

# Electrochemical Phenomenon on Stainless Steel in High and Low Conductivity Solutions

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## Abstract

Studies were done on the corrosion rate of M50 steel samples in various solutions. The studies were done using the potentiodynamic and electrochemical impedance spectroscopy (EIS) experimental techniques and appropriate data was graphed for each. The information was used to calculate corrosion rate related to the solution.

## Introduction

Currently most aircraft engines are made of stainless steels. Traditionally, corrosion rates of these metals have been tested using the salt spray test. This test involves spraying a sample with salt water and observing it for an extended period of time. The change in weight of the sample is used to calculate corrosion rate. The main problem with this test is that it takes a long time and there are a number of environmental factors that can affect it.

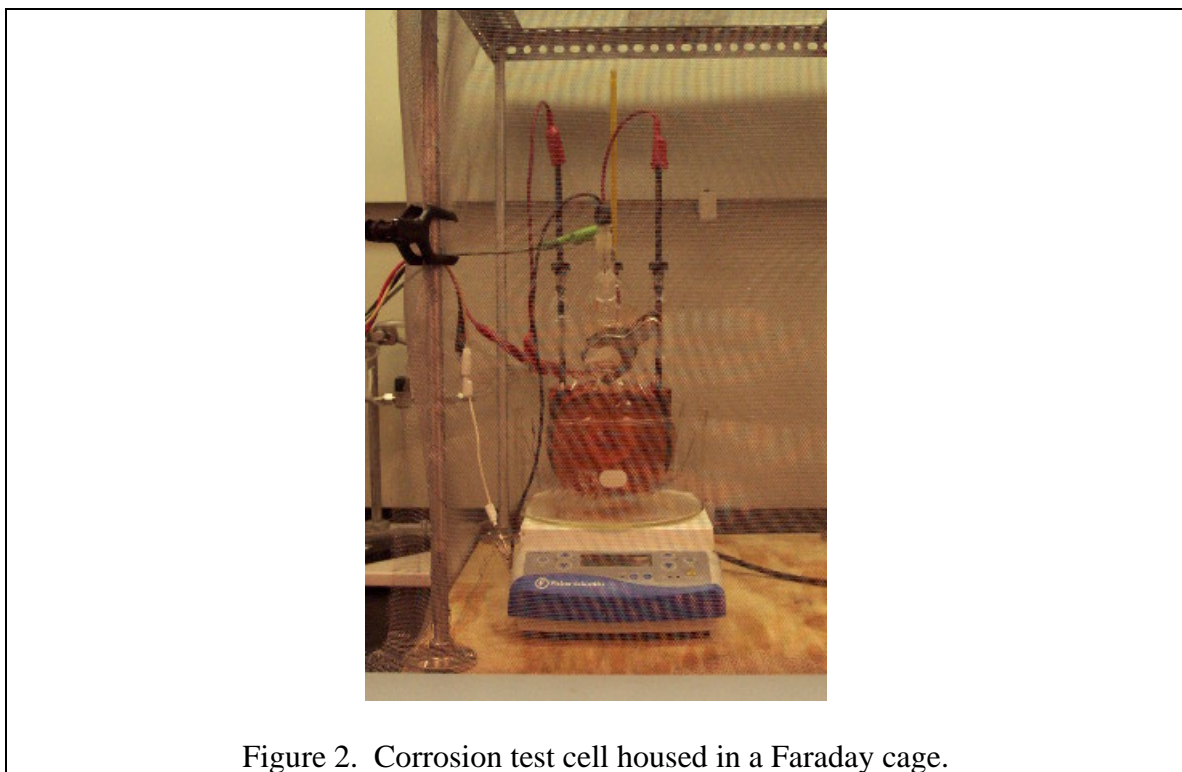
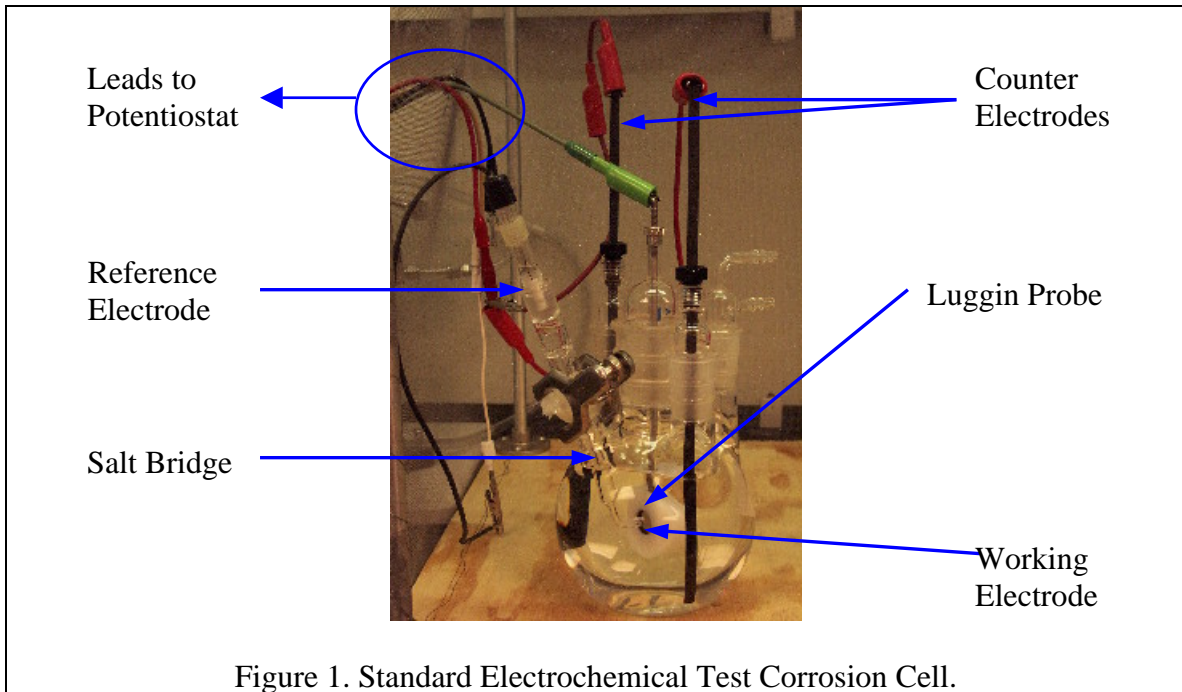
A better way of measuring corrosion is through electrochemical methods of which there are two main types: potentiodynamic and electron impedance spectroscopy (EIS). Both techniques take advantage of the electrochemical behavior associated with corrosion. However, both techniques have disadvantages in different situations. The objective of this work was to determine the corrosion rate and polarization behavior of the selected metal.

