EVALUATION OF CORROSION RESISTANCE OF 6101 ALUMINUM ALLOY AFTER DIFFERENT PRECIPITATION HEAT TREATMENTS

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Abstract: Currently, 6xxx series aluminum alloys have been occupying an important place in the global production of aluminum alloy. Recently, more than 80% of the alloys used in the worldwide production of extruded profiles are made of the Al-Mg-Si. The wide use of this class of alloys is a result of improvement on mechanical properties when subjected to heat treatments. Furthermore, characteristics such as low density, good thermal and electrical conductivity, high reflectivity to light and heat, and corrosion resistance are key factors for the wide applicability of these alloys. The corrosion resistance of alloys in the Al-Mg-Si, is due to the formation of a compact thin film of alumina oxide (Al_2O_3) which grows naturally on the surface of these alloys under normal atmospheric conditions, protecting the entire surface of material. However, the thermal treatments of solubilization and precipitation of metastable phases during aging may impair the corrosion resistance of this class of alloys. Moreover, the presence of certain corrosive agents, such as chloride and sulfate, can also affect the kinetics of corrosion process. Therefore, the present study aims to evaluate the corrosion behavior of aluminum alloy 6101 subjected to solubilization heat treatment at temperatures of 450° C, 520° C, 550° C, 580° C and 520° C aged for 5 minutes at 170° C. It was investigated by corrosion tests of weight loss, potential vs time and anodic polarization curves. The the formation of pits was evaluated by scanning electron microscopy(SEM) and optical microscopy. Solutions of 3,5% NaCl and 0,1M Na₂SO₄ were carried out in order to simulate conditions of high aggressiveness.

Keywords: Aluminum Alloys 1, Pitting corrosion 2, heat treatment of solubilization 3

1. INTRODUCTION

The extensive use of aluminum alloys is associated, among other factors, with the excellent combination of low specific weight and good mechanical strength, which in some cases the mechanical resistance are similar to structural steel. This feature is very important for many industrial applications, such as: ships, planes, cars, etc. (Smith, 1992).

The 6xxx series alloys are highlighted and most used aluminum alloys. their mechanical strength and hardness increased considerably when subjected to heat treatment of solubilization, natural and artificial aging. According to Ezuber et al. (2007), corrosion resistance is an important characteristics of this group of alloys because of the presence of an adherent film of alumina (Al_2O_3) and develops naturally on the alloy surface under normal atmospheric conditions. In that case, this film protects the entire structure of certain corrosive agents.

However, Vera et al. (2006) and Svenningsen et al. (2005) argue that the presence of specific pollutants, particularly SO_2 and CI, can affect the corrosion kinetics because of the dissolution of the passive film. Therefore, the attacks of localized corrosive can cause loss of mechanical strength and fail of the material.

2. **REVIEW OF LITERATURE**

Aluminum alloys represent an important class of materials as a result of their high technological value and wide range of industrial applications, especially in naval, aerospace, automotive and domestic. (Badawy et al.1998). The low densities coupled with some features like: good electrical and thermal conductivity, high reflectivity to light and heat and high corrosion resistance. These factors explain the large scale of production these alloys.

2.1. Solubilization Heat Treatment

Aluminum alloys 6xxx series have been occupying an important place in global production of aluminum alloys. Recently more than 80% of the alloys used in the worldwide production of extruded profiles are part of the Al-Mg-Si. (Usta et al. 2004). According Marioara et al. (2006) the widespread use of these alloys is due to improvement of its mechanical properties when subjected to heat treatments.

The solubilization heat treatment (TT) in aluminum alloys 6000 series, is characterized by heating the material and maintaining the elevated temperature for a given time, providing a complete dissolution of secondary phases initially present in the alloy. (InfoMet, 2010b). Smith (1992) argues that the system of precipitation hardening in this class of

alloys is possible due to the decrease in solubility of the solid intermetallic compounds, according to the temperature decreases.

The precipitates formed after the TT alloys in the system Al-Mg-Si, can have different characteristics according to the temperature of TT performed. Karabay (2007) says that the coherent precipitates and semi-coherent with the matrix promote small distortion in the crystal structure and are associated with hardening of the alloy. However, the incoherent precipitates do not have strong connection with the crystal structure and does not hinder the movement of dislocations and is thus related to the softening of the alloy.

