

Dissolution of Second Phase Particles in 319-Type Aluminum Alloy

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Abstract

The dissolution of second phase particles in a 319-type (Al-Si-Cu-Mg) aluminum casting alloy has been quantified by image analysis of metallographic specimens as well as an electron microprobe technique. The initial phase content of the as-cast material, and the change in volume fraction of each phase following solution treatment for various times at 480°C and 505°C, was determined by analysis of particles observed by backscattered electron microscopy. Furthermore, the change in dendritic alloy content during solution treatment was measured using electron microprobe analysis in order to estimate the relative volume fraction of second phase particles dissolved. Finally, a non-isothermal dissolution model was used to predict the dissolution behaviour during solution treatment and comparisons are made between the model predictions and experimental measurements.

Introduction

Aluminum casting alloys based on the Al-Si eutectic system are widely used in automotive components as a result of their high castability, good mechanical properties and low density. These alloys typically contain magnesium and/or copper as alloying elements, and cast components are usually heat treated to improve their mechanical properties. The most common heat treatment applied to these alloys is the T6 temper, which consists of solution treatment at high temperature to dissolve any copper- and magnesium-rich intermetallic phase particles, followed by quenching and ageing at an intermediate temperature to promote a fine dispersion of strengthening precipitates in the alloy matrix. The dissolution of intermetallic phases during solution treatment is a critical stage in this process because it controls

the amount of each alloying element taken into solution and used for subsequent precipitation strengthening.

A number of investigations have been carried out over the years to determine the as-cast phase content of Al-Si casting alloys containing copper and magnesium, and the behaviour of these particles during solution treatment, and these are reviewed in a recent paper by Sjolander and Seifeddine [1]. Typically the as-cast structure consists of α -Al dendrites surrounded by an Al-Si eutectic phase that also contains coarse CuAl_2 , Mg_2Si , $\text{Q-Al}_5\text{Mg}_8\text{Si}_6\text{Cu}_2$ and Fe-rich intermetallic particles. The CuAl_2 phase is usually present in two different morphologies; blocky, and eutectic, and the proportion of each has been shown to depend on several factors including cooling rate, level of modification and Cu and Mg content [2-5].

Solution treatment temperatures for Al-Si-Mg-Cu alloys are typically limited to about 500°C in order to avoid localized melting of low melting point CuAl_2 and Q-AlMgSiCu intermetallic particles [6,7]. This imposes a significant limitation in the ability to rapidly dissolve the intermetallics and longer solution treatment times impact the cost of production and reduce productivity. Researchers have investigated this problem and found that CuAl_2 and Mg_2Si dissolve relatively quickly, whereas dissolution of $\text{Q-Al}_5\text{Mg}_8\text{Si}_6\text{Cu}_2$ occurs more slowly [8], while the dissolution kinetics of the blocky CuAl_2 particle morphology are slower than those of the eutectic morphology [5,9]. Long solution treatments may also be problematic in other ways, as researchers have reported the formation of Al_7FeCu after long times at soak temperatures, which removes copper from solid solution and reduces the strengthening potential of the alloy [10,11]. In high Mg content alloys, the Fe-rich intermetallics may also transform to π - $\text{Al}_8\text{Mg}_3\text{FeSi}_6$ reducing the solute magnesium content.

Sokolowski et al [2] performed an experimental study to reduce the solution treatment time of 319-alloy components. They developed a two stage solution treatment procedure in which an initial lower temperature step allowed the dissolution of low melting intermetallic phases, and a second higher temperature step results in faster homogenization and dissolution rates. Chaudhry and Apelian [12] used fluidized bed heat treatments to study the effect of rapid heating on dissolution in Al-Si-Mg and Al-Si-Cu-Mg casting alloys. They found the high heating rates increase the dissolution rate and proposed an optimal treatment for the 319 alloy of 45 minutes soaking at 493°C using the fluidized bed. Others have shown that the response of the alloy during aging is improved if a fluidized bed is used for solution treatment [13].

Some researchers have used experimental techniques to analyze the evolution of dissolution processes during solution treatment. Lasa et al [14,15] have compared metallographic analysis of solution treated specimens with the results of calorimetry tests for two hypereutectic Al-Si-Cu-Mg alloys, and found that the heat flow characteristics of the alloy can be used to monitor the progress of dissolution, ideally when thermodynamic modeling is also used to provide information regarding the stability of the various intermetallic phases present in the alloy. Han et al [9] used electron microprobe and x-ray spectroscopy techniques to study the dissolution of CuAl_2 during solution treatment in 319-type alloys containing different magnesium additions. The maximum dendritic copper content was measured after 8 hours of soaking which was the longest time investigated at which point full dissolution of Cu-rich phases had not been achieved.

Although several models have been developed to predict dissolution in aluminum casting alloys [11,16,17], relatively little work has been targeted at the Al-Si-Cu-Mg alloy system. The benefit of developing models to predict dissolution during solution treatment include an improvement in our understanding of alloy behaviour during multi-stage heat treatment, which in turn can enable process optimization from the standpoint of the material and result in lower variability in final component properties. This is particularly important when casting large components with complex solidification patterns and non-uniform as-cast microstructures that respond to solution treatment locally at different rates [18]. In the work presented here, the dissolution behavior of an industrial 319-type alloy has been investigated by several experimental techniques, and a model has been developed to predict the evolution of the CuAl_2 particles as they dissolve. The model predictions and experimental results have been compared to assess the suitability of the model to make predictions for industrial solution treatment processes.

