SOLUBILIZATION TEMPERATURE INFLUENCE ON THE MECHANICALS PROPERTIES OF AGED AL-SI-MG 6101 ALLOY

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Abstract: This study investigates the mechanical behavior of aluminum alloy 6101 according to the solubility temperature and natural aging. The heat treatments of solubility were performed in temperatures of: 450°C°, 480 ° C, 520 ° C, 550 ° C, 580 ° C and 520 ° C aged for 5 minutes at 170 ° C. The micro-hardness Vickers was performed at intervals of 10 days at the temperature of solubilization determined. Tensile tests were made on samples with variations of aging times for its natural temperature of solubilization. The images of the precipitates were obtained using scanning electron microscopy (SEM). It was observed a relationship between increasing the solubility temperature and corresponding hardness. It was also observed a relationship between increased mechanical strength with increasing temperature and time of natural aging. This response on the mechanical properties is justified based on the phase coherent precipitation in the aluminum matrix.

Palavras- Chave: *aluminum alloy, solubilization of heat-treatment, aging of heat-treatment.*

1. INTRODUCTION

The extensive use of aluminum alloys is related to the excellent combination of low specific weight and good mechanical strength, which in some cases may reach the structural steel. This characteristic is very important for industrial applications in ships, planes, automobiles and power transmission networks (Smith, 1992).

According to Marioara *et al* (2006), the 6xxx series alloys are among the most commonly used aluminum alloys in the world, mainly due to present a significant gain in hardness and strength when subjected to the heat treatment defined as solubilization, always followed by aging (natural or artificial).

Smith (1992) argues the property of increasing the hardness and strength is due to precipitation of different types of metastable phases in the aluminum matrix. This precipitation is due to the decrease of solid solubility of other phases in the aluminum matrix when the temperature decreases. This is the main mechanism of precipitation hardening alloys in the system Al-Mg-Si.

This study evaluates the influence of the temperature of solubilization heat treatment on precipitation behavior and consequently on the mechanical properties of aluminum alloy 6101. The analysis of mechanical properties was made from performig Vickers hardness and tensile tests. The images of the precipitates were obtained using scanning electron microscopy (SEM).

2. EXPERIMENTAL PROCEDURES

The experimental procedures were performed on 6101 aluminum alloy provided by LSM Brazil, which has the chemical composition shown on Table 1.

Table 1. Chemical analysis of aluminum alloy 6101

	%Mg	%Si	%Fe	%Cu	%Mn	%Cr	%B	%E.O*	%T.O**
6101 Alloy	0,57	0,51	0,11	0,018	< 0,01	0,004	< 0,003	--	--

*E.O = Each Others ; ** T.O = Total Others

This alloy presented the following percentage in weight:
Mg_2Si = 0,90% and Si livre = 0,13%
Both calculated.

2.1. Heat Treatment

The test samples used were taken from the rod of a 9.5 mm diameter of 6101 aluminum alloy, and submitted to solubilization heat treatment at specifics temperatures of 450°C, 480°C, 520°C, 550°C, 580°C. Furthermore, the sample solubilized at 520°C, was submitted to artificial aging for 5 minutes at 170°C. They were hold for 30 minutes in a muffle furnace after reaching the desired temperature, and then 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. Also, were conducted precipitation heat treatments on samples naturally aged for 4.7 months. The precipitation heat Treatment involves heating the metal at 170°C and maintain this temperature for a period of 2 hours.

2.2. Metallographic Preparation

The samples were cut from cross-sectional and longitudinal rod. After deep drawing, the samples were sanded on abrasive papers number 200, 400 and 600. The polishing step was performed in a suspension of colloidal silica for 7 minutes. Subsequently the samples were washed in water and dried in hot air.

2.3. Vickers Microhardness

Metallographic samples were prepared for each temperature of solubilization. The Vickers micro-hardness was measured according to ASTM E384-89, every interval of 10 days for about 5 months. A Mitutoyo MVK-G1 microhardness under a load of 0.5 kg and 55x lens was used for the microhardness testing. The behavior of hardness increasing was related to the solubility temperature and aging time.

