

# MICROSTRUCTURAL AND MECHANICAL STUDY OF ALUMINIUM ALLOYS SUBMITTED TO DISTINCT SOAKING TIMES DURING SOLUTION HEAT TREATMENT

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## Abstract

This work studies the microstructural characteristics and mechanical properties for different aluminium alloys (1100, 3104 and 8011) hot rolled sheets that were subjected to a solution heat treatment with distinct soaking times, in order to promote microstructural and mechanical changes on these alloys with solute fractions slightly above the maximum solubility limit. Scanning Electronic Microscopy (SEM) / Energy Dispersive Spectroscopy X-Ray (EDS), X-Ray Diffraction (XRD) and Hardness Tests were employed to observe the microstructural / compositional and mechanical evaluation. For the 1100 and 8011 alloys the more suitable soaking time occur between 1 and 2 hours, and for the 3104 alloy occurs between 2 and 3 hours.

**Keywords:** Aluminum; Microstructure; Hot rolling; Solution heat treatment.

## ESTUDO MICROESTRUTURAL E MECÂNICO DE LIGAS DE ALUMÍNIO SUBMETIDAS A DISTINTOS TEMPOS DE SOLUBILIZAÇÃO

## Resumo

Este trabalho estuda as características microestruturais e mecânicas de distintas ligas de alumínio (1100, 3104 e 8011) na forma de chapas laminadas a quente que foram submetidas a um tratamento térmico de solubilização com distintos tempos de encharque, de modo a promover modificações microestruturais e mecânicas nestas ligas com percentuais de soluto ligeiramente acima do limite máximo de solubilidade. A avaliação microestrutural/composicional e mecânica foi feita por Microscopia Eletrônica de Varredura (MEV) / Espectroscopia de Energia Dispersiva de Raios-X (EDS), Difração de Raios-X (DRX) e ensaios de durezas. Para as ligas 1100 e 8011 o tempo mais adequado de encharque ocorreu entre 1 e 2 horas, e para a liga 3104, ocorreu entre 2 e 3 horas.

**Palavras-chave:** Alumínio; Microestrutura; Laminação a quente; Tratamento térmico de solubilização.

## 1 INTRODUCTION

Aluminum is one of the most metals applied in various industry sectors, and such applicable is due to a big range of properties obtained by their chemical composition and thermomechanical treatments, combined with a low density [1].

Among the heat treatment existing for aluminum alloys, the solution heat treatment is used to produce a structural single-phase supersaturated without precipitates, more easily for cold forming, or as a precondition for aging

heat treatment [2-4]. In some cases where the solute exceeds the solubility limit, this initial condition cannot be got (single-phase structure without precipitates), but it allows to obtain a supersaturated matrix with a partial fraction of precipitates [5,6]. This condition allows a strength reduction [7] and a ductility increase [8-10].

The solution heat treatment consists in most cases, heating an alloy between 573 to 773 K, keep for a certain soaking time (which can vary from a few minutes to several

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hours), and after this, the alloy is cooled down to room temperature or lower temperatures [2,4,11,12]. Some solution heat treatments use two distinct heating stages [10,12].

This heating depends on the alloy chemical composition, and is made up to a sufficiently high temperature associated to single-phase field, during a sufficiently time to achieve a nearly homogeneous solid solution which all precipitates are dissolved, so that one single-phase structure is attained [4,9].

The temperature or/and the soaking time must be well controlled, because the over temperature or the over time may not cause the spheroidization, coarsening and an precipitate decrease, and this way obtain the microstructural characteristics which are not expected after the solution heat treatment [7,12].

An important characteristic obtained by the solution heat treatment in aluminum alloys, is that depending on the parameters of the heat treatment, there are different corrosion mechanisms or rates [12].

This study evaluated the influence of a solution heat treatment (ST), with different soaking times (1, 2 and 3 hours) in the mechanical and microstructural properties for the 1100, 3104 and 8011 alloys. For this purpose the techniques used were: Scanning Electronic Microscopy (SEM) / Energy Dispersive Spectroscopy X-ray (EDS), X-ray diffraction (XRD) and hardness tests.

## 2 MATERIALS AND METHODS

### 2.1 Materials

The materials used for this study were 1100, 3104 and 8011 aluminum alloys, a 6.00, 10.00 and 6.95 mm thickness hot-rolled sheet, respectively, donated by Votorantim

Metals (originally from Aluminum/SP - Brazil). Table I shows the sheets chemical compositions.

Samples nomenclatures are as follow:

- (i) The first four digits represent the alloy analyzed = 1100, 3104 or 8011;
- (ii) The second two digits indicate the initial condition of the alloy: AR = As Received and ST = submitted to a Solution heat Treatment).
- (iii) The last digit indicate the soaking time = 1 for 1 hour, 2 for 2 hours or 3 for 3 hours.