Recent studies have been made and consider that the behavior of precipitation in alloys of Al-Mg-Si obeys the following scheme: (Karabay et. al., 2007) and (Gaber et. al., 2009):

Supersaturated solid solution (SSSS) => Clusters => GP Zone => B'' (coherent precipitates) => B'(semicoherent precipitates).

Moreover, Karabay (2007) argues that the most effective hardening phase for these kinds of materials is characterized by precipitates fine B" which hinders the movement of dislocations. Although studies to define the maximum hardness obtained is given by a good combination of GP zone and B" phase (Gaber et al., 2009).

2.2 Corrosion Resistance in Aluminum Alloy in the Al-Mg-Si System

Another important characteristic of aluminum alloys 6xxx series is their good corrosion resistance caused by the phenomenon of passivation. However, the stability of this film can be affected by thermal treatments and the presence of certain corrosive agents that could influence the corrosion kinetics. (Svenningsen et al. 2005).

The passivation of aluminum alloys is a complex and controversial process. Some authors consider that the passivation is caused by an oxide film present on the metal surface. On the other hand, other authors concluded that an oxidized state is responsible for preventing the contact between the alloy and the corrosive atmosphere (Badawy, 1998, Gentil, 2007 and Szklarska, 1998). Recent studies consider that the corrosion resistance of aluminum alloys is associated with the presence of a compact thin film of alumina oxide (Al₂O₃), which develops naturally on the surface of alloys under normal atmospheric conditions. (Ezuber et al., 2005), (Vera et al., 2006) and (Infomet, 2010).

The corrosion protection provided by this film varies, among others, according to the alloy composition, microstructure and corrosive medium. Changing any of these factors the corrosion properties will be directly affected, such as morphology and the rate of corrosion.

According to (Szklarska, 1998), the pitting formation in aluminum alloys is associated with a division into specific points of the oxide film, when the alloy is exposed to environments containing aggressive ions, for example chloride ions that is most common. The intensity of pitting corrosion attack is influenced by the concentration of halogen, temperature, fluid velocity and pH. Generally, the pitting formation in aluminum alloys occurs in the presence of an electrolyte with pH between 4.5 and 8.5 and tends to increase with increasing temperature and concentration of aggressive ions.

Unlike pitting corrosion, the intergranular corrosion (IGC) is characterized by occur in tunnels "subsurface" around grain boundaries, visible only in metallographic analysis. This type of corrosion is very common in aluminum alloys 6xxx series with excessive content of silicon due to the strong character of cathodic constituent of insoluble silicon. (Infomet, 2010).

Studies stated that the IGC occurs when the metal corrodes preferentially to the grain boundaries, which act as an anode while the interior of the grain as a cathode and forming a galvanic cell. The region of the grain boundary becomes anode due to the presence of a phase that covers the boundary or due to precipitation of phases in the contours. (Infomet, 2010). Svenningsen et al. (2005) believes that one of the leading factors for the occurrence of IGC is the formation of precipitates with different corrosion potential in the matrix grain boundaries, which generates a galvanic couple and promotes corrosion.

The occurrence of the IGC is associated with mechanisms that increase the probability of precipitation at grain boundaries. The rapid cooling after the solubilization heat-treatment reduces the precipitation at grain boundaries. However, higher cooling rates are not desirable because they can introduce residual stresses in the material. The susceptibility to IGC decreases when occurs the coarsening of particles on grain boundaries and the matrix, causing a reduction at the differences on the corrosion potential and breaking the continuity of the galvanic couple formed. (Svenningsen et al. 2005).

Another factor that contributes for changing on conditions of crevice corrosion is a decrease on the free corrosion potencial in relation to the free corrosion potential of metal surface. The difference of potential between the gap and the surface can reach 270mV on aluminum alloys in solutions of 0.5 M NaCl. This corrosion deterioration case in the crack is caused predominantly by a contact with the free surface. (Sinyavskii and Kalinin, 2004).

The corrosive media in which the metal is used is crucial in the corrosion resistance. Among the natural environments where aluminum alloys can be widely used, the sea water is more corrosive electrolyte. The mineral

composition of sea water contains a complex composition of sea salts, they stand out among the NaCl and $MgCl_2$ for its importance in the corrosion process. (Sinyavskii and Kalinin, 2004).

