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DEVELOPMENT OF AN ACCELERATED AGEING TEST ON AN Al-Si-Cu-Mg ALLOY FOR AERONAUTICS

L. Grosset[1, 2], C. Desrayaud[1], A. Fraczkiewicz[1], C. Bosch[1], L. Anssems[2], B. Guérin[2], S. Becquerelle[3]
[1]École Nationale Supérieure des Mines de Saint-Étienne, Laboratoire Georges Friedel, CNRS UMR 5307, 158 Cours Fauriel CS 62362, 42023 Saint-Étienne Cedex 2, France
[2]Hispano-Suiza (Groupe Safran), Direction Technique Matériaux et Procédés, 18 Bd Louis Seguin, 92707 Colombes Cedex, France
[3]Hispano-Suiza (Groupe Safran), Recherche et Technologie, 18 Bd Louis Seguin, 92707 Colombes Cedex, France

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Abstract

Because of the evolution of structural requirements for some aircraft components, which are required for longer life time and high temperature conditions operation (up to 90 000 hours at up to 250°C), developing an accelerated ageing process becomes essential to observe and understand the ageing of aluminum alloys and predict the influence of such process on the mechanical behaviors of these alloys under temperature. The alloy considered in this study is an as cast Al-Si-Cu-Mg aluminum alloy.
In this presentation, microstructures and mechanical properties of the material in its initial non-aged state are compared to those obtained after different ageing treatment. Changes in mechanical properties are related to changes in terms of precipitation through precipitate size. Higher temperature conditions accelerate the ageing phenomenon and thus enable us to reach an infinite ageing state from which, with a time – temperature equivalency, we will be able to build a computer model of accelerated ageing.

Introduction

Since the mid-nineties, transportation is the bigger market for aluminum in the western world before packaging, construction and other application such as electricity, mechanics, electronics etc. [1]. Among aluminum alloys, foundry alloys had their development intensified due to their high strength to weight ratio (2.6 to 2.8), as well as their good castability, corrosion resistance and recycling [2], [3]. The current context in aeronautics is such that requirements in terms of designing aircraft parts are constantly changing. Al-Si-Cu-Mg alloys are used for structural parts in engine power transmissions, such as accessory gearboxes, required for longer life time and high temperature conditions (up to 90 000 hours at up to 200°C – 250°C).
In the literature, many authors such as Sjölander and Seifeddine [4], [5], Alexopoulos [6] or Elsebaie *et al.* [7] have studied ageing of foundry alloy but over relatively short ageing periods. Current industrial practices are thus to carry out ageing in ovens for periods up to 20 000 hours and to mechanically test the parts, a process which represents a relatively heavy experimental campaign that industry would like to lighten. The goal is to develop a model of accelerated ageing that can closely reproduce the actual flight conditions of aircraft parts.
This study focuses on the mechanical and microstructural properties of an Al-Si-Cu-Mg cast alloy, and on their evolution in response to different over-ageing conditions. In his work on a 319 foundry aluminum alloy, Martinez [8] has studied ageing treatment up to 1 000 hours and proposed a theoretical model founded on the coarsening modeling of Al_2Cu precipitates in the α-phase which gives results close to TEM measurements and tensile tests, and found that mechanical properties such as yield stress is a function of the precipitation state and is time-temperature dependent. Thus, the microstructural parameters that will be under consideration here are the strengthening precipitates. The precipitation sequence for Al-Si-Cu-Mg precipitates is quite complex and not well defined in the literature, but can be summarized as follows [9]:

$$_{SS} \rightarrow GP, GPB\ Zones \rightarrow \begin{cases} \theta'' \\ \beta'' \\ Q'' \end{cases} \rightarrow \begin{cases} \theta' \\ \beta' \\ Q' \end{cases} \rightarrow \begin{cases} \theta - Al_2Cu \\ \beta - Mg_2Si \\ Q - Al_5Cu_2Mg_8Si_6 \end{cases}$$

Clusters of atoms form rapidly from the supersaturated matrix and evolve into GP zones [10], [11], which rapidly dissolve to form metastable coherent and semi-coherent precipitates, and finally stable phases [9], [12]. The growth of these precipitates is controlled by atoms diffusion in the aluminum matrix [13].
Scanning Electron Microscopy (SEM) technique will be used to follow this microstructural evolution, while mechanical properties will be determined by tensile tests.

