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CHARACTERIZATION OF SECOND PHASE PARTICLES IN AN ALUMINUM ALLOY AA7050 FORMED AFTER AGEING TREATMENT

S. C. Jacumasso[1]
A. L. M. Carvalho[2]
L. O. R. Lima[3]
Universidade Estadual de Ponta Grossa - UEPG - Avenida General Carlos Cavalcanti, 4748 - Uvaranas, Ponta Grossa - PR, 84030-900, Brasil.
sheijacumasso@gmail.com[1], andrelmc@uepg.br[2], luisorlima@hotmail.com[3]

J. P. Martins
Universidade Tecnológica Federal do Paraná - UTFPR - Campus Ponta Grossa - Avenida Monteiro Lobato, s/n, km 04 - Jardim Carvalho, Ponta Grossa - PR 84016-210, Brasil.
julianadepaulamartins@hotmail.com

Abstract. *The 7XXX series alloys, AA7050 class, based on the Al-Zn-Mg-Cu, are widely used in aerospace industries, airframe structures, mobile equipment and components under high stress loading due to its high ratio of resistence mechanical and density, in addition to its corrosion resistance. The properties of these alloys are obtained after processing involving heat treatment and ageing of solubilization, precipitation by means of a thin phase and dispersed, coming from its own chemical composition. In this context, the present work has as main objective the realization of different aging treatments with subsequent characterization of precipitated phases. The aim is mainly to establish the conditions of phase precipitation eta line (η'), because it is responsible for increasing the mechanical properties of these alloys without significant loss of elongation. The techniques used for characterization were precipitated by Optical Microscopy (OM), Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM).*
Keywords: *aluminum alloy AA7050-T7451, precipitation hardening, artificial ageing, phase η'.*

1. INTRODUCTION

The characteristics of aluminum allow it to have a diverse range of applications. Material lightweight, durable, aluminum shows an excellent performance and superior properties in most applications. [1]

The main function of aluminum alloys is to increase strength without affecting other properties.

The 7XXX series alloys, the basic Al-Zn-Mg-Cu, is widely used in the aerospace industry, particularly alloy AA7050-T7451, increased resistance in these alloys is by precipitation of a fine dispersed phase, from of the composition itself, in a process called aging heat treatment where this increase is strongly dependent on the alloy composition, size, morphology and distribution of the phases present in the microstructure. The particles η and η 'are the main stages involved in 7XXX series alloys, and phase η' is mainly responsible for the increased resistance. [2]

When the Zn content is higher than the Mg precipitation sequence tends to the formation of stable phase $MgZn_2$ (η). On the other hand, if the Mg content exceeds the content of Zn sequence will lead to the formation of $Mg_3Zn_3Al_2$ phase (T). If the Cu content is higher than the Mg precipitation tends to result in formation of Al_2CuMg phase (S). [3]

In addition to Zn, Mg and Cu are found in aluminum alloy additives Cr, Mn, Fe, Si, Zr and even. [3]

The AA7050 alloy contains Zr in the form of Al_3Zr dispersoids forming hard particles which increase the strength of the alloy. The Fe and Si are considered impurities, forming intermetallic phases with Al and other alloying elements. [4]

Greater resistance is achieved in alloys that respond to heat treatment solubilization / aging. First, the metal is heated uniformly to 500°C, with the exact temperature depends on the particular alloy. This causes the dissolution of alloying elements in solution (solubilization). Then follows a rapid cooling (quenching), usually in water, which temporarily prevents these constituents precipitate. This condition is unstable and gradually constituents precipitate so extremely thin, thus achieved the maximum effect of hardening (aging). In some alloys it occurs spontaneously after a few days (natural aging), while in other occur by reheating for a few hours (artificial aging). [5-6]

The main types of second phase particles that occurs in aluminum alloy 7XXX series are intermetallic dispersoids and precipitates. [5]

Intermetallic occur from the interaction of impurities such as Fe and Si with alloying elements, have irregular shape, size between 1 and 20µm, non-uniform distribution. [5]

Dispersoids are formed during homogenization because the reaction of Cr, Zr and Mn with the alloying elements have size between 10nm and 2µm, non-uniform distribution. In the AA7050 alloy composition has dispersóide Al_3Zr.

