

Changes in Lattice parameter in Aluminium cast Alloys due to aging

Cambios en el Parametro de Red en Aleaciones de Colada debido a su Envejecimiento

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Abstract

Aluminium alloys are widely used due to their combination of low weight and high strength resulting from heat treating, which takes place by heating-up the material at temperatures high enough for the alloying elements to dissolve into the aluminium matrix; the material is then cooled-down to room temperature at a rate fast enough for the alloying elements to remain in solution; strengthening occurs by aging, in which particles of different size, shape and nature precipitate from the supersaturated matrix promoting changes in the lattice parameter of the aluminium. X-ray diffraction analyses were conducted in different aluminium cast alloys after aging them at different temperatures for up to 100 hours, revealing that changes in the value of the lattice parameter of the aluminium matrix depends on the alloying elements. Aluminium- copper alloys have the tendency to increase the parameter due to precipitation of Al₂Cu particles, whereas those that harden by precipitation of Mg₂Si are affected to a lesser degree.

Resumen

Las aleaciones de aluminio son ampliamente usadas por su combinación de bajo peso y alta resistencia mecánica resultante de su tratamiento térmico, que se lleva cabo al calentar al material a temperaturas suficientemente altas como para disolver a los elementos de aleación en la matriz de aluminio; el material se enfría a temperatura ambiente a una tasa lo suficientemente alta como para asegurar que los elementos aleantes permanezcan en solución; el endurecimiento se produce por envejecimiento, en que partículas de diferente tamaño, forma y naturaleza precipitan de la matriz supersaturada promoviendo el cambio en el parámetro de red del aluminio. Análisis de difracción de rayos X se realizaron en diferentes aleaciones de colada de aluminio a diferentes temperaturas por hasta 100 horas, revelando que los cambios en el valor del parámetro de red de la matriz de aluminio depende de los elementos aleantes, siendo aquellas con cobre las que tienden a incrementar su parámetro por la precipitación de partículas de Al₂Cu; aquellas que endurecen por la precipitación de Mg₂Si tienen un efecto menor.

Keywords:

Aluminium, heat treating, lattice parameter, X-ray diffraction

Introduction

Aluminium alloys are preferred materials when light weight and high mechanical demands are required, as is the case for transport industry, such as automotive and aeronautic. Environmental agencies all over the world have established the normative to reduce fuel consumption and emission of greenhouse effect gases in light vehicles [1]. However, CO₂ emissions from the transportation sector in the European Union increased in a 17% in the period from 1990 to 2014, as light passenger transport vehicles increased their emissions in 12%, whereas heavy load transport vehicles doubled it [2]. Several automotive companies have turned into aluminium to replace parts and pieces originally made and designed to be manufactured from iron or steel; replacement of cast iron by aluminium alloys has become commonplace for

Palabras clave:

Aluminio, tratamiento térmico, parámetro de red, difracción de rayos X

parts such as cylinder heads, engine blocks and manifolds [3,4]. An additional advantage that takes place in the use of aluminium alloys in engine power train components is that of the enhancement of the power rating. This is due to the higher thermal conductivity of these alloys, in comparison with cast irons, that allows for higher working temperatures in the combustion chamber, which contributes in delivering more power per cylinder size while requiring lower amounts of coolant liquids [5-7].

Changing from iron to aluminium has not been an easy task for industry, as it is required for aluminium parts to withstand more stringent conditions than those to which iron castings are subjected, which are promoted by the higher working temperatures of the engine that enhance the reduction in strength of the material at the time that increases the

