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Development of Spraycast Al-Mg-Li Alloys

I.G. Palmer, S.C. Hogg, P.S. Grant

Department of Materials, University of Oxford, Parks Road, Oxford, OX1 3PH

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Abstract

Spraycast Al-Li alloys offer the potential to reduce weight and improve performance in aerospace structures. This paper describes an investigation to develop new alloys based on the Al-Mg-Li-Zr system. The overall objective is to develop a non-heat-treatable alloy with similar properties to those of the mechanically alloyed AA5091 alloy by the potentially cheaper spraycasting route. Relatively high Zr concentrations have been used to produce an as-sprayed microstructure which after primary processing contains fine dispersoids to control grain structure during secondary processing. Since the alloys are non-heat-treatable, hot working under different temperature and strain rate conditions has been used to generate a range of microstructures for evaluation. Preliminary mechanical properties have been obtained for an Al-5.31Mg-1.15Li-0.28Zr alloy.

1. Introduction

Al-Li alloys offer the potential to reduce weight and consequently improve performance in aerospace structures. The advantages of spraycasting for the production of Al-Li alloys include microstructural refinement, extended alloying capability and reduction in the levels of the embrittling elements Na and H [1]. However, as-sprayed alloys always contain a small fraction of residual porosity, typically <1% in alloys made on a semi-commercial scale, that can be successfully healed by hot working. Previous investigations of spraycast Al-Li alloys concerned the development of heat treatable alloys with relatively high Li contents [1]. Improvements in composition, microstructure and mechanical properties over conventionally processed alloys were demonstrated for alloys with Li and Zr concentrations in the range 2.5–5wt% and 0.2–0.5wt% respectively, and with grain sizes in the range 15-50µm. Na and H concentrations were reduced to a few ppm [1]. These alloys were processed primarily by extrusion and forging, and characterisation and mechanical property investigations are continuing.

The overall objective of the current work is to develop a non-heat-treatable Al-Mg-Li alloy with similar properties to those of the mechanically alloyed AA5091 (Al-4Mg-1.3Li-1.1C-0.4O) alloy, by the potentially cheaper and more scaleable route of spraycasting and hot working. In the study here, higher concentrations of Mg (5-6wt%) are used to increase the strength, and Zr (0.3-0.5wt%) additions are used to produce a distribution of stable, fine dispersoids after primary processing, as an alternative to the carbide and oxide-based dispersoids present in AA5091. The Li concentration is maintained at the AA5091 level of 1.3wt%. Since the alloys are designed to have high strength without complex heat treatments, hot working under different temperature and strain rate conditions has been used to develop a range of microstructures for evaluation.

In particular, the dynamic recrystallisation behaviour of the alloys is critical, in order to produce fine grain sizes of 1-5µm to provide a balance of strength and resistance to fatigue crack propagation.

Fine grain sizes have been produced in Al-Mg-Li 1420 alloy by multi-step forging [2], and in related spraycast Al-Mg-Li-Zr-Ti alloys by equal channel angular extrusion [3].

2. Experimental Procedure

Spraycasting was performed using a 60kg Al capacity Osprey spraycasting plant installed at Oxford University. The charge was melted in an induction heated crucible under an Ar inert atmosphere. A disposable ceramic fibre liner in the crucible prevented cross-contamination from one alloy to another, and improved safety. The melt was heated to the required temperature, and then degassed for 30min by bubbling Ar through the melt. The degasser consisted of an impermeable SiAlON shaft that resisted attack by the Al-Li melt and a porous graphite diffuser. After degassing, the melt was heated to the required temperature and poured into a ceramic fibre tundish containing a ceramic foam filter and maintained under an inert atmosphere. The melt flowed through a ceramic nozzle into the spray chamber, which was purged previously with N_2, and was atomised by high pressure N_2 jets in a scanning atomiser. The spray was collected on a steel collector plate and solidified into a ~30kg preform with a fine, uniform microstructure. The atomiser scanned between the centre and edge of the collector plate that was mounted on a ram and rotated and withdrew in order to maintain a constant spray distance between the atomiser and the preform top. The spraycasting variables were manipulated in order to obtain the required preform microstructure. They included the gas to metal ratio, the pour temperature, the spray distance and the metal flow rate.