### 2.2 Methods

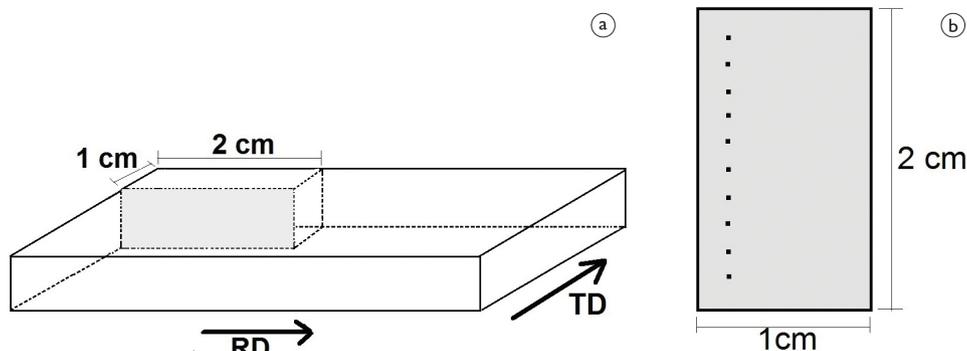
Samples with 1 cm (transverse direction - TD) × 2 cm (rolling direction - RD) were extracted from plates and subjected to solubilization heat treatment. Figure 1a shows how the samples were taken from the hot rolled sheets.

The heat treatment was performed in an electric resistance furnace at soaking temperature at 773 K (According to the Metals Handbook [2]), for 1, 2 and 3 hours, and cooled down in water at room temperature under agitation. This soaking temperature was chosen based on the binary phase diagrams [13] associated to the principal solute for each alloy (3104, 8011 and aluminum commercially pure (1100)), in order to promote the maximum precipitates dissolution, without occur local melting (below their eutectic lines). Consequently, verify significant microstructure changes on the alloys during the soaking times longer than 1 hour [2].

The samples, associated with the rolling direction (RD) (indicated by the gray color in Figure 1a), were hot encapsulated with phenolic resin, sanded with 220-4000 mesh SiC sandpaper and mechanically polished with diamond pastes (6 μm, 3 μm and 1 μm).

**Table I.** 1100, 3104 and 8011 aluminum alloys - chemical compositions (weight percent)

Alloy	Chemical Composition (%)										
	Si	Fe	Cu	Mn	Mg	Ti	Cr	Ni	Zn	Al	Others
1100	0.140	0.520	0.096	0.009	0.001	0.012	0.005	0.005	0.005	99.200	0.007
3104	0.230	0.640	0.072	0.770	0.810	0.018	0.034	0.004	0.021	97.380	0.016
8011	0.590	0.690	0.027	0.005	0.001	0.019	0.003	0.004	0.006	98.620	0.035



**Figure 1.** (a) Samples extraction from the hot rolled sheet and (b) 1/4 of the thickness position where the Vickers hardness tests were taken.

The SEM / EDS analysis was performed with the secondary electron detector: 12 KeV, spot size of 500 and a working distance of 11 mm.

The Vickers hardness test (Manufacturer Spectrum Instrumental Scientific LTD) was performed with a load of 100 g (0.1 kgf) and a dwelling time of 18 seconds. For samples on AR, ST-1, ST-2 and ST-3 conditions, 10 measurements were performed at 1/4 of the thickness as shown in Figure 1b.

The X-Ray diffraction (XRD) analysis was performed with a Shimadzu diffractometer (XRD-6000) with Co-K $\alpha$  radiation, standard goniometer, 30 kV, 30 mA,  $\theta/2\theta$  coupling scans in the range of 20° to 110° for 2 $\theta$ , with 2 $\theta$  step of 0.02° and speed of 2° per minute (equivalent to acquisition time of 0.6 s per point). The JCPDS cards (*Joint Committee on Powder Diffraction Standards*) used were 89-4037 ( $\alpha$  phase), 89-1981 (Al<sub>2</sub>Cu), 65-5188 (AlFe<sub>3</sub>), 89-7376 (FeSi), 89-9056 (Si) and 89-9056 (Mg<sub>32</sub>Al<sub>47</sub>Cu<sub>7</sub>). For the XRD analysis results shown in Results and Discussion, the “relative intensity (%)”, represents the percentage value of each peak compared to the highest intensity obtained during the measurement.