possibility of promoting thermal fatigue [3,8,9]. Cast aluminium alloys may be produced from commercial primary ingots or from scrap, due to the strong policy in reducing costs of the automotive industry. This problem is of particular interest for Al-Si-Cu alloys, which may have considerable amounts of residual elements (mainly Fe, Mn and Zn). Al-Si-Mg and Al-Cu alloys are normally prepared from primary ingots and are cast with lower contents of residual elements [10,11]. Casting is carried out by pouring the liquid metal into a mould made from materials ranging from sand to high strength tool steel, which modify the solidification and cooling rates that affect the microstructure of the solid alloy. The microstructure in silicon containing alloys is made of primary aluminium dendrites surrounded by the Al-Si eutectic aggregate. Mg is added to some alloys to strengthen by precipitation of Mg_2Si particles, but should be avoided in castings hardened by Al_2Cu precipitates, as these elements tend to form a low melting point eutectic that takes place at temperatures below 500°C. Modifying elements are added to change the aspect of the Al-Si eutectic by promoting the formation of fibrous silicon branches; titanium and boron are added to refine grain size by the nucleation of new grains in front of the solidifying front; the presence of tramp elements from scrap promotes the formation of different intermetallic phases [3,4,10-14]. Al-Cu alloys are developed for their enhanced mechanical properties that are able to withstand the higher pressures that advanced engines have to sustain [15].

Mechanical properties in aluminium cast alloys that contain copper and silicon and magnesium can be enhanced by heat treating. This treatment proceeds by heating the material to temperatures at which either copper or silicon and magnesium are dissolved; the material is cooled down to room temperature at a rate that will maintain the supersaturated solution. The final stage in aging consists in keeping the material at a temperature high enough to allow precipitation from the supersaturated solution. The alloys after solution will be cooled down to room temperature at a rate fast enough to maintain the alloying elements in solution, and this is conducted by immersing the material in water or by the use of forced air or steam. Precipitation of the hardening particles will proceed during aging [3,4,10-5]. The desired aging condition will also depend on the type of alloy; T7 treatment is preferred in Al-Si-Cu and Al-Cu alloys, as this condition allows achieving the strength requirements in powertrain applications, whereas Al-Si-Mg alloys have to be treated to a T6 condition to achieve similar values of strength. T7 condition is also referred as stabilization, and this is due to the changes in dimensions that occur during heat treating [16,17]. Copper dissolves in the matrix during the solution treating and precipitates while aging promoting dimensional changes, a full discussion on this phenomenon is found elsewhere [18]. It is a well-known fact in cast shops that copper bearing aluminium alloys grow when treated to T6, and reduce their size while in operation during overaging [16,17]. Al-Si-Mg alloys do not exhibit this behaviour as the effects of Mg and Si oppose each other [18].

The changes that take place in the mechanical properties during aging are normally followed by means of tensile or hardening tests [3,4,10-12,16,17], however, X-ray diffraction has been used to evaluate the effect of precipitation in aluminium alloys [19-21]. The aim of this work is to present the results of a study conducted to evaluate the effect that aging exerts on cast aluminium alloys aged at different temperatures for up to 100 hours by means of the analyses of X-ray diffraction spectra, aging was also be evaluated by microhardness tests.

Experimental procedure

A series of experimental alloys were prepared in a 250 kg gas-fired furnace. The melt was degassed with nitrogen before adding master alloys of titanium and boron, to refine the grain size, and strontium to modify the Al-Si eutectic aggregate. Liquid metal was poured at 740°C into silica sand moulds designed to impose a one-direction thermal gradient by a grey iron chill plate at their bottom [15,22]. The samples used in the present study were cut from the bottom of the ingot to assure that the secondary dendrite arm spacing (SDAS) in the Al-Si alloys varied within the 16-21 μm range. The selected experimental alloys harden by the precipitation of Al_2Cu , **A**, **B** and **D**, and Mg_2Si , **C** and **D**; their chemical composition is shown in Table 1.