Particles of precipitates are formed during aging of aluminum alloy 7XXX series, their size varies between 1nm and 100nm. Three different types of particles are observed: GPs zones, metastable precipitates (η'), stable precipitates (η). [5]

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The sequence of precipitation of particles precipitated in 7XXX series alloys is as follows:

α-Supersaturated solid solution (α -sss) → Guinier–Preston zones (GP zones) → metastable phase η' → equilibrium phase η ($MgZn_2$). [5]

The nuclear zones GPs can be homogeneously or heterogeneously in the matrix gaps and disagreements. The metastable precipitates (η') are formed directly by transforming areas GPs or by heterogeneous nucleation. The precipitates stable (η) are formed by the transformation of metastable precipitates or by heterogeneous nucleation on dislocations, grain boundaries or interfaces between matrix and dispersoids. [5]

Aging temperature, terms of nucleation and aging time are responsible for variation in the final formation of the precipitates in the alloy. The completion of aging heat treatments can be carried out in several stages at different temperatures. [6]

The method most commonly used alloys aging Al-Zn-Mg-Cu is aging in two steps, namely the double aging, usually the first aging is performed at 110 ° C and the second aging at 175°C (condition T7451), the heat treatment has a deficit of about 15% in the mechanical properties compared to those obtained by the aging treatment in one step at 130 ° C (condition T6), but results in a better combination of strength, corrosion resistance and fracture toughness. According to the literature (condition T 614-65) when the secondary aging is carried out at 65 ° C, ie at a temperature below the primary aging at 130°C is an improvement with respect to tensile properties, fracture toughness and ductility. The primary aging at 130ºC aims to promote the formation of GP zones, and secondary aging 65°C is the formation of particles η 'from zones GPs. [7]

It aims of this study were to characterize the second phase particles size, morphology, composition, chemical and crystallographic condition to obtain a heat treatment condition T7451 alternative currently in use in the aerospace industry, with minimal losses of properties tensile.

2. EXPERIMENTAL PROCEDURE

The development of the experimental work was carried out with a block of aluminum AA7050, with T7451 temper, provided by Empresa Brasileira de Aeronáutica - Embraer SA, from which samples were taken to perform the proposed work. The chemical composition of the alloy is: 6.06 Zn, 1.90 Mg, 2.19 Cu, 0.15 Zr, 0.10 Mn, 0.14 Fe, 0.04 Cr, 0.12 Si (in wt.%) and Al bal.

Conditions, treatments and ageing solubilization performed in this work are presented in Table 1:

Table 1. Description of ageing heat treatments

Ageing Heat Treatment	*Solution Treatment*	*First Step*	*Second Step*
T7451	10ºC/min to 485ºC	110ºC for 2h	110ºC for 2h
T6	485ºC for 4h	Ageing at 130ºC	-
T614-65	485ºC for 4h	130ºC for 30 min	Ageing at 65ºC

The Vickers hardness tests were conducted following ASTM E 92 -82, in a durometer of the type LEICA VMHT MOT with a load of 1 kgf and penetration time of 12 seconds.

The tests were performed on DSC equipment Shimatzu brand, model DSC-60 with scan between 40 and 400 ° C, an increase of 10 º C / min and a minute's residence at 400ºC.

The samples selected for microstructural characterization are described in Table 2:

Table 2. Description of samples groups for microstructural characterization

Specimens Group	*First step*	*Second step*
T7451	As received	As received
T6	130ºC for 2h	-
T614-65	130ºC for 30min	65ºC for 24h

For the metallographic characterization, small samples were cut and abraded with SiC abrasive paper in particle sizes 240, 400, 600, 800, 1200 and 1500, polished with alumina 1 and 0.3 µm in a semiautomatic polishing machine (AROTEC Ind. Com. , Brazil) and etched with Keller reagent, the attack time was around 30 seconds. The micrographs were recorded by optical microscopy (OM, Olympus BX51) and scanning electron microscopy (SEM, Shimadzu SSX-550). The atomic force microscopy analysis was performed in a Shimadzu SPM-9600 microscope with 400 nm minimum resolution, equipped with a 125 µm scanner operating in non-contact mode.

For each test were used to characterize two samples for each condition.

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3. RESULTS AND DISCUSSION

3.1 Age hardening

The hardness values, which were recorded for the various samples are shown in Figure 1.

