Study of 6N HCl Corrosion on Commercial 316 SS, Hastelloy C-22 and TrueTube™ Variants

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Testing Conducted by:

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Test Report

Testing was conducted to determine the relative corrosion resistance of Hastelloy C-22, commercial 316L SS, commercial tube with fused silica coating, electropolished TrueTube EP, and electropolished TrueTube EPS with a deactivated layer of bonded amorphous silicon applied via CVD technology (Siltek™) provided by Restek Corporation.

Corrosion Test Protocol and Results

The test specimens were ½" OD tubing with 0.049" wall thickness. They were cut, faced and deburred to approximately 8" in length. Each sample was rinsed in DI water and blown dry using 0.003 µm filtered N_2 gas. The samples where weighed and one end was capped using LDPE Tube Caps. The samples where then placed in a tube holder and filled with 6N HCl. The other end of the tube was then capped using a tube cap that had a pinhole in it to let the sample breath. The samples where exposed to the HCl for 72 hours. During the exposure the samples where agitated using vibration every 8 hours. After the 72-hour period the samples where drained rinsed and dried. The final weights where then recorded. The tubing that was tested is listed as follows:

Hastelloy C-22
Commercial Grade 316L Tubing
Commercial Grade 316L Tubing with Fused Silica Coating
Electropolished TrueTube EP Tubing
Electropolished TrueTube EP Tubing with a Siltek Coating

Table 1 Corrosion Rates for Tubing Tested

Material	Weight Loss (g)	Corrosion Rate (g/hr cm^2)	MPY (mils per year)	Sample Variance
Hastelloy C-22	0.0075	1.69E-06	0.6733	0.001622
Commercial Grade 316L Welded Tubing	0.3085	6.93E-05	29.9400	0.640267
Commercial Grade 316L Tubing with Fused Silica Coating	0.0492	1.1E-05	4.7567	13.19002
Electropolished TrueTube EP Tubing	0.1669	3.6E-05	16.5733	0.795022
TrueTube EP with Siltek Coating (TrueTube EPS)	0.0031	6.65E-07	0.2867	0.000622

Determination of Test Data

The corrosion rate was determined using the ASTM equation established in ASTM G31.

$$CorrosionRate = \frac{(K \cdot W)}{(A \cdot D \cdot T)}$$

Where: K is a constant =3.45 E6
W is the weight loss in grams
A is the exposed surface Area cm2
D is the density g/cm2
T is the time in hours

The variance of each sample population was computed to determine a confidence level for the results. The variance was less than 1 for all samples except fused silica coating on commercial tubing. Variance was calculated by the equation: $\frac{\sum(x-\overline{x})}{n}$ where x is the sample mean and n the sample size.

Micrographic Inspection of Test Samples

In addition to the corrosion testing micrographs of the samples where taken at 500x after the exposure to 6N HCl. All the micrographs taken are of the surface after exposure to 6N HCl.

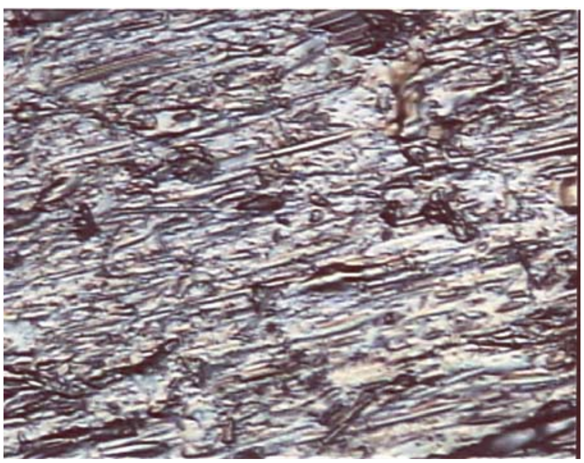

Figure 1: Hastelloy C-22 500x

Figure 1 is the micrograph of Hastelloy C-22. It clearly shows that the 6M HCl has barely touched the surface.

Figure 2 shows the surface of the commercial 316L SS sample. There are several corrosion mechanisms occurring here. The darken lines indicate grain boundary attack. There is also general corrosion due to the irregular surface and some pitting. Comparing the micrographs of the commercial 316L SS tubing and the TrueTube EP tubing illustrates the importance of minimizing nucleation sites in which pitting and general corrosion can occur. In the case of the commercial tubing the nucleation sites are the peaks and valley's on the surface and process contamination. The electropolished TrueTube EP process minimizes these nucleation sites.

