EUROPEAN COMMISSION

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"STRUCTURAL APPLICATIONS OF FERRITIC STAINLESS STEELS"

Report on laboratory accelerated corrosion tests (Deliverable WP7.3)

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Report on laboratory accelerated corrosion tests (Deliverable WP7.3)

SAFSS-WP7.3 : Report on laboratory accelerated corrosion tests

- ANNEX I : Pictures of samples after one cycle
- ANNEX II : Pictures of samples after three cycles
- ANNEX III : Pictures of samples after four cycles
- ANNEX IV : EN 1.4003 Chemical cleaning

1 INTRODUCTION

A comparative corrosion study is being carried out in the SAFSS project.

The participation of Acerinox as leader of the corrosion work package (WP7) includes three different tests: exposure fields (atmospheric test), accelerated tests (climatic chamber) and electrochemical tests.

This document only includes the accelerated test in climatic chamber. By means of this assessment, comparative information about corrosion resistance from the different ferritic grades in saline environment is obtained.

2 TEST MATERIALS

The stainless steels have been delivered by the three industrial partners involved in the project (Aperam, Outokumpu and Acerinox).

Table 1 shows the identification in accelerated tests of the ferritic stainless steels. The grade, origin, line, finish and thickness are specified.

Ferritic Stainless Steel	Line	Finish	Thickness (mm)	Identification
EN 1.4003	Hot rolled	1D	4.0	1
	Hot rolled	1D	6.0	2
	Cold rolled	2B	0.8	3
	Cold rolled	2B	1.0	4
EN 1.4509	Hot rolled	1D	3.5	5
	Hot rolled	1D	6.0	6
	Cold rolled	2B	0.6	7
	Cold rolled	2B	1.0	8
EN 1.4521	Cold rolled	2B	1.2	9
	Cold rolled	2B	0.8	10
EN 1.4621	Cold rolled	2M	1.0	11
EN 1.4016	Cold rolled	2B	1.2	12
	Cold rolled	BA	1.0	13
EN 1.4509	Cold rolled	BA	1.0	14
	Cold rolled	BA	0.8	15
EN 1.4521	Cold rolled	BA	0.7	16
EN 1.4301	Cold rolled	2B	0.7	17

Table 1.- Stainless steels identification

Samples from 1 to 11 are taking part on the atmospheric test on the four test stations of the project, Seville (urban), Isbergues (industrial), Ljubljana (rural) and Tornio (marine). The ferritic EN 1.4016 (12, 13) has been decided to test, because it is a ferritic grade widely used and studied. The grade EN 1.4509 in BA finish

(14,15) has been included in order to evaluate the BA finish in this grade, and finally, the EN 1.4521 grade in BA finish (16) has been included so as to improve the information gathered from the test. The austenitic stainless steel EN 1.4301 (17) has been considered as a reference material. The inclusion of more specimens improves the repeatability and the information obtained from the test.

In order to check materials, by means of x-Ray fluorescence spectroscopy and LECO automatic detectors (carbon, nitrogen and sulphur) the chemical composition has been analysed (table 2).

	Weight %										
	C	**Si**	**Mn**	**Sn**	**Ni**	**Cr**	**Mo**	**Ti**	**Nb**	**S**	**N**
1	0.011	0.29	1.40	0.011	0.55	11.02	0.03	0.004	0.017	0.003	0.0146
2	0.019	0.29	1.40	0.011	0.55	11.05	0.03	0.003	0.017	0.002	0.0124
3	0.024	0.46	0.59	0.009	0.53	10.80	0.03	0.004	0.007	0.001	0.0154
4	0.014	0.26	1.42	0.010	0.48	11.05	0.01	0.004	0.002	0.002	0.0111
5	0.016	0.43	0.26	0.010	0.27	17.85	0.01	0.170	0.475	0.001	0.0210
6	0.017	0.57	0.32	0.010	0.26	17.64	0.01	0.149	0.402	0.002	0.0143
7	0.015	0.46	0.26	0.009	0.39	17.65	0.04	0.135	0.464	0.001	0.0255
8	0.019	0.52	0.44	0.015	0.32	18.14	0.03	0.120	0.443	0.001	0.0176
9	0.019	0.59	0.28	0.004	0.24	17.78	1.92	0.156	0.408	0.001	0.0237
10	0.027	0.55	0.54	0.007	0.41	18.02	1.98	0.138	0.395	0.003	0.0241
11	0.017	0.29	0.26	0.009	0.29	20.36	0.02	0.003	0.452	0.002	0.0230
12	0.066	0.37	0.64	0.011	0.35	16.35	0.01	0.003	0.008	0.003	0.0321
13	0.050	0.37	0.34	0.010	0.26	16.26	0.01	0.003	0.005	0.002	0.0352
14	0.022	0.48	0.37	0.011	0.47	17.98	0.03	0.185	0.459	0.002	0.0164
15	0.025	0.60	0.29	0.017	0.28	17.71	0.02	0.152	0.446	0.002	0.0176
16	0.022	0.66	0.28	0.007	0.34	17.88	1.84	0.137	0.351	0.002	0.0142
17	0.048	0.33	1.73	0.009	8.07	18.12	0.22	0.005	0.012	0.001	0.0564

Table 2.- Chemical Composition

The right composition is confirmed in each stainless steel.

3 ACCELERATED TEST

3.1. TEST CONDITIONS

The test is based on VDA 621-415 standard "Prüfung des Korrosionsschutzes von Kraftfahrzeuglackierungen bei zyklisch wech selnder Beanspruchung". This document explains an accelerated test into climatic chamber. The procedure consists in introducing samples into chamber where they will be exposed to alternate cycles of salt spray fog, humidity and temperature variations. The steps are shown in table 3.

Step	Conditions	Environment	Time (hours)	Temperature (ºC)	Pressure (psi)	Humidity (%)
1	Salt spray	NaCl	24	35	21-24	--
2	Humidostatic	Air	8	40	--	100
	Climatic	Air	16	23		96
3	Humidostatic	Air	8	40	--	100
	Climatic	Air	16	23		96
4	Humidostatic	Air	8	40	--	100
	Climatic	Air	16	23		96
5	Humidostatic	Air	8	40	--	100
	Climatic	Air	16	23		96
6	Climatic	Air	48	23	--	50

Table 3.-Test Conditions into the climatic chamber

The sodium chloride solution has a concentration of 50 ± 5 g/l with a pH value of 7.

3.2. SAMPLE PREPARATION

The samples are cut with dimensions of 150x100mm^2. The edges are polished up to a fine-grained finish by 120, 180, 320 and 600-grit SiC paper, consecutively.

The samples are tested in order to get as wide information as possible about their behaviour versus phenomenon of generalized, pitting and crevice corrosion. For this purpose, there are three groups. In the first of them, the surface is flat. In the second of them, the surface has a hole with stainless steel screw and metallic washers. And in the third of them, the surface has a hole with stainless steel screw and Teflon washers.

Table 4 shows samples identification.

Identification	Group 1 Flat	Group 2 Stainless steel washer	Group 3 Teflon washer
1	1-A	1-D	1-G
	1-B	1-E	1-H
	1-C	1-F	1-I
2	2-A	2-D	2-G
	2-B	2-E	2-H
	2-C	2-F	2-I
3	3-A	3-D	3-G
	3-B	3-E	3-H
	3-C	3-F	3-I
4	4-A	4-D	4-G
	4-B	4-E	4-H
	4-C	4-F	4-I
5	5-A	5-D	5-G
	5-B	5-E	5-H
	5-C	5-F	5-I
6	6-A	6-D	6-G
	6-B	6-E	6-H
	6-C	6-F	6-I
7	7-A	7-D	7-G
	7-B	7-E	7-H
	7-C	7-F	7-I
8	8-A	8-D	8-G
	8-B	8-E	8-H
	8-C	8-F	8-I
9	9-A	9-D	9-G
	9-B	9-E	9-H
	9-C	9-F	9-I
10	10-A	10-D	10-G
	10-B	10-E	10-H
	10-C	10-F	10-I
11	11-A	11-D	11-G
	11-B	11-E	11-H
	11-C	11-F	11-I
12	12-A	12-D	12-G
	12-B	12-E	12-H
	12-C	12-F	12-I
13	13-A	13-D	13-G
	13-B	13-E	13-H
	13-C	13-F	13-I
14	14-A	14-D	14-G
	14-B	14-E	14-H
	14-C	14-F	14-I
15	15-A	15-D	15-G
	15-B	15-E	15-H
	15-C	15-F	15-I
16	16-A	16-D	16-G
	16-B	16-E	16-H
	16-C	16-F	16-I
17	17-A	17-D	17-G
	17-B	17-E	17-H
	17-C	17-F	17-I

Table 4.- Identification of samples

Before putting screws and washers, specimens are washed by means of soap and distilled water, dried by cellulose paper and weighed. Table 5 shows the weight of every sample.

Group 1		Group 2		Group 3	
Flat	**WEIGHT (g)**	**Stainless steel washer**	**WEIGHT (g)**	**Teflon washer**	**WEIGHT (g)**
1-A	455,6700	1-D	453,3900	1-G	450,5500
1-B	456,7900	1-E	448,8000	1-H	456,6700
1-C	456,2200	1-F	454,1100	1-I	448,9000
2-A	674,1300	2-D	667,4200	2-G	666,4800
2-B	700,8600	2-E	672,4800	2-H	675,7100
2-C	677,3400	2-F	674,7900	2-I	674,4400
3-A	91,1514	3-D	90,9121	3-G	90,9967
3-B	90,7763	3-E	90,0266	3-H	90,7554
3-C	90,5092	3-F	88,4187	3-I	91,5553
4-A	115,2924	4-D	114,8722	4-G	115,0327
4-B	114,0643	4-E	114,8298	4-H	114,9236
4-C	115,1308	4-F	114,7580	4-I	114,9178
5-A	398,6200	5-D	403,2800	5-G	401,2600
5-B	697,6400	5-E	399,5500	5-H	403,3600
5-C	404,1200	5-F	404,7300	5-I	403,5700
6-A	692,5800	6-D	678,7900	6-G	663,0000
6-B	691,2400	6-E	691,6900	6-H	692,7300
6-C	687,8300	6-F	691,5200	6-I	679,9100
7-A	68,6747	7-D	68,4051	7-G	68,3176
7-B	67,9981	7-E	68,0114	7-H	68,0809
7-C	68,8295	7-F	67,8716	7-I	68,2576
8-A	113,9952	8-D	113,6061	8-G	113,5091
8-B	114,0272	8-E	112,6067	8-H	112,4515
8-C	113,6122	8-F	112,8716	8-I	113,4318
9-A	135,2753	9-D	135,0325	9-G	133,3149
9-B	134,1666	9-E	134,7347	9-H	134,5193
9-C	135,2049	9-F	134,1434	9-I	134,3433
10-A	89,0770	10-D	89,8218	10-G	89,6827
10-B	89,6444	10-E	89,1578	10-H	89,9632
10-C	89,7718	10-F	89,7007	10-I	89,1780
11-A	113,4078	11-D	113,3670	11-G	111,1961
11-B	114,1169	11-E	113,6994	11-H	113,9255
11-C	114,2910	11-F	112,8452	11-I	112,8435
12-A	139,9362	12-D	139,4497	12-G	139,8314
12-B	139,9297	12-E	136,7637	12-H	139,9096
12-C	141,3187	12-F	140,6613	12-I	138,5003
13-A	115,4145	13-D	115,2109	13-G	114,6015
13-B	115,1957	13-E	114,4987	13-H	114,6184
13-C	114,4231	13-F	113,7524	13-I	115,4601
14-A	117,1841	14-D	116,4464	14-G	116,2482
14-B	117,1745	14-E	116.3844	14-H	116,0674
14-C	117,0388	14-F	116,6187	14-I	116,7355
15-A	91,9584	15-D	90,6126	15-G	90,8014
15-B	91,3919	15-E	91,2242	15-H	91,6349
15-C	91,7980	15-F	91,6383	15-I	91,6890
16-A	70,0120	16-D	69,3686	16-G	69,7669
16-B	69,8820	16-E	69,6412	16-H	69,6976
16-C	70,2182	16-F	70,2508	16-I	70,1397
17-A	80,8497	17-D	79,6857	17-G	80,0358
17-B	80,6870	17-E	79,3673	17-H	79,9519
17-C	80,2654	17-F	80,5964	17-I	80,5924

Table 5.- Weights of the samples before climatic chamber test

3.3. CLIMATIC CHAMBER

The samples should be placed in a way that they do not touch each other to allow free flow of fog and no contamination between samples. The specimens are placed randomly inside the chamber with upright rolling direction. The distribution of the samples in the chamber is shown in table 6 and in figure 1.

