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Effect of heat treatment on properties of thixoformed high performance 2014 and 201 aluminium alloys

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Tempering treatments T4, T5 and T6 have been applied after thixoforming to investigate the effects of heat treatment on microstructure and mechanical properties of thixoformed wrought aluminium alloy 2014, a 'modified' 2014 and high strength casting aluminium alloy 201. The 'modified' 2014 was a simplified version of 2014 with composition (wt%) Cu 3.87, Mg 0.6 and Si 0.14. The results showed that standard solution time for conventional wrought alloy 2014 (1 h at 500°C) gave incomplete homogenisation of thixoformed material. Longer times (5 and 17 h) at 500°C are required for complete solutionisation of $CuAl_2$. Low hardness values in the as-thixoformed 2014 were increased by subsequent heat treatment. After 18 h ageing at 160°C, the hardness of material solutionised for 5 and 17 h is higher than that for the standard 1 h. The tensile properties of the thixoformed 2014 were improved and close to those of forged 2014 using optimised post-forming heat treatment (solution treatment for 17 h at 500°C followed by water quenching and then ageing for 24 h at 160°C). Thixoformed alloy 201 in the standard T6 condition (2 h at 513°C followed by 17 h at 527°C and water quenching and then ageing 20 h at 153°C) showed a significant improvement over permanent mould casting. Good elongation to fracture (19.6%) was found for thixoformed 'modified' 2014 in the T6 condition.

1. Introduction

The production, rheology and processing of semi-solid metals have been widely studied since the original discovery of semi-solid metal processing [1]. Conventional casting alloys (A356, A357) are used to produce millions of thixoformed automotive parts every year. Although producers would like to be able to thixoform components with a higher performance comparable to that of wrought aluminium alloys, or high strength casting aluminium alloys, such as 201, these alloys are not currently thixoformed because there is no available commercial source of feedstock and they are difficult to thixoform [2]. However, there is significant progress towards reaching the goal of thixoforming alloys with performance approaching that of the conventional wrought alloys [3]. Three alloys were used in this work: alloy 2014, one of the most important aircraft alloys, containing copper as the primary alloying addition with smaller quantities of magnesium, silicon and manganese; 'modified' 2014 (no Fe or Mn and reduced Si; these elements form the insoluble intermetallic compound $CuFeMn_3Si_2Al_{15}$); and high strength silver-containing casting Al alloy 201. Alloy 201 has a particularly high response to age-hardening because silver completely changes the precipitation process, causing an orthorhombic form of the tetragonal phase θ (Al_2Cu) to precipitate as very thin, coherent plates on the {111} matrix planes rather than the {100} planes [4]. Thixoforming requires starting material which has been treated in such a way that when it is in the semi-solid state the microstructure is spheroidal rather than dendritic. In this work, the Recrystallisation and Partial Melting (RAP) route [5, 6] and Cooling Slope (CS) casting [7] were used to obtain the semi-solid feedstock from extruded material and DC-cast dendritic material. Compared with the SIMA (Strain-Induced Melt Activated) route, RAP involves warm working below the recrystallisation temperature (rather than hot working)

TABLE I Compositions of aluminum alloys used in this study (wt%)

											Ingot diameter (mm)	
Alloy	Cu	Mg	Si	Fe	Mn	Zn	Cr	Ti	Ag	Bal.	1st round	2nd round
2014	3.91	0.47	0.83	0.29	0.55	0.06	0.01	0.07		Al	63.5	50
Modified 2014	3.87	0.6	0.14	<0.01	<0.01	<0.001	–	–		Al		73
201	4.35*	0.36	<0.05	<0.02	0.34	<0.02	<0.01	<0.01	0.60	Al		97

Compositions measured using inductively coupled plasma (ICP) spectroscopy method.
*4.72 by wet chemistry.

and no cold working is needed before the alloy is rapidly heated to the semi-solid state.

Alloys 2014 and 201 are generally not used in the as-formed condition, being instead subjected to heat treatment. Here the thixoformed alloys are solutionised, quenched and then aged. Only limited work has been reported on the effects of heat treatment on spheroidal microstructures and mechanical properties [8–14].

Cerri *et al.* [10] studied the effect of heat treatments on the mechanical properties of 319 thixocast alloy. Solution heat treatment at 500°C resulted in incomplete dissolution of the CuAl$_2$ phase in the eutectic regions and its eventual disappearance at a slightly higher temperature 510°C. The mechanical properties of the thixocast 319 alloy were higher than those obtained from traditional cast 319 alloys.

