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INFLUENCE OF HEAT TREATMENT PARAMETERS ON THE METALLURGICAL QUALITY OF EN AW 7068 EXTRUDED BARS

M. Rosso[1]*, I. Peter[1], C. Castella[1]
[1]Politecnico di Torino, Department of Applied Science and Technology,C.so Duca degli Abruzzi 24 – 10129 Torino – Italy, mario.rosso@polito.it

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Abstract

In the present research activity EN AW 7068 alloy samples have been analyzed, from morphological and mechanical point of view. Extrusion process has been employed for the alloy production. As industrial routine, following the manufacturing the alloy has been submitted to different thermal treatments. Thermal treated samples have been extracted by cutting procedures and the obtained samples, have been submitted to standard laboratory preparation and then to morphological and mechanical characterization. Some preliminary SEM analysis reveal the presence of secondary particles. The secondary phase grains are mostly uniformly distributed in both longitudinal and transverse sections. Moreover, especially in the longitudinal section, the grains appear organized along parallel lines to the extrusion direction. According to the EDS analysis results these particles are prevalently made of Al, Cu, Zn and Mg. With the goal to achieve the dissolution of Cu-rich phases, two new solution heat treatments were performed at 470°C for 48 h and at 470°C for 24 h. Effectively, the new solution heat treatments have allowed to reach the dissolution of Cu-rich phases and secondly allowed to obtain a more homogeneous microstructure, especially for as regard the microstructure of cross section. So these thermal treatments could be adopted to dissolve Cu-rich phases that are deleterious for the mechanical properties.

1. Introduction to heat treatments of aluminium alloys

Heat treatment is a critical process to improve mechanical properties of certain aluminium alloy components. Heat treatment technology is currently facing numerous challenges such as energy conservation, environmental impact and the more strict market needs such as reliability, higher performance and production costs. Controlled heat treatment of aluminium alloys can significantly influence properties such as strength, ductility, fracture toughness, thermal stability, residual stresses, dimensional stability, resistance to corrosion and stress corrosion cracking. The main heat treatment procedures are homogenization, annealing and precipitation hardening which involves solution heat treatment, quenching and aging. The influence of heat treatments parameters and their mechanisms, as well as possible defects arising after heat treatments have been quite well studied [1 – 8], however here some indications are here introduced just as reminder.

A heat treatment and temper designation system has been developed by the Aluminum Association to describe the processing of wrought and cast aluminium alloys. Every single physical state or degree of work hardening is individuated from a letter placed after the initials of the material, separated from a hyphen and followed from one or more numbers that indicate the subdivision of the heat treatments. In Table 1 are reported the temper designation system for aluminium alloys.

Table 1 Heat treatment and temper designation system for aluminium alloys

Suffix		Treatment
F		As fabricated (as cast….)
O		Annealed
H		Strain hardened by cold work
T		Heat treated to a stable condition, excluding annealing (O)
	T1	Cooled from an elevated temperature forming process (partial solution) followed by natural ageing
	T2	Cooled from an elevated temperature forming process (partial solution), cold worked and naturally aged
	T3	Solution heat treated, quenched, cold worked and naturally aged
	T4	Solution heat treated, quenched and naturally aged
	T5	Rapidly cooled from elevated forming temperature and then artificially aged
	T6	Solution heat treated, quenched and then artificially aged
	T7	Solution heat treated, quenched and overaged
	T8	Solution heat treated, quenched, cold worked and then artificially aged (amount of cold work in % is indicated by subsequent digit)
	T9	Solution heat treated, quenched, artificially aged and then cold worked
	T10	Cooled from an elevated temperature forming process, cold worked and then artificially aged
W		Unstable temper applied for alloys which age spontaneously at room temperature after solution heat treatment. Only specific if followed by the time of natural aging.

The annealing heat treatment, indicated by capital “O” is more specific for wrought alloy, as well as the strain hardening by cold working, capital letter “H”; while capital letter “T” indicates series of processes that can be applied both to cast and wrought alloys. For the heat treated alloys, temper state T, an additional numbers can eventually be added to the first one to indicate significant differences with respect to the original state T. This more detailed designation of the heat treatment state is mainly used for high performance wrought alloy, especially for aeronautical and aero spatial applications.