Experimental Methodology

Start Material

The alloy was a 319-type alloy, supplied in the form of industrially cast ingots by Nemak Engineering Centre, Windsor, Ontario. These were remelted and cast into a wedge

mold at CANMET Materials Technology Laboratory, Hamilton, Ontario using an apparatus and procedure outlined in detail elsewhere [19]. No strontium or grain refiner additions were made for this research. The overall alloy composition, measured by an optical emission spectroscopy technique at CANMET is shown in Table 1. A quarter-section schematic of the copper wedge mould and resulting casting geometry is shown schematically in Figure 1. Metallographic specimens for investigation of the as-cast microstructure were taken vertically from the bottom of the chill at distances of 12mm, 25mm, 50mm and 75mm as shown in the diagram.

Table 1: Alloy Chemistry. One line separates the final line of the table and continuation of the narrative.

Element	Al	Si	Cu	Mg	Fe	Mn
Amount (wt%)	Bal.	8.3	2.8	0.5	0.45	0.34

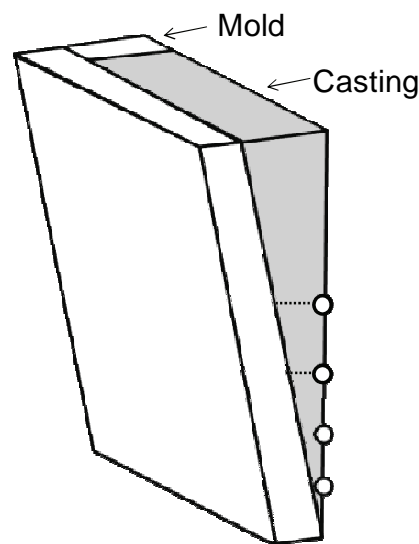


Figure 1: Schematic of the wedge mould and cast ingot showing the dimensions. Locations where metallographic investigation of the as-cast structure are shown (O), and the region of the casting where heat treatment specimens were taken is shown by the shaded area.

Heat Treatment Experiments

Specimens for the heat treatment investigation were taken from a section of the casting located 50mm to 75mm vertically from the bottom of the chill as given by the shaded region in Figure 1. Specimen dimensions were 25mm (l) x 25mm (w) x 4mm (h). Heat treatments were done using a Carbolite furnace at soak temperatures of 480°C and 505°C for various times up to 24 hours duration. In order to simulate typical industrial heating conditions, a 70 minute heating ramp was applied from room temperature to the soak temperature. Following heat treatment, the specimens were removed from the furnace and immediately quenched in water at room temperature.

Metallography and Image Analysis

All metallographic examinations were carried out on vertical sections which expose the microstructure perpendicular to the solidification direction. The specimens were mounted using a Buehler Epocixure acrylic resin, then ground and polished by hand to a 0.06 μm finish using a colloidal silica suspension.

Photomicrographs of the microstructures were taken using a Nikon EPIPHOT 300 series inverted metallurgical microscope equipped with a digital camera. Post-processing of the micrographs was carried out using Clemex Professional Imaging and Adobe Photoshop 7.0 software. Several microstructural characteristics were quantified by analysing the 2-D micrographs. In the as-cast alloys, the secondary dendrite arm spacing of the primary $\alpha\text{-Al}$ dendrites and the average size of the as-cast intermetallic particles were measured using the Imagetool software package.

As-cast and heat treated specimens were examined using a Hitachi S-3000 electron microscope in backscatter electron (BSE) mode. The imaging parameters used included an accelerating voltage of 20keV, working distance of 15mm. Backscatter electron micrographs were taken at random across the polished surface of the specimens and the volume fraction of each intermetallic phase present in the as-cast and solution treated specimens was quantified using a point count method. A minimum of 1600 points were used to estimate these values.

Microprobe

Electron probe microanalyses of the alloy microstructure were done using a fully automated CAMECA SX-50 electron-probe microanalyser, operating in wavelength-dispersion mode at the Electron Microbeam/X-ray Diffraction Facility in the Department of Earth and Ocean Sciences at the University of British Columbia. Quantitative analyses were made for Al Si, Mg, Cu and Fe using standards of known composition and the following operation conditions were used; excitation voltage, 15kV; beam current, 20nA; peak count time, 60s (10s for Al); background count-time, 30s (5s for Al); spot diameter, 1 μm .

The interaction volume was estimated to have an approximate diameter and depth of 2.5 μm and 2 μm , respectively. Consequently, measurements were made across the width of the dendrite arms at spacings of 4 μm , as well as, selected measurements in the eutectic regions and at the location of large (i.e. greater than 1 μm) second phase particles. These results allow the spatial alloying element content across the dendrite to be estimated so as to determine the evolution of the dissolution and homogenisation processes. Between four and six dendrites were examined for each experimental condition.

Model Development

The dissolution of CuAl_2 particles has been modeled under the assumption that it is controlled by the diffusion of magnesium atoms in the $\alpha\text{-Al}$ matrix. The model is based on the steady-

state dissolution of a single spherical particle of radius, r , in an infinite matrix, following the approach by Whelan [20] and applied to Mg_2Si dissolution in an A356 alloy previously [21].

$$\frac{dr}{dt} = - \left(\frac{C_o - C_i}{C_p - C_o} \right) \frac{D}{r}$$

The compositional terms C_i , C_o , and C_p are shown schematically in Figure 2. In the model formulation the microprobe measured copper content at the centre of the as-cast dendrite is substituted for the solute concentration at the infinite point, C_i . Furthermore, the temperature dependences of C_o and D have been described using linear and Arrhenius relationships respectively. The dissolving particle is assumed to have CuAl_2 stoichiometry, and the initial particle size has been estimated using a mass balance approach in which the amount of copper contained in the particle is equated to the difference between the mass of copper measured in the as-cast and fully solution treated dendrite arms. The calculated mass of copper is then converted into the radius of the CuAl_2 particle assuming it is spherical and fully dense, with a density of 4.34 kg/m^3 [22]. For simplicity it is assumed that no silicon or Fe-rich particles exist in the system.

