

Research Article

Effect of Aluminum Addition on the Microstructure, Tensile Properties, and Fractography of Cast Mg-Based Alloys

M. H. Abdelaziz,¹ M. Paradis,¹ A. M. Samuel,¹ H. W. Doty,² and F. H. Samuel¹

¹Département des Sciences Appliquées, Université du Québec à Chicoutimi, Chicoutimi, QC, Canada ²Materials Engineering, General Motors, 823 Joslyn Avenue, Pontiac, MI 48340, USA

Correspondence should be addressed to F. H. Samuel; fhsamuel@uqac.ca

Received 14 March 2017; Revised 2 May 2017; Accepted 6 June 2017; Published 10 August 2017

Academic Editor: Antonino Squillace

Copyright © 2017 M. H. Abdelaziz et al. This is an open access article distributed under the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

The present study was performed on Mg-based alloys containing Zn and Mn. The alloys were cast in a permanent metallic mold preheated to 200°C and with a protective atmosphere of dry air, CO_2 , and SF_6 . Two main phases are observed in the as-cast condition: Mg-Al-Zn and Mn-Al intermetallics. The size and morphology of the Mg-Al-Zn phase are significantly affected by the concentration of Al. Tensile properties, using standard ASTM B-108 samples, are directly related to the size, morphology, and density of the existing phase particles. The alloy ductility is reduced with increase in the Al concentration, whereas the ultimate tensile strength and the yield strength are more or less stable. The fracture surface of the tested tensile bars is mostly ductile for low Al-containing alloys and tends to be brittle with the increase in Al content as evidenced by an increase in the density of cleavage ruptured areas.

1. Introduction

Commercial Mg-based alloys normally contain 3–13% aluminum, 0.1–0.4% Mn, and 0.5–3% Zn. The AZ series (e.g., AZ63, AZ92, AZ31, and AZ61) is normally used in sand castings and die castings as well as extrusion. The M1 alloy (Mg alloy with 1% Mn) is applied for castings that require low strength or welding [1–5]. The influence of additives in commercially available magnesium alloys (AZ31, AM50) on their microstructure and mechanical properties has been investigated [6–8].

Yamashita et al. [9] studied the mechanical properties of magnesium and its alloys subjected to heavy plastic deformation. Pure Mg and Mg alloys have limited ductility at ambient temperature. Magnesium alloys may be considered for aeronautical applications due their high mechanical properties provided by a fine-grained structure [10]. High strength coupled with high ductility at room temperature is achieved by grain refinement. Furthermore, fine-grained magnesiumbased materials exhibit superplastic behavior at high strain rates ($\geq 10^{-1} \text{ s}^{-1}$) or low temperatures ($\leq 473 \text{ K}$).

The present study was aimed at understanding the effect of increasing Al and Mn in Mg-based alloys on their microstructure and tensile properties. Fracture surfaces of selected tensile bars were examined to arrive at a better understanding of alloy failure mechanism.

2. Experimental Procedure

It should be mentioned here that throughout the text all elements are given in wt%. Table 1 shows the chemical composition of the experimental alloys used in the present study. Pure Mg, Al, and Zn were used as base metals. Manganese was added in the form of Al-25 wt% Mn master alloy. The Mg alloy was held at 720°C to prepare the alloys shown in Table 2. Melting was carried out in an Fe crucible of 25 kg capacity. The crucible was placed in an electrical

Alloy code Al% Mn% Mg% Zn% А 95.56 4.000.44 0.13 В 92.22 7.00 0.78 0.20 С 90.00 9.00 1.00 0.26 D 87.78 11.00 1.22 0.27

14.00

1.56

0.27

TABLE 1: Chemical composition of the Mg-Al-Zn alloys used in the present study (wt%).

TABLE 2: Average grain size of the phases in the alloys used in the present study.

84.44

Alloy code	Grain size (μ m)		
A	105		
В	110		
С	115		
D	130		
E	138		

resistance furnace. The surface of the molten alloys was protected from oxidation using a special gas (a mixture of dry air (78% N_2 and 22% O_2), CO₂, and SF₆).

