Microstructure and age hardening response of cast Al-Mg-Sc-Zr alloys

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Al-Mg-Sc-Zr alloys have been investigated in recent years [1–3]. Sc and Zr additions to Al-5%Mg alloy were shown to refine the as-cast grain structure, increase strength of rolled sheets and reduce sub-grain size [4]. A series of alloys containing 0.30–0.35%Sc, 0.10–0.16%Zr and 1.1–6.3%Mg were produced in rolled sheet form and shown to possess higher tensile properties compared to conventional Al-Mg based alloys [3]. In addition, the grain size of Zr containing alloy was found to be more stable at high temperatures than that of Zr-free Al-3%Mg-0.2%Sc alloy under superplastic deformation conditions [5].

The effect of Mg content in the Al-Mg-Sc-Zr alloy system on as-cast grain structure and hardening on direct aging has not been studied systematically. Such a study is considered important, since it can lead to the development of alloys with improved castability and weldability combined with high strength compared to conventional casting alloys. Also hardening on direct ageing of the as-manufactured components may be an added advantage in situations where a separate solution treatment may not be possible or may be undesirable due to problems associated with residual stresses and distortion.

Chemical compositions of various alloys investigated are given in Table I. The alloys are designated by the nominal Mg content, and will be referred as such. The starting materials used in making the alloys were: 99.85% pure Al, Mg-30%Zr and Al-2%Sc ingots. The alloys were melted in an air induction melting furnace under flux cover, superheated to 760 °C, cooled to 700 °C and cast in dried M.S. molds as 25 mm diameter × 200 mm long ingots. Samples for metallography and age hardening studies were extracted leaving 25 mm height from the bottom of the ingots. Standard metallographic procedures were used. Samples were aged without prior homogenization or solution treatment in muffle type furnaces with job temperature variations within ±2 °C. Vickers hardness using 5 kg load was measured on the cross section of the ingots. Foils were

prepared from selected samples for transmission electron microscopy using standard electro-polishing techniques. A Philips EM300 TEM was used for studying the microstructures.

As-cast grain structures of OMg and 0.2Mg alloys were studied previously [6], and are reproduced in Fig. 1 for reference. These samples were etched using concentrated Keller's reagent. Coarse columnar grain structure was observed in both the alloys, with a small equiaxed zone near the ingot center in the Zr containing (0.2Mg) alloy. The same study had revealed that a minimum of 0.45%Sc with ≈0.14%Zr was required for grain refinement. The grain refinement was related to the presence of coarse (5–15 μm) Al$_3$(Sc$_{1-x}$Zr$_x$) particles which had a cube-cube orientation relationship with α-Al [6].

The as-cast microstructures of the 1Mg-8Mg alloys are shown in Fig. 2. The grain boundaries could not be clearly identified due to their slower response to
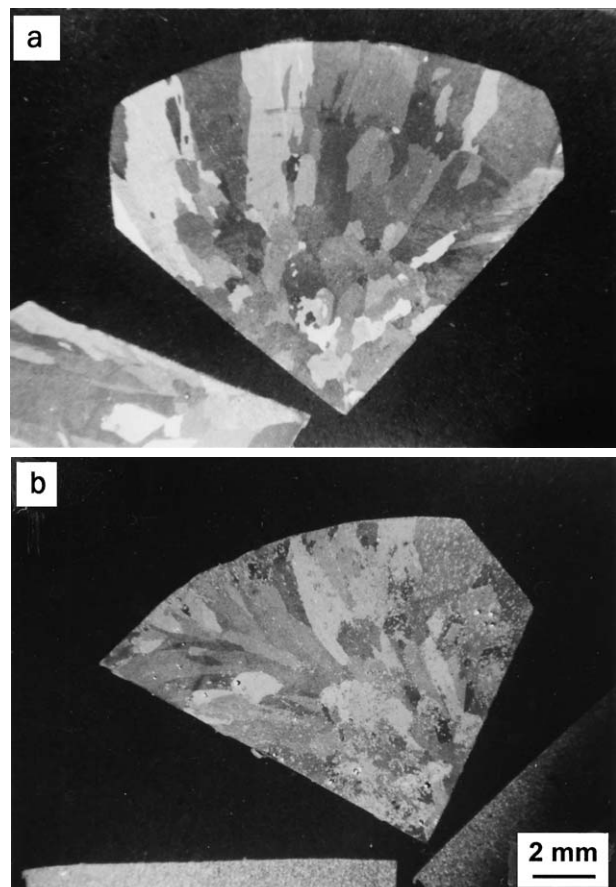
TABLE I Alloy compositions (in wt%), balance Al

Alloy	Mg	Sc	Zr
0Mg	–	0.24	–
0.2Mg	0.22	0.24	0.14
1Mg	1.06	0.26	0.14
3Mg	2.76	0.26	0.14
4Mg	4.11	0.26	0.14
8Mg	8.10	0.25	0.13

Figure 1 Optical microstructures of (a) 0Mg and (b) 0.2Mg alloys in as cast condition.