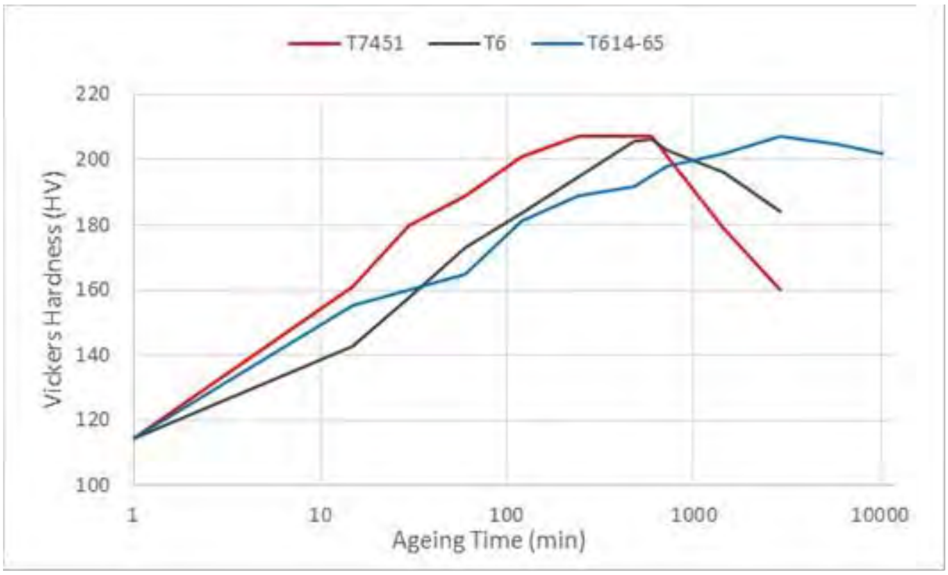

Figure 1. Hardness evolution of sample T6, T614-65 and T7451 tempers with increasing treatment time.

It is possible to observe a variation in hardness in relation to the treatment time, which indicates the change in the sample microstructure and mechanical properties. In all conditions is observed hardness increase over time to a maximum of 200 HV. The curve shows that primary heat treatment at high temperature resistance increase is accelerated, but the loss of properties occurs in more definite.

3.2 Differential Scanning Calorimetry (DSC)

Figure 2 shows the DSC curves obtained from samples of table 2. The curve shows exothermic peaks in the intense condition T614-65 and T6 condition, but less intense, to the condition T7451 curve shows no peaks. The curve shows three peaks related microstructural changes in the alloy AA7050 during heating. Next at a temperature of 165°C is possible to observe the formation of a small peak, indicating that the exothermic reaction corresponding to the phase formation η' between the temperatures of 200 to 230°C another peak is observed concerning the transformation η' → η, morphology plates, this reaction confirms the presence of the η phase. The peak at 250°C refers to the same processing, but the final morphology of the precipitates η is referred to as irregular blocks.[7] The presence of these peaks indicates that the samples with T6 and T614-65 tempers had particles of metastable η' particles meantime T7451 samples doesn't shows these peaks, indicating that all η' has been transformed into η or the residual η' are not detected by the equipament.

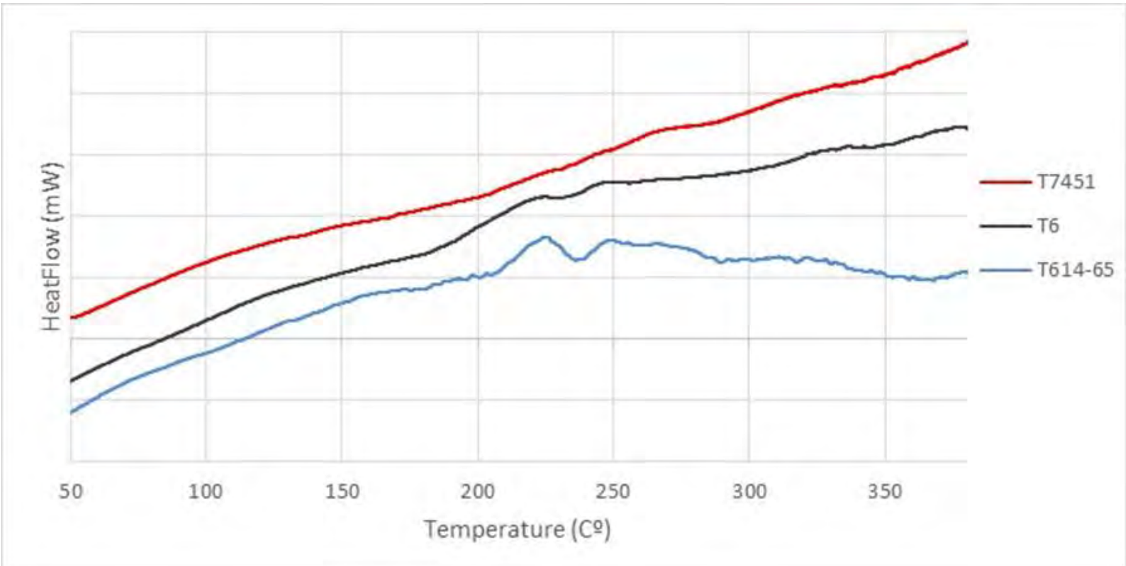

Figure 2. DSC scans for T6, T614-65 and T7451 tempers sample.

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3.3 Microstructural Characterization

Figure 3 shows the microstructures observed in the optical microscope in condition T7451, T6 and T614-65.

It can be seen in both figures a, b and c that there is no visible change in microstructure optical microscope to justify changing properties. The heat treatment process applied not change the morphology of the grains of the samples.