Gentil (2007), considers that the effect of sodium chloride on the corrosion occurs due to this salt is a strong electrolyte, causing an increase of conductivity which is fundamental in the mechanism of electrochemical corrosion. Ezuber et al. (2007), states that when the aluminum alloy is exposed in media containing halide ions, the oxide film breaks at specific points, leading to formation of small cavities (pits) on the surface of aluminum.

3. EXPERIMENTAL METHOD

The experimental procedures were carried out with aluminum alloy 6101 provided by LSM Brazil, which has the following chemical composition:

Table 1. Chemical analysis of aluminum alloy 6101									
	%Mg	%Si	%Fe	Cu	Mn	Cr	В	O.E.*	O.T.**
aluminum assotiation	0,35 a 0,8	0,30 a 0,7	0,50 máx	0,10 máx	0,03 máx	0,03 máx	0,06 máx	0,03 máx	0,10 máx
Liga 6101	0,57	0,51	0,11	0,018	< 0,01	0,004	< 0,003		
	*O.E. = Each Other: ** O.T. =Other Total								

The following experimental procedures were carried out in order to evaluate the corrosion behavior of aluminum alloy 6101 thermally treated at different temperatures:

3.1 Heat Treatment

The test samples used were taken from the rod of 9.5 mm diameter of alloy 6101, and submitted to solubilization treatment at following temperatures of 450°C, 480°C, 520°C, 550°C, 580°C. Moreover, the sample TT at temperature of 520°C, was performed an artificial aging for 5 minutes at a temperature of 170°C. They stayed for 30 minutes in a muffle furnace after reaching the desired temperature, and then were cooled in water at room temperature. The temperature variation obtained was about 10°C. This procedure was repeated every 10 days for a period of five months to obtain samples at various times of natural aging.

Samples	S19	S20	S21	S22	S23	S24
Temperatures of solubilization	450°C	480°C	520°C	550°C	580°C	520°C + 5min a 170°C

Table 2. Solubilization temperatures of samples

3.2 Vickers Microhardness (HV)

In order to infer the kinds of precipitates formed after the heat treatment in 6101 aluminum alloy, the Vickers microhardness was performed.

Metallographic specimens were prepared referring to the solubilization temperatures. The Vickers microhardness was measured according to ASTM E384-89, an each interval of 10 days for approximately 5 months. It was used a Mitutoyo MVK-G1 microhardness with a load of 0.5 kg and 55x lens. It was evaluated the behavior of the increase in hardness related to the solubilization temperature and the time of natural and artificial aging.

The mechanical behavior is just an initial parameter for evaluating the type of precipitate formed. The microscopic analysis are being undertaken to determine the precise kinds of precipitates formed.

3.3 Scanning Eletron Microscopy (SEM)

The visualization of the precipitates formed was made using scanning electron microscopy (SEM). For analysis in the SEM, the specimens were prepared metallographic samples and then they were attacked by 10 to 15 seconds on a 0.5% solution of HF (198ml water + 2 ml HF 48%).

Views occurred in samples solubilized at 480°C and 580°C with 4.7 months and 1 day of natural aging. The choice of temperatures was based on the results of mechanical tests.

The micro-structural characterization of all samples using SEM and optical microscopy is underway.

3.4 Corrosion Tests

The solutions used in the tests are presented on table 3:

Table 3. Chemical composition of solutions used in tests.

3,5% de NaCl pH 6,81	0,1 M Na ₂ SO ₄ pH 6,99
965mL of distilled water	1000 mL of distilled water
35g de NaCl	14,204 g de Na ₂ SO ₄

3.4.1 Polarization curves

It was made six specimens, one for each temperature of solubilization, all with exposed area of 70.88 mm². They were welded to a wire and insulated with enamel and resin on the spot weld to prevent contact of the wire with the solution. The polarization curves were performed by AUTOLAB PGSTAT101 using the method of linear voltammetry in a conventional electrochemical cell with three electrodes. The scan rate was 50 mV/min.

3.4.2 Potential vs Time

The specimens were immersed in solutions of 3.5% NaCl and 0.1 M Na2SO4. The corrosion potentials were measured three times per day for 1 month, using a reference electrode saturated calomel and a digital multimeter.