Material And Experimental Procedures

Material

The material used in this study is a commercial cast Al-Si-Cu-Mg alloy, which composition is given in Table I. All the test samples were taken into plates of 325 mm x 325 mm x 22 mm from the same casting ingot. These plates were made by SFU smelter using precision sand casting process, and they were then solution heat treated, water quenched and finally artificially aged at 200°C during 8 hours to reach a T1R2 temper. The role of the solution heat treatment is to homogenize the as-cast structure, dissolve intermetallic phases and change the eutectic Si morphology [9].

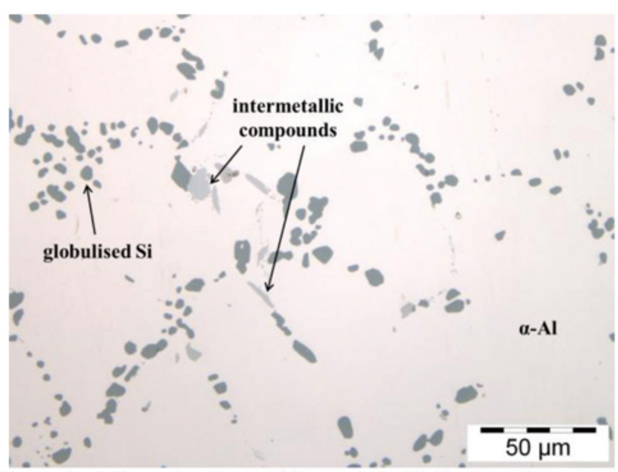

Figure 1 : Microstructure of the cast Al-Si-Cu-Mg alloy

The microstructure of the alloy is shown in Figure 1, and consists of α-Al dendrites and globular Si eutectic [14], along with intermetallic compounds. These compounds contain iron and exist in two forms: β-Al_5FeSi phase which is needle or plate shaped, and $Al_{15}(Fe,Mn)_3Si_2$ which looks like "chinese scripts" [8], [15], but that are not detected here.

Table I : Chemical composition of the Al-Si-Cu-Mg alloy (mass percent)

Elements (wt. %)	*Cu*	*Mg*	*Si*	*Ti*	*Fe*	*Mn*	*Others*	*Al*
min	0.7	0.45	6.5	0.08	-	-	-	bal.
max	1.7	0.8	7.5	0.2	0.1	0.05	0.05	

Experimental Procedures

This study aims at comparing microstructural and mechanical properties of the alloy in its initial state, i.e. T1R2-tempered, and after over-ageing. Plates of the studied alloy were T1R2-tempered and subsequently subjected to isothermal exposure treatment in furnaces, at temperatures ranging from 120°C to 200°C for different times up to 5 000 hours, to simulate the ageing of aircraft parts in service.
Tensile tests were carried out on an Instron 1186 machine at room temperature, with a deformation rate of 0.01 $mm.s^{-1}$. An extensometer was used on flat tensile specimens to measure ultimate tensile strength R_m, yield strength at 0.2% strain $R_{p0.2}$ and elongation to fracture A%. Scheme of the specimen is presented in Figure 2.
The microstructures of the materials are studied using an optical microscope and scanning electron microscopes JEOL 6500 and ZEISS. All the samples for microstructural analysis are cold mounted and polished with sandpaper and then finish with a 1 µm diamond paste to reach a mirror state. For SEM observations, a slight etching with colloidal silica is made to reveal the microstructure.
Note that the Secondary Dendrite Arm Spacing (SDAS) is also measured on the different alloys to check its evolution over ageing conditions. The measurement is made by manual counting of dendrite arms; a mean value is calculated for each aged alloy based on ten experimental values.
Finally, accelerated ageing tests were made in furnaces at 250°C for different times, and hardness measurements were carried out to follow the evolution of mechanical properties. A sample of the Al-Si-Cu-Mg alloy is placed in the oven for a given time, then water quenched before carrying out the hardness measurements, and then replaced in the oven to continue thermal ageing treatment. Table II summarizes the characteristics of each test, and Figure 3 presents the thermal cycle experienced by the sample during the first (A) and the third test (C). SEM observations are also made on the material at the end of each accelerated ageing treatment.