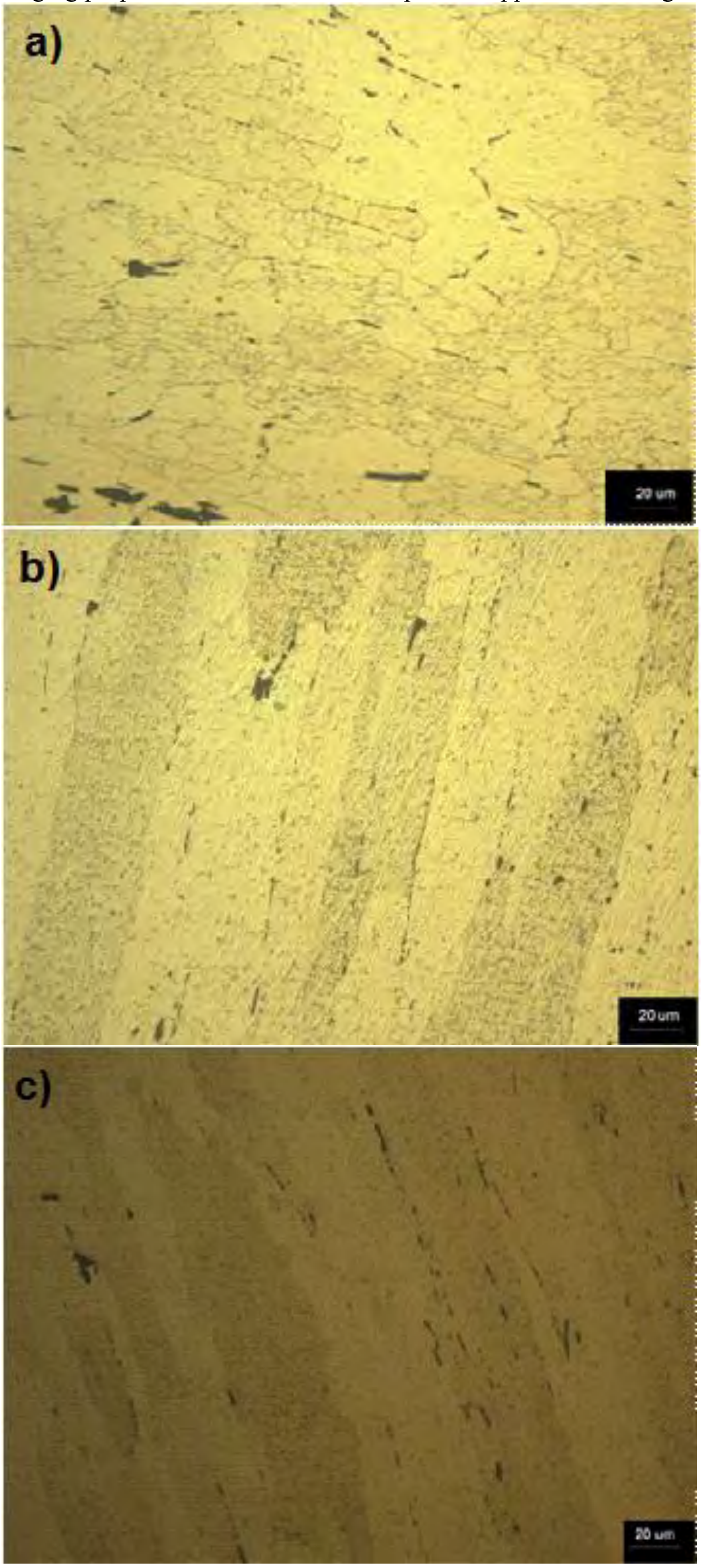

Figure 3. Microstructure of samples with temper: a) T7451, b) T6 and c) T614-65. Etching: Keller reagent

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3.4 Morphology Analysis by Scanning Electron Microscopy

SEM micrographs of the as received condition (T7451) are presented in Figure 4 (a and b) shows some dark spots, which are the holes left by uprooted intermetallic compounds during metallographic preparation of the material. The light regions, in turn, represent intermetallic constituent particles that were not removed during the preparation stage of the material.

Note that these particles have size ranging between 0,5 and 10μm and are preferably distributed in the grain boundaries, which are high-energy regions, such particles may be Al_7Cu_2Fe constituents $(Al,Cu)_6(Fe,Cu)$, Mg_2Si, Al_6Fe, $Al_{12}(Fe, Mn)_3Si$, Al_2CuMg and $Al_{40}Cu_{15}Mg_{25}Zn_{20}$ or the dispersóide Al_3Zr, in the case of smaller particles having a size of up to 2μm.