	3-A	8-A	2-B			3-C	11-C	5-C	
1-A	3-D	8-D	2-E	9-B	7-B	3-F	11-F	5-F	9-C
1-D	3-G	8-G	2-H	9-E	7-E	3-I	11-I	5-I	9-F
1-G	9-A	11-A	6-B	9-H	7-H	4-B	7-C	10-C	9-I
2-A	9-D	11-D	6-E	1-B	10-B	4-E	7-F	10-F	4-C
2-D	9-G	11-G	6-H	1-E	10-E	4-H	7-I	10-I	4-F
2-G	10-A	7-A	5-B	1-H	10-H	11-B	6-C	1-C	4-I
4-A	10-D	7-D	5-E	8-B	2-C	11-E	6-F	1-F	8-C
4-D	10-G	7-G	5-H	8-E	2-F	11-H	6-I	1-I	8-F
4-G	6-A	3-B	16-A	8-H	2-I				8-I
5-A	6-D	3-E	15-B	16-B	12-B	15-C	17-B	14-C	13-C
5-D	6-G	3-I	15-E	16-E	12-E	15-F	17-E	14-F	13-F
5-G	16-G	12-A	15-H	16-H	12-H	15-I	17-H	14-I	13-I
16-D	14-A	12-D	13-A	17-A	14-B	13-B	16-C	12-C	17-C
15-A	14-D	12-G	13-D	17-D	14-E	13-E	16-F	12-F	17-F
15-D	15-G	14-G	13-G	17-G	14-H	13-H	16-I	12-I	17-I

Table 6.- Distribution of the samples in the chamber

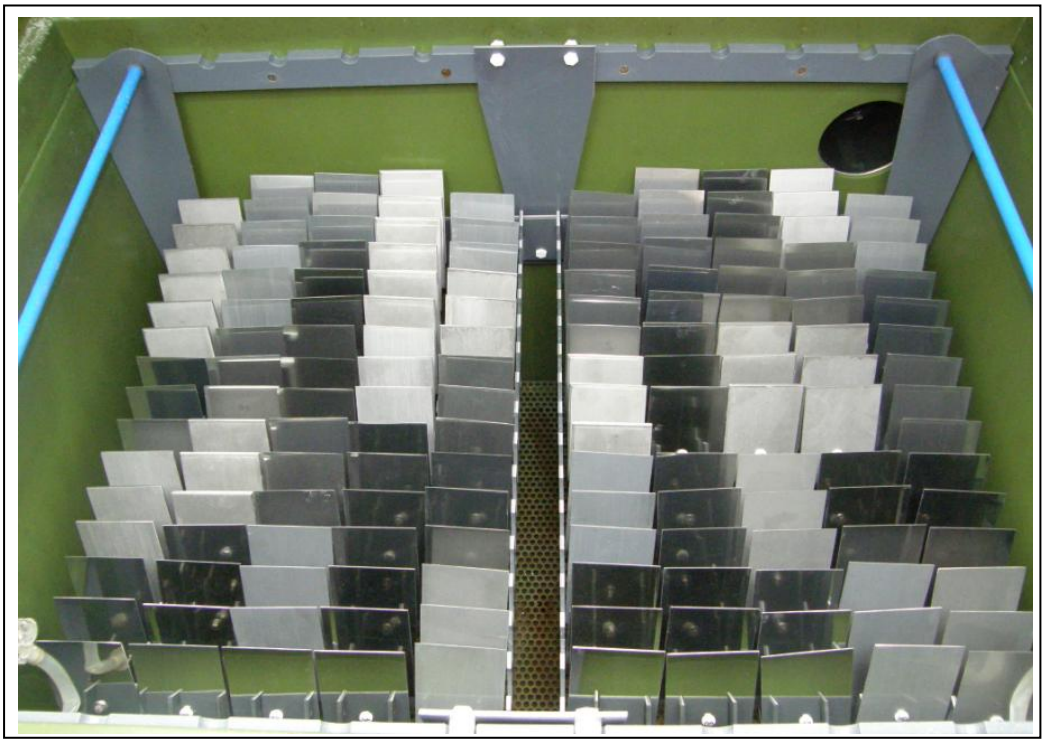


Figure 1.-Samples inside of climatic chamber

3.4. RESULTS AND EVALUATION

The samples were tested during 4 cycles. Every one consists of 6 steps explained in 3.1. The samples stayed inside the climatic chamber 672 hours. At the end of each cycle (168 hours), they have been observed and pictures of them have been taken in order to evaluate the different behaviors. In the annexes I, II and III pictures from the samples after first, third and fourth cycle respectively can be seen.

3.4.1. Qualitative evaluation

3.4.1.1. Flat samples

From the beginning of the test, the EN 1.4003 has been the most damaged grade. The stains appeared on the first 24 hours to salt salinity fog exposure. The classification from higher to lower deterioration is 1D finish from sample 1 (the highest rusty sample), 1D sample 2 and 2B sample 3, with nearly similar behavior, and finally 2B sample 4 (the lower rusty EN 1.4003 specimens).

The EN 1.4016 is another grade which shows brown points in its surface. The rusty products come, either on surface or edges. The 2B finish (12) is lower resistant than BA (13) regarding to the damage showed.

The EN 1.4509 is the following according to rusty products appearance. The 1D finishes are the lower resistant (5, 6). The 2B finish from sample 8 has some stains on its surface and some rusty products from the edges. The 2B finish from sample 7 only shows a few brown points on its surface. Finally the BA finish from sample 14 and 15 are the most resistant in this grade.

The EN 1.4521 2B finish from sample 9 showed some stains on its surface. Sample 10 have some of those stains on its surface. The BA finish (16) does not show any stains on their surface or edges.

The EN 1.4621 with 2M finish (11) does not show any significant stains on its surface.

The austenitic grade EN 1.4301 (17), used as reference, does not show any stains on its surface neither.

To sum up, the grade EN 1.4003 has been the most corroded during the test, followed by the EN 1.4016 in both finishes. The EN 1.4509 is the next according to stains appearance. The grade EN 1.4621, and the BA finishes in grades EN 1.4509 and EN 1.4521 do not show significant stains. The reference material EN 1.4301 does not show stains.

3.4.1.2. Bolted samples

The pictures about bolted samples before screws removing are included in the annexes I, II and III beside the flat samples. Detailed pictures about area under washers are shown following.

In the EN 1.4003 appears a high corrosion under either stainless steel or Teflon washers. All the area under washers is corroded (figure 2).

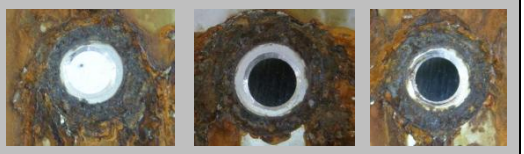
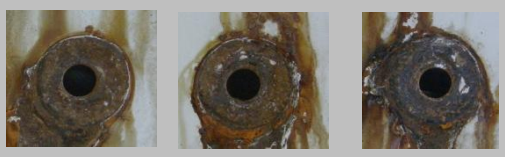
EN 1.4003		
IDENTIFICATION	STAINLESS STEEL WASHER	TEFLON WASHER
1 1D		
2 1D		
3 2B		
4 2B		

Figure 2.- Area under washers in EN 1.4003

The EN 1.4016 is the second highly attacked grade (figure 3). Both finishes 2B (12) and BA (13) have a high corroded area under washer, where there is a not clear different behavior between them. In figure 4 it can be seen that there is a clear lack of material on surface as a result of crevice corrosion. Again, there is not a clear difference between stainless steel and Teflon washer influence.

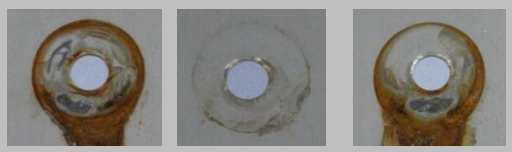
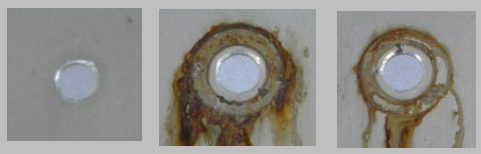
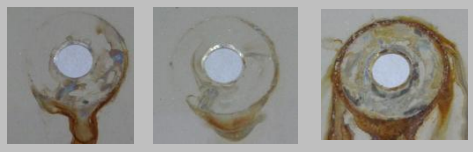
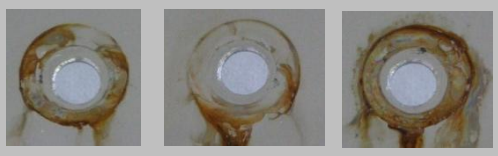
EN 1.4016		
IDENTIFICATION	STAINLESS STEEL WASHER	TEFLON WASHER
12 2B		
13 BA		

Figure 3.- Area under washers in EN 1.4016

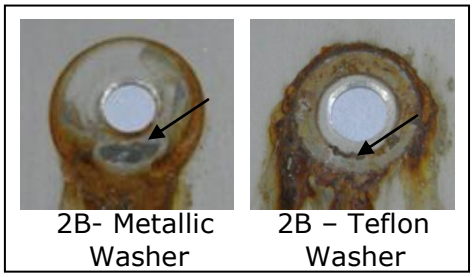


Figure 4.- Removed area by crevice corrosion in EN 1.4016

In the EN 1.4509 grade, it can not be observed a big difference between finishes influence according to corrosion under washers. The 2B finish (7, 8) may seem with a very similar behavior than the 1D (5, 6). In the BA finish (14, 15) there are a high number of specimens with less corrosion attack on the crevice area under washers. There is not any difference between stainless steel and Teflon washer.

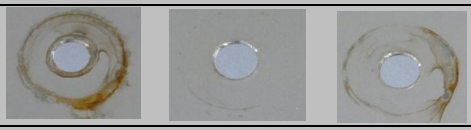
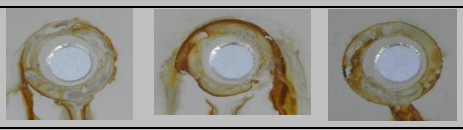
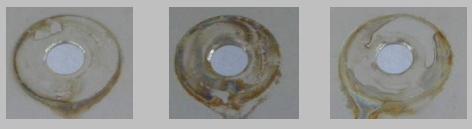
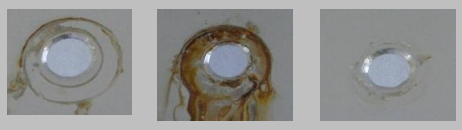
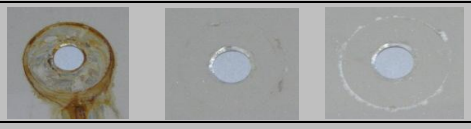
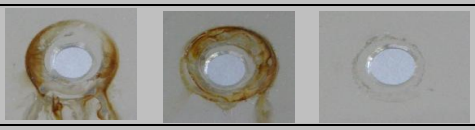
EN 1.4509		
IDENTIFICATION	STAINLESS STEEL WASHER	TEFLON WASHER
5 1D		
6 1D		
7 2B		
8 2B		
14 BA		
15 BA		

Figure 5.- Area under washers EN 1.4509

The EN 1.4521 only shows slight rusty products around samples in some specimens. The area under metallic washers looks more stained that under Teflon ones. No clear differences between 2B and BA finish are observed. In general the EN 1.4521 behavior looks better than the EN 1.4509.

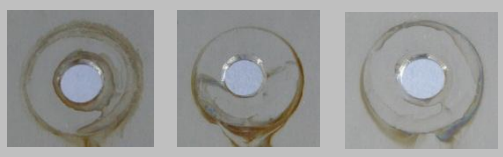
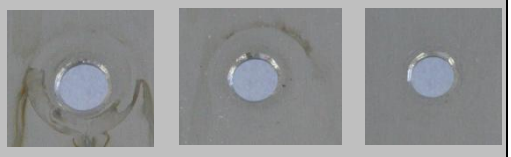
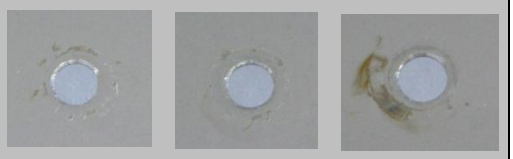
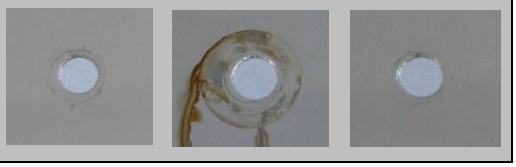
EN 1.4521		
IDENTIFICATION	STAINLESS STEEL WASHER	TEFLON WASHER
9 2B		
10 2B		
16 BA		

Figure 6.- Area under washers EN 1.4521

The EN 1.4621 shows a similar behavior than the EN 1.4521 attending to area under washers. Only slight corrosion is found in some specimens around washers, under metallic ones the specimen is slightly more stained than in Teflon ones, although the attack does not produce pouring of rusty products as another stainless steel grades (figure 7).

EN 1.4621		
IDENTIFICATION	STAINLESS STEEL WASHER	TEFLON WASHER
11 2M		

Figure 7.- Area under washers EN 1.4621

In the austenitic grade EN 1.4301, only very slight stains are found around or under washers in some specimens, either in the case of stainless steel or Teflon washers (figure 8).

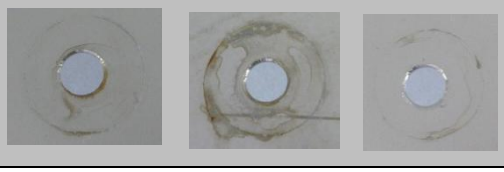
EN 1.4301		
IDENTIFICATION	STAINLESS STEEL WASHER	TEFLON WASHER
17 2B		

Figure 8.- Area under washers EN 1.4301

3.4.2. Quantitative evaluation

3.4.2.1. Flat samples

In the flat samples the quantitative evaluation is carried out by means of the weight variation in samples. The specimens have been weighted before and after the test. When the test finished, the samples were rinsed by water in order to remove salts deposition, then the samples were weighted. In table 8 the weight variation is showed. Red color means samples with mass loss, blue color samples with weight gain and white color samples without weight variation.

Flat	WEIGHT (g)
1-A	0,14
1-B	-0,41
1-C	-1,13
2-A	0,11
2-B	0,34
2-C	0,08
3-A	0,2140
3-B	0,1077
3-C	0,0388
4-A	-0,0101
4-B	0,0331
4-C	0,0176
5-A	0,03
5-B	0,02
5-C	0,04
6-A	0,01
6-B	0,01
6-C	0,00
7-A	0,0000
7-B	0,0009
7-C	0,0021
8-A	0,0018
8-B	0,0036
8-C	0,0012
9-A	0,0000
9-B	0,0007
9-C	0,0008

Flat	WEIGHT (g)
10-A	0,0015
10-B	0,0002
10-C	0,0030
11-A	0,0009
11-B	0,0007
11-C	0,0010
12-A	0,0002
12-B	0,0001
12-C	0,0002
13-A	0,0012
13-B	0,0009
13-C	0,0003
14-A	0,0008
14-B	0,0001
14-C	0,0006
15-A	0,0004
15-B	0,0014
15-C	0,0002
16-A	0,0002
16-B	0,0001
16-C	0,0002
17-A	0,0018
17-B	0,0017
17-C	0,0018

Table 8.- Weights variation after climatic chamber test

In order to ease result analysis, in figure 9 the percentage of weight loss is shown, this means the weight loss according to the mass of every specimen.

