Microstructural characterisation and thermal stability of an Mg-Al-Sr alloy prepared by rheo-diecasting

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Abstract

A commercial Mg-6Al-2Sr (AJ62) alloy has been prepared by a semisolid rheo-diecasting (RDC) process. The microstructure of the RDC alloy exhibits typical semisolid solidification features, i.e., 8.4 vol% primary α-Mg globules (23 μm in diameter), formed in the slurry maker at the primary solidification stage, uniformly distributed in the matrix of fine α-Mg grain size (8.2 μm) and intergranular eutectic Al_4Sr lamellae, which resulted from secondary solidification inside the die. A ternary Mg-Al-Sr phase was also observed. Heat treatment revealed the extreme thermal stability of the RDC AJ62 alloy. The hardness showed little change up to 12 hours at 450°C, whilst the Al_4Sr eutectic lamellae were broken up, spheroidised and coarsened during the annealing. The RDC alloy offers superior mechanical properties, especially ductility, over the same alloy produced by high pressure die-casting.

Keywords: Microstructural characterisation, thermal stability, Mg-Al-Sr (AJ62) alloy, rheo-diecasting.

1. Introduction

In recent years, Mg alloys face a challenge in meeting performance requirements for components used in automobiles at elevated temperature. Strength, creep resistance and corrosion resistance as well as castability are the key issues. Much attention has been paid to research and development of cost competitive and high temperature creep resistant Mg based alloys. Such alloys would enable the automotive industry to use more Mg alloy components in order to produce lightweight and fuel efficient vehicles. Among them, Mg-Al-Sr alloys have shown superior creep performance and tensile strength at temperatures as high as 175°C [1–7], with the particular AJ62 (Mg-6Al-2Sr) alloy giving an excellent combination of creep performance, tensile properties and castability [1,3,4,6,7].

Semisolid metal (SSM) processing is a promising technology capable of producing high integrity components [8–10]. Rather than a liquid metal, as used in conventional die casting technology, SSM processing uses a semisolid slurry with substantially increased viscosity, resulting in controlled die filling and close to zero porosity in the final components. Compared with conventional die-casting, SSM processing has a number of advantages, such as low porosity, heat treatability, consistency and soundness of mechanical properties, the ability to make complex component shapes and longer die life. During the last two decades, SSM processing has been of considerable scientific and industrial interest, with a number of novel SSM technologies being developed. Among them, the rheo-diecasting process (RDC), developed recently at BCAST, Brunel University, has been shown to have advantages over other SSM technologies in terms of technological flexibility, component integrity, microstructural homogeneity and production cost efficiency [11–14]. In the present study, a commercial AJ62 alloy (Mg-6.3Al-2.5Sr) has been prepared by the semisolid RDC process. The microstructure and its evolution during heat treatment of the AJ62 alloy have been investigated. The tensile properties have also been evaluated and discussed in terms of the microstructure.

2. Experimental

The composition of the AJ62 alloy used in the present study was Mg-6.3Al-2.5Sr-0.36Mn, which was provided by MEL (Magnesium Elecktron, Manchester, UK). The alloy was prepared using both RDC and HPDC processes with similar processing parameters for comparison. The RDC process is an innovative one-step SSM processing technique for manufacturing near net shape components of high integrity directly from liquid alloys. Detailed description of the RDC process can be found elsewhere [15–18]. The RDC equipment consists of two basic functional units; a twin-screw slurry maker and a standard cold chamber high pressure die casting (HPDC) machine. The twin-screw slurry maker has a pair of screws rotating inside a barrel. The specially designed screw profiles are fully intermeshing and self-wiping. The fluid flow inside the slurry maker is characterised by high shear rate and high intensity of turbulence, with a cyclic variation of shear rate. In the RDC process, a predetermined dose of alloy melt is fed into the slurry maker, which is set to a desired temperature between the liquidus and solidus. The liquid alloy is cooled continuously to the processing temperature while being mechanically sheared by the pair of screws. Depending on the barrel temperature, a certain volume fraction of the alloy melt solidifies inside the slurry maker, converting the liquid into semisolid slurry. After being sheared for a period of time, the slurry is then transferred to the shot sleeve of the HPDC machine for component shaping. In the present study, the AJ62 alloy ingot was melted at 715°C under the protection of N_2 + 0.5 vol%SF_6 gas mixture and fed into the slurry maker. The slurry maker was operated at 612°C. The rotation speed of the twin-screw was 500 rpm, and the shearing time between 20 and 40 s. A 280-ton cold chamber HPDC machine was used to produce standard tensile test samples. The mechanical properties were evaluated by tensile tests using an Instron tensile machine.

