting temperature (CPT) and critical crevice temperature (CCT) values. PRE no. indicates resistance of a material against pitting attack while CPT and CCT, in °C, are the lowest temperatures at which pitting and crevice corrosion respectively occurs on the steel surface after 24 hours of exposure in 6% $FeCl_3$ solution. The CPT and CCT values were determined experimentally using ASTM procedure (18). These are observed to be similar to those reported in literature (17,19) with slight difference which could related to the composition of stainless steels. PRE nos., CPT and CCT values for the tested materials are given below.

Material	PRE No. (16)	PRE No. (17)	CPT	CCT
316L	23.893	24.313	5-10	<5
317L	29.205	29.863	33	<5
904L	34.478	35.108	45	10-12
2205	34.397	36.553	52-54	15
254SMO	43.179	46.021	75	45

Accordingly, 316L is expected to show least resistance while 254SMO the highest resistance to pitting. Pitting resistance of 317L should be better than that of 316L. According to PRE no. (17) and CPT value, 904L should pit more than 2205. This is observed in the test results also. Better assessment of the pitting resistance on the basis of PRE no.(17), appears to emphasize the inclusion of N vis-à-vis Mo in enhancing pitting resistance of the stainless steels.

By and large, CCT values of the tested stainless steels and the predicted resistance against crevice corrosion is in accordance with the test results. This shows the importance of Mo and N together in imparting resistance against crevice corrosion.

Another test performed in near neutral D-bleach media (4), however, shows the importance of Mo content in resisting crevice attack. Beneficial effect of Mo in imparting pitting resistance has also been demonstrated in another study (5), which suggests breakdown of passivation in case of 904L even at lowest Cl level and 50C while 254SMO still showing passivation even for higher Cl and up to 65C in service conditions of D-washer. The present in-plant test, however, show 254SMO also experiencing localised attack although to a lesser degree than 904L in temperatures range 50-60C.

Material Selection

Material selection involves not only the comparison of corrosion resistance of the materials but also their mechanical properties and cost along with their fabrication aspect so as to provide a maintenance free long life. As such, effort has been made to propose about material selection considering their mechanical properties and cost (**Table 7** and **8**) also.

Materials	316L	317L	904L	2205	254SMO
Tensile Strength*	490	515	500	680	650
0.2% proof stress*	210	220	220	480	300

***** Values measured at 20°C, in MPa

Table 7. Mechanical properties of bleach plant materials.

Materials	316L	317L	904L	2205	254SMO	654SMO
Plates*	1.0	1.4	2.0	1.4	2.5	3.5
Tube(seamless)*	1.0	1.2	1.6	1.2		
(welded)*	0.5	0.6	1.3	0.6		
General**	1.0			0.85		

***** cost for equal weight , ** cost for equal strength

Table 8. Cost of bleach plant materials.

Mill A

C-washer: For exposure in gaseous phase e.g. for pipings, valves and other equipment, one should consider a material more resistant than 254SMO for instance 654SMO which has been observed to show required corrosion resistance (20). This material is also less costlier than the Nickel based alloys which could also be potential resistant materials. For vats, 254SMO will be the premium brand. In order that types of fabrication materials are limited to as low as possible, all except 316L will be suitable choice. A comparison of strength and cost (Table 7 and 8), makes 2205 better (for equal strength requirement) because of its superior strength in comparison to 317L and 904L and lower cost with respect to 904L. Similarly, for washer drum and machinery to be exposed to wet/dry environment, 2205 will be better choice.

D-washer: For liquid media in vat, crevice corrosion tendency on all the tested stainless steels except 254SMO is on the higher side, although in satisfactory limit. Hence, 254SMO will be better material for vat and related process equipments. For hood or equipment exposed to gaseous media, 316L onwards could be suitable materials.

However, 2205 is suggested on the basis of strength and cost. For drum washer and related machinery likely to expose to wet/dry environment, all except 316L are acceptable though only 254SMO is in premium range. Since crevice attack on 317L is lower than that on 904L and 2205, the former will be better acceptable. Initially it may be a little more costlier than 2205 but may turn out cost-effective in longer duration.

Mill B

C_D -washer: For C_D-vat, the premium brand will be 254SMO. However 2205 should be preferred on the basis of cost, strength and degree of attack. For hood and other equipment likely to be exposed to gaseous media, 2205 will be best acceptable material. This material experiences least attack and is cheapest if compared for equal strength barring 254SMO.

D- washer: For vat, hood and process equipment likely to be exposed to gaseous and liquid media 2205 is best material on comparing degree of attack, strength and cost.

In nutshell, one observes the suitability of duplex stainless steel 2205 in most of the environment except for exposure in (i) C-hood, (ii) C-vat and (iii) D-drum in Mill A. An additional advantage in using 2205 is in its having better resistance for chloride stress corrosion cracking (CSCC), due to lesser Ni content.

From fabrication aspect, welding of 2205 is not much different than the conventional material 316L except that cost of welding consumables in former case is higher. This is balanced by considering thinner gauges of 2205 (for equal strength), which will require less welding consumables and lesser efforts in edge preparation. Fabrication of 254SMO is more costly due to restricted heat input at welding and more time consuming machining. Overall, the proposition on material selection for different bleaching stages as per the conditions prevailing in the present studied plants, is given in **Table 9**.

		vat	2205
	C	hood	654 SMO
Mill A		drum	2205
		vat	254 SMO
	D	hood	2205
		drum	317L
	C_D	vat	2205
Mill B		hood	2205
	D	vat	2205
		hood	2205

Table 9. Suggested material of construction for bleach washers.

CONCLUSIONS

The gaseous media of C-washer appears most corrosive. The corrosivity is lesser in D-vat and D-gaseous media. The corrosion environment experienced by alternative exposure to gaseous and liquid media in D-washer is still lesser but of the same degree as experienced by coupons in liquid media of C-washer. Alternate exposure in liquid/gaseous media in C-washer is least corrosive in this mill (Mill A). In mill B, C_D washer seems to pose more problems than D-washer. Replacement of chlorine by chlorine-dioxide seems to increase crevice corrosion as compared to a washer having only chlorine.

The degree of corrosion resistance shown by various stainless steels is in following order of increasing resistance:

316L < 317L < 904L ≤ 2205 < 254SMO.

On comparing corrosion attack, mechanical strength, cost and fabrication aspect, duplex stainless steel 2205 turns out to be appropriate material for most media except in some environment of Mill A namely (i) C-hood where 654SMO is expected to perform better (ii) D-vat where 254SMO seems a better choice.

The results of some of the tests performed earlier show 254SMO to be the more resistant material for washer and filtrate piping of bleach plants using chlorine dioxide. But a comparison of cost and other properties along with the corrosion resistance may suggest a lesser resistant material e.g. 2205 for many of the media present in these plants.

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