

SALT SPRAY AND IMMERSION CORROSION TESTING OF PM STAINLESS STEEL MATERIALS

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ABSTRACT

Corrosion testing of four PM stainless steel materials (SS-316N1, SS-316N2, SS-316H, and SS-434H) has been conducted under the auspices of the MPIF Standards Committee.

Test specimens were subjected to salt spray testing in accordance with ASTM B117. Additional test specimens were tested for corrosion in 5% NaCl solution in accordance with ASTM B895.

A summary of the results obtained will be presented. The intent of the work is to provide data for the Engineering Information section of MPIF Standard 35- Materials Standards for PM Structural parts.

INTRODUCTION

MPIF Standard 35, Materials Standards for PM Structural Parts contains a section titled "Engineering Information" that contains data on such items as Jominy hardenability, axial fatigue, rolling contact fatigue (RCF), machinability, coefficient of thermal expansion (CTE), fracture toughness, corrosion resistance, steam oxidation of ferrous PM materials, and guidelines for specifying a PM part.¹

The current corrosion resistance data relates to the corrosion resistance of various PM stainless steel materials immersed in a 2% sulfuric acid solution at room temperature in accordance with ASTM G31.²

The MPIF Standards Committee has decided to expand the data available relative to the corrosion resistance of PM stainless steel materials to include information related to corrosion resistance to immersion in a 5% NaCl solution and to salt spray testing. This paper summarizes the results of a

preliminary exercise to compare the performance of some PM stainless steels materials tested in accordance with ASTM B895 (immersion in 5% NaCl) and ASTM B117 (salt spray testing).³⁻⁴ This initial work will be used to define the procedure for evaluating additional PM stainless steel materials.

EXPERIMENTAL PROCEDURE

The four PM stainless steel materials selected for this preliminary exercise were:

1. SS-316N1
2. SS-316N2
3. SS-316H
4. SS-434 (processed in the manner of an “H” grade material)

The N1, N2, and H designations relate to the chemical composition limits specified in MPIF Standard 35, Material Standards for PM Structural parts.¹ The chemical and physical characteristics of the two base powders used are summarized in Tables 1, 2, and 3.

Table 1: Chemical Composition (mass %)

Material	Cr	Ni	Mo	Mn	Si	S	C	P	Fe
SS-316L	16.6	13.4	2.2	0.1	0.8	0.01	0.03	0.01	Bal.
SS-434L	17.1	-	0.02	0.1	0.9	0.01	0.02	-	Bal.

Table 2: Sieve Analysis of the Base Powders (mass %)

Material	+ 150 μm	+ 106 μm	+ 75 μm	+ 45 μm	- 45 μm
SS-316L	2.9	8.4	16.7	30.7	41.3
SS-434L	2.9	11.1	15.8	24.2	46.0

Table 3: Physical Properties of the Base Powders

Material	Apparent Density (g/cm^3)	Flow (s/50 g)	Green Density at 550 MPa (g/cm^3)
SS-316L	2.69	37	6.57
SS-434L	2.87	33	6.34

Transverse rupture strength bars were compacted at 550 MPa (40 tsi) for each material in accordance with MPIF Standard 41 following the procedures outlined in MPIF Standard 60.⁵⁻⁶ The green densities were:

SS-316L = 6.53 g/cm³

SS-434L = 6.36 g/cm³

The SS-316H and the SS-434H grade materials were sintered at 1150 °C (2100 °F) for about 18 minutes in a 100% hydrogen atmosphere at Atlas Pressed Metals.

The SS-316N1 grade materials were sintered at 1190 °C (2170 °F) for about 30 minutes in a dissociated ammonia atmosphere at SMC Powder Metallurgy, Galeton.

The SS-316N2 grade materials were sintered at 1270°C (2325 °F) for about 30 minutes in a dissociated ammonia atmosphere at SMC Powder Metallurgy, Galeton.

The green and sintered densities of the materials are summarized in Table 4.

Table 4: Green and Sintered Densities of the Various Materials

Material	Green Density (g/cm ³)	Sintered Density (g/cm ³)
SS-316H	6.53	6.62
SS-434H	6.36	6.43
SS-316N1	6.53	6.57
SS-316N2	6.53	6.69

The carbon, oxygen, and nitrogen contents of the test bars after sintering are summarized in Table 5.

