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The Influence Of Light On The Electrochemical Characteristics Of Pure Aluminum

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The Influence Of Light On The Electrochemical Characteristics Of Pure Aluminum

4250:497-003 Honor Project

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April 28, 2017

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1. Executive Summary

1.1 Problem Statement

Aluminum is a very useful and efficient element that has been used extensively in many of our manufacturing industries. Its many positive characteristics make it an ideal metal for the applications that require a lightweight metal with the absence of magnetism, superior strength, heat resistance, flexibility, malleability, versatility, and corrosion resistance. As a result, aluminum is used ubiquitously in the automotive, aircraft, and food packaging industries that affect us all.

Previous studies have found that the presence of light has a direct effect on the electrochemical characteristics of aluminum and the protective passive film, which develops as the result of aluminum's exposure to air or aqueous solutions. The passive film has a relatively large band gap of 8.7 eV compared to other semi-conducting materials and has insulating properties in bulk. It is also recognized that the passive films of Al, like most metals, will exhibit semi-conductive properties, which are believed to play a role in its passivity and passivity breakdown.

Light has been found to amplify the corrosion potential of metals like Al through the emission of electrons from its surface. This effect is known as the photoelectric effect. The effect of light on the characteristics of the passive film and the corrosion parameters (i.e., pitting potential, transition potential, corrosion potential, corrosion current density, and metastable pitting behavior) must be recognized and quantified to be able to establish an accurate evaluation of the corrosion of aluminum.

1.2 Summary of Results:

Five different experimental methods were utilized to test for the influence of light on the electrochemical characteristics of pure aluminum's passive film while immersed in a 0.1 M NaCl solution: open circuit potential (OCP) measurements, galvanic potential difference, galvanic current (using zero resistance ammetry), cyclic potentiodynamic polarization (CPP), and electrochemical impedance spectroscopy (EIS). The exposure of a single aluminum electrode (in a 0.1 M NaCl solution) to the LED light of a wavelength 480-525 nm at 60 minute intervals (Off/ On) produced an increase in OCP of ~ 108 mV from its initial, stable non-illuminated state (-808 mV vs. SCE) to its LED illuminated value (-700 mV vs. SCE) (see Fig. 6 in the results section). Then when the aluminum electrodes were exposed to additional ambient daylight, the change in OCP increased to ~ 135 mV, an overall increase of ~ 27 mV over the LED only state. The effect of the same LED light, on the galvanic potential of two electrodes (illuminated vs. non-illuminated) was measured using 5-minutes (Off/ On) and 15 minute (Off/ On) intervals and found to increase by ~ 110 and ~ 100 mV, respectively (see Fig. 7a-b). The effect of light on the galvanic current was also examined using the ZRA method which produced a total decrease in current density of $\sim -0.225 \mu\text{A}/\text{cm}^2$ on the two aluminum electrodes as illustrated in Fig. 9 in the results section. The effect of light on CCP revealed that the average illuminated E_{pit} value (-553.19 ± 70.09 mV vs. SCE) is higher than the comparable non-illuminated pitting potential (E_{pit}) value (602.17 ± 52.70 mV vs. SCE) and there is a slight increase in the transition potential E_{trans} for illuminated versus non-illuminated conditions. The calculation of the average E_{pit} and E_{trans} and their standard deviation were illustrated in the Appendix A, Table 1. Like the other four tests, the final EIS test, used to evaluate the effect of light on the electrochemical characteristics of the passive film. This impedance increase due to light was observed as a semi-

circular radial increase of the Nyquist plot as compared to the comparable non-illuminated electrode (see Fig. 12 in the results section).

1.3 Conclusions

- The OCP of Al increased on illumination with LED (480-525 nm) by about 108 mV in a 0.1 M NaCl solution; and further increased by 27 mV due to the addition of ambient daylight.
- The galvanic potential of two identical Al electrodes increased by about ~100 mV due to the influence of LED illumination.
- The ZRA test revealed that the current density for the two-electrode setup decreased by $0.225 \mu\text{A}/\text{cm}^2$ from a positive ($0.075 \mu\text{A}/\text{cm}^2$) to negative current density ($-0.15 \mu\text{A}/\text{cm}^2$).
- The pitting potential of Al in a 0.1 M NaCl solution, increased by ~49 mV due to illumination with an LED light.
- The impedance of Al in a 0.1 M NaCl solution, increased under LED illumination as compared to the non-illuminated state.

1.4 Implications

This project provided the opportunity for technical laboratory skill development within a number of areas, including sample preparation, electrode specimen molding, electrical conductivity testing, and automatic/ manual operation of the grinder. More specifically this project refined my hands-on technical abilities with regard to the four electrochemical test methods (OCP, CPP, ZRA, and EIS) and honed my understanding of the type of data they produce. Utilization of the Bio-Logic VMP 300 Potentiostat and the Zenith microscope provided me with the opportunity to master their technical intricacies and requirements. Valuable

computer application of the Origin Lab software provided further opportunities to transform the project data into visually conclusive plots and results.

On a more personal level, the project created opportunities for my personal growth through the need for creative solutions to technical problems, equipment sharing and maintenance, and interpersonal cooperation and safety within the laboratory space. Normal and inevitable difficulties also provided me with opportunities to practice patience, self-motivation, perseverance, a deeper knowledge base after extensive review of the literature, and the opportunity to present this study as a poster at the 2017 NACE conference in New Orleans.

The broader implications of this study reveal the confounding role that light can play in the estimation of aluminum corrosion parameters and, therefore, the need to control and quantify for the effect of light through its interference with the protective, aluminum passive film. Recognition of this phenomenon should aid in the accuracy of future aluminum research that involves illumination.

1.5 Recommendations

Taking into account the results of this project and recognizing the effect that both microscopic and ambient light have on the electrochemical characteristics of aluminum passive films, I have many suggestions about possible future projects. First, applying the results from this project, I would recommend completion of my original plan to image and record the metastable pitting phenomena of aluminum wire of variable diameter (50 μm , 100 μm , and 500 μm) simultaneously with the collection of electrochemical data of its passive film. This would give a better understanding of the metastable mechanism through the identification of its initiation and progression. A secondary recommendation would be to examine the effect of a wide variety of different types of light on the electrochemical characteristics of aluminum passive films. A third option would be to study the effect of impurities or the use of different

aluminum alloys on the electrochemical characteristics of the aluminum passive film. Fourthly, I recommend, a Mott-Schottky analysis of the surface of the protective, passive film.

My advice to other students who may wish to work on this type of project would be to pay close to the electrode preparation remembering that a bad connection can create noise and an unpredictable drop in the OCP. Therefore, the quality of the wire used for the connection must be well isolated and resistant to breakage.

2.0 Introduction

It has been reported that metastable pitting can be a reliable and accelerated method for determining the pitting susceptibility of the materials.^{[1],[2],[3],[4],[5]} However, metastable pitting characteristic of the alloys are not well understood. The original purpose of this project was to study the mechanism that leads to the metastable pitting of aluminum in order to improve and optimize the measurement and quantification of the pitting mechanism. In order to accomplish this objective, multiple microelectrodes were created for use in the development of the electrochemical cells. Simultaneously, throughout the metastable pitting process, videos were recorded using a high-resolution camera attached to an optical microscope. Both the electrochemical data and the recorded videos were then compared, in order to identify the exact location and point in time when the metastable pitting began with monitoring continuing until they either re-passivate or transform into a stable pits. Some difficulties were encountered with the initial creation of the microelectrodes, the setup of the electrochemical cell, the detection of a small current density without the interference of ambient noise, and the detection of the impact of hydrogen evolution utilizing an optical microscope. However, the major difficulty encountered was the influence of the focused light applied by the microscope, over and above the level of the ambient room light.

Upon examination of the literature, it was found that the effect of light changes the density of the current flowing through metal as well as the potential difference between illuminated and non-illuminated electrodes. The effect of light on the potential between the two electrodes is known as the Becquerel effect.^[6] Bastidas and Scantlebury recorded an increase in the corrosion rate of mild steel in citric acid with the exposure of the electrode to light.^[7] Similarly, Macdonald et al. also established that UV light caused passivity breakdown of 304 stainless steel in 1.0 M NaCl.^[8] Given the proven effect of light on the corrosion rate of mild steel and stainless steel, this study was focused on establishing the probable similar effect of light on the OCP, and the CPP of pure aluminum electrode, as well as its effect on the galvanic current between illuminated and non-illuminated aluminum electrodes using the ZRA measurement method.

