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EFFECTS OF ALUMINIUM CONTENT ON STRAIN RATE SENSITIVITY OF AZ SERIES WROUGHT MAGNESIUM ALLOYS DURING SUPERPLASTIC FORMING

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ABSTRACT

During studying homogenised and hot rolled magnesium alloys with two different aluminium contents (3 and 6%) at 350 and 400 ^{o}C at a strain rate of $5x10^{-4}$ s^{-1}, a subtle difference was found in the strain hardening region during tensile testing. This occurs due to the solid solution strengthening effect of aluminium. However, an estimation of the apparent activation energy of deformation shows that both alloys are deformed by a single deformation mechanism. Therefore, the mode of deformation is not altered by the addition of aluminium. On the other hand, addition of aluminium is found to promote grain growth in the alloys. This certainly adversely affects *m* values which is obvious from the calculated sensitivity values. The accelerated grain growth observed is likely to be a result of the increased flow stress by the addition of aluminium. It is suggested that more segregation of solute atoms away from the mobile dislocations, together with rapid grain growth for higher aluminium, reduces *m* values.

Keywords: Magnesium Alloys, Superplasticity, Strain Rate Sensitivity.

1. INTRODUCTION

Magnesium is the lightest structural metal and magnesium alloys are therefore obvious candidates in weight critical applications. The environmental imperative to reduce vehicle emissions has recently led to intensified research interest in magnesium, since weight reduction is one of the most effective ways of improving fuel efficiency [1]. The hexagonal close-packed structure of magnesium results in poor room temperature formability. However, on heating, several magnesium alloys show superplastic properties, with the ability to deform to very high strains (up to 3000%) [2]. This opens up the possibility of forming complex components directly by superplastic forming (SPF). As a result, SPF of magnesium is a highly active research topic.

Large elongation, typical of superplastic deformation, is associated with a high strain rate sensitivity (m). m is defined as the variation in flow stress by strain rate as

$$m = \frac{\delta ln\sigma}{\delta ln\,\dot{\varepsilon}}$$

where σ is the flow stress and $\dot{\varepsilon}$ is the strain rate. Higher m gives a higher degree of resistance toward flow localisation. Localised deformation (necking) starts at maximum load, since strain hardening may increase the load-bearing capacity during deformation. At maximum load, the effect of stress increasing by the reduction of specimen cross-sectional area overcomes the load-bearing capacity by strain hardening. It is noteworthy that in sheet materials, where the thickness reduction is lower than elongation, a diffuse neck is produced. This type of neck may lead to fracture or transform into another instability process known as localised necking. The effect of m is more pronounced in retarding neck development. A higher m provides more resistance to neck growth. A higher m means that as the local strain rate increases in a forming neck, the flow stress increases rapidly. This increment of local strain rate requires a higher local stress to propagate the neck. Therefore, the growth of the neck is retarded as the applied stress is insufficient to continue its growth.

The effect of solute aluminium in magnesium alloys on strain rate sensitivity values is not clear yet. In this work, the effect of addition of solute aluminium in aluminium-zinc (AZ) series wrought magnesium alloys is studied to understand any variation of strain rate sensitivity—important flow property of deformation.

2. EXPERIMENTAL

Two alloys from Magnesium AZ, AZ31 (Al 2.8, Zn 0.92, Mn 0.369, Fe 0.0073, Ni 0.001 wt%) and AZ61 (Al 5.88, Zn 0.93, Mn 0.26, Fe 0.004, Ni 0.001 wt%), used in this work was provided by Magnesium Elektron, UK in chill-cast plate form. The major difference between these two alloys was aluminium content. The alloys were homogenised at 420 ^{o}C for 24 h in an argon gas atmosphere prior to hot-rolling at 300 ^{o}C to obtain sheet of thickness 2 mm from 40 mm plate thickness by 22 roll passes. The compressive strain developed during rolling