Table 5: Carbon, Oxygen, and Nitrogen Contents After Sintering (mass %)

Material	Carbon	Oxygen	Nitrogen
SS-316H	0.04	0.13	0.07
SS-434H	0.02	0.24	0.01
SS-316N1	0.05	0.17	0.29
SS-316N2	0.03	0.08	0.33

IMMERSION TESTING IN 5% Aqueous NaCl

ASTM B895 comprises two methods for evaluating the ability of sintered PM stainless steel parts/specimens to resist corrosion when immersed in an aqueous solution of sodium chloride (5% NaCl).

In Method 1, the test pieces are examined periodically and the time to the first appearance of rust is used to indicate the end point.

In Method 2, continued exposure to the sodium chloride solution is used to monitor the extent of corrosion as a function of time.

One test bar was placed per glass jar on top of some glass beads and the 5% NaCl solution was added to each jar such that the volume in mL was at least five times the mass of the specimen in grams. The distance from the surface of the TRS bars to the top of the solution was at least 25 mm. The ratio of the volume of the air to the volume of the solution in the jar was about 1:2 to 1:3. An example of the experimental set-up is shown in Figure 1. Any air bubbles were removed by gently swirling the solution.



Figure 1: Example of the experimental set-up for immersion testing in accordance with ASTM B895).

The jars were closed and the date and time of the test was recorded. The immersed test bars were maintained at a temperature between 21 to 24 °C (70 to 75 °F). The test bars were examined after ½, 1, 2, 4, 8 h, and 24 h intervals thereafter. After that the time interval was lengthened up to the first appearance of rust or a stain. The corrosion life per Method 1 is the time of the last examination before the observation of a stain or rust. Five TRS bars were tested for each material/process condition.

Method 2 follows the procedure of Method 1 then continues with periodic examination beyond the first appearance of a stain or rust. In Method 2, specimens are ranked for degree of staining or corrosion according to the following:

- A – The part or specimen is free from stain or rust
- B – The first sign of a stain or rust appears or up to 1% of the surface is covered by stain or rust
- C – More than 1% and up to 25% of the surface is covered by stain or rust
- D – More than 25% of the surface is covered by stain or rust

An example of the ratings for various amounts of stain or rust, taken from ASTM B895, is shown in Figure 2.

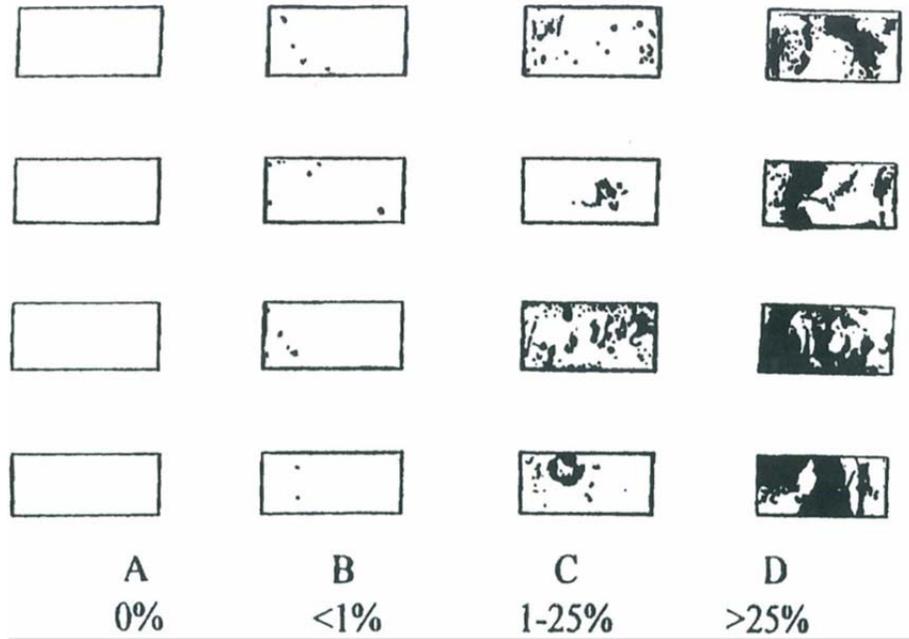


Figure 2: Examples of ratings for various amounts of rust or stain – from ASTM B895.

An example of a corrosion rating chart, taken from ASTM B895 is shown in Figure 3. Based on this chart, a plot is made of the percentage of replicate specimens with a given rating versus the immersion time – Figure 4.