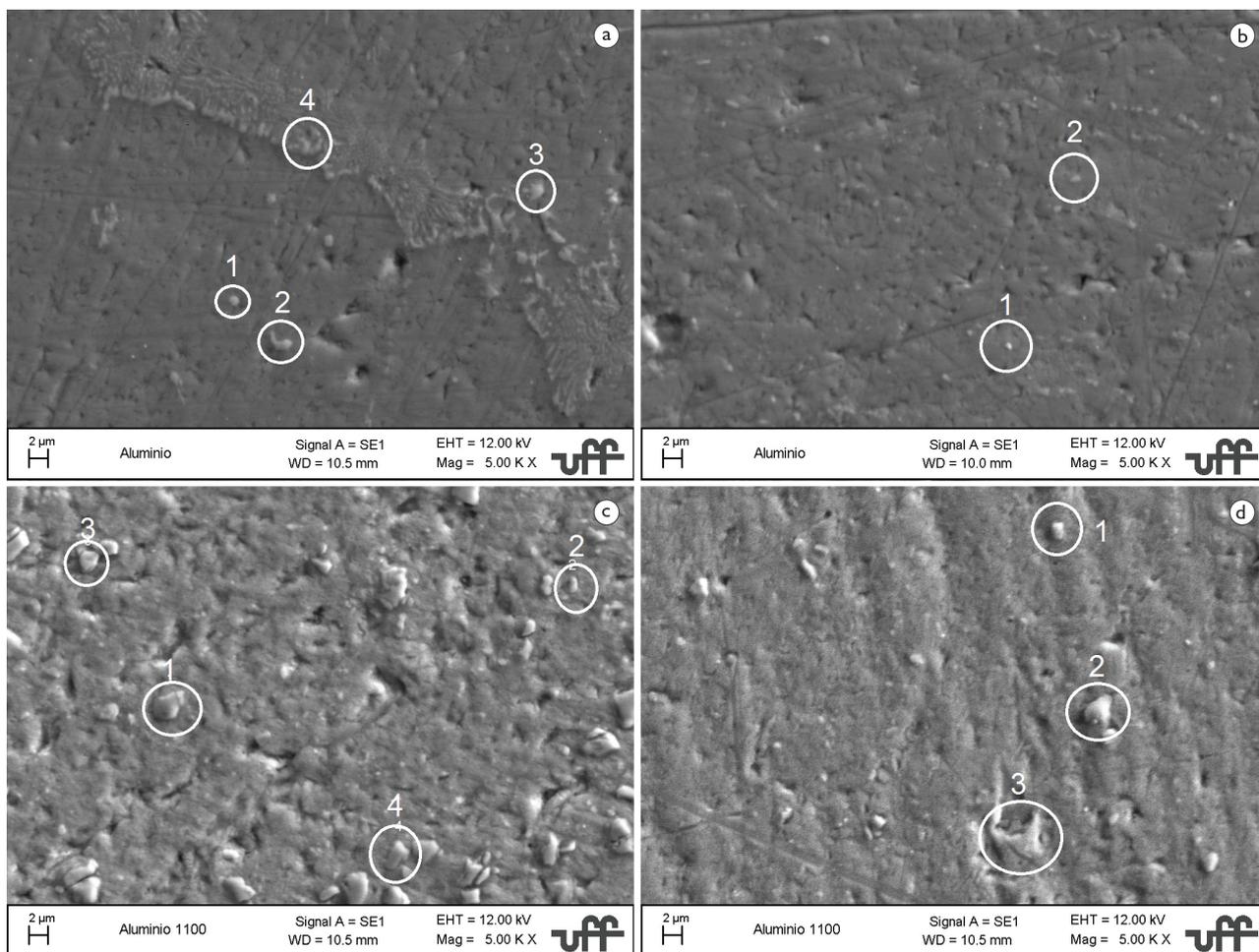
### 3 RESULTS AND DISCUSSION

#### 3.1 Microstructure

The Figures 2, 3 and 4 show the microstructure for the 1100 (Figure 2), 3104 (Figure 3) and 8011 (Figure 4) alloys, in AR (Figures 2a, 3a, 4a), ST-1 (Figures 2b, 3b, 4b), ST-2 (Figures 2c, 3c, 4c) and ST-3 (Figures 2d, 3d, 4d) conditions, obtained by the secondary electrons and EDS detectors coupled to SEM.

Punctual chemical compositions analyzes were performed on light gray points associated to the precipitates on matrix (dark gray), which the matrix showed high aluminum levels. The principal precipitates were stood out by white circles and indicated by numerical indices on the images (Figures 2, 3 and 4), which are referenced by their chemical composition analysis measured with the EDS probe, and listed in Tables 2, 3 and 4.