The liquid alloy was mechanically stirred for 5 minutes at 120 rpm prior to casting in a metallic mold preheated at 200°C (Figures 1(a) and 1(b)). The casting was mainly used for microstructural characterization as well as chemical analysis. Another set of castings was made using a standard ASTM B-108 mold (made of cast iron) to produce tensile bars (Figures 1(d)(i) and 1(d)(ii)). The mold was preheated at 500°C whereas the molten alloy was poured at 720°C. In both cases the solidification rate was more or less the same as inferred from measuring the alloy grain size. Figure 1(c) shows the procedure followed to measure the grain size.

Microstructures were examined by means of a Leica DM LM optical microscope. The grain-size measurements were carried out using a Clemex image analyzer in conjunction with the optical microscope. The grain size was obtained from the average of 200 measurements taken over 20 fields (10 measurements per field) at 100x magnification for each alloy sample. Phase identification was carried out using an electron probe microanalyzer (EPMA) in conjunction with energy dispersive X-ray analysis (EDX) and wavelength dispersive spectroscopic analysis (WDS) where required, integrating a combined JEOL JXA-8900l WD/ED microanalyzer operating at 20 KV and 30 nA, where the size of the spot examined was $\sim 2 \mu$ m.

The tensile test bars were pulled to fracture at room temperature at a strain rate of $4 \times 10^{-4} \text{ s}^{-1}$, using an MTS servohydraulic mechanical testing machine. A strain gauge extensometer (with a 50.8 mm range) was attached to the test bar to measure percentage elongation as the load was applied. The tensile properties of each alloy condition were represented by the average yield stress (YS) at 0.2% offset

strain, ultimate tensile strength (UTS), and fracture elongation (%El) values, which were calculated from five tested tensile bars.

The fracture surface of selected samples was examined using the Hitachi Cold FE SU-823000 SEM. Fracture surface analysis aims at investigating the nature of the fracture for selected samples and identifying the main source of cracking and fracture for these alloys. Samples for SEM examination were prepared from the tensile-tested specimens by sectioning them 1 cm immediately below the fracture surface and mounting them carefully for subsequent fracture surface examination.

3. Results and Discussion

The distribution of the Mg-phase particles in the present alloys is shown in Figure 2. When Al is about 4%, only traces of Mn-Al phase [11, 12] can be observed, in the A alloy (Figure 2(a)). Figures 2(b)-2(d) reveal the gradual increase in the volume fraction of the Mg-Al-Zn phase particles, mainly $Mg_{17}(Al,Zn)_{12}$ [13–15], with the increase in the amount of added Al, Figure 2(e) clearly showing the eutectic morphology of the phase. It is evident from Figure 3 that the morphology of the Mg-Al-Zn phase has been changed from spongy-like into a more or less flat surface when the increase in the Al content exceeds 9 wt%. Figure 4 displays the fraction of the Mg phase as a function of Al concentration. It is evident from Figure 4 that the surface fraction of the Mg-Al-Zn phase increased from about 0.3 to 11 wt% with the increase in the Al concentration from 4 to 14 wt%. Also, the standard deviation is less than 7% indicating the uniform distribution of this phase throughout the matrix regardless of the aluminum content. Figure 5 illustrates the distribution of Al, Zn, and Mn in the C and E alloys. As can be seen, over the aluminum range of 7 to 14 wt%, both Al and Zn are distributed uniformly within the Mg-Al-Zn phase particles. The Mn-Al phase appears in the form of scattered particles away from the Mg-Al-Zn phase particles (see white circled areas). Table 2 shows the grain size has not been significantly affected by the increase in the aluminum content.

The stress-strain curves obtained from the five studied alloys are shown in Figure 6(a). It is evident that the alloy with low aluminum content, that is, 4% (the A alloy), achieved the maximum ductility (approximately 16%) which is very close to pure Mg [16]. With the increase in the concentration of Al and the corresponding increase in the amount of Mg-phase particles, both YS and UTS parameters revealed degradation reaching minimum at 11 wt% Al. In contrast, % elongation increased more or less linearly with the increase in the added amount of Al [17].

Figure 6(b) summarizes the results presented in Figure 6(a). The yield strength of the A alloy is the lowest among all alloys (approximately 120 MPa), whereas those of B–D are almost similar, in the range of 135–138 MPa. The E alloy exhibited somewhat higher YS, about 153 MPa (with a standard deviation of about 1.6 MPa), solid line in Figure 6(b).

