Corrosion Study Using Electrochemical Impedance Spectroscopy

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ABSTRACT

Corrosion is a common phenomenon. It is the destructive result of chemical reaction between a metal or metal alloy and its environment. Stainless steel tubing is used at Kennedy Space Center for various supply lines which service the orbiter (2). The launch pads are also made of stainless steel. The environment at the launch site has very high chloride content due to the proximity to the Atlantic Ocean. Also, during a launch, the exhaust products in the solid rocket boosters include concentrated hydrogen chloride. The purpose of this project was to study various alloys by Electrochemical Impedance Spectroscopy in corrosive environments similar to the launch sites. This report includes data and analysis of the measurements for 304L, 254SMO and AL-6XN in primarily neutral 3.55% NaCl. One set of data for 304L in neutral 3.55%NaCl + 0.1N HCl is also included.
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1. INTRODUCTION

Stainless steel tubing is used at Kennedy Space Center for various supply lines which service the orbiter (2). The environment at the launch site has very high chloride content due to the proximity to the Atlantic Ocean. Also, during a launch, the exhaust products in the solid rocket boosters include concentrated hydrogen chloride. The acidic environment is aggressive to most metals and cause pitting in some of the common stainless steel alloys. Pitting corrosion can cause cracking and rupture of both high-pressure gas and fluid systems, which can result in the safety of shuttle launches.

The purpose of this project was to study various tubing alloys by Electrochemical Impedance Spectroscopy in corrosive environments similar to the launch sites. This report includes data and analysis of the measurements for 304L, 254SMO and AL-6XN in primarily neutral 3.55% NaCl. One set of data for 304L in neutral 3.55%NaCl + 0.1N HCl is also included. Three more alloys in similar environments are to going to be studied. 3.55% NaCl +1N HCl is also going to be studied.
2. NEXT SECTION

Electrochemical Impedance Spectroscopy

EIS. A low voltage 5 – 10mV (to keep it non-destructive) of varying frequency (100K to 10mHZ) is applied between the working electrode and the counter electrode, of course in the presence of the electrolyte. The data provided by the measuring equipment is in two forms. Impedance modulus |Z| and phase angle phi-vs-frequency, Bode plots. Real part of Z vs imaginary part of Z, Nyquist plots.

Bode plots points to a single time constant, hence the data is analyzed using a simple model, $R_s + R_p || C_p$, where $R_s$ is the resistance of the solution (electrolyte), $R_{EDL}$ resistance of the electrical double layer and $C_{EDL}$ capacitance of the electrical double layer. Software package PowerSuite was used to analyze the data. It regresses the appropriate equation around experimental data, by iteratively adjusting parameters, such as $R_p$ and $C_p$, until the closest fit of the equation to experimental data is obtained.

Three alloys 304L, 254SMO and AL-6XN are currently being studied. Eletrolytes used/to be used are, 3.55% neutral NaCl solution, 3.55% NaCl + 0.1N HCl and 3.55% NaCl + 1N HCl.

3. RESULTS AND DISCUSSION

1. Alloys in 3.55% neutral NaCl.
   As is expected, corrosion is a random process and reproducibility is not very good. Figure 3 shows a plot of $R_p$ - VS - Time for the second run of 304L. For this particular reason, a particular alloy is being studied three times under similar conditions. The resistance increases with time pointing to an increase in the thickness of the corrosive film.

![Figure 3. Change of resistance of the corrosive film with time](image)

Figure 4 shows its capacitive behavior. As expected the capacitance goes down with time but not in a well behaved manner.

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Figure 4. Change of capacitance of the corrosive film with time

Figure 5 shows change of open circuit potential (OCP) with time. OCP becomes more and more positive becoming less and less prone to corrosion.

Figure 5. Change of open circuit voltage of the corrosive film with time

Figure 6 shows Rp –VS- Time for another run for 304L and shows that initially its resistance hence the thickness of the corrosive layer goes up but then the thickness decreases due to etching of the layer and it increases again etc.

Figure 6. Change of resistance of the corrosive film with time

Alloys in 3.55%NaCl in 0.1N HCl

So far only 304L has been studied and it shows an interesting behavior. Figure 7 shows how the resistance with time changes. It appears that in the first 24 hours the passive film grows but then it gets etched away with time.
4. CONCLUSIONS

1. With time the thickness of the passive film grows initially but this trend does not continue for longer times. Even different coupons of the same alloy do not show consistent behavior.

2. Alloys show a different behavior in 3.55%NaCl+0.1NHCl.

Recommendations for future work

1. The behavior of alloys in 3.55% NaCl + 0.1N HCl should be looked at more closely between the time it is immersed in the electrolyte and 24 hours. Time interval for EIS data collection is too long.
2. 0.05N HCl should also be studied.
3. If a visible passive film appears then it should not only be photographed but also analyzed for its composition.
4. Find the concentration of the active ion (chloride) in the electrolyte at the end of a run.
5. Analyze the electrolyte to determine which of the element/elements from the alloy are being attacked. This should help to determine which alloy tubing to use in the launch pads.
REFERENCES