The metal that was studied, M50 stainless steel, is used for a number of different applications. Iron was the main component of M50, accounting for almost 90 percent by weight. Main alloying agents included Chromium and Molybdenum, which both made up about 4 percent of the alloy. The following alloying agents made up less than one percent each. They included Carbon, Manganese, Silicon, Phosphorus, Sulfur, Nickel, Vanadium, Cobalt, Tungsten, and Copper.

## Experimental Procedure

For both types of scans, a standard electrochemical test cell, pictured in Figure 1, was used. Our setup consisted of a saturated calomel reference electrode, two spectroscopically pure graphite counter electrodes, and a working electrode. The working electrode consisted of a Teflon holder in which the samples were mounted. It allowed a 1 cm<sup>2</sup> area of the sample to be exposed. The reference electrode was connected to the salt bridge which was connected to the Luggin probe. This was positioned approximately 1 mm from the sample. The cell was placed in a fume hood. The experiments were carried out using a Princeton Instruments Potentiostat/Galvanostat Model 263A. All components of the cell were immersed in a solution during each run. This solution

included NaCl solutions of varying concentrations, new oil, and used oil. Before each run, the samples were polished to a 600 grit finish, cleaned in acetone, methanol, and deionized water.



For the impedance runs, a Faraday cage (Figure 2) was used to reduce surrounding electromagnetic fields, which would otherwise disrupt readings. This was due to the high resistance of the oil, which caused the current going through the sample to be very low.

## Results

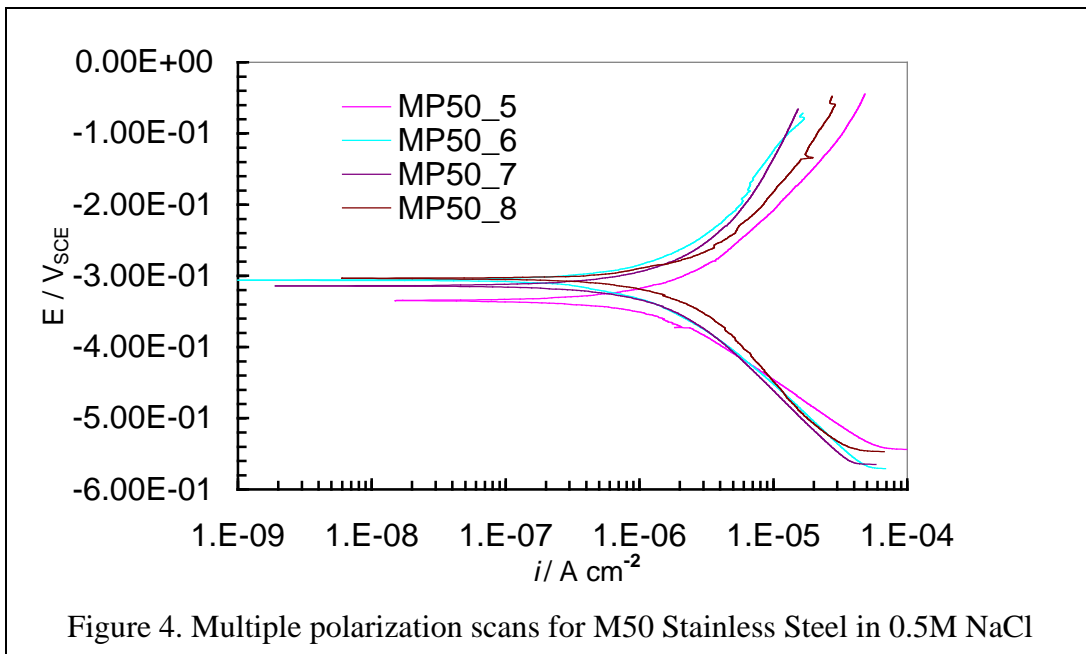
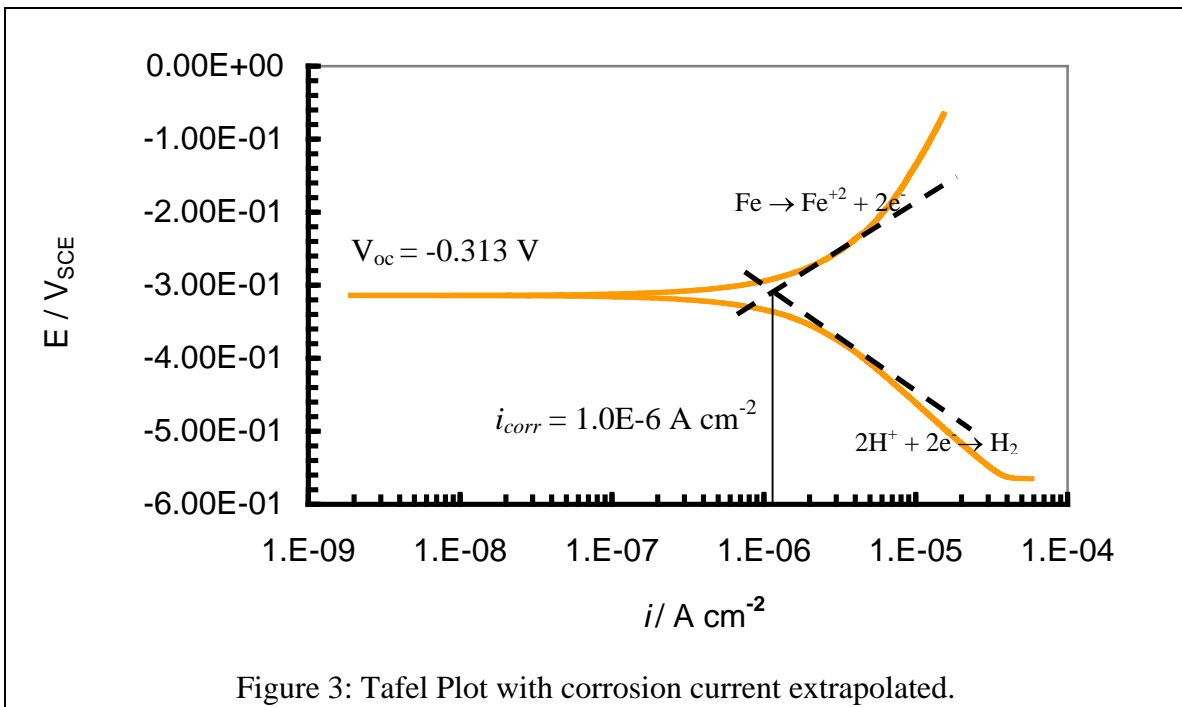
### ***Potentiodynamic Results***