3.0 Background

Previous electrochemical corrosion studies have focused on the behavior of aluminum and its alloys because of the expansion of their use within the construction, automotive, aircraft, and food processing industries. Their use for multiple industrial applications is possible due to the formation of its stable, passive Al_2O_3 films that form in both air and aqueous solutions ^[6]. (This film has a relatively large band gap of 8.7 eV compared to other semi-conducting materials and is known to have insulating properties in bulk. However, a previous study found a difference in the current density of Al in 0.5 M KSCN under illuminated versus non-illuminated conditions during cyclic potentiodynamic polarization (CPP)^[6]. (Fig1.)

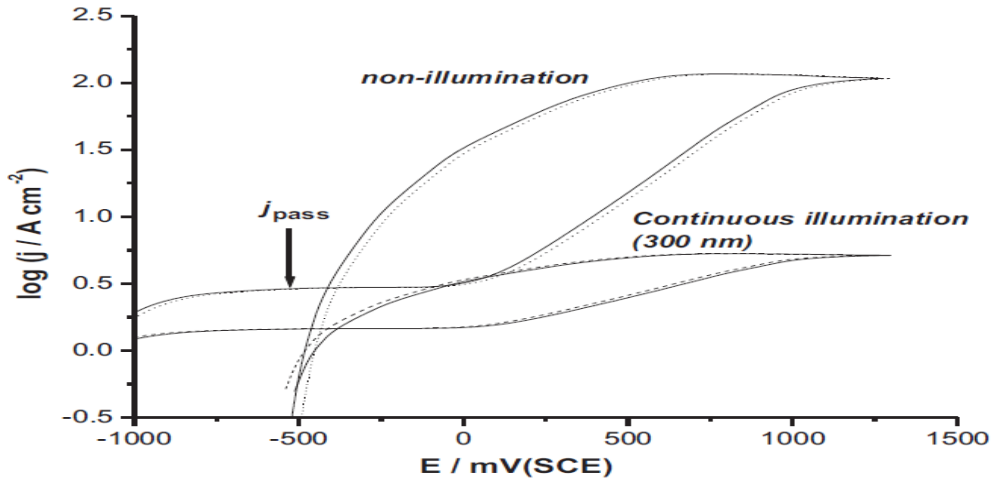


Fig 1. Cyclic potentiodynamic polarization curves recorded for Al in 0.5 M KSCN at 25 °C under the conditions of continuous illumination at 300 nm and non-illumination^[6].

It is also recognized that the passive films of Al, like most metals, will exhibit semi-conductive properties, which are believed to play a role in its passivity and passivity breakdown.

Light has been found to amplify the corrosion potential of metals like Al through the emission of electrons from its surface. This effect is known as the photoelectric effect (Fig1).

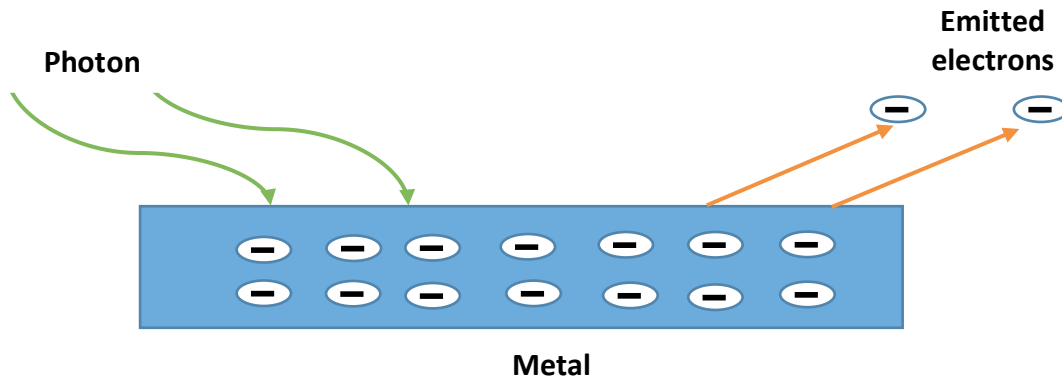


Fig 2. Schematic for the photoelectric effect showing emission of electrons when incident photons strike the metal surface

It has been established that light can cause a change in the electric current flowing through a metal, which results in the alteration of its potential. This change in potential difference due to

the influence of light has been referred to as the Becquerel effect^[7]. The basis of this phenomenon is the generation of electron-hole pairs as is illustrated below.

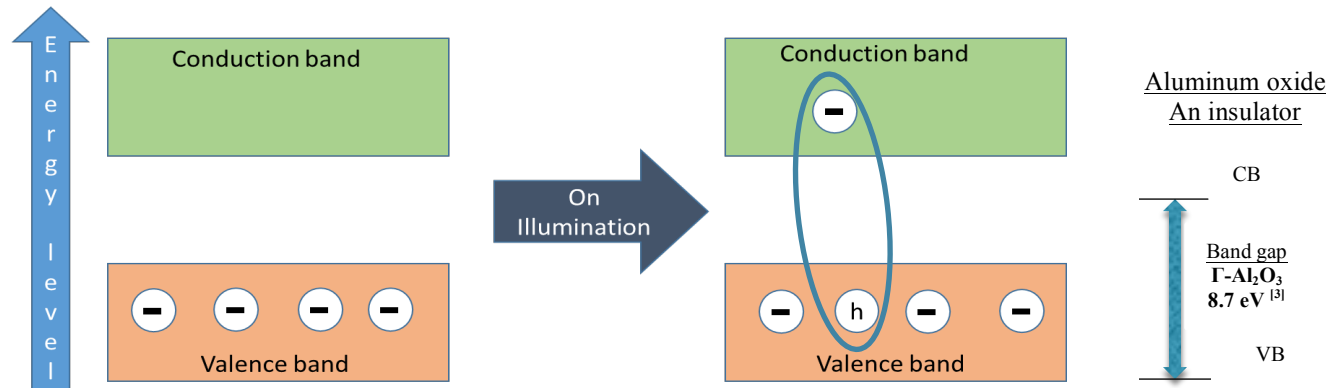


Fig 3. Schematic showing the effect of illuminating a semi-conductor oxide film with sufficiently energetic photons, which emit electrons from the valence to the conduction band generating electron-hole pairs

4.0 Experimental Method

4.1 Sample Preparation

Samples were prepared using disposable mounting cups and alligator wires. First the mounting cups are sprayed with mold release spray. Then the pure Aluminum wire was mounted vertically to the center of each mold using super-glue and a mixture of epoxy and epoxy hardener poured to cover $\frac{3}{4}$ of the mold, which was then left to harden overnight. Simultaneously, alligator clips were prepared by modifying one side of the clip by adding a 22-16 AWG connector to the side, thereby making it easier to connect the clip to the aluminum wire. The following day the alligator wire was connected to the aluminum wire and the final $\frac{1}{4}$ of the mold filled with the epoxy mixture, then again left to dry overnight. After the final drying period, the electrodes were released from the molds and ground flat on the bottom in order to expose the

aluminum wire. The conductivity of the electrode was then verified using a voltmeter. Similarly, double electrode samples were prepared in order to perform the ZRA measurements. All samples were then polished using 600 through 1200 silicon carbon paper (see fig 4).

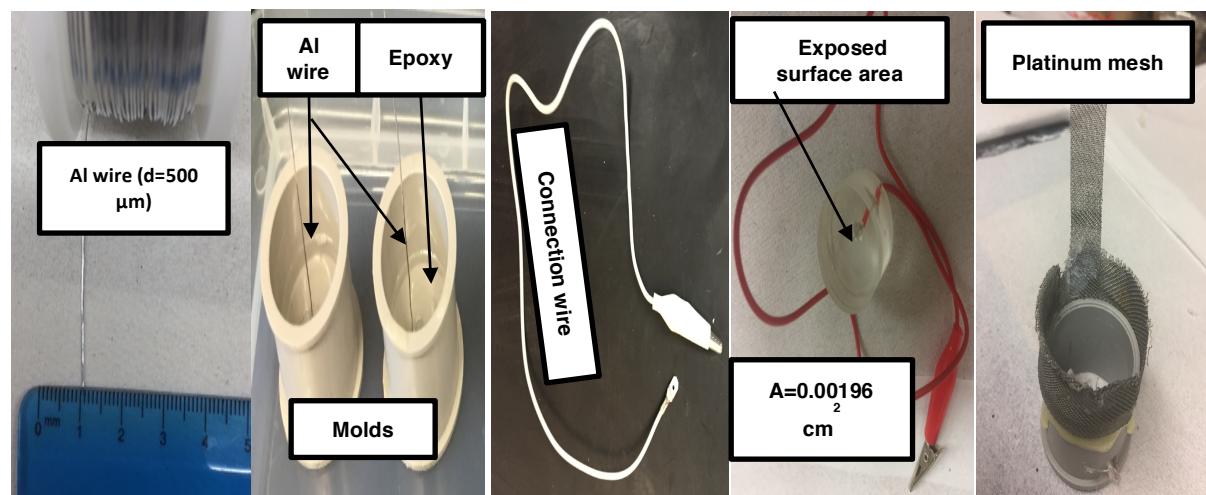


Fig 4. Working electrodes preparation

4.2 Electrochemical Techniques

The electrochemical measurements were carried out using the three electrode standard configuration^[6], where the working electrode was the prepared single electrode of pure aluminum, the counter-electrode was a platinum net mounted to the periphery of the epoxy mount of the working electrode, and all potentials were measured against a reference saturated calomel electrode (SCE)^[8]. Both the working and counter electrodes were mounted to the bottom of a circular glass dish of approximately 2 - 2 1/2 inches in depth. The three-electrode cell was then immersed in 0.1 M NaCl solution. The whole apparatus was then exposed to a controlled, monitored microscopic light source ($\lambda = 480-525$ nm). The first part of the experiment was conducted using the single electrode sample. The OCP was then measured