Zoqui *et al.* [13] determined the effect of solution and ageing treatments on the structure and properties of rheocast and conventionally cast Al-4.5 wt%Cu alloys, and found that rheocast material required more time for complete dissolution of the eutectic phase than cast structures. In addition, higher Cu contents were held in solution after T6 treatment and grain growth was inhibited during holding. The relative improvement in mechanical properties as a result of heat treatment is greater for rheocast material than for conventionally cast alloy with the same composition.

Blandin *et al.* [14] reported that thermal treatment of thixoformed magnesium alloy AZ91 led to partial or almost complete dissolution of the intermetallic phase depending on the temperature of the treatment. The mechanical behaviour was strongly influenced by the temperature and duration of the thermal treatment since the elongation to failure is controlled by the fracture of the brittle intermetallic phase.

Here, optimisation of the post-forming heat treatment (considering T4, T5 and T6 conditions) has been carried out in order to attempt to meet tensile mechanical property targets (YS 414 MPa, UTS 480 MPa, Elongation to fracture 13%) for forged alloy 2014 [15]. For alloy 201, a conventional T6 heat treatment (two-step solution 2 h at 513°C and 17 h at 527°C followed by water quenching and then ageing 20 h at 153°C) has been used.

2. Experimental procedures

The starting materials were as follows: for alloy 2014, two kinds of extruded bars of 63.5 mm and 50 mm diameter in the T6 condition; for alloy 201, DC cast 97 mm diameter; for 'modified' 2014, DC cast 73 mm diameter. The compositions and conditions of the starting materials are given in Table I.

The recrystallisation and partial melting (RAP) route and cooling slope (CS) casting were used to obtain the thixotropic feedstock for thixoforming. Extruded bars of 2014 were heated into the semi-solid state to cause recrystallisation and spheroidization of the microstructure by the RAP route. For CS casting, DC cast alloy 201 and 'modified' 2014 were melted to 720°C and cooled to 10 to 20°C above the liquidus in a bonded alumina crucible in an induction furnace and cast into a steel mould (ϕ50 mm × 150 mm) via a water-cooled slope fed from the bottom-stoppered crucible.

In the first round of thixoforming, ϕ60 mm × 70 mm slugs were machined from as-received extruded 2014 bar of 63.5 mm diameter. The slugs were rapidly heated to 632°C in 4 steps (to 500, 600, 625 and 632°C) in 2.5 min, then thixoformed into a cross-shaped graphite die (Fig. 1) in a thixoforming press which acts vertically upwards and has been described previously [16]. For the second round of thixoforming, ϕ50 mm × 70 mm slugs were sliced from as-received extruded 2014 bar of 50 mm diameter and from the ingots cast on the

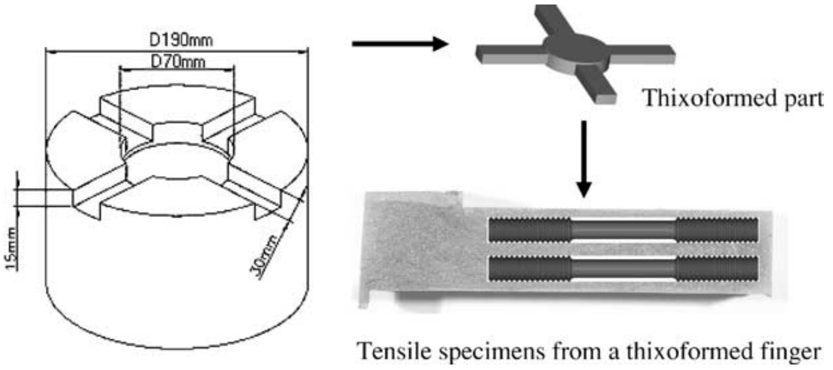

Figure 1 Graphite die (shown inverted) with location of tensile samples in thixoformed finger indicated.

cooling slope. The slugs were rapidly heated to 624, 631 and 634°C for extruded 2014, cooling slope cast 'modified' 2014 and cooling slope cast 201, respectively, then thixoformed into a cross-shaped die with a machinable Syndayo (ceramic composite) top (low thermal conductivity) and graphite bottom. The heating of the slug was monitored by using two K-type thermocouples, located at a depth of 15 mm from the top of the slug, one in the centre and the other 5 mm from the outer surface.

