# Effects of Homogenization and Cooling after Extrusion on Microstructure and Ageing Kinetics of Aluminum Alloy 2014

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#### Abstract

Homogenized billets of alloy 2014 were extruded under different conditions. The changes in grain structure and second phase particle distribution that took place during extrusion, cooling after extrusion and solution treatment, were analyzed by optical microscopy with polarized light (LOM) and by scanning electron microscopy (SEM with EDS). Also, ageing response at 170°C was assessed. Results showed that grain structure, as well as ageing response, are related to the homogenization treatment and conditions of cooling after extrusion. Extrudates produced from billets that were furnace cooled after homogenization have shown evidence of static recrystallization, even after press-quenching. As a consequence, maximum hardness in the T5 and T6 tempers was lower in comparison with bars produced from billets that were air cooled after homogenization. These findings emphasize the concept that the effects of thermal history prior to extrusion must be taken into account if the final mechanical properties of extruded 2014 alloy are to be optimized.

## 1. Introduction

The combination of high mechanical properties, low density and corrosion resistance makes extrudates of Al-Cu alloys very attractive to the transportation industry. However, in comparison to other Al alloys, the mechanical working of these alloys is difficult, especially when extrusion is considered. Therefore, systematic studies on the inter-relationships among processing, microstructure and mechanical properties for these alloys are of great importance, as the microstructures developed during extrusion processing must have great influence on the mechanical properties after ageing. The present work aims at the study of the microstructure of Al alloy 2014 in its as-cast and as-homogenized conditions, as well as the modifications brought about by the homogenization practice. The changes in grain structure and second phase particle distribution that take place during extrusion, cooling after extrusion and solution treatment, are analyzed by optical microscopy. Also, results on ageing kinetics of samples aged at 170°C are presented.

## 2. Experimental Procedure.

Industrial scale DC ingots of alloy 2014, of 6" in diameter with chemical composition (wt %) 4,15% Cu; 0,52% Mg; 0,46% Mn; 0,53% Si; 0,33% Fe; 0,065% Zn; 0,013% Pb, were used for the experimental work.

Two DC ingots were homogenized at 500°C for 24 h, followed by furnace cooling, and two DC ingots were homogenized at 500°C for 24 h, followed by air cooling. After homogenization, the DC ingots were preheated for extrusion in a muffle furnace at 350°C for 3 h. The extrusions were carried out in an horizontal 1,200 tons press, with its container heated to 450°C. The extrudates were square bars and extrusion ratio of 28 was used. The variables studied were homogenization condition and cooling after extrusion, as shown in Table 1 below.

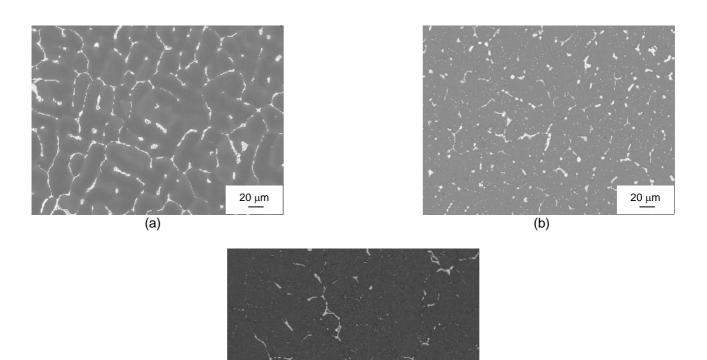
Condition	Homogenization practice	Cooling after extrusion
F / AIR	500°C for 24 h / furnace	in air
A / AIR	500°C for 24 h / air	in air
F / WAT	500°C for 24 h / furnace	in water
A / WAT	500°C for 24 h / air	in water

Table 1: Extrusions' experimental conditions.

Transverse sections were extracted from each extruded bar in the following positions, according to bar's length (~ 10 m): at 0,5 m from the beginning, at 0,5 m from the end and in the middle of each bar (the "beginning" of the bar is the first material that comes out of the extrusion press). Transverse sections were cut in halves, in order to analyze the grain structure in a longitudinal plane by means of optical microscopy with polarized light. Material extracted from the middle portion of each extruded bar was solution heat treated at 500°C for 2 h, then water quenched. After solution heat treatment samples were aged at 170°C, for several periods of time, then air cooled. Ageing response at 170°C was obtained from Vickers macrohardness measurements (load = 1Kg).