continuously while allowing 5 minute, intervals of light exposure followed by 15 minute intervals without microscopic light (although the ambient room light remained constant). Thus one light on/off cycle took a total of 30 minutes. The total measurement period covered five cycles of light on/off, after an initial period of potential stabilization. A second series of light on/off time interval measurements was then performed for the same single cell electrodes based on one-hour intervals of light on and then off for a total of five cycles.

The second part of the experiment using the double-electrode cell was carried out without using the platinum screen as a counter-electrode. Instead, one electrode was exposed to light and used as the working electrode, while the second was used as the counter electrode. The OCP was again measured against a reference saturated calomel electrode (SCE). The OCP measurement was taken at 5 minute intervals of light on/off and again for intervals of 15 minute light on/off, for a total period of five cycles each.

In the next portion of the experiment, the apparatus set-up is very similar to that described above except that the galvanic current between the two aluminum electrodes was to be measured using a Zero-Resistance Ammeter (ZRA) technique where the standard three electrode configuration was utilized. Initially, both Al electrodes were immersed in a 0.1 M NaCl solution for 4 hours in total darkness. Thereafter, the working electrode was exposed to an illuminating light of wavelength ($\lambda=480-525$ nm) emanating from an embedded LED microscope light bulb (see Fig 5.).

For the fourth cycle of experiments, cyclic potentiodynamic polarization (CPP) measurements were carried out using the same apparatus set-up detailed above for the OCP measurements, but with measurements of CPP taken instead. Scans of the potential were recorded by sweeping the potential linearly in the positive direction from the starting potential

and then reversing the scan direction at the equal rate of 0.50 mVs^{-1} until obtaining a hysteresis loop developed according to the following sequence: developing metastable pitting, followed by reaching potential pitting, transition potential, and potential passivation. These measurements were performed in order to evaluate the pitting potential (E_{pit}) and the transition potential (E_{trans}) of aluminum under illuminated and non-illuminated conditions.

The last set of experiments to be completed also involved the single-electrode set-up described above when measuring OCP and CPP, except the target measurement in this case would be the impedance parameters of aluminum under illuminated versus non-illuminated conditions using electrochemical impedance spectroscopy (EIS). Measurements were carried out using AC signals of amplitude 5 mV peak to peak at the OCP in the frequency range of 100 kHz to 1 mHz^[6].

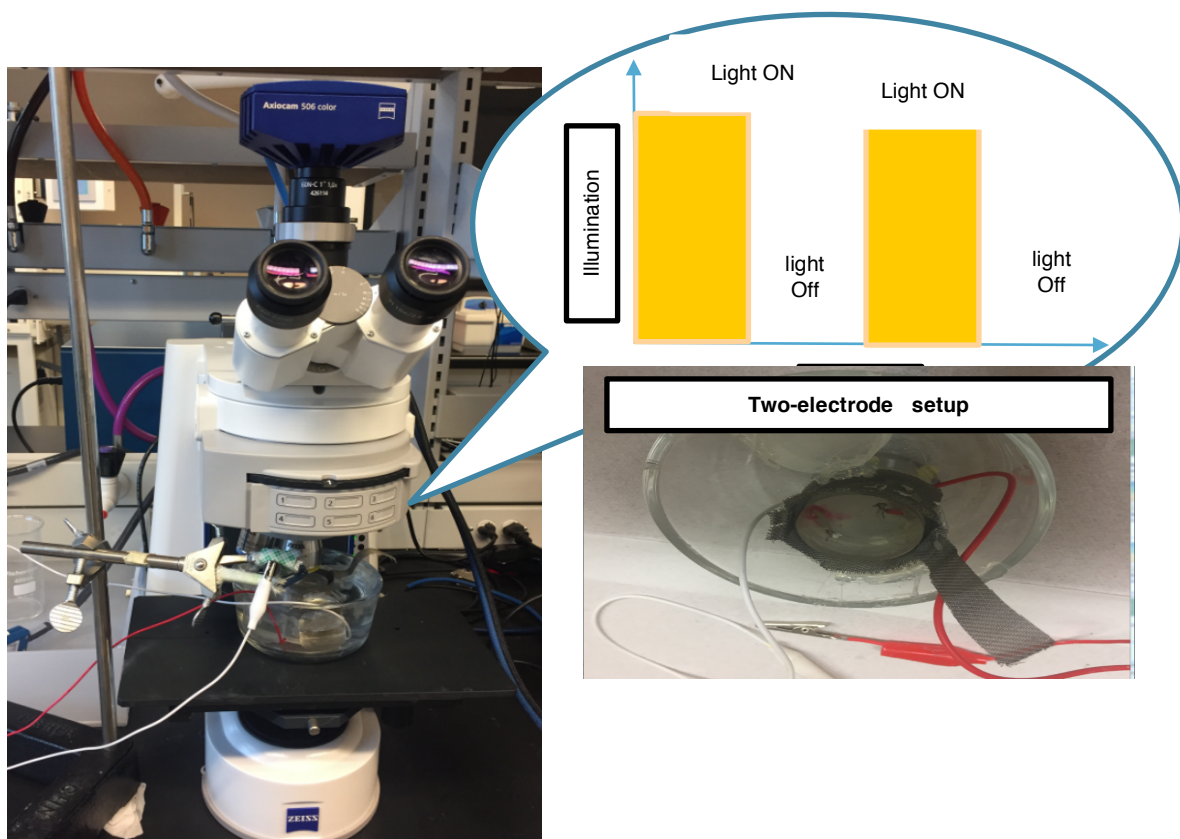


Fig 5. Three-electrode cell set up mounted under the microscope

5.0 Results and Discussion

5.1 Effect of illumination ($\lambda=480-525$ nm) on OCP

After an initial 5-hour stabilization period, the potential of the single aluminum electrode in a 0.1 M NaCl solution without the microscopic illumination (light off) was stabilized at a value of -808 mV versus SCE. Thereafter, light was introduced in cyclical 60-minute intervals of “light on” followed by “light off” for a total of 5 cycles. During the first 4 cycles an increase of approximately 108 mV was observed between the two conditions (lights on versus lights off) which were conducted during an overnight experiment; with an additional increase in the last cycle due to the influence of the extra ambient light produced by daylight

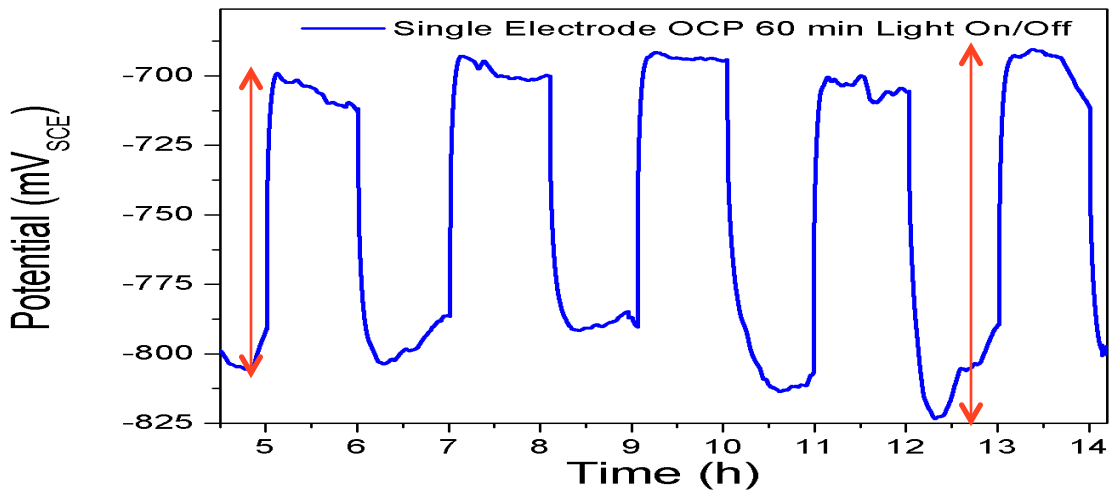


Fig 6. The change in the OCP of an Al electrode in a 0.1M NaCl solution exposed to LED light ON-OFF conditions was initially ~108 mV and later increased to ~135 mV due to the additional ambient daylight.