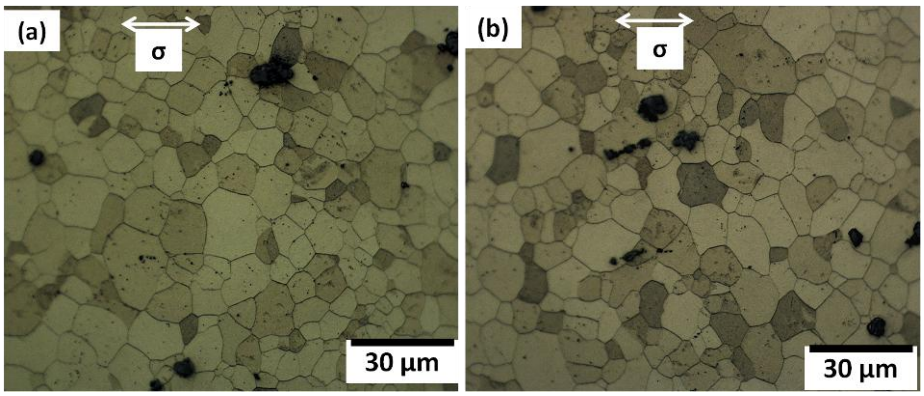

Fig 1. Optical micrographs of rolled (a) AZ31 and (b) AZ61

was 0.12 in each roll pass. Rolling was accomplished uni-directionally for first ten passes and was cross-rolled for the remaining passes. To keep the work-piece sufficiently warm, it was kept in furnace for 5-10 minutes before passing through the cold rolls.

Samples were prepared from rolled specimen using standard metallographic techniques and grain sizes were determined in linear intercept method using ImageJ [3]. To reveal grain boundaries, picral solution was used as etchant. Unetched specimens were observed under Phillips XL30 field emission gun scanning electron microscope and energy-dispersive X-ray spectroscopy (EDX) was used to obtain composition of the second-phase particles present in the microstructure.

Tensile specimens were prepared from the rolled sheets, with a gauge length of 12 mm and width of 6.3 mm with simple square tag ends (no blend radius at the end of the gauge). Tensile axis of the specimens was kept parallel to the final roll pass direction and tensile tests were conducted using a custom-built tensile machine (made by Alcan International Ltd), incorporated with an electrical resistant-heating furnace. Tensile tests were carried out with a base strain rate of 5 X 10^{-4} s^{-1} at 350 and 400 ^{o}C. The strain rate was varied ±10% of the nominal strain rate applied for each 0.1 strain (ε). Such variation allows determination of strain rate sensitivity (m) values at different true strains from stress-strain plots [4].

Grain sizes at the gauge and grip regions of the specimens were estimated after tensile tests to check growth of grains.

3. RESULTS and DISCUSSION

3.1 Hot Deformation Properties

Optical micrographs of the rolled AZ31 and AZ61 are shown in figure 1. Recrystallization took place during hot rolling, resulting in equiaxed microstructures of average grain sizes 8.04±0.44 and 8.92±0.65 µm respectively. Apparently, addition of aluminium does not have any prominent effect on recrystallization during hot rolling. In the optical images, there is clear evidence of presence of second phase particles mostly along the grain boundaries. The most likely reason for particles along the boundaries is the pinning of grains during recrystallization to impede grain growth. Composition of these particles were analysed using EDX in FEGSEM and at least 30 particles were analysed. To eliminate any effect from matrix magnesium, extrapolation method was used to determine composition of the particles [5]. Experimentally, particles found to contain approximately 71 at% Al and 28 at% Mn. This stoichiometry was close to predicted dominant and stable phase—$Al_{11}Mn_4$, estimated using JMatPro thermodynamic software and MgData database at the homogenisation temperature. The modes of the particle size data sets are 5 and 4 µm for AZ31 and AZ61 respectively. It appears that both alloys contain particles of similar characteristics. The volume fractions of the particles were approximately 0.50% in both cases, indicating more aluminium was dissolved in solution in the higher aluminium containing alloy—AZ61.