The first tests we ran were potentiodynamic scans. By running these scans, we were attempting to find the corrosion potential and the pitting potential so that we could know how resistive M50 is to corrosion. Potentiodynamic scans involve applying a potential between the counter and working electrodes, using the reference electrode for measurement. For these scans NaCl solutions of varying concentrations were used. The salt solutions used had a high conductivity, which translated into little or no ohmic resistance between the reference electrode and the working electrode. This is important, as the premise of these polarization scans is that the sample is scanned slowly as the applied potential becomes more positive.

Corrosion occurs due to the anodic oxidation of iron ( $\text{Fe} \rightarrow \text{Fe}^{2+} + 2\text{e}^-$ ). At the open-circuit potential,  $V_{oc}$ , the current generated by this reaction is balanced by current generated by the cathodic reduction reaction of hydrogen evolution ( $2\text{H}^+ + 2\text{e}^- \rightarrow \text{H}_2$ ). We first ran a Tafel scan in order to determine  $i_{corr}$ , the corrosion current. The corrosion current is the current that relates to the open circuit potential. For this scan the potentiostat ranged from  $-250 \text{ mV vs. } V_{oc}$  to  $250 \text{ mV vs. } V_{oc}$  with  $V_{oc}$  being the open circuit voltage. This voltage encourages the electrochemical reactions on the surface of the metal that corrode the metal. On the Tafel scan  $i_{corr}$  is found by first extrapolating the linear portions of the curve. Then the value of the current where they meet is  $i_{corr}$ . An example can be seen below.

Tests were performed in order to confirm our setup and system were working properly. Below (Figure 4) is an overlay of polarization tests that were performed, which shows that the tests were reproducible.

Another feature of the potentiodynamic scans is the ability to determine whether or not a metal pits. Pitting is the process of cavities forming on the surface and propagating downward into the metal. This occurrence is unfavorable because once it begins the corrosion rate locally increases dramatically. Additionally, pitting weakens the structure of the metal much more so than uniform corrosion since the pits grow into the metal rather than just corroding the surface. Figure 5 is a graph showing uniform corrosion of M50 at 2M NaCl and 0.6M NaCl.