Table 1. Chemical composition of the alloys

Element	Alloy			
	A	B	C	D
Si	0.02	8.1	7.4	7.2
Cu	4.9	3.1	0.01	0.47
Mg	0.31	0.32	0.41	0.32
Fe	0.15	0.69	0.15	0.18
Mn	0.2	0.4	0.06	0.9
Ti	0.05	0.18	0.18	0.14
Sr	0	140	140	140
B	30	30	20	20

Contents in wt.%, except for Sr and B that are in ppm

Samples of the alloys were solution-treated following different cycles. Alloy **A**, Al-Cu, was treated for 2 h at 495°Cs followed by 2 h at 520°C and another 12 h at 535°C; alloy **B**, Al-Si-Cu, was solution treated for 5 h at 495°C, whereas alloys **C**, Al-Si-Mg, and **D**, Al-Si-Mg-Cu, were held for 5 h at 530°C. All the samples were cooled-down to room temperature by immersing them in water at 90°C; aging was conducted at 150 and 240°C for up to 100 h.

The metallographic examination of the samples was conducted following standard procedures down to fine polishing (1 μm alumina) prior to their X-ray diffraction analyses. Preliminary runs were made in the 2θ interval of 20 to 90° to identify the various peaks, and in the interval of 77° to 80° to evaluate the shift of the {311} peak. The

step was of 0.02° in all cases with a 13 s time step; $K\alpha$ copper radiation, $\lambda=0.15418$ nm, was used.

The samples were etched after diffraction to reveal the microstructure using a solution of 5% HF in water for alloy A, and a solution of 5 ml HNO_3 , 3 ml HCl and 2 ml HF in 190 ml H_2O for the other alloys. Vickers microhardness tests were carried out using a 1 kg force for 15 s.

Results and discussion

Figures 1 and 2 show the microstructures of the four alloys in their as-cast condition. It is appreciated that solidification of the Al-Si alloys proceeds through dendrite growth, B, C and D, whereas that of the Al-Cu, A, occurs by cellular growth. Solution treatment contributes to dissolve some of the particles that formed during solidification [3,4,12]. The microstructures observed in the specimens after aging for 100 h at 170 and 240°C are shown in Figures 3 and 4 respectively. The main microstructural phenomenon that can be appreciated is the spheroidization of the silicon particles from the eutectic, in the Si-Al alloys, and the Cu-rich particles, in the Al-Cu alloy, due to the long aging time [16,23]. The changes in hardness as a function of time, aging, at 170 and 240°C are shown in Figure 5, in which the typical behaviour of age-hardening materials is exhibited [10,12].

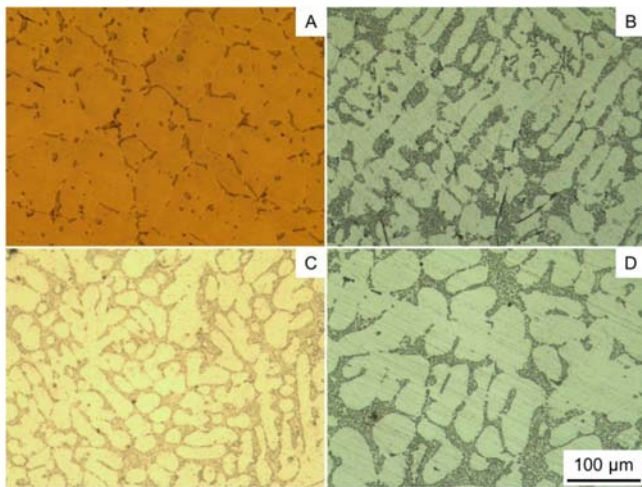


Figure 1. Microstructures of the alloys in their as-cast condition

Diffraction spectra were obtained from as-cast samples in the 2θ interval of 20 to 90° , Figure 6. The interval of analysis in aged samples was reduced and high definition scans were taken from 77 to 80° to evaluate the shift of the $\{311\}$ peak in the different alloys, as previous analyses showed the sensitivity of these peak to aging [20]. Figures 7 and 8 show the diffraction charts for selected times obtained in samples from the four alloys.