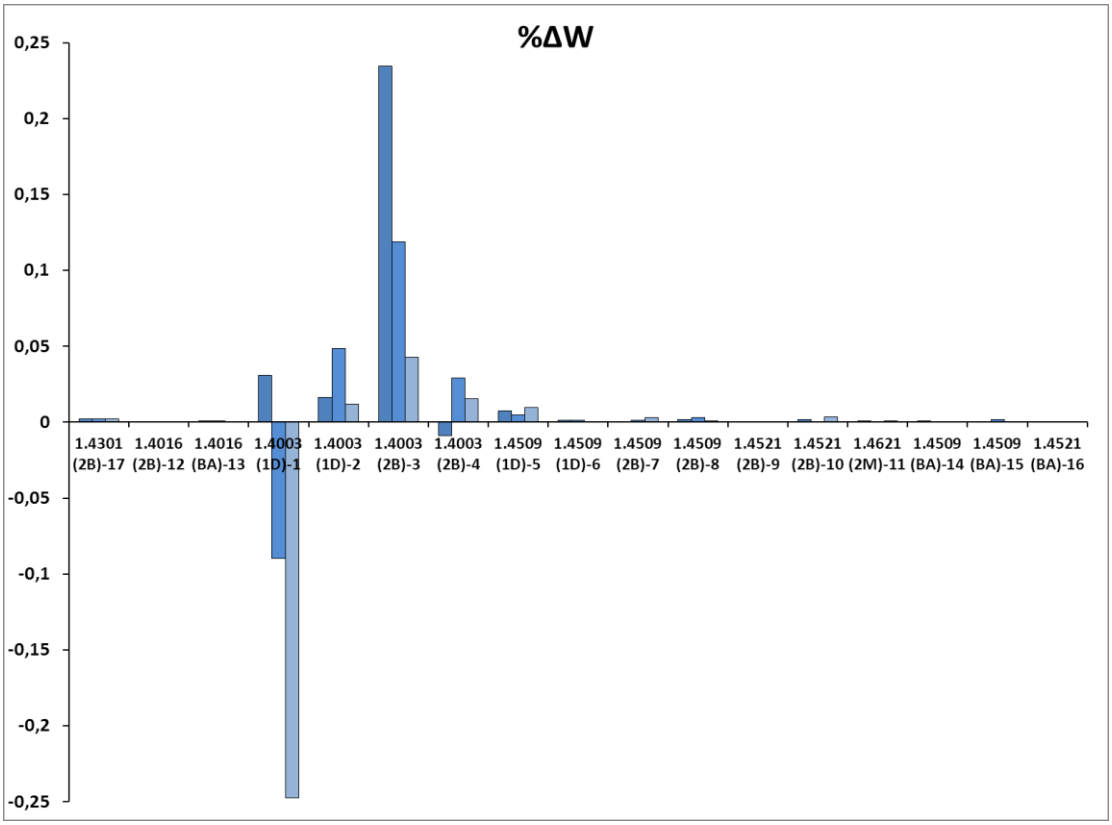


Figure 9.- Mass variation in flat samples

In the figure it can be observed as the sample 3 has suffered an increase in weight, this is due to corrosion products on the surface. On the other hand, the sample number 1 has shown a decrease in weight because of removing the high quantity of corrosion from its surface. In the rest of samples the mass variation is close to 0.

Due to EN 1.4003 samples have suffered weight variation with loss and gain, it has been decided to clean the samples in order to eliminate totally corrosion products.

3.4.2.2. Chemical cleaning

Specimens from 1 to 4 are decided to clean chemically in order to remove corrosion products and obtain the quantity of base material loss during the test.

In table 9 the samples which are going to be cleaned chemically are included.

Stainless steel	Finish	Identification
EN 1.4003	1D	1-A
		1-B
		1-C
	1D	2-A
		2-B
		2-C
	2B	3-A
		3-B
		3-C
	2B	4-A
		4-B
		4-C

Table 9.- Samples for chemical cleaning

Figure 10 shows the aspect of the specimens before cleaning procedure.

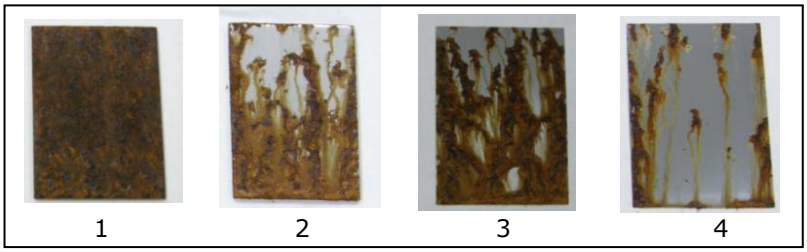


Figure 10.- EN 1.4003 samples with rust

The chemical cleaner is made by HCl (18% w/w), HNO_3 (4% w/w) and water. Samples are introduced in the cleaning solution during 10 minutes then they are washed by water, dried and weighted (this procedure is repeated up to mass loss is constant). When the mass loss is constant, oxides are removed from sample and the weight immediately before of that cleaning cycle is considered to obtain the final mass loss. Further information about the chemical cleaning is included in annex IV.

Finally, the corrosion rate under the artificial conditions applied on this test is obtained by means of equation 1.

$$R_{corr(mmpy)} = \frac{K \times W}{A \times T \times D}$$

Eq. 1

· K = 8.76×10^4 (constant millimetres per year, mmpy)
· T = exposure time (hours)
· A = area (cm^2)
· W = mass loss (g)
· D = density (g/cm^3, 7.7)

The mass loss and corrosion rate is gathered to table 10.

Identification	Mass loss (g)	R_{corr} (mmpy)
1-A	5,51	0,2915
1-B	6,86	0,3629
1-C	6,16	0,3259
2-A	1,41	0,0723
2-B	1,96	0,1006
2-C	2,28	0,1170
3-A	2,3630	0,1316
3-B	3,6208	0,2016
3-C	4,4709	0,2490
4-A	1,7732	0,0984
4-B	3,2931	0,1828
4-C	4,8773	0,2707

Table 10.- Mass loss after cleaning process

Corrosion rate comparison from the specimens can be observed in figure 11.

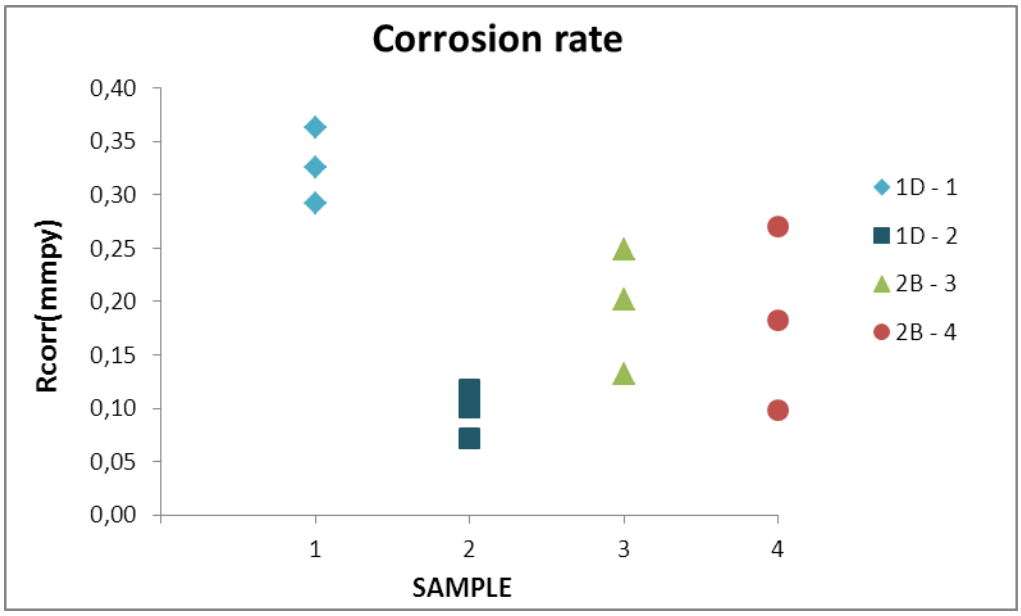


Figure 11.- Corrosion rate of EN 1.4003 samples

It can be observed as the hot rolled (1D) sample 1 has a higher corrosion rate. It can be noticed the good performance of the 1D specimens from sample 2, and the nearly similar performance from the 2B finish specimens with a lower repeatability of results in sample 4.

The worst performance of sample 1 may be associated to the quality of the surface. A lower quality eases chlorides deposition and they attack stainless steel surface.

3.4.2.3. Bolted samples

3.4.2.3.1. Weight variation

The bolted samples have been evaluated by two ways. The first of them is a simple weight measurement before and after test (table 11, figure 11).

Stainless steel washer	WEIGHT (g)	Teflon washer	WEIGHT (g)
1-D	-0,47	1-G	0,35
1-E	-0,71	1-H	-1,27
1-F	-1,41	1-I	-1,22
2-D	0,07	2-G	0,08
2-E	0,29	2-H	0,09
2-F	0,08	2-I	-0,04
3-D	0,2142	3-G	0,7133
3-E	0,0993	3-H	0,0167
3-F	0,2471	3-I	0,1554
4-D	-0,0411	4-G	-0,0153
4-E	0,0078	4-H	-0,0060
4-F	0,0068	4-I	0,0074
5-D	0,05	5-G	0,00
5-E	0,01	5-H	0,05
5-F	0,00	5-I	0,00
6-D	0,01	6-G	0,00
6-E	0,00	6-H	0,00
6-F	0,01	6-I	0,01
7-D	0,0007	7-G	0,0000
7-E	0,0014	7-H	0,0011
7-F	0,0032	7-I	0,0018
8-D	0,0045	8-G	0,0000
8-E	0,0016	8-H	0,0029
8-F	0,0070	8-I	0,0018

Stainless steel washer	WEIGHT (g)	Teflon washer	WEIGHT (g)
9-D	0,0009	9-G	0,0002
9-E	0,0023	9-H	0,0009
9-F	0,0003	9-I	0,0003
10-D	0,0009	10-G	0,0018
10-E	0,0018	10-H	0,0020
10-F	0,0022	10-I	0,0032
11-D	0,0013	11-G	0,0013
11-E	0,0015	11-H	0,0011
11-F	0,0011	11-I	0,0008
12-D	0,0010	12-G	0,0010
12-E	0,0003	12-H	0,0012
12-F	0,0022	12-I	0,0008
13-D	0,0184	13-G	0,0079
13-E	0,0146	13-H	-0,0114
13-F	-0,0146	13-I	-0,0174
14-D	0,0015	14-G	0,0016
14-E	0,0021	14-H	0,0024
14-F	0,0017	14-I	0,0012
15-D	0,0007	15-G	0,0012
15-E	0,0002	15-H	0,0014
15-F	0,0003	15-I	0,0090
16-D	0,0011	16-G	0,0001
16-E	0,0009	16-H	0,0053
16-F	0,0013	16-I	0,0008
17-D	0,0021	17-G	0,0029
17-E	0,0056	17-H	0,0027
17-F	0,0053	17-I	0,0048

Table 11.- Weight variation after climatic chamber test in bolted samples

Data from table 11 are represented in figure 12 as percentage of weight loss (weight loss/initial weight of the specimen).

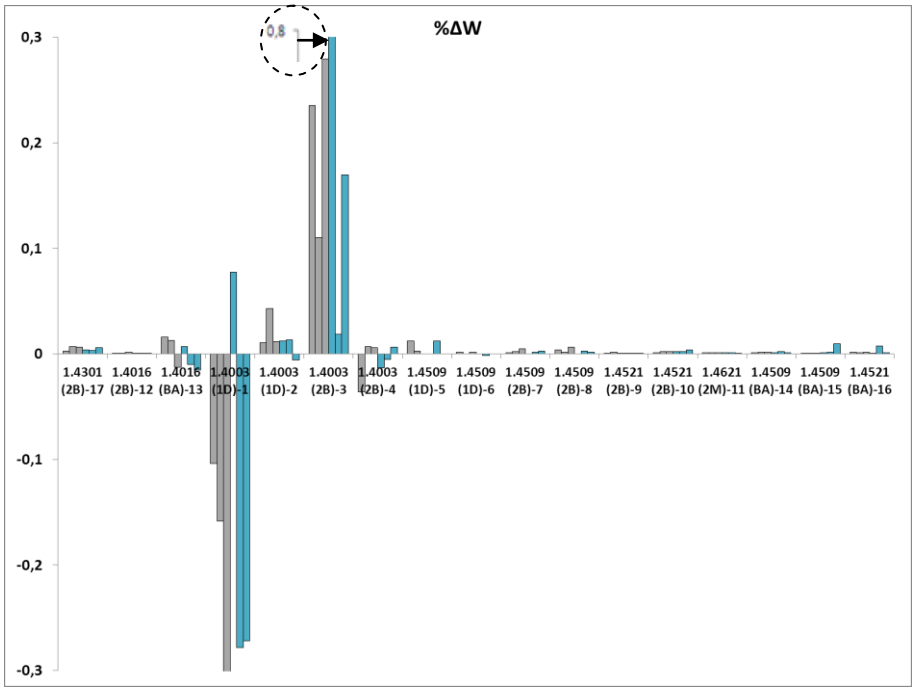


Figure 12.- Mass variation in bolted samples

In this figure, the samples 1 have suffered a high corrosion attack and the removing of the corrosion products explains their mass loss. In the case of the sample 3 their highest weight increase can be explained by a high quantity of corrosion products but these products over the surface.

If data from EN 1.4003 samples are removed (figure 13), it can be observed as sample 13 (EN 1.4016) has lost weight too. This fact is explained by the high crevices corrosion under washers, because when washers are removed some corrosion products are removed too, so it has lost base material.

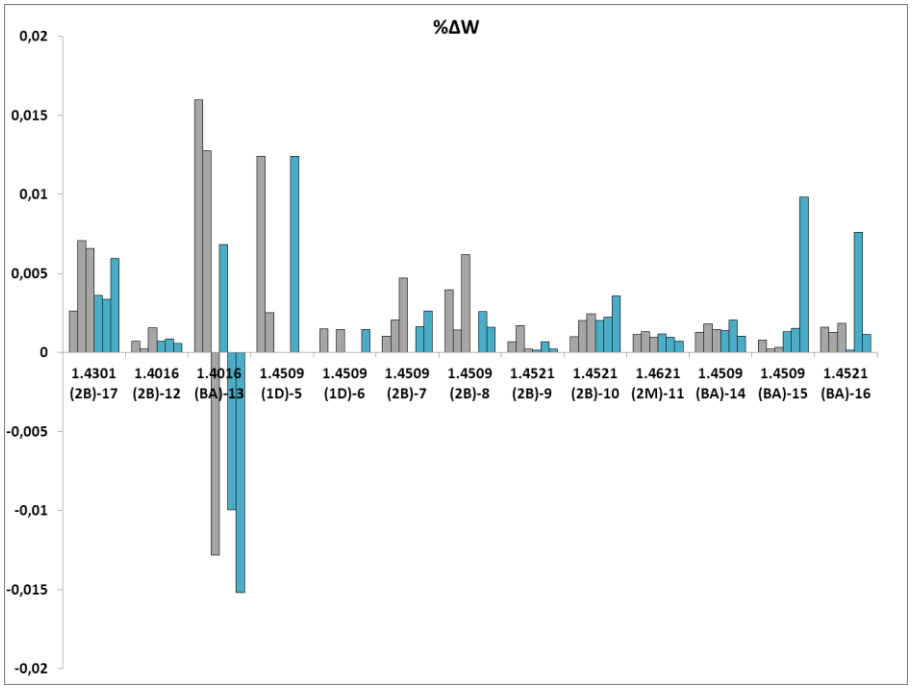


Figure 13.- Mass variation in bolted samples (EN 1.4003 removed)

3.4.2.3.2. Crevice corrosion

In order to obtain as many information as possible from the test, the second way to evaluate bolted samples consists of quantifying the crevice corrosion under washers. For this purpose, in the laboratory of Acerinox, an easy and suitable procedure to perform this task has been elaborated.