TABLE 1 Example of Corrosion Rating Chart for a Set of Ten Replicate Specimens of Sintered 316L Stainless Steel^A

Specimen #	Hours submersed in 5% aq. NaCl																				
	0.5	1	2	4	8	24	31	50	74	104	168	240	336	496	696	984	1368	1804	2282		
1	A	A	A	A	A	A	A	A	B	B	B	B	C	C	C	C	D	D	D		
2	A	A	A	A	A	A	A	A	A	A	B	B	B	B	C	C	C	D	D		
3	A	A	A	A	A	A	A	B	B	B	B	C	C	C	D	D	D	D	D		
4	A	A	A	A	A	A	A	A	A	B	B	B	B	C	C	C	C	C	D		
5	A	A	A	A	A	A	A	A	A	B	B	B	B	C	C	C	C	C	C		
6	A	A	A	A	A	A	A	A	A	B	B	B	B	C	C	C	C	C	D		
7	A	A	A	A	A	A	B	B	B	B	B	B	C	C	C	C	D	D	D		
8	A	A	A	A	A	A	A	B	B	B	B	B	C	C	C	D	D	D	D		
9	A	A	A	A	A	A	A	A	B	B	B	B	B	C	C	C	C	D	D		
10	A	A	A	A	A	A	A	A	A	B	B	B	B	C	C	C	C	C	D		
		% Holding 'A' Rating																			
		100	100	100	100	100	100	90	70	50	10	0									
												% Holding 'B' Rating									
												100	90	60	10	0					
												% Holding 'C' Rating									
												100 90 80 70 40 10									

^ASee Section 10.1.1 for definition of ratings.

Figure 3: Example of corrosion rating chart – from ASTM B895.

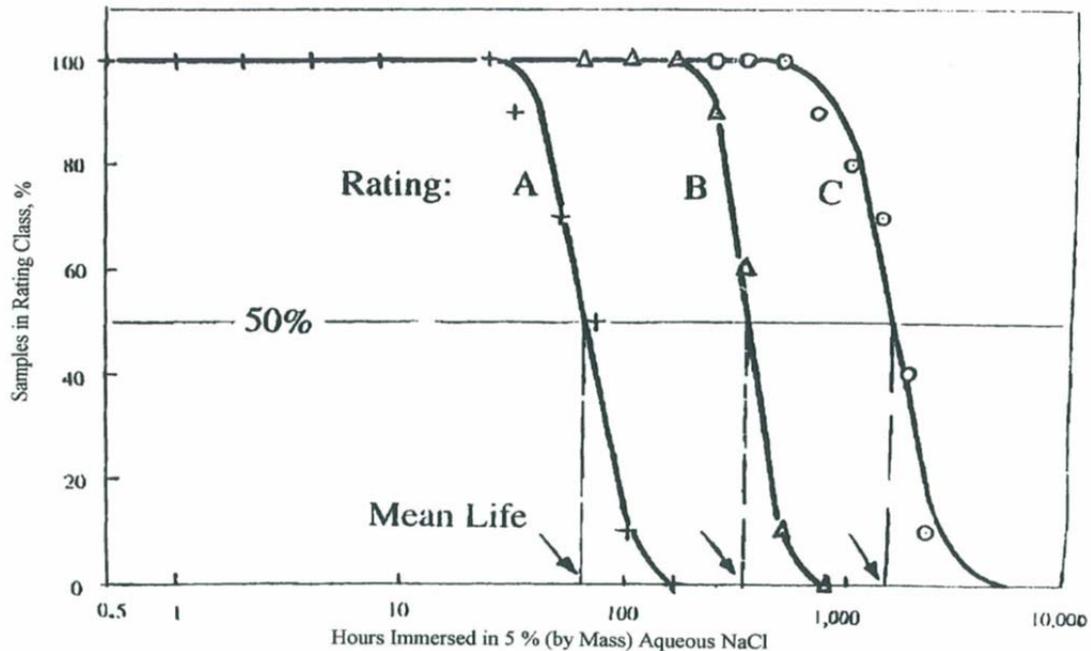


Figure 4: Plot of percentage of replicate specimens with a given rating versus immersion time. (from ASTM B895)

SALT SPRAY TESTING

Salt spray testing was conducted in accordance with ASTM B117 at Assured Testing Services, Ridgway, PA. This practice provides a controlled corrosive environment that has been utilized to produce relative corrosion resistance information for specimens of metals exposed in a test chamber. Prediction of performance in natural environments has seldom been correlated with salt spray results when used as stand-alone data.