Table II : Total exposure time and frequency of hardness measurements for the three accelerated ageing test

Test	*T°C*	*Total exposure time*	*Hardness measurement*
A		48 hours	Every hour
B	250	384 hours	Every 24 hours
C		2 hours	Every 15 minutes

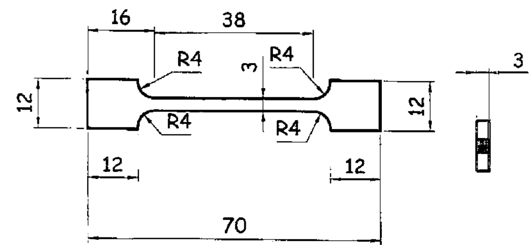

Figure 2 : Scheme of flat tensile specimens (mm)

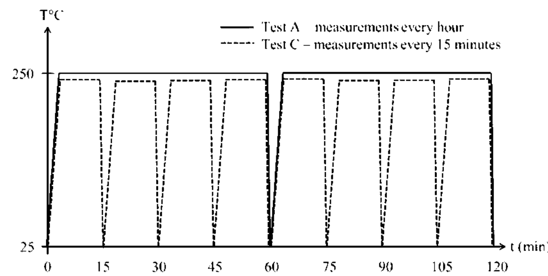

Figure 3 : Thermal cycle undergone by the Al-Si-Cu-Mg alloy on a two-hour period for accelerated ageing test A and C

Results And Discussion

Evolution Of The Secondary Dendrite Arm Spacing (SDAS)

The SDAS value has an influence on mechanical properties, as a smaller SDAS gives higher mechanical properties, just like for grain size [16]–[18]. No specification is provided on the SDAS except that it must guarantee the desired mechanical properties. Typical values are around 40 µm, and values above 60 µm or 70 µm can compromised some mechanical characteristics. The SDAS is not supposed to evolve during any aging treatment, for exposure temperatures between 120°C and 200°C. That is why it is important to check this point. Figure 4 presents this evolution of SDAS over exposure time at different temperatures.

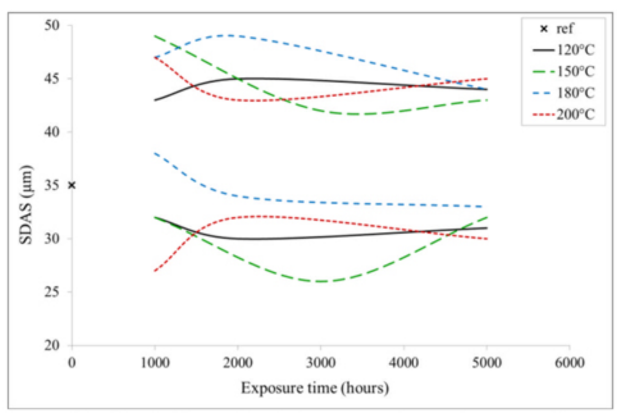

Figure 4 : Evolution of minimum and maximum values of SDAS over exposure time at different temperatures

The reference Al-Si-Cu-Mg T1R2-tempered alloy has a SDAS mean value of 35 µm, giving mechanical strengths of around 350 MPa for tensile strength and 330 MPa for yield strength. Figure 4 shows that all the SDAS values measured for any thermal exposition are between 26 and 49 µm, which are common values for casting alloys. Fluctuations between values are only due do measurement uncertainty. This confirms the hypothesis that SDAS do not evolve during aging treatment.