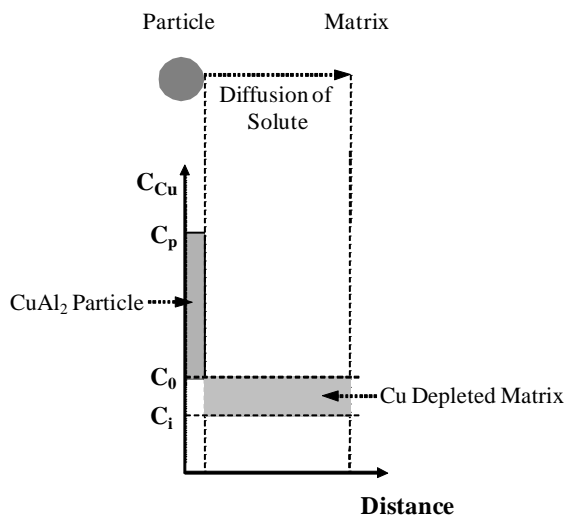


Figure 2: Schematic diagram showing the solute concentration surrounding a dissolving second phase particle.

The model calculates the rate of change in particle radius with time and hence the evolution of particle size can be determined. The relative volume fraction of the particle, f_r , during dissolution can be calculated using a similar approach to Nolfi et al. [23].

$$f_r = \frac{f}{f_0} = \frac{r^3}{r_0^3}$$

where the instantaneous particle radius, r , is predicted at each time step.

Results and Discussion

The results of the as-cast microstructure investigation are presented first, followed by the images and results of the point count analysis that indicate the changes in intermetallic content during solution treatment. The microprobe data is then presented, followed by estimates of the mean Cu and Mg content for the as-cast and solution treated conditions studied. Finally, model predictions for the dissolution of CuAl_2 are presented and compared with the results of the image analysis and microprobe investigations.

As Cast Microstructure

The optical micrographs show that the as-cast alloy has an unmodified microstructure consisting of the α -Al matrix, Al-Si eutectic and several intermetallic phases including CuAl_2 , Q-phase and α -AlFeSi. Small Mg_2Si particles were also observed at the interfaces of other phases. The as-cast alloy is shown in Figure 3 and the phases observed are identified. Backscatter electron micrographs can be used to clearly indicate the differences in the various Cu-rich intermetallics. The blocky and eutectic morphologies of CuAl_2 are shown in Figure 4. The Q-phase is also observed to be closely associated with eutectic CuAl_2 particles. The script morphology of the α -AlFeSi particles is also clearly seen.

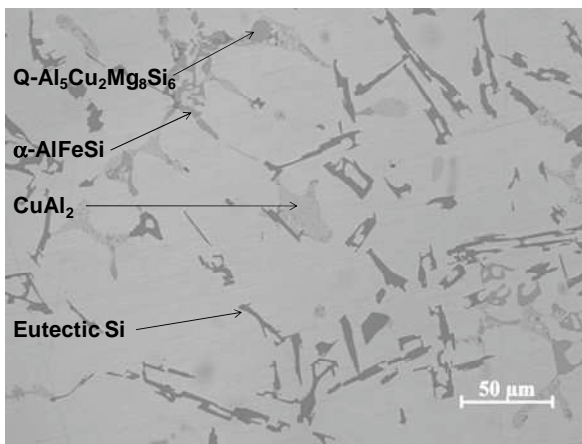


Figure 3: The as-cast microstructure of the 319-type aluminum alloy. The eutectic Si particles and intermetallic phases CuAl_2 , $\text{Q-Al}_5\text{Mg}_8\text{Si}_6\text{Cu}_2$ and α -AlFeSi are identified using arrows. The Mg_2Si particles consist of small black particles at the interfaces of other second phase particles.

Microstructure Evolution

The dissolution of second phase particles in the 319-type alloy during solution treatment can be seen using backscatter electron micrographs. Figure 5 shows the change in phase content between the as-cast alloy and after solution treatment at 480°C for 150 minutes and 1440 minutes. The amount of

CuAl_2 is seen to decrease significantly. The α -AlFeSi intermetallic is not affected under these conditions.

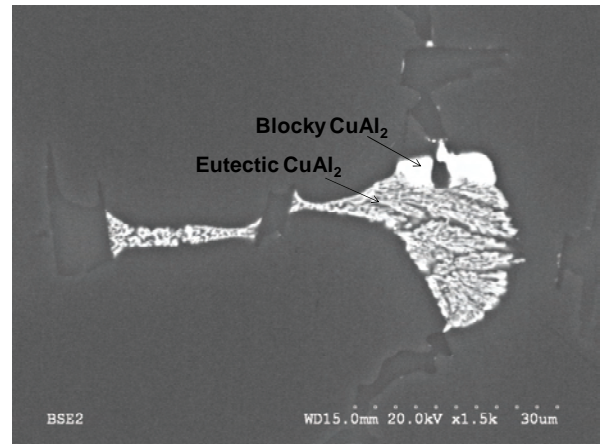


Figure 4: Backscatter electron micrograph of the as-cast 319 alloy showing the blocky and eutectic CuAl_2 morphologies.

The results of the point-count analysis to estimate the volume fraction of each intermetallic are presented in Table 2. The volume fraction of CuAl_2 reported does not differentiate between blocky and eutectic morphologies. Estimates for the volume fraction of Mg_2Si are omitted as these particles are difficult to see in an aluminum matrix using the backscatter electron technique. The results presented in Table 2 confirm the observations of the backscatter electron micrographs shown in Figure 5. The amount of CuAl_2 decreases during solution treatment to near zero after 24 hours soaking at both temperatures investigated. In contrast, the volume fraction of the Q-phase only decreases at 505°C and remains fairly constant throughout solution treatment at 480°C . The Fe-rich intermetallic phases remain unaffected by solution treatment. Figure 6 shows a plot of the changes in volume fraction for each intermetallic phase.