5.2 Effect of light causing galvanic potential difference

Using dual electrodes to study the effect of light on the galvanic potential difference, OCP measurements revealed a ~ 110 mV increase in potential difference with 5-minute lights On-Off intervals as illustrated in Fig 7-a. A similar difference in OCP of ~ 100 mV was also observed when illumination was altered along a 15 minute interval schedule as illustrated in Fig 7-b. It should be noted that the two Al electrodes were in the same epoxy mold and the potential difference between the illuminated and non-illuminated electrodes was measured. The illuminated electrode was working electrode whereas non-illuminated electrode was reference electrode.

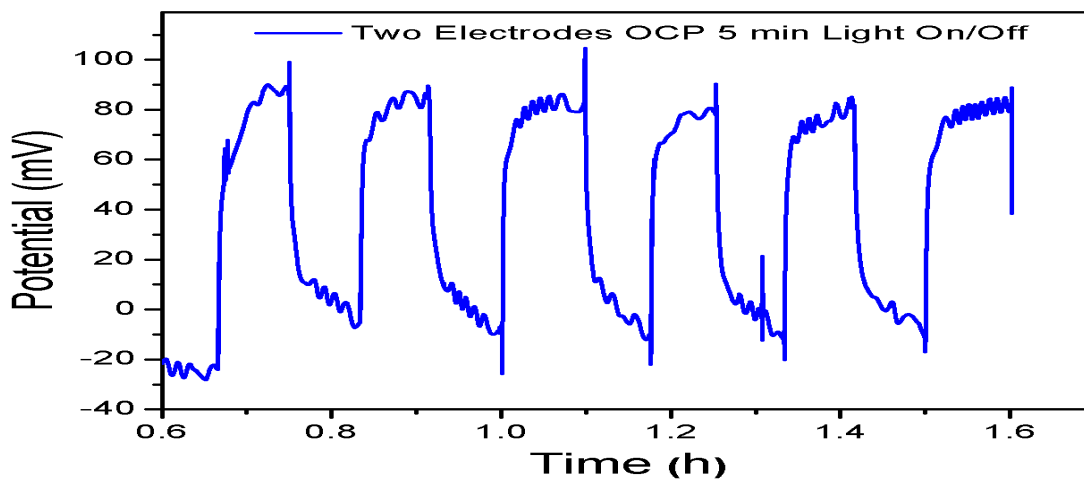


Fig 7-a. The change in the OCP between two Al electrodes in a 0.1 M NaCl solution is ~ 110 mV under ON-OFF LED light conditions, while alternating illumination every 5 minutes.

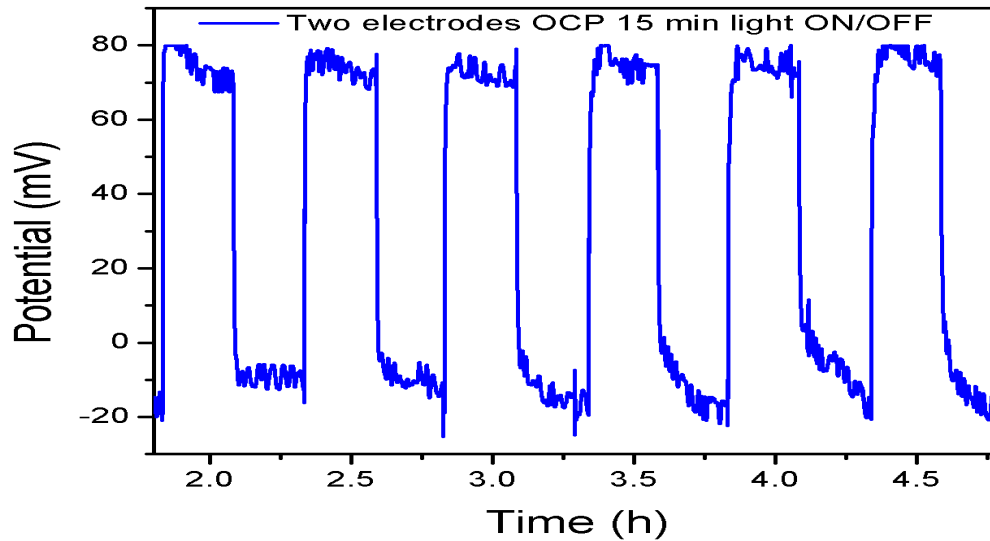


Fig 7-b. The change in the OCP between two Al electrodes in a 0.1M NaCl solution is ~ 100 mV under ON-OFF LED light conditions, while alternating illumination every 15 minutes.

5.3 Effect of light on galvanic current density

Using the same double electrode set-up, the change in current density between the two aluminum electrodes in 0.1 M NaCl solution was measured using the ZRA technique. Initially, the change in current density was positive ($\sim 0.15 \mu\text{A}/\text{cm}^2$) and stabilized at $\sim 0.075 \mu\text{A}/\text{cm}^2$ after about 1 hour and remained constant over the remaining 4 hours of darkness. At the 5th hour the LED light was switched on, which led to a rapid decrease in the current density to an initial negative value of $\sim -0.18 \mu\text{A}/\text{cm}^2$ and eventually stabilized at a value of $-0.15 \mu\text{A}/\text{cm}^2$. These results suggest that the effect of light caused a total decrease in current density of $\sim -0.225 \mu\text{A}/\text{cm}^2$ on the two aluminum electrodes as illustrated in Fig. 9. Illumination causes a change in the sign of the current density, i.e. the electrode was anodic which became cathodic due to the illumination.

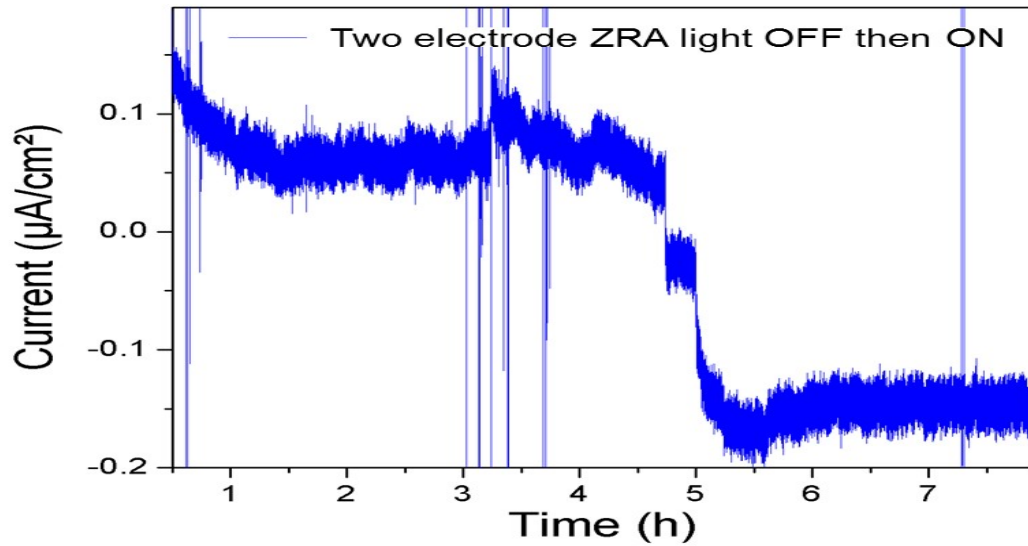


Fig 9. Zero resistance ammetry (ZRA) showing positive current for two Al electrodes in the absence of illumination, which decreases by $0.225 \mu\text{A}/\text{cm}^2$ to become negative current of $-0.15 \mu\text{A}/\text{cm}^2$ in the presence of illumination

5.4 Effect of light on the cyclic potentiodynamic polarization (CPP)

In order to evaluate the pitting corrosion of the Al test samples immersed in the 0.1M NaCl solution, pitting potentials (E_{pit}) and pitting transition potentials (E_{trans}) were estimated from the generated cyclic potentiodynamic curves as a function of sample composition under both illuminated and non-illuminated conditions. Multiple cyclic potentiodynamic polarizations were completed for each condition using the single electrode apparatus described above in the procedure section for the purposes of verification and reproducibility of the data. Figure 10 illustrates the comparison between two of the many generated CPP curves for illuminated and non-illuminated samples. The table that follows represents the calculated average for all values of E_{pit} , E_{trans} , and their standard deviations. From these values, it is evident that the presence of illumination has a positive effect on the pitting resistance of Al in a 0.1 M NaCl solution. The results indicate that the average illuminated E_{pit} value ($-553.19 \pm 70.09 \text{ mV vs. SCE}$) is higher than the comparable non-illuminated E_{pit} value ($602.17 \pm 52.70 \text{ mV vs. SCE}$). There is also a

slight increase in the E_{trans} for illuminated versus non-illuminated conditions. The noble shift of E_{pit} and E_{trans} in the presence of light is due to the semiconducting nature of the passive film, where quenching of vacancies within the electric field by electron-hole pairs results in a decrease in the flux of cation vacancies across the protective film^[6] (the Becquerel Effect).