Figure 3 Optical microstructure of 8Mg alloy in as cast condition, showing presence of an $Al_3(Sc_{1-x}Zr_x)$ particle.
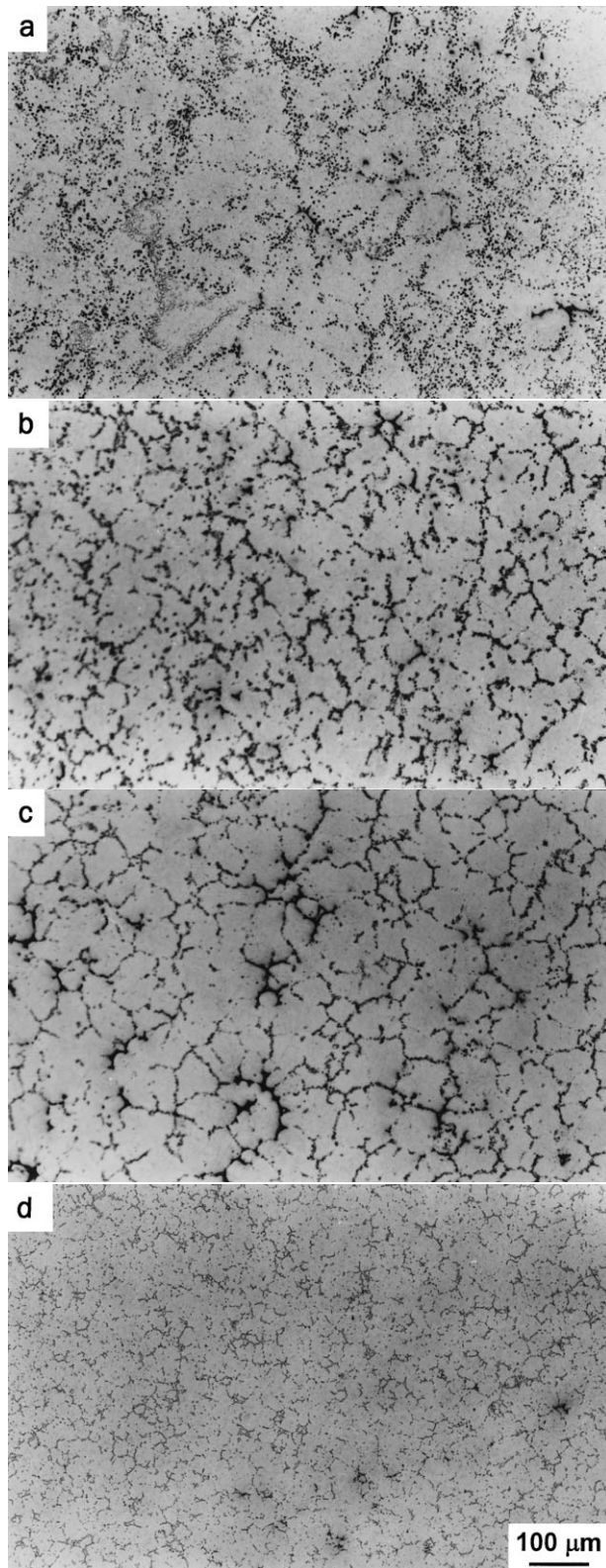
Figure 2 Optical microstructures of (a) 1Mg, (b) 3Mg, (c) 4Mg and (d) 8Mg alloys in as cast condition.