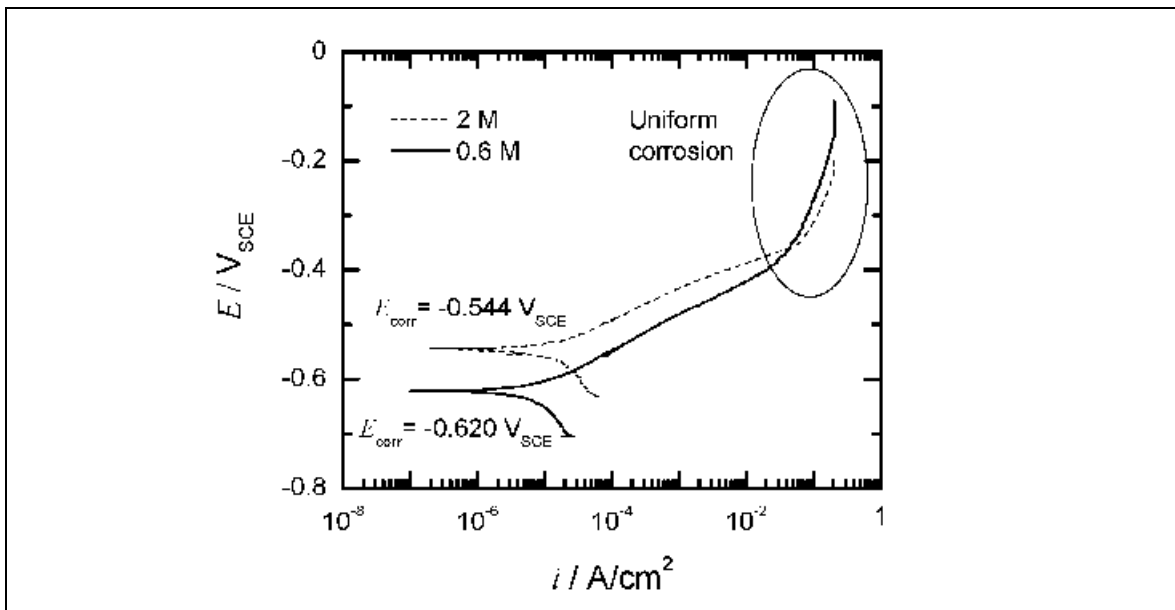


Figure 5: Potential as a function of current density for M50 SS with concentration of NaCl electrolyte as a parameter.

If the M50 had pitted, there would have been a sharp increase in current in the circled area of the graph in Figure 5. Since the slope remains mostly vertical rather than horizontal, pitting did not take place during the test and therefore will not take place anywhere. As the graph shows, this result will hold up even in a solution with a high concentration of salt.

Below are some pictures of the uniform corrosion. If pitting had taken place, the dark spots in the picture would be much more distinct.

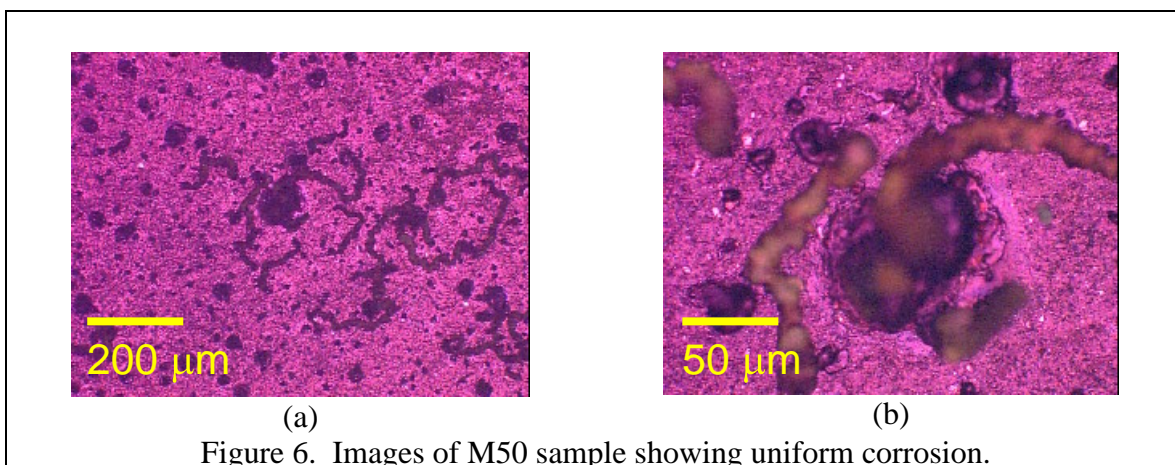


Figure 6. Images of M50 sample showing uniform corrosion.