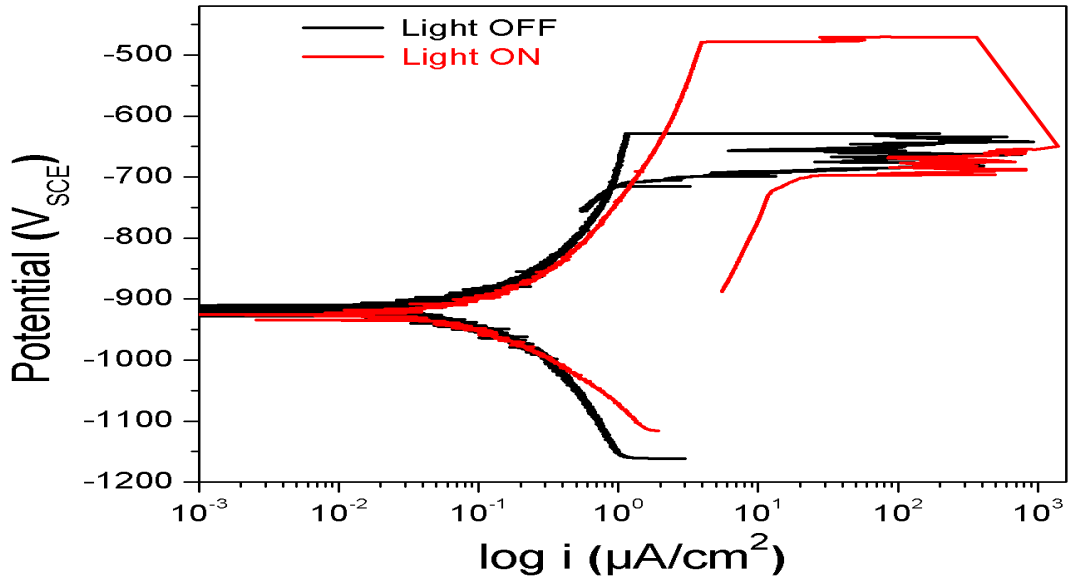


Fig 10. The CCP plots of illuminated and non-illuminated Al electrodes in a 0.1M NaCl solution showing a ~ 0.1 V increase in the illuminated pitting potential, but no effect in either transition potential

Table 1. The calculated averages and standard deviations of pitting potential and transition potential values for Al electrodes in a 0.1M NaCl solution under illumination.

L. On	E_{pit} (mV)	E_{trans} (mV)
Avg	-553.19	-706.46
S.D.	70.09	6.82

Table2. The calculated averages and standard deviations of pitting potential and transition potential values for Al electrodes in a 0.1M NaCl solution without illumination.

L. Off	Epit (mV)	Etrans(mV)
Avg	-602.17	-713.73
S.D.	52.70	27.79

5.5 Effect of light on the EIS

Like the other four tests, the final EIS test, used to evaluate the effect of light on the electrochemical characteristics of the passive film, likewise confirmed the positive increase of the potential as concomitant with a decrease in the current density exhibited by an increase of the impedance surface^{[10],[11]}. This impedance increase was observed as a semi-circular, radial increase of the “lights on” Nyquist plot as compared to the comparable non-illuminated electrode (see Fig. 12 in the results section).

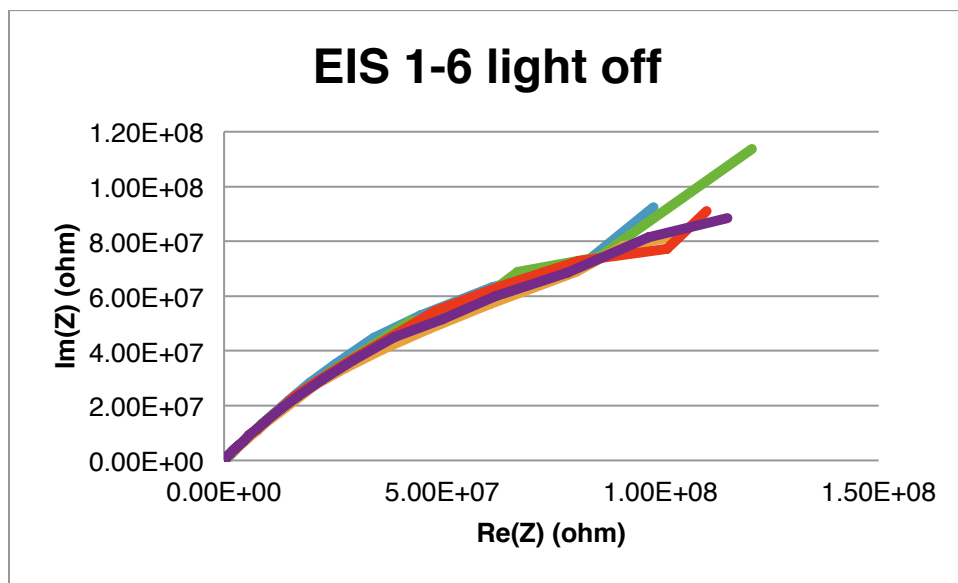


Fig 11. Multiple Nyquist plots of non-illuminated Al electrodes in a 0.1M NaCl solution show consistency of the impedance surface.

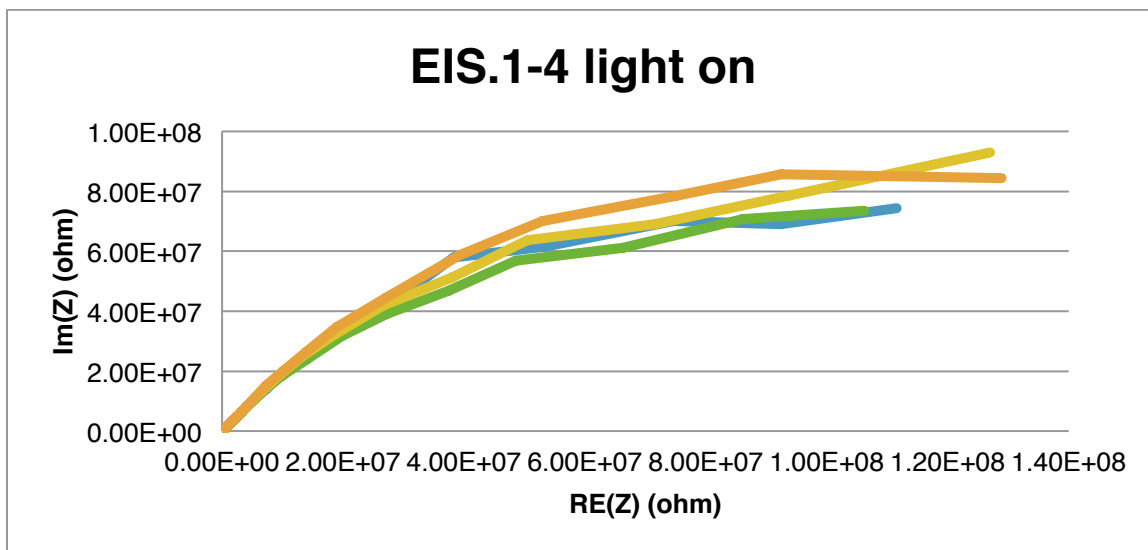


Fig 12. Multiple Nyquist plots of illuminated Al electrodes in a 0.1M NaCl solution show inconsistency of the impedance surface due the inconsistency of the density of light.