2.4. Tensile Tests

The tensile tests were done in a universal testing machine, using a load cell of 2.5 Ton. The samples were removed from the rods heat-treated at different temperatures and with different aging times. The dimensions of test specimens were 300 mm long, 9.5 mm in diameter and length is 250 mm. The tests were performed for all temperatures of solubilization and at certain discrete aging time intervals to determine the influence of aging on the mechanical properties of the alloy.

2.5. Scanning electron microscopy (SEM)

To perform the test, the samples were prepared by metallographic routine and then attacked by 10 to 15 seconds on a 0.5% solution of HF (198ml water + 2 ml HF 48%).

The scanning electron microscopy (SEM) was performed on all samples heat treated. Images were done after heat treatment of solubilization, after a period of 4.7 months of natural aging and after the artificial precipitation.

3. RESULTS AND DISCUSSION

3.1. SEM Analisys

The Figure 4 shows the several dark precipitates in the aluminum matrix for samples solubilized at temperatures of 450°C and 480°C. After calculating the percentage of Mg2Si and using the equilibrium diagram (Fig. 2), was observed that temperatures of 450°C and 480°C are slightly below the solvus line for the system Al-Mg2Si, ie, the samples were not completely solubilized to their temperatures. Thus, it can infer that the precipitates dark cited are Mg2Si particles. According to Karabay *et. al.* (2007), these phases are characterized by being coarse and incoherent with the aluminum matrix.

In addition, the samples solubilized at temperatures of 450°C and 480°C did not present any significant increase in the number of precipitates after 4.7 months of natural aging and after artificial aging. According to Andersen et. al (1998), this behavior is a consequence of the presence of B phase of equilibrium system, which is characterized by presenting mainly Mg2Si particles.

The sample heat treated at temperature of 520°C shows little incoherent precipitates with the matrix after the completion of the heat tretment. Similarly, samples solubilized at lower temperatures, the coarse phases still remain after natural aging and artificial aging.

As a consequence of complete solubilization, the other temperatures do not present the precipitates coarse as shown in Fig 4. There is evidence of, presented at the mechanical behavior of the samples, some presence of fine precipitates and coherent with the aluminum matrix. However, analysis of X-ray diffraction and EDS (energy dispersive spectroscopy) of the SEM will be performed to verify the presence of these phases in a next investigation.