The apparatus for salt spray (fog) exposure consists of a fog chamber, a salt solution reservoir, a supply of suitably conditioned compressed air, one or more atomizing nozzles, specimen supports, provision for heating the chamber, and necessary means of control – see Figure 5. Drops of the solution that accumulate on the ceiling or cover of the chamber shall not be permitted to fall on the specimens being exposed. Drops of solution that fall from the specimens shall not be returned to the solution reservoir for re-spraying.

Ten specimens (TRS bars) were tested per material/process condition. The specimens, placed in the specimen supports for testing, are shown in Figure 6.

RESULTS

IMMERSION TESTING

The results for Method 1 of the immersion testing in 5% NaCl are summarized in Table 6 and the average time for the first sign of rust in Table 7.

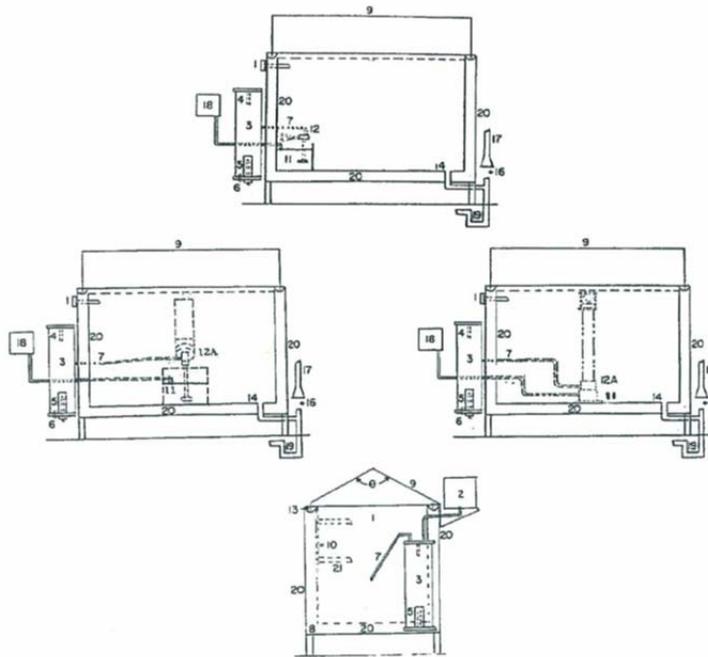


Figure 5: Schematic of salt spray chamber – from ASTM B117.



Figure 6: TRS bars in their racks prior to salt spray testing.

Table 6: Immersion Test Results for Method 1 of ASTM B895
Hours to First Sign of Rust

Material	Bar 1	Bar 2	Bar 3	Bar 4	Bar 5
SS-316H	>1512	>1512	>1512	>1512	>1512
SS434H	0	0	0	1	0
SS-316N1	0	0	½	0	6
SS-316N2	½	120	½	129	6

Table 7: Average Time for the First Sign of Rust (hours)

Material	Average Time for First Sign of Rust (h)
SS-316H	>1512
SS-434H	<0.5
SS-316N1	1.3
SS-316N2	51.2**

** Considerable scatter in the data

Examples of the condition of one of the SS-434H test bars after half an hour and after 4 hours are shown in Figure 7.



Figure 7 (a): SS-434H test bar after ½ h
Class B = 0-1% rust



Figure 7 (b): SS-434H test bar after 4 h
Class D = >25% rust

Examples of some of the other test bars are shown in Figure 8.



Figure 8 (a): SS-316N1 test bar after ½ h
Class B = 0-1% rust



Figure 8 (b): SS-316N2 test bars after 96 h
On left Class D = >25% rust
On right Class C= 1-25% rust

The SS-434H test bars exhibited signs of rust fairly quickly. The results for Method 2 with the SS-434H test bars are summarized in Table 8 and the results for the SS-316N1 test bars are summarized in Table 9.

Table 8: Rating Results for Method 2 – SS-434H

	½ h	1 h	2 h	4 h	6 h
Bar 1	B	B	C	D	D
Bar 2	B	B	C	D	D
Bar 3	B	B	C	D	D
Bar 4	A	A	B	B	D
Bar 5	B	B	C	D	D

As the SS-434H test bars showed signs of rust fairly quickly there is no point in plotting the results in the manner proposed in ASTM B895. The data for the SS-316N1 test bars (Table 9) are, however, plotted in Figure 9.