The heat treatments of main interest for casting are the homogenization and annealing if strengthening effects are not a priority, while when a controlled improvement of strength is required the heat treatment to a stable condition (T series temper state) must be executed.

The heat treatment determine a strengthening by precipitation, in fact in the heat treated aluminium alloys is obtained a fine and homogeneous distribution of precipitates in a deformable matrix, the presence of these precipitates hinders the movement of the dislocations and thus strengthens the heat treated alloys.

The heat treatment is divided in three steps:

1. Solution treatment at relatively high temperature to back in solution the alloying elements and to achieve a high and homogeneous concentration of these alloying elements in solid solution;
2. Quenching usually to room temperature to obtain a supersaturated solid solution of solute atoms and vacancies,
3. Age hardening, generally indicated as aging or ageing, to cause precipitation from the supersaturated solid solution, either at room temperature (natural ageing) or at an elevated temperature (artificial ageing).

In some cases, in order to reduce the costs, it is possible to avoid the solution treatment and substituting it by a rapid cooling step performed immediately after the hot working or casting operation, but the final attained properties will not reach the highest level attainable performing the suitable solution treatment.

Solution treatment

Solution treatment is carried out at a high temperature, close to the eutectic temperature of the alloy. This temperature has to be chose with high accuracy, in fact as can be seen in the Al-Cu phase diagram shown in Figure 1, the solubility of alloying elements increase when temperature increase. Generally this temperature is comprised between the solvus temperature and the solid temperature, and the components have to be maintained to this temperature for a certain period of time necessary to obtain a homogeneous solid solution.

The purpose of the solution heat treatment is to:

1) dissolve soluble phases containing Cu and Mg formed during solidification;

2) homogenize the alloying elements;

3) spheroidize the eutectic Si particles.

The rate of these three processes increases as the solution treatment temperature increases. The maximum solution treatment temperature that can be used depends on the Cu and Mg concentrations of the alloy and is limited by incipient melting of phases formed from the last solidified melt which is rich in solute elements due to segregation.

Localized melting results in distortion and substantially reduced mechanical properties.

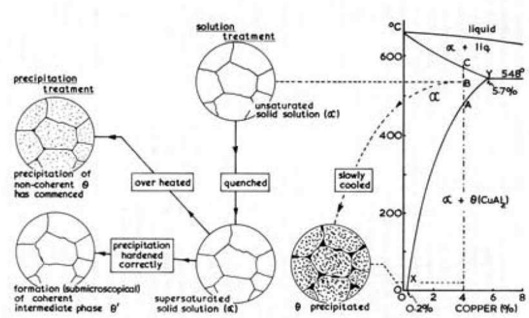

Figure 1. Schematic representation of the mechanisms associated with solution quench and aging in the case of Al-Cu alloys [9].

Quenching

The goal of the quenching step is that to suppress precipitation upon cooling of the casting from the high solution treatment temperature to room temperature. Is necessary to chose accurately the quenching rate as a matter of fact if cooling is too slow particles precipitated at grains boundaries and this results in a reduction in supersaturation of solute and a lower maximum yield strength after ageing, while on the other hand a quick cooling induces residual stresses into the components. Generally water is used as a quenching medium. When a slower quench rate is needed other quenching media such as oil, salt baths and organic solutions can be adopted.

Ageing

Aging is the controlled decomposition of the supersaturated solid solution to form finely dispersed precipitates in heat-treatable alloys. Aging takes place at room temperature (natural aging) or at an elevated temperature in the range of 100–210 °C artificial aging. The objective of aging is to obtain a uniform distribution of small precipitates, which gives a high strength. The strength of an alloy is derived from the ability of precipitates to impede mobile dislocations. The strength is determined by the size and distribution of the precipitates and by the coherency of the precipitates with the matrix.