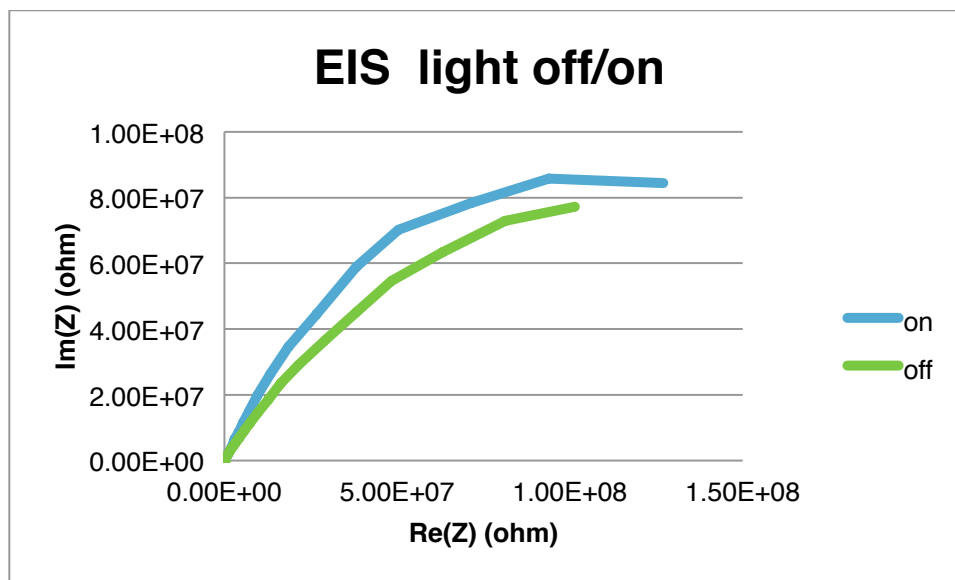


Fig 13. Nyquist plots of illuminated vs. non-illuminated Al electrodes in a 0.1M NaCl solution show an increase in the impedance surface due to the density of light.

6.0 Conclusion

The effect of LED light of wavelength (480-525 nm) on the electrochemical characteristics of the aluminum passive film have been studied in 0.1M NaCl solution based on OCP, CPP, ZRA, and EIS methods of chemical analysis. The changes in the corrosion

parameters of the Al passive film discussed above, can be explained by effect that the LED light has on the suppression of the pitting attack at the passive layer. Breslin and Macdonald suggested that this phenomenon occurred as result of a photo-induced quenching of the electric field strength, which modifies the vacancy structure of the passive film^{[8],[9]}. Therefore, the influence of light was found to be protective of the passive layer through the increase in passivation due to the increase in photon energy during illumination and the overall decrease in pitting.

7.0 Acknowledgement

I would like to acknowledge Mohammad Umar Farooq Khan, Dr. Scott Lillard, Dr. Rajeev Gupta, and his entire lab team for their effort and support during the time I was sharing the equipment and the space to get this project done.

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Appendix A: Plots of the raw data

Please note, due to the large amount of raw data that would need to be presented in table form, all the experimental data presented here (in plot form) represents and supports the generated plots in the body of the work above:

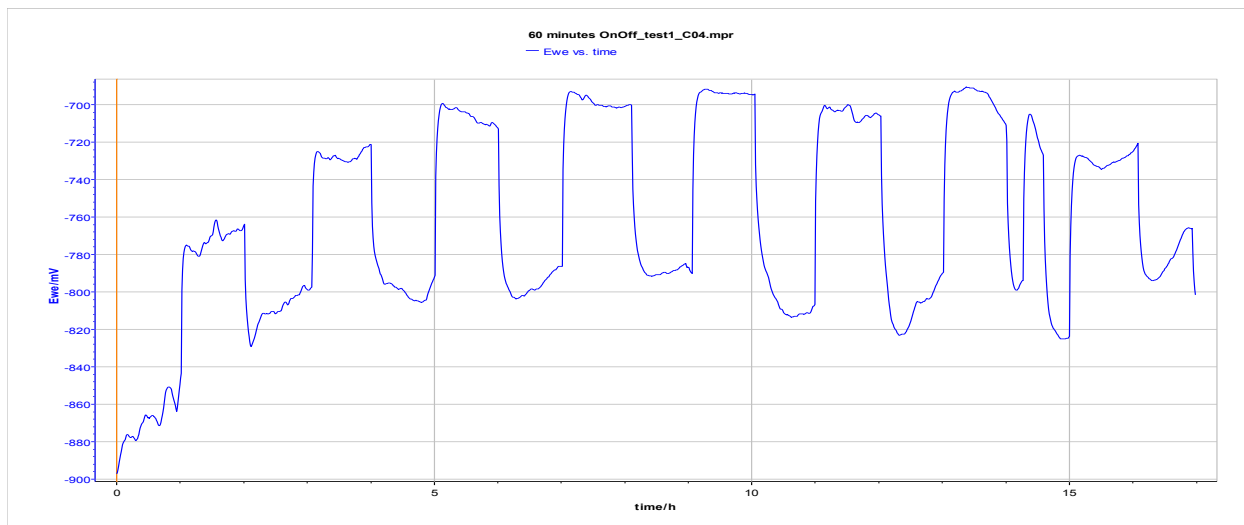


Fig 14. Original plot representing the change in the OCP of the Al electrode in a 0.1M NaCl solution exposed to LED light ON-OFF conditions, as presented in Fig.6 of the Results and Discussion section.

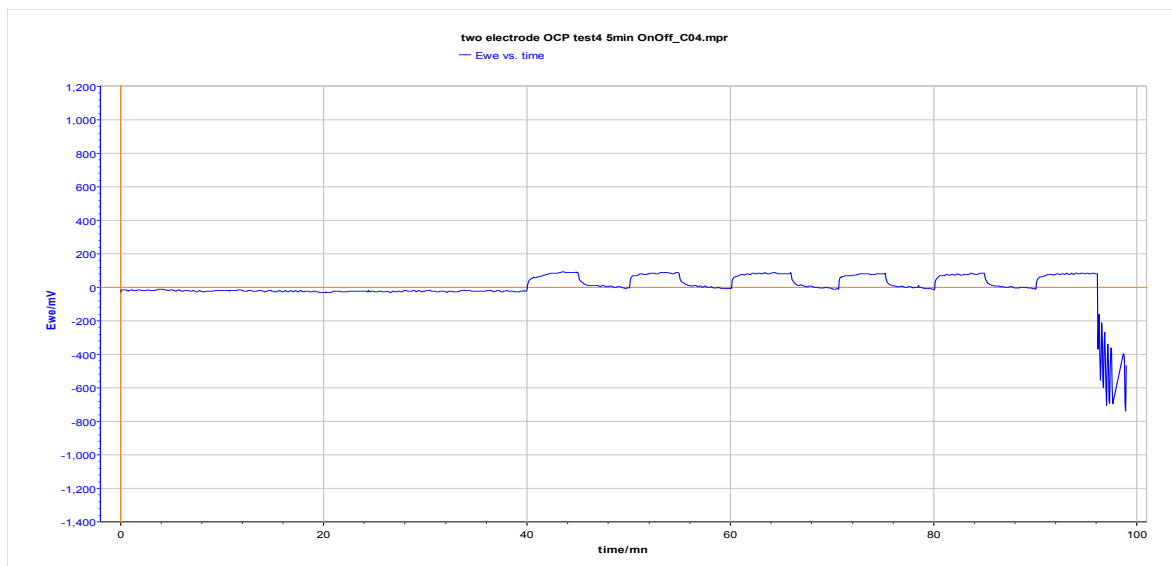


Fig 15. Original plot representing the change in the OCP between two Al electrodes in a 0.1 M NaCl solution under ON-OFF LED light conditions, while alternating illumination every 5 minutes (as presented in Fig 7.a of the Results and Discussion section).

