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Effects of Sensitization on Intergranular Boundaries of Aluminum Alloy 5083

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EFFECTS OF SENSITIZATION ON INTERGRANULAR BOUNDARIES OF ALUMINUM ALLOY 5083

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Executive Summary

The 5XXX series aluminum alloy (Al-Mg) are a common material used in the transportation industry, especially within marine applications. This alloy is non-heat treatable, and exhibits good strength to weight ratio, solid solution strengthening, and cold work.¹ Despite these features, the 5XXX series aluminum alloy contain rich magnesium deposits which are susceptible to intergranular corrosion (IGC) and intergranular stress corrosion cracking (IGSCC).³ This susceptibility is due to sensitization which is the process of a material to change phases causing precipitation of a metal. This is problematic since the magnesium rich deposits force a phase shift, β -phase (Mg_2Al_3), which attacks the grain boundaries to form precipitation.⁴ This presents risks especially in marine environments where AA5083 is used in naval ship hulls.¹²

The effects of sensitization on intergranular boundaries of AA5083 can be quantified in a multitude of ways. The most common method to assess the susceptibility of the 5xxx series to IGC or IGSCC is the nitric acid mass lost test (NAMLT).¹⁰ In acid media, β -phase dissolves preferentially, leading to higher weight loss values for AA5083 with greater amounts of β -phase precipitates.¹⁰ According to ASTM G67-18, alloys are considered to be immune to intergranular attack if the degree of sensitization (DOS) is less than $15\text{mg}/\text{cm}^2$ and susceptible to IGC if greater than $25\text{mg}/\text{cm}^2$.^{3,10,11} Values in-between $15\text{mg}/\text{cm}^2$ and $25\text{mg}/\text{cm}^2$ are considered to be uncertain.¹² It is worth mentioning that reproducibility of NAMLT values tend to decrease with increasing sensitization, especially at values above $25\text{mg}/\text{cm}^2$ and could produce DOS values with a large error.¹³ Another test to help quantify the effect of sensitization is the Cyclic Potentiodynamic Polarization (CPP). This test is a direct current test which is great for determining resistance to corrosion in short times. The E_{corr} or resting potential can be used to indicate how susceptible the material is to corrosion. These values have a standard deviation demonstrating a source of error.

The pitting potential can help quantify when pitting occurs in a material. This value has a standard deviation demonstrating a source of error. Another test called Electrochemical Impedance Spectroscopy (EIS) is a powerful non-destructive technique that can study the effects on chemical and physical reactions on materials. This test is run with an alternating current (AC) where frequency is changing to measure current and potential simultaneously. The results from this test can be presented in the form of a plot called a Nyquist plot. This complex plane plot depicts impedance on the x-axis as a real part and the y-axis as the imaginary part. The impedance values can help demonstrate how resistant a material is to corrosion.¹³ These values include a standard deviation showing a source of error when testing. **Table 2** shows the values collected by the CPP and EIS tests. **Figure 1-2** shows SEM micrographs collected during tests. These micrographs can help characterize the surface of the material by scanning for pits, IGC, and precipitates. This test is subjective indicating a source of error when evaluating the results.

Definitive conclusions can be made by reviewing the results. The DOS for the 0 days sample was below 15 mg/cm² and the DOS for the 7 days sample was above 25 mg/cm². This indicates that at increased heat treatment times promotes higher β -phase precipitation, thus higher DOS values.^{2,12,13} The SEM micrographs displayed no clear grain boundaries but instead showing a small number of voids indicating dissolution of β -phase precipitates formed during the manufacturing process of the alloy. The 7 day sensitized specimen shows defined grain boundaries and pitting showing the effects from sensitization. The E_{corr} values for the AR and 7 days sample are -737 mV and -874mV respectively. This indicates that sensitization causes E_{corr} values to decrease meaning the material is more susceptible to corrosion.^{23,24} The pitting potential values for the AR and 7 days samples were -728 mV and -734 mV respectively. The decrease in pitting potential infers that as samples are more sensitized, pitting becomes easier to initiate, making the

sample more susceptible.²⁴ The impedance plot in **Figure 4** shows that higher DOS shows lower impedance, therefore lower corrosion resistance. These results are also corroborated by the CPP measurements; The sensitized sample shows a lower E_{corr} , higher i_{corr} , and lower corrosion resistance, thus the sensitized sample is much more susceptible to corrosion.