The thixoformed 2014 fingers were given T4, T5 and T6 heat treatments to assess their effects on the microstructure and mechanical properties of thixoformed parts. A resistance furnace was used. T4 solution treatments at 500°C for 1 to 17 h were performed on a set of samples in order to study the dissolution of the eutectic phase. For the T6 temper, the specimens were solution heat treated at 500°C for 5 or 17 h followed by water quenching at room temperature and then artificially aged at 160 or 170°C for different times (2 to 64 h). In addition, the T6 heat treatment which would be used for conventional wrought alloy 2014 (1 h solutionising at 495 or 500°C followed by water quenching and 8 or 18 h ageing at 160 or 170°C) was performed along with the T5 ageing treatment of 18 h at 170°C. An optimised heat treatment was then identified and applied to thixoformed 2014 and 'modified' 2014. For thixoformed alloy 201, the conventional T6 heat treatment for cast material (two-step solutionising with 2 h at 513°C and 17 h at 527°C followed by water quenching and then ageing for 20 h at 153°C) was used.

Samples for optical observation were mounted, ground, polished and then electro-etched in Barker's reagent (7 ml $HBF_4$48%, 93 ml H_2O) at 20 V for 90 s using a stainless steel cathode. For Scanning Electron Microscopy (SEM) observations, specimens were final polished with silco liquid carefully and not etched. After heat treatment, the specimens were examined by SEM to identify the phases present and Differential Scanning Calorimetry (DSC) was used to identify the temperature of the eutectic reaction during heating. The distribution of constituents was observed with Transmission Electron Microscopy (TEM). The compositions of intermetallic phases were obtained by quantitative EDS analysis on a Jeol 6400. Hardness values were measured at 50 g load using a Leica VMHT Vickers hardness tester. Each hardness value is the average of at least five measurements. Specimens for tensile testing were machined from thixoformed fingers after T6 heat treatment and were tested using an H100KS material testing machine. Specimens (ϕ4 mm) were held at two ends by automatic hydraulic clamps, and the test conditions were input from a computer. Crosshead speed was 5 mm/min, and the gauge length was 25.00 mm. Quoted results are for single tests. Optical metallography on longitudinal sections of tensile samples was carried out.

3. Results

3.1. As-received microstructures

Fig. 2 shows the optical microstructures of the as-received extruded 2014 in the T6 condition and DC-cast 'modified' 2014 and DC-cast 201. The elongated grains are aligned with the extrusion direction in extruded 2014. Quantitative EDS analysis showed the two main constituent intermetallic phases present to be '$CuAl_2$' and insoluble $(CuFeMn)_3Si_2Al_{15}$. For DC-cast 'modified' 2014 and DC cast alloy 201, the structures consisted of α-aluminium with dendritic morphology and eutectic located between the dendrite arms.

3.2. Thixoformed microstructures before and after post-forming heat treatment

The first round of thixoforming at 632°C resulted in successful filling of the graphite die, but with varying degrees of porosity exhibited throughout the fingers as indicated by radiography. Fingers where no defects were evident from radiography were subjected to heat treatment before mechanical testing. Typical microstructures from a thixoformed finger with defects are shown in Fig. 3. Fine, equiaxed, spheroidal grains are dispersed in the liquid matrix (Fig. 3a). Occasional unrecrystallised elongated grains are retained in the finger (Fig. 3b) and porosity (Fig. 3c) can be observed. There is more liquid fraction near the surface of the finger than elsewhere and liquid segregation can be observed at the end of the finger (Fig. 3d). In the second round of thixoforming successful filling of the ceramic die resulted for 2014, 'modified' 2014 and 201. Less porosity occurred for thixoformed 2014 than in the first round with the graphite die but some elongated grains were still retained.

SEM Back-Scattered Electron images of as-thixoformed 2014 and material solutionised for 5 and 17 h at 500°C (quenched after solutionising but not aged) are shown in Fig. 4. In the as-thixoformed 2014, primary α-Al presents a globular morphology, with eutectic phase located in interglobular regions, as well