In figure 2, the true stress and true strain plots for the alloys at 350 and 400 ^{o}C are shown. The steps in the plots correspond to the imposed strain rate jumps. An increase in temperature reduces flow stress and hence, typically an augmentation of failure strain is observed. However, in the current work, increase of temperature reduces maximum elongations of the specimens (in AZ61). This occurs due to promoted diffusion of cavities at higher temperature. The effect of adding more aluminium is mostly limited up to the strain hardening region. Due to the solid solution strengthening, the strain hardening region is shortened by adding more aluminium. This means that the peak stress was reached comparatively earlier than the low aluminium alloys.

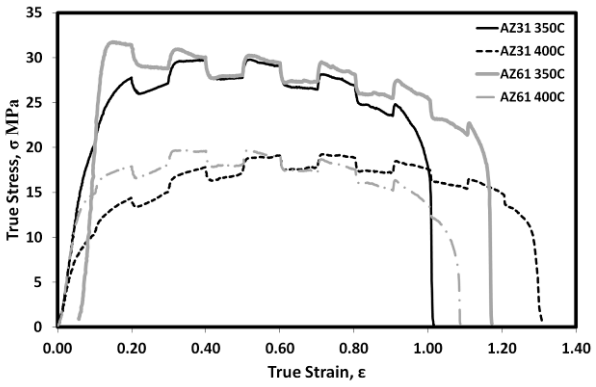

Fig 2. True stress-strain (σ-ε) plots for AZ31 and AZ61 at 350 and 400 ^{o}C at a strain rate of 5 X10^{-4} s^{-1}

The activation energy for deformation provides information about the underlying rate controlling mechanism. The activation energy (Q) of deformation can be calculated from the flow stress dependency at elevated temperature as [6]

$$\dot{\varepsilon} = A_1 \sigma^n \exp\left(-\frac{Q}{RT}\right)$$

After rearranging and taking ln in both sides,

$$\ln\sigma = \frac{mQ}{RT} + \ln A_2 + m\dot{\varepsilon}$$

where $\dot{\varepsilon}$ is the strain rate, R is the molar gas constant, $A_2 = 1/A_1^m$ are constants and n=1/m. The peak flow stresses of the alloys at different temperatures were used to plot lnσ against 1/RT to obtain the slope mQ. From figure 3, the slope obtained is 21 kJ mol^{-1}. Using this value, the apparent activation energies can be determined from the average strain rate sensitivity (m) values of the alloys at different temperatures. 75.19±14.66 and 83.99±10:96 kJ mol^{-1} are the average activation energies of the alloys (averaged from data for all temperatures) for both alloys respectively. The activation energy of lattice diffusion of pure magnesium is 135 kJ mol^{-1} and that of grain boundary diffusion is 92 kJ mol^{-1} [6]. This indicates the deformation mode of these alloys is likely to be dominated by grain boundary diffusion at all test temperature conditions.

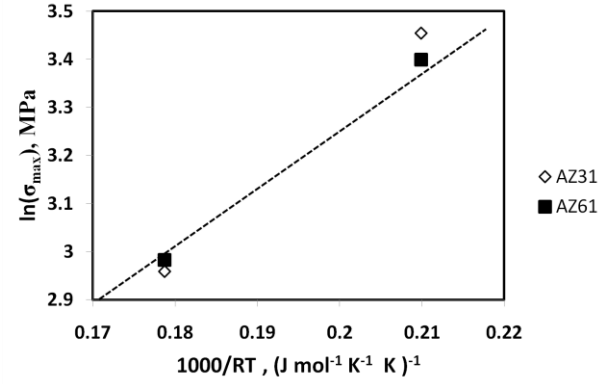

Fig 3. A plot of the logarithmic maximum flow stress, σ_{max}, as a function of the reciprocal of the absolute temperature (T) of the alloys deformed at the strain rate of 5 X 10^{-4} s^{-1}. The slope of the curve, mQ, is 21 kJ mol^{-1}. 1/T was normalised by 1000=R before plotting.

From the flow curves, the strain rate sensitivity (m) values of the alloys were determined. To find the relationship between composition and m values, analysis of variance (ANOVA) was performed on the m data. The consequence of the addition of aluminium on m is shown in Table 1. At 350 ^{o}C, addition of aluminium is not significant, in terms of m, considering the associated error bars. At 400 ^{o}C, the addition of aluminium has reduced m slightly. Interactions that involve the combined effect of aluminium and temperature may also be critical but are not easily identified from these plots. To get rid of this ambiguity, ANOVA was performed.