etching compared to that of the solute enriched interdendritic regions. Further examinations of the as-cast microstructures revealed the presence of blocky particles in the 8Mg alloy (Fig. 3) but not in the remaining alloys. These particles were similar in appearance to those found in the 0.45%Sc + 0.14%Zr containing alloy [6], and are believed to be $Al_3(Sc1_{1-x}Zr_x)$, known to nucleate α-Al. Considering the present results and those of Yin *et al.* [4], where the authors reported presence of 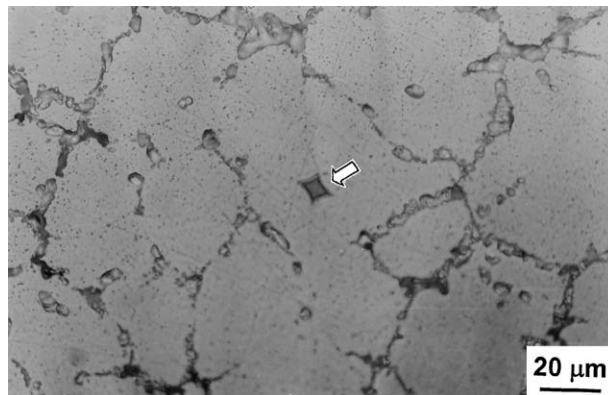$Al_3(Sc_{1-x}Zr_x)$ particles in Al-5%Mg-0.2%Sc-0.1%Zr alloys, it is felt that the presence of Mg (in addition of about 0.10–0.14%Zr) reduces the Sc content needed for the formation of the aluminide particles to about 0.2–0.3%Sc.

Examination of a pseudo-binary Al-Mg-Sc phase diagram in the range covering 0–1%Sc and simultaneously varying Mg from 17 to 0% [1] suggests that, for a 8%Mg + 0.5%Sc alloy, the first phase to form from the liquid is Al_3Sc. Though the section of the diagram corresponding to the exact composition from the current experiments was not available, it could be argued that a similar solidification sequence may follow for 8%Mg + 0.25%Sc alloy in the presence of 0.13%Zr. The basis for such an argument is that 35–50% Zr is known to replace Sc in Al_3Sc precipitates [1], and also the findings that Zr reduced Sc content required to form aluminide particles [4, 6]. The present work indicates that the minimum Mg content required for primary Al_3Sc particle formation is more that 4.11%, but this needs to be verified.

Fig. 4 shows microstructures of all the Mg containing alloys in the homogenized (430 °C/24 h/aircooling) condition. The grain boundaries can be seen clearly, since most of the interdendritic segregation products were dissolved. A qualitative comparison of the micrographs shows that the grain size remains largely unchanged up to 4%Mg, and drops significantly for 8%Mg. These results, and taking observations of Fig. 3 into consideration, confirm that the presence of aluminide particles is necessary for grain refinement in Al-Mg-Sc-Zr alloys.

Fig. 5 shows the isochronal and isothermal age hardening curves, respectively, for all the alloys. In general, hardness increased with increasing Mg content (even without aging), as would be expected from the known solid solution hardening effect of Mg in Al alloys [7]. The difference in the hardness between various alloys remained approximately constant over the aging temperatures studied. This indicates that the Mg induced hardness was purely additive and independent of the transformations related to Sc and Zr.

For each composition, the peak aging temperature was about 300 °C, which is much higher than the aging temperatures for most commercial alloys (120–190 °C). The peak aging time for 300 °C aging is 10 h.
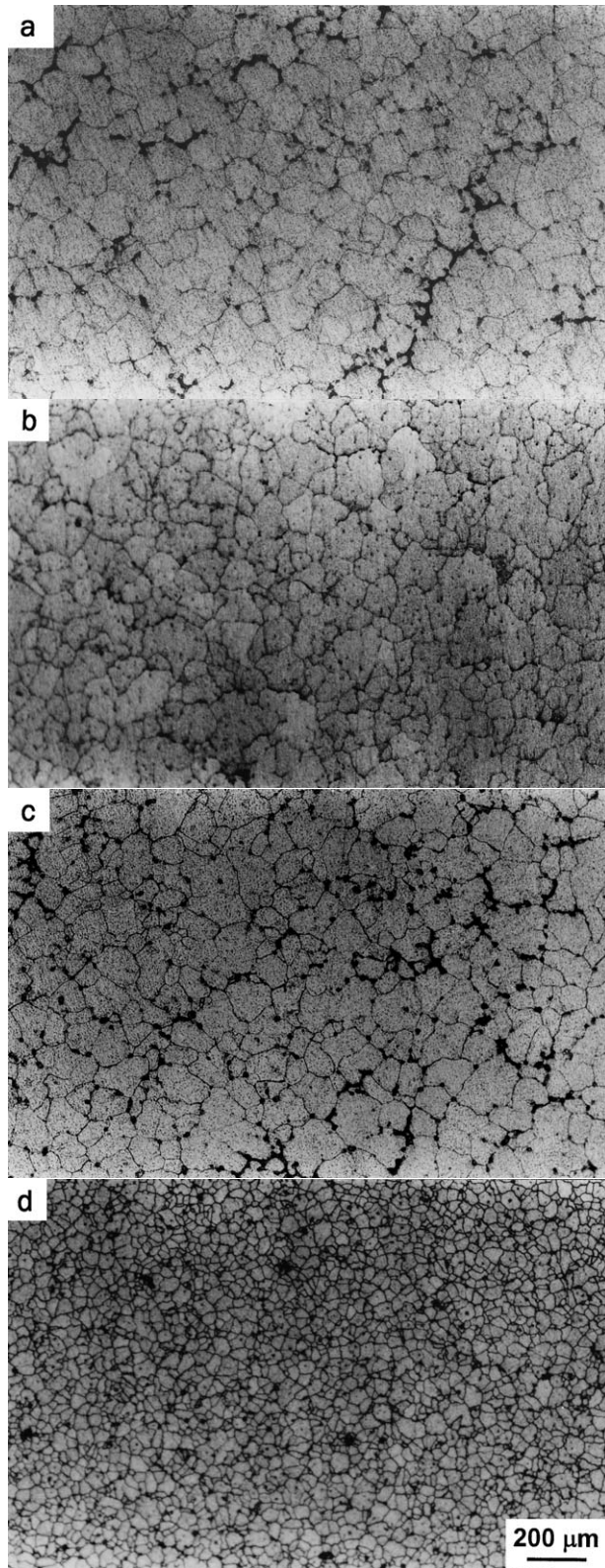
Figure 5 Hardness after direct aging (a) for 24 h at different temperatures and (b) at 300 °C for different times.