The microstructure of the samples was examined by optical microscopy (OM) with quantitative metallography. The specimens for OM were prepared by the standard technique of grinding with SiC abrasive papers and polishing with an Al_2O_3 suspension solution. A Zeiss optical imaging system was utilised for the OM observations and the quantitative measurements. Scanning electron microscopy (SEM) was carried out with a FEG Zeiss Supera 35 machine, equipped with an energy dispersive spectroscopy (EDS) facility and operated at an accelerating voltage of 5 to 20 kV. EDS measurements were performed to obtain chemical information for the phases using a fixed accelerating voltage of 15 kV, calibrated with a standard sample at every session.

3. Results and Discussion

3.1 Solidification microstructure

Figure 1 shows the typical microstructure of AJ62 alloy produced by the RDC process, in comparison with the same alloy prepared by HPDC. The general view shows that the RDC alloy exhibits a non-dendritic and more uniform microstructure than the HPDC alloy. Apart from the relatively large α-Mg globules formed inside the slurry maker, the solidification structure of the RDC alloy is composed of fine α-Mg particles, contributed by solidification of the remaining liquid inside the die, which is referred to as secondary solidification. Detailed SEM observation revealed that the fine α-Mg particles, formed inside the die, have equiaxed morphology and are delineated by the lamellar eutectic phase, as shown in Figure 2. The fine α-Mg particles were measured to be 8.2 μm in size, compared to 23 μm for the primary globules. A dendritic morphology of the primary phase, typical of most conventional cast alloys, was hardly observed in the RDC alloy samples. In contrast, the AJ62 alloy prepared by HPDC exhibits a coarse and non-uniform