The interaction with the dislocations can be described by the Friedel effect and by the Orowan mechanism, both are illustrated in Figure 2. Small and not too hard precipitates are normally sheared by moving dislocations (Friedel effect), see Figure 2 (a). When the precipitates are larger and harder the moving dislocations bypass the precipitates by bowing (Orowan mechanism), see Figure 2 (b).

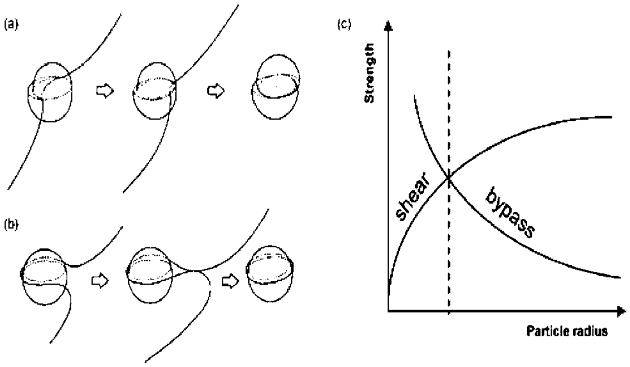

Figure 2. Dislocation bypass precipitates by : a) Friedel effect or b) Orowan mechanism [4].

The strength of the precipitates increases with their size as long as they are sheared by dislocations. Further increase of precipitate size makes the shearing processes rather difficult; thus, it is more favourable for the dislocations to pass the precipitates via the Orowan mechanism, leading to a decrease in strength with further increase in precipitate size, see Figure 2 (c). The highest strength is obtained when there is an equal probability for the dislocations to pass the precipitates by shearing and by bowing.

A precipitation hardened alloy after the quenching has an high energy level and it is instable, for this reason the alloy itself tries to achieve a low energy state through the spontaneous transformation of the supersatured solid solution in metastable phase or equilibrium phase. The driving force for the transformation is represented by the decrease of the system's energy that occurs as a result of the transformation itself. For certain alloys the driving force is so high to favours the precipitation already at room temperature during short period after quenching, these spontaneous transformations are indicated as natural aging.

The formation of the precipitates follows the traditional mechanism of nucleation and growth. After a certain period of time necessary for the formation of stable nuclei, the process rapidly continues up to a slowdown determined by the progressive impoverishment of solute in the solution. In Figure 3 can be observed the hardness-time curve summary of the aging process.

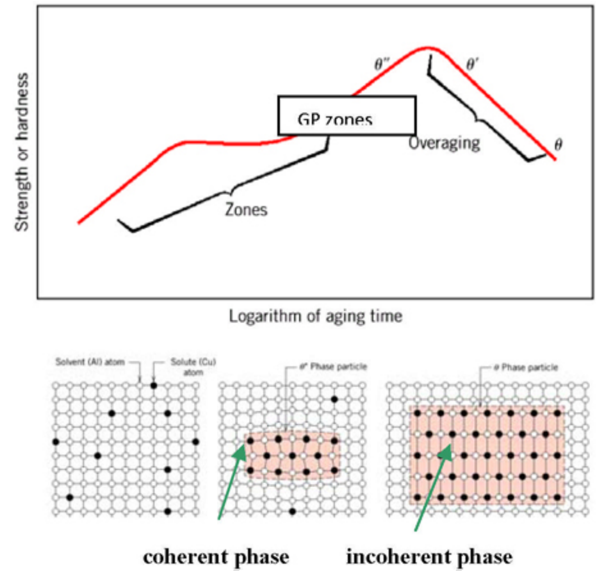

Figure 3. TOP, schematic diagram of the effects of the isothermal aging on hardness and mechanical strength. BOTTOM, sequential sketches of solvent and solute atoms in the solid solution state, coherent and incoherent phases respectively.

