

Effect of Zn Concentration and Grain Size on Prismatic Slip in Mg-Zn Binary Alloys

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Keywords: magnesium, slip, twinning, deformation, grain size, Hall-Petch

Abstract

Mg-Zn binary alloys with concentrations between 0 and 2.8wt% Zn have been prepared and processed via hot rolling and annealing to produce specimens with a strong basal texture and a range of grain sizes. These have been deformed in tension, a condition in which the deformation is dominated by prismatic slip. This data has been used to assess the Hall-Petch parameter as a function of Zn concentration for deformation dominated by prismatic slip. Pure magnesium showed non-linear Hall-Petch behaviour at large grain sizes, and this is compared to the values for prismatic slip measured on single crystals. The differences between critical resolved shear stress measurements made through single crystal, polycrystal and mathematical modelling techniques are also discussed.

Introduction

Due to the close packed hexagonal crystal structure of magnesium, alloys of this material deform by five principle deformation modes: basal $\langle a \rangle$ slip, prismatic $\langle a \rangle$ slip, second order $\langle c+a \rangle$ pyramidal slip, $\{10\bar{1}2\}$ twinning and $\{10\bar{1}1\}$ twinning [1,2]. The critical resolved shear stress (CRSS) values for these deformation modes have been measured in single crystals [3-9], and their values in polycrystals have been estimated by mathematical models such as the VPSC approach eg. [10]. It is noted in a number of publications that these two sets of data do not correlate well [11,12]. The ratio of the CRSS values for basal to prismatic slip, measured in single crystal studies, is in the region of 1:85, while VPSC modelling typically find values around 1:6 for polycrystal alloys [11]. This discrepancy could be either a result of differences in the solute level, or a result of the difference in grain size between the experimentally examined single crystals and the mathematically modeled polycrystals.

It is known that magnesium alloys typically increase in strength with a decrease in grain size, consistent with the Hall-Petch effect, see Fig. 1. However, due to the fact that a number of deformation modes operate in this system, it has not yet been quantified what effect grain size has on the individual deformation modes. In this short paper we restrict our discussion to prismatic slip, but this communication is part of a much larger body of work that also examines basal slip and twinning. The aim of this research is to develop a quantitative measure of the effect of grain size on the stress to activate the different deformation modes, in the case discussed here, prismatic slip. Our measurements are based on the notion that when strongly textured magnesium alloys are deformed in tension, the prismatic slip system is the dominant deformation mode. We therefore deliberately induce strong textures and then deform in tension to activate prismatic slip. These measurements are normalized for texture in order to

estimate as closely as possible the stress required to activate the prismatic slip system as a function of grain size.

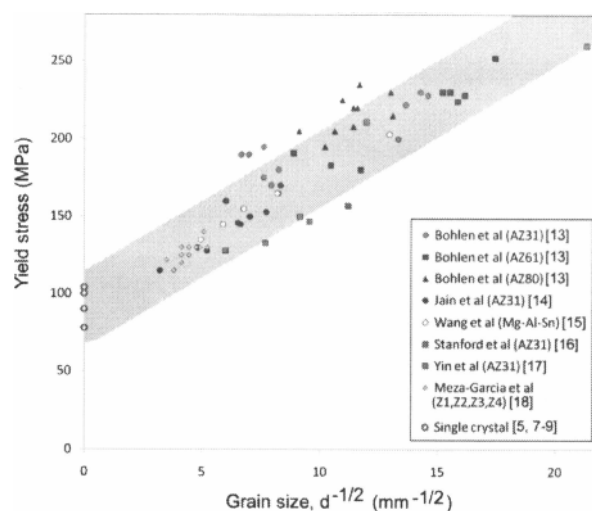


Figure 1 – Hall-Petch plot for a range of alloys from literature. All alloys have been processed by either rolling or extrusion, and are therefore expected to have strong textures. All alloys have been deformed in tension. Single crystal data from crystals orientated for prismatic slip

Experimental Methodology

This work is based on five alloy compositions (in wt%): Pure Mg, Mg-0.2Zn, Mg-0.5Zn, Mg-1.1Zn, and Mg-2.8Zn. The alloys were produced in 400 g castings that were chill cast into 25 mm thick plate. The alloys were solution treated for 4 hours at 450°C in a flowing argon atmosphere. The alloys were then sectioned and hot rolled at 400°C to 1.25 mm thickness. Tensile specimens with a gage length of 25 mm and width of 3 mm were cut from the rolled plate and then annealed to produce three different grain sizes for each alloy composition. Tensile testing was carried out on an Instron tensile tester equipped with a non-contact video extensometer and a 5 kN load cell. The tests were carried out at an initial strain rate of 10^{-3} s^{-1} . All specimens were deformed to failure, and were tested in the rolling direction.