Е



(a) Mild steel mold with copper base and casting for microstructure examination

(b) Schematic diagram of the mold used (dimensions are in inches)



(c) Line intercept method used for the measurement of grain size





Figure 1



(e) Optical microstructure of the C alloy showing (a) Al-Mn phase and (b) $Mg_{17}(Al,Zn)_{12}$ phase; note its eutectic morphology

FIGURE 2: Backscattered electron images showing the size and distribution of the Mg-Al-Zn phase in: (a) A alloy, 4 wt% Al; (b) C alloy, 9 wt% Al; (c) D alloy, 11 wt% Al; (d) E alloy, 14 wt% Al. Note: white particles are Mn-Al phase and gray particles are Mg-Al-Zn phase.

The variation in the alloy ultimate tensile strength (UTS) is not, however, proportional to the amount of the added Al. The UTS reaches its maximum level at 7% Al (B alloy) followed by gradual decrease with the increase in the Al percentage up to 11%, broken line #2. The %El revealed a marked decrease with the increase in aluminum content compared to the other two tensile parameters. The decrease in %El exhibited an exponential trend as shown by the broken line #3 in Figure 6(b). The present data are comparable with those obtained by Guangyin et al. [13] for the SJTU-HM1

(a rare earth-containing Mg-5%Al-0.9%Zn-0.71%Si-0.4%Sb-0.2%RE alloy) and AE42 alloys investigated by them and which showed superior ductility to that of the AZ91 alloy tested at room temperature. It should be mentioned here that neither Sb nor rare earth metals were added to the present alloys.

The quality of aluminum alloy castings may be expressed numerically. Drouzy et al. [18] first proposed the following equation:

$$Q = a \text{UTS} + d \log (\%\text{El at failure}), \qquad (1)$$



FIGURE 3: High magnification backscattered electron images of (a) 7% Al, (b) 9% Al, (c) 11% Al, and (d) 14% Al.

(d)

(c)



FIGURE 4: Surface fraction of the Mg-Al-Zn phase in the present alloys based on 20 fields/alloy at 100x.

where Q is the quality index in MPa, UTS refers to the ultimate tensile strength in MPa, %El is the percentage elongation to fracture, and d is a constant of 150 MPa for Al-7%Si-0.4%Mg alloys. Figure 6(c) illustrates the variation in Q-values as a function of the added aluminum revealing a negative relationship between Q and percentage of the added Al up to 11%.

Figure 7 shows the fracture surface of the A alloy with large dimples (marked 1 in Figure 7(a)) caused by coalescence of microporosity due to the alloy high ductility along with secondary cracks (marked 1 in Figure 7(b)). Some scattered particles of Mn-Al phase can also be seen in Figure 7(b), marked 2. These features are comparable with those obtained from pure Mg. With the decrease in the alloy ductility caused

CP50 um	Al50 um	CP Lv 1785 1713 1641 1569 1498 1426 1354 1282 1211 1139 1067 996 924 852 780 709 637 Ave 971	Area (%) 0.2 0.0 0.0 0.0 0.3 1.2 1.9 1.6 1.1 1.1 8.4 60.2 22.9 0.7 0.2 0.1 0.1	Al Lv 918 861 805 748 692 635 579 522 466 409 353 296 240 183 127 70 14 Ave 100	Area (%) 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.
Zn50 um	Mn50 um	Zn Lv 58 54 50 47 43 39 36 32 29 25 21 18 14 10 7 3 0 Ave 6	Area (%) 0.0 0.0 0.0 0.0 0.1 0.2 0.5 1.1 1.1 1.6 1.2 1.8 4.3 11.6 46.6 29.9 0.0	Mn Lv 68 64 60 55 51 47 43 38 34 30 25 21 17 12 8 4 0 Ave 3	Area (%) 0.7 0.0 0.0 0.0 0.1 0.1 0.1 0.1 0.1
	(a)	CP Lv	Area (%)	Al Lv	Area (%)
CP50 um	Al 50 um	1848 1777 1705 1633 1561 1490 1418 1346 1275 1203 1131 1059 988 916 844 772 701 Ave 973	$\begin{array}{c} 0.1\\ 0.0\\ 0.0\\ 0.0\\ 0.0\\ 0.0\\ 0.1\\ 0.8\\ 3.8\\ 4.9\\ 3.2\\ 2.6\\ 3.7\\ 46.5\\ 32.7\\ 0.7\\ 0.2\\ 0.4 \end{array}$	1109 1040 972 903 835 766 698 629 561 492 424 355 287 218 150 81 13 Ave 153	$\begin{array}{c} 0.0\\ 0.0\\ 0.0\\ 0.0\\ 0.0\\ 0.0\\ 0.0\\ 0.0$
		Zn Lv 48 45 42 39 36 33 30 27 24 21 18 15 12	Area (%) 0.0 0.0 0.0 0.1 0.2 0.6 1.4 2.5 3.4 3.8 3.5 3.7 6 9	Mn Lv 39 37 34 32 29 27 24 22 19 17 14 12 9	Area (%) 0.6 0.0 0.0 0.0 0.0 0.1 0.1 0.1 0.1

(b)

FIGURE 5: Continued.