These results suggest that 24 hours at 505°C is required for near-complete dissolution of the CuAl_2 and Q-phase particles, although most CuAl_2 is dissolved after 24 hours at temperatures below this. These results confirm other researchers results [2,9] that dissolution is not complete after 8 hours at temperatures below 500°C due to incomplete CuAl_2 dissolution but also because of the limited amount of total dissolution undergone by the Q-phase at these temperatures. Indeed, the two-step solution treatment procedure proposed by Sokolowski et al [2] may be beneficial due to dissolution of the Q-phase during the high second stage temperature. The differing dissolution behaviour of the two phases across this temperature range may explain some of the complexities of the heat treatment behavior of this alloy, including the level of strength achieved in the peak aged condition following different solution treatments. Increased copper and magnesium solute contents would be expected to arise from the slowly dissolving Q-phase, both of which contribute towards the later formation of strengthening precipitates.

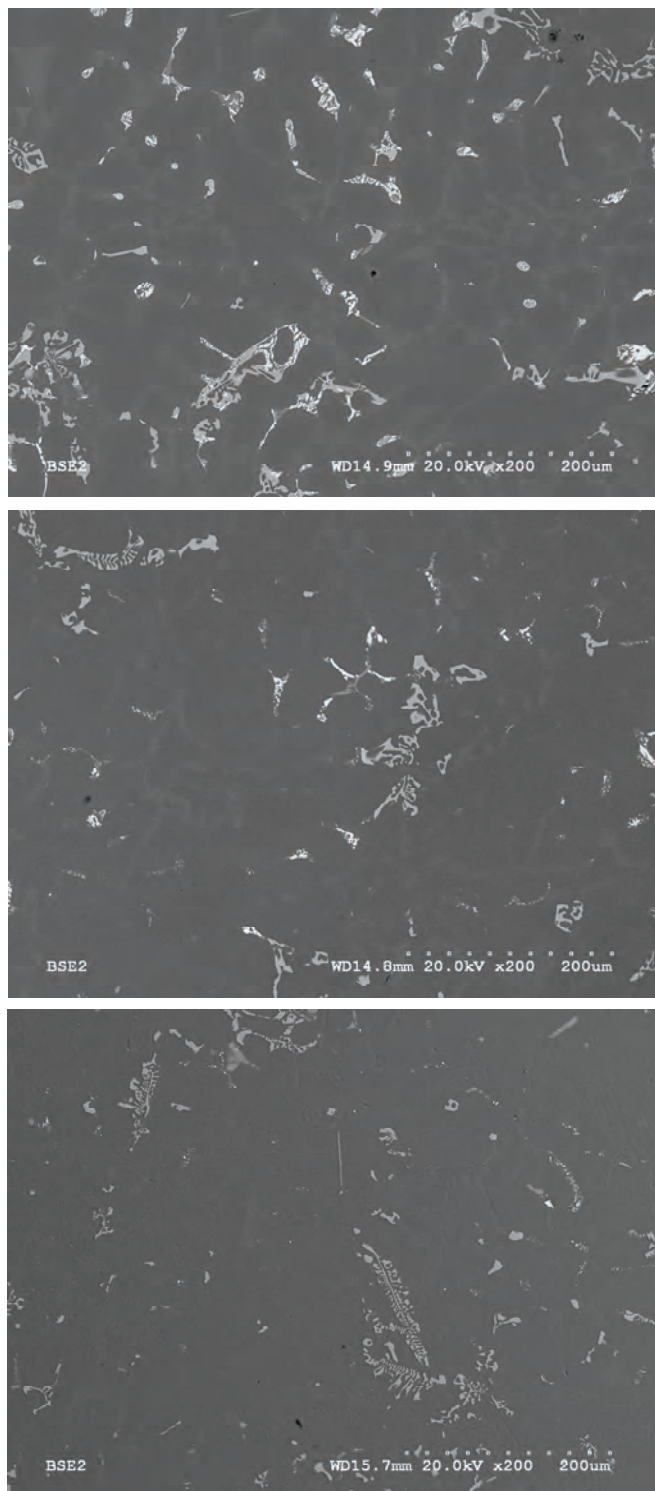


Figure 5: Backscattered electron micrographs for the 319 alloy: (a) in the as-cast condition and (b) following solution treatment for 150 minutes at 480°C, (c) for 1440 minutes.

Table 2: Estimated volume fraction for intermetallic phases in the 319-type alloy in the as-cast condition and following solution treatment using the point-count method.

Sol. Treat Temp. (°C)	-	480				505		
Sol. Treat Time (min)	AC	15	150	1440	15	150	1440	
Vol. Frac. CuAl ₂	0.039	0.020	0.015	0.004	0.018	0.004	0.002	
Vol. Frac. Q-AlCuMgSi	0.008	0.006	0.009	0.008	0.005	0.003	0.002	
Vol. Frac. α-AlFeSi	0.030	0.030	0.036	0.024	0.032	0.026	0.027	