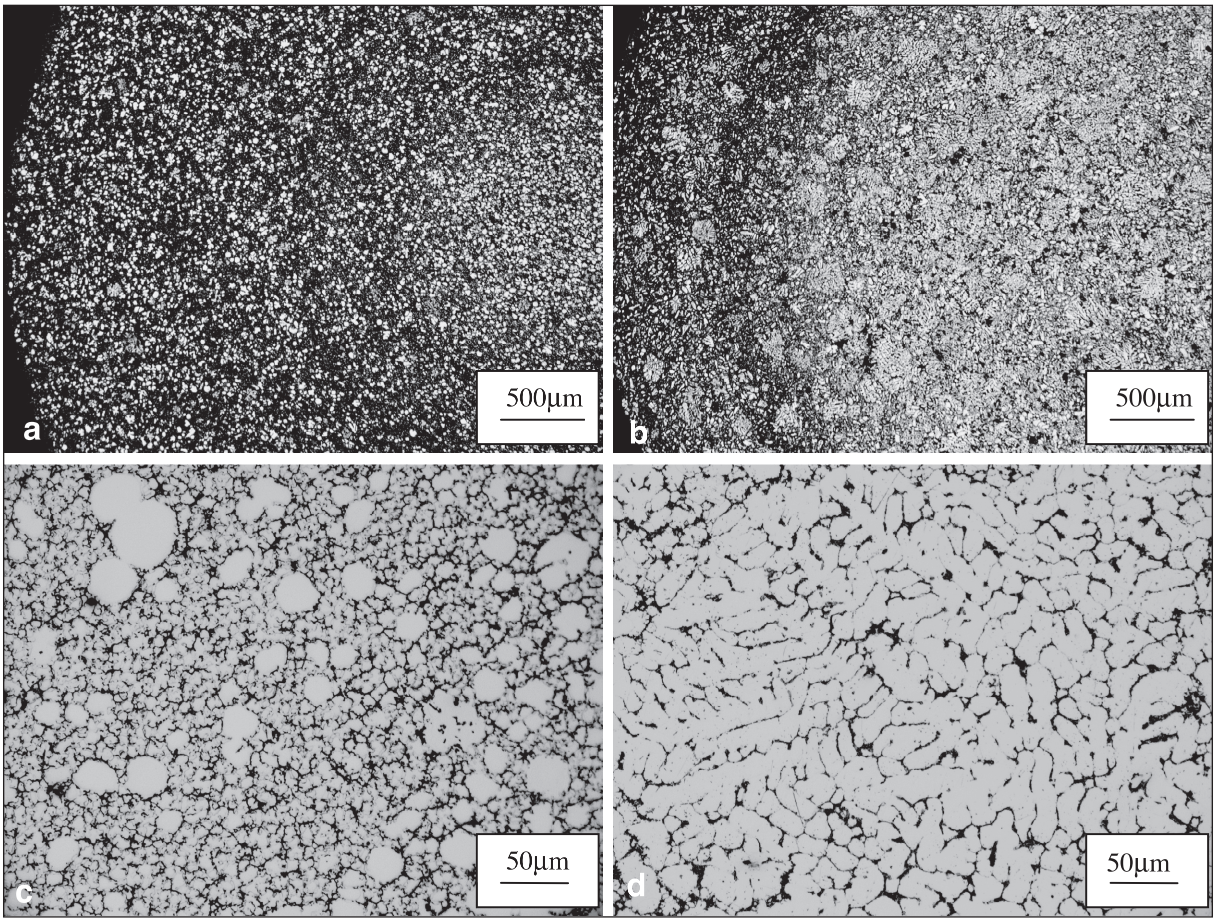

Figure 1: Optical micrographs show the microstructure of the AJ62 Mg-6Al-2Sr alloy produced by (a) and (c) rheo-diecasting (RDC), and (b) and (d) high pressure die casting (HPDC).

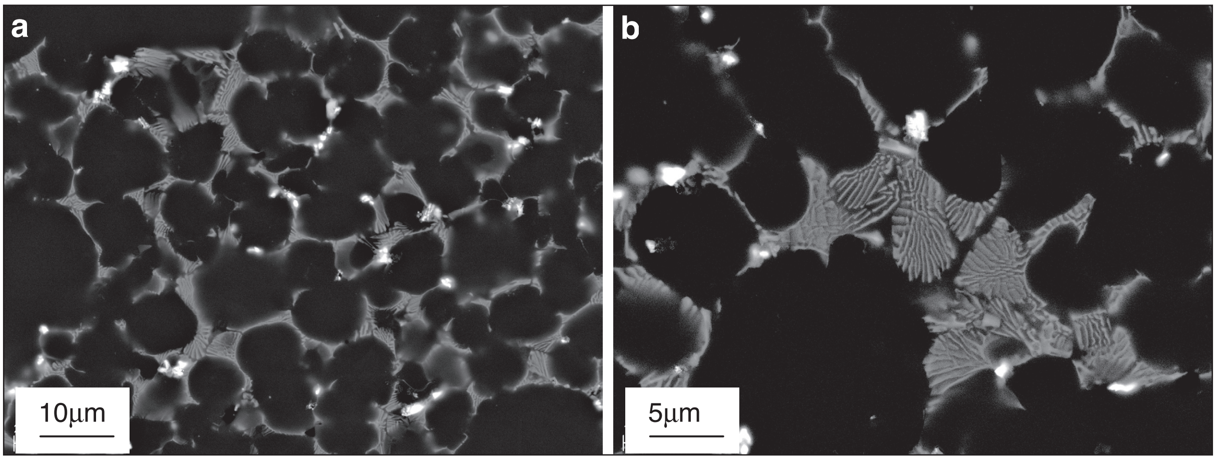

Figure 2: SEM backscattered electron images showing (a) fine α-Mg particles formed by secondary solidification and (b) lamellar structure for the eutectic Al_4Sr intermetallic.