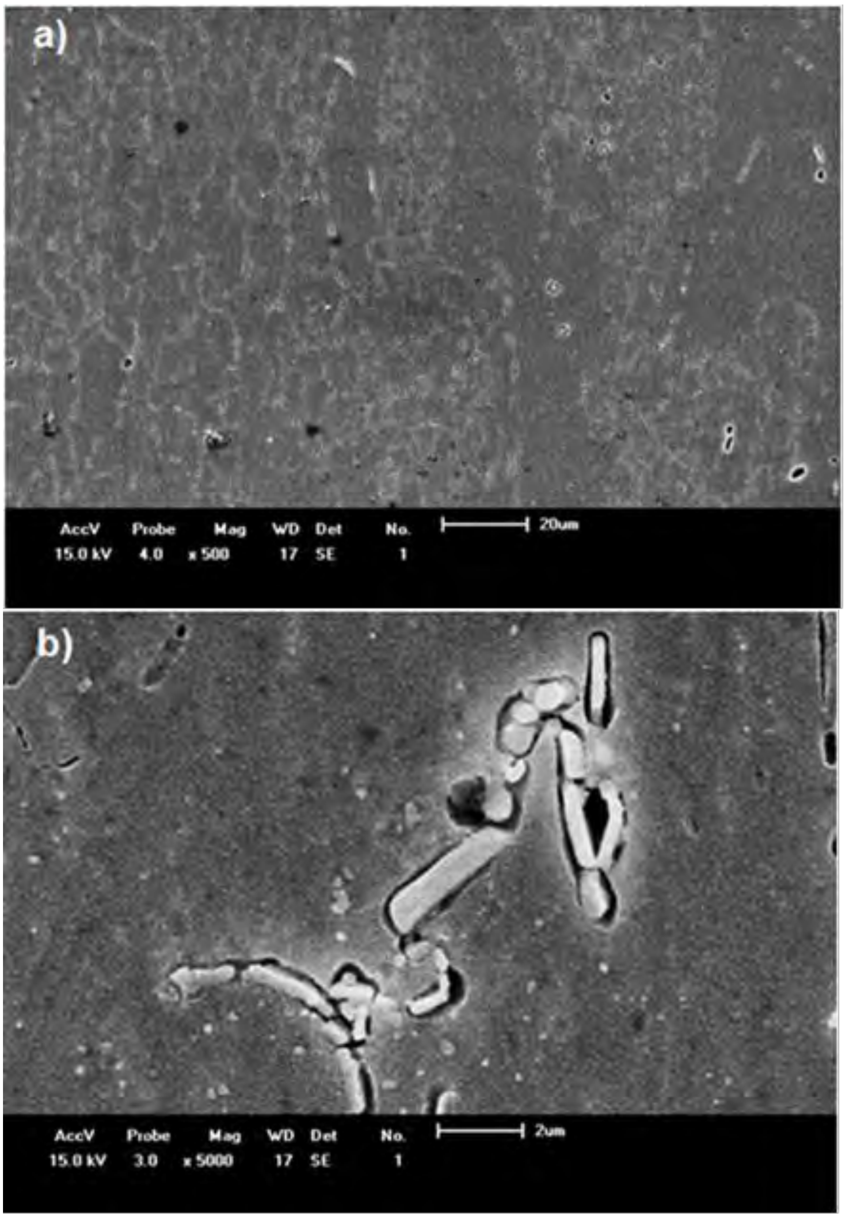

Figure 4. Scanning Electron Microscopy micrographs sample with T7451 temper: (a) Image from secondary electrons mag. x500, (b) Image from secondary electrons mag. x5000. Etching: Keller reagent.

SEM micrographs of the T6 condition are presented in Figure 5 (a and b). It is observed in Fig 5.a the size difference between the fine precipitates probably η' homogeneously dispersed in the matrix dark background, and second phase particles, white spots with rounded morphology and shaped slats in Fig. 5.b is possible to observe particle intermetallic constituent rich in Cu and Mg, confirmed by EDS analysis.