Table 1: Strain rate sensitivity (m) values of the alloys at different temperatures at the strain rate of of 5 X 10^{-4} s^{-1}.

Alloy	m value	
	350 ^{o}C	**400 ^{o}C**
AZ31	0.34 ± 0.04	0.40 ± 0.03
AZ61	0.32 ± 0.03	0.33 ± 0.02

To perform ANOVA, the half-effects (Δ/2) of the response (m) were first calculated and corresponding Pareto charts are shown in figure 4. The half-effects of two variables were considered, indicated as A (aluminium content (3 and 6 wt%)) and B (temperature (350 and 400 ^{o}C)). Aluminium (A) appears to be the most influential variable and it has a negative effect on m followed by temperature (B) with a positive effect. Moreover, combined response of aluminium and temperature (AB) is also negative.

As already discussed, addition of solutes increases strain hardening rate and this may reduce m due to the effect of solute drag [7]. Recently, it was claimed that in magnesium alloys a reduction in m depended on the mobility of solute atoms [8]. Since activation energy for diffusion of solute aluminium into magnesium is 143 kJ mol^{-1} [6], any diffusion of aluminium is unlikely to be rate controlling as the estimated Q was close to 92 kJ mol^{-1}. Moreover, solute structures, such as solute atmospheres and segregated solutes not attached to dislocations, have an adverse effect on m [9]. Therefore, it is probable that with an increase of aluminium content, more segregation of solutes occurs away from the mobile dislocations. This is believed to be the reason for reduction of m values at higher aluminium content. The combined effect from AB is significant since both single variables act in an opposite way on m-value but the observation that the combined effect is negative suggests that for the range of conditions used in this work, an increased temperature cannot overcome the effect of added aluminium solutes.

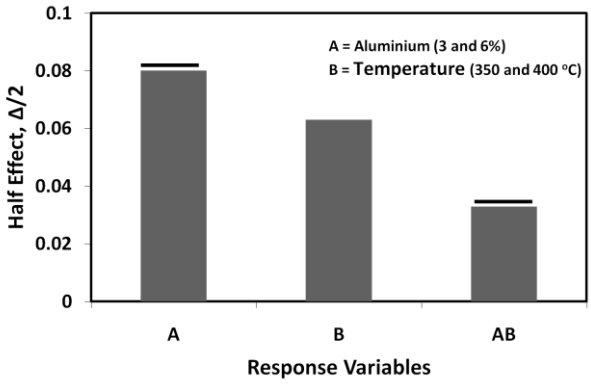

Fig 4. Pareto charts of the calculated half effects of the variables aluminium (A) and temperature (B) on the responses of strain rate sensitivity (m) values. The horizontal lines on top of the bars represent a negative effect of that variable.

3.2 Grain Growth of the Alloys

During hot deformation, significant grain growth sometimes occurred in the alloys, largely depending on

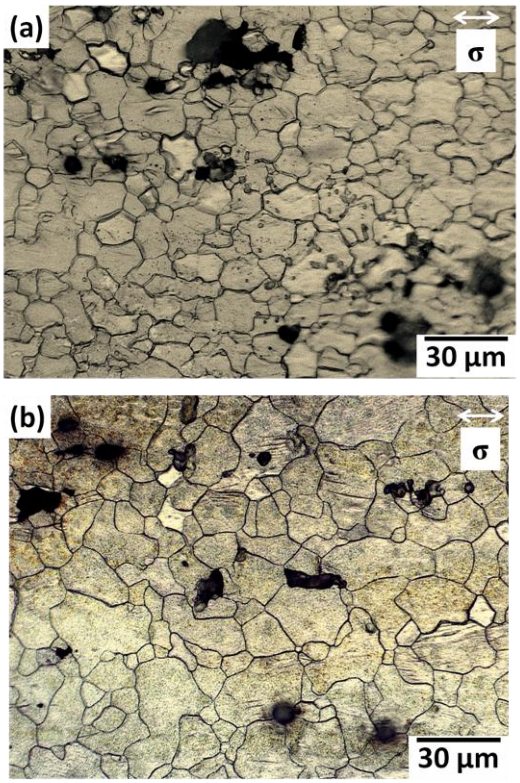

Fig 5. The growth of the grains in the gauge region of the failed specimens of (a) AZ31 and (b) AZ61 after testing at 350 ^{o}C at a strain rate of 5 X10^{-4} s^{-1}.