The procedure is described following.

1. At first, corrosion products are removed from the area under washer using a commercial stainless steel cleaner (figure 14).

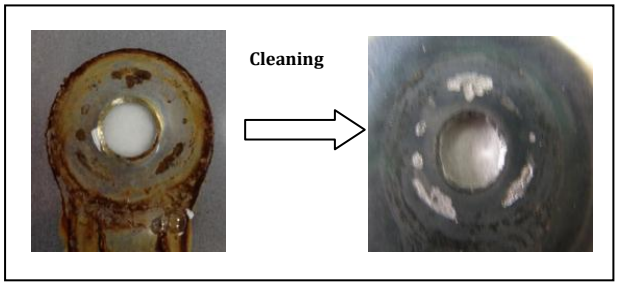


Figure 14.- Removing of rusty products

2. It is necessary to delimitate the area susceptible from suffer crevice corrosion, area under washer. On the surface of a clean specimen this area is drawn as figure 15 shows. This step is only performed once for metallic and another for Teflon washer.

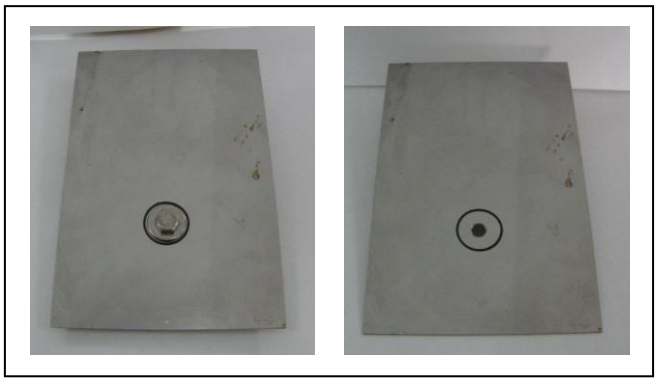


Figure 15.- Delimitation of the surface under washers

3. The specimen is photocopied with a magnification as big as possible.

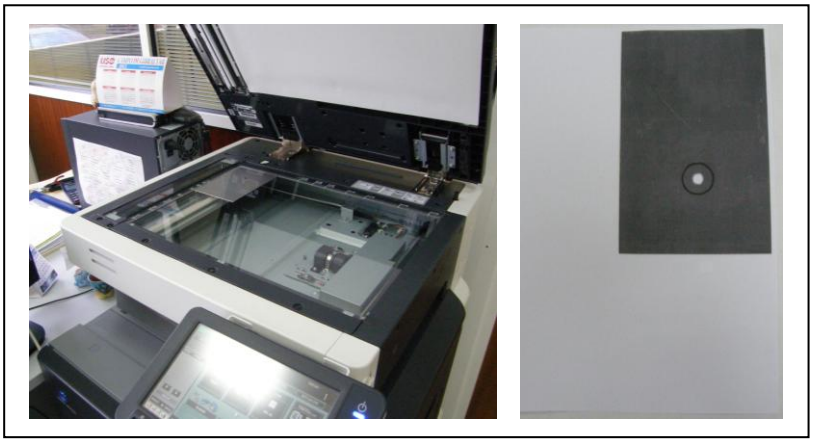


Figure 16.- Photocopy of the sample

4. The delimitated area corresponds to the 100% of the area which is able to suffer crevice corrosion. This area is cut from the photocopy and use to establish the 100 % of the area susceptible to suffer crevice corrosion in the photocopies of the rest of samples (figure 17).

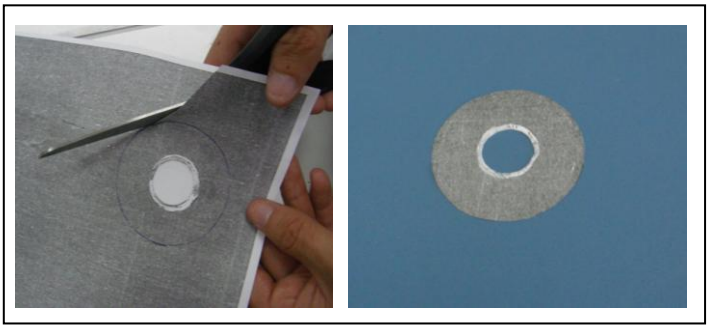


Figure 17.- Area used to establish the 100% of the area susceptible to suffer crevice corrosion

5. Every sample which has suffered crevice corrosion is photocopied with the same magnification of step 3. The samples have been cleaned previously.

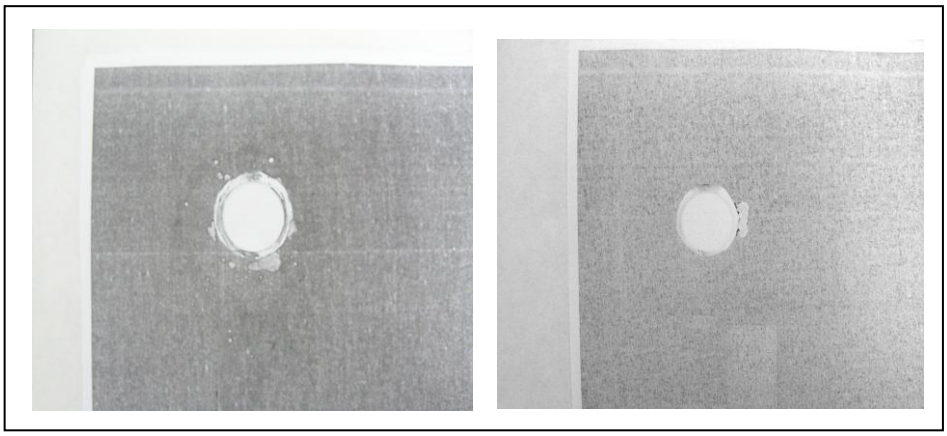


Figure 18.- Photocopy of samples

6. The area established in step 4 is used to delimitate the area under washer in the copy of the samples (figure 19).

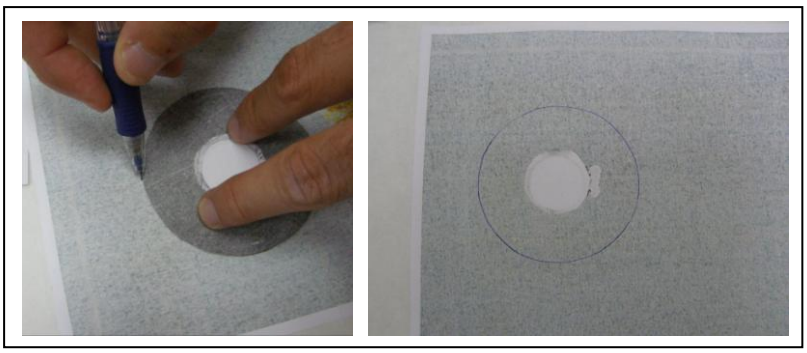


Figure 19.- Delimitation of the area under washers in photocopies

7. Then, the area is cut as figure 20 shows. The weight of this area is W1.

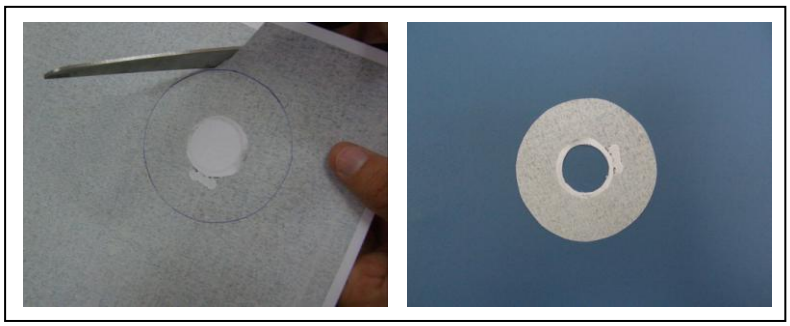


Figure 20.- Area under washers: W1

8. Corroded areas by crevice corrosion are identified in this photocopy. The specimens are inspected by microscope to check that the total corroded area is shown in the photocopy. Finally, corroded areas are cut and removed from the photocopy. The photocopy is weight and this value corresponds to W2.

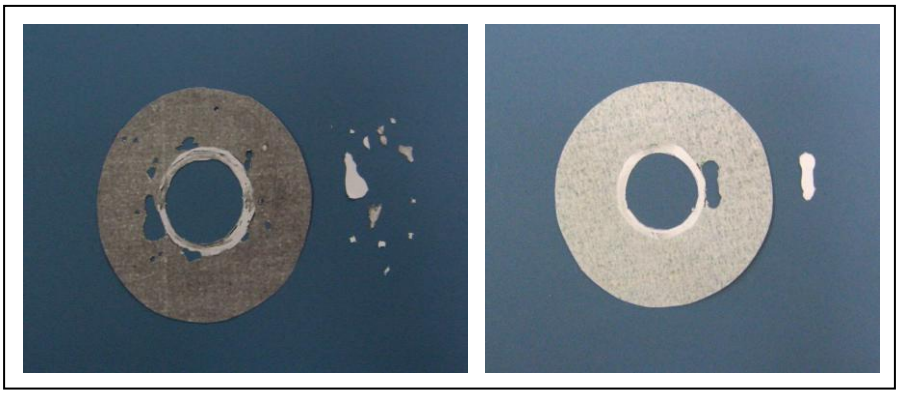


Figure 21.- Area under washers without crevice corroded areas: W2

9. With the aforementioned measures, the percentage of attacked area by crevice corrosion is calculated by equation 2.

$$\%\ Crevice = \frac{(W_1 - W_2)}{W_1} \times 100 \qquad \text{Eq.2}$$

The obtained data are gathered to table 12. Samples from 1 to 4 have not been analyzed by the aforementioned procedure, because all the area under washers has been corroded (100%).

Metallic washer			Teflon washer		
Sample	**% Crevice**	**Average**	**Sample**	**% Crevice**	**Average**
1 – D	100	100	1 – G	100	100
1 – E	100		1 - H	100	
1 - F	100		1 – I	100	
2 – D	100	100	2 – G	100	100
2 – E	100		2 - H	100	
2 - F	100		2 – I	100	
3 – D	100	100	3 – G	100	100
3 – E	100		3 - H	100	
3 - F	100		3 – I	100	
4 – D	100	100	4 – G	100	100
4 – E	100		4 - H	100	
4 - F	100		4 – I	100	
5 – D*	2,17	0,72	5 – G	0,00	2,97
5 – E	0,00		5 – H*	7,29	
5 - F	0,00		5 – I*	1,62	
6 – D	1,90	0,63	6 – G	0,00	0,24
6 – E	0,00		6 - H	0,00	
6 - F	0,00		6 – I	0,73	
7 – D	0,44	0,23	7 – G	1,42	1,49
7 – E	0,00		7 - H	1,42	
7- F	0,24		7 – I	1,62	
8 – D	0,16	0,24	8 – G	0,37	2,10
8 – E	0,10		8 - H	5,92	
8 - F	0,46		8 – I	0,00	
9 – D	0,51	0,47	9 – G	0,31	0,10
9 – E	0,64		9 - H	0,00	
9 - F	0,27		9 – I	0,00	
10 – D	1,87	0,66	10 – G	0,31	0,14
10 – E	0,10		10 - H	0,00	
10 - F	0,00		10 – I	0,10	
11 – D	0,00	0,09	11 – G	0,00	0,05
11 – E	0,17		11 - H	0,05	
11 - F	0,10		11 – I	0,10	
12 – D	9,50	3,40	12 – G	0,00	3,90
12 – E	0,34		12 - H	8,18	
12 - F	0,37		12 – I	3,51	
13 – D	0,75	3,11	13 – G	1,26	1,61
13 – E	0,81		13 - H	1,42	
13 - F	7,77		13 – I	2,15	
14 – D	2,14	0,71	14 – G	0,26	0,44
14 – E	0,00		14 – H	1,05	
14 – F	0,00		14 - I	0,00	
15 – D	1,02	0,40	15 – G	0,42	0,21
15 – E	0,17		15 - H	0,21	
15 - F	0,00		15 – I	0,00	
16 – D	0,07	0,15	16 – G	0,00	0,12
16 – E	0,27		16 - H	0,37	
16 - F	0,10		16 – I	0,00	
17 – D*	0,07	0,08	17 – G	0,00	0,03
17 – E*	0,07		17 – H*	0,10	
17 – F*	0,10		17 – I	0,00	

* Low depth of crevices.

No crevices % < 1 % > 1

Table 12.- Percentage of corroded area under washers

In figure 22 the values from table 12 are shown in order to draw conclusions according to crevice corrosion performance.