The angular position at which the maximum value of the $\{311\}$ peak was obtained from the diffraction tests in the restricted range and, from it; the lattice parameter of the aluminium in the alloys was computed. Table 2 summarizes the results of the analyses on the $\{311\}$ peak shift, data from a wrought alloy [20] are included for comparison. Figures 9

and 10 show, respectively, the position of the maximum value of the $\{311\}$ peak and that of the lattice parameter computed from this angular position; the straight lines drawn in these figures correspond to the values obtained in the solution treated samples. Changes in the lattice parameter reported in a wrought 2.3% copper containing alloy [24] are compared with those of alloy A, which contains 4.9%, in Figure 10.

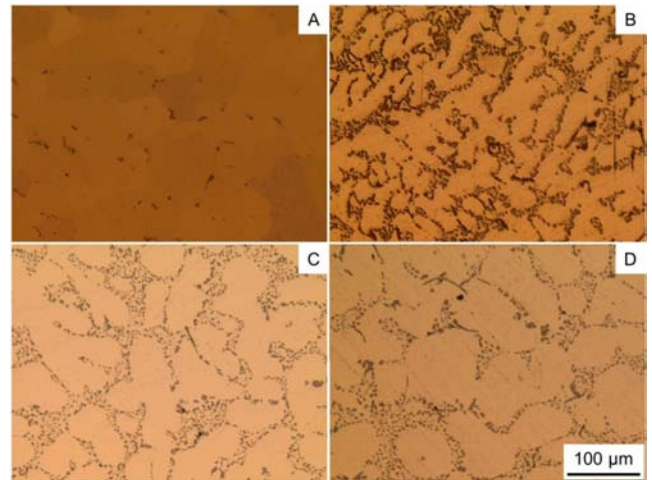


Figure 2. Microstructures of the alloys after their solution treatment

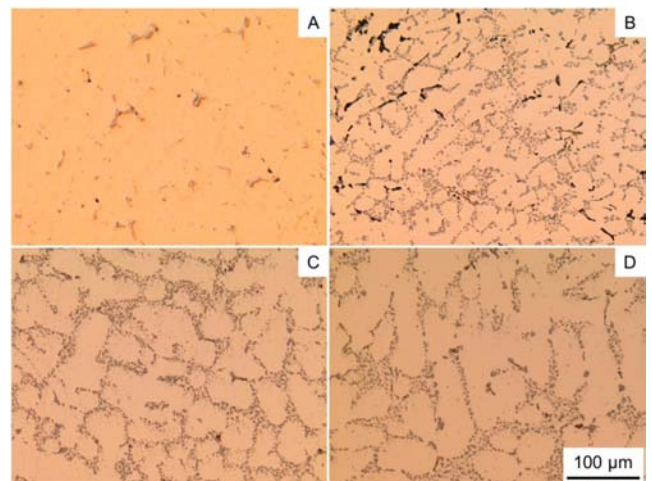


Figure 3. Microstructures of the alloys after aging for 100 h at 170°C

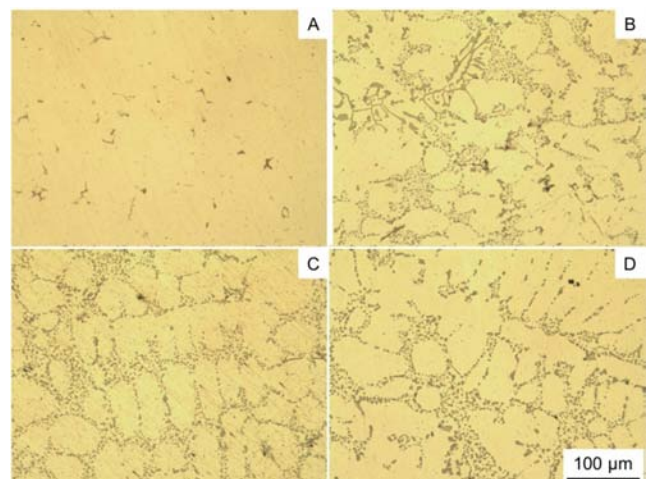


Figure 4. Microstructures of the alloys after aging for 100 h at 240°C

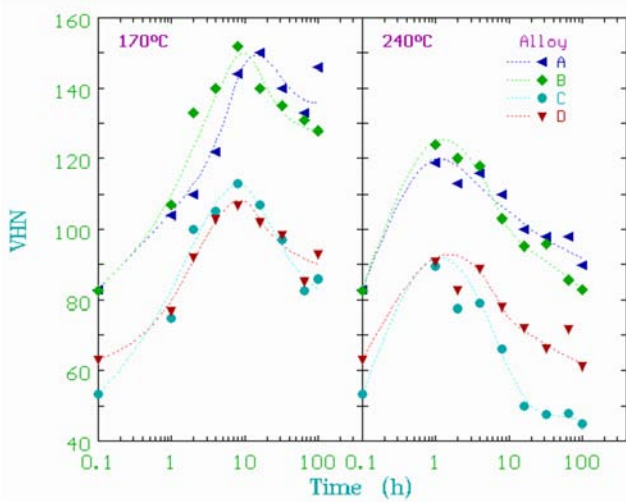


Figure 5. Hardness as a function of time when aging takes place at 170 and 240°C

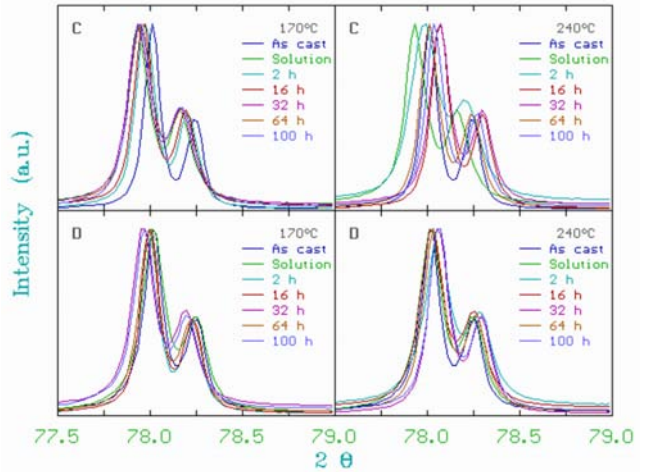


Figure 8. X-ray diffraction of the {311} peak for alloys C and D

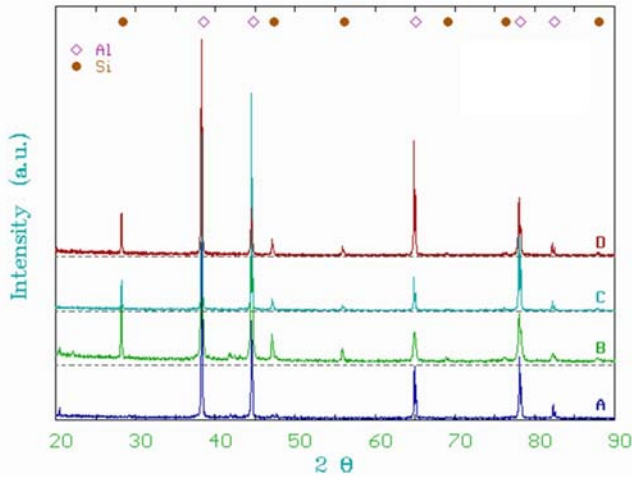


Figure 6. X-ray diffraction charts for the four alloys in the as-cast condition, the peaks corresponding to Al and Si are indicated