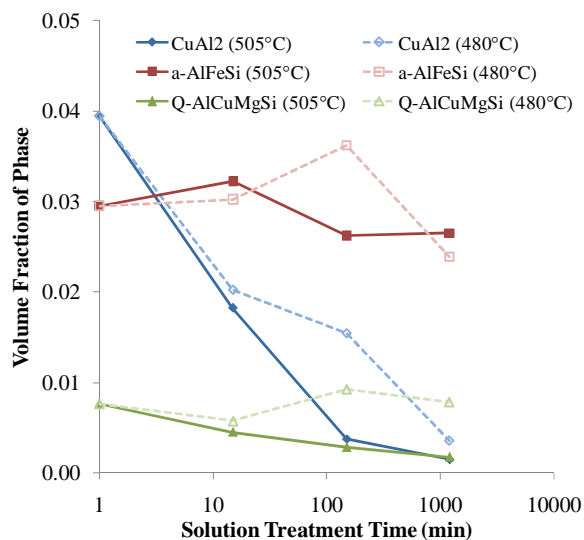
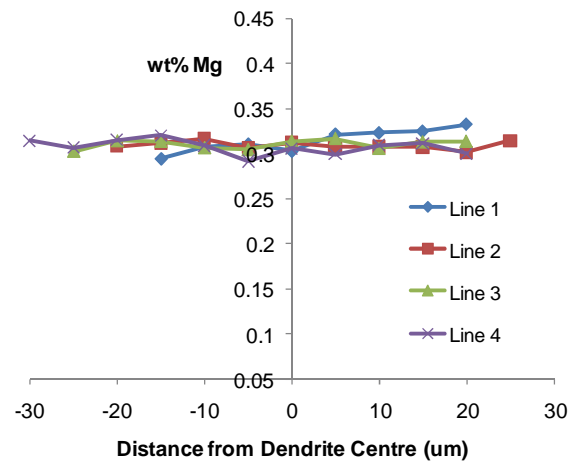
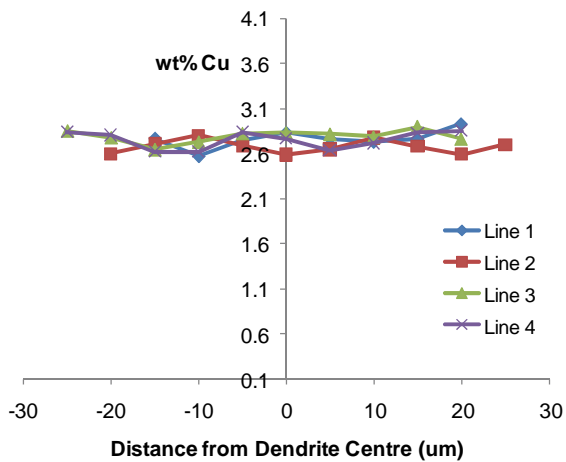
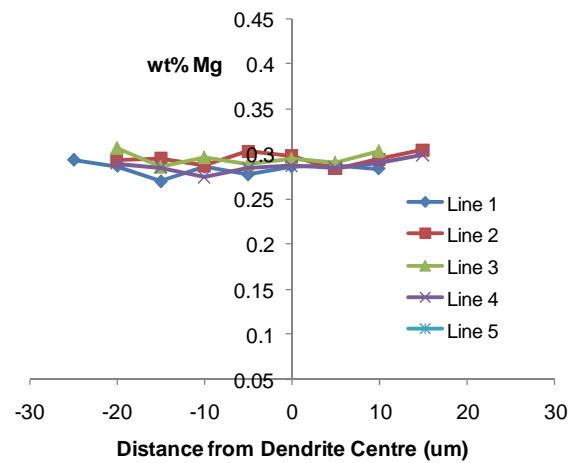
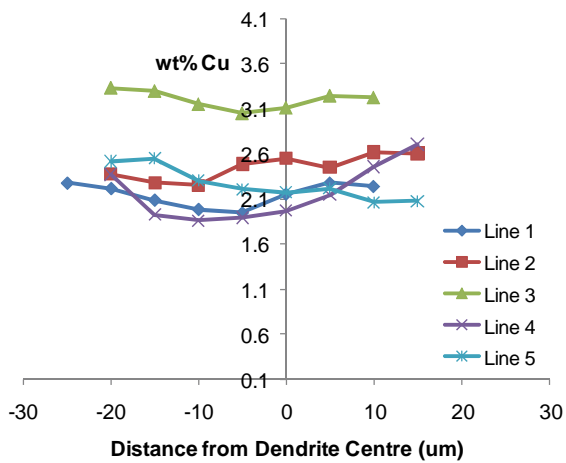
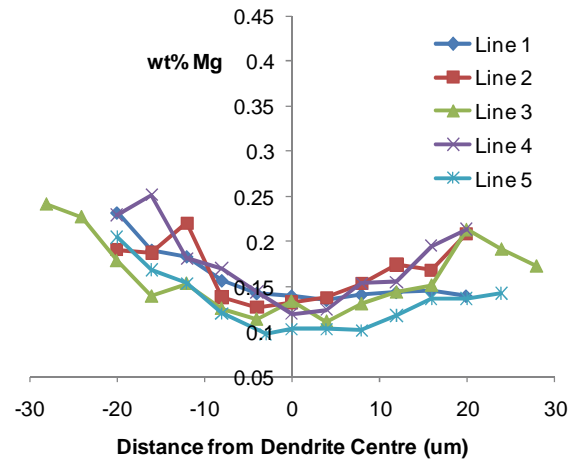
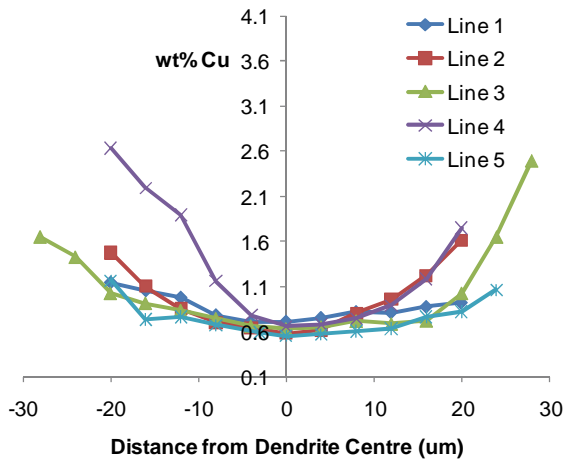


Figure 6: change in volume fraction of CuAl₂, α-AlFeSi and Q-phase in 319-type alloy during solution treatment at 480°C and 505°C.