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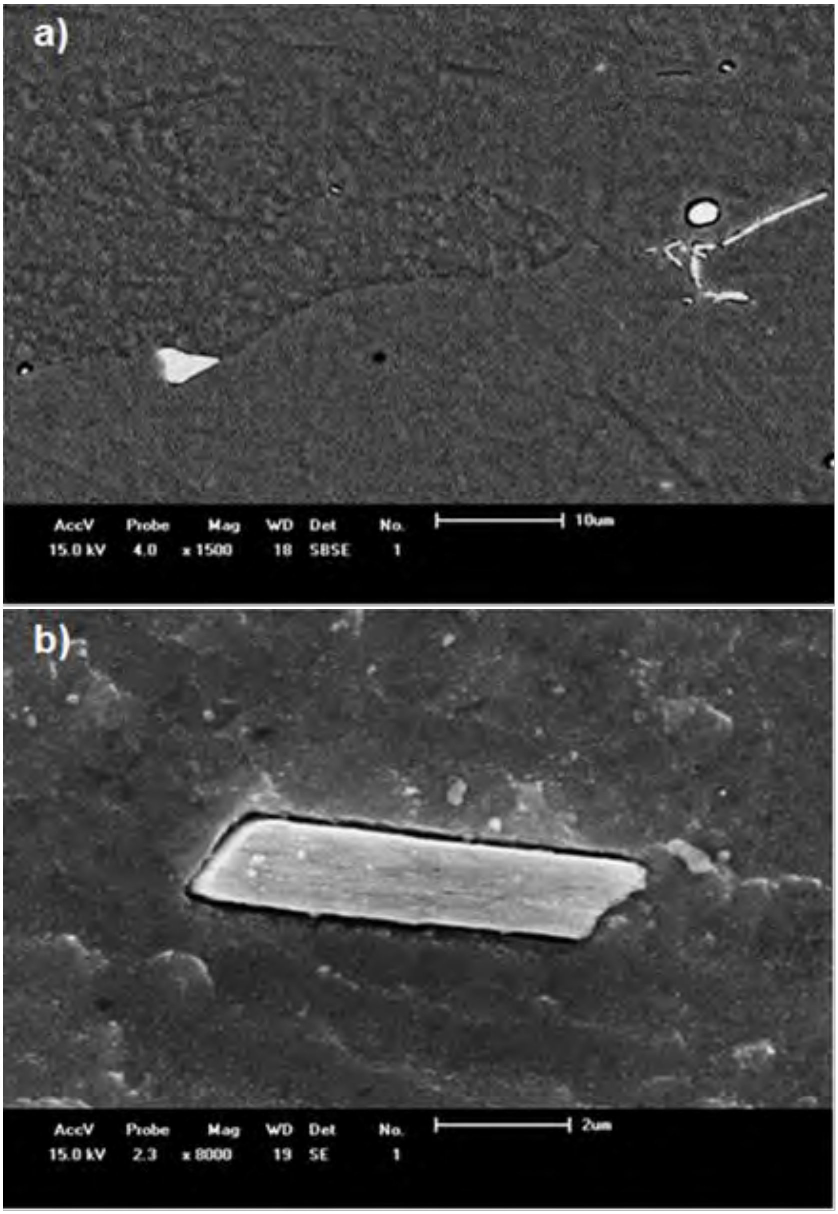

Figure 5. Scanning Electron Microscopy micrographs sample with T6 temper: (a) Image from backscattered electrons mag. x500, (b) Image from secondary electrons mag. x5000. Etching: Keller reagent.

SEM micrographs of the condition T614-65 is presented in Figure 6 (a and b). It is noted that the image 6.a is composed of white spots on a dark background, corresponding to α aluminum matrix. These white patches are composed of clusters of particles composed of chemical elements that have atomic weight higher than average dark background. Thus, these clusters of particles, which are homogeneously dispersed in the matrix, are probably phase particles η' which precipitated during the aging treatment. Fig. 6b corresponds to a particle of intermetallic constituent rich in Cu and Zn, confirmed by EDS analysis.

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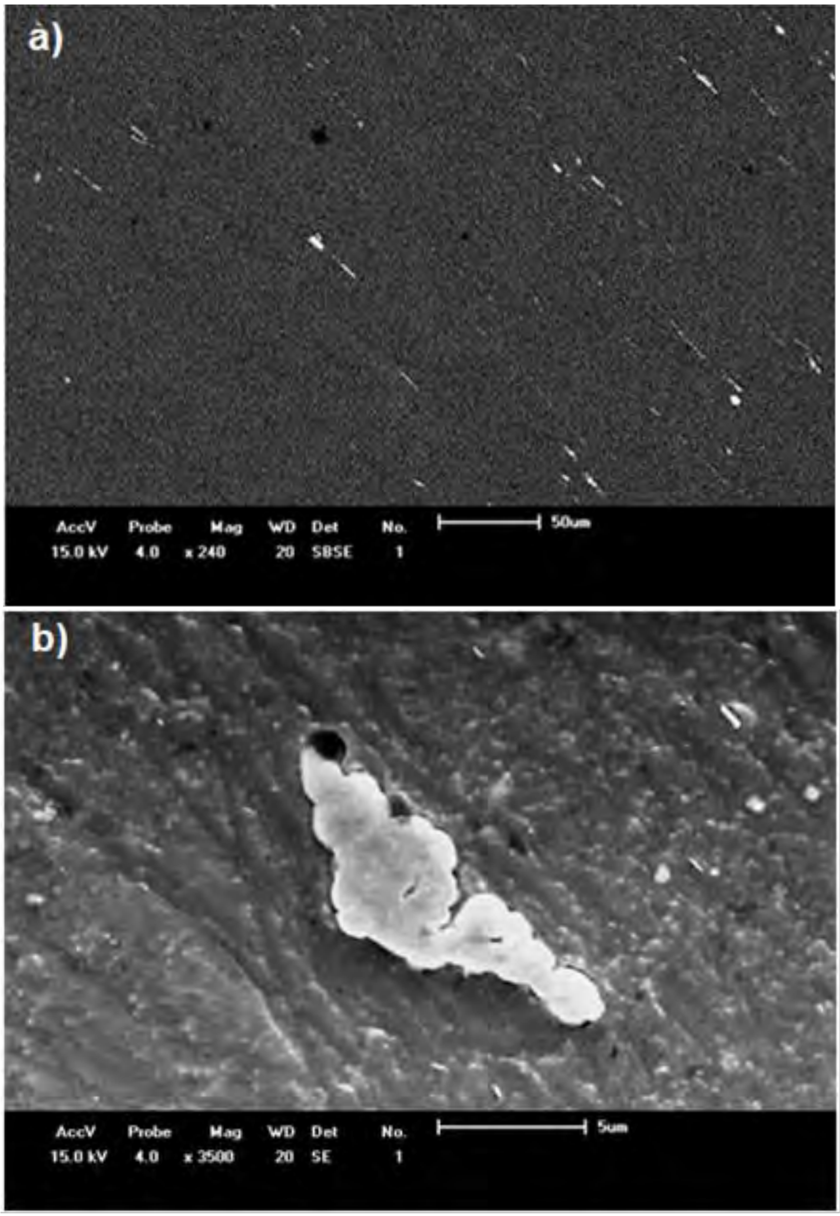

