Influence of texture on Hall-Petch relationships in a Mg alloy

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Abstract

The influence of changes in crystallographic texture on the Hall-Petch (H-P) relationship for a Mg alloy was investigated. First, the texture variations were facilitated by changing the uniaxial tensile loading orientation with respect to the normal direction of the rolled Mg plate. With a strong plane texture of the as-received material, the initial dominant deformation mechanisms were systematically varied from the basal slip, prismatic slip to extension twinning as well as combinations thereof. Second, different grain sizes were produced for each loading orientation through isochronal annealing at various temperatures up to 773K while closely monitoring grain size and texture distributions. The experimental results are presented for the grain growth kinetics during annealing; changes in yielding behavior as a function of grain size and initial texture; and H-P relationship as a function of the texture. Moreover, the effects of changes in texture and dominant deformation mechanism on H-P parameters; namely, friction stress, σ_0 , and strength coefficient, $k\sigma$, will be discussed. Finally, H-P relationships for each individual deformation mode including basal, prismatic, and pyramidal slips as well as extension twin will be identified.

Keywords: Magnesium; Texture; Grain size; Hall-Petch relation; Twin; Slip

1. Introduction

Thermo-mechanical processes; such as extrusion, forging, rolling, equal channel angular pressing (ECAP), and friction stir processing (FSP); are employed to shape components or modify the microstructure to improve the ductility and strength of magnesium (Mg) alloys [1, 2]. During thermo-mechanical processes, the grain size can be refined through dynamic recrystallization (DRX) as a result of severe plastic deformation at elevated temperatures [3, 4]. For example, FSP, a solid-state joining process, can significantly refine the grain size in the stir zone [5, 6], depending on the processing parameters [7, 8]. Moreover, the crystallographic texture is often significantly altered during thermo-mechanical processes [9, 10]. According to Yu [11], two distinctly different recrystallization textures, namely, shear or twin-assisted DRX textures, can develop with changes in thermo-mechanical inputs in terms of the Zener-Hollomon parameter during the FSP of a Mg alloy. In general, the changes in grain size and texture during a thermo-mechanical process are significant, which influence the yielding and hardening behaviors of Mg alloys considerably.

The deformation modes in hexagonal-close-packed (hcp) Mg crystals at ambient temperature include three distinct slip systems and one twin system, whose critical resolved shear stresses (CRSSs) have large differences: $0001\langle 11\bar{2}0 \rangle$ basal slip, $10\bar{1}0\langle 11\bar{2}0 \rangle$ prismatic slip, $11\bar{2}2\langle 11\bar{2}3 \rangle$ pyramidal slip, and $10\bar{1}2\langle 10\bar{1}1 \rangle$ extension twin. The selection of a dominant deformation mechanism is highly dependent on the initial texture for Mg alloys, and the relationship between the texture and active deformation mechanisms has been extensively studied using Taylor, Sachs, and self-consistent plasticity models [12-14]. For example, Agnew [15] and Jain [16] demonstrated that the activation of various deformation modes is strongly dependent on the initial texture by comparing the uniaxial compression behaviors along transverse direction (TD), rolling direction (RD), and normal direction (ND) at temperatures up to 200°C for a hot-rolled AZ31B Mg plate. It was reported that prismatic slip was dominant during compression along ND, while the extension twinning was most active during

compression along TD or RD. Also, Yi [17] reported compression and tension behaviors along 0, 45, and 90 degrees to the extrusion direction for a hot-extruded AZ31 alloy. The results showed that various deformation modes were activated with the changes in the angle between the loading direction and the c -axis of the crystal. Overall, it is well established that Mg alloys are plastically anisotropic, and different deformation modes result in distinctly different yielding and hardening behaviors.

The grain size refinement can increase strength of alloys, which follows the Hall-Petch (H-P) relationship:

$$\sigma_y = \sigma_0 + k\sigma D^{-1/2} \quad (1)$$

where σ_y is the yield strength, σ_0 is the friction stress for dislocation movement, $k\sigma$ is the strength coefficient for the normal stress, and D is the grain size. Several models have been developed to elucidate the physics of the H-P relationships including the dislocation pile-up model, which has been successfully applied to low-carbon steels and various other alloys [18]. On the other hand, the effect of grain size on twinning is still not very well understood. Meyer [19] suggested that the extension twin should have a greater grain-size sensitivity (H-P slope, $k\sigma$) than slip-dominant systems due to the unfavorable orientation factors and high twin nucleation stress. On the contrary, Armstrong [20] proposed that twins should have similar grain-size sensitivities since the twin accommodates only the microscopic plasticity within local regions, whereas slip is more uniform in deformation. Nevertheless, it is suggested that grain size generally affects the extension twin through two aspects. First, according to Barnett [21], the stress concentration along the grain boundaries (GBs) triggers the nucleation of the twin, and thus, the nucleation density of twin is sensitive to the grain size. Second, twin size is limited by grain size, which implies that grain size affects twin growth. However, a quantitative understanding of the H-P relationship of extension twin is still lacking and the literature is somewhat inconsistent. In particular, current literature presents vastly different H-P parameters for Mg alloys often without clearly identifying

dominant slip or twin systems. For example, a particular slip system has a specific Burgers vector and critical stress, which determine the grain boundary strengthening [22]. Thus, different deformation systems should result in different H-P relations.