Mechanical Behavior After Aging Treatments

Martinez reported that the mechanical properties of a 319 foundry alloy dramatically fall after aging at elevated temperature [8]. Figure 5 presents the mean tensile curves of the Al-Si-Cu-Mg alloy aged at different temperatures, for different exposure times.

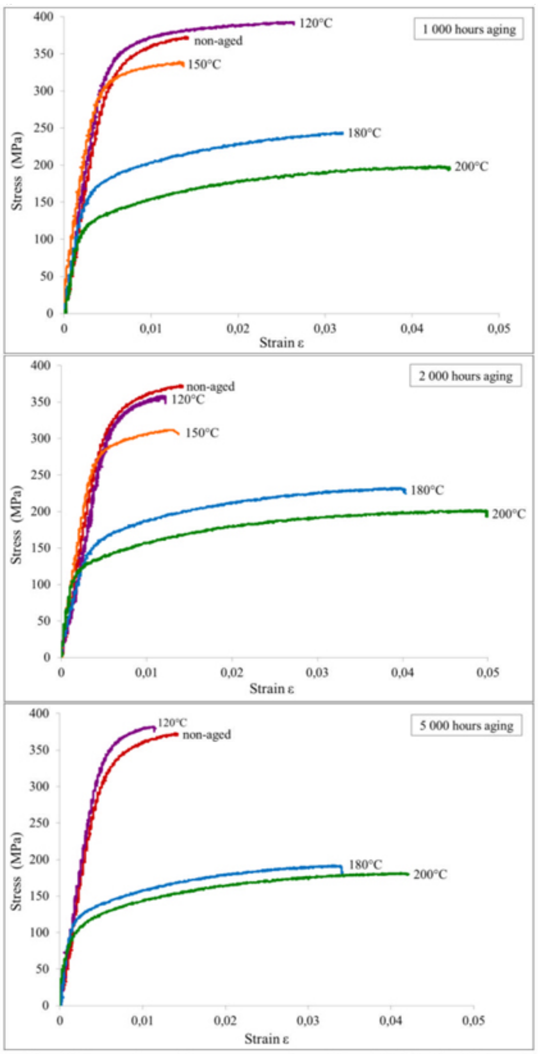

Figure 5 : Mean tensile curves for the Al-Si-Cu-Mg alloy at room temperature for the alloy aged at different temperature for 3 exposure times obtained with flat tensile specimens

Two trend stand out from the curves of Figure 5:
The desired mechanical properties are maintained up to around 150°C whatever the exposure time, however with a slight reduction of 10% - 20% with the exposure time at 150°C.
At 120°C, this is quite different. The exposure of the material at such temperature puts the alloy under peak hardening because of its T1R2 heat treatment, and is actually strengthening the materials, which results in a slight increase in R_m and $R_{p0.2}$, or at least in the same order of magnitude. Fluctuation in elongation at break is due to the variability of A% measurements commonly observed in tensile tests. Testing with cylindric specimens give slightly higher values of A% but still with fluctuation in measurements.
Over 180°C, mechanical characteristics drop considerably with a reduction of over 50% of R_m and $R_{p0.2}$. At elevated temperature, this loss of strength is offset by a gain in elongation to fracture, which is in agreement with Garat and Jacob observations [16], [19]. Fluctuation in A% is still present, but it is clear that elongation to fracture increases with temperature and time exposure, as plastic deformation becomes easier.

Microstructural Observations

For reminder, the microstructure of the Al-Si-Cu-Mg alloy is as illustrated in Figure 6. When microstructural observations are conducted at the same scale on the samples after over-ageing, no apparent changes are visible on the different micrographs. See for example Figure 7 representing the microstructures of the alloy aged at different temperatures for 5 000 hours.