Figure 6. Scanning Electron Microscopy micrographs sample with T6 temper: (a) Image from backscattered electrons mag. x500, (b) Image from secondary electrons mag. x5000. Etching: Keller reagent.

3.4 Morphology Analysis by Atomic Force Microscopy

AFM micrographs conditions T7451, T6, T614 and 65 are shown in Figures 7 (a and b), 8 (a and b) and 9 (a and b) respectively.

Figures 7.a, 8.a and 9.a show the AFM topographic image of three-dimensional (3D), it is possible to observe the roughness of the surface morphology indicates that this area is not uniform, revealing the presence of peaks and valleys, the condition T614-65 has a rougher surface compared to the T6 condition and T7451, it shows that the particles hardening η' are better distributed throughout the aluminum matrix.

The topography 2D illustrated in figures 7.b, 8.b and 9.b, shows the presence of possible clusters of second phase particles in the lighter area of the image, which according to the literature may be responsible for hardening the alloy. In Figure 8b, topographic image (2D), it is possible to observe the presence of "holes" which shows the presence of second phase particles, where the same by being more rigid than the matrix was possibly torn when the material was prepared during preparation for metallographic analysis.

The particles observed were measured and the results are summarized in Table 3.

Table 3. Average particle size by AFM

Temper	**T7451**	**T6**	**T614-65**
Width (µm)	0,14 – 2,00	0,42 – 2,15	0,32 – 0,91

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According to the literature (ANDREATTA, 2004) correspond to the values found intermetallic having sizes between 1 and 20 μm and constituents that have sizes between 10 nm and 2 μm.

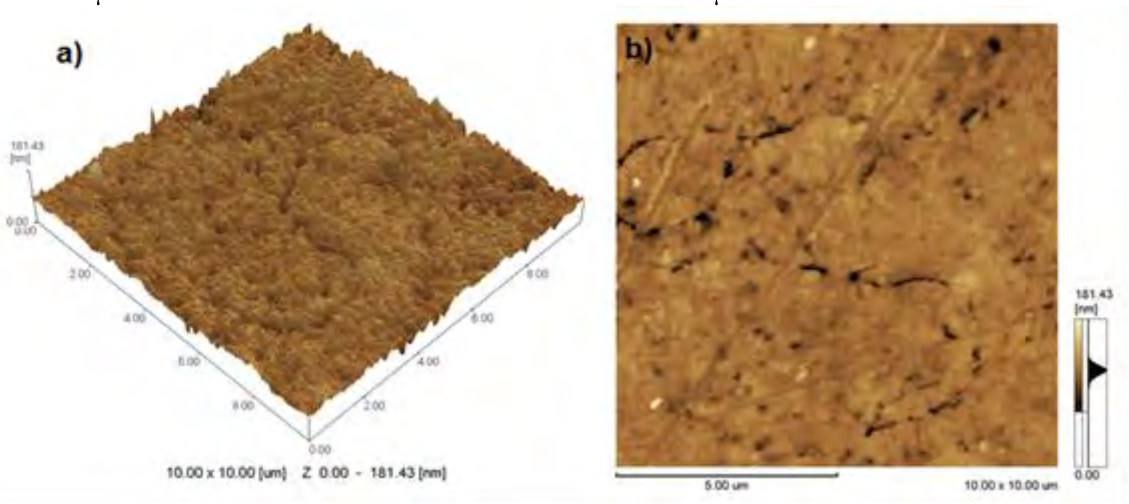

Figure 7. Atomic force microscopy topographical analysis sample with T7451 temper: (a) three-dimensional image and (b) bi-dimensional image. Etching: Keller reagent.