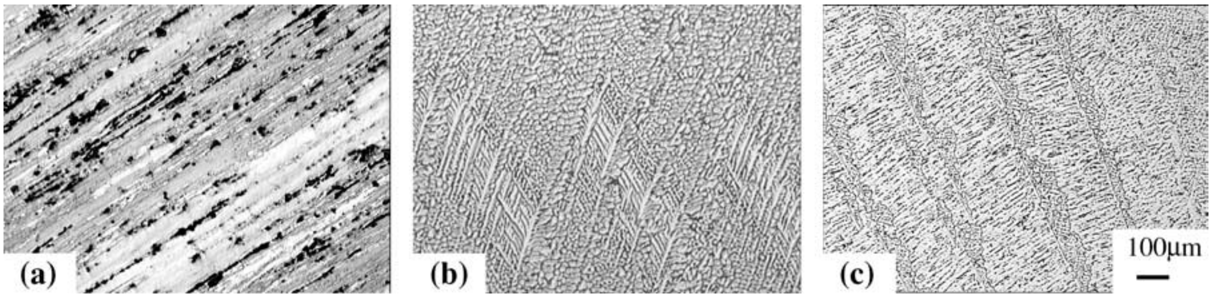

Figure 2 Optical microstructures of as received material: (a) Extruded 2014 (T6 condition), (b) DC-cast 'modified' 2014 and (c) DC-cast 201.

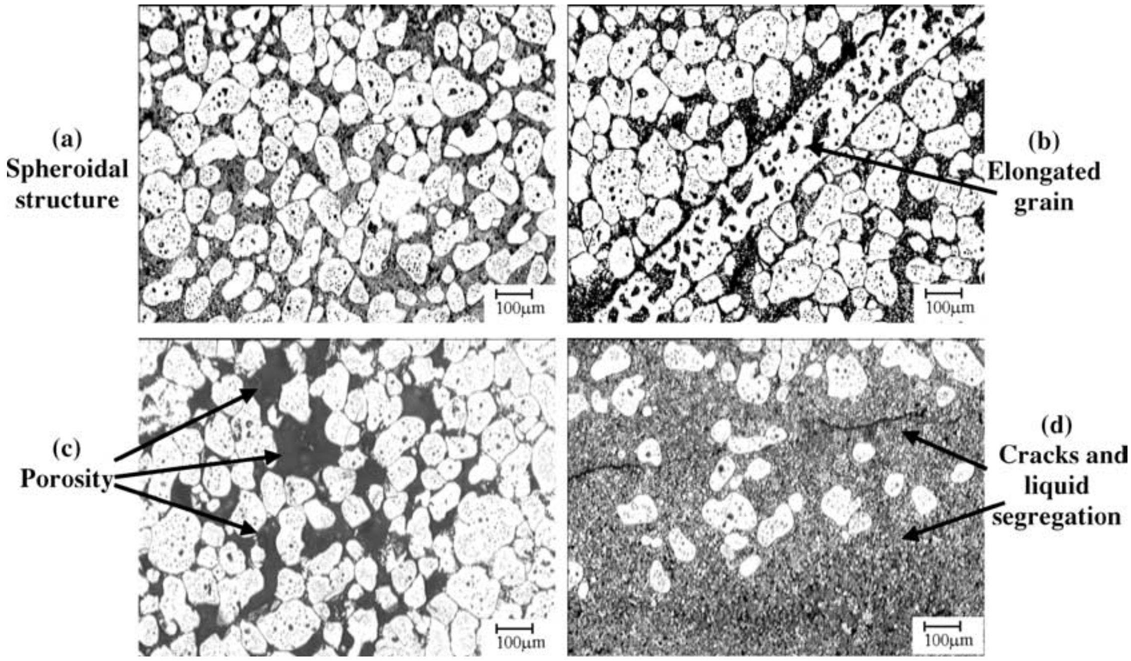

Figure 3 Optical microstructures of inadequately as-thixoformed 2014.

as in pools within the globules. The eutectic phase is derived from solidification of the "liquid" in the semi-solid slug. Quantitative EDS analysis showed that there is more Cu, Mg and Si in the interglobular eutectic in thixoformed 2014 than in extruded material. It also showed that some $CuAl_2$ phase still remained after 5 h solutionising at 500°C as shown in Fig. 4b, while it had completely dissolved after 17 h solutionising as shown in Fig. 4c. This was confirmed by Differential Scanning Calorimetry (DSC). Fig. 5 shows DSC traces for melting of thixoformed 2014 both before and after solution treatment. The relative area of the eutectic melting peak 3 over the total area of peaks 1, 2 and 3 is shown in Table II. The relative area percent decreases from 3.2% to 0 with the increase of the solution time to 17 h. No eutectic reaction occurred in thixoformed 2014 after 17 h solution treatment at 500°C. In agreement with this finding, eutectic liquid pools in grains were observed by TEM to have been retained in as-thixoformed 2014 after 1 h solution at 500°C, as shown in Fig. 6. This indicates that this standard solution treatment for conventional wrought alloy did not dissolve the Cu-rich eutectic entirely in thixoformed material.