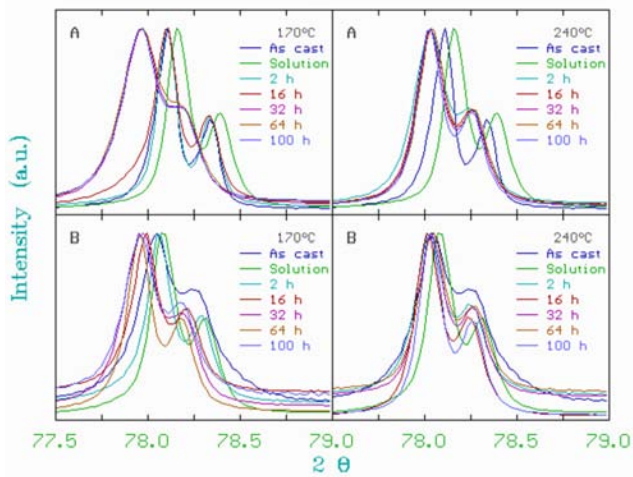


Figure 7. X-ray diffraction of the {311} peak for alloys A and B

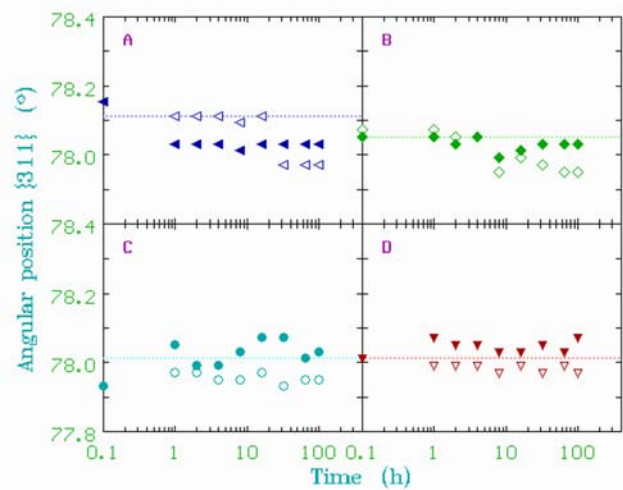


Figure 9. Angular position of the maximum values obtained from plots such as that in Figs. 7 and 8. The straight lines are those of the samples after solution; the open and full symbols correspond to samples aged at 170 and 240°C respectively

Table 2. Data related to the position of the {311} peak

Alloy	Aging temperature	Angle at solution	Angle (time) at maximum hardness	Angle at 100 h	Shift at maximum hardness	Shift at overaging
A	170°C	78.15°	78.09° (8 h)	77.97°	-0.06°	-0.18°
	240°C	78.15°	78.03° (1 h)	78.03°	-0.12°	-0.12°
B	170°C	78.07°	77.95° (8 h)	77.95°	-0.12°	-0.12°
	240°C	78.07°	78.05° (1 h)	78.03°	-0.02°	-0.04°
C	170°C	77.93°	77.95° (8 h)	77.95°	0.02°	0.02°
	240°C	77.93°	78.05° (1 h)	78.03°	0.12	0.10°
D	170°C	78.01°	77.97° (8 h)	77.97°	-0.04°	-0.04°
	240°C	78.01°	78.07° (1 h)	78.07°	0.06°	0.06°
6063*	180°C	78.07°	78.16° (8 h)	78.13° (24 h)	0.09°	0.06

* Cavazos and Colás, 2003

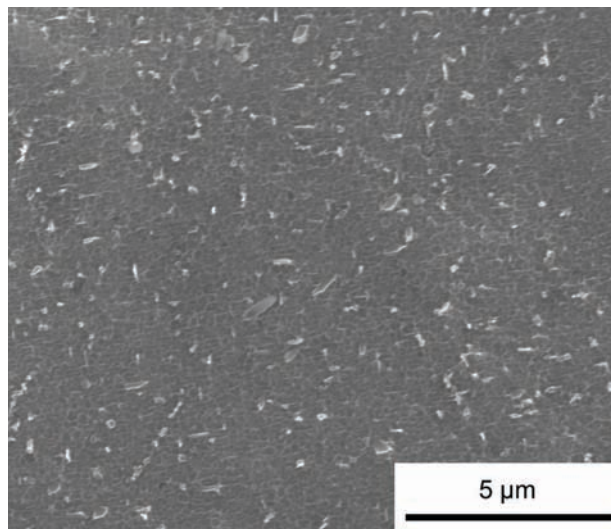


Figure 12. Scanning electron microscopy of a sample from the alloy D aged for 4 h at 170°C.