This work has strengthened the skill set for this author. This research helped strengthen my knowledge on corrosion and electrical circuits. In addition, I learned how to perform CPP, EIS, and NMLT at a high level and how to operate efficiently. Also, I learned how to polish samples effectively as well as working independent without relying on others. The biggest thing I learned is to read before you begin testing. This will not only save a lot of time but save money which is important in industry. The project itself helped identify to companies that AA5083 based items are corroding from sensitization. These skillsets will help me thrive in industry as well as make my engineering background more diverse.

The results concluded that sensitization increases aluminum alloy 5083 corrosion susceptibility by IGC, pitting from β -phase dissolution, and pitting corrosion of the aluminum alloy matrix. Future work should investigate changing the heat treatment temperatures to higher and lower degrees to see how DOS values and physical characterization differ. In addition, future work should investigate passive film thickness of the alloy to help determine its effect on sensitization. Future students working on this project or related projects should always ask questions to avoid confusion and to increase their knowledge on the subject. To enhance the experience, students should get as much lab time as possible since it will help increase their skillset and help prepare for industry work.

Introduction/Background

The 5XXX series aluminum alloy (Al-Mg) are a common material used in the transportation industry, especially within marine applications. Aluminum alloys are known to be lightweight, formable, high strength, and have high corrosion resistance.¹ This alloy is non-heat treatable, and exhibits good strength to weight ratio, solid solution strengthening, and cold work.² Since the 5XXX aluminum alloy series is formable, the produced microstructures will be nearly precipitate free. Despite these features, the 5XXX series aluminum alloys contain rich magnesium deposits (greater than 3.5 wt.% Mg) which are susceptible to intergranular corrosion (IGC) and intergranular stress corrosion cracking (IGSCC).³ This susceptibility is due sensitization which is the process of a material to change phases which causes precipitation of the metal. Sensitization occurs in the 5XXX aluminum alloys when the magnesium rich deposits force a phase shift, β -phase (Mg_2Al_3), which attacks the grain boundaries to cause precipitation.⁴ Sensitization does not occur at room temperatures, but growth of the β -phase can be found between 50 °C and 220 °C.⁵ Also, AA5083 samples sensitized for prolonged periods at higher temperatures will lead to a faster sensitization rate.⁶ Sensitization is a significant weakness for the alloy since marine environments could reach temperatures above 50 °C due to solar radiation, presenting a dangerous risk for AA5083 naval ship hulls.¹²

Aluminum alloy 5083 is worth prioritizing over other aluminum alloys due to it having the highest strength of the non-treatable alloys with an ultimate tensile strength (UTS) of 317 MPa and tensile yield strength of 228MPa.⁷ The composition of this alloy consists mostly of Al and Mg with traces of Mn and Cr. Manganese is added to aluminum alloys since the UTS increases without affecting ductility.⁸ The UTS increases since Mn forms a manganese dispersoid (Al_6Mn) which helps the matrix of alloy therefore increasing the UTS.⁸ Chromium is added to aluminum alloys to

control grain structure and prevent grain growth.⁷ In addition, chromium reduces stress corrosion susceptibility and increases toughness of the alloy.⁷

AA5083 is most commonly found in marine applications but in particular ship hulls⁹. The high corrosion resistance, damage tolerance, and strength makes this alloy attractive in these settings.⁹ Therefore, testing should be done to replicate marine environments to give accurate results for the alloy.