3.4.3 Microscopic analysis of the pits formation

Visualization of the pits formation was done using an optical microscopy. The test samples were polished in order to obtain good images of the surface of aluminum alloy 6101. This procedure was carried out before and after of the polarization tests.

4. **RESULTS AND DISCUSSION**

4.1 Vickers Microhardness

The objective was to infer the type of precipitates formed, after completion of the solubilization heat treatment in 601 aluminum alloy, using the Vickers microhardness and comparing the results of the samples.



Figura 1. Chart Hardness versus solubilization temperatures. A) Samples with 1 day of natural aging. B)Samples with 4,7 months of natural aging.

Already the samples S21, S22, S23 and S24 have higher levels of hardness, when compared to other samples, and presents evolution in microhardness over time. This response indicates that the samples were completely solubilized at these temperatures and may have the thin precipitated, semi-coherent or coherent with the aluminum matrix (Infomet, 2010).

Sample S23 showed the highest level of hardness between samples, and according to the images obtained in scanning electron microscopy (Fig. 3) can be inferred that the TT at this temperature promotes the precipitation of fine coherent phases as stated in literature Karabay (2007).

Figures 2 and 3 show scanning electron microscopy performed at temperatures of 480°C and 580°C respectively.



Figura 2. Scanning Electron Microscopy, samples solubilized at 480°C. A) 1 day of natural aging . B) 4,7 months of natural aging



Figura 3. Scanning Electron Microscopy, samples solubilized at 580°C. A) 1 day of natural aging . B) 4,7 months of natural aging

4.3 Measurements of corrosion potential (Ecorr) over time

Figures 1 and 2 show the evaluation of the open circuit potential versus time of the 6101 alloy with different temperature of heat treatment (Table 2) in solutions of 0,1 M Na₂SO₄ and 3,5% of NaCl respectively. Table 4 shows the values of corrosion potentials after 300 hours of immersion.



Figure 4- Open circuit potential versus time in the solution of 0.1 M Na₂SO₄.



Figure 5 . Open circuit potential versus time in the solution of 3,5% NaCl.

E (mV) – SCE							
Solutions	Samples						
Solutions	S19	S20	S21	S22	S23	S24	
0,1 M Na ₂ SO ₄	-648	-775	-545	-684	-830	-782	
3,5% NaCl	-885	-876	-869	-922	-829	-768	

Table 4. Open circuit Potential versus Time.

The corrosion potential (Ecorr) of all alloys in both solutions began in values ranging from -100 mV to - 600 mV (SCE). The potential stabilization process occurred only after an average of 800 hours of immersion, stabilizing the values described in table 4. The Ecorr had a cathodic tendency. In that case, the decrease on the potential probably occurs because of the passive film formation on the metal surface.

The corrosion potentials of alloys immersed in 3.5% NaCl were more negative than the solution of 0.1 M Na₂SO₄. This probably occurred due to reactions occurred with different solutions.

According to the electrochemical equilibrium diagram for the system Al/H_2O at 25°C (Pourbaix, 1976), the corrosion potentials of all alloys in both solutions were stabilized within the domain of stability of passivation with formation of Al_2O_3 and below the equilibrium line of H/H^+ . In this case, two thermodynamically spontaneous reactions occurred on the metal surface, such as the passivation of aluminum to oxide and hydrogen reduction.

The following shows the polarization curves of 6101 Al alloys in both media, where it is proven the passivation process on the metal surface.

4.2 Potentiostatic polarizations Curves

Figures 3 to 5 show the anodic polarization curves of Al alloys with different heat treatments, respectively obtained in solutions of 0,1 M Na_2SO_4 and 3,5% of NaCl. The tests were carried out in naturally aerated condition. Tables 5 and 6 show the pitting potential of each alloy respectively obtained in the solutions of 0,1 M of Na_2SO_4 and 3,5% of NaCl.





Figure 3. Anodic polarization curves of Al alloys with different heat treatments in 0.1 M Na₂SO₄ solution, naturally aerated.



Figura 4. Anodic polarization curves of Al alloys with different heat treatments in 3,5% NaCl solution, naturally aerated.