Some additional experiments were carried out only on pure magnesium with a larger grain size range. For these experiments the magnesium plate was hot rolled from 10 mm to a thickness of 5 mm and then recrystallisation annealed at various times and

temperatures to obtain the desired grain sizes. These larger thickness specimens were used for the largest grain sizes because it has been shown that a minimum number of grains, about 15, are required across the gauge thickness to ensure that the yield point is indicative of bulk material behaviour [19,20]. The texture of all specimens was measured using x-ray diffraction with CuK α radiation. Only the basal pole figures were measured.

Results

Typical examples of the stress strain curves obtained in tension are shown in Fig. 2. There was an increase in yield point with a decrease in grain size, and this was more marked in the higher concentration alloys. For the smaller grain sizes there was a well defined yield point in the more concentrated alloys, Fig. 2b.

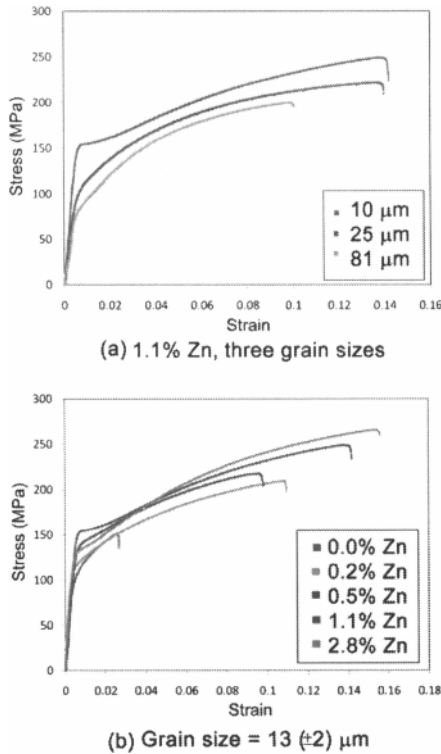


Figure 2 – Tensile curves for selected specimens

The ductility of the pure magnesium specimens was quite low, less than 0.05 in all cases, Fig. 3a. However the ductility was significantly improved at higher Zn concentrations, Figs. 3a and 3b. The work hardening rate was not significantly affected by the Zn concentration, with all the work hardening rate data fitting to a straight line when plotted against $d^{-1/2}$, Fig. 4.

For the pure magnesium, a larger range of grain sizes were tested in order to validate the Hall-Petch behaviour, Fig. 5. Also included on this plot is the stress to activate prismatic slip measured on single crystals [5, 7-9] assuming a Taylor factor of 2. The band of data from Fig. 1 is also included for reference. It is apparent from this plot that there is a plateau in the yield stress for larger grain sizes, and that this plateau corresponds well with single crystal data.

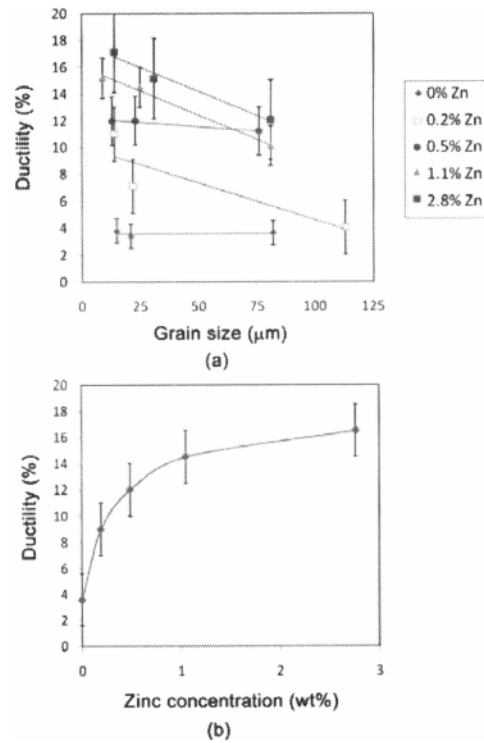


Figure 3 – (a) Measured ductility for all alloys and grain sizes tested. (b) Effect of Zn concentration on the tensile ductility for a grain size of 20 μm .