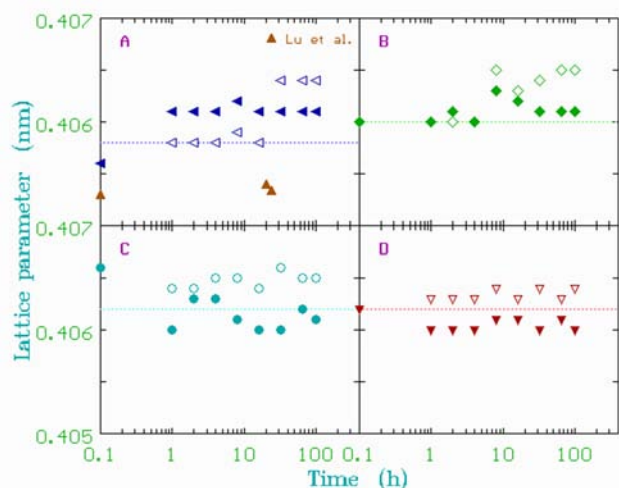


Figure 10. Changes in the lattice parameter of the various samples as function of aging; the straight lines are those of the samples after solution; the open and full symbols correspond to samples aged at 170 and 240°C respectively

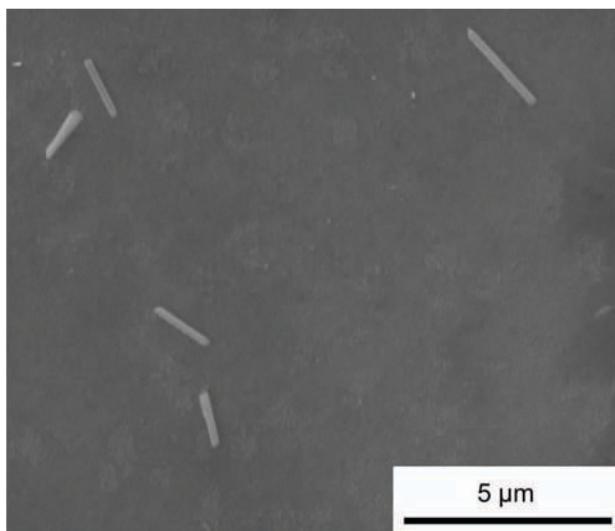


Figure 11. Scanning electron microscopy of a sample from the alloy C aged for 4 h at 170°C

The above made assumption may be confirmed by work being conducted using high definition electron microscopy, scanning and transmission, on heat treated samples of the various alloys. Figures 11 and 12 show the type of precipitates that were observed in alloys C and D, respectively, after being aged for 4 h at 170°C. The particles in Figure 11 are typical of Mg₂Si [19,26,27], whereas those in Figure 12 correspond to a mixture of Mg₂Si and Al₂Cu [26,28].

The results obtained in this work point in the direction that has been known from some time when heat-treating Al-Si-Cu alloys, of which the 319 type is the most common, as the growth of parts has been correlated with the amount of copper dissolved in the matrix. This can be attributed as the stable Al₂Cu phase forms during solidification, which results in parts with the desired dimensions, but, as this phase goes into solution and precipitate into metastable particles during aging, it will growth [29-32]. Therefore, T7 (stabilization) condition is favoured in powertrain, copper containing aluminium cast alloys parts as they will be close to their finished dimensions and will not grow and impair engine performance during their operational life [16,17,31].

Conclusions

Analyses of the heat treating process in cast aluminium alloy points out the effect that various alloying elements exert on distortion and dimensional changes in parts during their operation at high temperature.

It was found that copper-containing alloys, A and B, which harden by precipitation of Al₂Cu particles, increase the lattice parameter during aging, whereas C alloy that is hardened by precipitation of Mg₂Si particles do not. Alloys such as D, which are alloyed with copper and magnesium, apart from the silicon that provides fluidity to the molten metal, will exhibit grow when aged towards the higher temperature range, which can be attributed to precipitation of copper-rich particles over those of Mg₂Si.

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