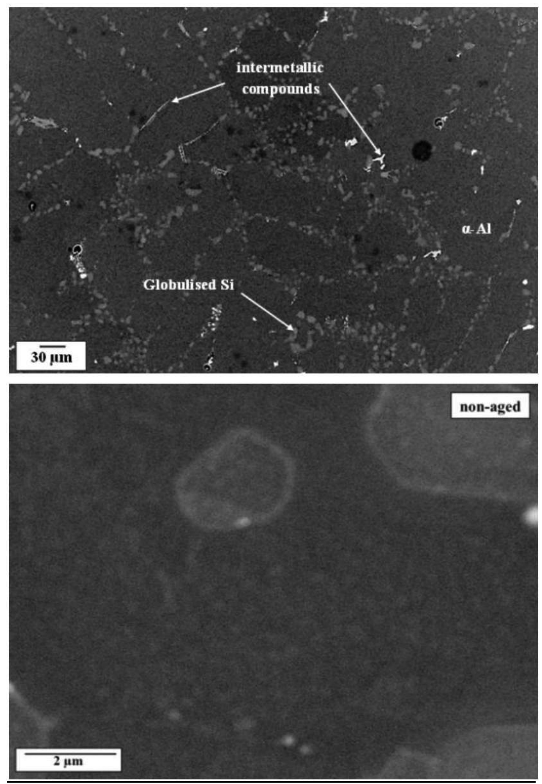

Figure 6 : SEM image of Al-Si-Cu-Mg alloy T1R2-tempered without additional ageing at medium (top) and high magnification (bottom)

Losses in mechanical properties necessarily come from microstructure evolution, through the presence of hardening precipitates, which are generally micrometer or nanometer – sized. Although nanometric resolution is not reachable with the SEM, the idea was then to investigate the microstructure more deeply to see what is happening. To achieve this, the working distance between the sample and the electron beam is minimized in order to achieve a good chemical contrast using backscattered electrons, and images are taken at high magnification. The results of these observations are presented in Figure 8.