FIGURE 5: X-ray electron images showing elements distribution in (a) B alloy, 7% Al; (b) C alloy, 9 wt% Al; and (c) E alloy, 14 wt% Al. The side bars indicate the intensity of each element. CP = backscattered image.

by the addition of Al, the fracture surface of B alloy shown in Figure 8(a) displayed a mixture of fine dimples, marked 1 corresponding to the precipitation of Mg-Zn-Al phase particles, together with large flat fracture areas corresponding to the Mg matrix, marked 2. Figure 8(b) shows the presence of both types of intermetallics, that is, Mg-Zn-Al (marked 1) and Mn-Al (marked 2).

The fracture surface of C alloy is shown in Figure 9(a) revealing the presence of particle cleavage covering most of the fracture surface. It should be mentioned here that the C alloy (9 wt% Al) exhibits only about 5.4% elongation (Figure 6(a)). Considering such a brittle alloy, it is expected that the crack would propagate very fast across the entire cross-section along the grain boundaries (intergranular fracture) as illustrated in Figure 9(b). The transition from transgranular (ductile alloys) to intergranular type becomes more evident with marked decrease in the alloy ductility as presented in Figure 9–11.

4. Conclusions

Based on the results obtained from this study, the following conclusions could be drawn:

- Alloys containing a low aluminum content, less than 4%, are characterized by a low volume fraction of Mg-Al-Zn phase particles (less than 0.3%). In this case, both Al and Zn are in solid solution in the Mg matrix.
- (2) Precipitation of the Mg-Al-Zn phase in the form of eutectic particles heterogeneously distributed in the Mg matrix is observed when the Al content is about 7%.
- (3) At higher Al content (9%), the Mg-Al-Zn phase particles are precipitated homogeneously throughout the matrix maintaining the same morphology as in Figure 2.
- (4) When the aluminum content exceeds 11%, the Mg-Al-Zn phase precipitates in the form of massive particles with smooth surfaces and high density.
- (5) Addition of Al and Zn to Mg alloys leads to a marked decrease in the alloy ductility.
- (6) The mechanical property plots show that 9 wt.% Al alloys have a good balance of strength and ductility corresponding to the widely used AZ91D alloy.
- (7) The fracture mechanism of the present alloys was found to be directly related to the amount of added





(a) Typical stress-strain curves obtained from the alloys used in the present study as a function of percentage of Al





(c) Variation in Q-values as a function of %Al; see Table 1

Figure 6



FIGURE 7: (a) Secondary electron image of the A alloy, 4 wt% Al; (b) backscattered electron image of (a).



FIGURE 8: (a) Secondary electron image of the B alloy, 7% Al; (b) backscattered electron image of (a).



FIGURE 9: (a) Secondary electron image of the C alloy, 9 wt% Al; (b) backscattered electron image of (a).



FIGURE 10: (a) Secondary electron image of the D alloy, 11 wt% Al; (b) backscattered electron image of (a).



FIGURE 11: (a) Secondary electron image of the E alloy, 14 wt% Al; (b) backscattered electron image of (a).

aluminum. The transition from ductile fracture to a brittle one takes place when the Al concentration exceeds 9%.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

The authors would like to thank Dr. Carlton Fuerst, Chief Scientist, General Motors Canada, for suggesting the present research topic. Thanks are also due to Amal Samuel for enhancing the images used in this article.