The most common method to assess the susceptibility of the 5XXX series to IGC or IGSCC is the nitric acid mass loss test (NAMLT).¹⁰ In acidic media, β -phase dissolves preferentially, leading to higher weight loss values for AA5083 with greater amounts of β -phase precipitates.¹⁰ According to ASTM G67-18, alloys are considered to be immune to intergranular attack if the degree of sensitization (DOS) is less than $15\text{mg}/\text{cm}^2$ and susceptible to IGC if greater than $25\text{mg}/\text{cm}^3$.^{10,11} Literature mentions that at longer testing times at a temperature of $50\text{ }^\circ\text{C}$ leads to a higher rate of β -phase precipitation at the grain boundaries.¹² Also, reproducibility of NAMLT values tends to decrease with increasing sensitization, especially at values above $25\text{ mg}/\text{cm}^2$ and could produce DOS values with a large error.¹³

Cyclic Potentiodynamic Polarization (CPP) is a direct current test which is great for determining resistance to corrosion in short times. This technique helps explain the passivity, dissociation of oxide film layer, susceptibility to repassivation, and pitting potential.¹⁴ Electrochemical Impedance Spectroscopy (EIS) is a powerful test that can study the effects on chemical and physical reactions on materials. This test is typically used in applications in corrosion studies, batteries, and fuel cells.¹⁵ EIS is a non-destructive technique meaning that the sample will not be damaged during testing. EIS is run with an alternating current (AC) where frequency is changing to measure current and potential simultaneously. The results from this test can be

presented in the form of a plot called a Nyquist plot. This complex plane plot depicts impedance on the x-axis as a real plane and on the y-axis as the imaginary part. Impedance values that are large correspond to a greater resistance to corrosion for the particular material.¹³ Experimentation requires a NaCl solution, replicating a marine environment, which causes AA5083 to precipitate aluminum (Mn, Fe, Cr) which act as permanent cathodes but due to the inhibition affect, the electrical response from the system is minimized for the cathodic intermetallics.¹⁶ In addition, the plots will shows pit initiation and dissolution of β -phase precipitates by a marked increase in the current density when anodically polarized, denoting a pitting potential, E_{pit} .² For instance, pit initiation for pure metals is generally believed to begin by the rupture of passive film of the surface and pitting resistance is dependent on the electrochemical stability of the passive film.¹⁷ Therefore, pitting occurs when the film layer of aluminum breaks which causes the matrix to dissolve.¹⁸ The cause for pitting can be linked to sensitization. This is due to the β -phase, which acts as cathodic intermetallics, where the anodic aluminum matrix dissolves granting exposure to pitting.¹⁹

Literature suggests that as sensitization exposure increases, pitting will occur more frequently on the outer layer.²⁰ When pits form, the film layer from the aluminum alloys breaks down which then causes the matrix to dissolve.¹⁸ Also, seeing deficiencies of aluminum is expected.

The aim of this report is to demonstrate the effects of sensitization on intergranular boundaries of aluminum alloy 5083 by performing NAMLT, physical characterization through SEM analysis, CPP, and EIS.

Experimental Procedures

Preparation

AA5083 specimens were cut into 40 mm × 20 mm × 3 mm. The specimens were polished using 1200 grit SiC paper. After polishing, the samples were placed into a furnace at 100 °C for 7 days. The as received (AR) sample had no heat treatment. Further details for the chemical composition of AA5083 are found in **Table 1** below.

Table 1- Chemical composition of commercial of AA5083.

Element	Mg	Si	Fe	Cu	Mn	Zn	Cr	Ti	Al
Wt.%	4.4	0.08	0.19	0.026	0.56	0.004	0.077	0.015	Bal.

NAMLT specimens were prepared and tested in accordance with ASTM G67-18 standards which provides insight on how to quantitatively measure susceptibility of intergranular corrosion of Al-Mg alloys¹⁰. The specimens were pre-treated in a 5 wt.% NaOH solution at 80 °C for 1 minute, rinsed in DI water, and air dried. The initial weight was recorded. The specimens were placed at a 45° angle in a 70 vol.% nitric acid solution for 24 hours at 30 °C. After the heat treatment, the specimens were rinsed with DI water and air dried. Mass loss was recorded, representing the DOS values obtained for each sample per ASTM G67-18 standards¹⁰.