The results for Method 2 testing of the SS-316N2 test bars are summarized in Table 10 and the data plotted in Figure 10.

Table 9: Rating Results for Method 2 – SS-316N1

	½ h	1 h	2 h	4 h	6 h	27 h	96 h	120 h	
Bar 1	B	B	B	B	B	C	D	D	
Bar 2	B	B	B	B	B	D	D	D	
Bar 3	A	B	B	B	B	C	D	D	
Bar 4	B	B	B	B	B	C	D	D	
Bar 5	A	A	A	A	A	B	C	C	
	% Holding "A" Rating								
	40	20	20	20	20	0			
	% Holding "B" Rating								
	60	80	80	80	80	20			
							% Holding "C" Rating		
							60	20	20
							% Holding "D" rating		
							20	80	80

NaCl Immersion Test for SS-316N1

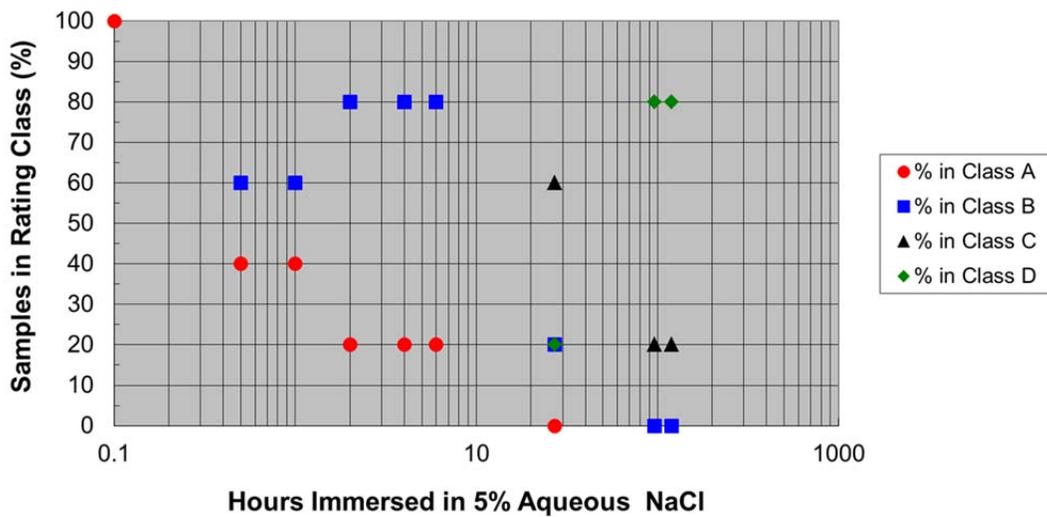


Figure 9: Plot of percentage of replicate test specimens with a given rating versus immersion time.

Table 10: Rating Results for Method 2 – SS-316N2

	½ h	1 h	2 h	4 h	6 h	27 h	96 h	120 h	129 h	264 h	288 h	312 h
Bar 1	A	B	B	B	B	C	D	D	D	D	D	D
Bar 2	A	A	A	A	A	A	A	A	C	C	C	C
Bar 3	A	B	B	B	B	B	C	C	C	C	C	D
Bar 4	A	A	A	A	A	A	A	A	A	B	B	B
Bar 5	A	A	A	A	A	C	C	C	C	C	D	D
	% Holding "A" Rating											
	100	60	60	60	60	40	40	40	20	0		
	% Holding "B" Rating											
		40	40	40	40	20	0			20	20	20
	% Holding "C" Rating											
						40	40	40	60	60	40	20
	% Holding "D" Rating											
							20	20	20	20	40	60