the test temperature. Fig. 5 shows the micrographs of the gauge regions of the alloys deformed at 350 ^{o}C at a strain rate of 5X10^{-4} s^{-1}. Substantial growth of grains is apparent in all alloys (note that the rolled grain size is 7 to 8 µm). Some cavities are also evident in all microstructures. Table 2 shows the average grain sizes of the alloys in the grip (d_{gr}) and gauge (d_g) regions at different temperatures. The grain growth in the grips is without any straining effect and therefore this reflects the static grain growth of the alloys at different temperatures. At the slow strain rate condition, the time inside the furnace chamber varied from 25 to 43 minutes, depending on the strain following the preheating of the specimens for 20 minutes. Depending on the annealing time, therefore, the size of the grains varied in the grip region. However, in the high aluminium alloys, more rapid grain growth is observed.

In the gauge region, faster growth of grains, compared to the grip region, is evident. The as-rolled average grain sizes of the alloys are 7 to 8 µm, whereas during straining, grains have increased in average size by approximately 2 to 3 times. From Table 2, it can be seen that grain growth in the gauge length region also appears more pronounced for the high aluminium content alloys. Moreover, a substantial growth of grains due to straining is observed in all alloys at 400 ^{o}C. The dynamic grain growth (DGG) rate therefore appears to be controlled mainly by temperature. Otherwise there is no reason that DGG has less influence at 350 ^{o}C than 400 ^{o}C, since the difference between failure strains at 350 and 400 ^{o}C is subtle. Therefore, two trends are identified: grain growth is larger in the higher aluminium content alloys and grain growth rate increases with temperature.

One interesting feature in the microstructures, at different strains and of the failed specimens, is that there is no evidence of grain refining for any alloys. In addition, the stress strain curves do not show a very long steady state during deformation, typical of recrystallization. Dynamic recrystallization does not therefore appear to occur in any of the alloys under the conditions studied.

Table 2: Grain size at grip and gauge regions of the tensile tested specimens

Alloy	Temperature, ^{o}C	Grain Size, µm	
		Grip (d_{gr})	Gauge (d_g)
AZ31	350	10.01±0.98	11.45±0.90
	400	12.79±0.74	19.20±2.13
AZ61	350	15.96±1.39	16.11±1.43
	400	17.95±1.72	20.33±1.77

To check if any elongation of the grains occurs, aspect ratios were measured for the AZ61 alloy deformed at 400 ^{o}C. Grain sizes were measured along the tensile direction and normal to the tensile direction separately in both grip (d_{gr}) and gauge (d_g) regions. The aspect ratio was 1.08±0.11 at the grip (non-deformed part) and 1.15±0.03 at the gauge (deformed part) regions. Considering the associated errors, there is no notable elongation of the grains.

The addition of more aluminium increases the growth of grains in the gauge region. This is the opposite effect to that usually expected for solute addition, when adding solute reduces grain growth rate by increasing drag opposing boundary migration [10]. However, in the present work, it is likely that all the alloys contained sufficient aluminium to saturate the solute drag effect. The addition of extra aluminium (i.e., in AZ61L) does not provide any extra relaxation, but accelerates grain growth in the gauge region probably as a result of the increased flow stress with extra aluminium.