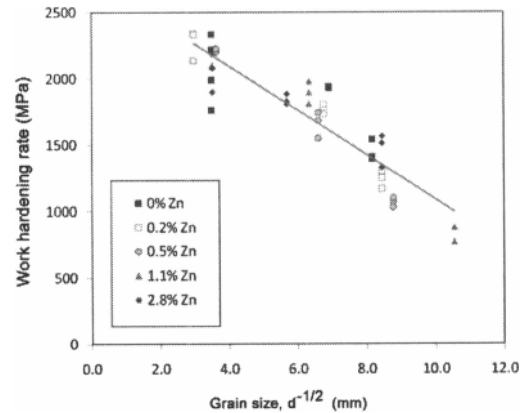


Figure 4 – Work hardening rate at a strain of 0.02 for all alloys and grain sizes tested.

A number of different fits could be applied to the data shown in Fig. 5. For example, this same data set plots well against the inverse of the grain size, d^{-1} . Another option would be a linear fit that crosses the y-axis at ~ 70 MPa. However, taking into consideration that the single crystal data are a minima estimate, we conclude here that the best fit to the available data is a plateau

at grain sizes above $\sim 30 \mu\text{m}$. We also draw weight for our argument to plot against $d^{-1/2}$ from the data shown in Fig. 1 where it is clear that the Hall-Petch relationship holds over a range of grain sizes and alloys.

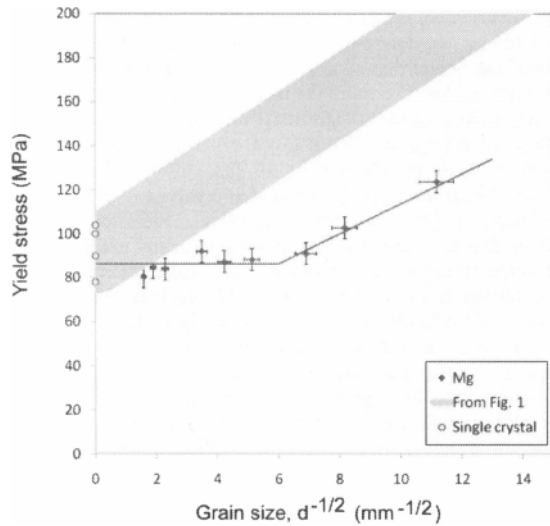


Figure 5 – Hall-Petch plot for pure magnesium. Data zone from Fig. 1 also included.

If we now assume that the strongly textured specimens are deforming predominantly by prismatic slip, we can use the measured textures to estimate the average Schmid factor, m . The average Schmid factors was measured from XRD texture data for each processing condition, and all fell within the range of 0.40 to 0.45. We can now estimate the stress required to activate prismatic slip by multiplying the yield point by the average Schmid factor. The $\sigma \cdot m$ data for each of the alloys are shown in Fig. 6. It can be seen that the three most concentrated alloys fall essentially on the same line, and that the weakest alloy, 0.2%Zn lies between the pure magnesium and high Zn lines. Extrapolation of the Hall-Petch line for the alloy compositions indicates they would have an intercept ~ 25 MPa. This value is far below those measured for single crystals. For this reason it is suggested that for the alloy compositions tested here, yielding behaviour at larger grain sizes must plateau, as it does in pure magnesium. We can predict the minimum value of the plateau from measurements made on single crystals by Akhtar *et al.* [5], see Table 1. Using these single crystal values as a guide, the stress to activate the prismatic system has been elucidated for a range of compositions and grain sizes, Fig. 6.

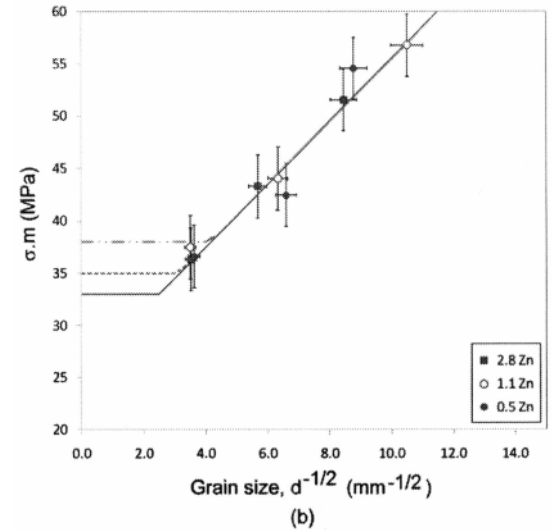
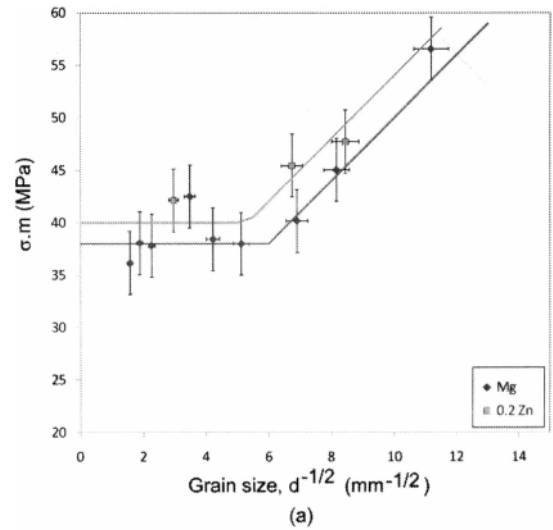


Figure 6 – Stress to activate prismatic slip in strongly textured magnesium plate estimated from the yield point multiplied by the average Schmid factor, m .