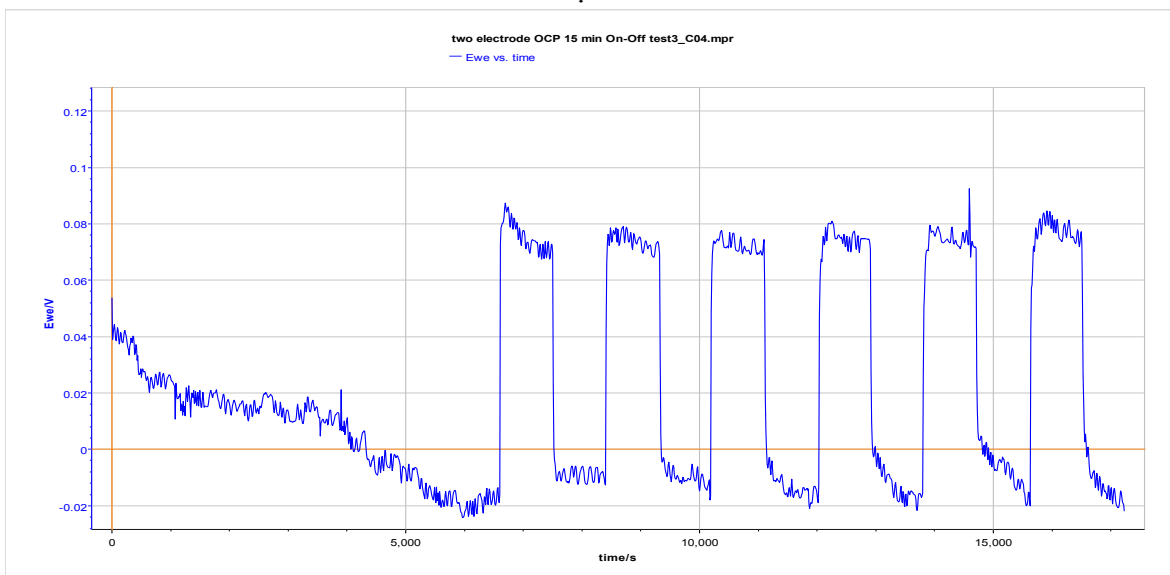


Fig 16. Original plot representing the change in the OCP between two Al electrodes in a 0.1 M NaCl solution under ON-OFF LED light conditions, while alternating illumination every 15 minutes (as presented in Fig 7.b of the Results and Discussion section).

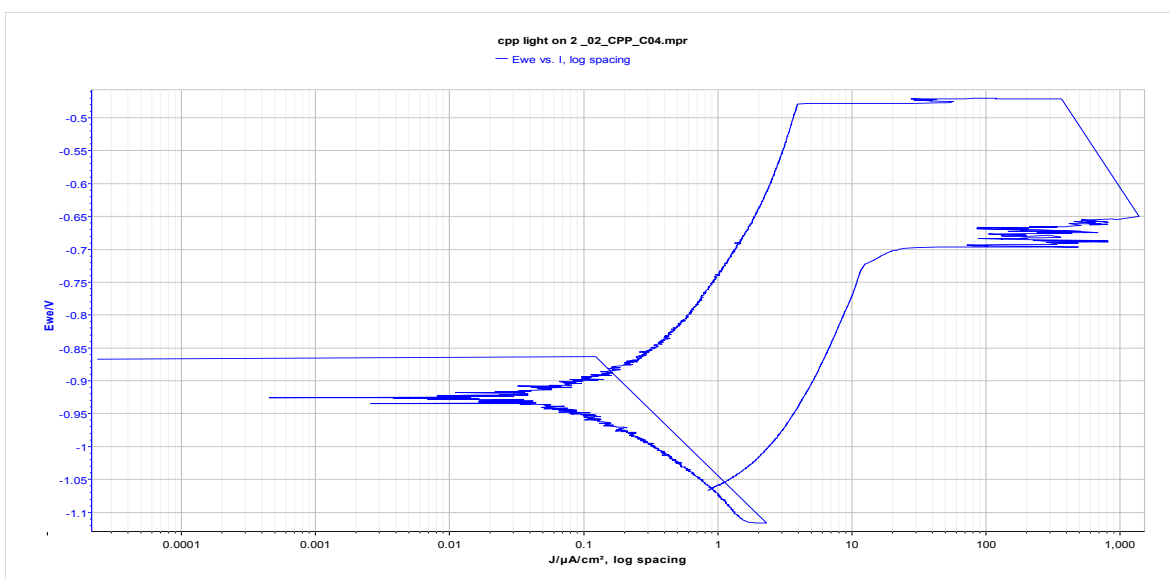


Fig 17. Original plot representing the pitting potential of an illuminated Al electrode in a 0.1M NaCl solution, as presented in Fig.10 of the Results and Discussion section.

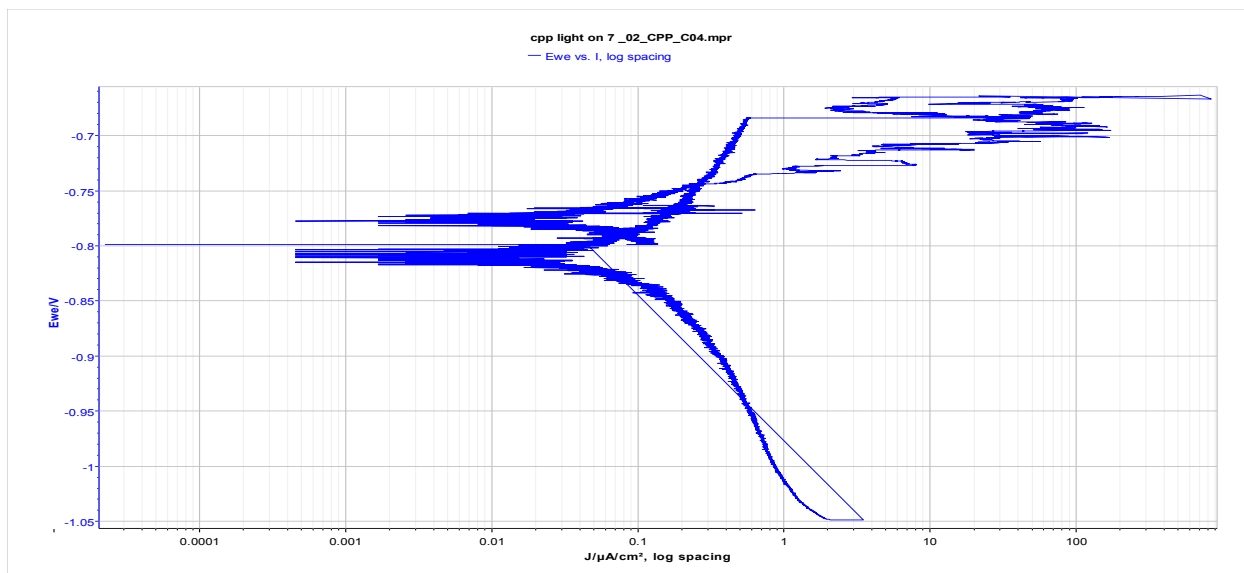


Fig 18. Original plot representing the pitting potential of a non-illuminated Al electrode in a 0.1M NaCl solution, as presented in Fig.10 of the Results and Discussion section.

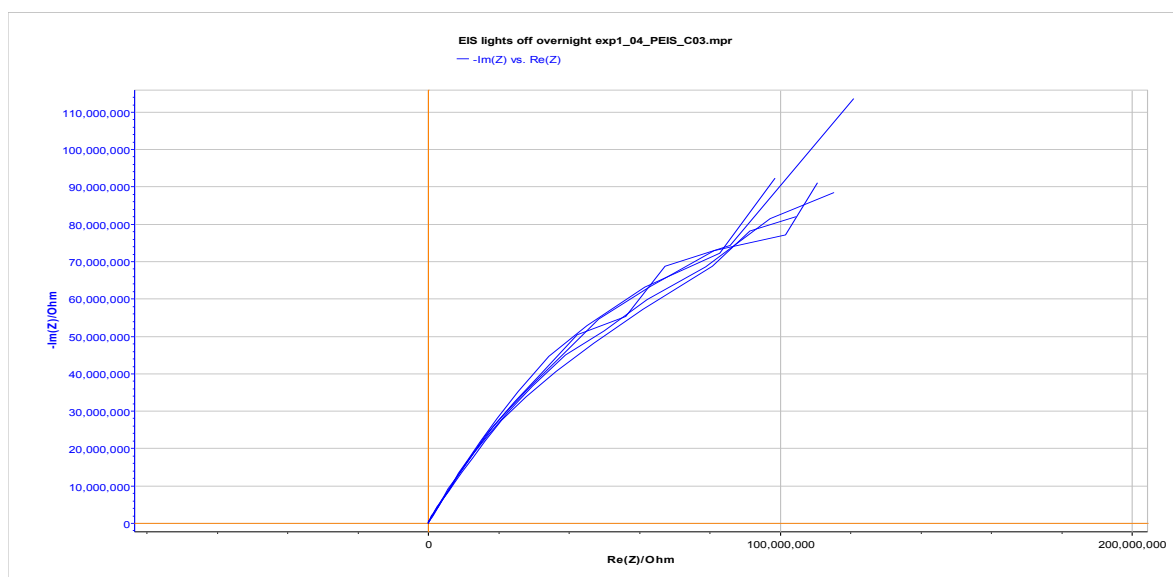


Fig 19. Original plot representing the multiple Nyquist plots of a non-illuminated Al electrode in a 0.1M NaCl solution, as presented in Fig 11 of the Results and Discussion section.