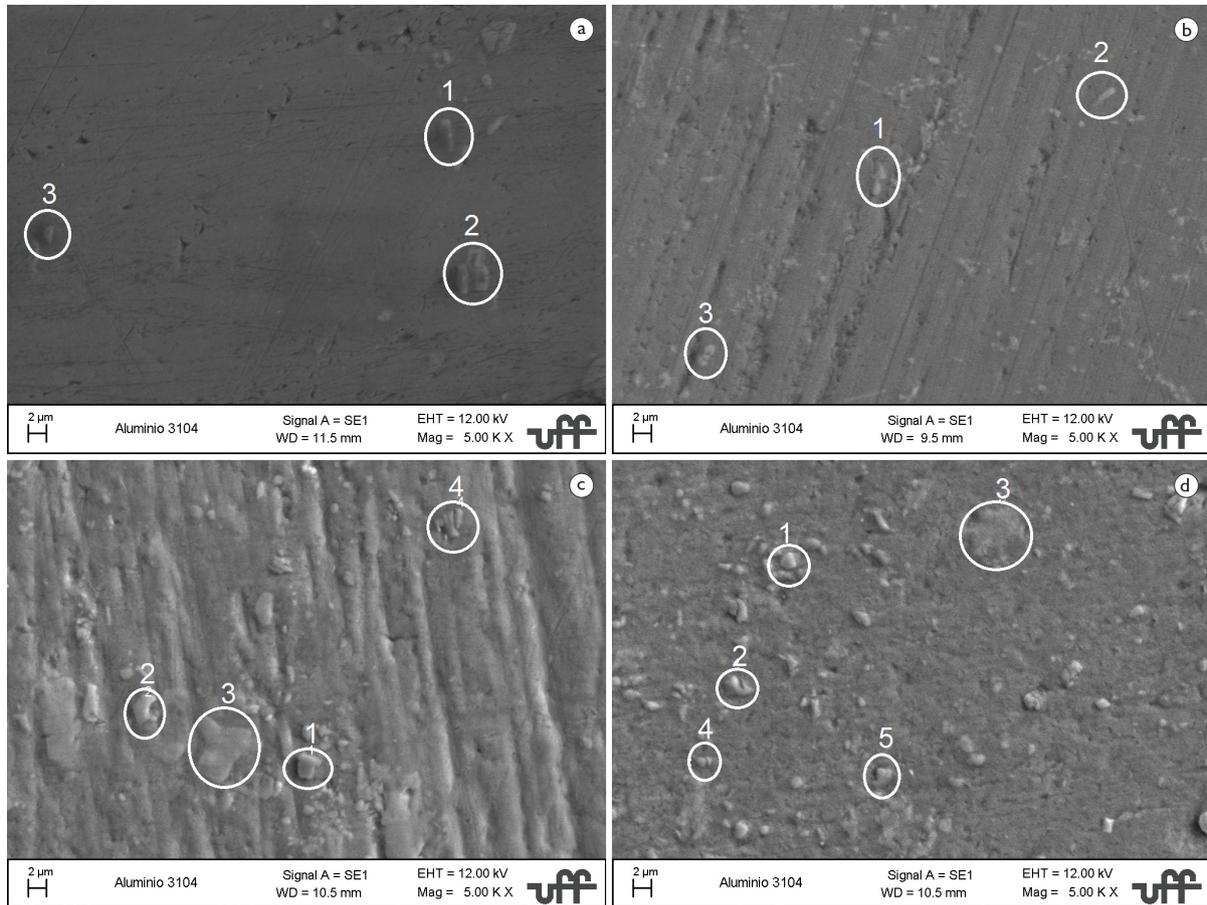
The presence of Al large percentages came from the matrix (Al rich  $\alpha$  phase) surrounding the precipitate, due to electron beam volume interaction larger than precipitates volume, especially for those smaller precipitates, implied



**Figure 2.** Microstructural aspects for the 1100 alloy at 1/4 of the thickness (a) AR, (b) ST-1, (c) ST-2, and (d) ST-3 conditions. The white circles stand out the precipitates analysed by EDS detector, as punctual chemical compositions results shown in Table 2.

**Table 2.** Punctual chemical composition by EDS for the 1100 alloy on AR, ST-1, ST-2 and ST-3 conditions associated to distinct analyzed points, according to the indications in Figure 2. Values in weight percent