The precipitation sequence for Al-Si-Cu alloys is the following:

1) GP zones: are small aggregations of solute (clusters) called Guiner Preston zones. These zones are formed a low temperature of aging and are formed from the segregation of Cu atoms in the supersatured solid solution α. The GP zones are composed of areas of thickening in the form of discs of a few atoms thick (0.4-0.6 nm) and they have a diameter of about 8-10 nm. Since cupper atoms have a diameter that is about 11% smaller than that of aluminum atoms, the matrix's lattice around these zone is deform in a tetragonal way. These zones are coherent with the lattice of the matrix. The GP zones can be observed with an electron microscopy thank to the strain fields that these zones create. In Figure 4 it's possible to observe a schematization of a solid solution and of a GP zone.
2) θ'': are nano-precipitates that are coherent with the matrix. Their dimensions ranging from about 1 to 4 nm thick and 10 to 100 nm in diameter.
3) θ': these phases nucleate in a non-homogeneous way especially on the dislocations, are incoherent with the matrix and have micrometric size.
4) θ: is the equilibrium phase, is incoherent with the matrix and has the composition of $CuAl_2$.

Finally there is a decrease in the mechanical strength (overaging), this is due to the coalescence of finely dispersed precipitates to form precipitates of biggest dimensions, that are visible with an optical microscope. The higher the aging temperature, greater is the activation energy available and faster are the transformation processes, with a rapid formation of precipitates of equilibrium characterized by the lowest energy level available.

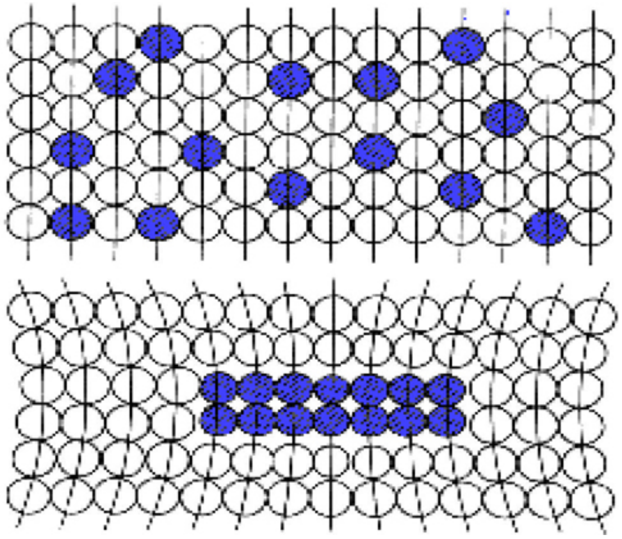

Figure 4. Schematization of a solid solution and of a GP zone.

In figure 5 to the CCT curve for a Al-Cu 4 wt.% alloy are add the curves that represent the start of the single stages of ageing, in this way is easy to individuate the optimal process's parameters: temperature and time, to conduct the ageing process with efficacy.

Too high heating rate during preheating treatment can generate dangerous levels of thermal stresses, favouring warp phenomena. Nominal commercial solution heat treating temperature is determined by the composition limits of the alloy and an allowance for unintentional temperature variation. Although ranges normally listed allow variations of ± 6 °C from the nominal, some highly alloyed, controlled toughness and high strength alloys require that temperature be controlled between more restrictive limits. Broader ranges may be allowable for alloys with greater melting range, that is the interval of temperature between their solvus and eutectic melting temperatures.

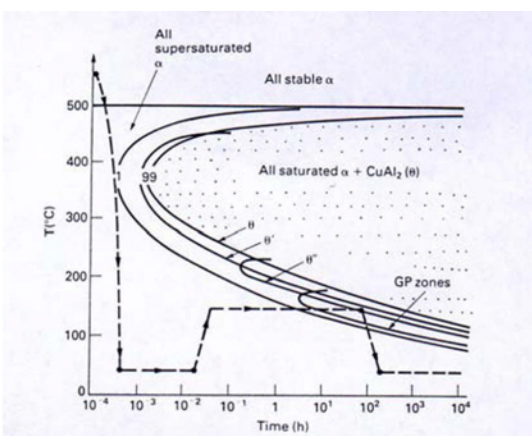

Figure 5. CCT curves for a Al-Cu 4 wt.% alloy with in addition the curves that represent the start of the single stages of ageing [11].