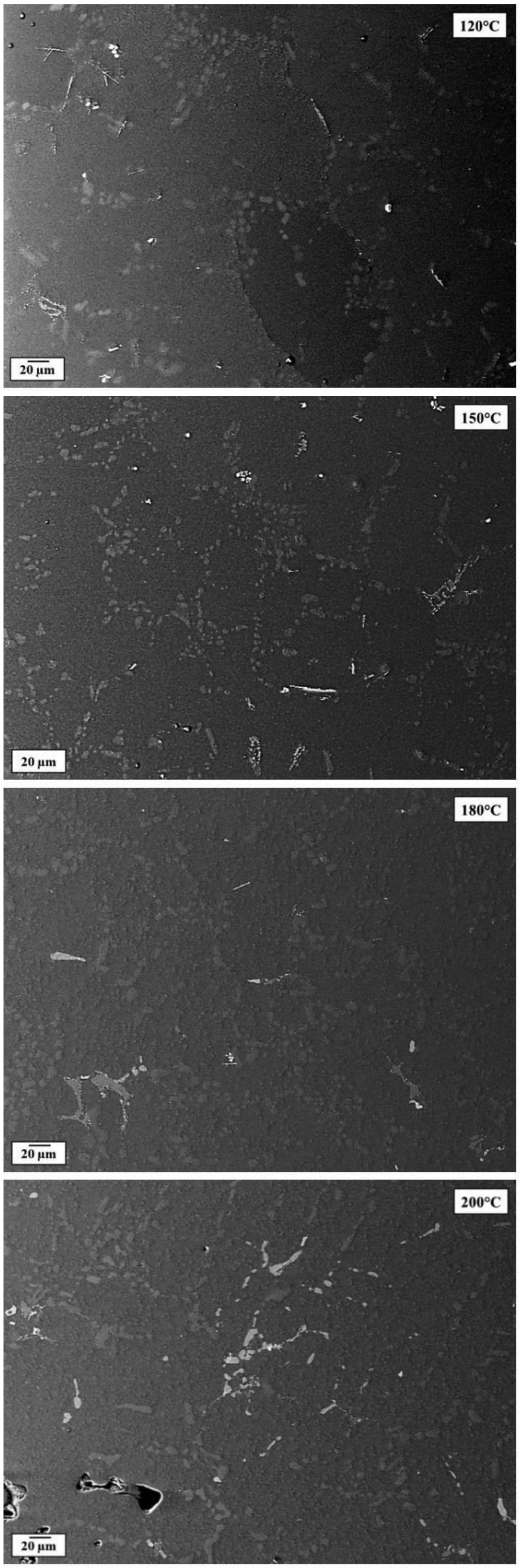

Figure 7 : SEM images of Al-Si-Cu-Mg alloy aged at different temperatures (120°C, 150°C, 180°C and 200°C) at medium magnification (x500)

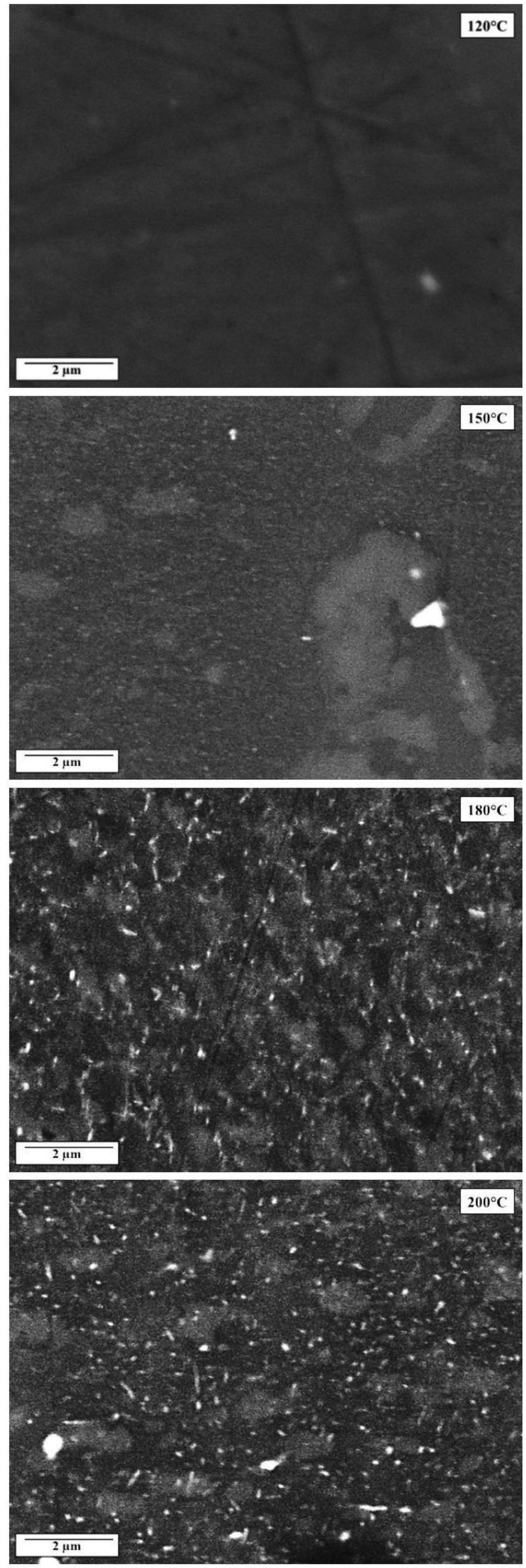

Figure 8 : SEM images of Al-Si-Cu-Mg alloy aged at different temperatures (120°C, 150°C, 180°C and 200°C) at very high magnification (x20000)