Discussion

Hall-Petch Effect

Let us firstly consider the measured yield stress values for these dilute binary alloys against those measured on more concentrated commercial alloys, Fig. 7. This figure combines the data from Figs. 1 and 5, and also includes some additional data for pure magnesium from the literature. It is firstly apparent that the strongly textured commercial alloys almost all lie within a single linear band, colored grey in Figs. 1 and 7. The different alloys do

Table 1 – Single crystal values for prismatic slip measured in [5]

| Composition | Measured CRSS [5] | CRSS used here |
|-------------|-------------------|----------------|
| Mg | 45 MPa | 38 MPa |
| Mg-0.2Zn | 47.6 MPa | 40 MPa |
| Mg-0.5Zn | 45.5 MPa | 38 MPa |
| Mg-1.1Zn | 41.5 MPa | 35 MPa |
| Mg-2.8Zn | Not measured | 33 MPa |

not shift up or down significantly from one another, see Fig. 1. These data are a combination of rolled and extruded materials, and all will be expected to have strong textures. Thus, they will be deforming predominantly by prismatic slip. This can therefore be considered a Hall-Petch plot of prismatic slip for these materials. It is clear that for the alloys that lie in this grey band, prismatic slip is not strongly affected by the compositional differences between them. It is also apparent that these data all tend towards the single crystal CRSS values at the y-axis, although it is not clear what the trend will be at large grain sizes, $> 100 \mu\text{m}$. This lack of sensitivity to composition in commercial type alloys has been noted in previously published literature [11,18]. However, it can be seen from Fig. 7 that pure magnesium is softer than the commercial alloys under conditions that promote prismatic slip. The addition of Zn strengthens the alloy, as shown in Fig. 7, but in the binary alloys tested here the strength exhibited by commercial alloys is not reached. The obvious difference between the alloys plotted in figs 1 and 7 and the Mg-Zn binaries examined here is the presence of large amounts of Al in the alloy systems. The presence of Al in the commercial alloys is likely to account for their higher strength.

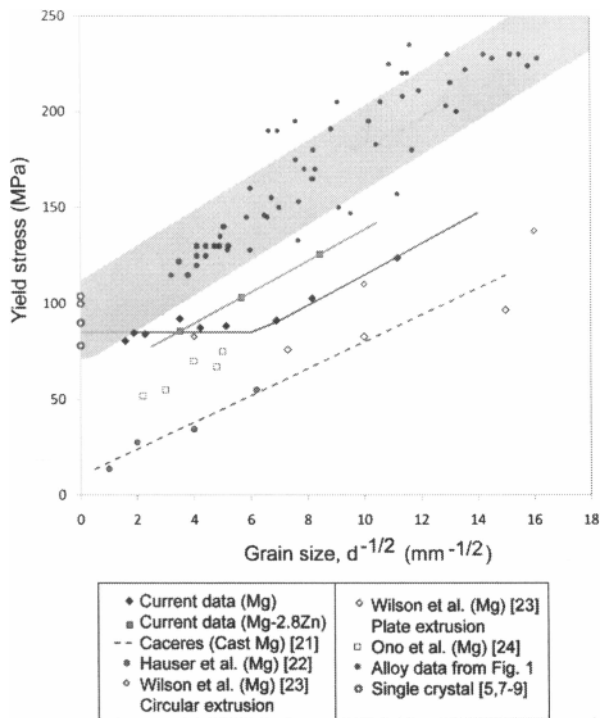


Figure 7 – Hall-Petch plot for pure magnesium taken from literature. Data points from current study also included for pure magnesium and Mg-2.8Zn alloys. Data zone for alloy compositions from Fig. 1 also included.