References

- J. T. Black and R. A. Kohser, DeGarmo's Materials and Processes in Manufacturing, 11th edition, 2012.
- [2] A. Lindemann, J. Schmidt, M. Todte, and T. Zeuner, "Thermal analytical investigations of the magnesium alloys AM60 and AZ91 including the melting range," *Thermochimica Acta*, vol. 382, no. 1-2, pp. 269–275, 2002.
- [3] S. Sankaranarayanan, S. Jayalakshmi, and M. Gupta, "Effect of individual and combined addition of micro/nano-sized metallic elements on the microstructure and mechanical properties of pure Mg," *Materials and Design*, vol. 37, pp. 274–284, 2012.
- [4] D. H. StJohn, M. Qian, M. A. Easton, P. Cao, and Z. Hildebrand, "Grain refinement of magnesium alloys," *Metallurgical and Materials Transactions A*, vol. 36, no. 7, pp. 1669–1679, 2005.
- [5] D. H. StJohn, A. K. Dahle, T. Abbott, M. D. Nave, and D. Qian, in *Magnesium Technology 2003, San Diego, CA*, H. I. Kaplan, Ed., pp. 95–100, TMS, Warrendale, PA, USA, 2003.
- [6] K. Neh, M. Ullmann, and R. Kawalla, "Effect of grain refining additives on microstructure and mechanical properties of the commercial available magnesium alloys AZ31 and AM50," in *Proceedings of the Materials Today*, vol. 2, 1, pp. S219–S224, 2015.
- [7] J. Hirsch and T. Al-Samman, "Superior light metals by texture engineering: optimized aluminum and magnesium alloys for automotive applications," *Acta Materialia*, vol. 61, no. 3, pp. 818– 843, 2013.
- [8] E. Essadiqi, M. T. Shehata, A. Javaid et al., Alloying and Process Design of Mg Sheet, CANMET Materials, Ottawa, Canada, 2011.
- [9] A. Yamashita, Z. Horita, and T. G. Langdon, "Improving the mechanical properties of magnesium and a magnesium alloy through severe plastic deformation," *Materials Science and Engineering: A*, vol. 300, no. 1-2, pp. 142–147, 2001.
- [10] K. Kubota, M. Mabuchi, and K. Higashi, "Processing and mechanical properties of fine-grained magnesium alloys," *Journal of Materials Science*, vol. 34, no. 10, pp. 2255–2262, 1999.
- [11] P. Cao, M. Qian, and D. H. Stjohn, "Effect of manganese on grain refinement of Mg-Al based alloys," *Scripta Materialia*, vol. 54, no. 11, pp. 1853–1858, 2006.
- [12] Y. Liu, G. Huang, Y. Sun et al., "Effect of Mn and Fe on the formation of Fe- and Mn-rich intermetallics in Al-5Mg-Mn alloys solidified under near-rapid cooling," *Materials*, vol. 9, no. 2, article no. 88, 2016.
- [13] Y. Guangyin, L. Manping, D. Wenjiang, and A. Inoue, "Mechanical properties and microstructure of Mg-Al-Zn-Si-base alloy," *Materials Transactions*, vol. 44, no. 4, pp. 458–462, 2003.

- [14] A. Bag and W. Zhou, "Tensile and fatigue behavior of AZ91D magnesium alloy," *Journal of Materials Science Letters*, vol. 20, no. 5, pp. 457–459, 2001.
- [15] A. Shukla and A. D. Pelton, "Thermodynamic assessment of the Al-Mn and Mg-Al-Mn systems," *Journal of Phase Equilibria and Diffusion*, vol. 30, no. 1, pp. 28–39, 2009.
- [16] H.-F. Sun, C.-J. Li, Y. Xie, and W.-B. Fang, "Microstructures and mechanical properties of pure magnesium bars by high ratio extrusion and its subsequent annealing treatment," *Transactions* of Nonferrous Metals Society of China (English Edition), vol. 22, no. 2, pp. s445–s449, 2012.
- [17] D.-H. Li, M.-L. Hu, H.-B. Wang, and W.-A. Zhao, "Low temperature mechanical property of AZ91D magnesium alloy fabricated by solid recycling process from recycled scraps," *Transactions of Nonferrous Metals Society of China (English Edition)*, vol. 21, no. 6, pp. 1234–1240, 2011.
- [18] M. Drouzy, S. Jacob, and M. Richard, "Interpretation of tensile results by means of quality index and probable yield strength," *AFS International Cast Metals Journal*, vol. 5, no. 2, pp. 43–50, 1980.







International Journal of Polymer Science



Smart Materials Research





BioMed Research International





Submit your manuscripts at https://www.hindawi.com

Nanomaterials







Materials Science and Engineering

Nanoscience







Journal of Crystallography



The Scientific World Journal



Journal of Ceramics



Journal of Textiles