Previous work on the degassing of Al-Li melts before spraycasting showed that degassing of the melt was essential in order to obtain low H concentrations and improved mechanical properties [1]. Even though there is a degassing effect as a result of the spraying process itself, degassing was still important as it may have also removed oxide films from the melt. In the present work, the PREFIL method [4] was used to investigate the effect of degassing on the melt cleanliness of an Al-5Mg alloy. Samples of liquid metal were taken before and after degassing, and the filtration behaviour through a standard filter measured. Figure 1 is a plot of mass filtered versus time for the degassed and non-degassed melt showing approximately 50% more metal filtered after degassing. The improved filtering confirmed that the degassing process also removed oxide films and other inclusions from the melt.

As-sprayed alloys contained a small amount of porosity, typically <1%, that was removed either by vacuum hot pressing (VHP) or by hot isostatic pressing (HIP). The thermal exposure during VHP or HIP also provided a precipitation treatment for the Al_3Zr dispersoid. The effect of heat treatments in the range 350-450°C on dispersoid precipitation and grain structure after hot working was investigated by microscopy and hardness indentation. Small 8mmØ x 10mm samples of consolidated material were compressed to strains in the range 1.3-2.0 at temperatures in the range 250-500°C, using a strain rate of approximately $10^{-3}s^{-1}$.

The samples were quenched in cold water within 30s of deformation and resulting microstructures were examined by a combination of optical microscopy, electron probe microanalysis (EPMA) in a JEOL JXA8800 and electron backscatter diffraction (EBSD) in a JEOL 6500 field emission gun SEM. Alloy compositions were investigated by inductively coupled plasma spectroscopy.

3. Results and Discussion

Two alloys designated 12 and 13 of target composition Al-(5-6)Mg-1.3Li-0.3Zr were spraycast in order to evaluate the effect of spraycasting parameters on the as-sprayed composition and microstructure, and for initial hot working experiments. Commercially pure Al was used for alloy 12, and high purity Al (Fe 0.018, Si 0.016) for alloy 13. Al-4Li, Al-50Mg and Al-15Zr master alloys were used. The weight of alloy 12 and 13 as-sprayed preforms was 19.5kg and 26kg respectively. 10mm thick vertical radial slices were cut from the preform centres for chemical analysis and microstructural evaluation. The as-sprayed preform compositions are shown in Table 1.

Table 1: Alloy composition (wt%).

Alloy	Mg	Li	Zr	Fe	Si
12	6.31	1.00	0.33	0.06	0.03
13	5.31	1.15	0.28	0.03	0.02

Figure 2 is an optical micrograph of the etched microstructure of alloy 12 showing equiaxed grains of ~11µm diameter and a small fraction of rounded porosity.

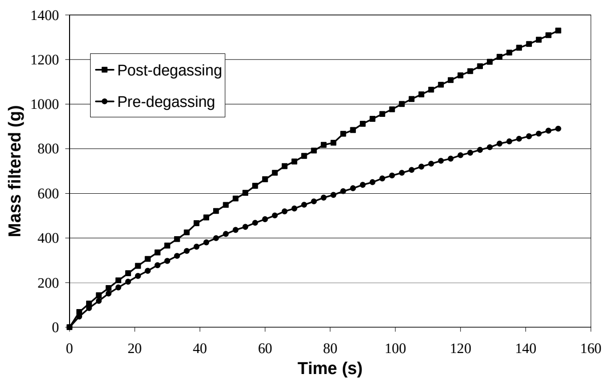


Figure 1: Effect of degassing on the filtration behaviour of an Al-5Mg melt.