The effect of ageing on the reinforcement of an aluminium alloy that can be hardened for precipitation and that has been subjected to a solution quenching, is generally describe by an ageing curve, showing the mechanical strength or the hardness as a function of ageing time (normally in a logarithmic scale) at a given temperature, as shown in Figure 6 [12].

The mechanical strength of the supersaturated solid solution is indicated on the vertical axis of the graph at the time zero. With increasing the ageing time, are formed the first Guinier-Preston zones and their dimensions increase, making the alloy more resistant and hard but less ductile. If the ageing temperature is sufficiently high, the maximum mechanical strength is achieved (conditions of ageing at the peak), generally associated with the formation of an intermediate metastable precipitate. If the ageing is continued, so that the precipitates coalesce and became enlarged, the mechanical strength of the alloy decreases respect to the maximum condition, the alloy has achieved the overageing condition.

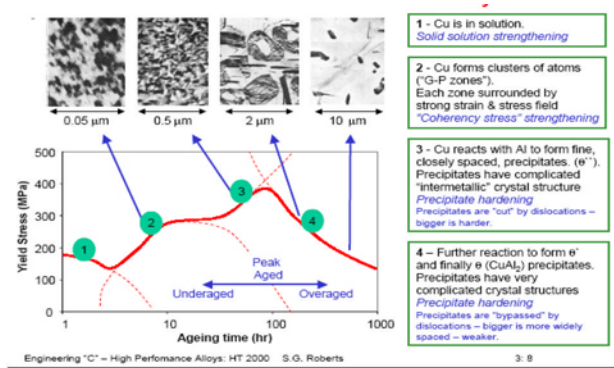

Figure 6. Ageing curve showing the mechanical strength as a function of ageing time at a given temperature and evolution of microstructures.

The kinetic of precipitation from a solid solution is function of the alloy and of the temperature, based on the values assumed by the temperature the ageing can be divided in: natural ageing conducted at room temperature and artificial ageing realized at high temperature.

Industrially the ageing is generally artificial, because being the precipitation a diffusion phenomenon, that depends in exponential way from the temperature, when the temperature is increased the time necessary for ageing decreases, and this allows to obtain a reduction of the treatment's costs.

Observing Figure 7 can be deduced as time and temperature influenced the ageing curves. At high temperatures the process is more rapid and the hardness's peak is achieved in a short time, this is due to the major diffusion rate; but on the other hand the maximum value of hardness decreases when the temperature is increases.

The compounds that can precipitated after the ageing vary with the alloys, some typical examples are:

- in the Al-Si-Cu alloys, the $CuAl_2$ phase;
- in the Al-Zn-Mg alloys, the $MgZn_2$ compound;
- in the Al-Si-Mg alloys, the Mg_2Si compound.

For as regard the Al-Si-Mg alloys the precipitation sequence starts with the formation of spherical GP zones consisting of an enrichment of Mg and Si atoms. The zones elongate and develop into a needle shaped coherent β'' phase. The needles grow to become semi-coherent rods (β’ phase) and finally non-coherent platelets (stable β phase).

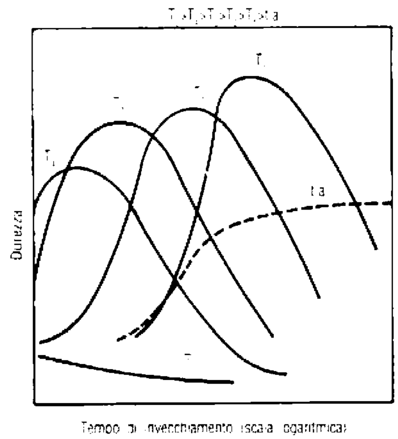

Figure 7. Hardness-time curves at different ageing temperatures.