At 120°C and 150°C, SEM images are not relevant, whereas at 180°C and 200°C, very fine precipitation is observed, with precipitates smaller than 1 µm (about 100 nm to 800 nm). Over 180°C, it appears that the detection limit of the fine precipitation is reached. This does not mean that the fine precipitation does not occur on the alloys aged at 120°C and 150°C, but that this precipitation is too small at this point to be detected by the SEM. It shows the beginning of the coarsening of hardening precipitates that become bigger and so visible through a scanning electron microscope. Moreover, this detection limit coincides with the drop of ultimate tensile strength and yield strength noticed in Figure 5.

First Tracks To The Establishment Of An Accelerated Ageing Test

The objective of this test is to overcome the heavy experimental campaign, which engages relatively long exposure time under temperature. A model based on a time-temperature equivalence would then be suitable, provided that the thermal treatment conditions (in service and accelerated ageing) only generate dissolution and coarsening mechanisms.
The ageing test is here accelerated by applying higher temperature, which cannot exceed 250°C in the case of aluminum alloys. Hardness measurements were performed on samples of the different tests because hardness is an effective and non-destructive means to first estimate mechanical behavior of the alloy.
First test was conducted in order to have a first trend of the evolution of mechanical properties of the Al-Si-Cu-Mg alloy, at high temperature (250°C). Hardness measurements were carried out over a period of 48 hours, the corresponding curve is plotted in Figure 9-*A*. A gradual drop in hardness is observed, with a loss of 45% in hardness after 48 hours relative to the initial value. This is a consequence of coarsening phenomenon, the nano-sized hardening precipitates grow and no longer participate in the strengthening of the alloy.
A second test was then performed at the same temperature but over a much longer period to reach an asymptotic state where the hardness no longer evolves. Thus, the Al-Si-Cu-Mg alloy has undergone a heat treatment at 250°C for 384 hours, or 16 days as seen in Figure 9-*B*. One can note a significant drop of hardness during the first four days (96 hours) followed by a gradual stabilization of the latter, to finally reach a hardness level at 9 days (216 hours) of thermal exposure. This bearing means that the alloy has achieved a thermal equilibrium state, where there is no more competition between coarsening and dissolution phenomenon.
Finally it seemed of interest to study the early stages of ageing at 250°C, and the third test was conducted by raising hardness measurements every 15 minutes for 2 hours, reported in Figure 9-*C*. The results are a bit surprising, showing constant hardness values hovering around the initial value, while a slight decrease in hardness was expected instead for a two-hour period. This phenomenon results from a simultaneous competition between coarsening and elements dissolution. Indeed, Figure 9 shows that during short maintaining (about 10 minutes), one must take into account the time for temperature to rise into the sample.
This result is important because it shows that continuous exposure to temperature is not fully equivalent to a stepped thermal cycle for short times (in the order of a minute). This will be of considerable interest for aviation companies which often based the parts structural requirements on thermal cycling.
If microstructural data of these accelerated ageing are compared to those of the previous alloys aged at 180°C and 200°C, the fine

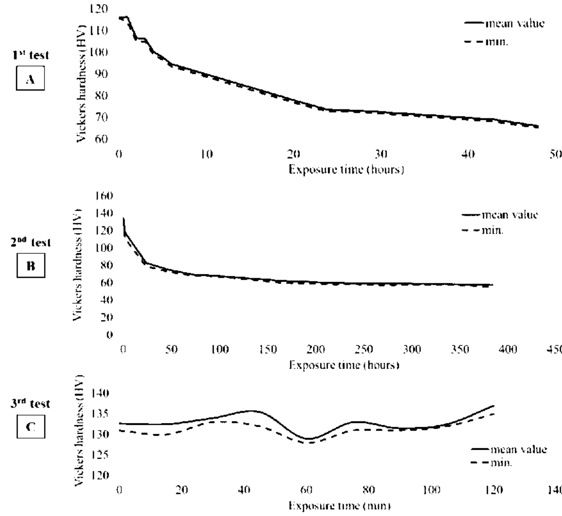

Figure 9 : Hardness evolution over time for the Al-Si-Cu-Mg alloy exposed to an ageing test at 250°C for 3 different exposure times A: 48 h – B: 384 h or 16 days – C: 2 h ***NB: changes in initial hardness values are due to a slight hardening of the alloy during its storage***