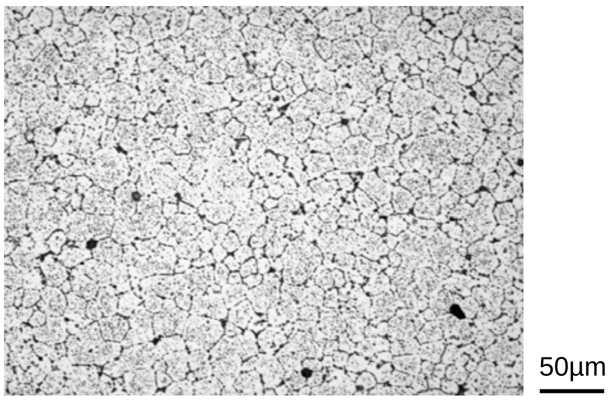
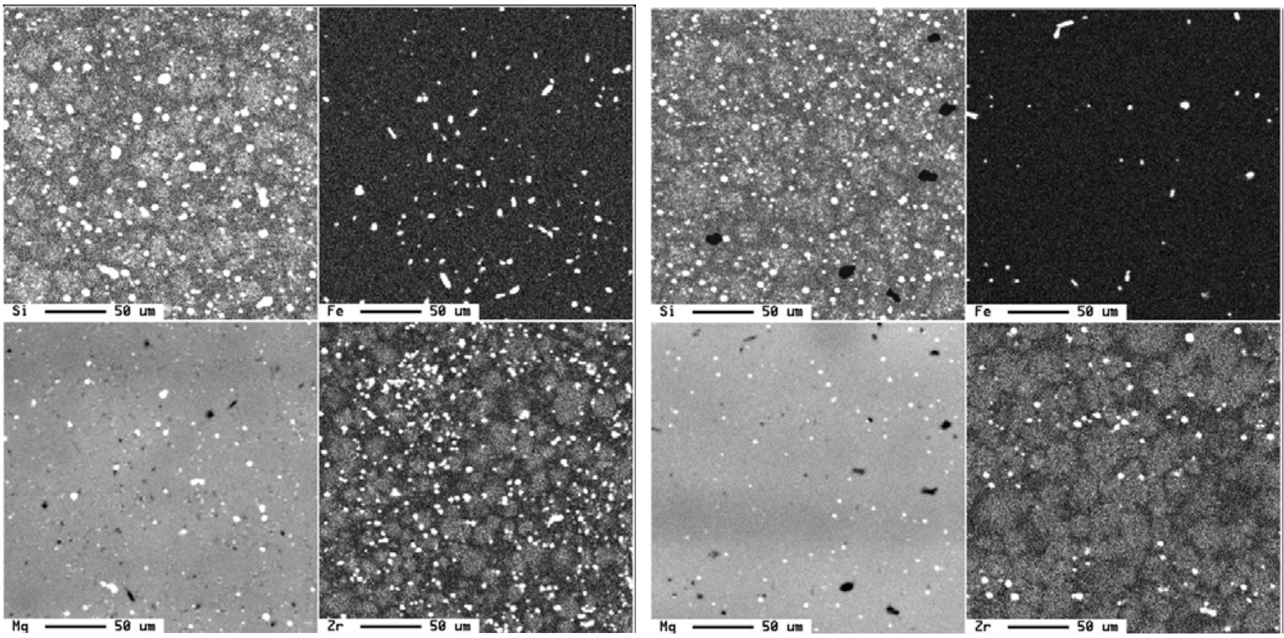


Figure 2: Microstructure of as-sprayed alloy 12.

Typical elemental distribution maps from EPMA for Fe, Si, Mg and Zr for both alloys are shown in Figures 3(a) and (b). Note that the apparent size of the particles observed is exaggerated because of the interaction zone effect. Most of the Si-containing particles were associated with a high Mg level, and were provisionally identified as Mg_2Si. The Fe-containing particles were not associated with Si or Mg, although there was some correlation with Zr distributions. Other than a weak correlation with Fe, the Zr-containing particles did not co-locate preferentially with other elements.

There were more Fe, Si and Zr-rich particles in alloy 12 than in 13, as expected from the higher concentrations of Fe, Si and Zr. Coarse (up to 0.5µm) Zr-containing grain boundary particles have been reported in spraycast Al-Li-Zr alloys and identified as the metastable Al_3Zr phase, precipitated in the liquid regions during solidification [5]. However, it was shown that most of the Zr remains in solid solution after spraycasting, and can be precipitated as fine coherent Al_3Zr dispersoid particles during heat treatment [5]. This precipitation was examined by hardness measurements and Figure 4 shows the variation in alloy 13 hardness as a function of time at 365 and 400°C. At 400°C, peak hardness of ~90VHN was reached after ~20h while at 365°C the hardness was ~95VHN and continued to increase slowly after 100h.