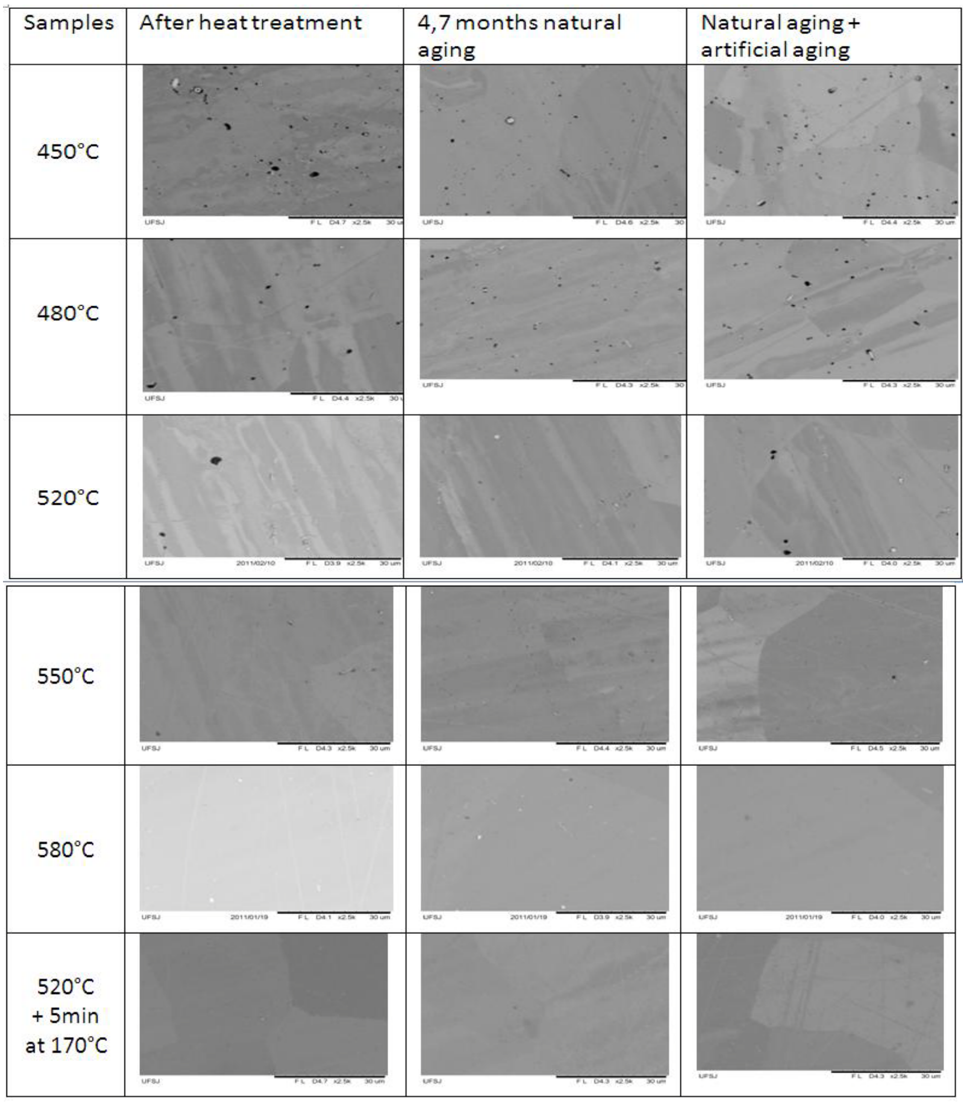


Figura 4. SEM micrograph of the 6101 Al alloy subjected the different heat treatment (5Kv X2500)

3.2 Vickers Micro-hardness

The Vickers microhardness testing has shown a trend of increasing in hardness with aging time. The sample solubilized at a temperature of 450°C presented the lowest value of hardness and there was no considerable increase in hardness over time. This result was not surprising, considering this temperature beeing very close or even below the solvus line as shown in Fig. 2. Thus, it is possible to infer that this alloy composition was not completely solubilized at this temperature.

The Figure 4 shows the results for Vickers microhardness related to the natural aging time for samples solubilized at different temperatures.

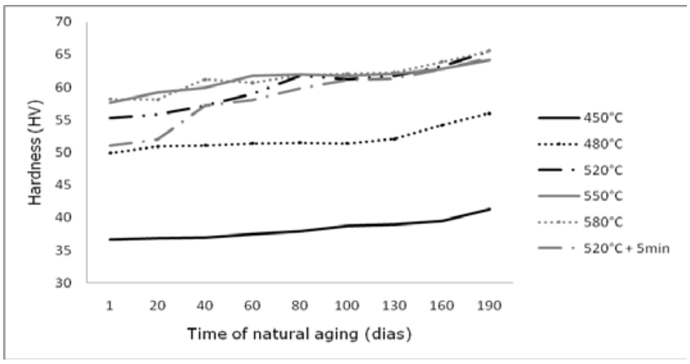
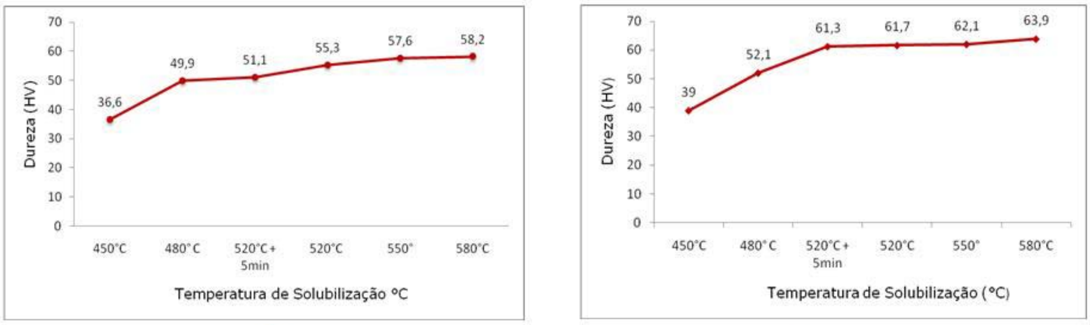