3.3. Mechanical properties

The evolution of hardness during T5 or T6 ageing at 160 or 170°C is shown in Fig. 7. Low hardness values in as-thixoformed alloy 2014 were improved by post-forming heat treatment. Substantial hardening takes place for both 5 and 17 h prior solutionising, the hardness after 17 h being slightly higher than that after 5 h for both ageing temperatures (160 and 170°C). For an ageing temperature of 160°C, the initial stages of ageing gave a slow increase in hardness in the first 8 h, and then a rapid rise in hardness between 8 and 16 h. For 18 h ageing, the hardness of 5 and 17 h prior solutionised material is higher than that of 1 h, which is the standard solution treatment time for conventional wrought alloy 2014. At the higher ageing temperature of 170°C, two over-ageing peaks appeared during ageing from 2 to 64 h. This is in contrast with ageing at the lower temperature. For 170°C, the initial stages of ageing gave a rapid increase in hardness in the first 8 h followed by a rapid decrease in hardness and then a slow increase to another peak after 16 h ageing. In the T5 condition (thixoforming followed by ageing without a solutionising treatment), the hardness value is very low compared with the T6 condition. This is attributable to the lack of a fully supersaturated α-Al solid solution for the T5 condition.

On the basis of the above results, the 'optimised' heat treatment for thixoformed 2014 was taken to be 17 h solutionising at 500°C followed by water quenching, then ageing for 24 h at 160°C.

Figs 8 and 9 show yield strength and tensile strength vs. elongation to fracture for thixoformed 2014, 'modified' 2014 and alloy 201 in standard T6 temper compared with 2014 wrought targets and 201 permanent mould cast targets. The graphs also show the results for the optimised T6 heat treatment. The tensile properties of the thixoformed 201 in the standard T6 condition show a significant improvement over permanent mould casting and are very close to wrought 2014 targets with higher yield strength and tensile strength and slightly lower elongation to fracture.

The tensile properties of the thixoformed 2014 were much improved (from 310 to 420 MPa and from 360 to 470 MPa in yield strength and tensile strength, respectively) and close to those of forged 2014 targets (YS 414 MPa, TS 480 MPa) by using the optimised post-forming T6 heat treatment, rather than the conventional T6 heat treatment. However, the elongation to fracture is still poor and needs to be improved. There is significant variation in the values of elongation for different tensile samples all thixoformed under the same conditions and all machined from radiographically sound thixoformed fingers. One source of this variation may