Strain rate sensitivity (m) depends on strain rate, temperature, concurrent grain growth and strain hardening and softening of flow stress [11]. At a fixed temperature and strain rate condition, grain size becomes the dominating variable. Increasing temperature typically increases m. From literature, it is confirmed that a decrease in initial grain size increases m due to enhanced sliding of grains [12, 13]. However, since the initial microstructures are similar in the current study, the effect of grain coarsening appears to adversely affect m. In an Al-5.76Mg aluminium alloy, m was increased with increasing temperature at a particular strain rate but above a certain temperature, m started to decrease due to a pronounced coarsening of grains [14]. For the alloys in the current study, the observed behaviour is similar, with the critical temperature above which m starts to decrease is 400 ^{o}C.

To understand the grain size effect during testing, specimens were deformed to different pre-set strains and grain sizes were measured and compared with the instantaneous strain rate sensitivity, m*. In figure 6, m* values at different strains are shown for two temperatures

(350 and 400 °C). The m* values at different strains were averaged from the repeat test results and the corresponding error bars are also shown. At 400 °C, m* is slightly higher than that at 350 °C for the strain range shown. A trend is common at both temperatures for all alloys—m* decreases during deformation. The observed grain growth can explain the observed reduction in m* with strain.

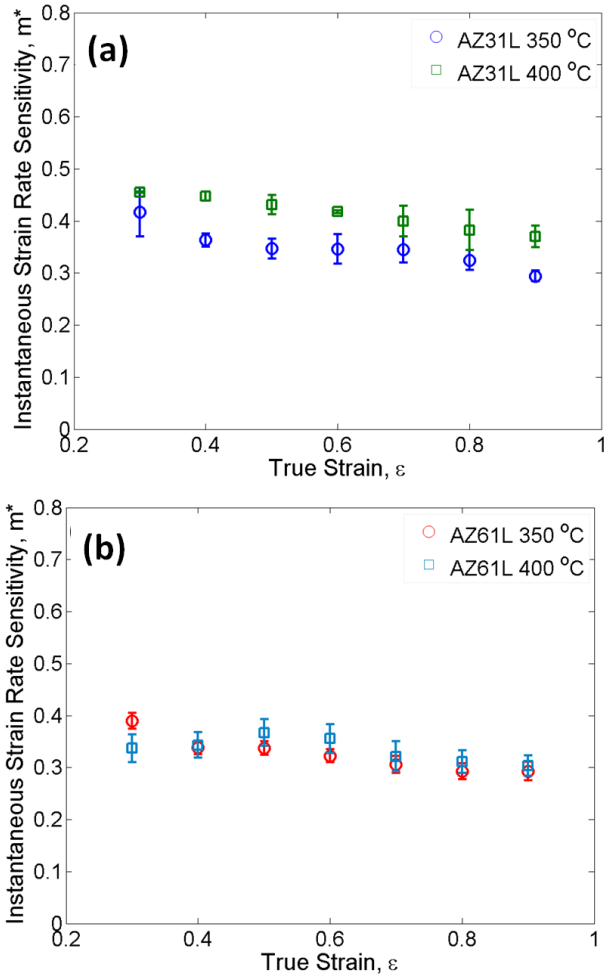

Fig 6. The instantaneous strain rate sensitivity (m*) values are plotted as a function of strain for (a) AZ31 and (b) AZ61 for two different temperatures (350 and 400 °C). The deformation strain rate was 5 X 10^{-4} s^{-1}.

4. CONCLUSION

(l) Hot rolling of the as-cast alloys refined the microstructure and a homogeneous grain structure (<10 µm) was obtained for all alloys. All alloys contained Al-Mn particles of approximately similar amounts.

(2) Flow stress of the alloys decreased with increasing temperature. Addition of solute aluminium showed a prolonged strain hardening to higher strain levels but this was a small effect. A marked difference was found in the strain softening regions attributed to cavitation.

(3) The activation energy for deformation was close to that for grain boundary diffusion in all alloys and a single mechanism of deformation was identified.

(4) Strain rate sensitivity, m, was reduced slightly during testing due to the growth of grains. Aluminium was identified as influencing m by the analysis of variance which is likely to be an effect of solute segregation.

(5) Grain growth was observed the extent of which was dependant on temperature. However, additional aluminium was found to accelerate growth kinetics in the gauge region probably due to slightly increase in flow stress with more aluminium.

5. ACKNOWLEDGEMENT

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