(a) (b)

Figure 3: EPMA elemental distribution maps for the as-sprayed alloys (a) alloy 12 (b) alloy 13.

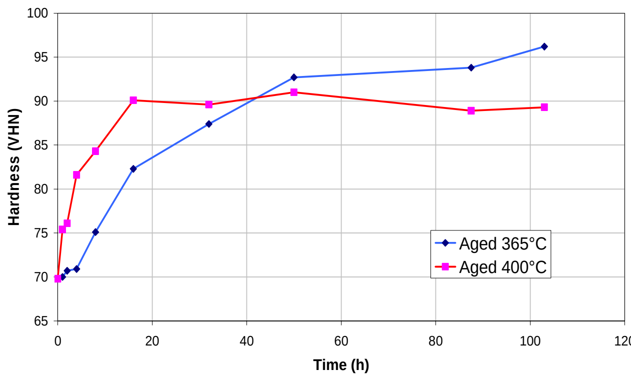


Figure 4: Ageing curves for the precipitation of Al_3Zr in alloy 13.

Hot compression experiments at a strain rate of $10^{-3}s^{-1}$ to a range of total true strains were performed on samples of alloy 12 given dispersoid precipitation treatments of 425°C/6h, or 450°C/6h. Figure 5(a) is the microstructure of a sample heat treated at 425°C/6h compressed to a strain of 1.3 at 250°C showing a largely unrecrystallised microstructure. Figures 5(b)-(d) show recrystallised microstructures, using 425°C/6h, strain = 2 at 500°C, 450°C/6h, strain = 2 at 425°C, and 450°C/6h, strain = 2 at 370°C respectively.

There was a progressive decrease in the dynamically recrystallised grain size, measured by EBSD, from ~10µm at 500°C to ~7µm at 370°C. Note that the black regions in the microstructures in Figure 5 are etch pits produced by electroetching, not residual porosity. A more comprehensive study of alloy 13 hot working behaviour is reported elsewhere [6].

Two hot working conditions were selected for processing larger samples of alloy 13 suitable for the evaluation of mechanical properties. Two cylinders 85mmØ x 150mm were HIPped to remove as-sprayed porosity and to precipitate the Zr. The HIP conditions were: temperature ramp at 120°C/h, hold at 400°C and 100MPa for 4h, and furnace cool.