Effects of Reheating

The precipitation characteristics of aluminium alloys must be considered frequently during evaluation of the effect of reheating on mechanical properties and corrosion resistance. Such evaluations are necessary for determining standard practices for manufacturing operations, such as hot forming and straightening, adhesive bonding, coating, painting and for evaluating the effects of both short and long term exposure at elevated temperatures.

The stage of precipitation that exists in an alloy at the time of reheating plays a significant role in the effects of reheating. Consequently, it is extremely dangerous to reheat material in a solution heat treated temper without first carefully testing the effects of such reheating [10]. It is not possible to establish general rules and indications. In the case of wrought alloys negative influences were observed during reheating of alloys in the W or T4 condition, because of further precipitation phenomena occurring during the reheating, but it was found that the strength of alloys in the T6 temper state was only slightly reduced depending on the reheating parameters. Anyway, because the behaviour to the reheating depends strongly from the alloy composition and from the particular temper state, previous and dedicated tests are always necessary.

In this paper have been evaluated the effects that two new solution heat treatments, performed respectively at 470°C for 48 h and at 470°C for 24 h, have had on the microstructure of EN AW 7068 aluminum alloy. Especially has been evaluated the possibility to reach the dissolution of Cu-rich phases, which are deleterious for the mechanical properties, through the adoption of these new solution heat treatments.

2. Experimental procedure

In table 2 is reported the chemical composition of the EN AW 7068 aluminum alloy that has been studied in the present work. Structural properties have been investigated while mechanical properties represent an on-going activity. Samples for the morphological analysis, have been extracted from components obtained by extrusion process and then have been prepared by a standard metallographic technique by mounting and polishing procedures and finally etched with Keller reagent for 15 s. The microstructure of the samples has been investigated using an optical microscope, (OM, MeF4 Reichart-Jung) and Scanning Electron Microscopy (SEM, Leo 1450VP) equipped with Energy X-rays Dispersive Spectroscopy unit (EDS, Oxford microprobe) used for compositional analysis. The microstructure obtained with a T6 heat treatment, which adopted parameters are reported in table 3, has been compared with those obtained with two new solution heat treatments (labeled in table 3 as A and B). This comparison has allowed to evaluate the microstructural differences existing and mainly evaluate the reliability of the two new solution heat treatments, to reach a completely dissolution of Cu-rich phases, which are particularly deleterious for the mechanical properties. The samples investigated in the present paper has been labeled as reported in Table 4.

Table 2 Chemical composition of the studied alloy

Chemical Composition										
Weight (%)	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Zr	Others
Min.			1.6		2.2		7.3		0.05	
Max.	0.12	0.15	2.4	0.10	3.0	0.05	8.3	0.10	0.15	0.05

Table 3 Parameters of the adopted A and B solution heat treatments and of the T6 heat treatment

Solution heat treatment	Parameters
A	470°C for 48 hours
B	470° for 24 hours
T6	475°C for 30 minutes followed by 135°c for 15 hours

On the polished samples hardness measurements have been performed using a Volpert DU01 tester. A force of 3 N has been applied for 15 s for each measurement and a minimum of 5 indentations were performed on each samples.

Table 4 Samples investigated in the present paper

Samples	Treatments
Series 1	T6 heat treatment
Series 2	Solution heat treatment A
Series 3	Solution heat treatment B

3. Results and discussion

Microstructure analysis

In figures 8 and 9 are reported the microstructures of the cross section and of the longitudinal section, respectively of all the samples analyzed in this work. The two solution heat treatments A and B have allowed to reach a finer microstructure with respect to those obtained with the T6 heat treatment, in both the cross and the longitudinal section. Moreover, especially in the longitudinal section, grains appear organized along parallel lines to the extrusion direction, in all the analyzed samples (see figure 9).