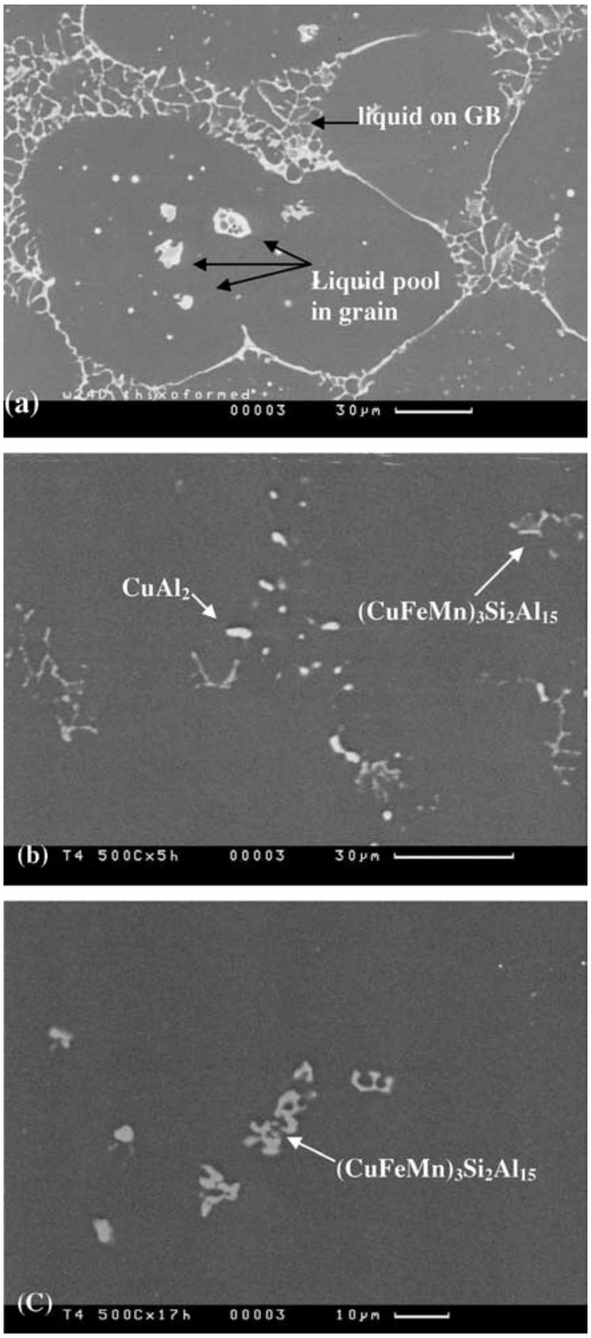

Figure 4 SEM back-scattered electron images of (a) as-thixoformed 2014: (b) and after T4 solution heat treatment 5 h, (c) 17 h at 500°C.

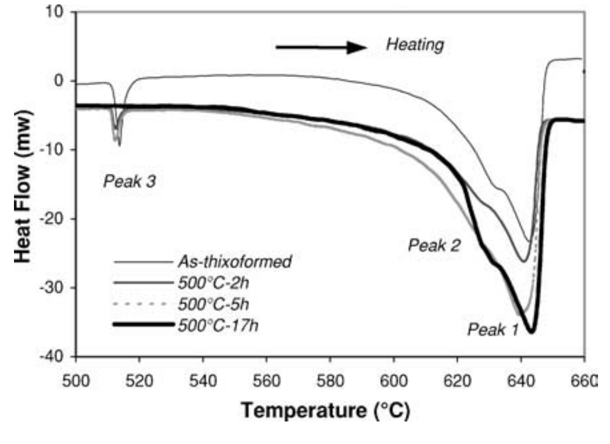

Figure 5 DSC traces for thixoformed 2014 before and after solution treatment at 500°C for 2, 5 and 17 h.

TABLE II Comparison of eutectic melting reaction ($L \leftrightarrow Al + \theta + Si + Q$) in DSC for different times of solution treatment of thixoformed 2014 at 500°C

Heat treatment	Melt onset temperature (°C)	Relative area[a] (%)
As-thixoformed	510°C	3.2 ± 0.1
T4 500°C-2 h	511°C	1.4 ± 0.1
T4 500°C-5 h	510°C	1.4 ± 0.1
T4 500°C-17 h	–	0

[a] Relative area = [(Area under peak 3/Total areas of peak 1, 2 and 3)] × 100 (see Fig. 5).

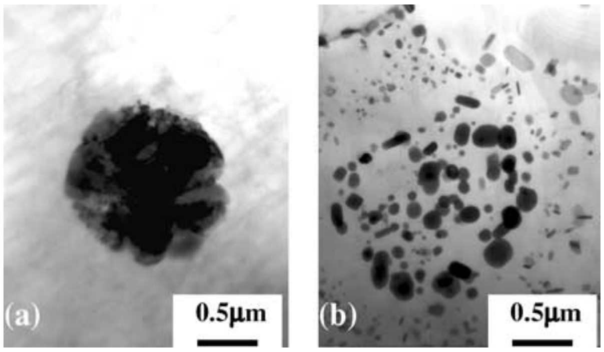

Figure 6 Solidified eutectic liquid pool in globule: (a) before and (b) after 1 h solution treatment at 500°C followed by water quenching.

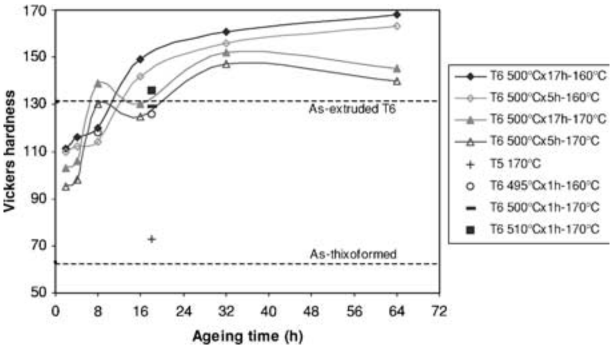

Figure 7 Evolution of hardness during ageing for thixoformed 2014 (error on hardness is ±3).

be associated with the fact that, when the fracture surface of a sample with poor elongation is examined, the appearance shows areas which have the appearance of uneven cobbled pavements [17]. This is thought to be associated with fracture through the liquid between the spheroids *during solidification.* The areas with the cobbled appearance are less than 1mm in diameter and therefore would not be revealed radiographically. They are however areas of weakness. It is not clear why some tensile samples contain these areas and not others.