Figure 4. Chart Hardness versus Time of natural aging for samples solubilized at different temperatures.

The sample heat-treated at 480°C also has shown lower hardness than the other samples, however there was a small increase of hardness with longer time as is shown in Tab.4. This increase in hardness is due to the greater proximity to the line that the solvus temperature of than the sample TT at 450°C. According to Infomet (2010), above the solvus line the Mg2Si particles are dissolved in the aluminum matrix, and after the rapid cooling appears the fine precipitates responsible for hardening.

Figures 5 (A) and (B) show the relationship between hardness and temperature of dissolution for the samples after heat treatment and samples naturally aged for 4.7 months:



A

B

Figure 5. Hardness versus Temperature of solubilization .

A) Samples with 1 day of natural aging. B) Samples with 4,7 months of natural aging.

The sample solubilized at 580°C showed the highest hardness among the other samples, specially for the alloy after heat treatment and aged naturally for 4.7 months. Moreover, this behavior was similar to samples solubilized at temperatures of 520°C +5 min, 520°C and 550°C. This response of the hardness is a consequence of the solubilization to be completed at the temperatures above. The appearence of the coherent and semi-coherent precipitated with the aluminum matrix has been reported before by Andersen *et. al* (1998) and Marioara *et. al* (2003). Furthermore, Karabay (2007) considers that the most effective hardening phase is B", which is characterized by the presence of precipitates fine and coherent with the aluminum matrix.

The EDS analysis in SEM will be made, and may confirm the presence of precipitates fine and coherent with the aluminum matrix.

The sample solubilized at a temperature of 520°C + 5 min aging at 170°C showed the greatest increase in hardness. This behavior is the result of artificial aging at temperature for 5 minutes at 170°C.

Table 4 presents the hardness values for samples after heat treatment and 4.7 months of natural aging. Moreover, the hardness increments is presented for all the temperatures studied.

Table 4. Hardness increments for the solubilization temperatures studied

Temperature of solubilization (°C)	**Hardness (HV) after the heat-treatment of solubilization**	**Hardness (HV) 4,7 months of natural aging**	**Percentage Increase of hardness(%)**
450°C	36,6 HV	39	6,5
480°C	49,9 HV	52,1	4,4
520°C + 5min 170°C	51,1 HV	61,3	19,9
520°C	55,3 HV	61,7	11,5
550°C	57,6 HV	62,1	7,8
580°C	58,2 HV	63,9	9,8

3.3 Tensile Tests

The Table 5 presents the results for tensile tests performed on 6101 aluminum alloy naturally aged for 4.7 months. It also presented the results for samples aged naturally and artificially precipitated at temperature of 170 ° C for 2 hours.

Table 5. Results of tensile tests conducted on 6101 aluminum alloy subjected to heat treatment.

	Yield Strenght (Mpa)		Tensile Strenght (Mpa)		Elongation (%)	
	Natural aging	Aging + Precipitate for 2hours a 170°C	Natural aging	Aging + Precipitate for 2hours a 170°C	Natural aging	Aging + Precipitate for 2hours a 170°C
450°C	77	78	139	134	13,8	9,6
480°C	94	127	178	197	16,2	11,6
520°C + 5min a 170°C	117	178	221	258	21,0	16,0
520°C	112	144	212	232	21,0	19,8
550°C	112	151	212	241	21,0	16,4
580°C	112	146	211	233	23,6	18,6

The table 6 shows the percentage increase for the mechanical properties presented in Table 5, when the samples are subjected to artificial aging.