Indeed, a current review of literature [23-48] shows that large discrepancies in the values of H-P parameters are evident as summarized in Table 1. The H-P parameters vary widely even for the same alloy AZ31B: the grain size sensitivity $k\sigma$ ($\text{MPa}\cdot\text{mm}^{1/2}$) varies from 5.0 to 12 and the friction stress σ_0 (MPa) ranges from 10 to 131. First, the H-P parameters change with the changes in processing methods; such as rolling, ECAP, and FSP. Moreover, the H-P parameters depend on the processing parameters. For example, for the rolled AZ31 Mg alloy, the H-P parameters change with the changes in reduction ratio presumably due to the changes in intensity of the plane texture [47]. The H-P parameters are particularly sensitive to the loading path, which is illustrated by comparing tension [33] and compression [38] along the extrusion direction (ED). It is apparent that the texture and deformation modes of Mg alloys have significant influences on the H-P parameters. A quantitative investigation of the effect of changes in initial texture; therefore, changes in initial dominant deformation modes; on the H-P relations of AZ31B Mg alloy would advance the basic understanding of the wide range of H-P parameters reported in the literature.

In this paper, we will first present the grain growth kinetics based on grain size and texture evolutions during the annealing of AZ31B. Then, we will analyze the tensile behavior of the Mg alloy with different initial texture and grain size, and finally, present H-P relationships as a function of the initial texture. Moreover, the variations in friction stress, σ_0 , and the strength coefficient, $k\sigma$, with the changes in texture will be discussed in the context of the changes in dominant deformation mechanisms and dislocation pile-up theory. In addition, a yield strength contour will be generated as a function of grain size and texture. Finally, H-P relationships will be identified in terms of shear stress for basal, prismatic, and pyramidal slips as well as extension twinning. The current study provides a basic understanding of the complex interplay between the grain size and texture that often change

simultaneously and significantly during the thermo-mechanical processing of Mg alloys. Moreover, the H-P relationships for various deformation mechanisms identified in this study can be directly applied to various polycrystal models, including the visco-plastic self-consistent (VPSC) model, to simulate the effect of grain size on the flow behavior under complex deformation conditions, where texture evolves with strain.

2. Experimental

2.1 *Material, heat treatment, and microstructure*

The material used in this study is a commercial AZ31B Mg alloy, which was hot rolled at 573K followed by O-temper annealing at 633K for 240 minutes to obtain fully recrystallized microstructure. The dimension of the rolled plate was 400×250×90mm, which was adequate for a series of tension tests at various angles between normal and transverse directions of the plate. Throughout the paper, ND, TD, RD, and LD refer to normal, transverse, rolling, and tensile loading direction, respectively.

The Mg specimens with different grain sizes were prepared by conducting a series of isochronal annealing of the as-received specimen for 96 hours at various temperatures ranging from 623K to 803K in a sealed quartz tube filled with pure argon gas to prevent oxidation. For the optical microscopy (OM), samples were polished and etched in an acetic picral solution (3g picric acid powder, 5ml acetic acid, 100ml ethanol, and 30ml water). The grain size was measured using the linear intercept method [49] for ND, TD, and RD cross-sections, and the process was repeated at 100 different locations for each annealing condition. The grain size distribution was obtained in terms of mean size and standard deviation from the Gaussian fitting of the measured data.

2.2 *Texture measurements*

The crystallographic texture was measured for the as-received and annealed samples (at 673K, 723K, and 773K) using synchrotron x-ray diffraction (S-XRD) at beamline 11-ID-C, Advanced Photon

Source (APS), Argonne National Laboratory. The wavelength was 0.107841Å, and the nominal distance between the sample and the 2D detector was set as 1540mm, which covered 22 reflections (01.0 to 03.2) for the hcp structure. The beam size was 600µm×600µm, and the cross section of the samples used for the texture measurement was 3mm×3mm. The Perkin-Elmer area detector was used to obtain the Debye-Scherrer rings using the transmission scattering geometry. The texture data was collected by mounting the pin samples on a rotary stage and by rotating ω from 0° to 90° with a 15° step size, obtaining full pole coverage. The Debye-Scherrer rings were converted into diffraction patterns using the Fit2D software with 10° caking. Then, Rietveld refinements were conducted for texture analyses using the Material Analysis Using Diffraction (MAUD) software and the E-WIMV algorithm. The raw pole figure data were further processed using the MTEX quantitative texture analysis software [50] for texture presentation in terms of pole figures. Further details of the texture measurement can be found in [51].

2.3 *Mechanical testing*

A series of dog-bone tensile specimens were prepared by electrical discharge machining (EDM) from the rolled Mg plate, with their tensile LD systematically changing with respect to the ND of the plate. A total of five different orientations were prepared as illustrated in Fig. 1. The angle, θ , between LD and ND of the plate varies from 0° to 90°. For example, 0° case is where the LD is along ND of the plate, while 90° case is where the LD is 90° to the ND and along the TD. The gauge dimensions of the tensile specimen are 3.0 mm wide, 1.6 mm thick, and 16.5 mm long. All tests were performed using a 1-kN Instron E-1000 high-resolution load frame to accurately measure the yield strengths as low as 40MPa. An extensometer was also used to record the strain. The initial strain rate used for the test was 10–3s–1. The tests were stopped when either fracture occurred or a maximum force (1kN) was reached. Several repeat measurements were performed for all five different loading orientations and showed an excellent reproducibility with a variation in the flow stress within $\pm 0.5\%$.

3. Results

3.1 Initial microstructure and texture

The grain size of the as-received specimen was measured on ND, TD, and RD cross-sections of the rolled plate. The grain morphology and size distribution are isotropic with equiaxed grains, which is typical of a hot-rolled plate. The equiaxed grains have a mean size of 26μm, Fig. 2 (a). Fig. 2 (b) shows pole figures of the as-received Mg plate, which exhibit a strong plane texture. Most of the basal poles $\langle 0001 \rangle$ are parallel to the ND with a symmetric angular distribution with the intensity higher than 80% of the maximum within 7–8 degrees.