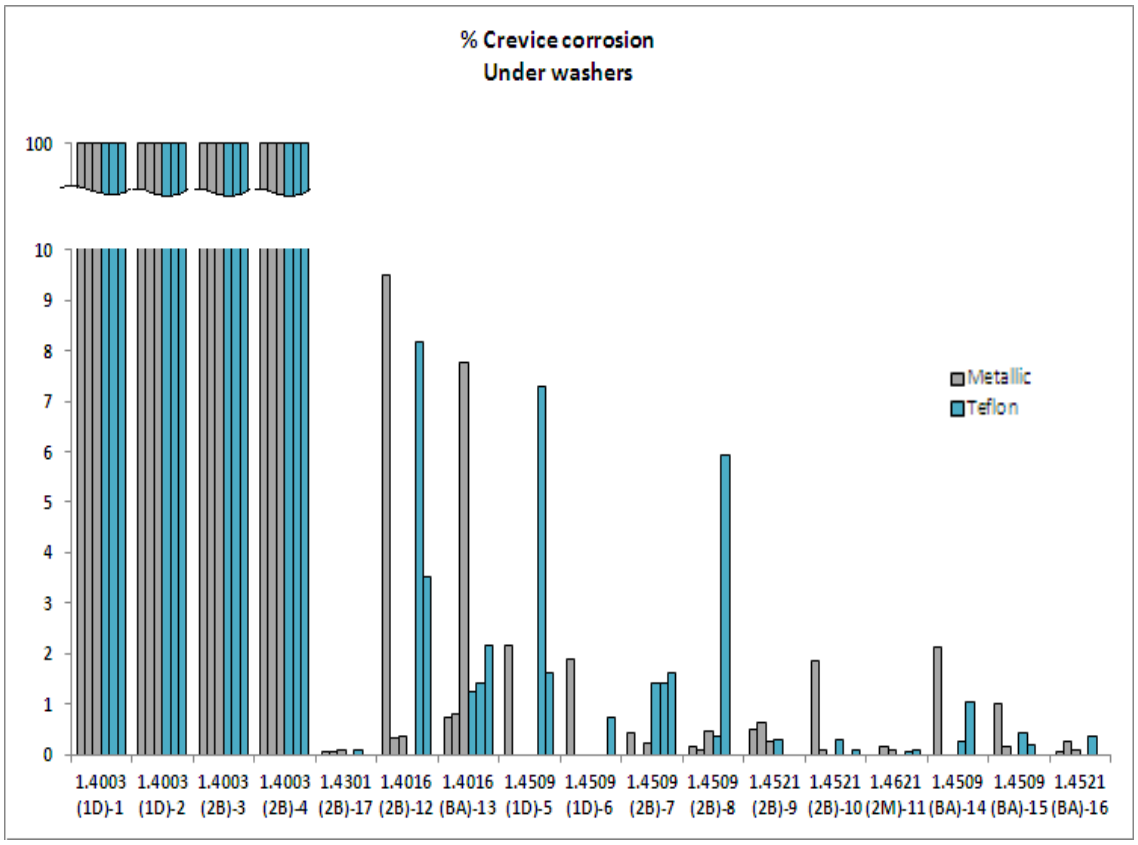


Figure 22.- Percentages of crevice corrosion

The samples 1, 2, 3 and 4 (EN 1.4003) are totally corroded under washers. The samples 12 and 13 (EN 1.4016 2B, and BA) are the most corroded, with either metallic or Teflon washers. The samples 5, 7 and 8 (EN 1.4509 1D, 2B, 2B) with Teflon washers have a slight significant percentage of area corroded too.

On the other hand, the samples 9, 16 (EN 1.4521 2B, BA), 11 (EN 1.4621 2M) and 17 (EN 1.4301 2B) shows the best behavior to crevice corrosion resistance with a similar performance of area under metallic and Teflon washers and practically 0 % of corroded area.

It should be noted that qualitative and quantitative procedures have shown similar results.

4 CONCLUSIONS

A laboratory accelerated test in climatic chamber has been carried out to evaluate the behavior of different ferritic grades. An austenitic grade has been included as a reference material. The design of samples included flat specimens, specimens with stainless steel screws and washers, and with stainless steel screw and Teflon washers. The duration of the test has been 672 hours.

Results are commented regarding to stainless steel grade resistance, finish influence, and different designs.

From a qualitative and qualitative evaluation in flat samples, the stainless steel grades may be ranked as follows from lower to higher corrosion resistance,

$$1.4003 << 1.4016 < 1.4509 \approx 1.4521 < 1.4621 < 1.4301$$

It must be commented the influence of finish in the result of the test. In highly corroded samples a clear different according to finishes is not found. The different ferritic with BA finish has not shown any significant stains in flat conditions.

By means of testing specimens with stainless steel and Teflon washers in this test, it is concluded that, the nature of the device for crevice formation is not clearly responsible for a higher or lower corrosion attack.

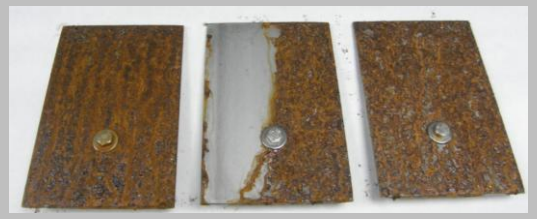
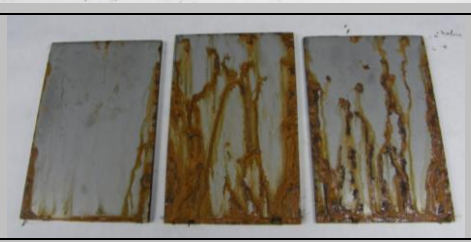
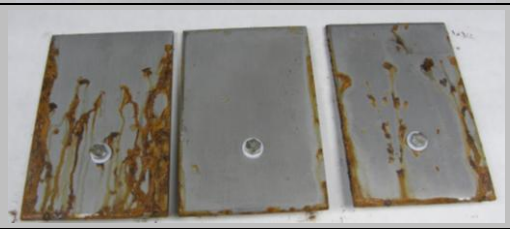
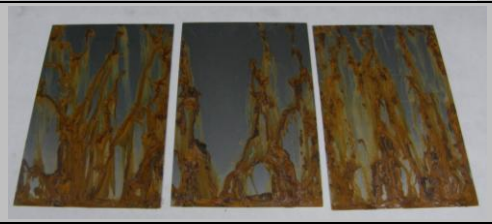
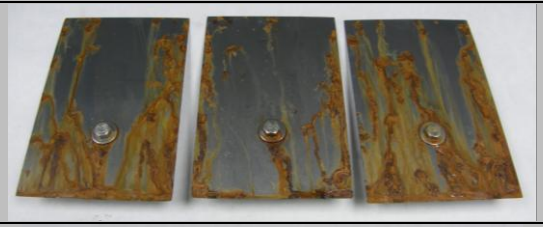
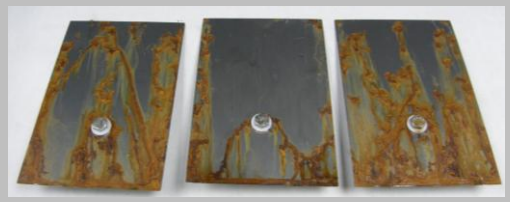
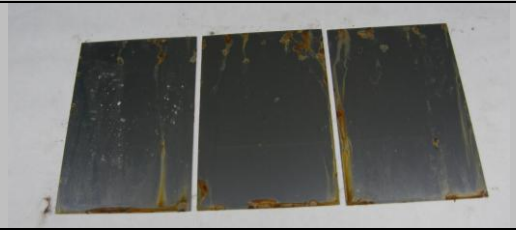
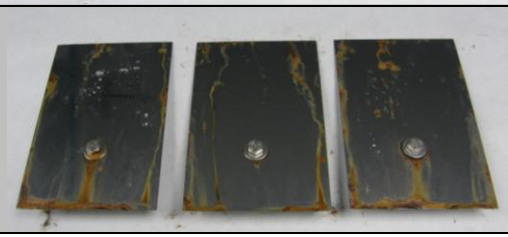
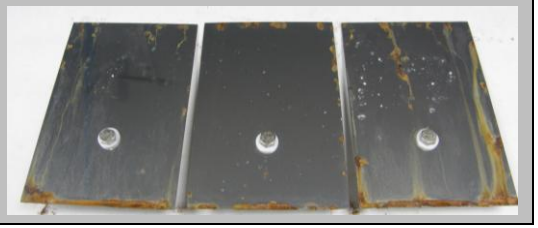
After evaluating surface under washers, the following classification from lower to higher crevice corrosion resistance may be concluded,

$$1.4003 << 1.4016 < 1.4509 < 1.4521 < 1.4621 < 1.4301$$

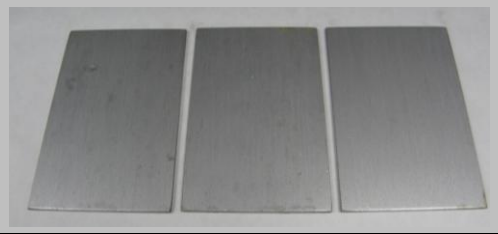
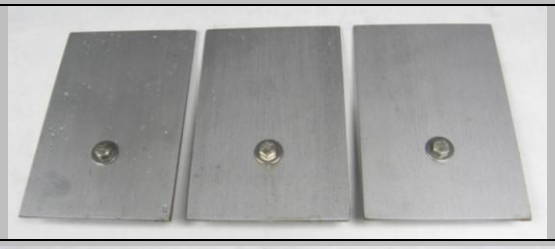
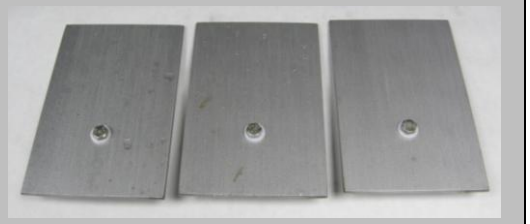
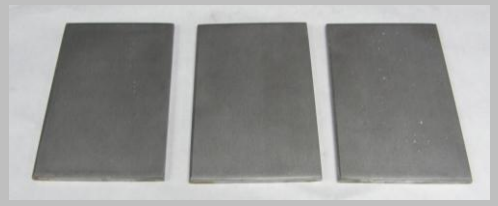
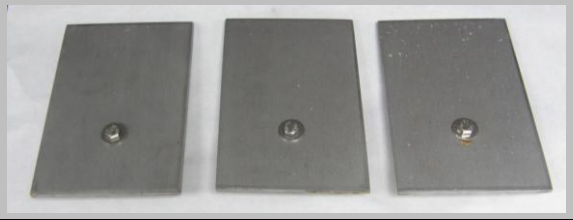
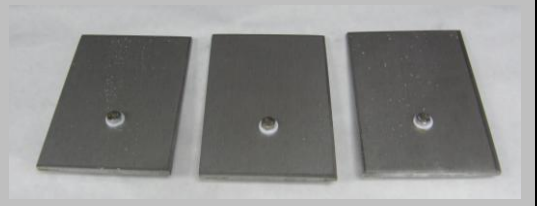
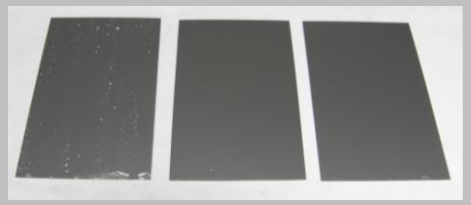
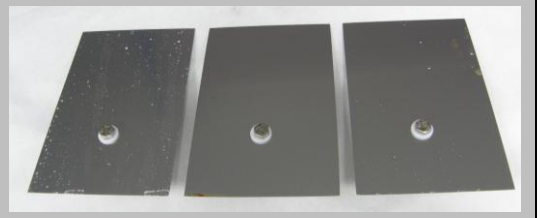
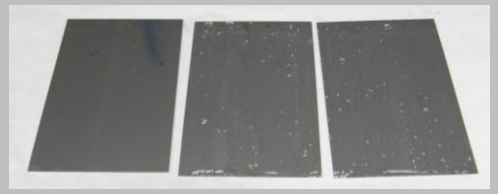
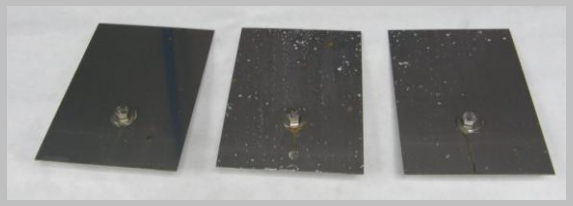
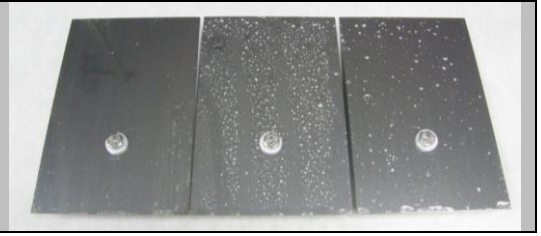
BA finish with stainless steel washer in EN 1.4521 has the closer behavior to EN 1.4301 with practically no signs of corrosion under washers.

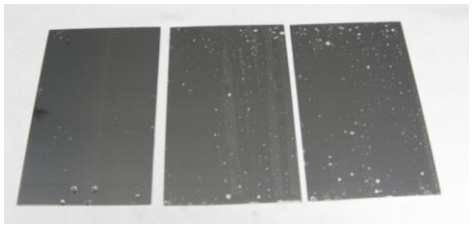
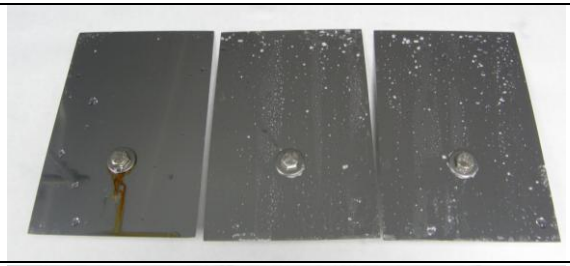
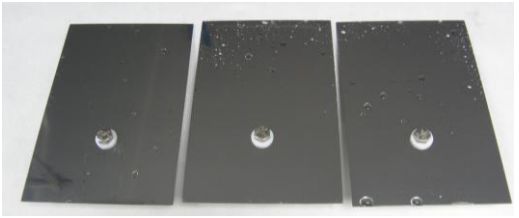
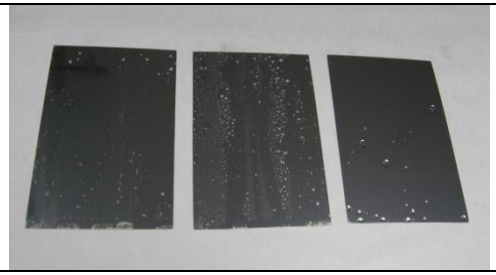
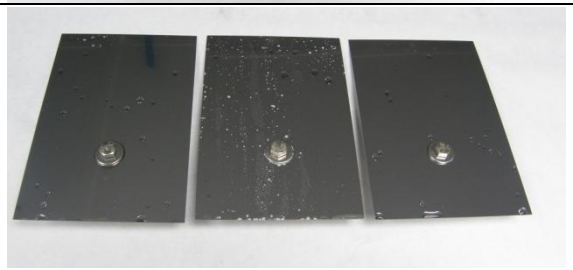
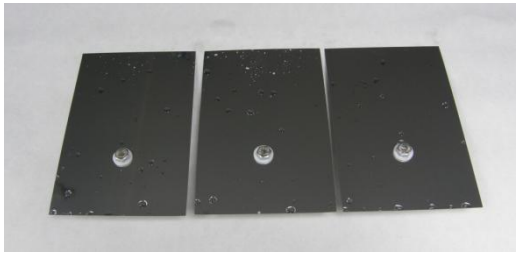
The test shows as crevices contribute to a worst behavior and corrosion appearance in stainless.