Microprobe Results

Microprobe plots showing the change in distribution of copper and magnesium in the α-Al dendrites during solution treatment at 480°C and 505°C are shown in Figures 7-10. Microsegregation of copper and magnesium atoms is seen in the as-cast dendrites. The copper and magnesium content at the centre of the as-cast dendrite were approximately 0.6wt% and 0.12wt% respectively. Alloy content was significantly higher closer to the dendrite edge in both cases. The volume-averaged dendritic alloy concentration in the as-cast condition was calculated to be 1.05wt%Cu and 0.17wt%Mg assuming the dendrites are cylindrical. These values increase during solution treatment as second phase particles dissolved and the figures show increasing alloy content and decreasing microsegregation as homogenization occurs.



480

Figure 7: Electron microprobe data for the distribution of copper concentration across dendrites (a) in the as-cast condition, and after solution treatment at 480°C (b) for 30 minutes and (c) 1440 minutes.

Figure 8: Electron microprobe data for the distribution of magnesium concentration across dendrites (a) in the as-cast condition, and after solution treatment at 480°C (b) for 30 minutes and (c) 1440 minutes.

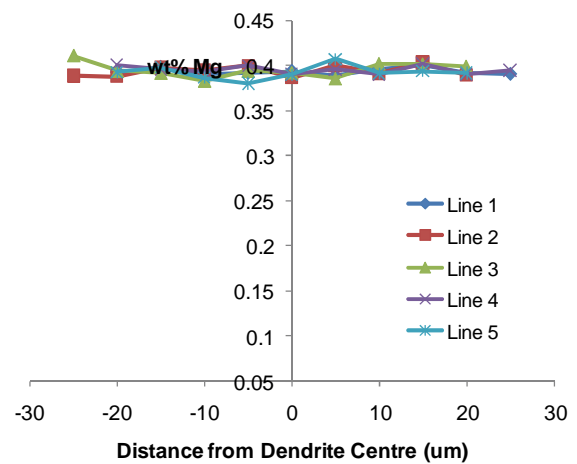
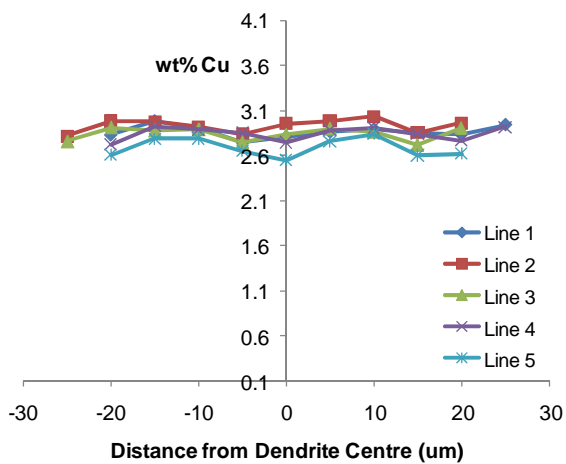
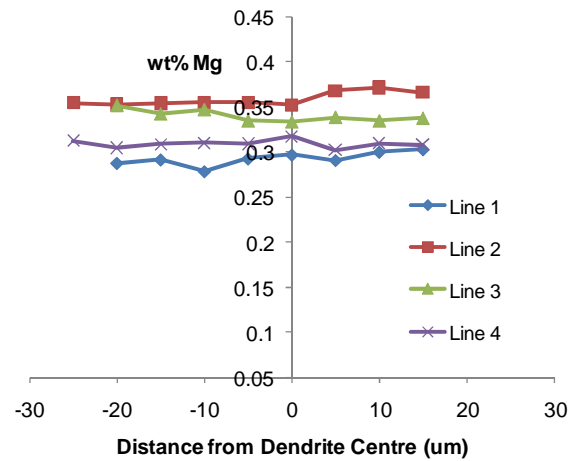
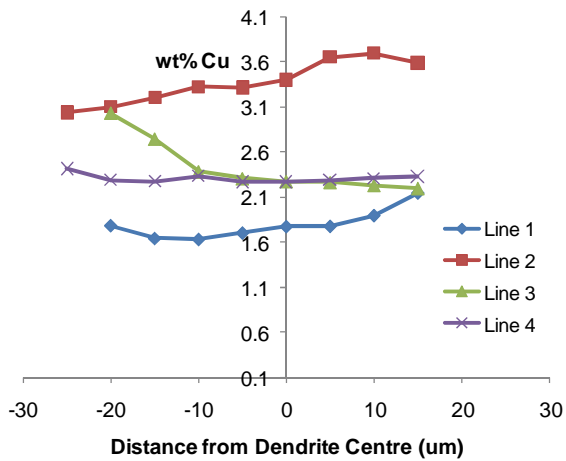
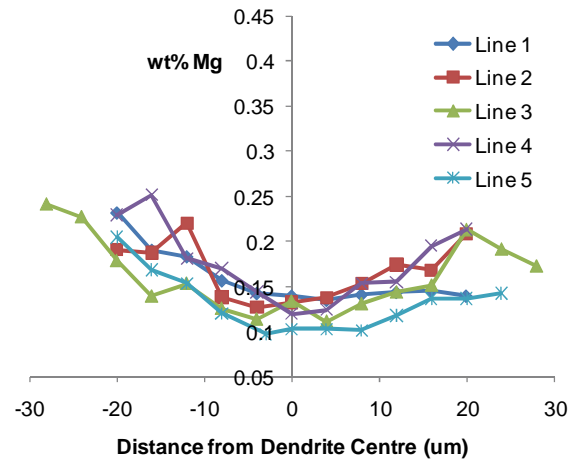
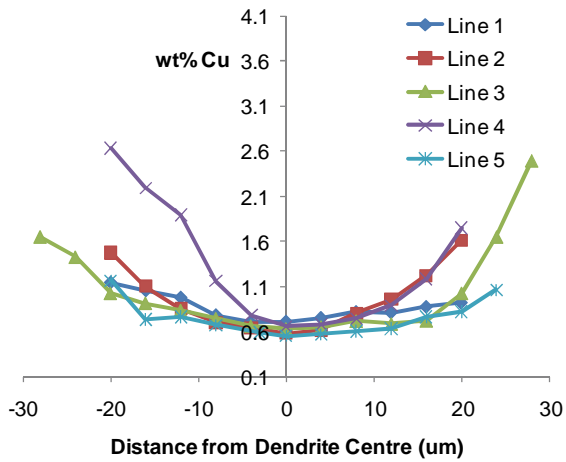