3.2 Grain growth kinetics and microstructure control

The grain size measurements of the specimens annealed at various temperatures (i.e., 623, 673, 723, 753, 773, and 803K) show equiaxed grains with almost the same size distributions in three orthogonal cross-sections except for the 803K case. The grain size distributions and OMs of the ND cross-sections for a few selected specimens are presented in Fig. 3. The measured grain size distributions were analyzed by the normal Gaussian statistical distribution function, and the mean and standard deviation values are summarized in Table 2. The grain size increases from 26μm for the as-received specimen to 127μm for the specimen annealed at 803K. However, for the specimen annealed at 803K, the grains grew “abnormally” and heterogeneously, and the size distribution could not be analyzed by the Gaussian fitting. Instead, a numerical average value was used to estimate the grain size at 803K.

The grain growth rate, K , was also determined following the Arrhenius equation:

$$K = A \cdot \exp(-Q/RT)$$

(2)

where A is the pre-exponential constant, Q is the activation energy for the grain growth, R is the gas constant, and T is the annealing temperature. K can be also written as [52]:

$$K = D^N - D_0^N t$$

(3)

, where D is the grain size after annealing for a period of time t , N is the grain growth exponent constant, and D_0 is the initial grain size before annealing, i.e., 26 μ m. Therefore, the relationship between the grain size, D , and the annealing temperature, T , can be expressed as:

$$\ln(D^N - D_0^N) = \ln A - Q \cdot 1/RT$$

(4)

The measured grain growth kinetics of AZ31B Mg alloy is shown in Fig. 4. For a pure metal, grain growth component N is 2 [53]; but it is usually higher than 2 for an alloy. It was reported that N ranges from 2 to 8 for Mg alloys [39]. To obtain the best linear fitting, $N=3$ was used in this study. The activation energy for the grain growth is 88 kJ/mol below 773K (Fig. 4 and Table 2), which is comparable to the activation energy for the boundary diffusion (92 kJ/mol) for pure Mg [54]. Thus, the boundary diffusion could be the dominant mechanism for the grain growth below 773K. At 803K, the data point in Fig. 4 is clearly off the linear regression established at lower temperatures with an apparent activation energy (256 kJ/mol) much larger than that of the boundary diffusion as expected from the abnormal grain growth observed in the microstructure studies [55]. The secondary recrystallization is often related to a presence of second-phase particles, texture, or surface effects [56]. Considering the low solid solution temperature (about 480K) of Mg-3%Al alloy, the sudden change in grain growth kinetics at 803K may not be due to the dissolution of precipitation particles. Instead, the presence of a large number of low-angle grain boundaries with the strong plane texture would play a key role. Such boundaries have relatively lower energy and mobility than the high-angle boundaries. During annealing at higher temperatures, the high-angle boundaries may grow preferentially to cause the abnormal grain growth and also result in a texture change.

The textures of the annealed specimens were also investigated to ensure that the initial strong basal texture is maintained during the grain growth process. First, potential changes in texture after the annealing processes were examined using a laboratory x-ray diffraction. The ratios of integrated intensities of 0002 reflections measured from ND and RD surfaces were examined for different annealing processes. The ND/RD intensity ratio is reasonably constant up to 773K, Table 2. However, the intensity ratio is significantly altered at 803K, consistent with the abnormal grain size distribution and changes in the apparent activation energy. Finally, we compared the 0002 pole figures and axial distributions of 0002 pole intensities (from ND to TD) for the specimens annealed up to 773K to those of the as-received specimen using S-XRD, Fig. 5. The consistency in the angular distribution and in the maximum intensity of the 0002 poles for the specimens with different grain sizes confirms that the strong plane texture of the rolled plate was maintained during the annealing processes up to 773K.

Therefore, the annealing temperatures for the grain size control were limited to 773K in the subsequent H-P study to ensure that the grain size distribution is reasonably homogenous and the initial texture is maintained.

3.3 *Tensile behavior as a function of θ*

Figure 6 shows tensile stress-strain curves for the as-received specimens ($D \approx 26\mu\text{m}$) measured as a function of θ , the angle between the tensile LD and ND of the Mg plate. It is clearly shown that the yielding (inset in Fig. 6) and hardening behaviors change with the changes in the tensile orientation.

For the 0° case (i.e., tension along ND to which most of the c -axes are parallel), 0.002 offset yield strength is low at 57MPa, followed by a plateau from 0.002 to 0.025 strain with little strain hardening. Following the plateau, the hardening rate increases significantly and a “concave-up” stress-strain curve is observed. The hardening rate decreases again when the strain increases to about 0.18. Such stress-strain behavior is different from ones dominated by slip systems, whose hardening rate continuously decreases with the increase in strain. The stress-strain behavior for the 0° case is a manifestation of an extension-

twinning dominant plastic deformation, similar to the compression along TD or RD of a rolled sheet [57, 58] or compression along the extrusion direction of an extruded bar [59]. The appreciable work hardening with the increase in strain is caused primarily by the crystal reorientation as a result of the extension twin [59]. The stress-strain behavior of the 22.5° case is similar to that of 0°, in that the plateau stage occurs after the yielding, while the subsequent hardening rate is slightly lower than that of the 0° case.

For the 45° case, the tensile behavior is qualitatively different from those of 0°/22.5° cases, even though all three cases show a similar level of yield strength. The hardening rate of the 45° case is the lowest among all five orientations and the plateau does not occur during the deformation.

For the 67.5° and 90° cases, the stress-strain curves are parabolic, similar to a multiple slip dominant plastic deformation behavior with a continuous decrease in hardening rate. The yield strength of the 67.5° case is between those of 45° and 90° cases.

3.4 *Hall-Petch relationship as a function of θ*

Figure 7 shows the tensile stress-strain behaviors of specimens with four different grain sizes for each loading orientation, θ . Also shown in the insets are magnified views of the yield behavior for different grain sizes. The characteristic yielding and hardening behaviors for each θ indicate that different deformation mechanisms dominate the plastic deformation with the changes in texture as discussed earlier. Moreover, it is clearly shown that the yield strength decreases systematically with the increases in grain sizes in all cases. However, in this study, the focus will be on the basic understanding of the yield behavior as a function of texture (i.e., “initial” dominant deformation mode) and its interplay with the changes in grain size. The investigation of the hardening behaviors for various orientations and grain sizes (including compressive behavior) will be a subject of our future publications based on S-XRD experiment and VPSC modeling.