The differences in the scales of figures 4 and 5 is a consequence of the current density observed in tests.
AA 6101 Alloy- Sample \$20
AA 6101 Alloy- Sample \$23



Figure 5. Anodic polarization curves of samples S20 e S23 of 6101 aluminum alloy in solution of 3,5% NaCl, naturally aerated.

The pitting potential was defined when there was a significant increase in current density .

Sample	рН	E pitting (V) (ECS)	I cor in Pot. pitting(A)
S19	6,99	-0,252	2,27E-06
S20	6,99	-0,244	6,36E-06
S21	6,99	-0,355	2,42E-06
S22	6,99	-0,168	2,07E-06
S23	6,99	-0,143	1,54E-06
S24	6,99	-0,288	8,59E-06

Table 5. Results of polarization curves in solution of 0,1 M Na₂SO₄.

Table 6. Results of polarization curves in solution of 3,5% NaCl.

Sample	рН	E pitting (V) (ECS)	I cor. in Pot. pitting(A)
S19	6,81	-0,612	5,32E-03
S20	6,81	-0,362	8,74E-05
S21	6,81	-0,586	3,84E-03
S22	6,81	-0,578	6,23E-03

S23	6,81	-0,029	3,22E-06
S24	6,81	-0,581	6,92E-03

All alloys showed passivation process in both solutions tested, passivation process were observed in the range of 1150 to 1350mV polarization for the solution of 0.1 M Na_2SO_4 , and from 800 to 1470mV for the solution of 3.5 % NaCl. Moreover, the corrosion potential did not vary from one alloy to another, in the two tested solutions.

Based on the electrochemical equilibrium diagram for the system at 25°C Al/H₂O, all alloys in both solutions had the corrosion potential, in natural aerated conditions, which were within the domain of passivation and below the equilibrium line of H/H^+ . In this case, the reaction of anodic passivation Al/Al₂O₃H₂O and hydrogen reduction was thermodynamically spontaneous. Thus, within the passivation domain, the Al alloys suffered combined effect of passive film formation of Al/Al₂O₃H₂O with concentrations of 10 g * L⁻¹ and hydrogen reduction on the surface of alloys.

On tables 5 and 6 presents the pitting potential (E_{pit}). In this case, note that the NaCl solution was more aggressive than 0.1 M Na₂SO₄ solution. It occurred because of the pitting potential was lower for the solution of 3.5% NaCl solution than 0.1 M Na₂SO₄. For this reason, it can be inferred that the Cl⁻ ions in the solution of 3.5% NaCl favored the process of breaking the passive film and initiation of the pitting corrosion.

Ezuber *et. al.* (Ezuber *et. al.*, 2007) reported that the oxide film formed on the aluminum surface is thin and not uniform, when exposed to aggressive environment, such as Cl⁻, this film breaks at specific points causing the pitting corrosion. Moreover, the author reported that the pitting corrosion is influenced by several factors, including the formation of second phase particles as a result of heat treatment.

As already presented in this work, the TT promotes the precipitation of metastable phases in the aluminum matrix. According to the solubilization temperature, the precipitate may be fine and coherent with the matrix or coarse and incoherent.

Guillaumin et. al. (2000) in his work proved that the coarse intermetallic precipitates are highly reactive in solution of NaCl. In addition, other authors Blanc et al. (1998) also showed that coarse intermetallic particles in aluminum alloy system Al-Mg-Si, presents active dissolution when in the middle of sulfate. To Svenningsen et. al. (2005), the reactivity of the coarse precipitates is a result of the corrosion potential difference between these precipitates and the matrix of aluminum, and consequently the formation of micro galvanic pairs that increase susceptibility to corrosion.

The pitting corrosion in aluminum alloys of the system Al-Mg-Si heat treated is a consequence of the preferably dissolution of coarse precipitates. The preferential corrosion of these particles causes the precipitation zones become fragile promote the occurrence of pitting. (Guillaumin et. al 2000).

At first the pits develop within the grains leading to the formation of corrosion products that are responsible for maintaining the environment within the acid pit. In this aggressive environment, the pits grow through microscopic tunnels and when they penetrate the grain boundaries promote intergranular corrosion. In addition, Guillaumin (2000) proved experimentally that the regions without the presence of precipitates no showed pitting and intergranular corrosion (IGC).