## 1. Results and Discussion

The microstructures developed during the homogenization treatments used are shown in Figure 1, where the interdendritic phase distribution can be observed by means of SEM. Prior to homogenization, as seen in Figure 1a, the as-cast microstructure is composed of aluminum matrix and interdendritic lamellae precipitates, typical of eutectic phases, in the form of an almost continuous network. The microanalysis of these lamellae showed that they can either be formed by  $\theta$  (Al<sub>2</sub>Cu) and Q (Al<sub>5</sub>Cu<sub>2</sub>Mq<sub>8</sub>Si<sub>6</sub>) phases, or by  $\theta$  and Al<sub>15</sub>Si<sub>2</sub> (Cu,Fe,Mn)<sub>3</sub> phases. According to Mondolfo [1], in Al-Cu alloys in which the relation %Mg / %Si  $\leq$  1, such as the allov studied here, the eutectic can be formed by  $\theta$  and Q phases. Also, some authors [2,3] report the existence of other eutectic phases, constituted by AI and Si, that can be identified as AI<sub>15</sub>Si<sub>2</sub> (Cu,Fe,Mn)<sub>3</sub> in the present work. It is to be expected that the above-mentioned coarse intermetallic compounds have a deleterious effect on the hot workability of alloy 2014. Aiming at the redistribution of coarse intermetallic compounds and improved mechanical properties of the extrudate, homogenization is a current practice for alloy 2014. When the microstructure after homogenization and furnace cooling is analyzed (Figure 1b), it can be seen that the interdendritic network changed to a broken configuration. Also, some of the interdendritic precipitates are coarser and a fine precipitation (~1  $\mu$ m) can be observed within the grains. The microanalysis of the interdendritic precipitates showed that they are the  $\theta$  (Al<sub>2</sub>Cu), Q (Al<sub>5</sub>Cu<sub>2</sub>Mg<sub>8</sub>Si<sub>6</sub>) and Al<sub>15</sub>Si<sub>2</sub> (Cu,Fe,Mn)<sub>3</sub> phases. The small, intragranular particles were identified as  $\theta$  (Al<sub>2</sub>Cu). Some Mg<sub>2</sub>Si precipitates (~0.5  $\mu$ m), that formed during cooling from the homogenization temperature, were also found in the AI matrix.



(c) Figure 1: Al 2014 micrographs / SEM of conditions: (a) as-cast; (b) homogenized and furnace cooled; (c) homogenized and air cooled. Transverse sections, mid-radius.

20 µm

The coarsening of interdendritic precipitates is less obvious in the homogenized, air cooled condition, as can be seen in Figure 1c. These precipitates were identified by EDS as  $\theta$  (Al<sub>2</sub>Cu) and Al<sub>15</sub>Si<sub>2</sub> (Cu, Fe, Mn)<sub>3</sub> phases. In terms of the precipitation within the grains, it seems that the particles were finer and in a higher fraction than for the homogenized, furnace cooled condition. Also, Mg<sub>2</sub>Si particles can be observed inside the grains. Although homogenization enabled changes in the distribution of interdendritic particles (e.g. from an almost continuous film to a more broken configuration), it wasn't effective in terms of the dissolution of such particles. Some authors [2,3] point out the difficulty of dissolving interdendritic phases present in Al-Cu alloys. Also, the cooling mode after homogenization has a major influence on the size and amount of second phase particles present in the microstructure, which is of great importance to the development of the microstructure in the as-extruded condition, as will be discussed later.

Microstructures after extrusion can be seen in Figure 2. A typical feature in the micrographs is the presence of elongated grains aligned in the extrusion direction. The slow cooling rate due to air cooling after extrusion enabled the occurrence of static recrystallization on F / AIR and A / AIR bars (Figures 2a and b, respectively). The grain growth in the transverse direction is an evidence of partial recrystallization. Since AI and its alloys have relatively high stacking-fault energies (SFE), they easily develop subgrains during extrusion, which characterizes the occurrence of intense dynamic recovery. Therefore, there is less driving force for dynamic recrystallization (e.g. low dislocation density), since recovery and recrystallization are competitive phenomena [4]. From the points discussed, it can be concluded that the recrystallized grains observed in F/AIR (Figure 2a) and A/AIR (Figure 2b) bars were formed during air cooling after extrusion. Recrystallized grains are also observed in F/WAT bar, despite the fact that this extrudate

was water cooled after extrusion. Probably, the time period from the extrudate exiting the press and its immersion in water was sufficient for static recrystallization to occur. Furthermore, it is known that the coarse particles, present in high amount in the ingots that were furnace cooled after homogenization, are preferable sites for nucleation of recrystallization through the mechanism of particle-stimulated nucleation (PSN) [5]. Figure 2d shows a micrograph of A/WAT bar, which does not show evidence of recrystallization. The explanation for this observation lies on the small volume fraction of coarse particles present in A/WAT bar, which rules out PSN.