The 0.002 offset yield strengths were measured for four different grain sizes at five different LDs; and the results are plotted as a function of the inverse square root of the grain size in Fig. 8. The error bars for the grain size (shown only for the 90° case for clarity) are produced from the standard deviation of the

Gaussian distribution fitting. The linear regression of measured data for each orientation clearly demonstrates that H-P relationship can be established as a function of θ . In Table 3, different intercepts (σ_0) and slopes ($k\sigma$) obtained for each θ are summarized. The measured σ_0 ranges from 10.5 to 41.1 (MPa) and $k\sigma$ ranges from 5.0 to 13.0 (MPa·mm^{1/2}) for different loading orientations. They are in good agreement with the wide ranges reported in Table 1. It is shown that the $k\sigma$ is lowest for the tension along 45°; while the 90° case exhibits the highest $k\sigma$ and σ_0 , implying the strongest grain size sensitivity. In summary, the grain-size effect on yield strength of the Mg alloy clearly obeys the H-P relationship, where the parameters are strongly dependent on the initial texture, and therefore, initial dominant plastic deformation mechanism.

4. Discussion

4.1 Dominant deformation mechanisms at yielding for different orientations

The deformation modes of Mg alloys at ambient temperature include three slip systems and one twinning system whose CRSSs are significantly different. Therefore, the yield strength is directly related to the activation of a particular deformation mechanism and is significantly affected by the angular relationship between the mechanical loading direction and its crystallographic texture in wrought Mg alloys [60].

According to the Schmid law of a single crystal, the yield strength, σ_y , can be written as:

$$\sigma_y = \tau_{CRSS} m$$

(5)

, where τ_{CRSS} is the CRSS of a specific deformation mode and m is the Schmid factor. For polycrystals with a strong texture, Schmid factors for different deformation mechanisms can still provide valuable insights to the understanding of the dominant mechanisms, in particular, at yielding. Assuming that the

rolled plate has an ideal plane texture $0001 \langle 1010 \rangle$, Schmid factors for the various deformation modes can be calculated for different LDs (see Table 4). In the “ideal” Schmid factor calculation, two points are worth noting. First, to simplify the calculation, it was assumed that the rolled Mg plate has the ideal $0001 \langle 1010 \rangle$ texture, which means the c -axes of all crystals are perfectly parallel to the ND without any texture distribution. Second, the largest Schmid factor is selected if several variants exist for each deformation mechanism. Therefore, the “ideal” Schmid factors shown in Table 4 are the upper boundary values, and the calculated σ_y using such m values would provide lower boundaries. The ideal Schmid factors of various deformation modes change sharply with θ and illustrate shifts in the dominant deformation mechanism clearly. However, if the actual crystal orientation distribution is considered, the effect of crystal orientation on the H-P parameters (σ_o and $k\sigma$) can be calculated more accurately. Therefore, Schmid factor distributions for each deformation mode at each loading orientation are obtained from the orientation distribution function measured using S-XRD and the average Schmid factor values are calculated (Table 4). Moreover, standard deviations are also obtained from the Gaussian distribution function analyses (Table 4). Typical standard deviation of a dominant deformation mode is about 10% of the average Schmid factor. The measured values show slightly smoother changes as a function of θ since the distribution of 0002 pole from the ND are taken into account.

For the 0° case (LD//ND), the extension twin has the highest m (0.499) except for the pyramidal slip whose CRSS is too high to be considered as a slip mode at yielding. With the increase in θ , m of extension twin continuously decreases. When θ is higher than 47° , the extension twin is impossible to be activated with c/a ratio close to or smaller than 1.633. For the 45° case, the basal slip is the most favorable deformation mechanism in terms of m . On the other hand, neither extension twin nor basal slip is possible when θ is 90° . Therefore, only prismatic slip is able to accommodate the plastic deformation, which is consistent with Jiang [60], Barnett [12], and Koike’s [61] studies. However, since the yield strength is determined not only by the crystal orientation but also by the CRSS, the much higher CRSS of the prismatic slip results in the increase in the yield strength for the 90° case. Moreover, as mentioned earlier,

the pyramidal slip and contraction twin are not taken into account further for the study of the yield behavior because of their significantly higher CRSSs compared to other three deformation modes. The current observation of the changes in dominant deformation mode as a function of θ is consistent with Oppedal's [62] and Jain's [63] studies using elasto-plastic self-consistent (EPSC) and VPSC simulations of the rolled Mg plate.

4.2 σ_o as a function of θ

In the H-P relationship, σ_o is the friction stress for dislocation movement, at which material yields if the grain-boundary effects are not considered. The significant variations in σ_o with the changes in θ are clearly shown in Table 3. According to Armstrong [64], the σ_o is related to $\tau_{CRSS}\beta$ (CRSS of a given deformation mode β) and orientation factor M :

$$\sigma_o = M \cdot \tau_{CRSS} \beta$$

(6)