Table 6.Porcentage increase tensile tests

	Porcentage Increase		
	Yield Strength (Mpa)	Tensile Strenght (Mpa)	Elongation (%)
450°C	1,29	-3,59	-30,43
480°C	35,12	10,67	-28,39
520°C + 5min a 170°c	52,14	16,74	-23,81
520°C	28,57	9,43	-5,71
550°C	34,82	13,68	-21,90
580°C	30,38	10,42	-21,18

The sample solubilized at temperature of 450 ° C did not show any significant increase in yield strength and the limit of tensile strength when subjected to artificial aging. This result is similar to the behavior of the hardness for this temperature and can also be considered as another indication that the sample was not solubilized for this temperature.

The sample heat treated at 480°C presented some increase in yield strength and tensile strength. This behavior was similar with that showed at the microhardness in Fig.5.

The best result obtained so far for the yield strength and Tensile strength, as observed in Tab.6, was achieved by the sample solubilized at 520°C + 170°C 5min, which obtained 52.1% increase in yield strength and 16.7% in the tensile limit. Thus, it is important to emphasize the effectiveness of pre-aging performed, it can be a viable alternative to achieve the mechanical response in a faster time.

The samples solubilized at temperatures of 520°C, 550°C and 580°C showed considerable increases in yield strength and tensile strength, especially for the sample heat treated at a temperature of 550 ° C, which showed 34.8% increase in yield strength and 13.7% in the limit of tensile strength. This behavior is very interesting, in view of the possibility of increasing the mechanical resistance beyond the levels obtained by nature aging.

The largest reductions in elongation were observed for the samples solubilized at temperatures of 450 ° C and 480 °. The smallest reduction in elongation was observed for the sample solubilized at a temperature of 520 ° C.

Further analysis consisting of X-Ray Diffarction and EDS will be made in a next step of investigation to identify the precipitates present after the completion of heat treatment in all samples, and thus being able to explain, based on the aging theory, the mechanical responses observed (Marioara *et al*.,2006), (Liu *et al*. 1996), (Edwards *et. al* 1998), (Karabay,2007), (Fukui and Takeda,1998), (Zhen and Kang, 1998) and (Gaber *et al*., 2009) .

4. CONCLUSIONS

- The samples solubilized at temperatures of 450°C and 480°C showed the lowest hardness and there was no considerable increase in hardness over aging time. The presence of coarse precipitates in the SEM images and the mechanical behavior suggested the alloy was not completely solubilized at the respective temperatures.

- The presence of coarse precipitates in the samples solubilized at 450°C, 480°C and 520°C, even after the treatment of natural aging and artificial, associated with the mechanical behavior indicates the presence of phase Mg2Si.

- The samples solubilized at temperatures of 520°C, 520°C + 5 min, 550°C and 580°C showed the highest values of hardness and hardness increments over time. It was not observed the presence of coarse phases in the SEM images, which indicate the complete solubilization for the respective temperatures. However, analysis of X-ray diffraction and EDS will be held in a next investigation in order to determine accurately the presence of fine and coherent phases with the aluminum matrix.

- The largest increase in hardness over time was observed for the sample solubilizated at temperature of 520 ° C + 5 min aging at 170 ° C. Moreover, the best result for the yield strength and tensile strength limit has been reached by this sample. These results are consequences of the artificial aging for 5 minutes at temperature of 170 ° C. Thus, the pre-aging of 5 min. performed, can be a viable alternative to achieve the mechanical response in shorter time

- The samples solubilized at temperatures of 520°C, 550°C and 580°C showed significant increases in yield strength and tensile strength, especially for the sample solubilized at a temperature of 550°C, which showed of 34.8% increased in the yield strength and 13.7% in the limit of tensile strength. This behavior is very interesting, in view of the possibility of increasing the mechanical resistance beyond the levels obtained by artificial aging.

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