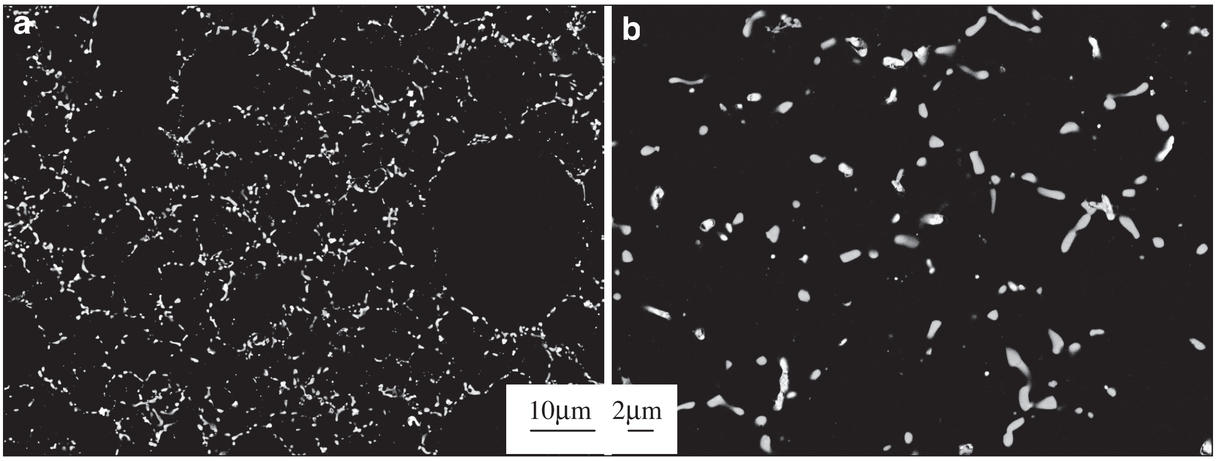

Figure 3: Microstructural evolution of the RDC AJ62 Mg alloy during heat treatment at 450°C for up to 12 h. (a) 450°C for 1 h, (b) 450°C for 12 h. It is seen that the eutectic intermetallic phase has spheroidised and coarsened with increasing treatment time.

microstructure with the primary α-Mg particles being dendritic, as shown in Figure 1. The dendrite arm spacing of the HPDC AJ62 alloy was measured to be 16.3 μm, double the size of the fine α-Mg particles of the RDC alloy.

The microstructure of Mg-Al-Sr alloys with different concentrations (varying Sr/Al ratios) of Sr and Al has been investigated before [1,4–6]. Two types of second phase were observed in the alloys with higher Sr contents. All these intermetallic phases are located at the grain boundaries of the α-Mg primary particles. The total volume fraction of second phase particles was measured to be 6.02 vol% for the Mg-6.3Al-2.5Sr alloy used in the present study. SEM/EDS analysis revealed the lamellar structure of the inter-granular Al_4Sr eutectic intermetallic [1,4,6], while an Mg-Al-Sr ternary intermetallic phase was also observed occasionally with a brighter contrast, a coarser size and blocky-shaped structure, as shown in Figure 2. This phase has been tentatively identified as $Al_3Mg_{13}Sr$ previously [1,6]. SEM/EDS and XRD analyses revealed no $Mg_{17}Al_{12}$ in the RDC alloy.

Sr in the AJ series alloys combines with Al and makes the formation of the $Mg_{17}Al_{12}$ phase more difficult, since the latter phase is thought to facilitate grain boundary migration and therefore decrease the creep resistance. The absence of $Mg_{17}Al_{12}$ phase in the microstructure of the AJ62 alloy is probably one of the main reasons for the superior creep resistance of the Mg-Al-Sr (AJ series) alloys [1]. In fact, Al is added to improve castability and room temperature tensile properties. The Sr content needs to be high enough to avoid any increase in Al supersaturation of the primary α-Mg phase and also to prevent formation of the β-$Mg_{17}Al_{12}$ phase [2]. The improvement in creep resistance of the Mg-Al-Sr alloy is due to strengthening by the thermally stable intermetallic phases [1].