In this section, the friction stress, σ_o , will be calculated as a function of θ using Equation (6) based on the measured average Schmid factors (Table 4) and representative CRSSs for each deformation mode from the literature [12, 65] (Table 5). Then, the calculated σ_o will be compared to the current measured values at five different orientations. The CRSSs for different deformation mechanisms in Mg alloys have been reported in previous studies. However, the data vary widely even for a given alloy system depending on the methods used in the study including single crystal stress-strain behavior measurements [66] and various plastic deformation modeling methods such as EPSC, VPSC, or Sachs polycrystal models [12, 65, 67]. For example, the CRSS reported for basal slip ranges from 5 to 40MPa. Note that the CRSS for a single crystal may not be directly applied to a polycrystal behavior as evidenced in the discrepancies between the CRSS of slip systems obtained from the single crystal stress-strain

behavior and the polycrystal simulations mentioned above [45, 65]. For many modeling schemes (e.g., VPSC), the CRSS inputs for each deformation mechanism are not usually the true CRSSs. In fact, the input parameters for the CRSSs are the resolved shear stresses for a given grain size, which provide best fits to the specific stress-strain curve measured experimentally. Therefore, the CRSSs used in such polycrystal modeling schemes have already been combined with the grain-boundary effects, which explains the reason for the relatively higher values of CRSSs used in many simulations. Since the Schmid factors for 1012 twinning, basal slip, and prismatic slip at 0°, 45°, and 90°, respectively, are all close to 0.5 (Table 4), the differences in σ_o shown in Table 3 for these three orientations must be mainly due to the differences in CRSSs. Indeed, the CRSSs for extension twinning, basal slip, and prismatic slip used in the calculation of σ_o (Table 5) show good agreements with the σ_o measured at $\theta=0^\circ$, 45°, and 90°, respectively, with a similar ratio of:

$$\tau_{CRSSBasal}:\tau_{CRSSPrism}:\tau_{CRSSTwin}=1:2.0:0.7$$

The friction stress, σ_o , is calculated for different deformation modes for the five different orientations, and the results are presented in Table 6. Typical deviation for the calculated σ_o , inherited from the standard deviations of measured Schmid factors, would be about 10% of the stress values. For example, the deviation of σ_o for extension twin at $\theta=0^\circ$ is 1.6 MPa and that for prismatic slip at 90° is 6 MPa. The dominant deformation modes are revealed for each θ by comparing the value of $M \cdot \tau_{CRSS} \beta$ for all possible modes. The minimum calculated σ_o in each loading orientation agrees well with the measured σ_o in that particular orientation. For the 0° and 22.5° cases, the calculated σ_o for twinning is the minimum. The basal slip is the easiest one to be activated at 45° and 67.5°. At $\theta=45^\circ$, both basal slip and extension twin may be activated at the same time since the values of $M \cdot \tau_{CRSS} \beta$ are quite similar. The

σ_o is higher at 67.5° than at 45° because it is a slightly harder orientation with a relatively lower Schmid factor. At 90° , prismatic slip has the lowest σ_o .

Moreover, Fig. 9 shows both the calculated (curves) and measured (symbol) σ_o as a function of θ .

The error bars for the measured σ_o comes from the standard deviation of the linear fitting of the H-P relations. The lower boundaries of the calculated lines across the θ represent dominant deformation mechanisms at yielding. The calculation indicates that from $\theta=0^\circ$ to 39° , extension twinning is dominant, while basal slip will contribute mostly at yielding between $\theta=0^\circ$ and 83° . The prismatic slip is dominant from 83° to 90° . Henceforth, 0° , 67.5° , and 90° cases will be designated as extension twin, basal slip, and prismatic slip cases, respectively.

4.3 $k\sigma$ as a function of θ

The slope of the H-P relation, $k\sigma$, which represents grain size sensitivity, has a wide range of values for different materials. In general, soft metals with face-centered cubic (fcc) structures have a lower $k\sigma$, while harder metals with body-centered cubic (bcc) structures have a higher value. For example, $k\sigma$ of Mo is more than 15 times that of Cu (about $3.5 \text{ MPa}\cdot\text{mm}^{1/2}$). The physical mechanisms of the H-P relationship discussed in the literature include: (1) dislocation pile-up theory [68], (2) GB source theory [69], (3) slip distance theory [70], and (4) geometrically-necessary dislocation theory [71]. One of the most commonly cited mechanisms for the grain-boundary effect is the dislocation pile-up theory proposed by Hall [72]. For Mg alloys, dislocation pile-ups near the grain boundaries have been investigated using transmission electron microscopy (TEM) [73, 74], which supported the dislocation pile-up theory in that the basal and non-basal dislocations are confined in the slip planes and pile up against grain boundaries in

the stressed AM50 and AZ31B with the spacing between each parallel dislocation at only several nanometers.

According to the dislocation pile-up model [18], the effective stress, τ_e , acting on the moving dislocation is:

$$\tau_e = \tau - \tau_0 \quad (7)$$

where τ is the applied shear stress and τ_0 is the friction shear stress of the dislocation. If n dislocations are piled up near the GBs, the cumulative stress due to the pile-up is represented as:

$$\tau_p = n\tau_e \quad (8)$$

According to the dislocation line tension energy expression, the line tension energy, TL , can be written as:

$$TL = \tau_e b L_p n \quad (9)$$

where b is the dislocation Burgers vector, and L_p is the pile-up length, which can be assumed to be similar to the grain size, D . Hence, Equation (8) can be rewritten as:

$$\tau_p = \tau_e 2b L_p TL \quad (10)$$

From Equations (7) and (10),

$$\tau_p = (\tau - \tau_0) 2b L_p TL \quad (11)$$

The dislocation line tension energy, TL , can also be expressed as:

$$\tau l = \alpha G b \lambda$$

(12)

where G is the shear modulus and α is a constant. For screw dislocation, $\alpha = 1/2$; while for the edge dislocation, $\alpha = 1/2(1-\nu)$. ν is the Poisson's ratio, which is 0.289 for Mg. It was observed that screw dislocations are more stable on the basal plane, while the edge dislocations are more common on the prismatic plane [75]. Also, for $\langle c+a \rangle$ dislocation, screw dislocations glide on $11\bar{0}1$, while edge dislocations glide on $11\bar{2}2$ planes according to the first principle simulation [76]. Accordingly, $\alpha = 1/2$ was used for the basal slip, while $\alpha = 1/2(1-\nu)$ was used for the prismatic and pyramidal slips.