Figure 4 Optical microstructures of (a) 1Mg, (b) 3Mg, (c) 4Mg and (d) 8Mg alloys in homogenized condition. Grain sizes for each of the alloys are also mentioned.

Fig. 5b also suggests that there is negligible drop in hardness even after further aging to 24 h. This suggests that the room temperature properties of the 300 °C/10 h peak aged alloy would remain substantially unaffected even after additional exposure for 14 h at 300 °C temperature.

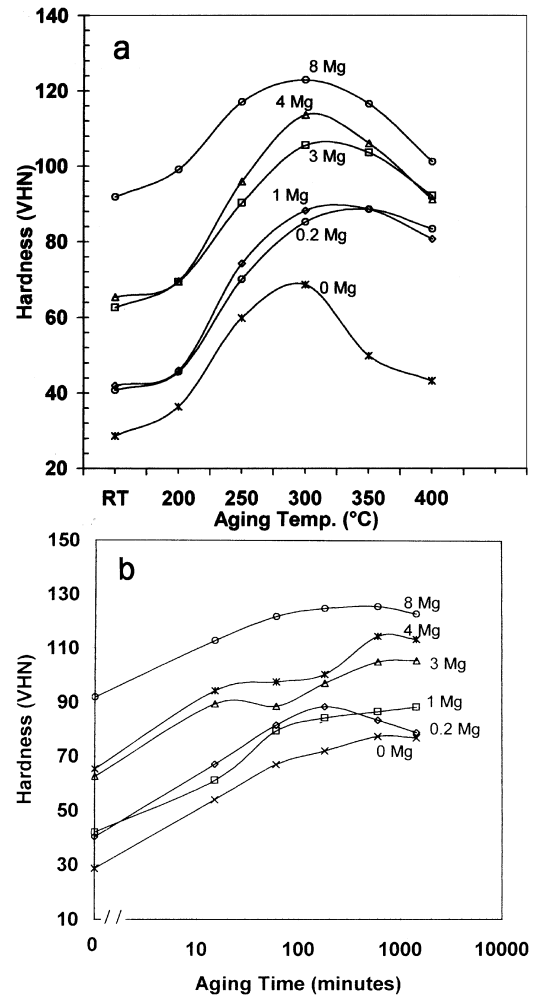There is agreement between the present work and that of Toropova *et al.* [1] that the optimum aging tem-

perature is about 300–350 °C. However, Toropova *et al.* found the hardness of the binary Al-Sc to be higher than that of ternary Al-Sc-Zr in the initial stages of aging, while in the present work, the ternary alloy (containing minor amount of Mg) showed higher hardness throughout the aging cycle. Further studies on early aging, involving detailed TEM observations, are required to understand these differences.