The solidification behaviour of the RDC AJ62 alloy is characterised by two solidification stages: primary solidification inside the slurry and secondary solidification inside the die cavity. Inside the slurry maker, a certain volume fraction of solid forms and takes the globular shape because of the intensive shearing [18]. The secondary solidification, which has been extensively investigated [19] starts when the slurry leaves the twin-screw slurry maker and occurs under no forced convection.

The remaining liquid in the slurry has uniform temperature and composition fields due to the prior intensive shearing. With the large cooling rate provided by the die, nucleation is expected to take place throughout the entire remaining liquid with a high nucleation rate giving numerous nuclei which grow and solidification finishes before growth instability even occurs, preventing them from developing dendritically. This produces the fine non-dendritic α-Mg particles shown in Figure 2.

3.2 Microstructural evolution during heat treatment

Heat treatment has been carried out to investigate the thermal stability of the RDC AJ62 alloy and its microstructural evolution. Figure 3 shows the microstructure of the alloy after treatment at 450°C for 1 and 12 hours. The only microstructural modification is breaking-up, spheroidisation and coarsening of the Al_4Sr eutectic phase.

Figure 4 shows hardness and particle size of the eutectic Al_4Sr phase against time during treatment at 350 and 450°C. The alloy is thermally stable at temperatures as high as 450°C with little change in hardness or in percentage of the eutectic phase. However, the eutectic intermetallic does spheroidise and coarsen with increasing time, as shown in Figure 4b. A power law was used to fit the experimental data and to extract the growth constants according to the well-established classic law, $D^n - D^n_0 = Kt$. It was found that the value of n is 2.75, in between 2 and 3.

Figure 5a shows a typical microstructure of the RDC alloy after treatment for 2 hours at 450°C. It can be seen that the majority of the eutectic lamellae have been broken-up and have started to coarsen. Particle size distribution of the modified eutectic Al_4Sr phase after treatment at 450°C for 2 hours is shown in Figure 5b, with the average size being measured to be 510 nm in diameter.

As mentioned above, two types of second phase compounds were observed in the RDC AJ62 alloy, with the major one being the Al_4Sr intermetallic. Quantitative metallography indicated that the total volume fraction of the second