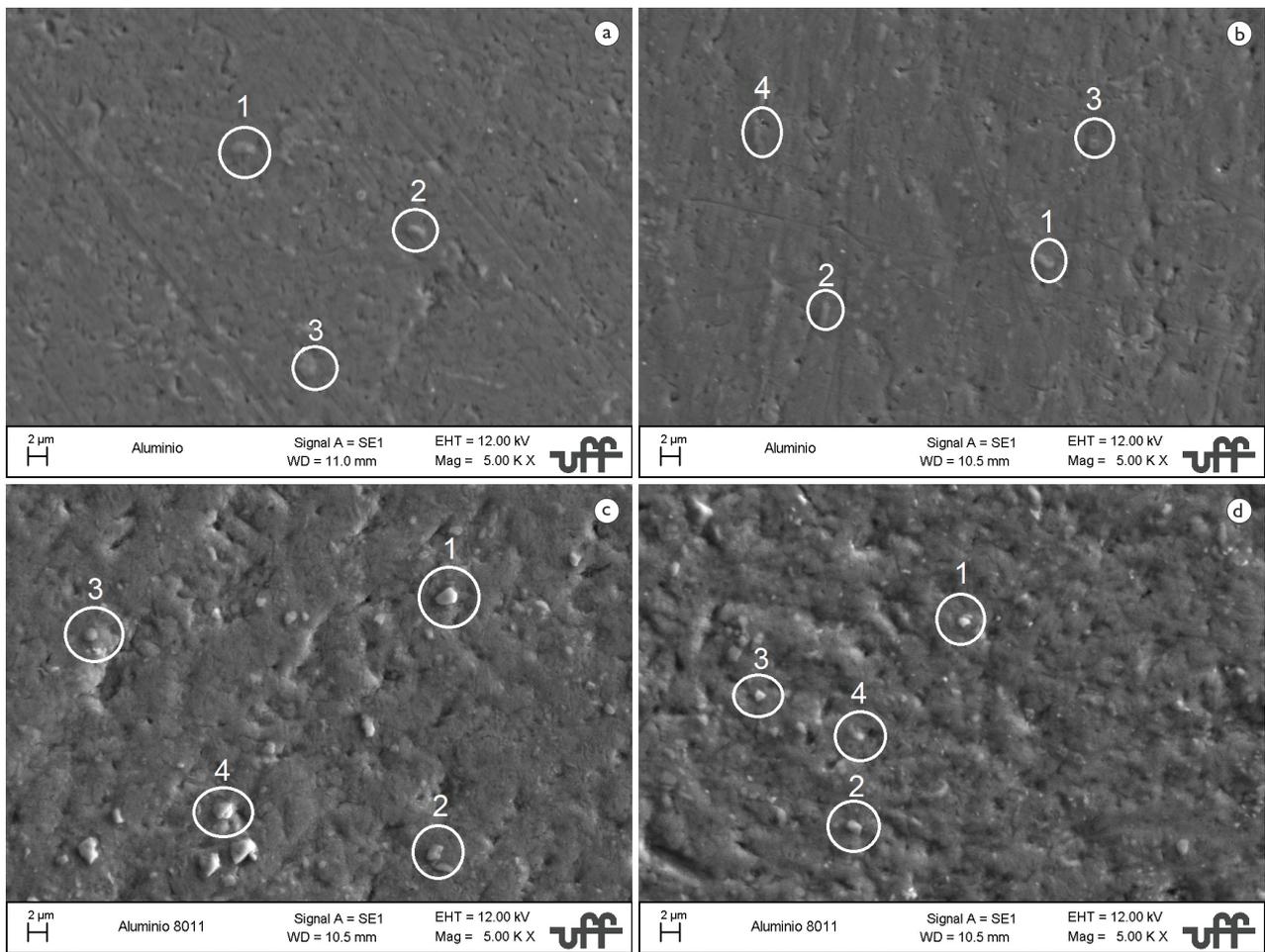
Sample Condition / Precipitate Point	1100 alloy - 1/4 of the thickness													
	AR (a)				ST - 1 (b)		ST - 2 (c)				ST - 3 (d)			
	1	2	3	4	1	2	1	2	3	4	1	2	3	
Element by weight (%)	Si	-	-	-	-	-	38.7	6.9	20.1	30.0	13.5	29.8	8.1	
	Fe	5.0	7.3	9.1	11.7	3.9	3.8	0.7	1.0	1.9	1.2	0.8	0.8	1.9
	Cu	1.5	1.7	2.1	1.8	1.4	1.4	-	-	-	-	-	-	
	Mg	-	-	-	-	-	-	-	-	-	0.4	0.3	0.4	
	Al	92.8	90.3	88.1	85.9	94.0	94.1	53.1	84.9	69.1	63.0	82.9	57.4	88.0
	Others	0.7	0.7	0.7	0.6	0.7	0.7	7.5	7.2	8.9	5.8	2.4	11.7	1.6



**Figure 3.** Microstructural aspects for the 3104 alloy at 1/4 of the thickness. (a) AR, (b) ST-1, (c) ST-2 and (d) ST-3 conditions. The white circles stand out the precipitates analysed by EDS detector, as punctual chemical compositions results shown in Table 3.