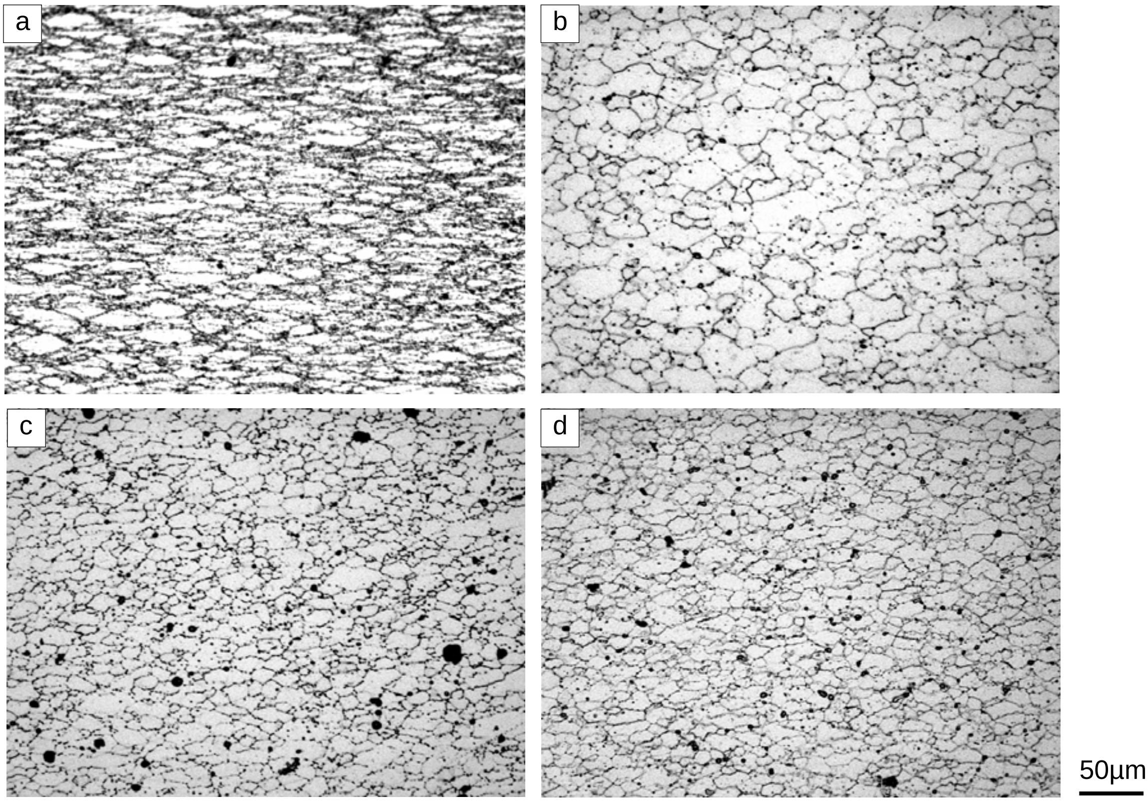


Figure 5: Microstructures of alloy 12 after hot compression tests (a) dispersoid precipitation 425°C/6h, true strain = 1.3 at 250°C (b) dispersoid precipitation 425°C/6h, true strain = 2 at 500°C (c) dispersoid precipitation 450°C/6h, true strain = 2 at 425°C (d) dispersoid precipitation 450°C/6h, true strain = 2 at 370°C

Each cylinder was then cut into two smaller cylinders 85mmØ x 72mm for upset forging at 400°C to slabs of thickness 23mm, representing a true strain of ~1, using strain rates of 10^{-2} s^{-1} and 10^{-3} s^{-1}. The tensile properties of the material forged at 10^{-2} s^{-1} were measured and are shown in Table 2. The 0.2% proof strength (PS) of 224MPa was relatively low, in comparison to the target alloy AA5091, but the work hardening rate, ultimate tensile strength (UTS) and elongation to failure of 29% were relatively high. This behaviour suggested that the alloy would be amenable to strengthening by cold work, as is often carried out on non-heat-treatable alloys. Thus, the effect of prior work in the temperature range 20 to -150°C on room temperature properties was investigated. Low temperature deformation was included because Al-Mg alloys have shown high flow strengths following deformation at cryogenic temperatures, together with reduced strain localisation effects and improved formability [7]. These improvements resulted from the higher work hardening rate and the suppression of dynamic recovery at cryogenic temperatures [7].

Samples were compressed to a 20% reduction in thickness at temperatures of 20, -100 and -150°C and the resulting tensile properties are again shown in Table 2. Significant increases in strength were obtained, for example a PS of 388MPa using prior working at room temperature, and an elongation to failure of 11%. Cryogenic working resulted in slightly higher levels of both strength and ductility. The properties achieved for all three processing conditions exceeded the target minimum property requirements for AA5091 of

PS=370MPa, UTS=455MPa, Elongation=6% [8]. In future work, the effect of higher levels of both Li and Zr will be examined and the processing route will be optimised further. It is envisaged that these modifications will result in further increases in strength.

Table 2: Tensile Properties of Alloy 13.

Condition	Working Temperature (°C)	0.2%PS (MPa)	UTS (MPa)	Elongation (%)
As-forged	-	224	370	29
Forged + worked	20	388	459	11
Forged + worked	-100	394	468	13
Forged + worked	-150	403	469	13

4. Conclusions

1. Al-Mg-Li-Zr alloys have been produced by spraycasting, with refined equiaxed grain structures, low levels of porosity and with sufficient control over alloy chemistry including impurities and inclusions.
2. Hot working at high temperatures in the range 250-500°C and a low strain rate of $10^{-3}s^{-1}$ resulted in dynamic recrystallisation. Within this range, lower deformation temperatures produced progressively finer recrystallised average grain sizes down to 7µm at 370°C.
3. Hot worked alloys have been strengthened further by cold work, which resulted in tensile properties that exceeded the target minimum specification for AA5091.

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