Good elongation to fracture (∼20%) was obtained for thixoformed 'modified' 2014 in the T6 condition with a yield strength of 270 MPa and tensile strength of 408 MPa. This yield strength is significantly short of the wrought 2014 target and the tensile strength a little short. Heat treatment needs further development to improve the yield strength and tensile strength for thixoformed 'modified' 2014 because the optimised heat treatment for thixoformed 2014 is not suitable for

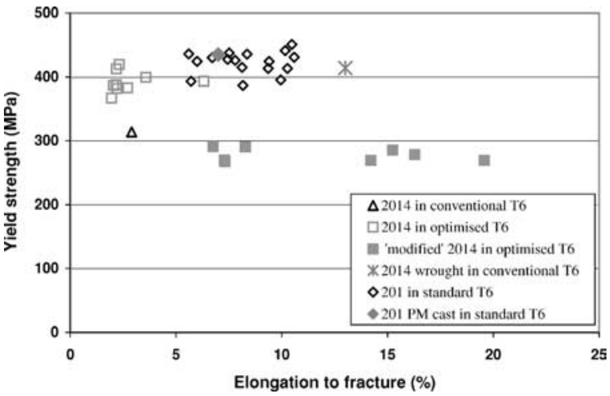

Figure 8 Yield strength vs. elongation to fracture for thixoformed Al alloys in T6 temper.

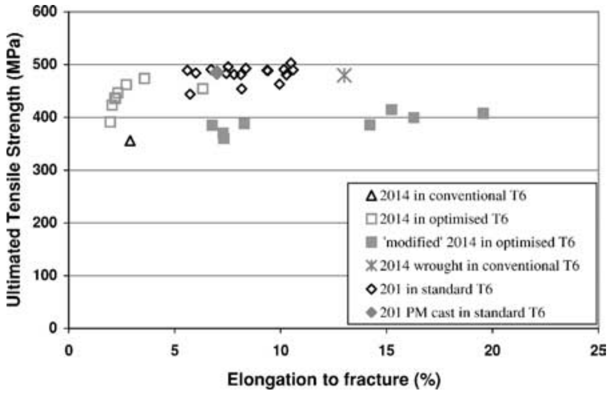

Figure 9 Tensile strength vs. elongation to fracture for thixoformed Al alloys in T6 temper.

thixoformed 'modified' 2014 due to the difference in compositions between the two alloys.

3.4. Fracture microstructure of tensile specimens

The longitudinal section of a thixoformed 2014 tensile specimen after the optimised T6 treatment (Fig. 10) showed that the failure is along the globule boundaries. No cracks were present in any of the globules. No significant globule growth was observed after the long solution treatment (17 h) in the optimised T6 treatment.

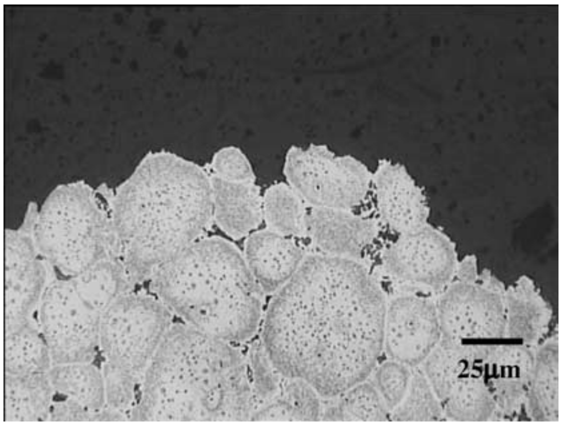

Figure 10 Longitudinal section of tensile fracture surface of thixoformed 2014 tensile specimen in optimised T6.