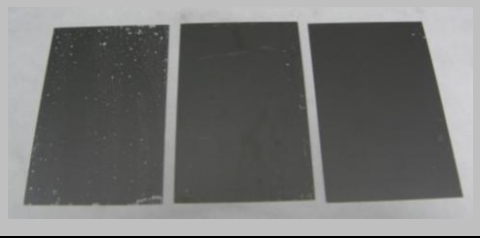
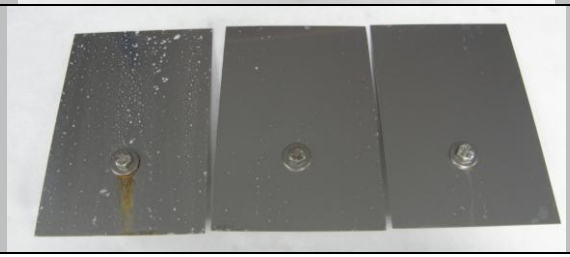
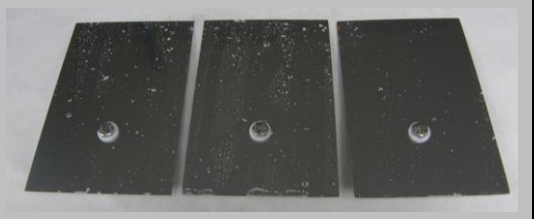
ANNEX I

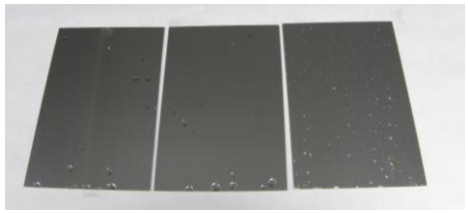
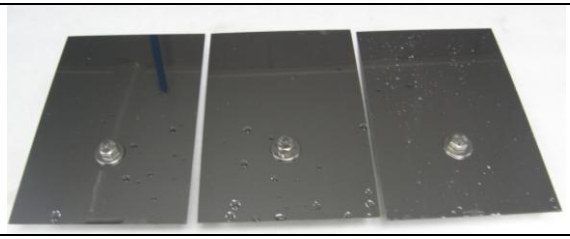
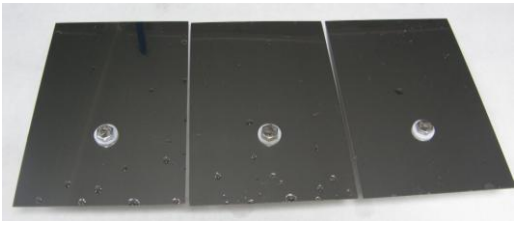
EN 1.4003			
IDENTIFICATION	FLAT	STAINLESS STEEL WASHER	TEFLON WASHER
1 1D			
2 1D			
3 2B			
4 2B			

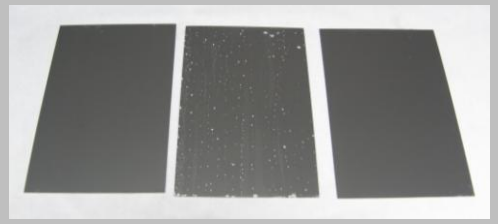
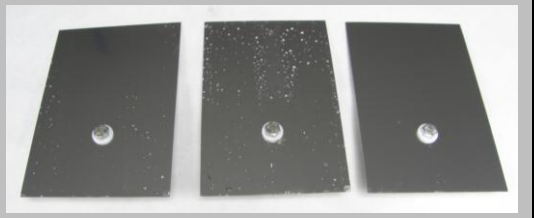
Gray background, samples on atmospheric corrosion test

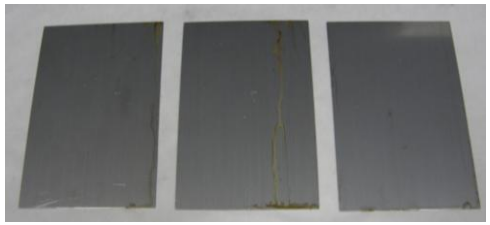
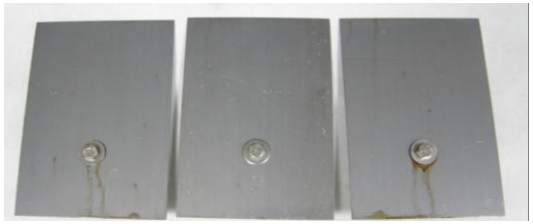
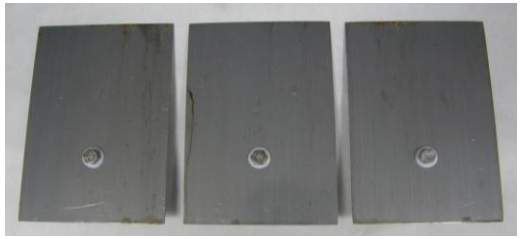
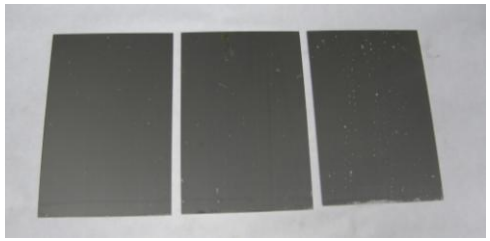
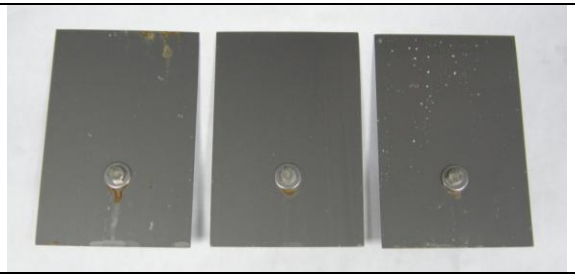
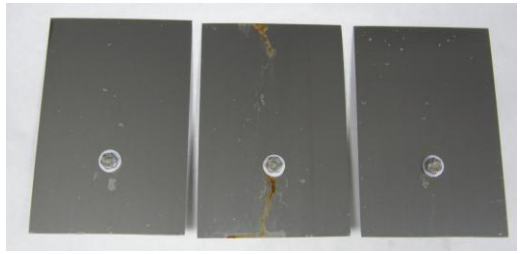
EN 1.4509			
IDENTIFICATION	FLAT	STAINLESS STEEL WASHER	TEFLON WASHER
5 1D			
6 1D			
7 2B			
8 2B			

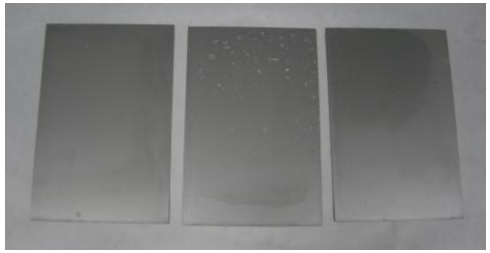
EN 1.4509			
IDENTIFICATION	FLAT	STAINLESS STEEL WASHER	TEFLON WASHER
14 BA			
15 BA			

EN 1.4521			
IDENTIFICATION	FLAT	STAINLESS STEEL WASHER	TEFLON WASHER
9 2B			
10 2B			

EN 1.4521			
IDENTIFICATION	FLAT	STAINLESS STEEL WASHER	TEFLON WASHER
16 BA			

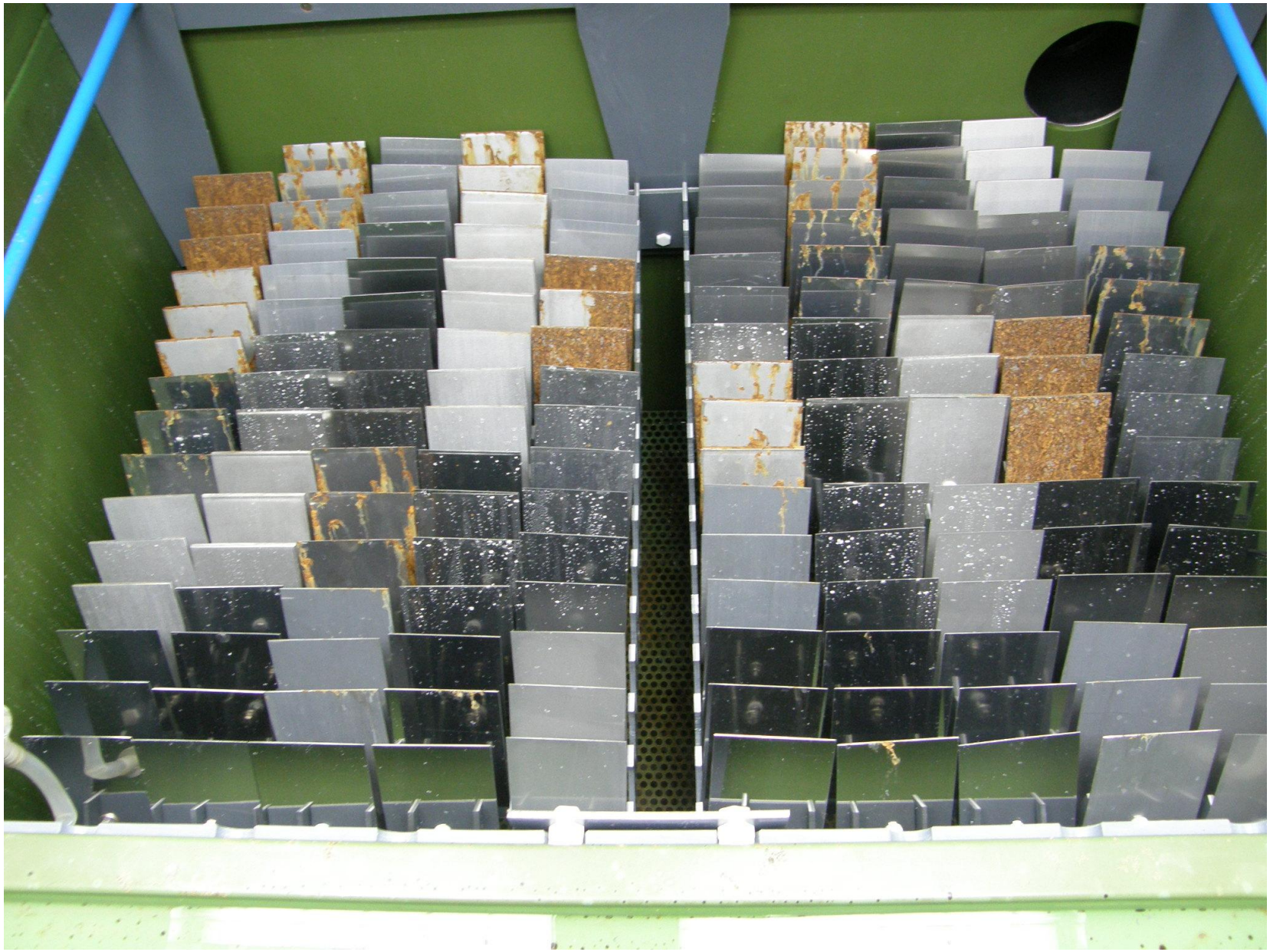
EN 1.4621			
IDENTIFICATION	FLAT	STAINLESS STEEL WASHER	TEFLON WASHER
11 2M			

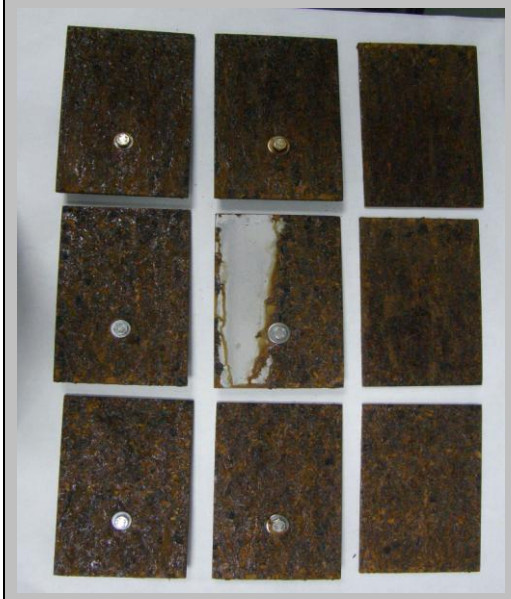
EN 1.4016			
IDENTIFICATION	FLAT	STAINLESS STEEL WASHER	TEFLON WASHER
12 2B			
13 BA			

EN 1.4301			
IDENTIFICATION	FLAT	STAINLESS STEEL WASHER	TEFLON WASHER
17 2B			

ANNEX II

General view of samples after three cycles:



EN 1.4003					
1 1D			2 1D		
TEFLON WASHER	*STAINLESS STEEL WASHER*	*FLAT*	*TEFLON WASHER*	*STAINLESS STEEL WASHER*	*FLAT*
					

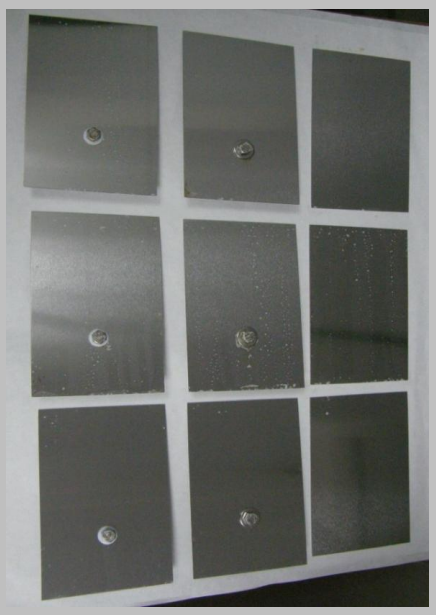
EN 1.4003					
3 2B			4 2B		
TEFLON WASHER	*STAINLESS STEEL WASHER*	*FLAT*	*TEFLON WASHER*	*STAINLESS STEEL WASHER*	*FLAT*
					