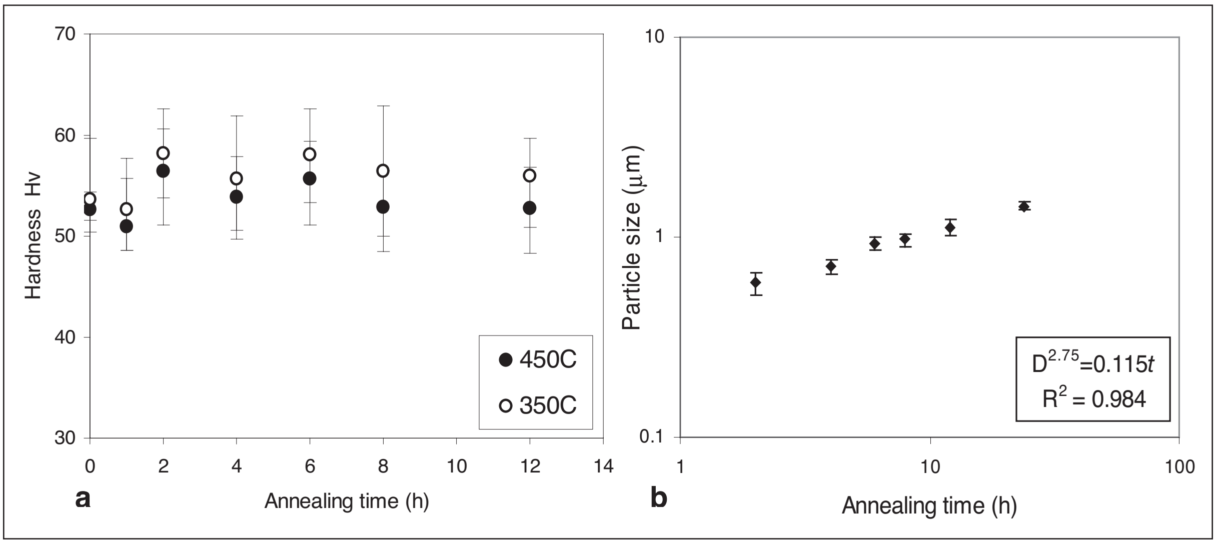

Figure 4: (a) Hardness of the RDC AJ62 alloy as a function of treatment time showing little change in hardness up to 12 h at 350 and 450°C. (b) Variation in particle size of the eutectic Al_4Sr intermetallic as a function of treatment time at 450°C. The mean size of the eutectic intermetallic increased from 0.51 to 1.43 μm as the treatment time increased from 2 to 24 h.

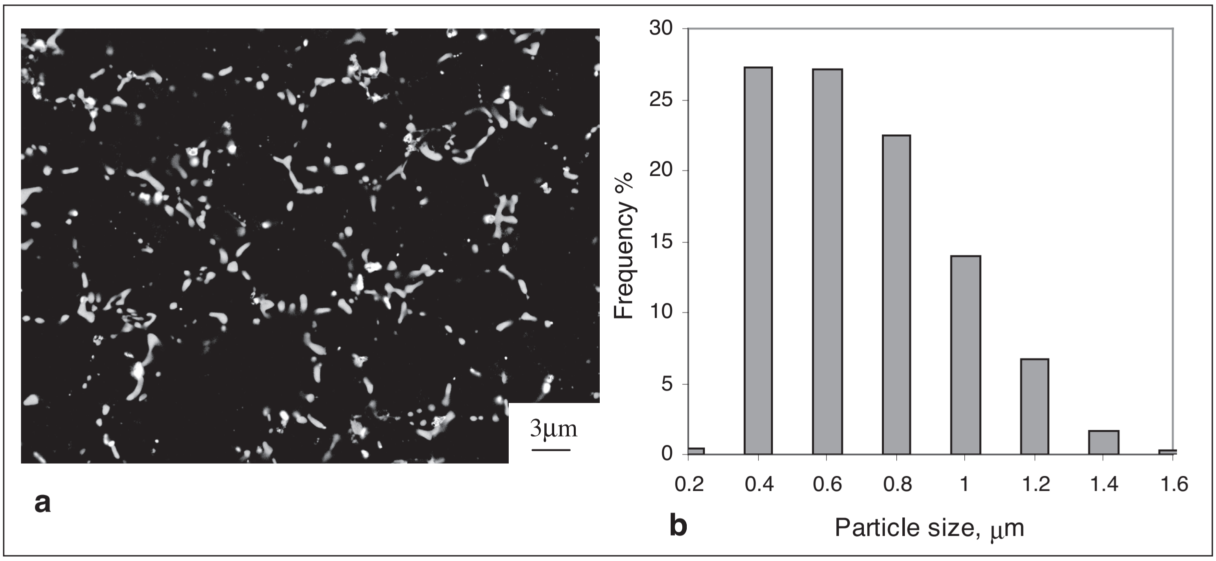

Figure 5: (a) SEM (BEI) image showing the typical microstructure of RDC AJ62 Mg alloy after treatment at 450°C for 2 h.
(b) Quantitative measurement of the modified eutectic particle size distribution. The morphology of the eutectic Al_4Sr phase changes from lamellar to spherical.

Table 1: Tensile properties of the RDC AJ62 alloy compared to that from HPDC.