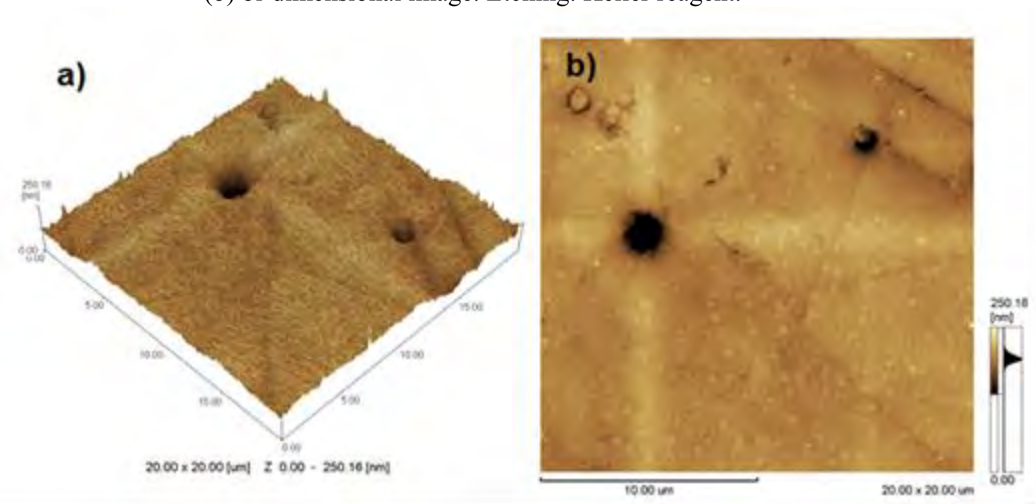

Figure 8. Atomic force microscopy topographical analysis sample with T6 temper: (a) three-dimensional image and (b) bi-dimensional image. Etching: Keller reagent.

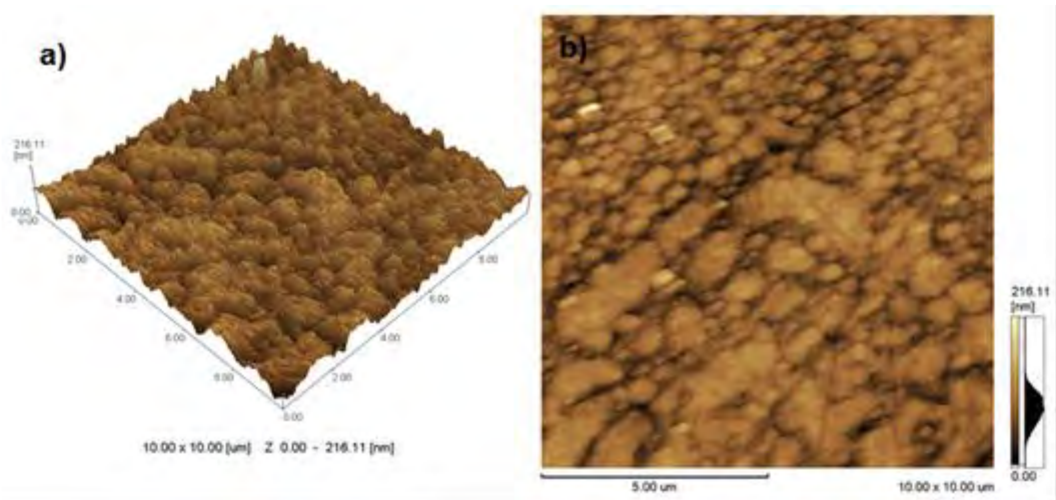

Figure 9. Atomic force microscopy topographical analysis sample with T614-65 temper: (a) three-dimensional image and (b) bi-dimensional image. Etching: Keller reagent.

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4. CONCLUSIONS

1. The aging heat treatment T614-65 proposed as an alternative to treatment condition T7451 proves efficient because exhibited high hardness peaks above 200HV.
2. DSC tests have identified the presence of η' phase the formation of exothermic peaks at characteristic temperatures only under the conditions T6 and T614-65.
3. The microstructures obtained by MO no visible change to justify the change of properties verified by DSC and characterization techniques.
4. The images obtained by SEM it was possible to identify intermetallic constituent particles, preferably located in the grain boundaries and also dispersed within the grains. The condition T614-65 showed agglomerates of particles homogeneously dispersed in the matrix probably phase particles η' which precipitated during the aging treatment.
5. The AFM images confirm what was observed in the condition where SEM T614-65 has higher surface roughness conditions T6 and T7451 due to higher particle density η'.

5. ACKNOWLEDGEMENTS

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