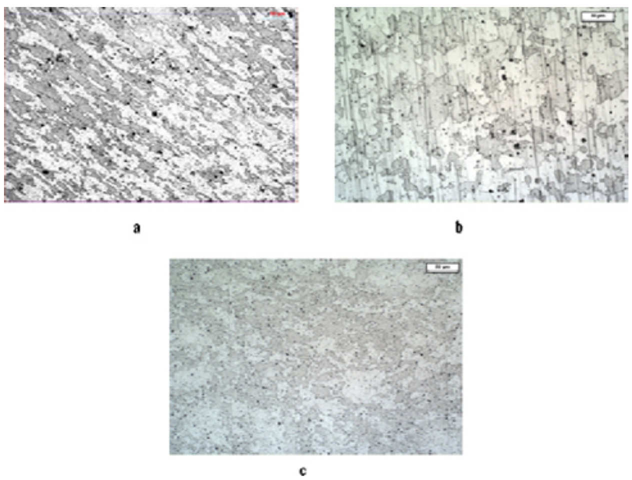

Figure 8. Optical microstructure of the cross section of semples: a) series 1; b) series 2 and c) serie 3 (200X).

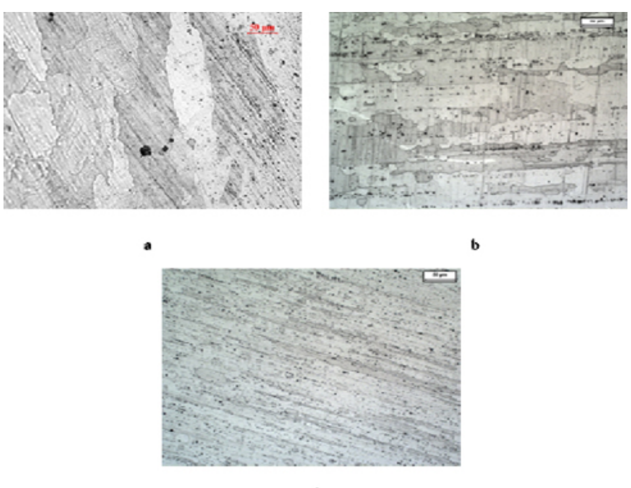

Figure 9. Optical microstructure of the longitudinal section of semples: a) series 1; b) series 2 and c) serie 3 (200X).

As can be observed, in the microstructure reported in figure 10, in the sample of the series 1 have been detected some Cu-rich phases, with an average dimension of some μm, uniformly distributed in both cross and longitudinal sections. These particles are extremely deleterious for the mechanical strength of the alloy and are probably due to the solution time used in the T6 heat treatment (30 minutes).

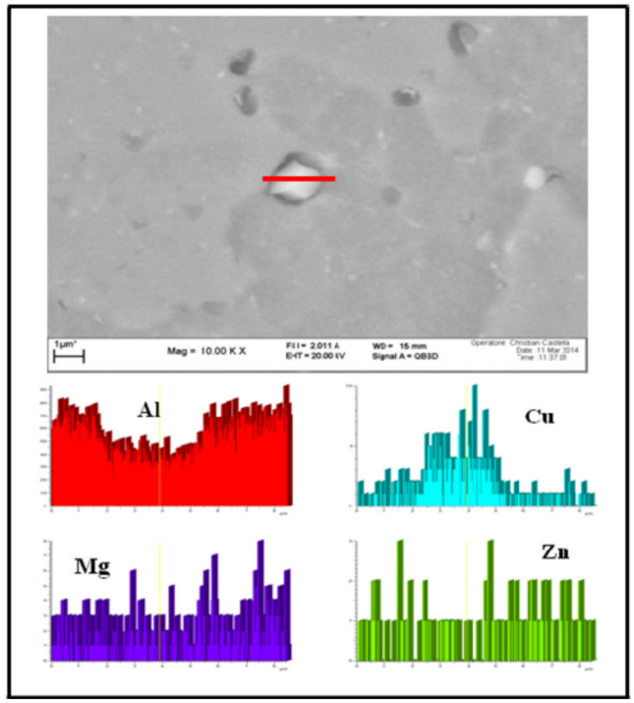

Figure 10. SEM microstructure and EDS analysis of Cu-rich phases individuated in sample of series 1.