In the solution of 3.5% NaCl was observed that alloys S20 and S23 showed the best resistance to pitting corrosion than other alloys. In this case, they showed highest pitting potential and lowest current density at the pitting potential. These alloys have a similar effect of alloys tested in 0.1 M Na_2SO_4 solution Tab.5 and 6. The other alloys when tested in NaCl showed pitting potential ranging from -578mV to -612mV, and the S24 alloy showed the highest current density and can be considered the most susceptible to pitting corrosion in NaCl solution.

The alloys showed higher resistance to pitting corrosion in 0.1 M Na_2SO_4 solution. This probably occurred because this solution does not have Cl⁻ ions, which favors the conductivity and breakdown of passive film. The pitting potential varied from -355 mV to -143mV, being highest for S23 alloy and lowest for the S21 alloy.

The alloy S23 showed the highest resistance to pitting corrosion of all alloys in both solutions. This is a consequence of complete solubilization and precipitation of fine coherent phases, which decrease the effect of micro galvanic couple created between the precipitates and Al matrix, as observed in mechanical behavior Fig.1 and SEM Fig. 2.

The alloys S19 and S21 had the lowest resistance to pitting corrosion in NaCl and Na2SO4 solutions respectively, ie had the highest current densities and the lowest pitting potential (Epit). This behavior is a consequence of precipitation of coarse phases which increases the susceptibility to pitting corrosion, according to Guillaumin *et. al* (Guillaumin *et. al*.,2000). Moreover, the largest current density presented by the sample S24 Tab.6 is also indicative of the presence of coarse precipitates.

Thus, the variation in pitting potential Tab.5 and Tab.6, ie the corrosion resistance of each alloy in the same solution results from the kind of heat treatment that each alloy was submitted. In this case, the TT process is directly influenced by the temperature of solubilization, which affects the kinetics of precipitation and consequently the size of the precipitates, which in turn interfere decisively in corrosion resistance of aluminum alloy 6XXX series.

After polarization testing the specimens, tested in 3.5% NaCl solution were evaluated in the optical electronic microscope to view the pits. In this case, the Figure 6 shows the pits observed in each alloy. Observed that the largest

pitting occurred in the sample S24 also had a higher current density at the pitting potential Tab.6. The alloy S23 showed only small pits confirming the higher resistance to corrosion verified in the polarization tests.



Figure 6. Optical microscope before and after polarization tests in the solution of 3.5% NaCl, samples: A) S19, B) S20, C) S21, D) S22, E) S23, F) S24.

5. CONCLUSION

• The corrosion potential had a cathode tendency. The decrease on the corrosion potential occurred probably by the formation of passive film on the metal surface.

• The corrosion potentials of all alloys in both solutions were stabilized within the domain of stability of passivation with stability of the oxide $Al_2O_3H_2O$ and below the equilibrium line H/H⁺. Therefore, two

thermodynamically spontaneous reactions occurred on the metal surface, the passivation of aluminum in oxide, and hydrogen reduction.

• All alloys showed passivation process in both solutions tested, the passivation process were observed in the range of 1150 to 1350mV polarization for the solution of 0.1 M Na2SO4, and from 800 to 1470mV for the solution of 3.5 % NaCl.

• The NaCl solution was more aggressive than $0.1 \text{ M} \text{ Na}_2\text{SO}_4$ solution, since the pitting potential was lower for the solution of 3.5% NaCl solution than $0.1 \text{ M} \text{ Na}_2\text{SO}_4$. As a result, it can be inferred that the Cl⁻ ions in the solution of 3.5% NaCl favored the process of breaking the passive film and initiation of pitting corrosion.

• The alloy S23 showed the highest resistance to pitting corrosion of all alloys in both solutions. This is a consequence of complete solubilization and precipitation of fine and coherent phases, which decrease the effect of micro galvanic couple created between the precipitate and the matrix of aluminum. However, to confirm this hypothesis are being carried out analysis of optical microscopy, scanning electron microscopy and X-ray diffraction in order to detect these phases and precipitates.

• The variation in pitting potential in the same solution results from the type of heat treatment that each alloy was submitted. In this case, the TT process is directly influenced by the temperature of solubilization, which affects the kinetics of precipitation and consequently the size of the precipitates, which in turn interfere decisively in corrosion resistance of aluminum alloy 6XXX series.

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