**Table 3.** Punctual chemical composition by EDS for the 3104 alloy on AR, ST-1, ST-2 and ST-3 conditions associated to distinct analyzed points, according to the indications in Figure 3. Values in weight percent

Sample Condition / Precipitate Point	3104 alloy - 1/4 of the thickness															
	AR (a)			ST - 1 (b)			ST - 2 (c)				ST - 3 (d)					
	1	2	3	1	2	3	1	2	3	4	1	2	3	4	5	
Element by weight (%)	Si	4.1	3.9	3.2	2.6	8.5	2.0	38.7	6.9	20.1	26.0	12.3	18.1	2.0	0.7	2.9
	Fe	17.5	14.5	13.4	17.7	15.9	9.8	0.7	1.0	1.9	1.5		6.2	3.3	6.5	
	Cu	0.4	0.4	0.3	0.2	0.4	0.5	-	-	-	-	-	-	-	-	
	Mn	8.0	7.9	6.5	3.4	5.1	3.5	-	-	-	-	-	3.1	1.8	3.2	
	Mg	0.5	0.7	0.8	0.4	0.5	0.8	-	-	-	-	0.8	0.8	0.9	0.9	0.8
	Al	59.0	57.5	58.7	70.3	60.7	78.0	53.1	84.9	69.1	66.7	76.4	74.9	84.7	90.1	84.0
	Others	10.5	15.1	17.1	5.4	8.9	5.4	7.5	7.2	8.9	5.8	10.5	6.2	3.1	3.2	2.6



**Figure 4.** Microstructural aspects for the 8011 alloy ¼ of the thickness. (a) AR, (b) ST-1, (c) ST-2 and (d) ST-3 conditions. The white circles stand out the precipitates analysed by EDS detector, as punctual chemical compositions results shown in Table 4.

**Table 4.** Punctual chemical composition by EDS for the 8011 alloy on AR, ST-1, ST-2 and ST-3 conditions associated to distinct analyzed points, according to the indications in Figure 4. Values in weight percent

Sample Condition / Precipitate Point	8011 alloy - ¼ of the thickness															
	AR (a)			ST - 1 (b)				ST - 2 (c)				ST - 3 (d)				
	1	2	3	1	2	3	4	1	2	3	4	1	2	3	4	
Element by weight (%)	Si	8.5	4,1	1.7	5.5	0.5	1.3	3.1	47.6	22.6	4.6	2.1	10.8	13.8	3.3	13.9
	Fe	3.2	2.4	5.6	12.1	2.1	4.7	5.4	-	-	8.6	4.5	-	-	10.2	6.6
	Al	76.7	75.8	79.5	74.6	85.7	81.2	78.0	36.3	68.4	77.3	87.1	86.2	79.8	77.5	72.9
	Others	11.6	17.7	13.2	7.8	11.7	12.8	13.5	16.1	9.0	9.5	6.3	3.0	6.4	9.0	6.6

by punctual EDS analyze and SEM parameters in used. For some points, associated to precipitates, analyzed on AR, ST-1, ST-2 and ST-3 conditions (Figures 2, 3 and 4), They were founded the presence of others chemical elements (indicated by ‘Others’ in Tables 2, 3 and 4), which are probably associated with oxides and carbides, resulting from impurities, contamination or inclusions derived from the preparation process of these alloys.

Comparing the precipitates observed in the alloys analyzed on distinct conditions (Figures 2, 3 and 4), it can be observed that the precipitates have a similar format, except

to CR- condition for 1100 alloy that exhibited eutectic cell format (Figure 2a). For the ST-1 and ST-2 conditions, there is an increase on the precipitates size in all alloys. These precipitates size increases, continued only for the 3104 alloy on ST-3 condition (Figure 3d).

For the 1100 (Figure 2) and 8011 (Figure 4) alloys, it can be seen that the precipitates became more evident and homogeneous on ST-2 condition (Figures 2c and Figure 4c). For the ST-3 condition (Figures 2d and Figure 4d) there are a precipitates number reduction, probably due to the soaking time for these alloys, therefore, the more suitable

soaking time for the solution heat treatment for this alloys should be between 2 and 3 hours. Thus this indicates evidence of initial event of precipitates slightly coarsening [7] on the first hour of solution heat treatment (1 hour for the 1100 and 8011 alloys, and 2 hours for the 8011 alloy) and dissolution the same in longer soaking times (3 hours for 1100 and 3104 alloys, and 2 and 3 hours for the 8011 alloy). Some evidences of volume fraction reduction with the soaking time can be observed in the work done by Xu et al. [12] in a 7150 aluminum alloy.