Microscope Characterization

Each sample was polished using 1 μm diamond powder solution. A solution for etching was made based on literature described by Buczynski MS thesis at the University of Virginia²¹. Each specimen was etched for 40 minutes in the solution at 30 °C. After the chemical etch was completed, the sample was rinsed with ethanol and water, and air dried. The specimens were placed in the Hitachi TM3030 SEM for imaging and characterization.

Electrochemical Characterization

The EIS and CPP were performed in 3.5 wt.% NaCl supporting electrolyte using a three-electrode configuration; a graphite counter electrode, a saturated calomel reference electrode, and AA5083 specimens as the working electrodes. The tests were performed using a Gamry series 600 potentiostat. EIS was performed at the OCP with 10 mV r.m.s, 5 points per decade, and a range of 10^5 – 10^{-2} Hz. The CPP tests were performed with a range of -200mV to $+200\text{mV}$ at a forward and reverse scan rate of 0.1667 mV/s.

Results and Discussion

Table 2 below presents the DOS values collected for both AR and 7 day specimens after the NAMLT by ASTM standards.¹⁰

Table 2. Values for the DOS, E_{corr} , and i_{corr} calculated from the cyclic potentiodynamic polarization (CPP) and electrochemical impedance (EIS) for AA5083 at 100 °C for a particular time. Standard deviation is in parenthesis if applicable.

Sensitization Time, Days	DoS mg cm ⁻²	E_{corr} mV _{SCE}	i_{corr} A cm ⁻²
0	8	-737 (±8)	1.12×10 ⁻⁷
7	57	-874 (±12)	5.71×10 ⁻⁷

At higher sensitization times, β -phase will precipitate in the grain boundaries and β -phase dissolution occurs, which corresponds to higher weight loss measurements.² According to the ASTM standard, alloys that have DOS values below 15 mg/cm² are considered to be immune to IGC, while values above 25 mg/cm² are susceptible to intergranular attack.¹⁰ Values between 15 mg/cm² and 25 mg/cm² are considered to be uncertain.¹² According to the DOS measured, the AR sample is immune to IGC since the DOS value is 8 mg/cm². However, the 7 day sensitized specimen is vulnerable to IGC since the DOS value is 57 mg/cm². These results conclude that increased heat treatment time promotes higher β -phase precipitation, thus the DOS values increase, which is supported by previous studies.^{2,12,13}

Figures 1-2 below show SEM micrographs of the AR and the 7 days sensitized specimen.

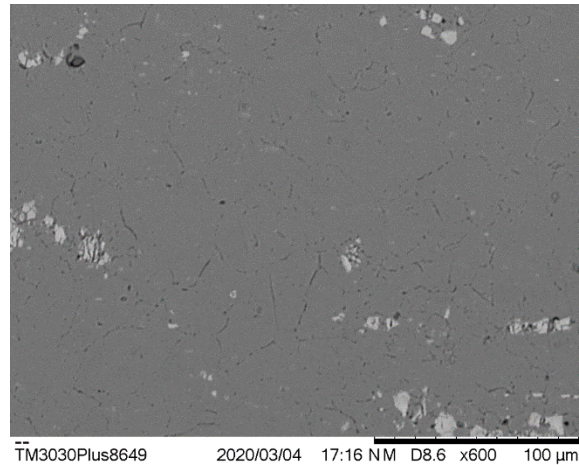


Figure 1. SEM image of the as received sample at 15kV using $\times 600$ magnification.