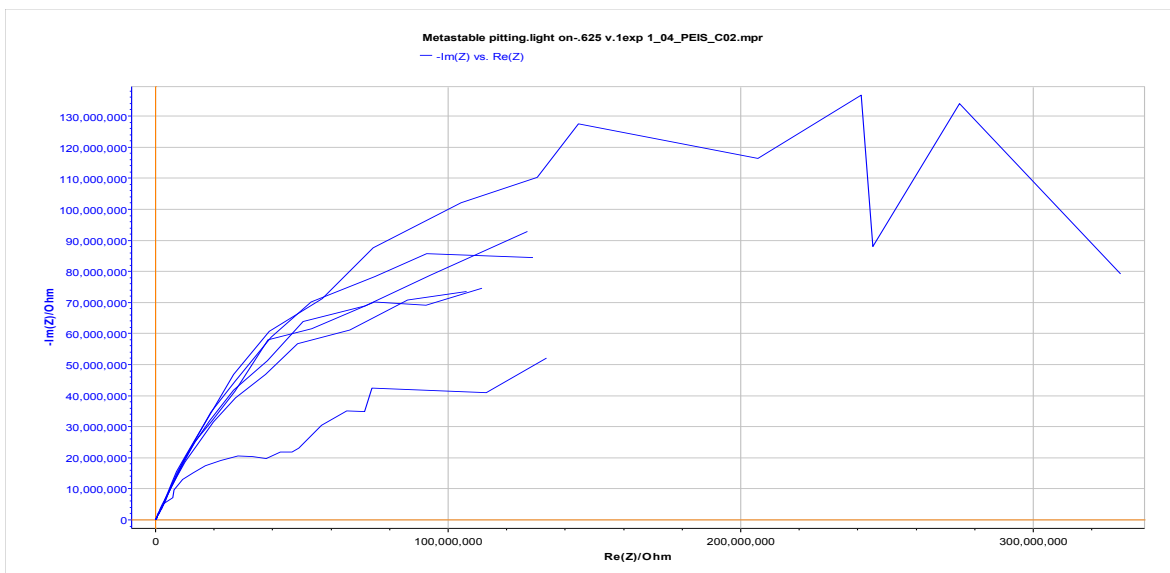


Fig 20. Original plot representing the multiple Nyquist plots of an illuminated Al electrode in a 0.1M NaCl solution, as presented in Fig 12 of the Results and Discussion section.

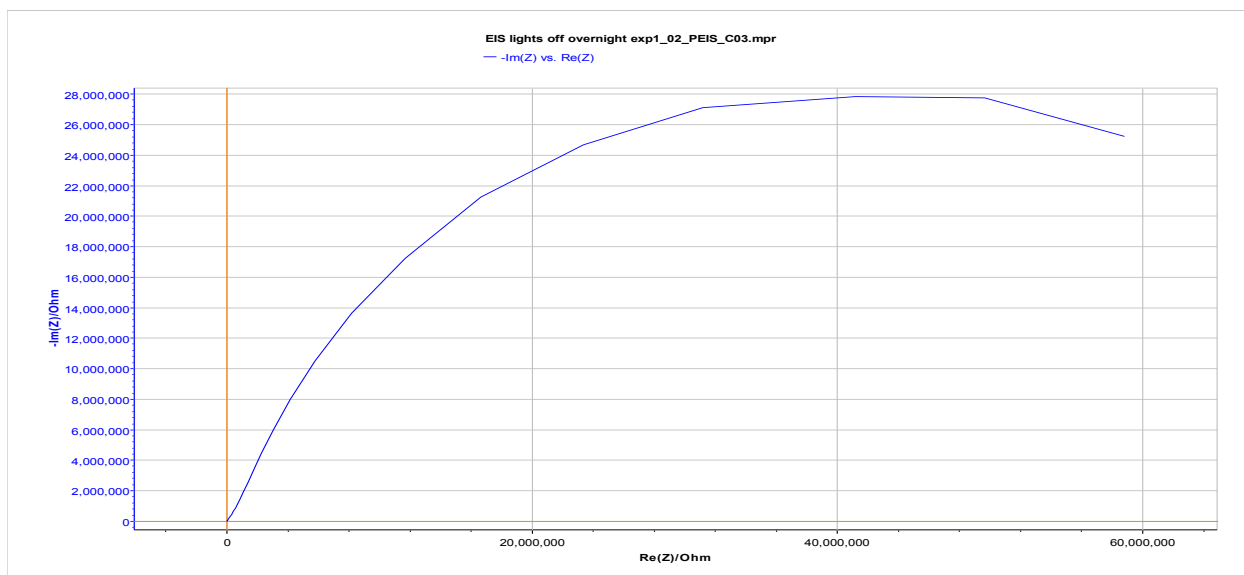


Fig 21. Original plot of the Nyquist plot of an illuminated Al electrode in a 0.1M NaCl solution, as presented in Fig 13 of the Results and Discussion section.

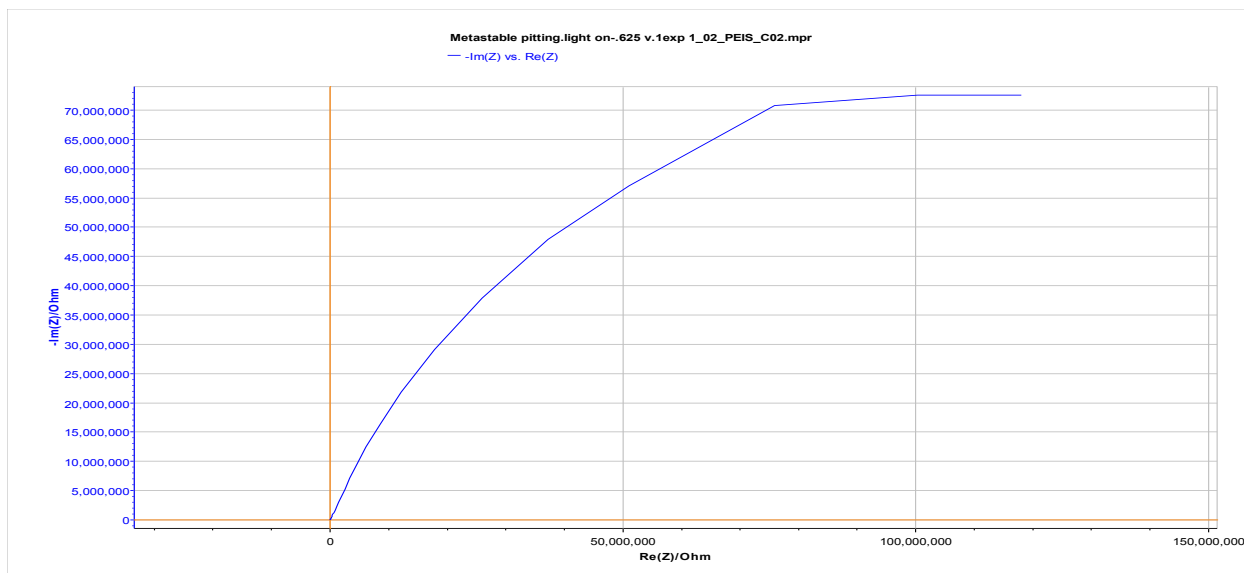


Fig 22. Original plot of the Nyquist plot of a non-illuminated Al electrode in a 0.1M NaCl solution, as presented in Fig.13 of the Results and Discussion section.

Appendix B: E_{pit} and E_{trans} average values and their standard deviations

Table 3. The calculation of the average pitting potentials and transition potentials and their standard deviations for illuminated and non-illuminated Al electrodes in a 0.1 M NaCl solution.

OCP	Light On		Light Off	
	Ep (mV)	Etrans(mV)	Ep (mV)	Etrans(mV)
1	-472.27	-698.46	-610.23	-671.55
2	-535.49	-714.99	-652.83	-718.96
3	-488.19	-698.63	-593.38	-743.81
4	-628.28	-712.06	-659.33	-689.18
5	-641.74	-708.17	-515.65	-734.66
6	-553.18	-706.45	-581.60	-724.22
Avg	-553.19	-706.46	-602.17	-713.73
S.D.	70.09	6.82	52.70	27.79