Also shown in Fig. 7 is previously published Hall-Petch data for pure magnesium. At first glance the data appears to not fit with the measurements made here. However, these discrepancies can be explained by the significant texture differences between the tested samples. The dashed line in Fig. 7 is the Hall-Petch slope measured by Caceres *et al.* on cast magnesium [21]. Being randomly oriented, the deformation of as-cast material will not be dominated by one deformation mode in particular, but will likely be significantly contributed to by both twinning and basal slip, the two softest modes. Therefore the measured Hall-Petch plot for the cast magnesium shows a significantly lower stress compared to the textured magnesium for a given grain size. This measured Hall-Petch slope by Caceres *et al.* fits closely with the data measured by Hauser *et al.* [22] and also that measured by Wilson and Chapman [23] on pure magnesium. We can therefore conclude that the pure magnesium measurements made in those cases differ to the present results due to texture differences. The data published recently by Ono *et al.* [24] sits between these two extremes of randomly textured and strongly textured materials, and it is likely that their samples had a texture that behaved somewhat between these two texture types. This demonstrates that a Hall-Petch plot for magnesium will only be useful in predicting the yield strength for a given grain size if the texture and orientation of testing are consistent with those used to develop the plot.

Solute Softening

The concept of solute softening, rather than strengthening, was reported by Akhtar and Teghtsoonian [5] who showed that the CRSS for prismatic slip of Mg single crystals decreased with the addition of Zn. Figure 8 shows the effect of Zn content on the stress to activate prismatic slip in the polycrystals examined in this study. This graph was produced by using the data shown in Fig. 6 and plotting it against Zn concentration. This figure shows that although large grain sizes may exhibit solute softening, at grain sizes below $\sim 50 \mu\text{m}$ the addition of Zn has a strengthening effect in magnesium. It is therefore concluded that for the grain sizes commonly used for wrought product, solute softening is not a phenomena that is likely to occur.

Single crystal and polycrystal CRSS values

One of the motivating factors for this work was to try and explain why the ratio of the CRSS values for prismatic slip compared to basal slip are so vastly different for single crystals compared to polycrystals. For the data examined here it appears that the plateau in stress required to activate prismatic slip in dilute magnesium alloys could be one contributing factor to this discrepancy. If the basal slip system hardened with increasing grain size, while the prismatic system remained unchanged, then the ratio $\text{Pris}_{\text{CRSS}} / \text{Basal}_{\text{CRSS}}$ would be reduced accordingly. However, at this stage there are no quantitative experimental studies on the effect of grain size on the CRSS for basal slip, so a quantitative evaluation of how this ratio changes with grain size cannot at this stage be made.

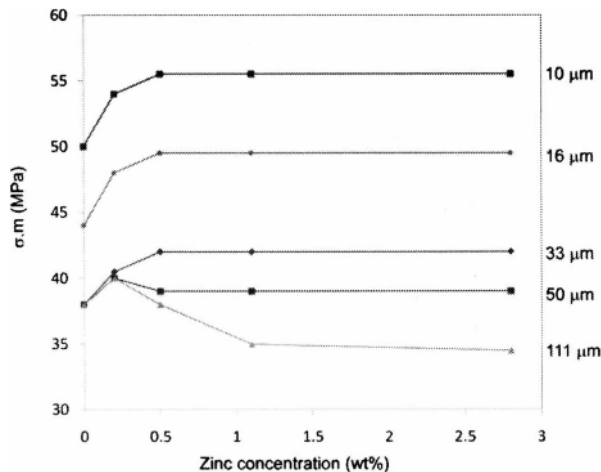


Figure 8 – Data from Fig. 6 re-plotted to show the effect of Zn concentration on the stress to activate prismatic slip for different grain sizes.

Conclusions

Five alloys in the range of pure Mg to 2.8 wt% Zn have been examined for a range of grain sizes. The strong texture developed during hot rolling ensured that during tensile testing the dominant deformation mode was prismatic slip. This has been used to examine the Hall-Petch effect on the prismatic slip system. It has been found that:

- The ductility of the alloys increased with increasing Zn concentration
- The work hardening rate was not affected by the Zn concentration
- For pure magnesium textured to favour prismatic slip, the Hall-Petch effect does not apply at grain sizes above ~50 μm. Grain sizes larger than this do not show a reduction in yielding stress with increasing grain size, but plateau at a constant yield stress.
- For pure magnesium textured to favour prismatic slip, the addition of Zn produces a solute softening effect for grain sizes above ~50 μm. At grain sizes smaller than ~50 μm the addition of Zn produces a solute strengthening effect.
- Collation of a range of data from literature has shown that there is a significant difference in the Hall-Petch plot for strongly textured and weakly textured materials.

Acknowledgements

The work described in this paper was funded by the Centre of Excellence for Design in Light Metals. The authors would also like to thank Mohan Setty, Mahendra Ramajayam, Lynton Leigh and Rob Pow for their assistance with the experimental work.

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