The dislocation can move across the GBs when the τ_p is as high as the critical stress τ_c . The applied shear stress τ can be replaced by resolved shear stress, τ_R , for a given grain size. Then, from equations (11) and (12), the resolved shear stress, τ_R , can be written as:

$$\tau_R = \tau_0 + (\alpha \tau_c G b) \lambda^{1/2}$$

(13)

which represents the H-P relationship in terms of shear stress, where $k\tau$ is defined as:

$$k\tau = (\alpha \tau_c G b) \lambda^{1/2}$$

(14)

The grain size sensitivity for the shear stress is proportional to the square root of the products of: shear modulus, Burgers vector, and critical shear stress, τ_c . Finally, the H-P slope for yield strength, $k\sigma$, can be written as:

$$k\sigma = M (\alpha \tau_c G b) \lambda^{1/2}$$

(15)

Using the CRSSs (as τ_c), shear moduli [77], Burgers vectors, and the inverse of the measured Schmid factors for basal and prismatic slip (Tables 4 and 5), $k\sigma$ is calculated as a function of the loading orientation, Fig. 10. However, the grain size effect of the extension twin cannot be simply explained by the same theory. For extension twin, the $k\sigma$ is calculated using: $k\sigma_{Twin}(\theta) = k\sigma_{Twin}(0^\circ) \cdot M(\theta) / M(0^\circ)$, since the physical basis of the H-P theory for the extension twin is still unavailable at this point.

The lower boundaries of the calculated $k\sigma$ across the θ show good agreements with the measured $k\sigma$ values (symbol), which demonstrate that the application of dislocation pile-up theory in the calculation of grain size sensitivity of the current Mg is a reasonable approach. The significant variations in the calculated and measured $k\sigma$ with θ are reflected well in the wide range of the H-P parameters reported in the literature (Table 1) for Mg alloys subjected to different thermo-mechanical processes and test conditions. For instance, Wang [33] reported that $k\sigma$ ($\text{MPa} \cdot \text{mm}^{1/2}$) decreased from 9.6 to 5.1 after the FSP of an extrusion bar of AZ31B. When the tensile loading axis was parallel to the initial extrusion direction, the normal direction of most 0001 planes was perpendicular to the applied stress, and prismatic slip dominated the plastic deformation. This behavior was similar to the tension along 90° case in the current study, and the highest grain size sensitivity was expected. After the FSP, the texture component changed to a condition that favored basal slip, which is similar to the tension along the 45° case here. Therefore, the decrease in $k\sigma$ was expected.

4.4 Yield strengths as a function of grain size and loading orientation

When θ is the angle between the LD and the c -axis of the crystal, the vector of loading is:

$$\mathbf{H} = (\cos\theta, 0, \sin\theta) \quad (16)$$

In the Cartesian coordinate system, slip plane normal **b** and the slip direction **c** for a given deformation mode can be expressed as:

$$\mathbf{I} = 1001/32/30001/1.624 \cdot \mathbf{P}\beta$$

(17)

$$\mathbf{J} = 1 - 0.5003/20001.624 \cdot \mathbf{U}\beta$$

(18)

where **U** β is the slip direction, **P** β is the slip plane normal, and 1.624 is the *c/a* ratio of the Mg alloy. Thus, the orientation factor *M* can be calculated by:

$$M = \mathbf{H} \cdot \mathbf{I} \mathbf{H} \cdot \mathbf{I} \cdot \mathbf{H} \cdot \mathbf{J} \mathbf{H} \cdot \mathbf{J}$$

(19)

Therefore, the yield strength for each slip mode and extension twin can be calculated as a function of grain size *D* and tensile orientation θ :

$$\sigma_y = M\tau_o\beta + \alpha G\beta\tau_o\beta b\beta^{1/2} \cdot D^{-1/2} \quad \text{Slip}$$

(20)

$$\sigma_y = M\tau_{otwin} + MM(O) \cdot k_{ttwin} \cdot D^{-1/2} \quad \text{Extension twin}$$

(21)

The tensile yield strength contours as a function of the grain size and loading orientation, θ , with respect to the normal direction (i.e., *c*-axis) of the hot-rolled Mg plate are presented in Fig. 11 for individual deformation modes as well as for the lower boundaries considering all possible modes. It is shown that highest yield strength can be obtained under tension along 90° with a small grain size, while yield strength will be lowest when tension is along 45° with a large grain size. The current result demonstrates that the yield contour for the AZ31B Mg alloy can be constructed as a function of the grain

size and tensile loading orientation when the texture of a thermo-mechanically processed component is known.

4.5 *H-P relationships for individual deformation mechanisms*

The resolved shear stress ($\tau R\beta$) for extension twin, basal slip, and prismatic slip at various grain sizes can be calculated from the measured yield strengths from the 0°, 67.5°, and 90° cases, respectively. Therefore, the H-P relationships can be expressed for these individual deformation mechanisms in terms of $\tau R\beta$ as a function of inverse square root of grain size, Fig. 12. The intercepts of the linear regression are $\tau_0\beta$ and the slopes represent the grain size sensitivity, $k\tau\beta$, for each deformation mode as summarized in Table 7. Moreover, the calculated $k\tau\beta$ for basal, prismatic, and pyramidal slip according to the equation (14) are also shown in Table 7. It should be noted that the H-P relation for the pyramidal slip is not easy to measure experimentally, since it is never the dominant yielding mode at room temperature at any initial texture conditions due to its high CRSS. However, the H-P relationship of pyramidal slip can represent the grain-size effect on flow stress during the hardening stage and it can be verified using a plasticity modeling scheme. We plan to discuss the VPSC modeling that incorporates H-P relationships in future publications.