The solution proposed, to solve this problem, has been that to increase the solution time up to 48 hours in order to encourage the dissolution of these particles. The two new solution heat treatments labeled as A and B, conducted at 470°C for 48 and 24 hours respectively, as shown in the microstructures of figure 11, have allowed to reach the dissolution of Cu-rich phases and secondly allowed to obtain a more homogeneous microstructure (see figure 10) with respect to that obtained by T6 heat treatment. An higher homogeneity of the microstructure affects positively the mechanical strength of the alloy. The solution temperature have to be lower than 485°C to avoid the incipient melting of the Cu-rich phases [13], because localized melting results in distortion and substantially reduced mechanical properties. While for as regard the time needed for dissolution and homogenization, this parameter depends on the composition, morphology, size and distribution of the phases present after solidification. From the results obtained up to now, the solution heat treatment B, resulted to be the most suitable treatment to achieve a complete dissolution of Cu-rich phases and a fine microstructure.

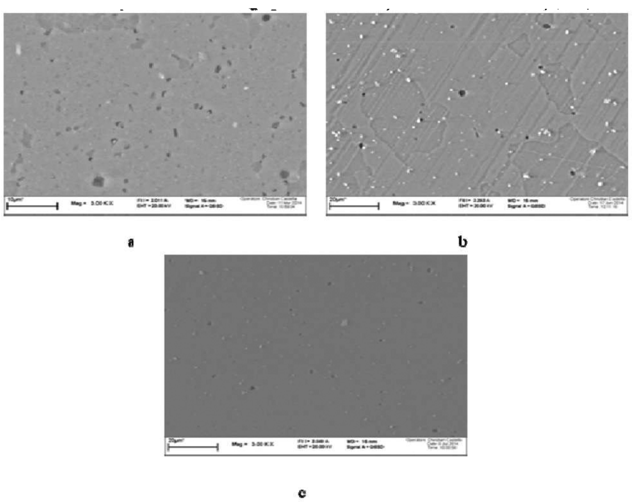

Figure 11. SEM microstructure of samples: a) series 1; b) series 2 and c) serie 3.

Moreover the solution heat treatments A and B have allowed to reach high hardness values, as can be observed from the graph of figure 12. This values represent a good starting point for the hardness, which could be further increased, through the adoption of an optimized artificial ageing step.

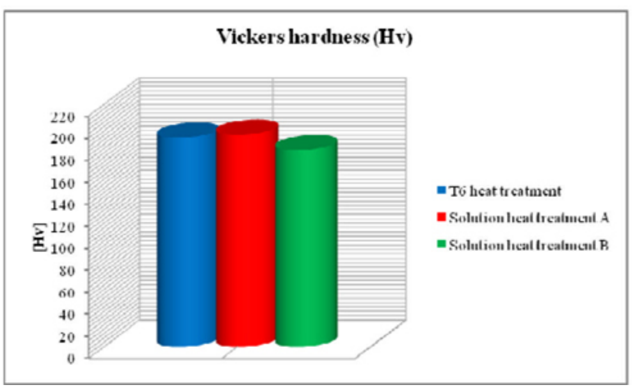

Figure 12. Vickers hardeness results.

4. Conclusions

In this paper the effects of two solution heat treatments on the microstructure of EN AW 7068 were studied. Based on the results obtained up to now the following conclusions can be drawn:

1. The microstructure of the EN AW 7068, subjected to a T6 heat treatment, has shown some Cu-rich phases, uniformly distributed in both cross and longitudinal sections. These particles are extremely deleterious for the mechanical strength.
2. With the goal to achieve the dissolution of these Cu-rich phases, two new solution heat treatments were performed at 470°C for 48 h and at 470°C for 24 h.
3. Successfully the new solution heat treatments have allowed to reach the dissolution of Cu-rich phases and also to obtain a more homogeneous microstructure.
4. The solution heat treatment B represents the most suitable solution to obtain a fine microstucture and the complete dissolution of Cu-rich phases. Moreover, with respect to the solution heat treatment A, gives rise to an economical advantage reducing the solution time from 48 h to 24h.

5. References

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