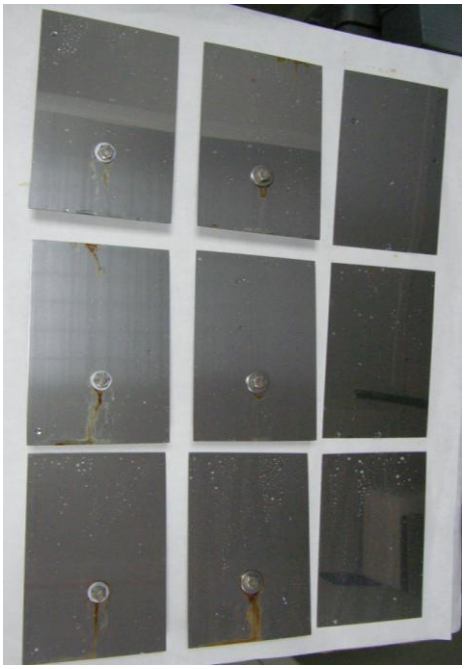
EN 1.4509					
5 1D			6 1D		
TEFLON WASHER	*STAINLESS STEEL WASHER*	*FLAT*	*TEFLON WASHER*	*STAINLESS STEEL WASHER*	*FLAT*
					

EN 1.4509					
7 2B			8 2B		
TEFLON WASHER	*STAINLESS STEEL WASHER*	*FLAT*	*TEFLON WASHER*	*STAINLESS STEEL WASHER*	*FLAT*
					

EN 1.4509					
14 BA			15 BA		
TEFLON WASHER	*STAINLESS STEEL WASHER*	*FLAT*	*TEFLON WASHER*	*STAINLESS STEEL WASHER*	*FLAT*
					

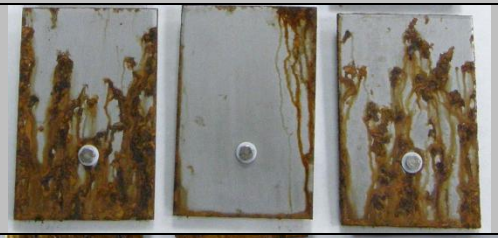
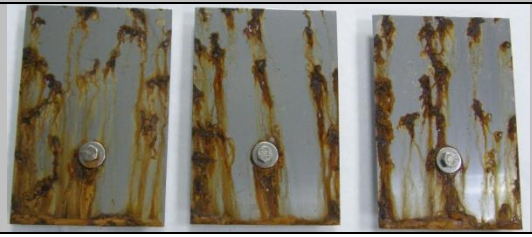
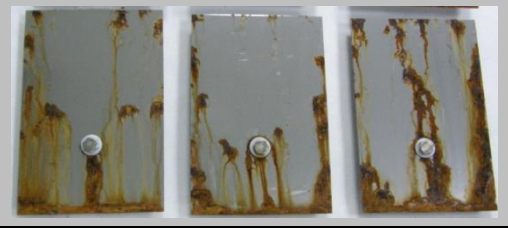
EN 1.4521					
9 2B			10 2B		
TEFLON WASHER	*STAINLESS STEEL WASHER*	*FLAT*	*TEFLON WASHER*	*STAINLESS STEEL WASHER*	*FLAT*
					

EN 1.4521			EN 1.4621		
16 BA			11 2M		
TEFLON WASHER	*STAINLESS STEEL WASHER*	*FLAT*	*TEFLON WASHER*	*STAINLESS STEEL WASHER*	*FLAT*
					

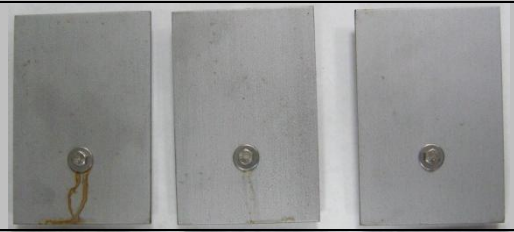
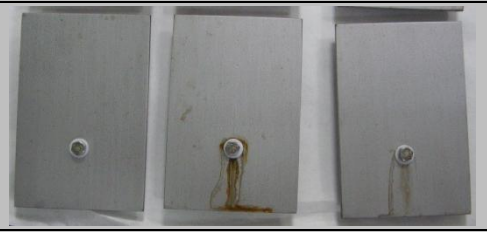
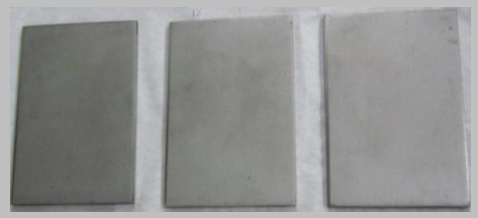
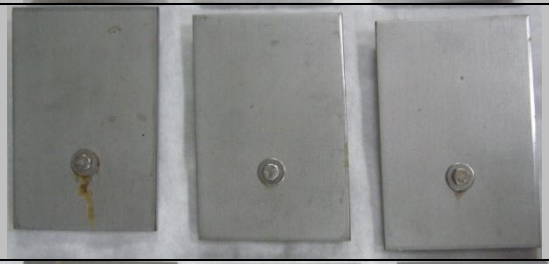
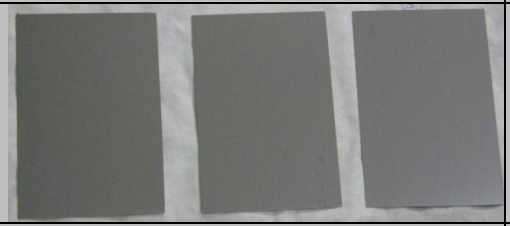
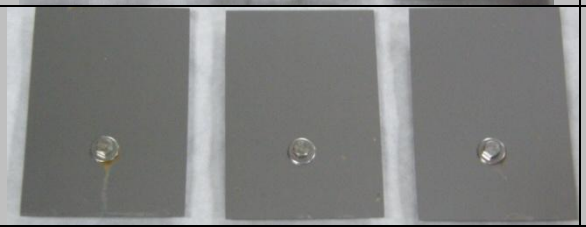
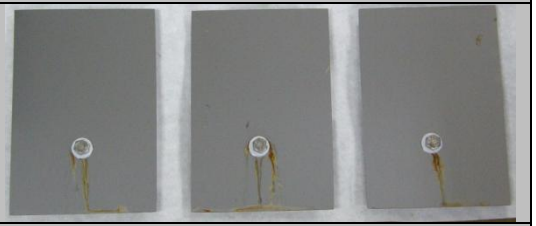
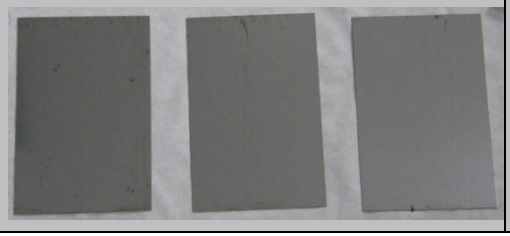
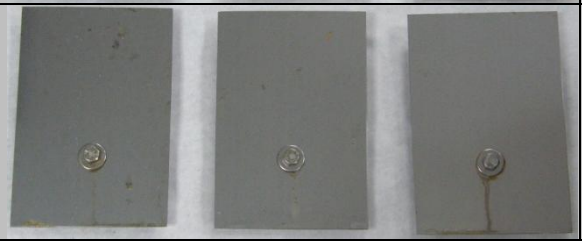
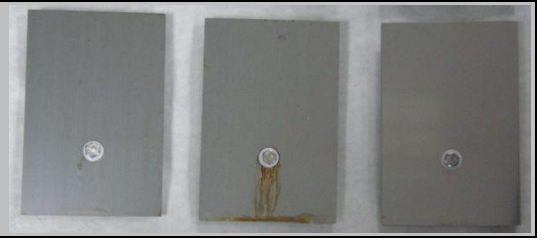
EN 1.4016					
12 2B			13 BA		
TEFLON WASHER	*STAINLESS STEEL WASHER*	*FLAT*	*TEFLON WASHER*	*STAINLESS STEEL WASHER*	*FLAT*
					

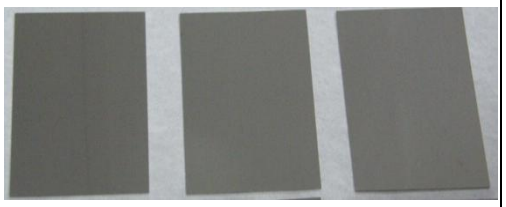
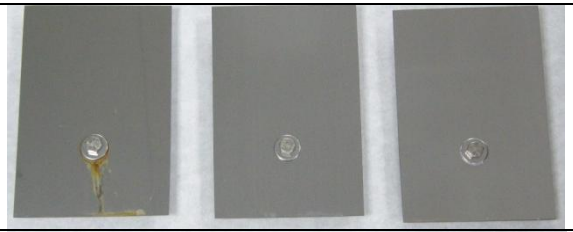
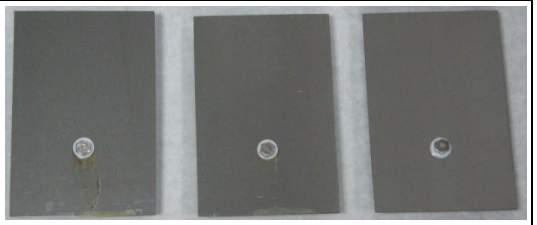
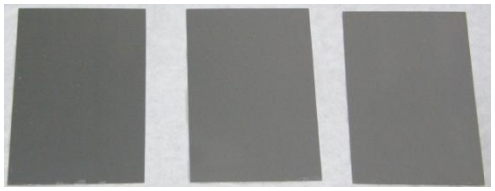
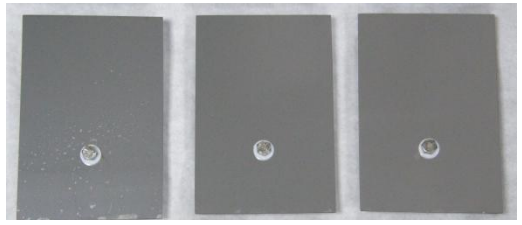
EN 1.4301		
17 2B		
TEFLON WASHER	*STAINLESS STEEL WASHER*	*FLAT*
		

ANNEX III

EN 1.4003			
IDENTIFICATION	FLAT	STAINLESS STEEL WASHER	TEFLON WASHER
1 1D			
2 1D			
3 2B			
4 2B			

Gray background, samples on atmospheric corrosion test

EN 1.4509			
IDENTIFICATION	FLAT	STAINLESS STEEL WASHER	TEFLON WASHER
5 1D			
6 1D			
7 2B			
8 2B			

EN 1.4509			
IDENTIFICATION	FLAT	STAINLESS STEEL WASHER	TEFLON WASHER
14 BA			
15 BA			

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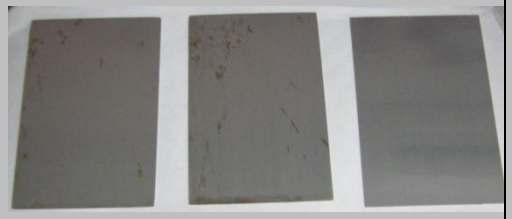
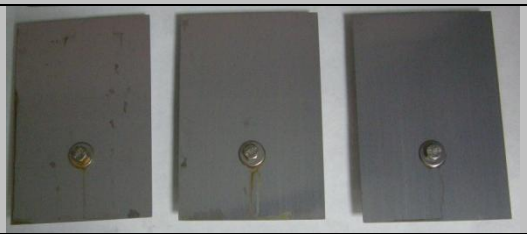
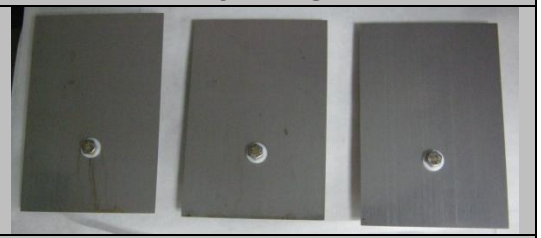
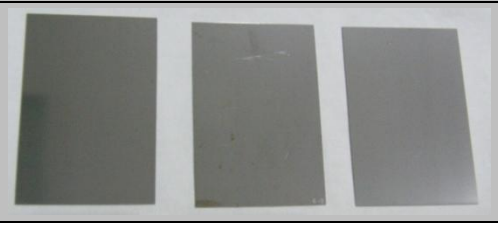
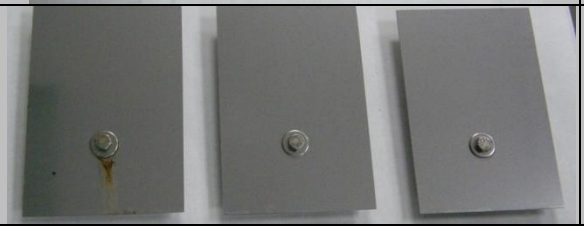
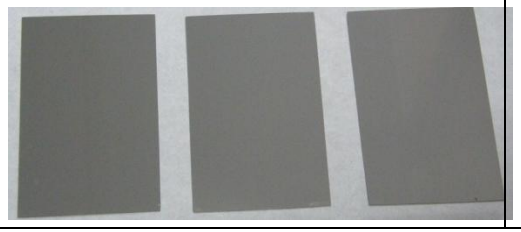
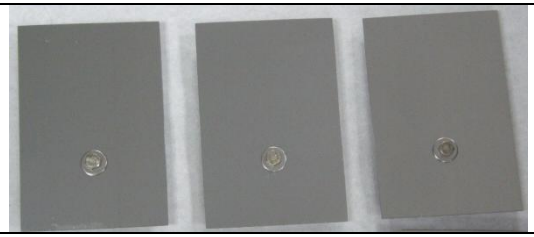
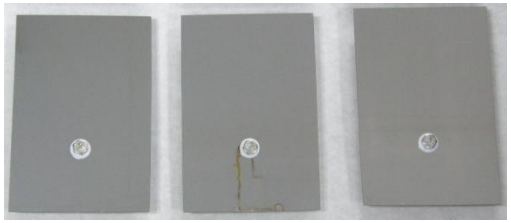
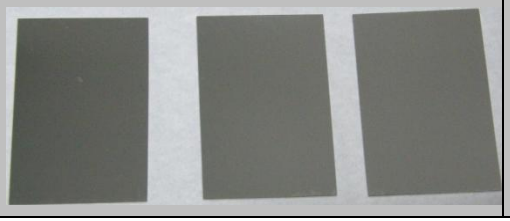
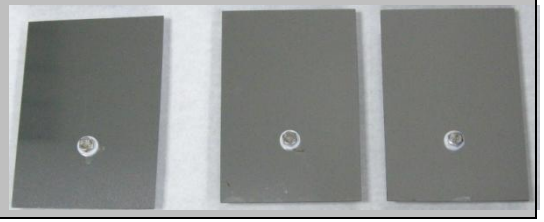
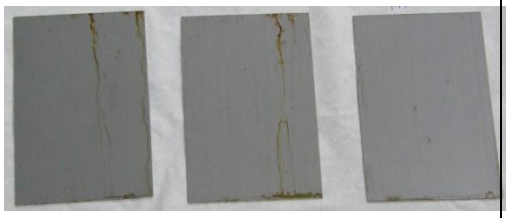
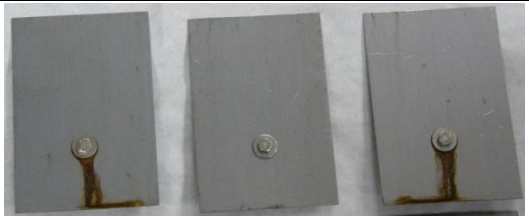
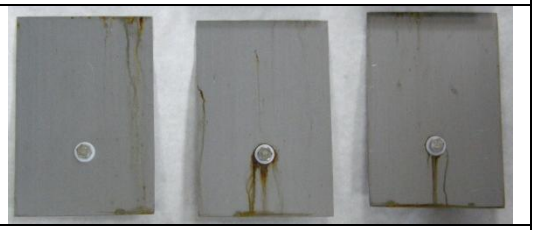
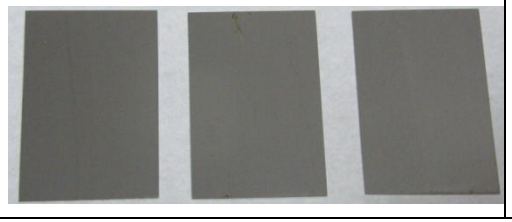
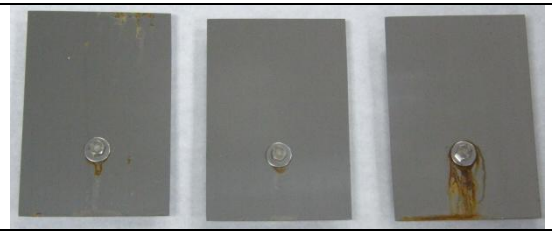
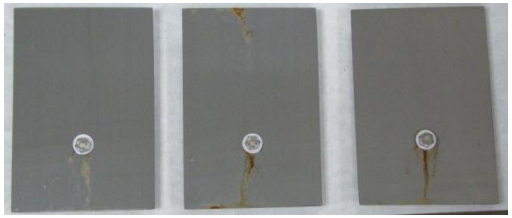
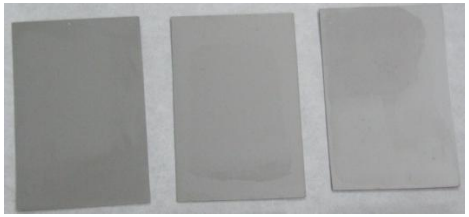
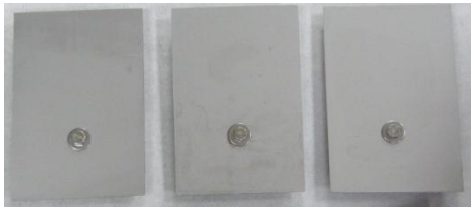
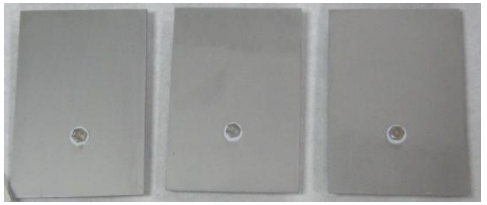
9 2B			
10 2B			