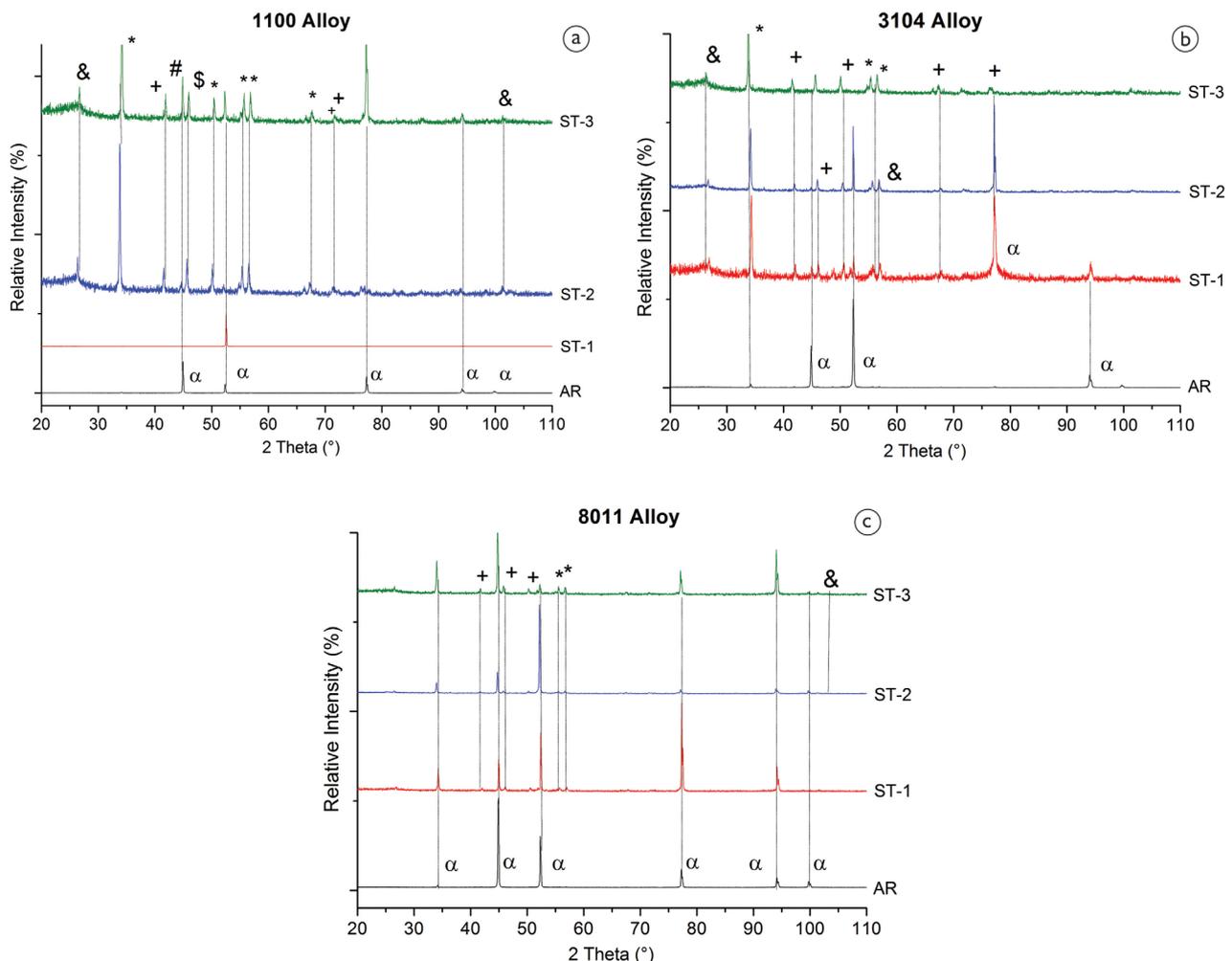
Based on the alloys global chemical composition (Votorantim Metals - Table 1) and the chemical composition analysis by EDS probe (Tables 2, 3 and 4), possibly occurred an alloying element atomic diffusion from the precipitates to the matrix. This evidence may be confirmed by the precipitates changing morphology on AR, ST-1, ST-2, and ST-3 conditions (Figures 2, 3 and 4), and the chemical elements (%) decrease obtained by the EDS probe (Tables 2, 3 and 4).

### 3.2 X-Ray Diffraction Analysis

The Figure 5 shows the X-Ray diffraction analysis results for the alloys on AR, ST-1, ST-2 and ST-3 conditions. In the Figure 5, ( $\alpha$ ) represents alfa phase, (\*) represents  $Al_2Cu$  precipitate, (&) represents Si precipitate, (+) represents  $Mg_{32}Al_{47}Cu_7$  precipitate, (#) represents  $AlFe_3$  precipitate, and (\$) represents FeSi precipitate.

Comparing the X-Ray Diffraction analysis (Figure 5) with the punctual EDS chemical composition (Tables 2, 3 and 4), it can be seen that the principal element for the precipitates formation is the Cu, Si, Fe and Mg.

According to the Figure 5, the  $\alpha$  phase diffraction peaks are present since the AR to ST-3 condition, and there was a significant intensity increase of the precipitates diffractions peaks for the 1100 and 8011 alloys on ST-1 to ST-2 condition. This behavior shows the evidence that the ideal soaking time to the phases stabilization is between 1 and 2 hours. However, for the 3104 alloy the significant



**Figure 5.** X-Ray diffraction analysis on AR, ST-1, ST-2 and ST-3 conditions: (a) 1100, (b) 3104 and (c) 8011 alloys. In the Figure ( $\alpha$ ) represents alfa phase, (\*) represents  $Al_2Cu$  precipitate, (&) represents Si precipitate, (+) represents  $Mg_{32}Al_{47}Cu_7$  precipitate, (#) represents  $AlFe_3$  precipitate, and (\$) represents FeSi precipitate.

