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Astro Pak Corporation

Comparison of Passivation Modalities

*An Independent Analysis of Results
Produced by Three Passivation Methods*

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Introduction

The Astro Pak Corporation has experience with many different passivation chemistries. Three commonly used passivation chemistries include nitric acid based, citric acid based, and phosphoric acid based.

Astro Pak Corporation obtained 12 small coupons (about 0.5" square, 0.125" thick) of 304L stainless steel from Lawrence Livermore National Laboratory. These pieces were cut from the same piece of stock and were supposed to be representative of stainless steel used in the NIF project.

The coupons were passivated by one of three methods (with three pieces unpassivated to serve as referee coupons). The coupons were sent to an independent laboratory, Charles Evans & Associates / Surface Science Laboratories, for analysis.

Treatment Modalities

The coupons were randomly assigned a number (1 through 12), which was engraved on one side of the coupon. The coupons were then cleaned ultrasonically in one batch. The coupons were then split into four groups. The coupons of the first group, numbers 1 through 3, were allowed to air dry and then were held for further analysis. The coupons of the second group, numbers 4 through 6, were allowed to air dry and then were passivated with a 30% (v/v) nitric acid solution per the ASTM A 967-99 method (as described in paragraph 6.1.1.2 of that document). The coupons of the third group, numbers 7 through 9, were allowed to air dry and then were passivated with a 10% citric acid solution (w/v) per the ASTM A 967-99 method (as described in paragraph 7.1.1.2 of that document). The coupons of the fourth group, numbers 10 through 12, were allowed to air dry and then were passivated in a 15% solution (w/v) of phosphoric acid (at room temperature, 90 minute dwell time).

All pieces were then kept in a dessicator cabinet for 72 hours. They were then packaged by group in ultra-low outgassing polyethylene film and sent to Evans & Associates for testing. Astro Pak commissioned Evans & Associates to perform XPS analysis (ESCA) for the materials of interest – specifically Cr/Fe ratios as well as $\text{Cr}^{\text{OX}}/\text{Fe}^{\text{OX}}$ ratios (oxide ratios).

Results

The results of the analysis are presented in spreadsheet format in the attached file **passivation test.xls**.

Discussion

The results of the tests show that chrome to iron ratios, as well as oxide ratios, are improved over baseline by all three passivation modalities. The selective removal of free iron atoms from the outer layer of the stainless steel results in a higher percentage of chromium atoms being exposed to the atmosphere or potential corrosion accelerants. In simple terms, this translates into enhanced corrosion resistance.

The three passivation modalities all improved the corrosion resistance of the coupons, though there were differences in efficacy. In terms of rank, the citric acid solution was the most efficacious, followed by nitric acid, and then phosphoric acid. It should be noted that the citric

acid method used was as described in the ASTM A 967-99 document paragraph 7.1.1.2, which is plain citric acid. The efficacy of the citric acid treatment can be further enhanced with proprietary solutions such as the Astro Pak Corporation's Ultra Pass solution, which contains added chelants and surfactants to accelerate and enhance the activity of the citric acid.

Conclusion

All three passivation modalities provided enhanced corrosion resistance on the 304L stainless steel test coupons. The citric acid passivation proved to be the most efficacious surface treatment tested.

SAMPLE ID	PROCESS - 304 SS	Cr/Fe RATIO	Cr/Fe OXIDE RATIO
1	CLEAN - 304	0.8	0.9
2	CLEAN	1	1.3
3	CLEAN	1	1.2
4	CLEAN, NITRIC - 304	1.5	2
5	CLEAN, NITRIC	1.5	2.1
6	CLEAN, NITRIC	1.5	2.2
7	CLEAN, CITRIC - 304	1.8	2.6
8	CLEAN, CITRIC	1.6	2.2
9	CLEAN, CITRIC	1.5	2.3
10	CLEAN, PHOSPHORIC - 304	1.4	1.9
11	CLEAN, PHOSPHORIC	1.2	1.6
12	CLEAN, PHOSPHORIC	1.2	1.5
13	CLEAN, CITRIC GEL - 316L	2.1	3.2
14	CLEAN, CITRIC GEL	1.9	2.6
15	CLEAN, CITRIC GEL	1.7	2.5

PASSIVATION COMPARISON

■ Cr/Fe RATIO □ Cr/Fe OXIDE RATIO