Conditions	Ultimate Tensile Strength (MPa)	Yield Strength (MPa)	Elongation (%)	Reference
HPDC (F condition)	227	138	7.0	[6]
HPDC (F condition)	225.3 ± 11.3	123.5 ± 4.0	7.43 ± 1.49	This work
RDC (F condition)	243.9 ± 10.4	129.8 ± 3.4	9.05 ± 1.01	This work
RDC (Heat treated, 2 hrs@450°C)	257.6 ± 7.7	121.4 ± 5.2	12.01 ± 1.06	This work

phase particles was 6.23 vol% for Mg-6.3Al-2.5Sr alloy used in the present study. Long time treatment at elevated temperatures up to 450°C gave little change in the volume fraction of the intermetallic phases present in the alloy. The Al_4Sr intermetallic is extremely stable at these temperatures only with its morphology being spheroidised, similar to the modification of eutectic silicon in A356/357 Al alloys during T6 treatment [20,21]. It was proposed that the creep resistance of the AJ alloys is related to the low Al supersaturation of the primary α-Mg and the absence of the β-$Mg_{17}Al_{12}$ phase due to the precipitation of thermal stable Al- and Sr-rich phases.

3.3 Mechanical properties

The mechanical properties have been evaluated by tensile testing for both RDC and HPDC AJ62 alloy under as-cast and heat treated conditions with the results given in Table 1. It is seen that the RDC alloy offers superior mechanical properties, i.e., higher ultimate tensile strength (UTS), higher yield strength, and especially significant improvement in ductility, over the same alloy produced by traditional HPDC. More importantly, the present study shows that specially designed heat treatment schedule, such as treatment at a temperature as high as 450°C, can be used to enhance the mechanical properties, particularly to significantly improve the ductility, as shown in Table 1.

The increase in elongation after heat treatment is attributed to the modification of the eutectic morphology. Previous studies [13,14] have shown that, for RDC Mg alloy, T_x treatment modified the $Mg_{17}Al_{12}$ phase network into isolated particles located along the grain boundaries, and resulted an increase in the elongation. In the present study, similar treatment makes the Al_4Sr eutectic morphology change from lamellar to spherical, which will reduce the interface between the eutectic and the α-Mg matrix, and then reduce the possibility of crack initiation and crack propagation along the interfaces.

Sr content plays an important role in determining the mechanical properties of the AJ alloys. When Sr concentration is low, the binary $Mg_{17}Al_{12}$ phase will form. This phase is thought to facilitate grain boundary migration and therefore decreases the creep resistance. Therefore the Sr content needs to be high enough to avoid the formation of the Mg-Al binary compound. On the other hand, the elongation of the AJ alloys is sensitive to the Sr content. A modified AJ62 alloy, AJ62Lx (1.9 wt%Sr), has been proposed and it has elongation up to 9 to 11% [6], higher than for AJ62x (2.4 wt%Sr [6]). In the present study, however, the elongation of the RDC AJ62 alloy with 2.5 wt%Sr is as high as 9.05% in the as-cast condition. Furthermore, the UTS and elongation can be significantly improved by heat treatment with little sacrifice of yield strength.

4. Conclusions

The microstructure of the rheo-diecast Mg-6.3Al-2.5Sr (AJ62) alloy exhibits typical semisolid solidification features, i.e., 8.4 vol% primary α-Mg globules (23 μm in diameter), formed in the slurry maker at the primary solidification stage, uniformly distributed in the matrix of fine α-Mg grain size (8.2 μm) and intergranular eutectic Al_4Sr lamellae, which resulted from secondary solidification inside the die. A ternary Mg-Al-Sr phase was also observed.

Heat treatment revealed the extreme thermal stability of the rheo-diecast AJ62 alloy. The hardness showed little change up to 12 hours at 450°C, whilst the eutectic Al_4Sr lamellae were broken up, spheroidised and coarsened during the heat treatment.

The rheo-diecast AJ62 alloy offers superior tensile mechanical properties, especially ductility, over the same alloy produced by conventional high pressure die-casting. Moreover, specially designed heat treatment schedules can be used to enhance the mechanical properties, particularly elongation.

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