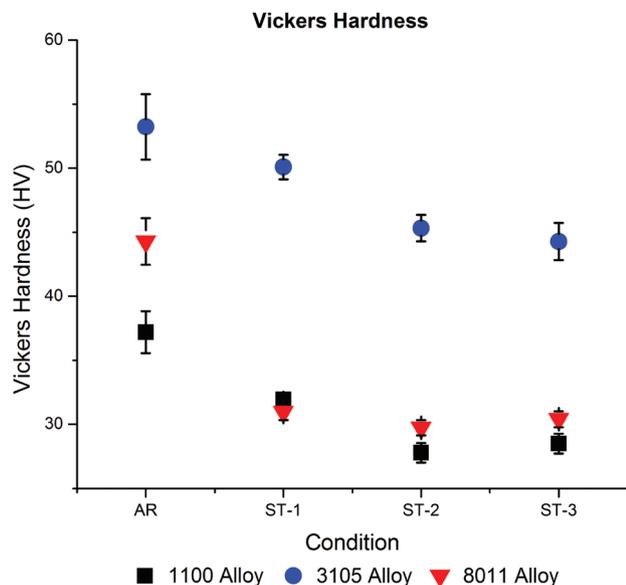
intensity increase of the precipitates diffractions peaks occurs between the ST-2 to ST-3 conditions, giving the evidence that the ideal soaking time to achieve the phases stabilization is between 2 and 3 hours. These results confirm the analysis by SEM / EDS (Figure 2 and Table 2).

Comparing the XRD and EDS analysis results, it can be seen that in some conditions, the XRD analysis showed precipitates peaks related to some chemical elements that were not related on EDS analysis. An example of this, are the ST-1, ST-2 and ST-3 (Figure 5c) conditions for the 8011 alloy, that showed the X-Ray diffraction peaks related to the  $Mg_{32}Al_{47}Cu_7$  and  $Al_2Cu$  precipitates, and the chemical element Cu is not observed on EDS analysis. The difference between both analyzes can be explained by the sensitivity of each technique, which to be detected in the XRD analysis is required a certain precipitates size and volume fraction. Another possible explanation is the no complete homogeneity of the alloys analyzed that were produce in industrial scale.

### 3.3 Vickers Hardness Test

Figure 6 shows the Vickers hardness for the 1100, 3104 and 8011 alloys on AR, ST-1, ST-2 and ST-3 conditions at  $\frac{1}{4}$  of the thickness.

In relation to hardness measurement positions, it can be seen that there were no significant differences in hardness. The standard deviations of the hardness measure-



**Figure 6.** Vickers hardness measurements (average and standard deviation) for the 1100, 3104 and 8011 alloys on AR, ST-1, ST-2 and ST-3 conditions at  $\frac{1}{4}$  of the thickness.

ments were small; it is shown some structural homogeneity along the sheet on AR, ST-1, ST-2 and ST-3 conditions.

According to the Figure 6 the 3104 alloy showed the highest Vickers hardness, the 1100 and 8011 alloys showed the lowest and intermediate Vickers Hardness, respectively. These differences on the Vickers hardness are due to the alloying elements present in each alloy in study [14].

According to the Figure 6, the biggest changes in the Vickers hardness (decrease) for the 1100 and 3104 alloys occurs until the ST-2 condition, while the Vickers hardness on ST-3 condition are very close to the ST-2 condition. The biggest change in the Vickers hardness (decrease) for the 8011 alloy occur until ST-1 condition and remains approximately constant until the ST-3 condition.

Vickers hardness decreased from AR to ST-2 conditions (Figure 6) for the 1100 and 3105 alloys and from AR to ST-1 conditions (Figure 6) for the alloy 8011, indicating a possible solute incorporation on the matrix by precipitate dissolution during solution heat treatment. However, this change can also be associated with grain growth promoted by the temperature and soaking time during the solution heat treatment.

## 4 CONCLUSIONS

Based on MEV / EDS, microstructural and XRD analysis for the 1100, 3104 and 8011 alloys on AR, ST-1, ST-2, and ST-3 conditions, it can be concluded that the solution heat treatment:

- resulted in spheroidal precipitates with a higher dispersion;
- decrease the needle precipitates volume fraction;
- decrease the samples hardness by dissolving and / or size reduction for the precipitates by the solute incorporation in the matrix;
- for the 1100 and 8011 alloys the more suitable soaking time occur between 1 and 2 hours, and for the 3104 alloy occurs between 2 and 3 hours.

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