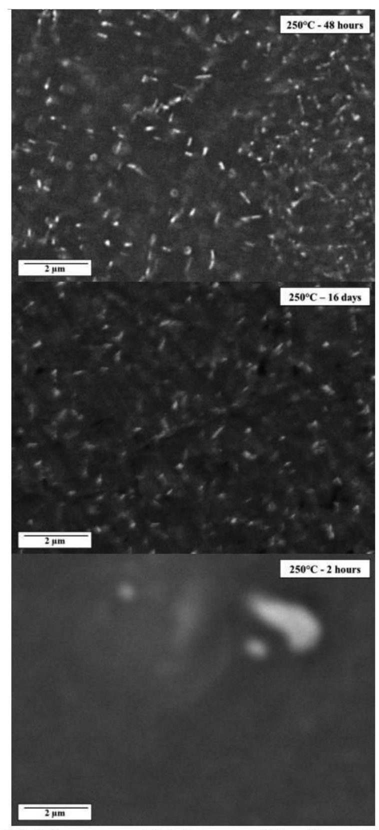

Figure 10 : SEM images at high magnification of the Al-Si-Cu-Mg alloy for each accelerated ageing test

precipitation of hardening precipitated is detected after 48 hours of ageing at 250°C, but not after only 2 hours.
This is in agreement with evolution of hardness measurements: as soon the fine precipitation is detected by SEM, a drop in hardness, and so in mechanical properties, is observed. Moreover, we note

that after 16 days of ageing at 250°C, the fine detected precipitation appears to present larger but less numerous precipitates (300-500 nm), result of the coarsening which may be a final equilibrium state usable to establish our accelerated ageing model.

Conclusions

Mechanical properties of an industrial casting Al-Si-Cu-Mg alloy were investigated over various ageing treatments, and have been linked to microstructural evolutions.

First, secondary dendrite arm spacing (SDAS) was found not to evolve during the different ageing treatments experienced by the studied alloys, meaning that the SDAS is not a factor to monitor during ageing in flight conditions.

Concerning static mechanical properties, two trends were revealed: Up to 150°C, there is no significant change in the microstructure at the micrometer level, and therefore no huge impact on mechanical properties. The alloy keeps mechanical properties suitable with the company specifications, but with a slight reduction between 10 and 20 % in R_m and $R_{p0.2}$, due to the early beginning of coarsening. Then, at higher temperature, over 180°C, a progressive drop in mechanical properties is observed with a 50 % loss in tensile strength and yield strength, offset by a gain in elongation at break. About microstructural changes, a fine precipitation of hardening Al_2Cu and Mg_2Si precipitates could be detected, which means that precipitates keep coarsening over a critical size where they are no longer efficient in the alloy strengthening, significantly reducing mechanical properties of the Al-Si-Cu-Mg alloy.

Regarding the accelerated ageing, the tests have shown that at a fairly high temperature, of 250°C for aluminum alloys, the drop in hardness occurs very rapidly during the first few hours of ageing. Then the hardness stabilizes to reach a hardness bearing around 9 days of thermal ageing.

SEM technique turned out to be very interesting and useful through the observation at high magnification of the fine precipitation coalescence, witnessing the decline in mechanical properties.

Prospects of this study are to draw a parallel between the observable fine precipitation using scanning electron microscopy and the same precipitation analyzed more quantitatively (precipitates nature and volume fraction) by transmission electron microscopy to build a model of accelerated ageing.

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