Figure 9: Electron microprobe data for the distribution of copper concentration across dendrites (a) in the as-cast condition, and after solution treatment at 505°C (b) for 30 minutes and (c) 1440 minutes.

Figure 10: Electron microprobe data for the distribution of magnesium concentration across dendrites (a) in the as-cast condition, and after solution treatment at 505°C (b) for 30 minutes and (c) 1440 minutes.

The variation in alloy content between dendrites in the same specimen increases initially from the as-cast condition, and then decreases again. This is attributed to the non-uniform distribution of second phase particles and resulting enrichment of dendrites close to dissolving particles. This is an important observation in terms of the downstream processing of partially solution treated components in that some areas of the components will have significantly higher solute content than others, and therefore, not only would the values of the peak strength be higher in the solute enriched areas, but the kinetics of precipitation would be faster and resulting in more rapid arrival at the peak and overaged conditions compared to the less solute-rich areas.

The volume averaged copper and magnesium content of the dendrites studied in the microprobe examination have been calculated and plotted in Figure 11. Both similarities and differences in the evolution of the two alloying elements can be seen. In all cases, the solute content of the dendrite increases, however, the difference between the evolution of copper and magnesium at the two temperatures is noticeable in that the magnesium content increases much more at 505°C than at 480°C relative to the increases in copper content. This effect may be related to the dissolution of the Q-phase which contains a higher mass of magnesium than copper. It also suggests that the increased strength measured in this alloy following solution treatment at higher temperature is related to the formation of a larger amount of Mg-rich precipitates during aging, rather than copper-rich precipitates.

In comparison with the volume fraction calculations based on the backscatter electron micrographs, it is clear that the Mg and Cu content increases as the CuAl_2 and Q-phase dissolve. The increase in Mg content at 480°C measured by the microprobe is assumed to be related to the dissolution of the Mg_2Si particles observed on the interfaces of other intermetallics, and the increase of Cu at this temperature appears to be directly related to the dissolution of CuAl_2 . At 505°C, dissolution of the Q-phase also occurs, resulting in an increased amount of Cu and Mg released into the dendrite.

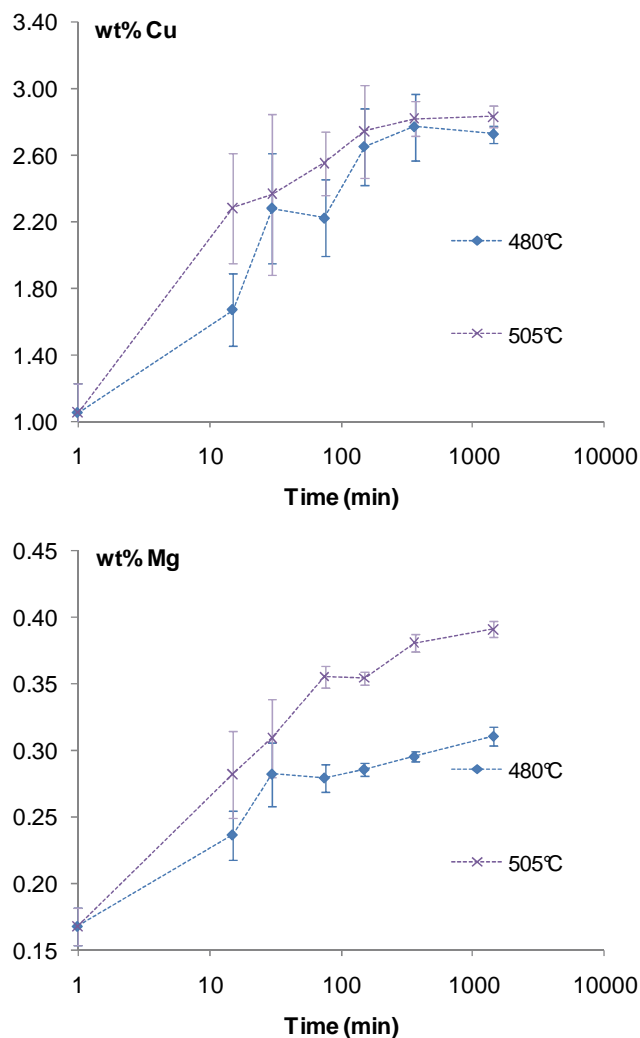


Figure 11: Volume averaged alloy content in dendrites examined by the microprobe: a) copper, b) magnesium.