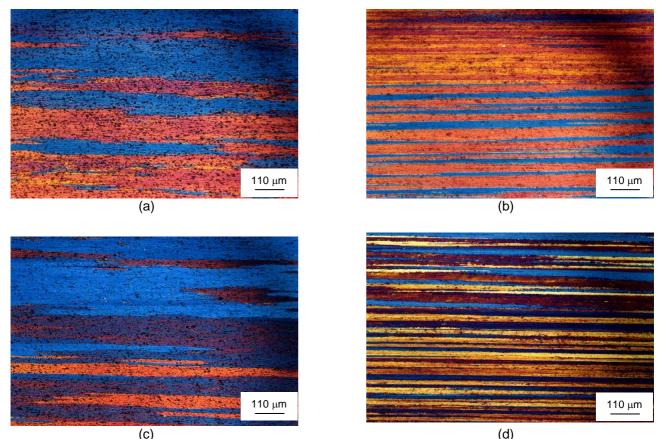


Figure 2: Al 2014 micrographs / LOM of conditions: (a) F / AIR; (b) A / AIR; (c) F / WAT; (d) A / WAT. Middle of bars. Longitudinal sections, mid-radius. Reagent: Barker's.

As showed in Figure 2, the cooling mode after homogenization (furnace or air) has an effect on particles size, which impacts on the occurrence of static recrystallization during cooling after extrusion, due to the effect of particle-stimulated nucleation of recrystallization.

Ageing curves at 170°C were obtained for T5 (press quenching followed by artificial ageing) and T6 (solution heat treatment followed by artificial ageing) tempers. For all extruded bars the hardness at T6 was higher than at T5 temper. In Figure 3, it can be seen how differences in particle and grain structures affect hardness after artificial ageing. This figure shows ageing curves at 170°C for F/WAT and A/WAT bars. It demonstrates that cooling mode after homogenization affects particle size; when the homogenization was followed by furnace cooling, second phase particles were coarser than when homogenization was followed by air cooling. In that way, F/WAT bar has coarser  $\theta$  (Al<sub>2</sub>Cu) and Mg<sub>2</sub>Si second phase particles than A/WAT bar, which implies depletion of Cu (and Mg) in solid solution to form hardening precipitates during artificial ageing in F/WAT bar. Moreover, F/WAT and A/WAT bars were press quenched and artificially aged after extrusion (both bars are in T5 temper). It is expected that no evolution of recrystallization

took place during artificial ageing, which means that F/WAT bar shows occurrence of recrystallization whereas A/WAT bar has a non-recrystallized grain structure (e.g. has a retained subgrain structure). Previous work has pointed out the importance of subgrain structure on the hardness of Al-Cu alloys, according to this work, the presence of retained substructure in aged material, apart from the direct response in hardness, leads to finer hardening precipitates and narrower precipitate-free zones (PFZ), which account for better mechanical properties [3,6].

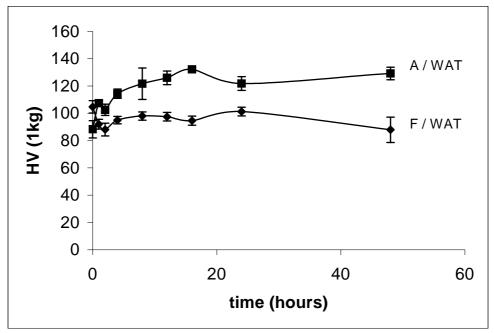


Figure 3: Ageing curves at 170°C (T5 temper).

## 4. Conclusions

Comparison of micrographs from as-cast and as-homogenized conditions showed that homogenization treatment changed the distribution of interdendritic particles. Homogenization followed by furnace cooling resulted in coarsening of interdendritic particles and precipitation of  $\theta$  (Al<sub>2</sub>Cu) and Mg<sub>2</sub>Si within the grains. Analysis of microstructures from extruded bars showed great influence of second phase particle size on the occurrence of static recrystallization during cooling after extrusion. Bars extruded from ingots that were homogenized and furnace cooled showed evidence of static recrystallization, even when press quenching was carried out. The explanation for this behavior lies in the occurrence of particle-stimulated nucleation of recrystallization in these extrudates.

The influence of thermal history prior to extrusion on the final mechanical properties was also analyzed. Bars with different homogenization conditions (homogenization followed either by furnace or air cooling) showed different hardness evolution at T5 temper. The lower hardness of the bar extruded from an ingot that was homogenized and furnace cooled is related to the homogenization condition, since it has less Cu (and Mg) in solid solution to form hardening precipitates during ageing and its coarser particles enable recrystallization (preventing subgrain boundary strengthening).

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