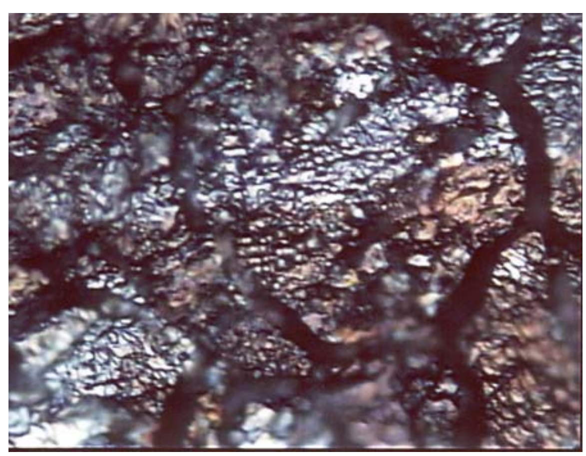

Figure 2: Commercial 316L SS Tubing 500x

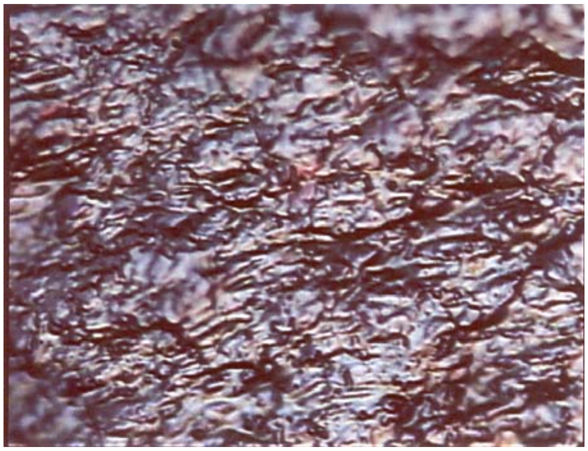

Figure 3: Commercial Grade SS with Fused Silica Coating 500x

Figure 3 is the surface of commercial grade 316L tubing with fused silica coating. Since the coating is transparent the image shows the metallic surface and not the coating surface. Because of the irregular surface roughness of the tubing it is difficult to distinguish any area of preferential attack. Several locations where inspected with no evidence of total coating failure.

Figure 4 shows the surface of the Electropolished TrueTube EP Tubing. Primary corrosion is evident at the grain boundaries with some pitting internal to the grains.

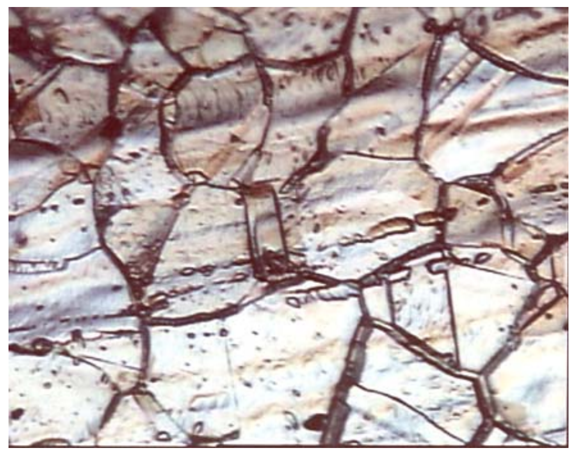

Figure 4: Electropolished TrueTube EP Tubing 500x

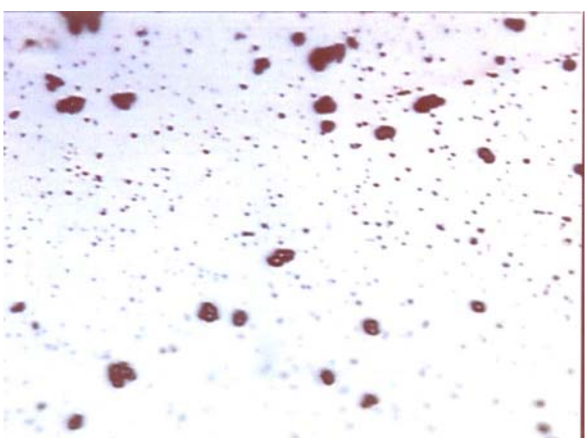

Figure 5: TrueTube EPS – Electropolished Tubing with Siltek Coating 500x

Figure 5 is of the TrueTube EP with Siltek Coating (TrueTube EPS). The micro pitting of the metallic surface is seen. This is the result of the electropolishing and coating process not due to corrosion. The base metal surface appears untouched by the HCl. Comparing the samples commercial grade fused silica (Figure 3) explains the variance in corrosion rate. The discontinuities in the coating for the fused silica coated commercial grade tubing are major sources of weight loss and directly affect the corrosion rate.

Notes on the Resulting Test Data

Commercial 316L SS is used as the base for all relative corrosion resistance rankings.

Hastelloy C-22 is included in the test because it is recognized as the 'material of choice' for HCl service. Test results illustrate a 44 fold improvement in corrosion resistance to 6N HCl over the base commercial 316L SS.

The corrosion rate of the commercial grade fused silica coated tube varied between samples, more than all other tubes tested. The average corrosion rate for commercial grade fused silica was 4.75 mpy or a 6 fold improvement over commercial 316L SS.

Test results clearly illustrate that TrueTube EP improves the corrosion resistance by approximately 2 fold over commercial 316L SS.

Using TrueTube EP as the substrate of the Siltek coating improved the corrosion resistance by 27 fold over commercial grade fused silica coated tubing and 172 fold over commercial 316L SS in the milled condition. The resulting TrueTube EPS showed far less variance then the commercial grade fused silica coated tubing. The average corrosion rate was calculated to be 0.28 mpy. This gives TrueTube EPS a 104 fold advantage over commercial 316L SS material with a very high confidence level.

Conclusion

The corrosion resistance of austenitic stainless steel can be improved by electropolishing or by applying a coating of fused silica or by combining both processes. The combination of O'Brien Corporation electropolished TrueTube EP and the Siltek coating by Restek provided corrosion resistance superior to that of Haselloy C-22.

Surface preparation will affect the ability of any subsequent process to provide additional corrosion protection. This assertion is supported by the variance found in the testing of the fused silica coating on commercial grade tubing. The corrosion rate of these samples varied sixteen times more than all other sample sets. The samples of fused silica applied to commercial tubing showed corrosion resistance changes from a low of 3 fold to a high of 24 fold. While the average corrosion rate for commercial grade fused silica computed to 4.75 mpy or a 6 fold improvement over commercial grade tubing, ranking by the average value belies the uncertainty of predicable performance.

While very different in their values both TrueTube EP and TrueTube EPS provided constant results and enhanced corrosion protection.

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