Model Results

Model predictions for dissolution of CuAl_2 at soak temperatures of 480°C and 505°C following a 70 minute constant heating ramp are shown in Figure 12. The accuracy of the model predictions can be assessed by comparing them against estimates of the fraction dissolved from microprobe data of specimens in the partially solution treated condition. The average dendritic copper content is calculated from the microprobe data assuming the measured values from the microprobe are taken from a section that passes through the centre of a cylindrical dendrite, thereby capturing the minimum copper content at the dendrite core. The fraction dissolved is then calculated from the relative increase in copper content of the dendrite between the minimum and maximum cases (i.e. the as-cast and fully solution treated specimens) and assuming that the relative fraction increase is equal to the volume fraction of particle dissolved.

The model predictions fit the experimental data very well, although they slightly under-predict the rate of dissolution at the beginning of dissolution and over-predict the fraction dissolved at longer times, leading to an overall under-prediction of the total dissolution time. The under-prediction at short dissolution times is likely due to inaccuracies in the thermal dependence of the kinetic constants used for the model, as some dissolution would be expected during heating. Alternatively, the experimental heating rate the specimens were exposed to was not linear as has been applied to the model. In contrast, the over prediction at long solution treatment times is likely to be related to the assumption of a constant copper content at the infinite point (i.e. the dendrite centre), which in reality will increase significantly during solution treatment as the dissolved solute diffuses towards the low-solute regions. This effect causes a reduction in driving force for further dissolution, which also explains the difference in the overall dissolution time between the model and experimental results.

The results of the image analysis investigation have been converted to relative volume fraction based on the initial volume fraction measured in the as-cast material. This data has been compared with the model predictions and microprobe estimates in Figure 12. There is reasonably good agreement between the three techniques, suggesting that the microprobe approach used in this work to calculate the dendritic alloying element composition can be used to obtain good approximations of the relative fraction of second phase particles dissolved. The comparison also indicates that the modeling approach provides good estimates of the progress of dissolution. This is an important step towards the development of models that aim to improve our understanding of the complex metallurgical phenomena occurring during solution treatment of these alloys, with the long-term aim of increasing efficiency by finding optimum heat treatment conditions.

Conclusions

The following conclusions can be made based on the work presented here.

The as-cast microstructure of the 319 alloy studied here contained CuAl_2 , $\text{Q-Al}_5\text{Mg}_8\text{Si}_6\text{Cu}_2$, Mg_2Si and $\alpha\text{-AlFeSi}$ as the main second phase particles.

CuAl_2 , $\text{Q-Al}_5\text{Mg}_8\text{Si}_6\text{Cu}_2$ and Mg_2Si were found to dissolve during solution treatment. Mg_2Si and CuAl_2 dissolved more easily than the Q-phase. The Fe-rich intermetallic phase was unaffected by solution treatment.

Microprobe tests were able to provide information regarding the solute content of the dendrite throughout solution treatment. The copper and magnesium levels were observed to increase with solution treatment, and the increase in magnesium appears to be more sensitive to temperature than the increase in copper.

The point count method accurately described the change in second phase content during solution treatment. The higher sensitivity of the magnesium to temperature was related to the dissolution of the $\text{Q-Al}_5\text{Mg}_8\text{Si}_6\text{Cu}_2$.

The dissolution of CuAl_2 was modeled with reasonable accuracy using a steady-state dissolution model with input parameters derived from the microstructure characteristics and experimentally measured solute content of the alloy dendrite.

Dissolution models based on microstructural mass balance calculations as presented may potentially be used to determine the optimum solution treatment conditions for efficient heat treatment processing.

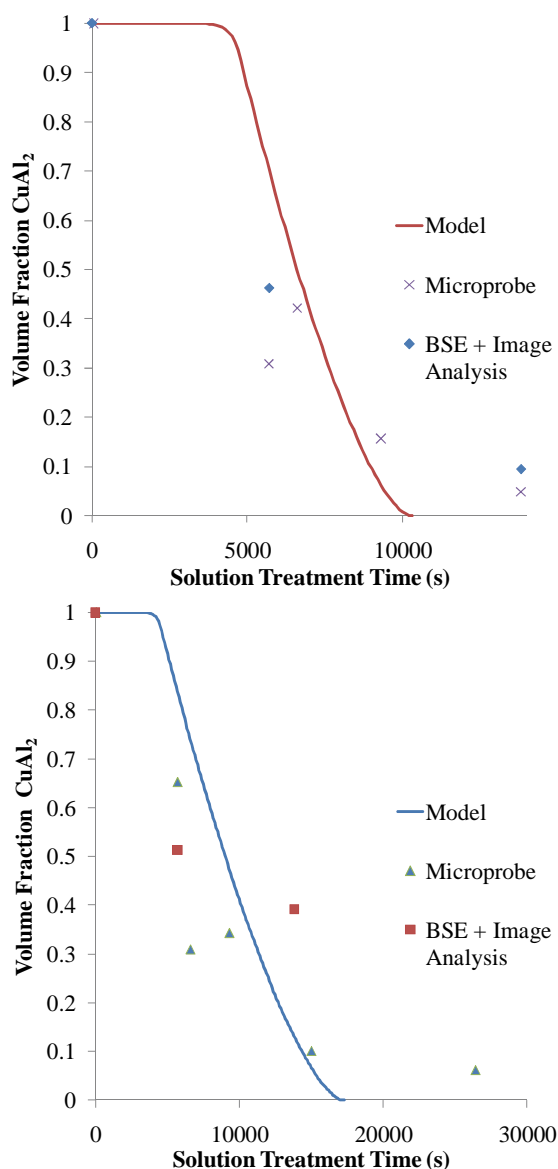


Figure 12: Model predictions for dissolution of CuAl_2 compared to calculated values from microprobe testing and image analysis of BSE micrographs: a) 480°C, b) 505°C.

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