The most important data gathered from the M50 was the lack of a pitting potential and the corrosion rate. This rate determined from the corrosion current using the following equation

$$r = 0.129 \cdot (a \cdot i) / (n \cdot D)$$

where  $r$  is the corrosion rate in mils per year,  $a/n$  is the equivalent weight of the alloy,  $D$  is the density in  $\text{g/cm}^3$ , and  $i$  is the current density in  $\mu\text{A/cm}^2$ . Using this equation along with the following values,  $i = 1.0$ ,  $a/n = .037$ , and  $D = 7.97$ , the calculated corrosion rate was  $r = .438$  mils/year for a solution of 0.5M NaCl.

## ***Electrochemical Impedance Spectroscopy (EIS) Scans***

Since the main objective was to test steels used in aircraft engines, a salt solution was not a practical solution in which to test the steel. Therefore, new and used oils were used as solutions as well. This however caused a major problem. Due to the high resistivity of the oil, the resistance to the solution,  $R_s$ , could not be assumed to be negligible as it previously was. Therefore, our simple single resistor model was not accurate and we could not run potentiodynamic tests. As a result, impedance techniques had to be used.

The physical setup of the system was the same for EIS as for the potentiodynamic runs. We used the same cell and electrodes although our solution was different. In addition to saltwater, we used new and used oil. The first EIS tests we ran we stuck with the 0.6 M NaCl solution. Even though we had already done tests in this solution, we wanted to be able to compare the EIS to the potentiodynamic scans.

EIS scans opened up a number of different possibilities for equivalent circuits due to the nature of the scan. By using an alternating current, low conductivity solutions could be modeled more complexly and therefore more accurately due to the information content of the EIS spectrum. While DC measurements dealt only with resistance, AC measurements dealt with resistance, capacitance, and inductance due to a modulating frequency. This enabled a much more complete model.

A popular way of representing electrochemical impedance data was through the Nyquist Plot. In this plot, the real impedance was plotted versus the imaginary impedance at each frequency used during the scan. This plot was helpful since the effects of capacitors, resistors, and inductors were known at different frequencies. As the frequency increased, the impedance of the capacitor decreased, the impedance of a resistor stayed constant, and the impedance of an inductor increased. By knowing this, an equivalent circuit can be modeled from the data gathered. A number of impedance scans were run in order to determine the effects of different conditions. The first scans we ran were the M50 in new and used oil at room temperature. These Nyquist plots are shown below.