NaCl Immersion Test for SS-316N2

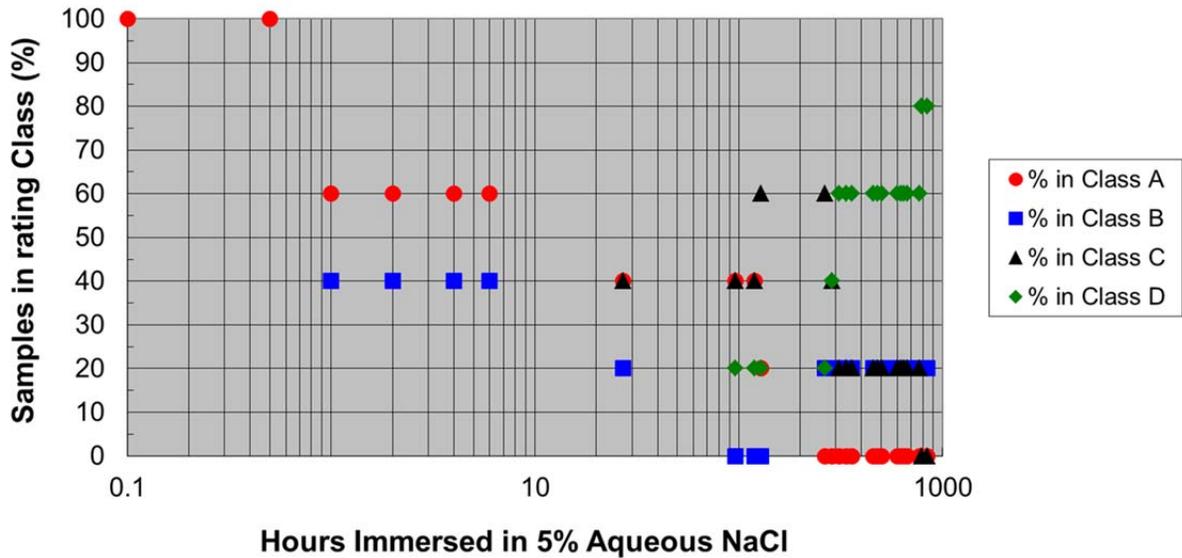


Figure 10: Plot of percentage of replicate test specimens with a given rating versus immersion time.

SALT SPRAY TESTING

Photographs of the test bars after various times are shown in Figure 11. The rows of test bars from top to bottom are – SS-316N1, SS-316N2, SS-316H, and SS-434H.



Figure 11: Photographs of the various test bars at different times of salt spray exposure.



Figure 12: Photographs of the various test bars after 168 h and 200 h of salt spray exposure.

DISCUSSION

It is generally accepted that extreme care needs to be taken in order to obtain good corrosion resistance with PM stainless steel materials – higher temperature sintering in a 100% hydrogen atmosphere in a furnace dedicated to sintering stainless steels. “H” and “L” grade processing should result in the best corrosion resistance with the “L” grade (highest sintering temperature) being better than the “H” grade. A more detailed discussion of the corrosion of PM stainless steels is provided by Klar and Samal in their book, “Powder Metallurgy Stainless Steels – Processing, Microstructures, and Properties”.⁷

The SS-316H material did well in immersion testing (>1512 h) and reasonably well in the salt-spray testing. This is despite the fact that the carbon and nitrogen contents of this material were above the limit specified in MPIF Standard 35.

While the SS-316N2 material did not do as well in the immersion testing (considerable scatter in the results) it performed reasonably well in the salt-spray testing. This may be due to the fact that the sintering temperature was 1270 °C (2325 °F).

As expected, the SS-434 material was inferior to the 300-series austenitic stainless steel grades.

The immersion test data exhibit considerably more scatter than the example provided in ASTM B895; the graphical method suggested may only be suitable when the transition from Class A to Class B occurs at relatively extended times (in our case, for SS-316N1 and SS-316N2 we had classes A, B, and C co-existing after a relatively short time). This also perhaps indicates the need for at least ten specimens – giving a lower percentage impact if one test bar changes class.

CONCLUSIONS

It is clear that there is generally considerable scatter in the results of corrosion testing – particularly when the test is being conducted on PM materials. While ASTM B895 indicates that a minimum of five specimens shall be used for each test, it appears that a minimum of at least ten specimens would be

preferable. Real life data are not always as well defined as the example given in the standard. Method 2 of ASTM B895 is really only suitable for those materials that have an extended life prior to the appearance of corrosion products.

Based on the results of this initial investigation it would appear that SS-316L should perform well in both the immersion and the salt-spray testing.

The next phase of testing on behalf of the MPIF Standards Committee will include SS-303H and L grades, SS-304H and L grades, and SS-316L. Test bars from each group will be checked for iron or low-alloy steel powder contamination using the ferroxyl test prior to immersion or salt spray testing.⁷

REFERENCES

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