The binary alloy (0Mg) softened after aging beyond 300 °C, while the Zr containing alloys having up to 1%Mg (0.2Mg and 1Mg) did not soften significantly even after aging at 400 °C. The resistance to overaging of the Zr containing alloys could be explained as follows. Since the lattice mismatch between Al and L1$_2$-Al$_3$Zr is lower ($\approx$1%) than that with L1$_2$-Al$_3$Sc ($\approx$1.5%), ternary Al$_3$Sc$_{1-x}$Zr$_x$ precipitates can be expected to have intermediate lattice mismatch (i.e. lower interface energy) and coarsen slower that binary Al$_3$Sc precipitates. The higher Mg alloys, however, showed over aging beyond 300 °C. This could be due to the formation of equilibrium Mg$_5$Al$_8$ type phase known to form and soften Al-Mg alloys [7].

Fig. 6 shows TEM bright field image of one of the alloys (0.2Mg) in the aged condition along with corresponding SAD pattern in the inset. The fine spherical precipitates, exhibiting the typical Ashby-Brown
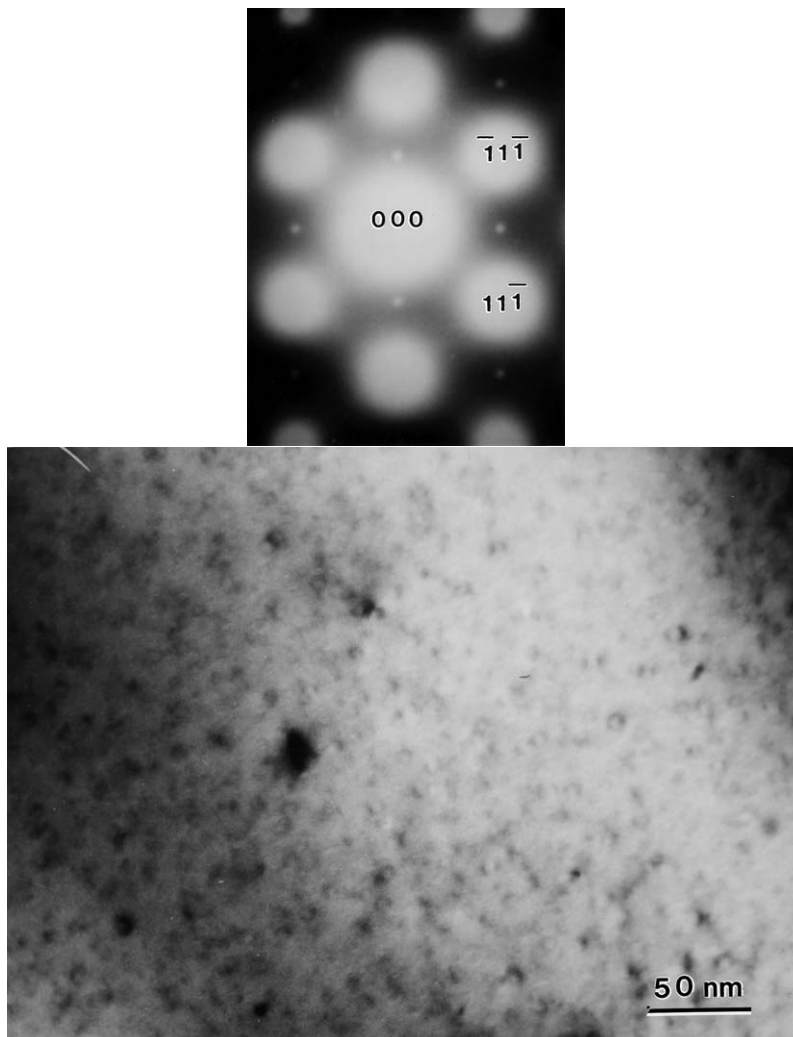
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Figure 6 Bright field TEM image (taken with [011] zone axis) of 0.2Mg alloy after 300 °C/24 h aging. Inset shows SAD pattern with super-lattice reflections due to Al_3Sc coherent precipitates.

coherency contrast, were observed in the aged condition, but not in the as-cast condition for both the alloys. The fact that the fine precipitates were not observed in the as-cast condition indicates that they are either absent or very fine and coherent (<6 nm) meeting the conditions of vanishing contrast for invisibility [8]. The coherent nature of the precipitates is also confirmed by the superlattice spots present in the diffraction pattern shown in Fig. 6 (inset). These fine precipitates are of $Al_3(Sc_{1-x}Zr_x)$ type shown by some investigators earlier [4, 9]. This confirms that the age hardening is solely due to fine $Al_3(Sc_{1-x}Zr_x)$ precipitates and the increased hardness of Mg containing alloys is due to solid solution hardening.

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