Table 7 shows that the grain size sensitivity of pyramidal slip is the highest (9.86 MPa·mm^{1/2}) among all deformation modes, while the basal slip (2.61) and extension twin (3.48) have the lowest values with the prismatic slip in between (5.54). The results are consistent with the general concept that hard slip modes have a more potent grain size dependence than soft ones [61]. Specifically, the grain size sensitivity is much higher for the pyramidal and prismatic slip systems with much higher CRSSs than the basal slip.

In the shear stress analysis, since the crystal orientation factor is not considered, the difference in grain size sensitivities comes solely from the physical differences in the deformation systems such as the

critical stress, Burgers vector, and shear moduli. Therefore, the grain size sensitivity of shear stress for each deformation mode should not be altered, even when the texture (or the grain orientation) changes. For example, both basal slip and prismatic slip are the movement of $\langle a \rangle$ dislocation, which has the Burgers vector of $a/3\langle 1120 \rangle$. The shear moduli of the basal plane and prismatic plane are only slightly different because of the asymmetry of the hcp crystal [77]. Therefore, differences in the grain size sensitivities for these two slip systems mainly come from the differences in τc .

However, it is important to note that even though the CRSS for the extension twin is lower than that of basal slip, the measured grain-size sensitivity is higher for the twinning. This observation suggests that the physical mechanism responsible for the H-P relationship for the extension twinning is different from that of the slip. According to Armstrong [20], twin is generated as a result of the microslip concentration due to the dislocation pileup, and it was proposed that $k\tau_{Twin} \approx (1.5-7)k\tau_{Slip}$ in bcc metals. In the current study, it was found that $k\tau_{Twin} = 1.3k\tau_{Basal}$, which is close to the lower boundary of the relation between the twinning and slip systems identified by Armstrong [20].

5. Conclusions

The influence of texture on the Hall-Petch relationship is investigated using a commercial hot-rolled AZ31B Mg alloy plate with a strong $0001\langle 1010 \rangle$ plane texture. The texture variations are facilitated by changing the angle, θ , between the uniaxial tensile loading direction (LD) and the normal direction (ND) of the plate. Also, different grain sizes are produced for each θ through isochronal annealings of 96 hours at various temperatures. The conclusions are as follows.

1. The as-received grain size of 26 μm increases systematically to 78 μm through a homogenous grain-growth process without altering the initial plane texture during annealing at various temperatures up to 773K. The grain growth kinetics study shows that the boundary diffusion is the dominant mechanism with the activation energy of 88 kJ/mol. However, when the annealing

temperature is higher than 773K, the grain growth is heterogeneous and the texture changes significantly due to the secondary recrystallization process with a much higher apparent activation energy.

2. The 0.002 offset yield strengths are measured for four different grain sizes ($D = 26, 43, 63$, and $78\mu\text{m}$) at five different tensile LDs ($\theta = 0, 22.5, 45, 67.5$, and 90°) and Hall-Petch (H-P) relationships are established as a function of θ . The measured H-P parameters of friction stress, σ_0 , and grain size sensitivity, $k\sigma$, vary significantly depending on the loading direction.
3. The σ_0 is calculated as a function of θ based on: (1) Schmid factors obtained from the texture measured using synchrotron x-ray diffraction and (2) critical resolved shear stresses (CRSSs) for basal, prismatic, and pyramidal slip as well as extension twinning. The calculation shows a good agreement with the measured values. Moreover, significant variations in the calculated σ_0 as a function of θ clearly identify changes in the dominant deformation mechanism at yielding. The extension twinning is dominant at yielding for $\theta=0^\circ$ to 39° , basal slip from 39° to 83° , and prismatic slip from 83° to 90° .
4. The grain size sensitivity, $k\sigma$, is also calculated based on the dislocation pile-up theory. The calculated $k\sigma$ shows considerable variations as a function of θ , illustrating its dependency on both characteristics of the dominant deformation mechanism (e.g., CRSS and Burgers vector of the dislocation) and crystal orientation factor. Both measured and calculated values of $k\sigma$ explain the wide range of H-P parameters reported in the literature for Mg alloys subjected to different thermo-mechanical processes and test conditions.
5. The yield strength contours are constructed for individual deformation modes (basal, prismatic, and pyramidal slip as well as extension twinning) as a function of grain size and tensile loading orientation based on the H-P parameters established in the current study. Moreover, the lower boundaries of the yield strength contours for all possible deformation modes are generated, which

show that highest yield strength can be obtained under tension along 90° with a small grain size, while yield strength will be the lowest under tension along 45° with a large grain size.

6. The H-P relationships for individual deformation mechanisms are identified. The grain size sensitivity $k\tau\beta(\text{MPa}\cdot\text{mm}^{1/2})$ increases from basal (2.61), prismatic (5.54) to pyramidal (9.86); which is consistent with the increase in the CRSS, $\tau_0\beta$, for each system. On the other hand, extension twinning has higher $k\tau$ (3.48) than the basal slip (2.61), even though the twinning requires smaller τ_0 (5.7 MPa) compared to that of the basal slip (9.9 MPa). Such discrepancy indicates that the physical mechanism for the grain size hardening of extension twinning is different from that of the slip modes.

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Figure 12. Hall-Petch relationships for basal slip, prismatic slip, and extension twinning for AZ31B Mg alloy in terms of the shear stress calculated from the yield strengths and measured Schmid factor. The y -intercept $\tau_o\beta$ and the slope $k\tau\beta$ are summarized in Table 7.

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Table 7. Hall-Petch parameters of various deformation modes for AZ31B Mg alloy.

Table 1. Hall-Petch parameters (σ_0 and $k\sigma$) for AZ31B Mg alloy as functions of the processing condition and loading path for a given grain size range: a literature review.