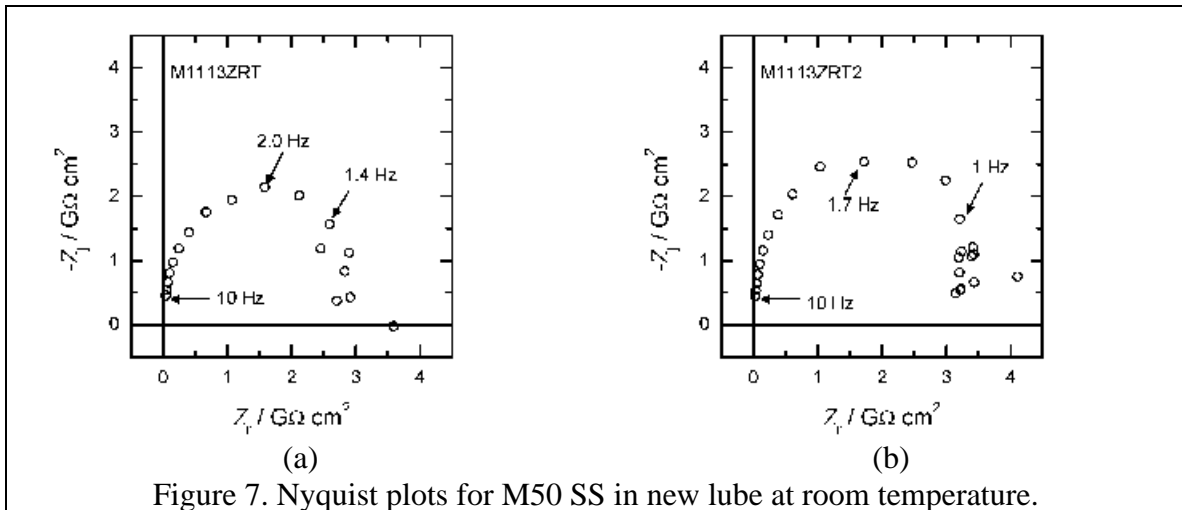


Figure 7. Nyquist plots for M50 SS in new lube at room temperature.

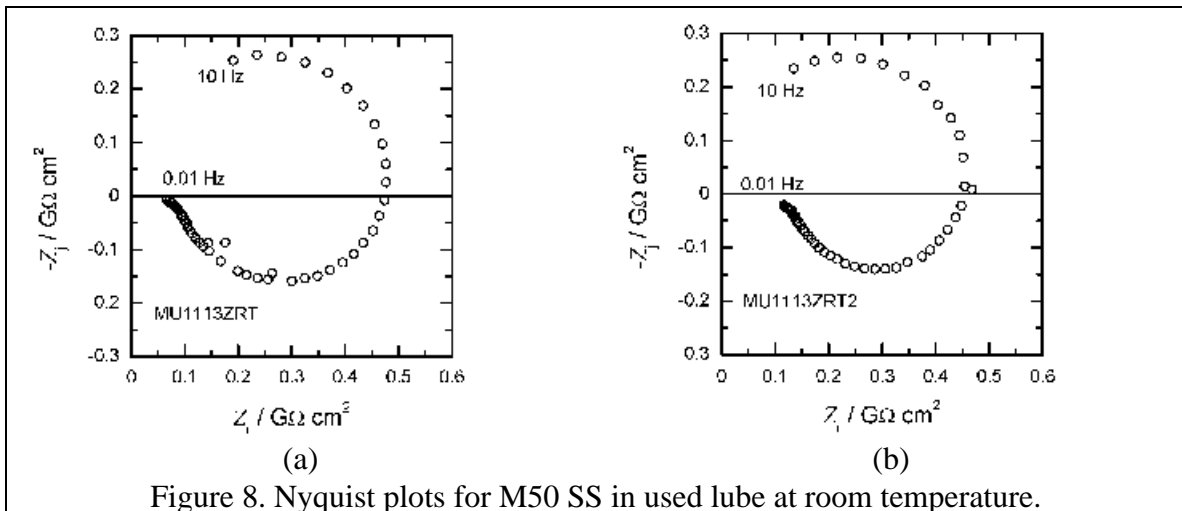


Figure 8. Nyquist plots for M50 SS in used lube at room temperature.

In the new oil a partial semi-circle above the real axis was associated with a capacitive effect. In the used oil on the other had there was a partial semi-circle below the real axis in addition to the one above the axis. This semi-circle below the axis was associated with an inductance effect. Therefore, the impedance response of the M50 in the used and new oils was very different. The main difference between the new and used oils is the concentrations of ions in the solutions. During use, salts dissolved in the oils making the solution more electrolytic. This decreased the resistivity of the oil, which made corrosion easier.

## Conclusion

The results demonstrated a number of things. First, it was shown the M50 does not pit. Second, the corrosion rate of M50 was found to be 0.438 mils/year in a 0.5M NaCl solution. Finally, the used oils showed an inductive feature that the new oils did not. This was caused by the increased conductivity of the oil, which was a result of the increased ion concentration. This can be avoided by changing oil more frequently.

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