4. Discussion

Thixoforming is the shaping of metal components in the semi-solid state. The processing temperature is between the solidus and liquidus and spheroidal grains are distributed in the liquid matrix uniformly in the thixotropic feedstock before forming. The nucleation and solidification of eutectic liquid after forming is a critical factor for thixoforming to obtain the ideal microstructure without defects and with good mechanical properties. For conventional casting (either casting alloys or wrought alloys), the solidification interval (ΔT) is a critical factor that affects the tendency to hot tearing and the castability [18–20]. A large freezing range will make the last region to solidify subject to contraction stresses over a larger temperature interval and hence make the material more prone to hot tearing. For 2014, the solidification interval is 131 K and DSC shows that the last 5% liquid takes ~70 K to solidify. The porosity in thixoformed 2014 is thought to be related to this long temperature range for the final stages of soldification. In addition, oxides (Al_2O_3, SiO_2, MgO-based) were also found in the pores by EDS in the SEM. These oxides could originate in the starting material (as found by EDS on the Jeol 6400 SEM) or be introduced with the Cooling Slope. Tzimas and Zavaliangos [21] showed that when spray-cast 2014 and Al-4 wt%Cu alloys were heated into the semi-solid state and then cooled down to room temperature, porosity was present although no forming had been carried out.

A key question is why the thixoformed material takes longer to homogenize than conventional wrought alloy. This issue is related to the size and amount of soluble intermetallics and the distance over which diffusion must take place once dissolution has occurred. For a forged material, both soluble and insoluble intermetallics will be present. Before forging, preheating often takes place and some soluble intermetallics dissolve at this stage. During forging, remaining intermetallics are often broken up and hence reduced in size. In addition, they are dispersed through the microstructure. Hence, during homogenization the time taken to dissolve these fine, dispersed intermetallics is relatively short and the diffusion is distance in the region of a few tens of microns. In a thixoformed microstructure, the soluble intermetallics are concentrated in the interglobular liquid and in some pools within the globules (e.g., Figs 4 and 6). The soluble intermetallics tend to be relatively coarse (Fig. 4) and the distance over which diffusion must occur for homogenization to be achieved is larger. The time t taken for this diffusion for spheroids of 100 microns diameter (a typical size—see Figs 3 and 4) can be roughly estimated as x^2/D where x is the diffusion distance (here $\sim 50\ \mu$m) and D is the applicable solute diffusivity (here 4.8×10^{-14} m^2/s [22] for Cu in Al at 500°C) which gives $t = 14$ h. This is consistent with the experimental results in this paper, which suggest that most soluble Cu-rich phases have been dissolved after 17 h at 500°C for thixoformed 2014.

The tensile properties of the thixoformed 2014 were much improved and close to those of forged 2014 targets by using the optimised post-forming T6 heat treatment. However, the elongation to fracture was poor.

This is thought to be related to retained occasional elongated grains, coarse insoluble intermetallic compounds ($(CuFeMn)_3Si_2Al_{15}$), fracture through the liquid during solidification, shrinkage cavities and oxides (either from the as-received extruded material or introduced in the Cooling Slope process). Good elongation to fracture (∼20%) was obtained for thixoformed 'modified' 2014 in the T6 condition. This may be associated with the fact that the modified alloy excludes elements such as Fe and Si, which form coarse insoluble intermetallic compounds in normal 2014.

For the thixoformed 201, the alloying elements are concentrated in the interglobular regions. Permanent mould cast 201 will have the alloying elements concentrated in the interdendritic regions and the scale of the microstructure will be similar. Hence, the homogenization treatment applied conventionally to 201 with a two-step solution (2 h at 513°C followed by 17 h at 527°C) is similarly effective for thixoformed 201. The tensile properties of the thixoformed 201 showed a significant improvement over permanent mould cast 201 and are very close to wrought 2014 targets with higher yield strength and tensile strength and slightly lower elongation to fracture. The improvement probably occurs because there is less porosity than in permanent mould casting.

5. Conclusions

The standard solution treatment for conventional wrought alloy 2014 (1 h at 500°C) gave incomplete homogenisation for thixoformed material. Longer solution times (5 and 17 h) are required for complete solutionisation of $CuAl_2$. Low hardness values in as-thixoformed 2014 were increased by full heat treatment. For 18 h ageing at 160°C, hardness of 5 and 17 h prior solutionised material was higher than that of 1 h, which is the standard solution heat treatment for conventional wrought alloy 2014. The optimised post-forming heat treatment for thixoformed 2014 was determined to be solutionising for 17 h at 500°C followed by water quenching and then ageing for 24 h at 160°C. The tensile properties of thixoformed 2014 were much improved and close to those of forged 2014 using the optimised post-forming heat treatment. Good elongation to fracture (19.6%) was achieved for thixoformed 'modified' 2014 heat treated with the optimised schedule developed for 2014. The tensile properties of the thixoformed alloy 201 showed a significant improvement over permanent mould casting even though the conventional T6 heat treatment for this alloy was used.

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