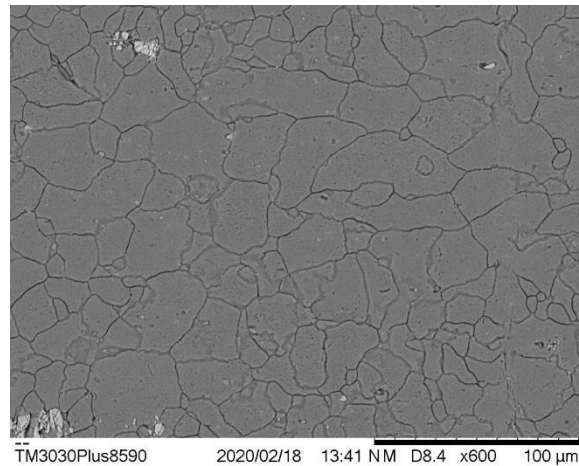


Figure 2. SEM image of the 7 days sample taken at 15 kV with magnification of $\times 600$.

The AR specimen in **Figure 1** displays no clear grain boundaries, instead showing a small number of voids indicating dissolution of β -phase precipitates formed during the manufacturing process of the alloy. However, the 7 day sensitized specimen in **Figure 2** shows defined grain boundaries which is due to β -phase dissolution.⁴ In addition, some pitting within the grains can be seen, this is caused by the development of intragranular β -phase precipitates due to the high DOS value.²² This pitting in the grain boundaries is due to β -phase development within the grain after the

saturation of the grain boundaries.⁵ The white precipitates on the specimens are known to be manganese oxide deposits (MnO_x).¹ These deposits form in Mn rich areas which oxidize when in contact with O_2 .

Figure 3 below shows the Cyclic Potentiodynamic Polarization (CPP) for the AR and 7 day sensitized specimens.

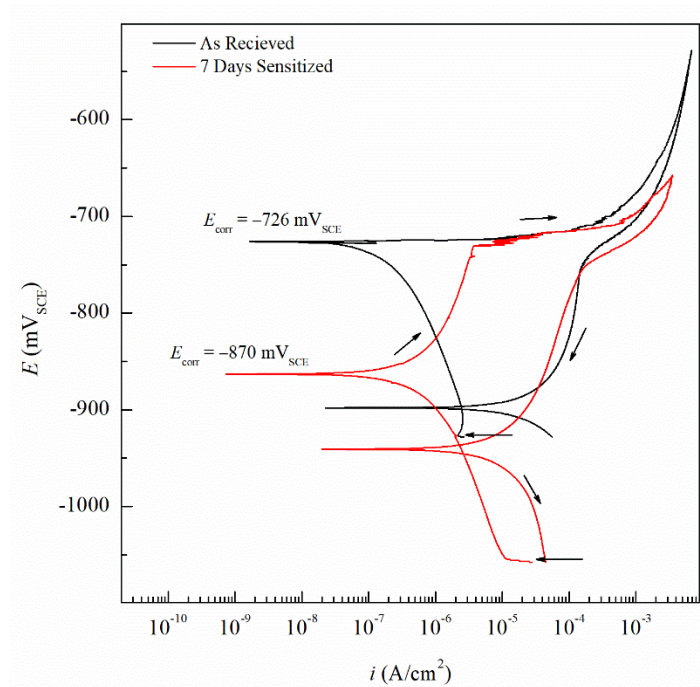


Figure 3. The CPP test results for the AR and 7 days sensitized specimens showing potential (E) in mV, and current (i) in A/cm^2 .

Table 2 summarizes the E_{corr} and i_{corr} values obtained by the CPP. Arrows display the direction of the current scan on the plot. The anodic branch is above the OCP and the cathodic branch is below. The E_{corr} , or resting potential, is the native potential of a metal from which the corrosion susceptibility can be qualitatively evaluated. The E_{corr} for the AR and 7 days samples were

measured to be -737 mV and -874 mV, respectively. The results demonstrate that sensitization causes E_{corr} values to decrease, thus indicating that the material is more susceptible to corrosion.^{23,24} The anodic forward sweep showed a pitting potential for both the AR and sensitized specimens. This potential is denoted by the large increase in current density due to the dissolution of aluminum matrix, thus causing pits to form.²³ The pitting potential values for the AR and 7 days specimens were very similar, -728 mV and -734 mV respectively. The small decrease in pitting potential can be explained by the greater amount of β -phase precipitates, thus providing a larger number of cathodic sites to cause anodic dissolution in the matrix.²⁵ This infers that as samples are more sensitized, pitting becomes easier to initiate, making the sample more susceptible.²⁴

Figure 4 below shows the Nyquist plot for the AR and 7 days samples.