EN 1.4521			
IDENTIFICATION	FLAT	STAINLESS STEEL WASHER	TEFLON WASHER
16 BA			

EN 1.4621			
IDENTIFICATION	FLAT	STAINLESS STEEL WASHER	TEFLON WASHER
11 2M			

EN 1.4016			
IDENTIFICATION	FLAT	STAINLESS STEEL WASHER	TEFLON WASHER
12 2B			
13 BA			

EN 1.4301			
IDENTIFICATION	FLAT	STAINLESS STEEL WASHER	TEFLON WASHER
17 2B			

ANNEX IV

Chemical Cleaner:

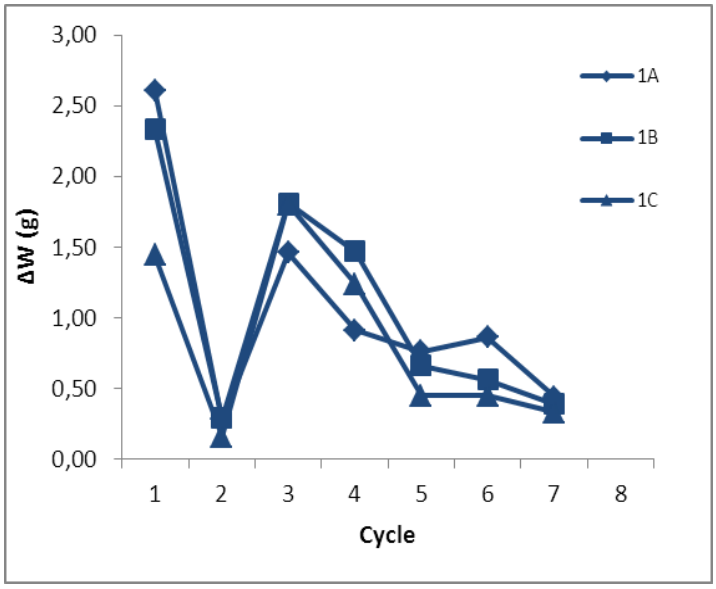
· 18% W/W HCl
· 4 % W/W HNO_3

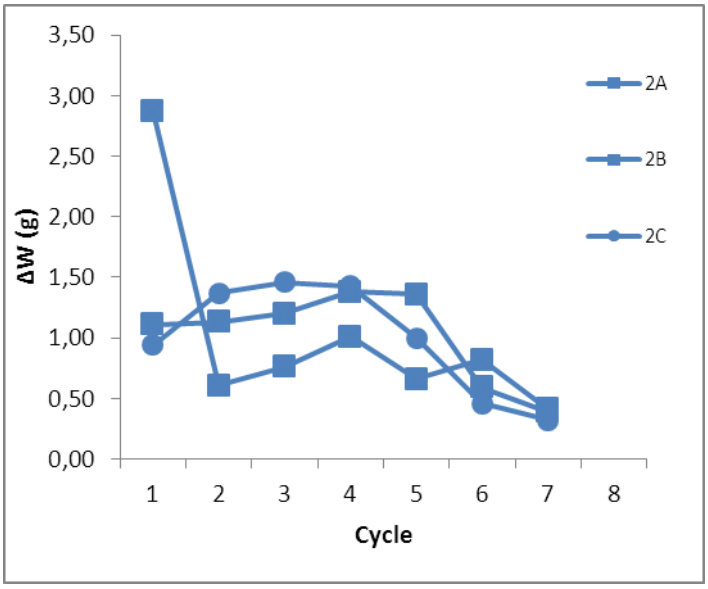
Data used to obtain mass loss and corrosion rate.

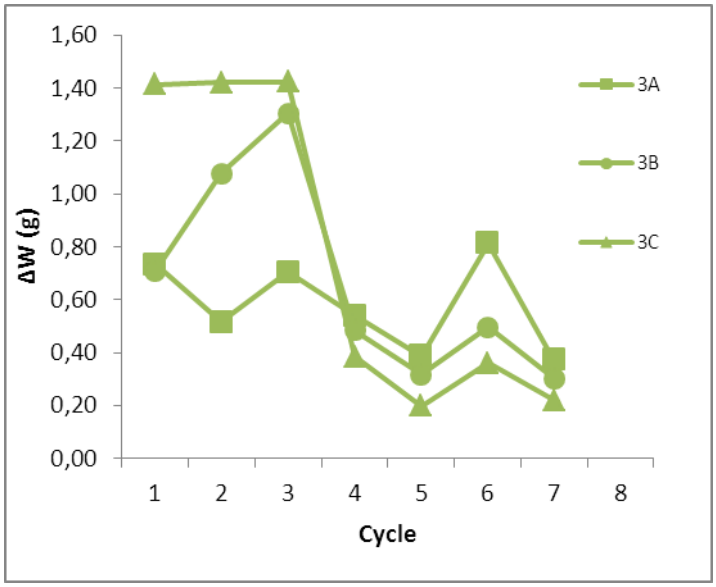
	P0 (g) Before cleaning	P1 (g) 10 min	P2 (g) 20 min	P3 (g) 30 min	P4 (g) 40 min	P5 (g) 50 min	P6 (g) 60 min	P7 (g) 70 min
1A	455,42	452,81	452,53	451,07	450,16	449,4	448,54	448,10
1B	455,83	453,5	453,21	451,4	449,93	449,27	448,71	448,32
1C	454,7	453,26	453,1	451,3	450,06	449,61	449,16	448,83
2A	676,2	673,33	672,72	671,96	670,95	670,29	669,47	669,06
2B	701,14	700,03	698,9	697,7	696,32	694,96	694,37	693,98
2C	677,37	676,43	675,06	673,6	672,17	671,17	670,71	670,39
3A	91,2777	90,5451	90,0302	89,3270	88,7884	88,4020	87,5895	87,2162
3B	90,7285	90,0208	88,9436	87,6385	87,1555	86,8396	86,3433	86,0410
3C	90,6791	89,2661	87,8464	86,4231	86,0383	85,8402	85,4809	85,2617
4A	115,2697	114,9907	114,7292	114,1012	113,5192	113,0924	112,3302	111,9879
4B	114,0861	113,5995	112,9417	111,3650	110,7712	110,4295	109,917	109,6145
4C	115,1375	114,5373	113,4690	110,6949	110,2535	110,025	109,63	109,4149

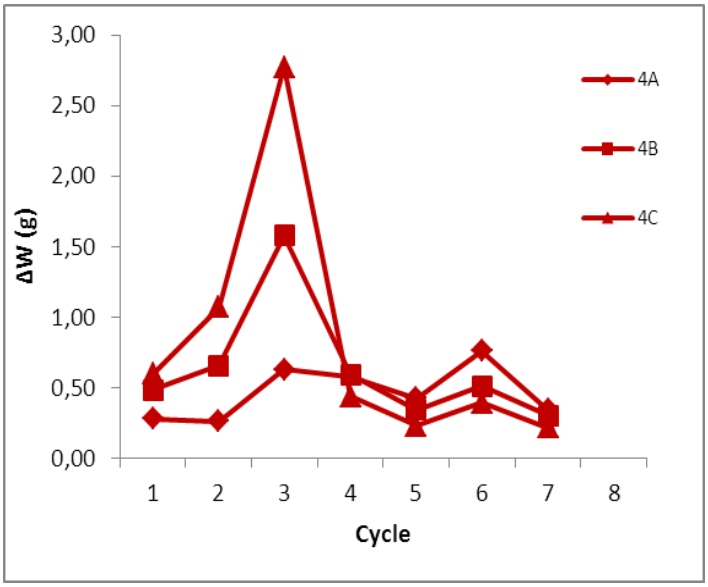
	P0-P1 (g)	P0-P2 (g)	P0-P3 (g)	P0-P4 (g)	P0-P5 (g)	P0-P6 (g)	P0-P7 (g)
1A	2,61	2,89	4,35	5,26	6,02	6,88	7,32
1B	2,33	2,62	4,43	5,9	6,56	7,12	7,51
1C	1,44	1,6	3,4	4,64	5,09	5,54	5,87
2A	2,87	3,48	4,24	5,25	5,91	6,73	7,14
2B	1,11	2,24	3,44	4,82	6,18	6,77	7,16
2C	0,94	2,31	3,77	5,2	6,2	6,66	6,98
3A	0,7326	1,2475	1,9507	2,4893	2,8757	3,6882	4,0615
3B	0,7077	1,7849	3,09	3,573	3,8889	4,3852	4,6875
3C	1,413	2,8327	4,256	4,6408	4,8389	5,1982	5,4174
4A	0,279	0,5405	1,1685	1,7505	2,1773	2,9395	3,2818
4B	0,4866	1,1444	2,7211	3,3149	3,6566	4,1691	4,4716
4C	0,6002	1,6685	4,4426	4,884	5,1125	5,5075	5,7226

	P0-P1 (g)	P1-P2 (g)	P2-P3 (g)	P3-P4 (g)	P4-P5 (g)	P5-P6 (g)	P6-P7 (g)
1A	2,61	0,28	1,46	0,91	0,76	0,86	0,44
1B	2,33	0,29	1,81	1,47	0,66	0,56	0,39
1C	1,44	0,16	1,8	1,24	0,45	0,45	0,33
2A	2,87	0,61	0,76	1,01	0,66	0,82	0,41
2B	1,11	1,13	1,2	1,38	1,36	0,59	0,39
2C	0,94	1,37	1,46	1,43	1	0,46	0,32
14A	0,7326	0,5149	0,7032	0,5386	0,3864	0,8125	0,3733
14B	0,7077	1,0772	1,3051	0,483	0,3159	0,4963	0,3023
14C	1,413	1,4197	1,4233	0,3848	0,1981	0,3593	0,2192
16A	0,279	0,2615	0,628	0,582	0,4268	0,7622	0,3423
16B	0,4866	0,6578	1,5767	0,5938	0,3417	0,5125	0,3025
16C	0,6002	1,0683	2,7741	0,4414	0,2285	0,395	0,2151









Corrosion rate

$$R_{corr(mmpy)} = \frac{K \times W}{A \times T \times D}$$

· K = 8.76×10^4 (constant millimetres per year, mmpy)
· T = exposure time (hours), 672 h.
· A = area (cm^2)
· W = mass loss (g)
· D = density (g/cm^3), 7.7 g/cm^3

	Before test (g)	After cleaning (g)	Weight loss (g)	Area (cm^2)	Thickness (cm)	R_{corr} (mmpy)
1-A	455,67	450,16	5,51	320	0,4	0,2915
1-B	456,79	449,93	6,86	320	0,4	0,3629
1-C	456,22	450,06	6,16	320	0,4	0,3259
2-A	674,13	672,72	1,41	330	0,6	0,0723
2-B	700,86	698,9	1,96	330	0,6	0,1006
2-C	677,34	675,06	2,28	330	0,6	0,1170
3-A	91,1514	88,7884	2,363	304	0,08	0,1316
3-B	90,7763	87,1555	3,6208	304	0,08	0,2016
3-C	90,5092	86,0383	4,4709	304	0,08	0,2490
4-A	115,2924	113,5192	1,7732	305	0,1	0,0984
4-B	114,0643	110,7712	3,2931	305	0,1	0,1828
4-C	115,1308	110,2535	4,8773	305	0,1	0,2707