Materials	Processing Condition	Loading Path	Grain Size (μm)	σ_0 (MPa)	$k\sigma$ ($\text{MPa}\cdot\text{mm}^{1/2}$)	Ref.
AZ31	Rolling	Tension (In-plane)	16-40	60	10.5	[46]
AZ31	Rolling	Tension //RD	5-25	45	10.1	[43]
AZ31	Single Rolling	Tension //RD	5-17	89	7.3	[47]
AZ31	Rolling	Tension //RD	5-21	131	7.9	[47]
AZ31	Rolling	Tension //RD	2-55	122	6.6	[32]
AZ31	Rolling	Tension //RD	13-140	88	8.9	[16]
AZ31	Rolling	Tension //TD	13-140	115	8.6	[16]
AZ31	Extrusion and Drawing	Tension //ED	3.5-12	120	5.0	[48]
AZ31	Extrusion	Tension //ED	2.5-8	80	9.6	[33]
AZ31	Extrusion	Compression //ED	3-11	22	12.3	[38]
AZ31	FSP	Tension //AD	2.6-6.1	10	5.1	[33]
AZ31	ECAP	Tension //ED	5-32	30	5.4	[49]
AZ31	ECAP	Tension //ED	6-22	50	10.8	[37]
AZ31	ECAP	Tension //ED	9-22	10	10.3	[37]
AZ31	ECAP	Tension //ED	2-8	30	5.7	[30]

Note: FSW, friction stir welding; ECAP, equal channel angular processing; RD, rolling direction; TD, transverse direction; ED, extrusion direction.

Table 2. Grain size distribution (mean and standard deviation), activation energy for grain growth, and texture (intensity ratio) measured after annealing at various temperatures.

Annealing Temp. (K)	Grain Size (μm)		Q (kJ/mol)	Texture: (0002) Integrated Intensity Ratio between ND and RD
	Mean	Std. Dev.		
As-rec.	26	3.5	-	52
623	33	3.2	88	-
673	43	3.4	88	57
723	63	3.4	88	-
753	72	4.0	88	-
773	78	5.5	88	59
803	127	-	>88	28

Table 3. Hall-Petch parameters (σ_0 and $k\sigma$) measured as a function of the angle, θ , between the LD and ND.

Angle, θ (Deg.)	Hall-Petch Parameters		R^2
	σ_0 (MPa)	$k\sigma$ (MPa $\cdot$ mm ^{1/2})	
0	12.2 (± 1.2)	7.2 (± 0.2)	0.996
22.5	10.5 (± 1.1)	7.3 (± 0.2)	0.996
45	18.2 (± 0.3)	5.0 (± 0.07)	0.999
67.5	26.5 (± 3.6)	7.0 (± 0.7)	0.965
90	41.1 (± 6.6)	13.0 (± 1.4)	0.964

Table 4. Ideal/measured Schmid factors, m , for basal slip, prismatic slip, pyramidal slip, and extension twin as a function of the tensile loading orientation, θ , between the LD and ND. Ideal Schmid factor is calculated assuming a perfect $0001\langle 10\bar{1}0 \rangle$ plane texture, while measured average Schmid factors are obtained from intensity distributions in the pole figures measured using S-XRD. The numbers shown in parentheses are standard deviations obtained from Gaussian distribution function analyses.

θ	Basal Slip	Prismatic Slip	Pyramidal Slip	Extension Twin
0	0 / 0.163 (± 0.047)	0 / 0.034 (± 0.075)	0.446 / 0.467 (± 0.022)	0.499 / 0.464 (± 0.050)
22.5	0.354 / 0.326 (± 0.072)	0.036 / 0.032 (± 0.038)	0.445 / 0.470 (± 0.038)	0.351 / 0.423 (± 0.044)
45	0.500 / 0.468 (± 0.055)	0.117 / 0.114 (± 0.058)	0.280 / 0.305 (± 0.077)	0.125 / 0.263 (± 0.065)
67.5	0.354 / 0.375 (± 0.068)	0.269 / 0.276 (± 0.055)	0.445 / 0.404 (± 0.070)	- / 0.075 (± 0.079)
90	0 / 0.078 (± 0.052)	0.433 / 0.428 (± 0.046)	0.446 / 0.427 (± 0.072)	- / 0

Table 5. Constitutive properties for basal, prismatic, and pyramidal slip as well as extension twin at ambient temperature obtained from the literature: typical critical resolved shear stresses (CRSS or τ_{CRSS}), their ratios with respect to the basal slip, shear moduli, and Burgers vectors [12, 66, 78].

Deformation Mode	Basal	Prismatic	Pyramidal	Extension Twin
CRSS (MPa)	10	20	40	7
Ratio of CRSSs	1	2	4	0.7
Shear Modulus (GPa)	16.4	16.6	17.6	-
Burgers vector (nm)	0.321	0.321	0.612	-

Table 6. Calculated σ_o for different deformation modes for the five loading orientations, θ , based on the measured Schmid factors (Table 4) and CRSSs from literature (Table 5). The minimum value in each orientation is shown in bold indicating the dominant plastic deformation mode at yielding.

Angle, θ (Deg.)	Calculated σ_o (MPa)				Dominant Plastic Deformation Mode at Yielding
	Basal	Prismatic	Pyramidal	Extension Twin	
0	61.4	588.2	85.7	15.1	Twin
22.5	30.7	625.0	85.1	16.5	Twin (+Basal slip)
45	21.4	175.4	131.1	26.6	Basal slip (+Twin)
67.5	26.7	72.5	99.0	93.3	Basal slip
90	128.2	46.7	93.7	-	Prismatic slip

Table 7. Hall-Petch parameters of various deformation modes for AZ31B Mg alloy.

Def. Mode	τ_o (MPa)	$k\tau$ (MPa $\cdot$ mm ^{1/2})	Calculated $k\tau$ (MPa $\cdot$ mm ^{1/2})
Basal Slip	9.9	2.61	2.89
Prismatic Slip	17.6	5.54	4.88
Pyramidal Slip	-	-	9.86
Ex. Twin	5.7	3.48	-