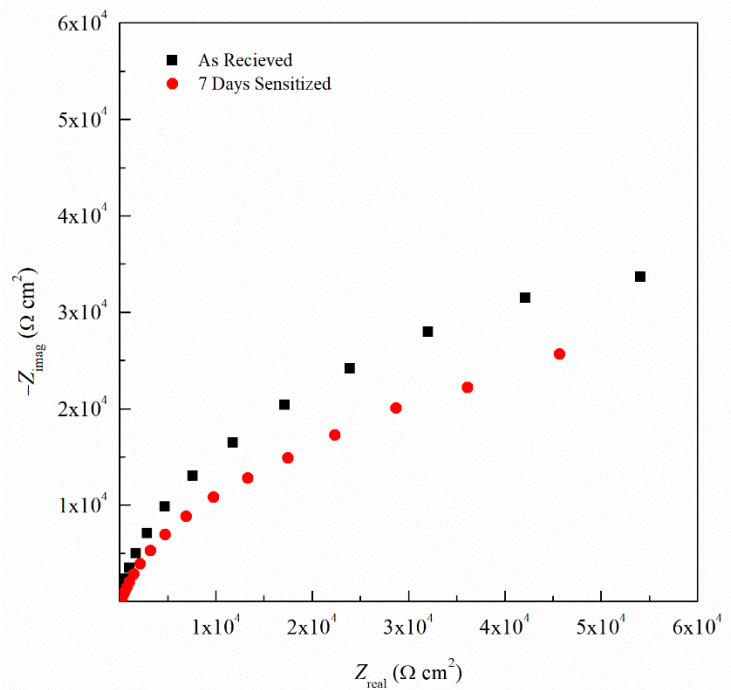


Figure 4- The Nyquist plot for the AR and 7 days sensitized specimens comparing the imaginary Z axis in Ωcm^2 to Z real in Ωcm^2 .

Results show that higher DOS shows lower impedance, therefore lower corrosion resistance. This is corroborated by Bazi and Mansfeld, who notes that lower impedance leads to lower resistance as the amount of sensitization increases.^{14,26} These results are also corroborated by the CPP measurements; The sensitized sample shows a lower E_{corr} , higher i_{corr} , and lower corrosion resistance, thus the sensitized sample is much more susceptible to corrosion.

Conclusions

This paper discussed the effects of sensitization on intergranular boundaries on aluminum 5083 by using physical and electrochemical characterization. The DOS was above 25 mg/cm² for the 7 days specimens, and below 15 mg/cm² for the 0 days sample. The SEM images displayed no clear grain boundaries for the AR sample, but the 7 days sample saw formation on grain boundaries due to IGC from β -phase dissolution. In addition, the 7 days specimens began seeing pitting due to development of intragranular β -phase precipitates. The CPP plot concluded that as DOS increased, E_{corr} decreased. The CPP plot concluded that sensitization influenced the pitting potential and resting potential. Sensitization showed an impedance decrease, therefore lower corrosion resistance for the sensitized sample. With this knowledge, sensitization played a major factor in pitting and IGC.

The results concluded that sensitization increases aluminum alloy 5083 corrosion susceptibility by IGC, pitting from β -phase dissolution, and pitting corrosion of the aluminum alloy matrix. Future work should investigate changing the heat treatment temperatures to higher and lower degrees to see how DOS values and physical characterization differ. In addition, future work should investigate passive film thickness of the alloy to help determine its effect on sensitization.

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